ISSN 2070--2515

Food and Agriculture Organization of the United Nations

World Health Organization

FAO PLANT PRODUCTION AND PROTECTION PAPER

165

# Pesticide residues in food 2000

## Joint FAO/WHO Meeting on Pesticide Residues

# EVALUATIONS

# 2000

# PART I - RESIDUES

Rome, 2001

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** Evaluated within the periodic review programme of the Codex Committee on Pesticide Residues

**2000 JOINT MEETING OF THE FAO PANEL OF EXPERTS ON PESTICIDE RESIDUES IN FOOD AND THE ENVIRONMENT AND THE WHO CORE ASSESSMENT GROUP**

Geneva, 20-29 September 2000

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**ABBREVIATIONS WHICH MAY BE USED**
(Well-known abbreviations in general use are not included)

| | |
|---|---|
| Ache | acetylcholinesterase |
| acute RfD | acute reference dose |
| ADI | acceptable daily intake |
| AFI(D) | alkali flame-ionization (detector) |
| ai | active ingredient |
| ALAT | alanine aminotransferase |
| AR | applied radioactivity |
| ASAT | aspartate aminotransferase |
| | |
| BBA | Biologische Bundesanstalt für Land- und Forstwirtschaft |
| bw | body weight |
| BOD | biological oxygen demand |
| | |
| CA | Chemical Abstracts |
| CAS | Chemical Abstracts Services |
| CCN | Codex Classification Number (this may refer to classification number for compounds or for commodities) |
| CCPR | Codex Committee on Pesticide Residues |
| CCRVDF | Codex Committee on Residue of Veterinary Drugs in Food |
| ChE | cholinesterase |
| CI | chemical ionization |
| CNS | central nervous system |
| cv | coefficient of variation |
| CXL | Codex Maximum Residue Limit (Codex MRL). See MRL. |
| | |
| DFG | Deutsche Forschungsgemeinschaft |
| DL | racemic (optical configuration, a mixture of dextro- and laevo-) |
| DP | dustable powder |
| DS | powder for dry seed treatment |
| DT-50 | time for 50% decomposition (i.e. half-life) |
| DT-90 | time for 90% decomposition |
| | |
| EBDC | ethylenebis(dithiocarbamate) |
| EC | (1) emulsifiable concentrate<br>(2) electron-capture [chromatographic detector] |
| ECD | electron-capture detector |
| EI | electron-impact |
| EMDI | estimated maximum daily intake |
| EPA | Environmental Protection Agency |
| ERL | extraneous residue limit |
| ETU | ethylenethiourea |
| | |
| $F_1$ | filial generation, first |
| $F_2$ | filial generation, second |
| f.p. | freezing point |

| | |
|---|---|
| FAO | Food and Agriculture Organization of the United Nations |
| FDA | Food and Drug Administration |
| FI(D) | flame-ionization (detector) |
| FP(D) | flame-photometric (detector) |
| | |
| g (not gm) | gram |
| µg | microgram |
| GAP | good agricultural practice(s) |
| GC-MS | gas chromatography - mass spectrometry |
| GC-MSD | gas chromatography with mass-selective detection |
| G.I. | gastrointestinal |
| GL | guideline level |
| GLC | gas-liquid chromatography |
| GLP | good laboratory practice |
| GPC | gel-permeation chromatograph or chromatography |
| GSH | glutathione |
| | |
| h (not hr) | hour(s) |
| ha | hectare |
| Hb | haemoglobin |
| hl | hectolitre |
| HPLC | high-performance liquid chromatography |
| HPLC-MS | high-performance liquid chromatography - mass spectrometry |
| HR | highest residue in the edible portion of a commodity found in the trials used to estimate a maximum residue level in the commodity |
| HR-P | residue in a processed commodity calculated by multiplying the HR of the raw agricultural commodity by the corresponding processing factor |
| | |
| i.d. | internal diameter |
| IEDI | international estimated daily intake |
| IESTI | international estimate of short-term intake |
| i.m. | intramuscular |
| i.p. | intraperitoneal |
| IPCS | International Programme on Chemical Safety |
| IR | infrared |
| IRDC | International Research and Development Corporation (Mattawan, Michigan, USA) |
| i.v. | intravenous |
| | |
| JMPR | Joint FAO/WHO Meeting on Pesticide Residues (Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group |
| | |
| LC | liquid chromatography |
| $LC_{50}$ | lethal concentration, 50% |
| LC-MS | liquid chromatography - mass spectrometry |
| $LD_{50}$ | lethal dose, median |
| LOAEL | lowest observed adverse effect level |
| LOD | limit of determination (see also "*" at the end of the Table) |
| LSC | liquid scintillation counting or counter |

| | |
|---|---|
| M | molar |
| µm | micrometre (micron) |
| MFO | mixed function oxidase |
| min (no stop) | minute(s) |
| MLD | minimum lethal dose |
| mo (not mth.) | month(s) |
| MRL | Maximum Residue Limit. MRLs include draft MRLs and Codex MRLs (CXLs). The MRLs recommended by the JMPR on the basis of its estimates of maximum residue levels enter the Codex procedure as draft MRLs. They become Codex MRLs when they have passed through the procedure and have been adopted by the Codex Alimentarius Commission. |
| MS | mass spectrometry |
| MSD | mass-selective detection or detector |
| MTD | maximum tolerated dose |
| n (not *n*) | normal (defining isomeric configuration) |
| NCI | National Cancer Institute (USA) |
| NMR | nuclear magnetic resonance |
| NOAEL | no-observed-adverse-effect level |
| NOEL | no-observed-effect level |
| NP(D) | nitrogen-phosphorus (detector) |
| NTE | neuropathy target esterase |
| OECD | Organization for Economic Co-operation and Development |
| OP | organophosphorus pesticide |
| PHI | pre-harvest interval |
| ppm | parts per million. (Used only with reference to the concentration of a pesticide in a diet. In all other contexts the terms mg/kg or mg/l are used). |
| PT | prothrombin time |
| PTDI | provisional tolerable daily intake. (See 1994 report, Section 2.3, for explanation) |
| PTT | partial thromboplastin time |
| PTU | propylenethiourea |
| RAC | raw agricultural commodity |
| RBC | red blood cell |
| r.d. | relative density. (Formerly called specific gravity) |
| RfD | reference dose (usually in the phrase 'acute reference dose') |
| s.c. | subcutaneous |
| SC | suspension concentrate (= flowable concentrate) |
| SD | standard deviation |
| SE | standard error |
| SG | water-soluble granule |
| SL | soluble concentrate |

| | |
|---|---|
| SP | water-soluble powder |
| sp./spp. | species (only after a generic name) |
| SPE | solid-phase extraction |
| STMR | supervised trials median residue |
| | |
| t | tonne (metric ton) |
| $T_3$ | tri-iodothyronine |
| $T_4$ | thyroxine |
| TADI | Temporary Acceptable Daily Intake |
| *tert* | tertiary (in a chemical name) |
| TLC | thin-layer chromatography |
| TMDI | theoretical maximum daily intake |
| TMRL | Temporary Maximum Residue Limit |
| TRR | total radioactive residue |
| TSH | thyroid-stimulating hormone (thyrotropin) |
| | |
| UDMH | 1,1-dimethylhydrazine (unsymmetrical dimethylhydrazine) |
| USEPA | United States Environmental Protection Agency |
| USFDA | United States Food and Drug Administration |
| UV | ultraviolet |
| | |
| WG | water-dispersible granule |
| WHO | World Health Organization |
| WP | wettable powder |
| | |
| < | less than |
| ≤ | less than or equal to |
| > | greater than |
| ≥ | greater than or equal to |
| * | at or about the limit of determination |

## USE OF JMPR REPORTS AND EVALUATIONS BY REGISTRATION AUTHORITIES

The summaries and evaluations contained in this book are, in most cases, based on unpublished proprietary data submitted for the purpose of the JMPR assessment. A registration authority should not grant a registration on the basis of an evaluation unless it has first received authorization for such use from the owner who submitted the data for JMPR review or has received the data on which the summaries are based, either from the owner of the data or from a second party that has obtained permission from the owner of the data for this purpose.

# INTRODUCTION

The report of the Joint Meeting of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Core Assessment Group (JMPR), held in Geneva, 20-29 September 2000, contains a summary of the evaluations of residues in foods of the various pesticides considered as well as information on the general principles followed by the Meeting. The present document contains summaries of the residues data considered, together with the recommendations made.

The Evaluations are issued in two parts:

Part I: Residues (by FAO)
Part II: Toxicology (by WHO)

For those interested in both aspects of pesticide evaluation, not only both parts but also the reports containing summaries of residues and toxicological considerations will be available. Special attention is drawn to Annex I containing updated ADIs, PTDI, MRLs, ERLs, STMR and HR levels which also appears in full as part of the report of the Meeting.

Some of the compounds considered at the Meeting have been previously evaluated and reported on in earlier publications. In general only new information is summarized in the relevant monographs and reference is made to previously published evaluations, which should also be consulted. In the case of older compounds which are re-evaluated as part of the periodic review programme of the CCPR a review of all available data, including data which may have previously been submitted, is carried out. Compounds evaluated for the first time are indicated by a single asterisk and those evaluated in the CCPR periodic review programme by double asterisks in the Table of Contents.

The name of the compound appearing as the title of each monograph is followed by its Codex Classification Number in parentheses.

References to previous Reports and Evaluations of Joint Meetings are listed in Annex II.

**Acknowledgements**

The monographs in these Evaluations were prepared by the following participants in the 2000 JMPR for the FAO Panel of Experts on Pesticide Residues in Food and the Environment:
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# ABAMECTIN (177)

## EXPLANATION

Abamectin was placed on the agenda of the 2000 JMPR at the request of the CCPR at its thirty-second session in 2000 for reconsideration of the residue definition for animal commodities with a view to removing avermectin $B_{1b}$ and 8,9-*Z*-avermectin $B_{1b}$ from the definition for residues in animal commodities.

## APPRAISAL

Abamectin is used both as a pesticide and as an anthelminthic drug in animals. It was evaluated toxicologically by the Meeting in 1992 and 1994, and an ADI of 0-0.0002 mg/kg bw was established on the basis of a NOAEL of 0.12 mg/kg bw per day for toxicity in pups in a study of reproductive toxicity in rats. A safety factor of 500 was applied because of concern about the teratogenicity of the 8,9-*Z*-isomer, a photodegradation product that has been detected as a residue in plants. MRLs were recommended for commodities of cattle (edible offal, 0.05 mg/kg; meat, 0.01* mg/kg; milk, 0.005 mg/kg) and goats (edible offal, 0.1 mg/kg; meat, 0.01* mg/kg; milk, 0.005 mg/kg). The residues were defined in 1992 as the sum of avermectin $B_{1a}$, 8,9-*Z*-avermectin $B_{1a}$, and avermectin $B_{1b}$. The 1992 Meeting was unaware of the existence of a photoisomer of avermectin $B_{1b}$.

In the analytical method, avermectin $B_{1a}$ is derivatized to a fluorescent compound for analysis by HPLC. As avermectin $B_{1a}$ and its 8,9-*Z*-isomer form an identical fluorescent derivative, they are not separated or distinguished in the analysis for residues. The method gives a single HPLC peak for the sum of avermectin $B_{1a}$ and its 8,9-*Z*-isomer. Avermectin $B_{1b}$ and its photoisomer behave analogously to produce a second, but smaller, peak. In 1992, the LOQ (known at that time as the limit of determination) for meat was 0.01 mg/kg.

The use of abamectin as a veterinary drug was considered by the Joint FAO/WHO Expert Committee on Food Additives (JECFA) at its forty-fifth meeting, in 1995. The Committee had intended to rely on the toxicological evaluation of the 1994 JMPR, but, on reviewing the data on the residues found when abamectin is used as a veterinary drug, it learned that the 8,9-*Z*-isomer is not present in animal tissues and that the major residue in cattle liver and fat is avermectin $B_{1a}$, accounting for 50% of the total residue 7 days after treatment. 24-Hydroxymethyl-$B_{1a}$ is a major part of a polar residue fraction that accounts for 22% of the total residue in liver 14 days after treatment and a major part of a fraction that accounts for 51% of the total residue in fat 21 days after treatment. Avermectin $B_{1b}$, which represents about 5% of the total residue in liver and fat 7 days after treatment, is a minor residue. Therefore, JECFA concluded that avermectin $B_{1a}$ is a suitable marker residue and recommended that consultations be held between representatives of JECFA and JMPR. At that meeting, held in September 1995, it was recognized that consideration should be given to establishing different ADIs for abamectin when it is used as a pesticide and as a veterinary drug.

As a consequence, the 1995 JMPR agreed that the ADI of 0-0.0002 mg/kg bw was not appropriate for abamectin residues that do not contain the 8,9-*Z*-isomer, and it allocated an ADI of 0-

0.001 mg/kg bw to abamectin, on the basis of a NOAEL of 0.12 mg/kg bw per day observed in the study of reproductive toxicity in rats, with a safety factor of 100.

JECFA at its forty-seventh meeting, in 1996, established MRLs of 0.1 mg/kg for cattle liver and fat, and 0.05 mg/kg for cattle kidney. The marker residue was avermectin $B_{1a}$. A validated analytical method (HPLC with fluorescence detection) for avermectin $B_{1a}$ is available. The residue defined for estimation of dietary intake is the total residue.

New toxicological data were evaluated by the JMPR in 1997. In view of the finding that rats are hypersusceptible postnatally, the Meeting agreed to reduce the interspecies safety factor in establishing an ADI. A safety factor of 50 was therefore applied to the NOAEL of 0.12 mg/kg bw in the multigeneration study in rats, which is corroborated by a NOEL of 0.24 mg/kg bw per day in a 1-year study in dogs, with a safety factor of 100. It was considered appropriate to establish a single ADI for abamectin and its 8,9-*Z*-isomer, since the potential teratogenicity of the isomer had been satisfactorily explained. An ADI of 0-0.002 mg/kg bw was established for the sum of abamectin and its 8,9-*Z*-isomer. In order to harmonize the MRLs with those proposed by JECFA, the Meeting suggested that the MRLs be modified as follows: cattle edible offal to be removed; cattle liver and cattle fat, 0.1 mg/kg; and cattle kidney, 0.05 mg/kg. These numerical values are the same as those proposed by JECFA, but the residue definition differs by including avermectin $B_{1a}$, avermectin $B_{1b}$, 8,9-*Z*-avermectin $B_{1a}$, and 8,9-*Z*-avermectin $B_{1b}$. The proposed MRLs for goat commodities remained unchanged.

The Meeting noted the large margin of safety between the estimated dietary intake of residues resulting from the accepted uses and the newly established ADI and concluded that the residue definition is not a matter of concern to public health but a question of analytical method and national enforcement measures for compliance with MRLs.

At a meeting to facilitate harmonization between JECFA and JMPR, held on 1-2 February 1999, the different definitions of the CCPR and the CCRVDF for residues of abamectin were noted, and the CCRVDF and JECFA were asked to consider expanding their residue definitions to include other isomers, such as the photodegradation isomer of avermectin $B_{1a}$.

JECFA at its fifty-fourth meeting, in February 2000, carefully considered the toxicological and chemical assessments of abamectin made by JMPR and concluded that inclusion of the photodegradation isomer in the residue definition would not be consistent with the assessment by JECFA of abamectin as a veterinary drug. Inclusion of other possible residues of abamectin will be reviewed at a future JECFA meeting.

The CCPR at its thirty-second session, in May 2000, noted that the CCRVDF at its 12th meeting had retained all draft MRLs at step 7 because of the different residue definitions for animal products proposed by JECFA and JMPR. The CCPR therefore decided to refer the question of the residue definition for animal products to the 2000 JMPR with the suggestion that avermectin $B_{1b}$ and 8,9-*Z*-avermectin $B_{1b}$ be removed from the definition for the sake of harmonization. In the meantime, the CCPR returned all draft MRLs for animal commodities to step 6 and advanced all draft MRLs for plant commodities to Step 8.

The following Table summarizes the residue definitions and MRLs currently recommended by the JMPR and JECFA.

| Commodity | Residue definition | | |
|---|---|---|---|
| | JMPR 1992 | JMPR 1997 | JECFA 1996 |
| | Sum of avermectin $B_{1a}$, $B_{1b}$ and 8,9-Z-avermectin $B_{1a}$ | Sum of avermectin $B_{1a}$, $B_{1b}$, 8,9-Z-avermectin $B_{1a}$ and $B_{1b}$ | Avermectin $B_{1a}$ |
| | MRL, mg/kg | MRL, mg/kg | MRL, mg/kg |
| Cattle edible offal | 0.1 | W | -- |
| Cattle meat | 0.01* | | -- |
| Cattle milk | 0.005 | | -- |
| Cattle liver | | 0.1 | 0.1 |
| Cattle fat | | 0.1 | 0.1 |
| Cattle kidney | | 0.05 | 0.05 |
| Goat edible offal | 0.1 | | -- |
| Goat meat | 0.01* | | -- |
| Goat milk | 0.005 | | -- |

W: the previous recommendation is withdrawn

## RECOMMENDATIONS

The Meeting recommended harmonizing the JMPR and JECFA proposals for abamectin in animal commodities by maintaining the recommended MRLs of 0.1 mg/kg for cattle liver and fat and 0.05 mg/kg for kidney, and simplifying the definition of the residue in animal commodities to include only avermectin $B_{1a}$ and 8,9-*Z*-avermectin $B_{1a}$ (i.e. eliminating avermectin $B_{1b}$ and 8,9-*Z*-avermectin $B_{1b}$).

Definition of the residue for compliance with MRLs for animal commodities: sum of avermectin $B_{1a}$ and 8,9-*Z*-avermectin $B_{1a}$.

Definition of the residue for compliance with MRLs for plant commodities and for estimating dietary intake: sum of avermectin $B_{1a}$, avermectin $B_{1b,}$ 8,9-*Z*-avermectin $B_{1a}$ and 8-9-Z-avermectin $B_{1b}$.

| CCN | Commodity | Recommended MRL, mg/kg[1] |
|---|---|---|
| MF 0812 | Cattle fat | 0.1 |
| MO 1289 | Cattle, kidney | 0.05 |
| MO 1281 | Cattle, liver | 0.1 |
| MM 0812 | Cattle meat | 0.01* |
| ML 0812 | Cattle milk | 0.005 |
| MO 0814 | Goat, Edible offal of | 0.1 |
| MM 0814 | Goat meat | 0.01* |
| ML 0814 | Goat milk | 0.005 |

[1] The numerical values remain unchanged but with a revised residue definition

The present Meeting recommended that the JMPR and JECFA continue to meet to harmonize recommendations for MRLs, definitions of residues and marker residues, and commodity descriptions for substances used both as pesticides and veterinary drugs.

# CAPTAN (007)

## EXPLANATION

Captan has been evaluated several times since the initial evaluation in 1965. It was identified as a candidate for re-evaluation by the 1995 CCPR (ALINORM 95/24 A) and scheduled for periodic review by the 1998 JMPR at the 1997 CCPR (ALINORM 97/24 A). The 28th (1996) Session of the CCPR returned all the proposed draft MRLs to Step 3, pending the evaluation of new data by the 1997 JMPR. The 1997 JMPR recommended MRLs of 20 mg/kg for apple replacing 10 mg/kg, 40 mg/kg for cherries replacing 20 mg/kg, 25 mg/kg for grapes replacing 20 mg/kg, and 30 mg/kg for strawberry replacing 15 mg/kg. Owing to the shifting of the rights from one company to another, it was requested that the re-evaluation of captan be deferred until the 2000 JMPR, and it is now evaluated in the Periodic Review Programme.

Data to support the existing CXLs (for apple, pear, cherries, peach, plums, nectarine, blueberries, strawberry, grapes, tomato) and other critical data required for the estimation of maximum residue levels have been provided by the manufacturers.

Relevant data have also been provided in support of new residue limits for oranges, lemons, grapefruit, apricot, raspberries, cucumber, melons, potato and almonds.

The governments of Australia, Germany, Poland and Thailand have submitted information on national GAP and/or residue data.

## IDENTITY

ISO common name: captan

Chemical name:

IUPAC: *N*-(trichloromethylthio)cyclohex-4-ene-1,2-dicarboximide

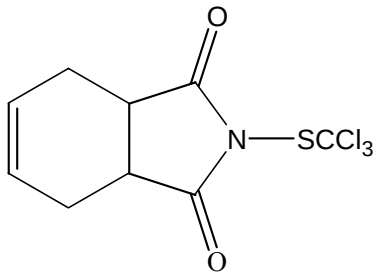
CAS: 3a,4,7,7a-tetrahydro-2-[(trichloromethyl)thio]-1*H*-isoindole-1,3(2*H*)-dione

CAS No.: 133-06-2

CIPAC No.: 40

Synonyms/trade names: SR-406, Merpan, Vanicide 89, Orthocide

Structural formula:




Molecular formula: $C_9H_8Cl_3NO_2S$

Molecular weight: 300.59

**Physical and chemical properties**

Pure active ingredient

Appearance: colourless crystals, white solid (Wollerton and Husband, 1995a)

Melting point: 172°C (Wollerton and Husband, 1995a)

Relative density: 1.71 (Wollerton and Husband, 1995a)

Vapour pressure: $4.2\times10^{-6}$ Pa at 20°C (Wollerton and Husband, 1995a)

Henry's law constant (Wollerton and Husband, 1995a):

$3\times10^{-4}$ Pa.m$^3$.mol$^{-1}$ in purified water
$3\times10^{-4}$ Pa.m$^3$.mol$^{-1}$ in purified pH 5 buffered water
$2\times10^{-4}$ Pa.m$^3$.mol$^{-1}$ in purified pH 7 buffered water
Partition coefficient (n-octanol/water) log $P_{ow}$ 2.5 (Wollerton and Husband, 1995a)

Solubility at 20°C, mg/kg solvent (Wollerton and Husband, 1995a,b)

| | |
|---|---|
| Purified water | 4.9 |
| Water buffered pH 5 | 4.8 |
| Water buffered pH 7 | 5.2 |

Hydrolysis (Yaron, 1985)

| | |
|---|---|
| Half-lives at 25°C: | 12 hours at pH 4 |
| | 2.6 hours at pH 7 |
| | too fast to measure at pH 9 |
| Half-lives at 40°C: | 1.7 hours at pH 4 |
| | 0.51 hours at pH 7 |
| | too fast to measure at pH 9 |

Photolysis

Not accurately measured owing to extensive hydrolysis in aqueous solution. The half-life, assuming a quantum yield of 1 and using experimental extinction values, was estimated to be about 880 days (Moffat, 1994).

Technical material (Wollerton and Husband, 1995b)

Appearance: cream solid

Melting point: 162-172°C

Solubility at 20°C (g/kg solvent):

| | |
|---|---|
| Hexane | 0.04 |
| Octan-1-ol | 1 |
| Methanol | 4 |
| Xylenes | 9 |
| Ethyl acetate | 25 |
| Acetonitrile | 31 |

| | |
|---|---|
| Acetone | 38 |
| 1,2-dichloroethane | 41 |

**Formulations**

The following types of formulation are available: suspension concentrate (SC), wettable powder (WP), dustable powder (DP) and water dispersible granule (WG).

## METABOLISM AND ENVIRONMENTAL FATE

### Animal metabolism

Metabolism studies on lactating goats and laying hens with [*trichloromethyl*-$^{14}$C]captan, [*cyclohexene*-$^{14}$C]captan and [*carbonyl*-$^{14}$C]captan were made available to the Meeting.

Abbreviations are used for some of the metabolites:

| | |
|---|---|
| THPI: | 1,2,3,6-tetrahydrophthalimide |
| 3-OH THPI | *cis/trans*-3-hydroxycyclohex-4-ene-1,2-dicarboximide |
| 5-OH THPI | *cis/trans*-5-hydroxycyclohex-3-ene-1,2-dicarboximide |
| 4,5-diOH HHPI | 4,5-dihydroxycyclohexane-1,2-dicarboximide |
| THPAM | *cis/trans*-6-carbamoylcyclohex-3-ene-1-carboxylic acid (*cis/trans*-1,2,3,6-tetrahydrophthalamic acid) |
| THPI epoxide | 7-oxabicyclo[4.1.0]heptane-3,4-dicarboximide |

Goats. Powell and Skidmore (1993) in a material balance study dosed a lactating goat orally by gelatine capsule with [*trichloromethyl*-$^{14}$C]captan, once daily for two consecutive days (equivalent to 55 ppm in the feed). The recovery of $^{14}$C for the period to 16 hours after the last dose was 78% with gastrointestinal tract contents and expired $^{14}CO_2$ accounting for most of the administered radioactivity at 20 and 43% respectively. Urine, faeces and milk accounted for 8.0, 4.6 and 0.2% of the radioactivity while tissue radioactivity was less than 1.4%. The low recovery of the administered radioactivity is likely to be due to the bacterial conversion of $CO_2$ to methane in the rumen. Samples were stored frozen and analysed within 6 weeks of slaughter.

Powell *et al.* (1994) dosed two lactating goats with [*trichloromethyl*-$^{14}$C]captan in gelatine capsules at a rate equivalent to 50 ppm in the diet for 7 consecutive days. Milk and excreta were collected throughout the dosing period and the animals slaughtered 16 hours after the final dose. 36% of the radioactive dose was recovered in the excreta, including cage washings. Total radioactive residues (TRR) in milk reached a plateau by day 4-5 of dosing at 2.2 mg captan/kg. The TRR in tissues were 0.46 mg captan equivalents/kg in muscle (fore- and hind-quarter), 0.11 mg/kg in subcutaneous fat, 0.09 mg/kg in perirenal fat, 0.06 mg/kg in peritoneal fat, 0.47 mg/kg in the diaphragm, 4.4 mg/kg in kidney and 4.7 mg/kg in liver. In the milk and tissues there was extensive incorporation of the radioactivity into natural products. These included fatty acids, cholesterol, glycerol, lactose, glucose, creatine, lactic acid, choline chloride, phosphatidylcholine and amino acids. (Samples were analysed within 6 months of dosing, and again 2 years later. The chemical profiles in liver and milk were the same for 6 months and 2 years storage).

A lactating goat was dosed orally with [*trichloromethyl*-$^{14}$C]captan at 1.4 mg/kg bw/day by capsule three times daily (3 × 0.47 mg/kg bw/day) for 3 days with an additional dose on the fourth day (Duan, 1988). Radioactivity in faeces, urine and milk collected until slaughter 4 hours after the last dose accounted for 21, 6.0 and 1.5% respectively of the administered dose. Tissue radioactivity accounted for 1.3% of the administered dose with highest residues in liver (2.0 mg/kg as captan) and kidney (1.6 mg/kg as captan). Characterization of tissue and milk radioactivity by extraction into solvents demonstrated that most of the radioactivity was incorporated into natural products. A metabolite identified in milk, liver, kidney and urine was thiazolidine-2-thione-4-carboxylic acid (TTC). TTC represented 0.4, 2.2, 4 and 24% of the TRR in milk, liver, kidney and urine respectively.

Radioactive residues were measured in the tissues, milk and excreta of a lactating goat dosed orally by capsule three times daily with [*carbonyl*-$^{14}$C]captan (Cheng, 1980). The daily dose was 1.4 mg/kg bw/day (equivalent to 50 ppm captan in the diet). The last dose (10th) was given after the morning milking on the 4th day and the goat slaughtered four hours later.

The major metabolites in urine, determined by derivatization with diazomethane and/or bis(trimethylsilyl)acetamide and characterization by GC-MS, were 3-OH THPI, 5-OH THPI and 4,5-diOH-HHPI. Milk samples were separated into fat, protein (casein), lactose and aqueous acetone-soluble fractions with >90% of the TRR located in the aqueous acetone fraction. The $^{14}$C in milk was not incorporated into natural products. 76-87% of the TRR was extracted from the tissues with methanol/water, indicating polar metabolites. The major metabolites were tentatively identified by chromatography by comparing relative retention times with authentic standards.

Table 1. Identity and distribution of metabolites in milk and tissues from a goat dosed with [*carbonyl*-$^{14}$C]captan equivalent to 50 ppm in the diet for 3 days (Cheng, 1980).

| | Milk | Liver | Kidney | Muscle (fq) | Muscle (hq) | Fat (peri) | Fat (subc) |
|---|---|---|---|---|---|---|---|
| TRR (mg/kg as captan) | 0.13-0.63 | 1.7 | 2.3 | 0.65 | 0.66 | 0.35 | 0.36 |
| Metabolite | % of TRR | | | | | | |
| THPI | 2.3-9.9 | 4.6 | 2.7 | 6.0 | 7.2 | 33 | 6.8 |
| THPI epoxide | 2.4-9.1 | 1.6 | 2.0 | 4.6 | 5.1 | 2.1 | 3.3 |
| 3-OH THPI | 27-33 | 10 | 17 | 36 | 44 | 16 | 28 |
| 5-OH THPI | 16-26 | 8.8 | 7.9 | 10.1 | 8.4 | 4.6 | 10 |
| 4,5-diOH HHPI | 4.7-8.2 | 2.1 | 2.0 | 0.0 | 0.0 | 4.8 | 8.6 |

fq = forequarter; hq = hindquarter; peri = peritoneal; subc = subcutaneous

Minor metabolites detected in urine and milk included THPAM as well as hydroxylated THPAM derivatives (3-OH THPAM, 5-OH THPAM and 4,5-diOH HHPAM).

Hens. A single hen was dosed with [*trichloromethyl*-$^{14}$C]captan for two days at a rate equivalent to 10 ppm in the diet and killed 16 hours after the last dose (Mathis and Skidmore, 1993). The total radioactivity recovered in excreta, expired-air traps and the carcase was 88% with the majority either expired as $^{14}CO_2$ (33%) or eliminated in excreta (50%). Only 2.8% of the dose was recovered in the carcase with a further 1.3% recovered from cage washings and 1.3% from the contents of the gastrointestinal tract. Samples were analysed within 2 months.

Mathis and Skidmore (1994) dosed 9 hens orally with [*trichloromethyl*-$^{14}$C]captan at a nominal rate equivalent to 10 ppm in the diet for 10 consecutive days. Radioactive residue in eggs reached a plateau by day 8 of dosing. Mean TRRs were 0.07, 0.04, 0.06, 0.68, 0.30, 0.40 and 0.07 mg/kg as captan for skin/subcutaneous fat, peritoneal fat, muscle (leg + breast), kidney, liver, egg yolk and egg white respectively. Much of the radioactive residue was incorporated into natural products. No single metabolite was present above 0.016 mg/kg in any sample of tissue or eggs. Samples were analysed within 2 months. Liver reanalysed after 23 months showed qualitatively the same results.

A group of 10 laying hens was dosed orally, by capsule, with [cyclohexene-$^{14}$C]captan at a nominal rate equivalent to 10 ppm in the diet for 10 consecutive days (Renwick and Skidmore, 1993). Birds were slaughtered 16 hours after the final dose. Radioactive residues in eggs reached a plateau at 2-4 days after the start of dosing. Samples were analysed after 2-4 months. Liver reanalysed after 7 months storage showed qualitatively the same results.

Radioactivity in the excreta collected over the 10-day dosing period accounted for 86% of the administered dose, and in the tissues and eggs 3.2% of the administered dose. Identification of the

residues in the excreta, tissues and eggs was by TLC and co-chromatography with authentic compounds.

Table 2. Identity and distribution of metabolites in tissues, eggs and excreta from hens dosed with [*cyclohexene*-$^{14}$C]captan equivalent to 10 ppm in the diet for 10 days (Renwick and Skidmore, 1993).

| TRR, mg/kg as captan | Excreta | Liver | Peritoneal fat | Muscle | Egg yolk day 9 | Egg white day 9 |
|---|---|---|---|---|---|---|
| | | 0.66 | 0.13 | 0.55-0.63 | 0.83 | 0.84 |
| Metabolite | %Total radioactive residue | | | | | |
| THPI | 8.9 | 64 | 77 | 52 | 74 | 61 |
| 3-OH THPI | 23 | 5.2 | 2.1 | 8.9 | 6.0 | 6.6 |
| 5-OH THPI | 10 | 1.3 | 0.5 | 1.5 | 1.3 | 1.6 |
| 4,5-diOH HHPI | 1.3 | 0.1 | 0.4 | ND | ND | ND |
| THPAM | 4.3 | ND | ND | 0.6 | ND | ND |
| THPI epoxide | 2.4 | ND | ND | 1.7 | 1.6 | ND |
| Total | 50 | 71 | 80 | 64 | 83 | 69 |

The metabolism of captan in goats and hens proceeds by cleavage of the N-S bond to form THPI and a derivative of the -$SCCl_3$ side chain. THPI and -$SCCl_3$ undergo further metabolism through independent pathways. The carbon of the side chain becomes incorporated into TTC and natural products. The cleavage partner THPI is oxidised to form THPI expoxide which is subsequently hydrolysed to form 4,5-diOH HHPI, or hydroxylated at the cyclohexene ring to form 3-OH and 5-OH THPI. The hydrolysis of THPI and its hydroxylated derivatives results in the formation of the corresponding THPAM derivatives. The proposed metabolic pathway in livestock is shown in Figure 1.

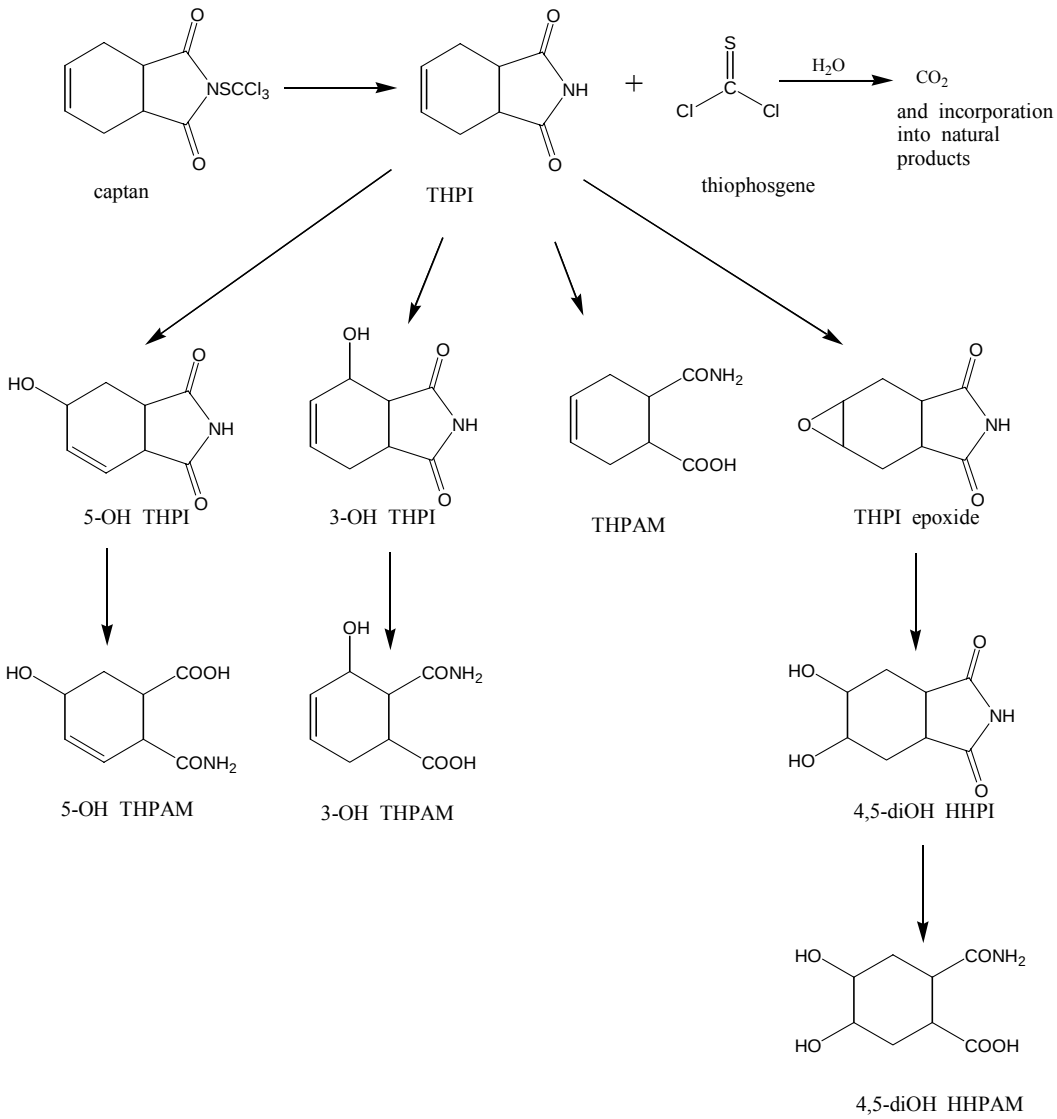



Figure 1. Proposed metabolic pathways of captan in livestock.

**Plant metabolism**

Metabolism studies on tomatoes, lettuce and apples were made available to the Meeting. Both [*trichloromethyl*-$^{14}$C]captan and [*cyclohexene*-$^{14}$C]captan were used to trace the fate of different parts of the captan molecule in tomatoes and lettuce while [*carbonyl*-$^{14}$C]captan was used in the apple study.

Lettuce and tomato plants were treated four times with [*trichloromethyl*-$^{14}$C]captan (Chen, 1988a) or [*cyclohexene*-$^{14}$C]captan (Chen, 1988b) at about 4.5 kg ai/ha (4.0 lb/acre) at 7 day intervals. The plants were harvested 3 hours after the last spray and separated into leaves, stems, roots and, in the case of tomatoes, fruit. Tomatoes were washed with acetone, blended and centrifuged to separate the juice from the pulp. Tomato pulp and other macerated plant samples were extracted with acetone, methanol and methanol/water. Tomato juice was extracted with ethyl acetate. Metabolites were characterized by TLC, HPLC and MS. Most of the radioactivity in the plants was found in the leaves and fruit of tomatoes and leaves of lettuce.

Table 3. Distribution and characterization of $^{14}C$ in acetone extracts of tomatoes and lettuce treated four times with [*trichloromethyl*-$^{14}C$]captan at 4.5 kg ai/ha (Chen, 1988a).

| Characterization | Tomato leaves and stem[1] | | Tomato fruit | | Lettuce leaves | |
|---|---|---|---|---|---|---|
| | % of TRR | Residue, mg/kg[2] | % of TRR | Residue, mg/kg[2] | % of TRR | Residue, mg/kg[2] |
| Captan | 81 | 93 | 77 | 5.3 | 76 | 52 |
| Captan epoxide | 0.3 | 0.34 | 0.2 | 0.01 | 0.3 | 0.21 |
| Other free metabolites | 6.9 | 7.9 | 9.5 | 0.66 | 5.2 | 3.6 |
| Polar and conjugates | 4.8 | 5.5 | 10 | 0.72 | 4.6 | 3.2 |
| Unextractable | 7.2 | 8.3 | 3.3 | 0.23 | 14 | 9.4 |

[1] Leaves constitute 8.0% and stems 7.0% of the total plant mass according to the reported distribution of radioactivity in leaves, stems, roots and tomatoes and on the TRR in these components. From the residues listed it appears that the plant material was 87% leaf matter
[2] Expressed as captan

Table 4. Distribution and characterization of $^{14}C$ in acetone extracts of tomatoes and lettuce treated four times at 4.5 kg ai/ha with [*cyclohexene*-$^{14}C$]captan (Chen, 1980b).

| Characterization | Tomato leaves and stem[1] | | Tomato fruit[2] | | Lettuce leaves | |
|---|---|---|---|---|---|---|
| | % of TRR | Residue, mg/kg[3] | % of TRR | Residue, mg/kg[3] | % of TRR | Residue, mg/kg[3] |
| Captan | 70 | 128 | 82 | 5.5 | 77 | 50 |
| Captan epoxide | 0.4 | 0.73 | 0.4 | 0.03 | 0.6 | 0.39 |
| THPI | 4.6 | 8.3 | 4.5 | 0.30 | 9.5 | 6.1 |
| Other free metabolites | 6.9 | 13 | 5.2 | 0.35 | 4.3 | 2.8 |
| Polar and conjugates | 8.9 | 16 | 7.5 | 0.50 | 4.5 | 2.9 |
| Unextractable | 8.8 | 16 | 0.9 | 0.06 | 3.0 | 1.9 |

[1] Leaves constitute 7.7% and stems 7.1% of the total plant mass according to the reported distribution of radioactivity in leaves, stems, roots and tomatoes and on the TRR. It appears plant material is 88% leaf matter.
[2] Calculated from the radioactivity in the acetone surface rinse, tomato juice and pulp using weight/volume ratios for whole fruit, pulp, juice and acetone rinse.
[3] Expressed as captan

When treated with [*cyclohexene*-$^{14}C$]captan, the unextractable residues accounted for less than 9% of the total radioactivity in all components except tomato pulp (not tabulated separately but included as a component of tomato fruit) in which unextractable radioactivity represented 42% of the total in the pulp. Fractionation of tomato pulp into carbohydrates, amino acids (proteins) and lignin fractions indicated that the radioactivity was distributed in all the sub-fractions with 71% associated with carbohydrates, 18% with amino acids and 3% with lignins.

With both labels most of the residue remained on the plant or fruit surface and was present as unmetabolized captan. In the plant captan was metabolized to form THPI which underwent further transformation. No specific steps were taken to avoid the hydrolysis of captan during the extraction procedure and the studies may overestimate the THPI contents.

DeBaun *et al.* (1975) treated branches of field-grown Golden Delicious apple trees with [*carbonyl*-$^{14}C$]captan at a rate equivalent to 0.12 kg ai/hl (1 lb ai/100 gal). The apples were washed with acetone and peeled to determine surface residues and residues in peel and pulp (peeled fruit). The residues were extracted with acetone, the extracts concentrated by rotary evaporation at 40°C, and the aqueous residue suspended in a saturated $(NH_4)_2SO_4$ solution. This was acidified and extracted with ethyl acetate before and after acidification. Residues in the combined ethyl acetate extracts were determined by TLC. Most of the residue was located on the surface of the fruit and was present as captan. THPI and THPAM represented 3.3-7.6% and 0.4-2.4% of the radioactive residue respectively. Radioactive residues in apple peel and pulp were low with captan accounting for 46 and 15% of the radioactive residue respectively. The main metabolites in peel and pulp were THPI and THPAM. The

extraction procedure may lead to the hydrolysis of captan and captan expoxide, leading to an overestimation of the THPI and THPI epoxide contents.

Table 5. Distribution of radioactivity in apples after application of [*carbonyl*-$^{14}$C]captan at 0.12 kg ai/hl (DeBaun *et al.*, 1975).

| No. of sprays (interval, days) | PHI, days | % of $^{14}$C | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Fruit | | | | | Foliage | |
| | | Surface wash | Peel extract | Peel residue | Pulp extract | Pulp residue | Extract | Residue |
| 1 | 0.13 | 96 | 3.3 | 0.9 | 0.8 | 0.2 | 99 | 1.5 |
| 1 | 20 | 90 | 4.5 | 2.6 | 2.7 | 0.7 | 85 | 15 |
| 2 (30) | 20 | 81 | 4.8 | 4.4 | 7.7 | 1.7 | 72 | 29 |
| 3 (30, 31) | 20 | 64 | 9.3 | 4.4 | 17 | 4.9 | 84 | 16 |

Table 6. Characterization of $^{14}$C radioactivity in apples after application of [*carbonyl*-$^{14}$C]captan at 0.12 kg ai/hl (DeBaun *et al.*, 1975).

| | % of TRR and (mg/kg as captan) | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Fruit surface wash | | | | Foliage extracts | | | |
| No. sprays | 1 | 1 | 2 | 3 | 1 | 1 | 2 | 3 |
| PHI, days | 0.13 | 20 | 20 | 20 | 0.13 | 20 | 20 | 20 |
| Compound | | | | | | | | |
| Captan | 78 (13) | 79 (14) | 68 (9.3) | 71 (4.3) | 84 | 74 | 67 | 71 |
| Captan epoxide | <1 (0.17) | <1 (0.19) | <1 (0.15) | <1 (0.06) | <1 | <1 | <1 | 1.1 |
| THPI | 7.6 (0.61) | 5.7 (0.50) | 6.1 (0.42) | 5.2 (0.16) | 5.4 | 4.4 | 3.6 | 3.3 |
| THPI epoxide | <1 (0.09) | <1 (0.10) | <1.2 (0.09) | <1.4 (0.05) | <1 | <1 | <1 | <1 |
| THPAM | 1.2 (0.11) | 0.4 (0.04) | 1.1 (0.09) | 1.3 (0.04) | 2.1 | 2.4 | 2.0 | 0.7 |
| | Peel | | | | Pulp | | | |
| Captan | 46 (1.6) | 37 (1.4) | 25 (0.74) | 21 (0.72) | 15 (0.02) | 6.1 (0.03) | 3.0 (0.03) | 2.8 (0.03) |
| Captan epoxide | 0.1 (<0.01) | 0.5 (0.02) | 0.2 (<0.01) | 1.0 (0.04) | 3.7 (0.006) | 5.0 (0.03) | 2.8 (0.03) | 1.0 (0.01) |
| THPI | 33 (0.59) | 17 (0.31) | 16 (0.24) | 15 (0.26) | 48 (0.04) | 29 (0.07) | 18 (0.09) | 13 (0.07) |
| THPI epoxide | 0.5 (0.01) | 2.3 (0.05) | 1.3 (0.02) | 1.1 (0.02) | 2.0 (0.002) | 0.5 (0.001) | 5.6 (0.03) | 3.3 (0.02) |
| THPAM | 8.1 (0.16) | 12 (0.24) | 12 (0.20) | 12 (0.24) | 0.5 (0.004) | 2.0 (0.005) | 2.4 (0.013) | 1.1 (0.007) |
| Total | 87 | 68 | 54 | 49 | 69 | 42 | 32 | 22 |

In apples, tomatoes and lettuce most of the residue was present on the surface of the leaves and fruit, mainly as unchanged captan. Metabolism in the plants includes cleavage of the thio-indole bond to form THPI and derivatives of the -$SCCl_3$ side chain. The carbon of the side chain is incorporated into natural products, and THPI is further metabolized to form THPAM. Captan is also oxidised to captan epoxide which undergoes hydrolysis to form THPI expoxide. The proposed metabolism in plants is shown in Figure 2.

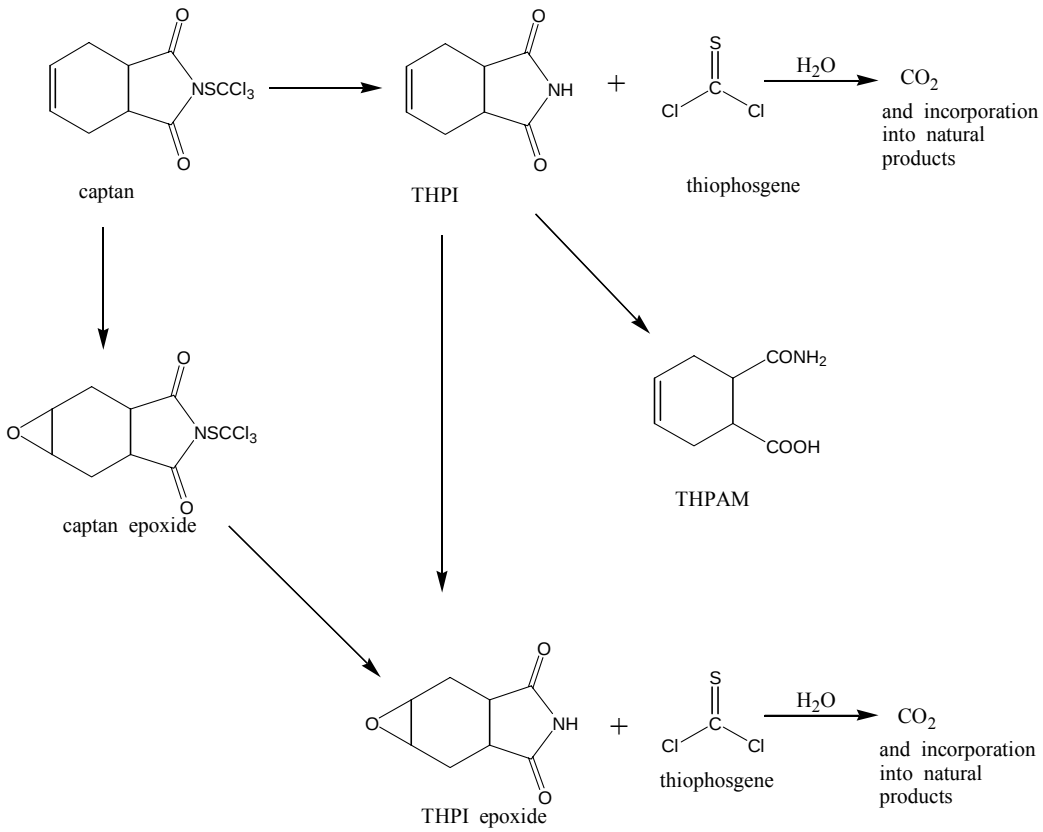



Figure 2 Proposed metabolic pathways of captan in plants.

## Environmental fate in soil

Residues in rotational crops

In a confined rotational crop study (Ewing *et al.*, 1990), beets, lettuce and wheat were planted in treated soil. Nine plastic-lined wooden boxes (76 × 91 cm × 67 cm deep) were filled with Huntington series sandy loam soil (73% sand, 18% silt, 9% clay; pH 7.4, 2% organic matter, from Fayette County, Kentucky) to a depth of 61 cm. Three boxes were treated with [*cyclohexene*-$^{14}$C]captan at 3.9 ± 2.2 kg ai/ha and three with [*trichloromethyl*-$^{14}$C]captan at 6.7 ± 4.1 kg ai/ha. The variability in the application rates was due to the inhomogeneity of the application solution (acetone/water). The remaining three boxes served as controls. Beet, lettuce and wheat seeds were planted in the boxes after fallow periods of 34 and 88 days after treatment (DAT) and the boxes maintained in a greenhouse at 10-29°C.

Samples were collected as immature trimmings and at maturity. Immature plants were analysed as whole plants. Mature beet and lettuce plants were separated into leaves and roots while mature wheat plants were separated into grain, chaff, straw and roots. The $^{14}$C in soil and plant components was determined by combustion and LSC. Crop samples were extracted twice with acetone followed by methanol, methanol/water and finally 1M HCl in methanol. The extracts were analysed by HPLC with a C-18 column and UV detection. Radio-chromatograms were constructed from LSC analysis of column fractions. Identification of compounds was by TLC on silica gel $F_{254}$ plates and co-chromatography with authentic standards. The extraction procedure may result in the hydrolysis of some of the captan to THPI.

Only low levels of radioactivity were found in the crops at harvest. Radioactive residues in immature plants were highest in lettuce and beet. The radioactive residues in crops planted 88 days after application to soil were lower than those in crops planted 34 days after application.

Table 7. Radioactive residues in rotational crops after application of [*cyclohexene*-$^{14}$C]captan (Ewing *et al.*, 1990).

| Planting DAT | Harvest DAT | $^{14}$C, mg/kg as captan | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Lettuce | | Beet | | | Wheat | | | |
| | | Immature | Mature leaf | Immature | Mature | | Immature | Mature | | |
| | | | | | Leaf | Root | | Straw | Chaff | Grain |
| 34 | 43 | 1.2 | | 1.2 | | | 0.39 | | | |
| 34 | 54 | 0.78/1.8 | | 0.31/0.40 | | | 0.26/0.51 | | | |
| 34 | 61 | 0.18/0.35 | | 0.06/0.16 | | | 0.14/0.23 | | | |
| 34 | 75 | | 0.02 | | | | 0.08/0.03 | | | |
| 34 | 126 | | | 0.07 | | | | | | |
| 34 | 131 | | | | 0.02 | 0.03 | | | | |
| 34 | 186 | | | | | | | 0.09 | 0.05 | 0.04 |
| 88 | 105 | 0.06 | | 0.05 | | | 0.03 | | | |
| 88 | 116 | 0.12 | | 0.07 | | | 0.03 | | | |
| 88 | 138 | 0.05 | | | | | | | | |
| 88 | 158 | | 0.01 | 0.01 | | | | | | |
| 88 | 186 | | | | 0.005 | 0.02 | | | | |
| 88 | 224 | | | | | | | 0.02 | 0.01 | 0.01 |

Table 8. Radioactive residues in rotational crops after application of [*trichloromethyl*-$^{14}$C]captan (Ewing *et al.*, 1990).

| Planting DAT | Harvest DAT | $^{14}$C, mg/kg as captan | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Lettuce | | Beet | | | Wheat | | | |
| | | Immature | Mature leaf | Immature | Mature | | Immature | Mature | | |
| | | | | | Leaf | Root | | Straw | Chaff | Grain |
| 34 | 43 | 0.11 | | 0.09 | | | 0.08 | | | |
| 34 | 54 | 0.07/0.15 | | 0.02/0.03 | | | 0.02/0.04 | | | |
| 34 | 61 | 0.01/0.04 | | 0.01/0.02 | | | 0.02/0.02 | | | |
| 34 | 75 | | 0.005 | | | | 0.02/0.06 | | | |
| 34 | 126 | | | 0.02 | | | | | | |
| 34 | 131 | | | | 0.003 | 0.007 | | | | |
| 34 | 186 | | | | | | | 0.05 | 0.03 | 0.02 |
| 88 | 105 | 0.03 | | 0.01 | | | 0.009 | | | |
| 88 | 116 | 0.04 | | 0.02 | | | 0.01 | | | |
| 88 | 138 | 0.008 | | | | | | | | |
| 88 | 158 | | 0.01 | 0.01 | | | | | | |
| 88 | 186 | | | | 0.003 | 0.01 | | | | |
| 88 | 224 | | | | | | | 0.03 | 0.03 | 0.02 |

The radioactive residues in immature crops were characterized by HPLC and comparison with authentic standards. The major metabolites were 4,5-diOH HHPI, THPAM and THPI.

Table 9. Characterization of [$^{14}$C] in acetone extracts at 43 DAT by HPLC.

| Compound | $^{14}$C residue, mg/kg as captan | | | | | |
|---|---|---|---|---|---|---|
| | Lettuce | | Beets | | Wheat | |
| | Ring label | $CCl_3$ label | Ring label | $CCl_3$ label | Ring label | $CCl_3$ label |
| Captan | <0.003 | <0.003 | <0.003 | <0.003 | <0.003 | <0.003 |
| THPI | 0.012 | <0.003 | 0.095 | <0.003 | 0.088 | <0.003 |
| THPI epoxide | <0.003 | <0.003 | <0.003 | <0.003 | <0.003 | <0.003 |
| THPAM | 0.012 | <0.003 | 0.044 | <0.003 | 0.027 | <0.003 |
| 4,5-diOH HHPI | 0.532 | <0.003 | 0.174 | <0.003 | 0.072 | <0.003 |
| THPAL | <0.003 | <0.003 | <0.003 | <0.003 | <0.003 | <0.003 |
| Other | 0.017 | 0.041 | 0.351 | 0.034 | 0.048 | 0.050 |
| Total | 0.573 | 0.041 | 0.665 | 0.034 | 0.234 | 0.050 |

Soil degradation

Aerobic. The aerobic degradation of [*trichloromethyl*-$^{14}$C]captan on Visalia sandy loam (sand 55%, silt 33%, clay 12%; pH 7.7; organic matter 0.7%; CEC 9.1 meq/100 g) and Greenville sandy loam (sand 58%, silt 30%, clay 12%; pH 7.2; organic matter 1.2%; CEC 7.7 meq/100 g) at 25°C in the dark has been studied by Diaz and Lay (1992) and Pack and Verrips (1988a) respectively. Captan was applied at a rate equivalent to 8.8 mg/kg to the Visalia soil and 4.6-6.1 mg/kg to the Greenville soil and incubated for periods up to 30 days. In both Visalia sandy loam and Greenville sandy loam the major product was $^{14}CO_2$, which accounted for 50-59% of the applied radioactivity after 1-3 days and reached 81-90% after 28-30 days incubation. The other compound detected was thiocarbonic acid which accounted for at most 1.1% of the applied radioactivity. Approximately 10% of the radioactivity was bound to the soil and could not be extracted. Volatile organic compounds accounted for about 0.2% of the applied radioactivity after 28 days incubation. The calculated degradation half-life for [*trichloromethyl*-$^{14}$C]captan was about 1-3 days at 25°C.

The aerobic degradation of [*carbonyl*-$^{14}$C]captan was studied on Oakley loamy sand soil (sand 67%, silt 17%, clay 16%; pH 6.8; organic matter 1.8%) at 25 °C using an initial captan concentration of 5.3 mg/kg (Pack, 1974). Approximately 20% of the applied radioactivity was evolved as $^{14}CO_2$ during the first 7 days of incubation and this accounted for 82% of the applied radioactivity at 37 days and 94% at 244 days. Captan represented less than 1% of the applied radioactivity after 7 days or more. Major compounds identified were THPI and THPAM. Residues of THPI reached a maximum of 66% of the applied radioactivity at 7 days, declining thereafter to less than 7% at 37 days and less than 0.2% at 224 days. THPAM reached a maximum of 17% of the applied radioactivity at 14 days, declining to less than 0.2% after 244 days incubation. Minor compounds identified were THPI epoxide, 4,5-diOH HHPI and THPAL.

Anaerobic. Lay (1992) studied the anaerobic degradation of [trichloromethyl-$^{14}$C]captan in both non-sterile and sterile Visalia sandy loam (sand 55%, silt 33%, clay 12%; pH 7.3; organic matter 0.7%; CEC 9.1 meq/100 g. In non-sterile soil, approximately 100% of the applied radioactivity was recovered as $^{14}CO_2$ after 14 days incubation. Residues of captan accounted for less than 0.1% of the radioactivity after 90 days. Bound residues accounted for 16-25% of the applied dose at all sample times. The variable recovery of radioactivity, 62-165%, was explained by problems in achieving homogeneity in soil moisture and applied captan. The recovery of radioactivity as $^{14}CO_2$ was lower for sterile soil, reaching 75% after 90 days incubation.

Pack and Verrips (1988b) investigated the anaerobic degradation of [*trichloromethyl*-$^{14}$C]captan in a Greenville sandy loam (sand 58%, silt 30%, clay 12%; pH 7.2; organic matter 1.2%; CEC 7.7 meq/100 g) at 25 °C. The amount of $^{14}CO_2$ evolved increased from 44-48% of the applied radioactivity after 1 day of incubation to 83-88% after 30 days. Bound residues accounted for 14-20% of the applied radioactivity.

The anaerobic soil degradation of [*carbonyl*-$^{14}$C]captan was studied on Oakley loamy sand soil (sand 85%, silt 6%, clay 9%; pH 7.3; organic matter 1.4%; CEC 7.5 meq/100 g) at 25°C in the dark with an initial captan concentration of 6.2 mg/kg (Pack, 1979). No captan was detected after 7 days of incubation. Compounds identified were THPI, THCY, THPAM and THPAL. The cyano acid THCY was not observed under aerobic conditions. Less than 9% of the applied radioactivity was converted to $^{14}CO_2$ over a period of 9 months. Extracts from the anaerobic soil that contained THCY were added to Oakley soil under aerobic conditions. The THCY was rapidly degraded with 98% loss occurring within 7 days.

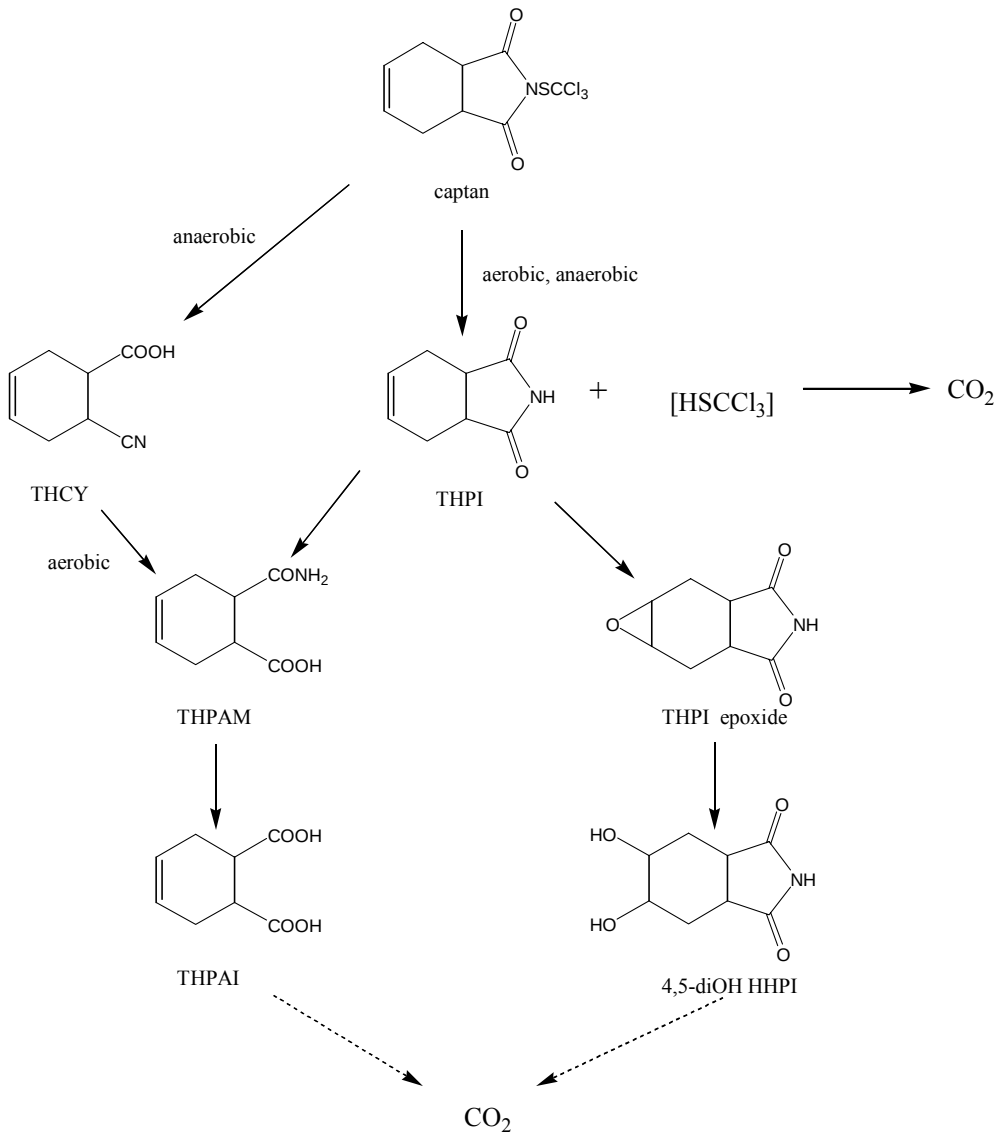



Figure 3. Proposed metabolic pathways of captan in soil.

Photolysis

The half-life of [*trichloromethyl*-$^{14}$C]captan in Greenville sandy loam soil exposed to sunlight was 15 days while the half-life for the dark control soil was 20 days (Ruzo *et al.*, 1988a). The photochemical half-life was estimated to be 54 days. Most of the radioactivity was accounted for as captan and carbon dioxide, with the remainder present as bound residues and unidentified compounds. In irradiated samples on day 21 captan accounted for 36-40%, $^{14}CO_2$ for 49-51%, bound residues for 7%, acid-released residues for 2-5% and unidentified residues for 1.4% of the applied radioactivity. In dark control soil samples captan accounted for 50-56% and $^{14}CO_2$ for 33-40% of the applied radioactivity.

Ruzo *et al.* (1988b) studied the natural sunlight photolysis of [*cyclohexene*-$^{14}$C]captan at 25°C on the surface of Greenville sandy loam soil treated at 4.48 kg ai/ha. The half-lives for photolysis were 130 and 236 hours for the light and dark conditions respectively, resulting in an estimated photochemical half-life of 287 hours. Products present at >3% were identified by HPLC and TLC. The major components formed under both light and dark conditions were THPI, THPI epoxide, THCY, THPAM and THPAL.

Degradation of major products

The aerobic degradation of the captan metabolite THPI was studied on Speyer 2.1 sand (sand 90%, silt 6%, clay 4%; pH 6.3; organic matter 0.7), Speyer 2.2 loamy sand (sand 85%, silt 8%, clay 7%; pH 6.0; organic matter 3.7%) and Hyde farm sandy loam (sand 59%, silt 24%, clay 17%; pH 7.1; organic matter 3.2%) at 20°C and 40% moisture holding capacity over a period of 50 days (Freeman and Jones, 1993a). The THPI degradation half-life, calculated using the Timme and Frehse model (Timme *et al.*, 1986), was 5-6 days for loamy sand and sandy loam, and 20 days for sand. A degradation half-life of 1 day was also observed for sand during the period 33-40 days after treatment. This was thought to be due to the microbial content of the soil. The half-lives for the aerobic degradation of *cis*-tetrahydrophthalamic acid studied under the same conditions as THPI were 4-5 days for loamy sand and sandy loam and 7 days for sand (Gallagher and Jones, 1993a).

Field studies

Soil residues from the confined rotational crop study described earlier were characterized by TLC, HPLC and MS (Ewing *et al.*, 1990). The total radioactive residues in the 0-7.5 cm soil layer decreased from 2.9 and 2.7 mg/kg (as captan) immediately after application to 0.71 and 0.17 mg/kg as captan respectively for the [*cyclohexene*-$^{14}$C]captan and [*trichloromethyl*-$^{14}$C]captan after 224 days. Significantly lower levels of radioactivity were found in the 7.5-15 cm soil layer which were thought to result from contamination during sampling. Residues of captan were detected 34 days after application at levels of 0.05-0.20 mg/kg but not at later samplings. Characterization of the radioactivity in the soil after application of [*trichloromethyl*-$^{14}$C]captan showed that most was associated with carbon dioxide which was retained in the soil in the form of carbonates. In the case of [*cyclohexene*-$^{14}$C]captan a variety of degradation products was formed.

Table 10. Distribution of radioactivity in extracts of the 0-7.5 cm soil layer after application of [*cyclohexene*-$^{14}$C]captan (Ewing *et al.*, 1990).

| DAT | $^{14}$C as captan, mg/kg soil | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Ethyl acetate | | | | | | | Water | NaOH | Unextracted |
| | Total | M1 | M2 | M3 | M4 | M5 | Bound | | | |
| 34 | 0.54 | 0.05 | 0.22 | 0.19 | 0.03 | 0.05 | 0.14 | 0.09 | 0.26 | 0.13 |
| 88 | 0.16 | - | 0.03 | 0.004 | 0.02 | 0.11 | 0.31 | 0.09 | 0.44 | 0.29 |
| 224 | 0.06 | - | 0.02 | 0.005 | 0.04 | 0.004 | 0.06 | 0.08 | 0.38 | 0.31 |

M1 = captan; M2 = THPI; M3 = THPAM; M4 = 4,5-diOH HHPI: M5 = THPI epoxide

The major products at 34 and 88 DAT were THPI and THPAM, while THPI epoxide was the main product after 224 days.

Captan was applied as eight sprays at 4.5 kg ai/ha and at intervals of 7 days to an apple orchard in New York, USA (Jones, 1988a). The soil (sand 81%, silt 14%, clay 5%; pH 5.5; organic matter 2.0%; CEC 3.7 meq/100 g) was sampled to a depth of 30 cm and analysed for captan and THPI. Air temperatures were 4-14°C during the study while rainfall totalled 34 mm. Residues of captan at 0-7.5 cm soil depth were 2.1 mg/kg on the same day as the last application, decreasing to 0.53 mg/kg by 7 days and 0.02 mg/kg by 59 days after the last spray. Residues of THPI at the same sample times were 0.78, 0.12 and less than 0.01 mg/kg respectively. Using the assumption of 1st order kinetics the degradation half-life for captan in soil at 0-7.5 cm depth was calculated to be 14 days. No captan was detected at depths of 15 cm or more. At soil depths of 15-30 cm THPI was detected only on the day of the last treatment and 1 day later at levels of 0.03 and 0.02 mg/kg.

After eight applications of captan at 3.4 kg ai/ha to strawberries in California, USA, the half-life in loamy sand (sand 78%, silt 13%, clay 9%; pH 7.1; organic matter 0.5%; CEC 3.8 meq/100 g) was determined to be 2.5 days (Jones, 1988b). The total rainfall and irrigation during the study was

170 mm. Captan was detected in soil at 7.5-30 cm depth on the day of the 6th application but only at low levels, 0.03-0.05 mg/kg. No captan was detected below 30 cm and no THPI below 7.5 cm.

Six sprays of captan were applied to grapes grown in Oregon, USA, in silt loam (sand 32%, silt 54%, clay 14%; pH 5.6; organic matter 3.8%; CEC 5.7 meq/100 g) at 4.5 kg ai/ha (Jones, 1988c). The air temperatures during the course of the study were 14-26°C while the rainfall during the sampling period of 6 months was 125 mm. Neither captan nor THPI were detected in soil below 7.5 cm. The degradation half-life of captan in soil at 0-7.5 cm depth was calculated to be 24 days.

The degradation of captan was also studied in clay soil (sand 14%, silt 26%, clay 60%; pH 7.8; organic matter 2.4%; CEC 8.2 meq/100 g) in which cantaloupes were grown in Texas, USA (Jones, 1988d). Captan was found in the 7.5-15 cm soil depth samples on the days immediately after the 3rd, 4th, 5th and 6th applications at 2.2 kg ai/ha at levels between 0.01 and 0.38 mg/kg. With the exception of the sample collected the day after the 6th spray, THPI was found in the same 7.5-15 cm samples at levels 0.03-0.08 mg/kg. Captan and THPI were not detected in soil samples of 7.5-15 cm depth at any interval after the last application. The degradation half-life of captan at 0-7.5 cm soil depth was calculated to be 4 days.

Tomatoes grown in loam soil (sand 36%, silt 40%, clay 24%; pH 6.9; organic matter 1.5%; CEC 14.5 meq/100 g) in California and clay soil (sand 14%, silt 26%, clay 60%; pH 7.8; organic matter 2.4%; CEC 8.2 meq/100 g) in Florida, USA, were treated 4 times with captan at 4.5 g ai/ha (Jones, 1988e,f). The degradation half-life of captan, assuming 1st order kinetics, was 6 days for the California site and 3 days for the Florida site.

Table 11. Field studies on the dissipation of captan in soil in the USA (Jones, 1988a-f).

| Crop/soil/location/year | Application, kg ai/ha /no. of sprays | Sample depth (cm) | DALA | Residues, mg/kg | |
|---|---|---|---|---|---|
| | | | | Captan | THPI |
| Apples/loamy sand/New York 1988 | 4.5/8 | 0-7.5 | 0 | 2.1 | 0.78 |
| | | | 1 | 1.8 | 0.54 |
| | | | 7 | 0.53 | 0.12 |
| | | | 14 | 0.84 | 0.10 |
| | | | 29 | 0.36 | 0.02 |
| | | | 59 | 0.02 | <0.01 |
| | | | 120 | <0.01 | <0.01 |
| | | 7.5-15 | 0 | 0.01 | 0.06 |
| | | | 1 | 0.06 | 0.10 |
| | | | 7 | <0.01 | <0.01 |
| | | 15-30 | 0 | <0.01 | 0.03 |
| | | | 1 | <0.01 | 0.02 |
| | | | 7 | <0.01 | <0.01 |
| Cantaloupe/clay/Texas/1987 | 2.2/7 | 0-7.5 | 0 | 0.16 | 0.74 |
| | | | 1 | 0.02 | 0.59 |
| | | | 7 | 0.01 | 0.20 |
| | | | 14 | <0.01 | <0.01 |
| Tomatoes/loam/California/1987 | 4.5/4 | 0-7.5 | 0 | 0.19 | 0.61 |
| | | | 1 | 0.24 | 1.2 |
| | | | 7 | 0.04 | 1.5 |
| | | | 14 | 0.01 | 1.3 |
| | | | 28 | <0.01 | 0.30 |
| | | | 59 | <0.01 | 0.46 |
| | | | 120 | <0.01 | <0.01 |
| | | 7.5-15 | 0 | <0.01 | <0.01 |
| | | | 1 | <0.01 | 0.01 |
| | | | 7 | <0.01 | <0.01 |
| Tomatoes/sand/Florida/1987 | 4.5/4 | 0-7.5 | 0 | 5.0 | 0.82 |
| | | | 1 | 3.7 | 1.1 |
| | | | 7 | 1.3 | 0.86 |
| | | | 14 | 0.14 | 0.20 |

| Crop/soil/location/year | Application, kg ai/ha /no. of sprays | Sample depth (cm) | DALA | Residues, mg/kg | |
|---|---|---|---|---|---|
| | | | | Captan | THPI |
| | | | 28 | <0.01 | 0.03 |
| | | | 91 | <0.01 | <0.01 |
| Strawberries/loamy sand/California/1987 | 3.4/8 | 0-7.5 | 0 | 0.22 | 0.20 |
| | | | 1 | 0.22 | 0.25 |
| | | | 7 | 0.06 | 0.12 |
| | | | 14 | <0.01 | <0.01 |
| Grapes/silt loam/Oregon/1987 | 2.2/6 | 0-7.5 | 0 | 2.2 | 0.44 |
| | | | 1 | 2.0 | 0.45 |
| | | | 7 | 0.44 | 0.46 |
| | | | 14 | 0.17 | 0.35 |
| | | | 28 | 0.49 | 0.57 |
| | | | 63 | 0.05 | 0.19 |
| | | | 119 | 0.05 | 0.18 |
| | | | 184 | <0.01 | 0.02 |

DALA: days after last application

Adsorption/desorption

Adsorption/desorption experiments with soil/water systems are not applicable to captan owing to its rapid hydrolysis (Spillner, 1988). The degradation of captan in soil-water mixtures was found to be pH-dependent, being most rapid at the highest pH studied, pH 7. The only degradation product detected was THPI. The presence of soil in the test solutions resulted in an increased rate of degradation.

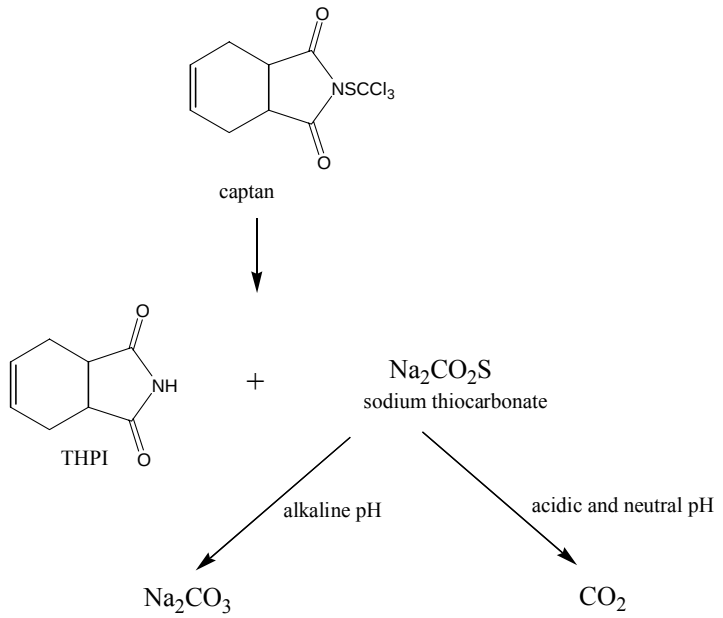



Figure 4. Proposed pathway of captan hydrolysis.

The adsorption/desorption properties of two major degradation products of captan, THPI and THPAM, were studied in six pre-sterilised soils (Rowe and Lane, 1983). Three of the soils were characterized as high pH (sandy loam pH 7.7%, 5.3 organic matter; sandy loam pH 8.1, 3.2% organic matter; loamy sand pH 7.9, 3.1% organic matter) and three as being low pH (sand pH 5.7, 0.8% organic matter; silty clay loam pH 5.0, 2.5% organic matter; sandy loam pH 4.7, 2.7% organic matter). Five rates of application were used for $^{14}C$-labelled THPI and THPAM.

THPI was weakly adsorbed by each of the soils with a clear relationship observed between THPI absorbed and % organic matter. The average adsorption coefficients (Kd) ranged from 0.04 for the soil with lowest % organic matter to 0.24 for the soil with the highest % organic matter while the Freundlich adsorption coefficients (K') ranged from 0.01 to 0.17. The coefficients corrected for the organic matter contents ranged from 7.6 to 13 for Kd and 2.2 to 11 for K'. Desorption was not completely reversible with a 2-3 fold increase observed in Kd values between adsorption and desorption.

THPAM was also weakly adsorbed by soil. The Kd values increased with decreasing soil pH ranging from 0.10 for the high pH sandy loam (pH 8.1) to 1.1 for the low pH sandy loam (pH 4.7). K' values showed a similar relationship with pH, ranging from 0.14 for the high pH soil to 1.2 for the low pH soil. When corrected for organic matter contents the range of Kd values was 3.8 to 110 while for K' the range was 4.5 to 100. As with THPI the desorption of THPAM was not entirely reversible with average 2-5 fold increases in Kd.

Mobility

The mobility of aged captan residues was studied in sandy loam, sand and loamy sand. $^{14}$C-ring-labelled captan was incubated in the soils in the dark at 20°C under aerobic conditions and at 40% moisture holding capacity for 30 days (Verity *et al.*, 1995). Samples were collected at intervals of 0, 1, 7, 14 and 30 days incubation. After 30 days incubation with sandy loam 21% of the radioactivity was associated with captan, 28% was converted to $^{14}CO_2$ and 30% remained unextracted. Captan constituted 59% of the radioactive residue in sand with 12% converted to $^{14}CO_2$ and 6% unextracted. In loamy sand, 51% of the radioactivity was due to captan, 21% to $^{14}CO_2$ and 11% was unextracted. THPI was the major product in the three soils representing 5, 11 and 6% of the radioactive residue in sandy loam, sand and loamy sand respectively.

The aged soils were placed on the top of duplicate 30 cm columns of the corresponding soils. The columns were leached with the equivalent of 200 mm of rain over a period of 48 hours and the soils in the columns were then analysed. The 0-5 cm layer of each of the soils contained 30-44% of the applied radioactivity. Of this radioactivity up to 25% was extractable with organic solvents. Captan was detected only in the first 0-5 cm of the sand and loamy sand. In material extractable with organic solvents, up to 12% of the radioactivity was from captan, 6% from THPI and 1% from THPAM.

No captan was found below the 0-5 cm layer and no individual compound accounted for more than 2% of the applied radioactivity. The radioactivity in the leachates from sandy loam, sand and loamy sand accounted for 1, 25 and 8% of the applied radioactivity respectively. The radioactivity in the leachates from sand and loamy sand were further characterized. THPI was the major compound, accounting for approximately 15 and 5% of the applied radioactivity from sand and loamy sand respectively. THPAM accounted for ≤3% of the applied radioactivity while no other compound accounted for more than 2%.

Biological degradation

The degradation of captan was studied in aqueous sediment systems (Travis and Simmons, 1993). The two systems studied were Old Basing (22% organic matter) and Virginia Water (5.4% organic matter). $^{14}$C-ring-labelled captan was applied at an initial concentration of 1.2 µg/ml, equivalent to an application at 3.6 g ai/ha being evenly distributed over a water body to a depth of 30 cm. Both non-sterile and sterile systems were used. Each water sediment system contained 10% dry matter in stream water.

After 24 hours incubation captan was not detected in any of the systems and was determined to have been rapidly hydrolysed to THPI. Three other products identified after 24 hours incubation

were THPAM, THPAL and THPI epoxide with similar levels observed in both the non-sterile systems. The levels of THPAM, THPAL and THPI epoxide reached maxima after 14 days incubation and represented up to 25% of the radioactivity for THPAM and 5 - 11% of the radioactivity for THPAL and THPI expoxide. Degradation in the non-sterile water-sediment systems was such that no THPI, THPAM, THPAL or THPI epoxide could be detected after 59 days incubation. By 90 days incubation about 50% of the ring-labelled captan had been mineralized to $^{14}CO_2$. Most of the remaining radioactivity was tightly bound to the sediment and not extracted by the solvents used.

Negligible amounts of $^{14}CO_2$ were evolved in the sterile systems. Most of the radioactivity present after 90 days incubation was associated with THPI, 64% in the Virginia Water and 36% in the Old Basing system.

Volatility (route and rate of degradation in air)

Air, 100 ml/min at 25°C, was passed over the surface of sandy soil (sand 92%, silt 6%, clay 2%; pH 7.2; organic matter 1.8%; CEC 3.6 meq/100 g) that had been treated with [*cyclohexene*-$^{14}$C]captan or [*trichloromethyl*-$^{14}$C]captan (Pack, 1987a). After 9 days, analysis of scrubber solutions revealed an average of 0.0003% and 0.4% of the radioactivity from [*cyclohexene*-$^{14}$C]captan and [*trichloromethyl*-$^{14}$C]captan respectively was trapped. There was no significant volatilization of captan from soil.

The proposed degradation pathways for captan in sediment/water systems are shown in Figure 5.

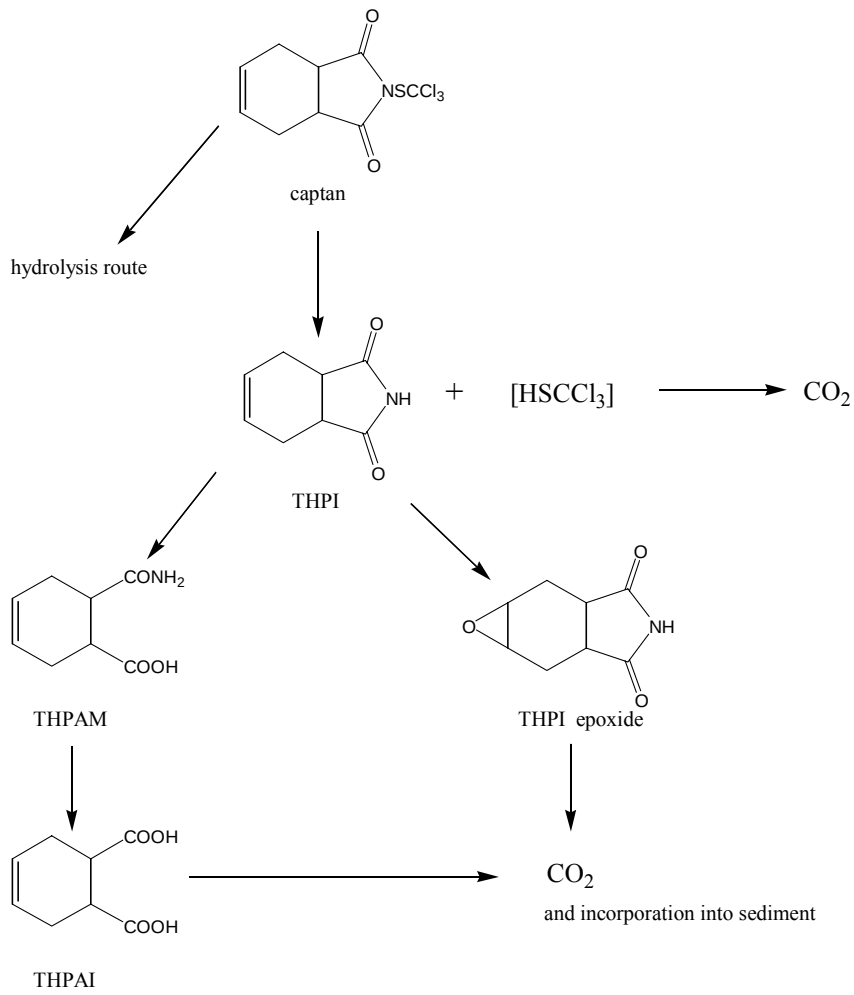



Figure 5. Degradation of captan in sediment/water systems.

## METHODS OF RESIDUE ANALYSIS

The determination of captan and THPI in non-oily crops (lettuce, tomatoes, melons, apples, squash, potatoes, grapes and strawberries) was described by Schlesinger (1992a). Samples are macerated with sodium sulfate, ethyl acetate and a small quantity of phosphoric acid. For captan, the filtered extract is evaporated to dryness and the residue dissolved in n-hexane before clean-up on a Florisil solid-phase extraction cartridge. The cartridge is eluted with 1% methanol in dichloromethane and the eluate evaporated to dryness before dissolving the residue in n-hexane for determination of captan by GLC with an ECD. For THPI, the filtered ethyl acetate extracts are partitioned with pH 11.5 aqueous buffer and the aqueous phase is treated with concentrated phosphoric acid before partitioning with dichloromethane which is evaporated to dryness. The residue is dissolved in ethyl acetate for determination of THPI by GLC with a TID. The limits of quantification are 0.02 mg/kg for captan and 0.1 mg/kg for THPI. The specificity of the method was tested by analysis of untreated crops. Captan and THPI were not detected above their respective limits of quantification. The presence of 25 common pesticides did not interfere with the analytical method. Recoveries from samples fortified with captan were 93-125% from lettuce at 0.02-20 mg/kg, 72-120% from tomatoes, 88-96% from melons and 92-125% from potatoes at 0.02-2.5 mg/kg, 76-110% from apples, 92-130% from grapes and 79-130% from strawberries at 0.02-10 mg/kg, and 88-110% from squash at 0.02-5 mg/kg. Recoveries from samples fortified with THPI at 0.1-2.0 mg/kg were 69-105% from lettuce, 74-82% from tomatoes, 70-114% from melons, 71-88% from potatoes, 69-86% from apples, 70-109% from grapes, 59-78% from strawberries and 87-120% from squash.

Captan and THPI were determined in crops and processed commodities by Iwata (1989). Samples are macerated with anhydrous sodium sulfate and ethyl acetate in the presence of phosphoric acid. For non-oily crops the filtered extract is washed with phosphoric acid. The ethyl acetate is dried over anhydrous sodium sulfate, the solvent removed and the residue dissolved in dichloromethane. For oily crops the filtered extract is evaporated and partitioned with acetonitrile and hexane, the solvent is removed and the residue taken up in dichloromethane. Clean-up of extracts of both crop types is on a nuchar/silica column. The captan is eluted with 5% ethyl acetate in dichloromethane while THPI is eluted with 20% acetone in dichloromethane. Quantification is by GLC with ECD. The limit of quantification is 0.05 mg/kg for both captan and THPI. Recoveries from apple samples fortified with captan at 0.05-100 mg/kg were 82-111% while those from samples fortified with THPI at 0.05-10 mg/kg were 71-115%.

Captan residues in liver, kidney, muscle, fat, eggs and milk are extracted by blending samples with acetone and phosphoric acid (Mende, 1997). The acetone in the filtered extract is removed by evaporation and the sample purified by passage through a chromatography column containing sodium sulfate and “Extrelut”. Further purification is by gel permeation chromatography. Muscle and liver samples require an additional clean-up on a silica gel solid-phase extraction cartridge. Quantification of captan residues is by GLC with an ECD. The limit of quantification is 0.005 mg/kg for milk, 0.02 mg/kg for kidney and fat and 0.03 mg/kg for muscle and liver. Mean recoveries from samples fortified at 0.005-0.5 mg/kg for milk, 0.02-0.5 mg/kg for kidney and fat and 0.03-0.5 mg/kg for liver and muscle were 93, 87, 94, 86 and 94% respectively.

THPI, *cis*- and *trans*-3-OH THPI, and *cis*- and *trans*-5-OH THPI were determined in bovine tissues and milk (Wiebe *et al.*, 1992). Samples are macerated with acetone, an aliquot of the extract diluted with ethyl acetate is filtered through anhydrous sodium sulfate and the solvent removed under a stream of nitrogen gas. The residue is dissolved in hexane and partitioned into acetonitrile and the solvent is evaporated. The residue is taken up in toluene/ethyl acetate and cleaned up on a silica column. The solvents are removed and the residue dissolved in acetonitrile for derivatization with *N*,*O*-bis(trimethylsilyl)trifluoroacetamide (BSTFA) and trimethylchlorosilane before quantification by GC-MS. The limit of quantification for each compound was 0.01 mg/kg in milk, liver, kidney, muscle and fat. Mean recoveries of the individual analytes were 84-95% for all substrates and fortification

levels studied (0.01-0.5 mg/kg in tissues and milk except THPI, *trans*-3-OH THPI and *trans*-5-OH THPI which were determined at 0.005-0.5 mg/kg in milk).

Determination of captan residues in the tissues and eggs of hens dosed with [*cyclohexene*-$^{14}$C]captan were in good agreement when carried out by measurement of radioactivity and by the method of Wiebe *et al.* (1992). No residues of *cis*-3-OH or *cis*-5-OH THPI were detected.

Table 12 Comparison of an analytical method suitable for enforcement with $^{14}$C measurement for the determination of captan metabolites in tissues and eggs of hens dosed at the equivalent of 10 ppm in the diet for 10 days with [*cyclohexene*-$^{14}$C]captan (Renwick and Skidmore, 1993).

| Sample | Residue, mg/kg as captan | | | | | |
|---|---|---|---|---|---|---|
| | THPI | | *trans*-3-OH THPI | | *trans*-5-OH THPI | |
| | GC/MS | $^{14}$C | GC/MS | $^{14}$C | GC/MS | $^{14}$C |
| Liver | 0.40 | 0.39 | 0.05 | 0.03 | <0.02 | ND |
| Peritoneal fat | 0.14 | 0.11 | <0.02 | 0.003 | <0.02 | 0.001 |
| Muscle | 0.26 | 0.29 | 0.05 | 0.05 | 0.04 | 0.01 |
| Eggs | 0.40 | 0.5 egg yolk<br>0.5 egg white | 0.05 | 0.04 egg yolk<br>0.05 egg white | <0.02 | ND egg yolk<br>ND egg white |

A method was provided for the determination of residues of captan and THPI in soil (Breault and Robinson, 1987). Extraction with acetone and acidic methanol was followed by selective partitioning of captan into hexane and THPI into dichloromethane. The captan extract was cleaned up on a Florisil column while clean-up of the THPI extract was by liquid-liquid partition at pH 11. Captan was determined by GLC with an ECD, THPI by GLC with an NPD. The limit of quantification was 0.02 mg/kg for both captan and THPI.

Captan was determined in buffered aqueous solutions by extraction of the residues by shaking with toluene and analysis by GLC with an NPD (Kleinschmidt, 1983).

As captan is rapidly hydrolysed in water to produce THPI, the analytical method described for potable water determined residues of THPI (Freeman and Jones, 1993b). THPI was sorbed from water onto a C-18 solid-phase extraction cartridge and eluted with ethyl acetate. The eluate was evaporated to dryness and reconstituted in a solution of citral in acetone. Residues were determined by GLC with mass-selective detection. The limit of quantification was 0.1 µg/l. The mean recovery for THPI over the concentration range 0.1-1.0 µg/l was 89%.

Jones and Freeman (1994) determined residues of captan in air, extracting the residues by passing air through an XAD-2 sorbent tube for 6 hours at a flow rate of 2 l/min (total volume 0.72 m$^3$). Captan residues were eluted with acetone and determined by GLC with an ECD. The limit of quantification was 0.06 µg/m$^3$. The overall mean recovery for captan over the concentration range 0.06-5.6 µg/m$^3$ was 92%.

**Stability of pesticide residues in stored analytical samples**

The stability of residues in a variety of crops and processed commodities during freezer storage was studied by McKay (1990a). Both samples fortified with captan and THPI and samples with field-incurred residues were analysed. Results were not corrected for procedural recoveries. The results were presented as the means of 2-4 replicates with the initial values reported as the unadjusted analytical results and not the fortification levels. The study was divided into three parts.

In the first part macerated samples of apple, cucumber, lettuce, spinach and strawberry with field-incurred residues were stored at -20°C for 14 months. The stability of the residues varied. Acceptable stability was observed in acidic samples such as apple and strawberry. Although captan

residues in spinach remained constant during freezer storage, the THPI residue level increased from 2.7 to 12 mg/kg. As captan is hydrolysed to THPI, the total residue of captan and THPI expressed in captan equivalents should not increase on storage. The total residue in spinach after 14 months storage was 55 mg/kg while the initial value was 37 mg/kg, indicating problems with the procedure. If the initial result is discarded, captan was stable in spinach samples for at least 11 months of freezer storage. There was a sharp decrease in the captan levels in cucumber and lettuce on storage for 3 months although the residue levels were stable thereafter.

Table 13. The stability of captan and THPI in macerated field-treated samples stored at -20°C (McKay, 1990a).

| Storage interval, months | Mean residues, mg/kg | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Apple | | Cucumber | | Lettuce | | Spinach | | Strawberry | |
| | Captan | THPI | Captan | THPI | Captan | THPI | Captan | THPI | Captan | THPI |
| 0 | 2.7 | 0.01 | 1.1 | 0.12 | 13 | 0.22 | 32 | 2.7 | 8.7 | 0.24 |
| 3 | 2.8 | 0.11 | 0.43 | 0.22 | 8.8 (4) | 0.43 (4) | 34 | 9.9 | 6.7 | 0.36 |
| 6 | 2.8 | 0.12 | 0.55 (5) | 0.28 (5) | 8.1 | 0.72 | 30 | 7.6 | 6.4 | 0.41 |
| 14 | 2.9 (13) | 0.13 (13) | 0.39 (13) | 0.34 (13) | 7.4 | 0.58 | 32 | 12 | 6.4 | 0.50 |

In the second and third parts of the study samples were obtained from local markets and producers, as items of commerce (apple juice, apple sauce, tomato sauce, raisin, fruit, nut and vegetable samples) and from processing plants (dry tomato and grape pomace). Soya beans and soya bean forage, sugar beet tops and wheat forage were obtained from crops that had not been treated with captan. In the second part of the experiment samples were fortified with a mixture of captan and THPI as well as captan and THPI separately and stored in glass bottles with polyethylene-lined lids in the dark at -20 ± 10 °C. The stability of cherry and tomato samples with field-incurred residues was also studied. As in the first part of the study, residues of captan were most stable in acidic samples such as apple juice and cherries and least stable in the more basic beet tops and corn grain.

Table 14. Residues of captan and THPI in macerated fortified and field-treated samples stored at -20°C (McKay, 1990a).

| Crop | Compound | Residue, mg/kg, uncorrected for recovery | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Storage period, months | | | | | | |
| | | 0 | 1 | 3 | 6 | 12 | 15 | 20 |
| Almond | Captan | 0.38 | <0.05 | <0.05 | <0.05 | | | |
| | THPI | 0.44 | 0.64 | 0.36 | 0.42 | | | |
| Apple juice | Captan | 0.41 | 0.41 | 0.58 | 0.48 | | 0.31 | |
| | THPI | 0.24 | 0.26 | 0.15 | 0.15 | | 0.28 | |
| Beet tops | Captan | 0.55 | 0.10 | 0.19 | 0.13 | | <0.05 | |
| | THPI | 0.69 | 0.24 | 0.32 | 0.35 | | 0.35 | |
| Cherry[1] | Captan | 21 | 25 | 19 | 12 | 16 | | |
| | THPI | 0.26 | 0.30 | 0.35 | 0.36 | 0.30 | | |
| Maize grain | Captan | 0.38 | 0.46 | 0.08 | 0.03 | | | |
| | THPI | 0.44 | 0.46 | 0.41 | 0.45 | | | |
| Potato tubers | Captan | 0.47 | 0.32 | 0.25 | 0.27 | | 0.14 | |
| | THPI | 0.17 | 0.14 | 0.23 | 0.24 | | 0.25 | |
| Soya bean forage | Captan | 0.38 | 0.38 | 0.35 | 0.22 | | 0.36 | |
| | THPI | 0.44 | 0.39 | 0.40 | 0.22 | | 0.38 | |
| Soya bean grain | Captan | 0.29 | 0.21 | 0.14 | 0.12 | | 0.04 | |
| | THPI | 0.24 | 0.21 | 0.32 | 0.21 | | 0.10 | |
| Tomato[1] | Captan | 0.88 | 0.40 | 0.30 | 0.28 | 0.14 | | 0.15 |
| | THPI | 0.09 | 0.22 | 0.25 | 0.35 | 0.34 | | 0.30 |

[1] Field-incurred residues

The third part of the experiment studied the effect of maceration on the stability of residues. Samples were fortified with captan or THPI and stored in glass bottles with polyethylene-lined lids in the dark at -20 ± 10 °C. Samples were stored whole, coarsely chopped, finely chopped or ground to

determine whether greater exposure to plant enzymes would accelerate the decomposition of residues. THPI was stable in all the samples. The total residue of captan and THPI, expressed as captan equivalents, was fairly constant. Residues of captan in almond samples fortified with captan were more stable in whole nuts than in coarse ground nuts.

Table 15. Residues of captan and THPI in whole, coarsely or finely chopped or ground and homogenised samples fortified with captan or THPI and stored at -20°C (McKay, 1990).

| Crop | Compound | Residue, mg/kg, uncorrected for recovery | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Storage period, months | | | | | | | | | |
| | | 0 | 1 | 2 | 3 | 3.5 | 6 | 8 | 9 | 10 | 12 |
| Almond whole | Captan (THPI) | 0.41 | 0.27 | 0.42 (0) | 0.38 (0) | | 0.33 (0.02) | | 0.36 (0) | | |
| | THPI | 0.44 | 0.36 | | 0.31 | | 0.35 | | 0.45 | | |
| Almond coarse ground | Captan (THPI) | 0.50 | 0.21 | 0.33 (0.07) | | | | | | | |
| | THPI | 0.38 | 0.38 | | | | | | | | |
| Apples | Captan | 0.52 | 0.41 | | 0.39 | | 0.38 | | 0.43 | | |
| | THPI | 0.42 | 0.44 | | 0.40 | | 0.36 | | 0.49 | | |
| Apple sauce | Captan | 0.47 | 0.40 | | 0.37 | | 0.37 | | 0.35 | | |
| | THPI | 0.43 | 0.37 | | 0.36 | | 0.42 | | 0.38 | | |
| Maize grain, whole | Captan (THPI) | 0.40 | 0.13 | 0.20 (0.08) | 0.13 (0.08) | 0.14 (0.09) | | 0.19 (0.08) | | | 0.05 (0.21) |
| | THPI | 0.46 | 0.35 | | 0.46 | 0.40 | | 0.40 | | | 0.46 |
| Maize grain, coarse | Captan | 0.44 | 0.05 | | | | | | | | |
| | THPI | 0.40 | 0.36 | | | | | | | | |
| Grape pomace, dry | Captan | 0.41 | 0.35 | | 0.41 | | 0.38 | | 0.37 | | |
| | THPI | 0.42 | 0.36 | | 0.43 | | 0.38 | | 0.41 | | |
| Potato tubers | Captan | 0.46 | 0.37 | | 0.37 | | 0.37 | | 0.34 | | |
| | THPI | 0.45 | 0.40 | | 0.37 | | 0.35 | | 0.36 | | |
| Raisins | Captan | 0.46 | 0.36 | | 0.37 | | 0.42 | | | 0.36 | |
| | THPI | 0.39 | 0.44 | | 0.53 | | 0.49 | | | 0.41 | |
| Spinach, chopped coarse | Captan (THPI) | 0.48 | 0.06 | 0.12 (0.10) | 0.14 (0.08) | | | 0.06 (0.14) | | | 0.03 (0.18) |
| | THPI | 0.44 | 0.39 | | 0.47 | | | 0.36 | | | 0.47 |
| Spinach, chopped fine | Captan | 0.49 | 0.15 | | | | | | | | |
| | THPI | 0.43 | 0.37 | | | | | | | | |
| Sugar beet tops | Captan (THPI) | 0.48 (0) | 0.43 (0.02) | | 0.46 (0.05) | | 0.42 (0) | | | | 0.32 (0.05) |
| | THPI | 0.47 | 0.35 | | 0.42 | | 0.37 | | | | 0.33 |
| Tomato | Captan | 0.46 | 0.38 | | 0.38 | | 0.38 | | 0.40 | | |
| | THPI | 0.37 | 0.42 | | 0.46 | | 0.43 | | 0.42 | | |
| Tomato pomace, dry | Captan | 0.43 | 0.42 | | 0.38 | | 0.36 | | 0.38 | | |
| | THPI | 0.43 | 0.38 | | 0.40 | | 0.39 | | 0.38 | | |
| Tomato sauce | Captan | 0.42 | 0.51 | | 0.44 | | 0.42 | | 0.36 | | |
| | THPI | 0.41 | 0.38 | | 0.53 | | 0.46 | | 0.39 | | |
| Wheat forage | Captan (THPI) | 0.41 (0) | 0.39 (0.07) | | 0.37 (0.04) | | 0.10 (0.18) | | | | 0.04 (0.09) |
| | THPI | 0.39 | 0.46 | | 0.39 | | 0.54 | | | | 0.35 |

Samples for captan determination should be stored whole if possible and residues of captan should be determined as soon as possible after the collection of samples for analysis.

Schlesinger (1992a) studied the freezer storage stability of both captan and THPI in macerated samples of potato, tomato and melon fortified at 1 mg/kg. Recoveries of captan from samples stored for about 1 year were 65, 73 and 78% for potato, tomato and melon respectively.

Recoveries of THPI from the fortified samples stored frozen for about 1 year were 66, 87 and 72% respectively.

Meyers and Wiebe (1995) studied the freezer storage stability of captan in milk. Residues of captan decreased rapidly with a half-life of approximately 29 days. However, there was a concomitant increase in THPI concentration so that the total residue of captan + THPI expressed as captan remained fairly constant throughout the storage period. The slight variation in the total residue can be attributed to the analytical method and differences in concurrent recoveries. The hydrolysis of captan in milk is not unexpected as the pH of milk is typically 6.8 and the half-life for hydrolysis in aqueous solution at pH 7 is 2.6 hours.

Table 16. Captan and THPI residues in milk fortified at 0.4 mg/kg during freezer storage at -20 ± 10°C (Meyers and Wiebe, 1995).

| Storage time, days[2] | Residue, mg/kg[1] | | | |
|---|---|---|---|---|
| | Captan | THPI | Total (mg captan equivalents/kg) | Total % remaining on storage |
| 0 | 0.43 | 0.02 | 0.46 | 115 |
| 14 | 0.31 | 0.006 | 0.42 | 105 |
| 29 | 0.21 | 0.07 | 0.34 | 85 |
| 90 | 0.17 | 0.11 | 0.39 | 96 |
| 181 | 0.10 | 0.13 | 0.36 | 91 |
| 367 | 0.06 | 0.17 | 0.35 | 87 |

[1] Means of three determinations
[2] From fortification to extraction

The freezer stability of THPI and the *cis*- and *trans*-isomers of 3-OH THPI and 5-OH THPI was determined in milk and bovine tissues (Wiebe *et al.*, 1992). The compounds were stable on freezer storage at -20 °C for at least 3 years.

Table 17. The stability of THPI and hydroxylated metabolites in milk and bovine tissues fortified at 0.4 mg/kg during freezer storage at -20 ± 10°C (Wiebe *et al.*, 1992).

| Sample | Storage time, days | % remaining after storage | | | | |
|---|---|---|---|---|---|---|
| | | THPI | *trans*-3-OH THPI | *cis*-3-OH THPI | *trans*-5-OH THPI | *cis*-5-OH THPI |
| Milk | 0 | 88 | 96 | 105 | 93 | 88 |
| | 14 | 86 | 101 | 100 | 95 | 90 |
| | 75 | 89 | 109 | 106 | 114 | 100 |
| | 181 | 89 | 90 | 91 | 97 | 98 |
| | 362 | 82 | 104 | 102 | 102 | 101 |
| | 734 | 85 | 98 | 89 | 91 | 96 |
| | 1185 | 89 | 93 | 90 | 90 | 78 |
| Fat | 0 | 82 | 100 | 103 | 100 | 85 |
| | 14 | 78 | 76 | 80 | 76 | 64 |
| | 122 | 80 | 84 | 86 | 92 | 80 |
| | 243 | 94 | 95 | 85 | 126 | 90 |
| | 368 | 81 | 75 | 74 | 76 | 61 |
| | 734 | 87 | 94 | 92 | 95 | 90 |
| | 1373 | 90 | 100 | 100 | 89 | 77 |
| Kidney | 0 | 77 | 89 | 88 | 86 | 81 |
| | 14 | 86 | 98 | 98 | 128 | 131 |
| | 122 | 74 | 69 | 74 | 79 | 77 |
| | 243 | 67 | 71 | 76 | 120 | 84 |
| | 372 | 74 | 90 | 88 | 112 | 105 |
| | 938 | 71 | 72 | 70 | 78 | 79 |
| | 1233 | 88 | 76 | 70 | 72 | 73 |
| Liver | 0 | 65 | 63 | 73 | 90 | 94 |
| | 14 | 78 | 75 | 86 | 111 | 114 |
| | 120 | 78 | 67 | 75 | 107 | 106 |
| | 243 | 73 | 65 | 74 | 105 | 105 |

| Sample | Storage time, days | % remaining after storage | | | | |
|---|---|---|---|---|---|---|
| | | THPI | *trans*-3-OH THPI | *cis*-3-OH THPI | *trans*-5-OH THPI | *cis*-5-OH THPI |
| | 365 | 76 | 75 | 81 | 81 | 85 |
| | 932 | 68 | 72 | 78 | 71 | 67 |
| | 1188 | 74 | 61 | 70 | 80 | 77 |
| Muscle | 0 | 87 | 80 | 83 | 83 | 81 |
| | 14 | 87 | 93 | 92 | 89 | 90 |
| | 142 | 86 | 80 | 90 | 79 | 86 |
| | 245 | 78 | 85 | 82 | 79 | 77 |
| | 367 | 86 | 81 | 80 | 73 | 65 |
| | 902 | 80 | 76 | 72 | 65 | 58 |
| | 1359 | 80 | 81 | 84 | 72 | 68 |

**Definition of the residue**

Captan is metabolized by cleavage of the N-S bond to form THPI and derivatives of the *trichloromethyl*thio side chain followed by further degradation of the cleavage products. THPI is the major metabolite detected in plants but is generally present at much lower levels than those of captan. The definition of the residue for commodities derived from plants should be "captan" both for compliance with MRLs and the estimation of dietary intake.

## USE PATTERN

Captan is registered as a fungicide in many countries. It is a broad-spectrum fungicide with activity against many diseases including scab (*Venturia spp*.), sooty blotch (*Gleodes pomigena*), leaf spot (*Alternaria mali*), fly speck (*Schizothyrium pomi*) and bitter rot (*Glomerella cingulata*) in pome fruit; brown rot/blossom blight (*Monilinia spp*.), scab (*Fusicladium carpophila*) and shot hole (*Stigmina carpophila*) in stone fruit; downy mildew (*Plasmopara viticola*), grey mould (*Botrytis cinerea*), and black rot (*Guignardia bidwellii*) in grapes; leaf spots (*Cladosporium* spp., *Mycospaerella* spp.), grey mould (*Botrytis cinerea*), anthracnose (*Colletotrichum spp*.) and damping off (*Pythium spp*., *Rhizoctonia spp*., *Penicillium spp*. and *Phythophthora spp*.) in vegetable crops.

The information available to the Meeting on registered uses is summarized in Table 18.

Table 18. Registered uses of captan.

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| Alfalfa | Paraguay | TS 750 | Foliar | | 0.35 | | |
| Almond | Argentina | WP 800 | Foliar | | 0.12 | | 43 |
| Almond | Belgium | WP 830 | Foliar | | 0.12 | | 30 |
| Almond | Greece | WP 830 | Foliar | | 0.13 | 4 | 30 |
| Almond | Portugal | WP 830 | Foliar | | 0.15-0.2 | | 14 |
| Almond | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Almond | USA | WP 500 | Foliar | 2.2-4.9 | | | 30 |
| Apple | Argentina | WP 800 | Foliar | | 0.12 | | 14 |
| Apple | Australia | WP 800 | Foliar | | 0.1 | 5 | 7 |
| Apple | Belgium | WG 800 | Foliar | | 0.1-0.12 | | 14 |
| Apple | Brazil | WP 500 | Foliar | | 0.11-0.12 (1-3 l/tree) | | 1 |
| Apple | Canada | WP 500 | Foliar | 1.5-3 | | | 7 |
| Apple | Chile | WP 800 | Foliar | | 0.14 | | (1-10)[2] |
| Apple | Colombia | WP 500 | Foliar | | 0.11 | | |
| Apple | Costa Rica | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Apple | Ecuador | WP 800 | Foliar | 0.8-1.2 | | | 15 |
| Apple | El Salvador | WP 500 | Foliar | 0.75 | 0.12 | | 7 |

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| Apple | Guatemala | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Apple | Honduras | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Apple | Ireland | WP 800 | Foliar | 2.7 | | 12 | 14 |
| Apple | Israel | WP 500 | Foliar | 1.3-3.8 | 0.13 | | 7 |
| Apple | Japan | WP 800 | Foliar | 2-8 | 0.07-0.13 | 6 | 14 |
| Apple | Mexico | WP 500 | Foliar | | 0.1-0.13 | 2-4 | |
| Apple | Netherlands | WP 830 | Foliar | 0.5-2.1[1] | 0.05-0.21 | | 7 (0.06%)/ 21 (>0.1%) |
| Apple | Nicaragua | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Apple | Panama | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Apple | Poland | WP 500 | Foliar | 1.5-2.3 | | | 7 |
| Apple | Portugal | WP 830 | Foliar | | 0.15-0.2 | | 21 |
| Apple | Romania | WP 500 | Foliar | | 0.13 | | 14 |
| Apple | South Africa | WP 500 | Foliar | | 0.08-0.1 | | 14 |
| Apple | Syria | WP 830 | Foliar | | 0.1 | | 14 |
| Apple | Turkey | WP 500 | Foliar | | 0.08 | 2+ | 3 |
| Apple | UK | WG 800 | Foliar | 2.7 | | 10-12[1] | 14 |
| Apple | Uruguay | WP 800 | Foliar | | 0.1-0.13 | 2 | 7 |
| Apple | USA | WP 500 | Foliar | 2.2-4.4 | | | Nil |
| Apple | USA | WP 500 | Post-H dip | | 0.15 | | Nil |
| Apricot | Canada | WP 500 | Foliar | 3-3.6 | | | 2 |
| Apricot | Poland | WP 500 | Foliar | 1.5-2.3 | | | 7 |
| Apricot | Turkey | WP 500 | Foliar | | 0.15 | 2+ | 3 |
| Apricot | Uruguay | WP 800 | Foliar | | 0.13 | | 7 |
| Apricot | USA | WP 500 | Foliar | 1.6-2.7 | | | 2 |
| Avocado | Costa Rica | WP 500 | Foliar | 1.4 | 0.12 | | 3-7 |
| Avocado | El Salvador | WP 500 | Foliar | 1.4 | 0.12 | | 3-7 |
| Avocado | Guatemala | WP 500 | Foliar | 1.4 | 0.12 | | 3-7 |
| Avocado | Honduras | WP 500 | Foliar | 1.4 | 0.12 | | 3-7 |
| Avocado | Mexico | WP 500 | Foliar | | 0.13 | | |
| Avocado | Nicaragua | WP 500 | Foliar | 1.4 | 0.12 | | 3-7 |
| Avocado | Panama | WP 500 | Foliar | 1.4 | 0.12 | | 3-7 |
| Avocado | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Bean | Bolivia | TS 750 | Foliar | | 0.14 | | |
| Bean | Chile | WP 800 | Foliar | 1.6 | | | 1 |
| Bean | Dominican Republic | WP 500, WP 800 | Foliar | 1-3 | | | 3 |
| Bean (broad) | Ecuador | WP 800 | Foliar | 0.8-1.2 | | | 15 |
| Bean | Hungary | WP 500 | Foliar | 0.4-1.5 | 0.1-0.13 | | 14 |
| Bean | Paraguay | TS 750 | Foliar | | 0.14 | | |
| Bean | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Bean | Poland | WP 500 | Foliar | 0.6 | | | 7 |
| Bean (broad) | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Bean (French) | Spain | SC 475 | Foliar | | 0.13-0.15 | | 21 |
| Bean | Venezuela | WP 500 | Soil | 6.0-7.0 | | | 30 |
| Blackberry | Belgium | WG 800 | Foliar | | 0.12 | | 4 |
| Blackberry | Canada | WP 500 | Foliar | 1.8 | | | 2 |
| Blackberry | Netherlands | WP 830 | Foliar | | 0.13 | | 4 |
| Blueberry | Canada | WP 500 | Foliar | 1.8 | | | 2 |
| Blueberry | USA | WP 500 | Foliar | 1.1-2.7 | | | Nil |
| Broccoli | Bolivia | TS 750 | Foliar | | 0.17 | | |
| Broccoli | Paraguay | TS 750 | Foliar | | 0.17 | | |
| Cantaloupe | Argentina | WP 800 | Foliar | 2.2 | 0.12 | | 7 |
| Cantaloupe (melon) | Brazil | WP 500 | Foliar | | 0.1 | | 1 |
| Cantaloupe | Colombia | WP 500 | Foliar | | 0.11 | | |
| Cantaloupe | Costa Rica | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Cantaloupe (melon) | Dominican Republic | WP 500, WP 800 | Foliar | 2.3-3.6 | | | 3 |

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| Cantaloupe | El Salvador | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Cantaloupe | Guatemala | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Cantaloupe | Honduras | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Cantaloupe | Mexico | WP 500 | Foliar | 1-1.5 | | | Nil |
| Cantaloupe | Nicaragua | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Cantaloupe | Panama | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Melon | Philippines | WP 500 | Foliar | | 0.5-1 | | |
| Melon | Venezuela | WP 500 | Foliar | 2.0-2.5 | | | 30 |
| Carrot | Costa Rica | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Carrot | El Salvador | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Carrot | Guatemala | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Carrot | Honduras | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Carrot | Mexico | WP 500 | Foliar | 2 | | | Nil |
| Carrot | Nicaragua | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Carrot | Panama | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Carrot | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Cauliflower | Bolivia | TS 750 | Foliar | | 0.17 | | |
| Cauliflower | Paraguay | TS 750 | Foliar | | 0.17 | | |
| Celery | Argentina | WP 800 | | | 0.12 | | Ns |
| Celery | Colombia | WP 500 | Foliar | | 0.11 | | |
| Celery | Costa Rica | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Celery | El Salvador | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Celery | Guatemala | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Celery | Honduras | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Celery | Nicaragua | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Celery | Panama | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Celery | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Cherry | Argentina | WP 800 | Foliar | | 0.12 | | 14 |
| Cherry | Belgium | WG 800 | Foliar | | 0.12 | | 4 |
| Cherry | Canada | WP 500 | Foliar | 3-3.6 | | | 2 (sweet)<br>5 (sour) |
| Cherry | Colombia | WP 500 | Foliar | | 0.11 | | |
| Cherry | Greece | WP 830 | Foliar | | 0.13 | 4 | 20 |
| Cherry | Italy | WP 450 | Foliar | 1.1-2.3 | | | 20 |
| Cherry | Japan | WP 800 | Foliar | 3-6 | 0.1 | 5 | 14 |
| Cherry (sour) | Poland | WP 500 | Foliar | 1.5-2.3 | | | 7 |
| Cherry | Romania | WP 800 | Foliar | | 0.13 | | 14 |
| Cherry | USA | WP 500 | Foliar | 1.1-2.2 | | | Nil |
| Cherry | USA | WP 500 | Post-H dip | | 0.15 | | Nil |
| Chili | Costa Rica | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Chili | El Salvador | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Chili | Guatemala | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Chili | Honduras | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Chili | Mexico | WP 500 | Foliar | 1-1.5 | | | Nil |
| Chili | Honduras | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Chili | Nicaragua | WP 800 | Foliar | | 0.14-0.23 | | 14 |
| Chili | Panama | WP 800 | Foliar | | 0.14-0.23 | | 14 |
| Citrus | Argentina | WP 800 | Foliar | | 0.14-0.23 | | 14 |
| Citrus | Brazil | WP 500 | Foliar | | 0.11-0.12<br>(2-5 l/tree) | | 7 |
| Citrus | Costa Rica | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Citrus | Dominican Republic | WP 500, WP 800 | Foliar | 4-6.5 | | | 3 |
| Citrus | El Salvador | WP 500 | Foliar | 0.75-1.4 | 0.12 | | 3-7 |
| Citrus | Guatemala | WP 500 | Foliar | 0.75-1.4 | 0.12 | | 3-7 |
| Citrus | Honduras | WP 500 | Foliar | 0.75-1.4 | 0.12 | | 3-7 |
| Citrus | Japan | WP 200 | Foliar | 0.67-2 | 0.27-0.4 | 5 | 5 |
| Citrus | Nicaragua | WP 500 | Foliar | 0.75-1.4 | 0.12 | | 3-7 |
| Citrus | Panama | WP 500 | Foliar | 0.75-1.4 | 0.12 | | 3-7 |
| Citrus | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| Citrus | Spain | SC 475 | Foliar | | 0.15-0.25 | | 10 |
| Citrus | Turkey | WP 500 | Foliar | | 0.15 | 2+ | 3 |
| Citrus | Venezuela | WP 500 | Foliar | | 0.1 | | 30 |
| Coffee | El Salvador | WP 500 | Foliar | 1-1.5 | | | 3 |
| Coffee | Guatemala | WP 500 | Foliar | 1-1.5 | | | 3 |
| Coffee | Honduras | WP 500 | Foliar | 1-1.5 | | | 3 |
| Coffee | Nicaragua | WP 500 | Foliar | 1-1.5 | | | 3 |
| Coffee | Panama | WP 500 | Foliar | 1-1.5 | | | 3 |
| Corn | Bolivia | TS 750 | Foliar | | 0.11 | | |
| Corn | Kenya | WP 800 | Seed treatment | | | | |
| Corn | Paraguay | TS 750 | Foliar | | 0.11 | | |
| Corn | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Cotton | Bolivia | TS 750 | Foliar | | 0.11-0.16 | | |
| Cotton | Paraguay | TS 750 | Foliar | | 0.11-0.16 | | |
| Cotton | Thailand | WP 500 | Seed treatment | 0.35 kg ai/100 kg seed | | | |
| Cotton | Venezuela | WP 500 | Soil | 6.0-7.0 | | | 30 |
| Cucumber | Argentina | WP 800 | Foliar | 2.2 | 0.12 | | 7 |
| Cucumber | Brazil | WP 500 | Foliar | | 0.1 | | 1 |
| Cucumber | Canada | WP 500 | Foliar | 1.6-3.4 | | | 2 |
| Cucumber | Dominican Republic | WP 500, WP 800 | Foliar | 2.3 | | | 3 |
| Cucumber | El Salvador | WP 500 | Foliar | 1-2 | | | 3 |
| Cucumber | Guatemala | WP 500 | Foliar | 1-2 | | | 3 |
| Cucumber | Honduras | WP 500 | Foliar | 1-2 | | | 3 |
| Cucumber | Japan | WP 800 | Foliar | 1.5-4 | 0.1-0.13 | 5 | 1 |
| Cucumber | Mexico | WP 500 | Foliar | 1-1.5 | | | Nil |
| Cucumber | Nicaragua | WP 500 | Foliar | 1-2 | | | 3 |
| Cucumber | Panama | WP 500 | Foliar | 1-2 | | | 3 |
| Cucumber | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Cucumber | Romania | WP 800 | Foliar | | 0.1 | | 14 |
| Cucumber | Venezuela | WP 500 | Foliar | 2.0-2.5 | | | 30 |
| Cucurbits | Greece | WP 830 | Foliar | | 0.13 | | 20 |
| Cucurbits | Venezuela | WP 500 | Foliar | 2.0-2.5 | | | 30 |
| Currants | Belgium | WG 800 | Foliar | | 0.12 | | 10 |
| Currants | Netherlands | WP 830 | Foliar | | 0.13 | | 10 |
| Eggplant | Argentina | WP 800 | Foliar | 1.6 | 0.12 | | 7 |
| Eggplant | Mexico | WP 500 | Foliar | 1-1.5 | | | Nil |
| Eggplant | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Eggplant | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Endive | Netherlands | WP 830 | Foliar | | 0.13 | | 21 |
| Endive | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Escarole | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Fruit[3] | Hungary | WP 500 | Foliar | 1-1.5 | 0.1-0.15 | | 10 |
| Garlic | Brazil | WP 500 | Foliar | | 0.11 | | 7 |
| Garlic | Dominican Republic | WP 500, WP 800 | Foliar | 1.5-4.8 | | | 3 |
| Garlic | El Salvador | WP 500 | Foliar | 1-2 | | | 3 |
| Garlic | Guatemala | WP 500 | Foliar | 1-2 | | | 3 |
| Garlic | Honduras | WP 500 | Foliar | 1-2 | | | 3 |
| Garlic | Mexico | WP 500 | Foliar | 1.3-1.5 | | | Nil |
| Garlic | Nicaragua | WP 500 | Foliar | 1-2 | | | 3 |
| Garlic | Panama | WP 500 | Foliar | 1-2 | | | 3 |
| Garlic | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Grape | Australia | WP 800 | Foliar | | 0.1 | 5 | 7 |
| Grape | Argentina | WP 800 | Foliar | | 0.14 | | 14 (table) 25 (wine) |
| Grape | Belgium | WG 800 | Foliar | | 0.12 | | 42 |
| Grape | Brazil | WP 500 | Foliar | | 0.11-0.12 (1-3 | | 1 |

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| | | | | | l/vine) | | |
| Grape | Canada | WP 500 | Foliar | 1.6-2.8 | | | 7 |
| Grape (table) | Chile | WP 800 | Foliar | 3.2 | | 3-4[1] | (1-15)[2] |
| Grape (wine) | Chile | WP 800 | Foliar | 3.2 | 0.14 | 3-4[1] | 30 |
| Grape | Colombia | WP 500 | Foliar | | 0.11 | | |
| Grape | France | WP 830 | Foliar | 1.9 | | | (14/21)[1] |
| Grape | Greece | WP 830 | Foliar | | 0.13 | 3-4[1] | 40 |
| Grape | Hungary | WP 500 | Foliar | 1-1.5 | 0.1-0.15 | 2-3[1] | 10 (table)<br>30 (wine) |
| Grape | Israel | WP 500 | Foliar | 1.3-3.8 | 0.13 | | 14 |
| Grape | Japan | WP 800 | Foliar | 2-3 | 0.1 | 5 | 30 |
| Grape | Mexico | WP 500 | Foliar | | 0.01-0.013 | 2 | Nil |
| Grape | Morocco | WP 500 | Foliar | | 0.13 | | |
| Grape | Peru | WP 800 | Foliar | | 0.16-0.2 | | 3-7 |
| Grape | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Grape | Portugal | WP 830 | Foliar | | 0.16 | | 21 (table)<br>42 (wine) |
| Grape | Romania | WP 800 | Foliar | | 0.1 | | 21 |
| Grape | Spain | SC 475 | Foliar | | 0.14-0.19 | | 21 |
| Grape | Thailand | WP 500 | Foliar | 3.1 | 0.13 | | |
| Grape | Turkey | WP 500 | Foliar | | 0.15 | 2+ | 3 |
| Grape | Uruguay | WP 800 | Foliar | 0.6-1.2 | | | 21 |
| Grape | USA | WP 500 | Foliar | 1.1-2.2 | | | Nil |
| Grape | Venezuela | WP 500 | Foliar | | 0.5-0.62 | | 30 |
| Leek | Netherlands | WP 830 | Foliar | | 0.25 | | 14 |
| Leek | Spain | SC 475 | Foliar | | 0.13-0.15 | | 21 |
| Lettuce | Costa Rica | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Lettuce | El Salvador | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Lettuce | Guatemala | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Lettuce | Honduras | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Lettuce | Nicaragua | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Lettuce | Panama | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Lettuce | Spain | SC 475 | Foliar | | 0.13-0.15 | | 21 |
| Lettuce | Turkey | WP 830 | Foliar | | 0.15 | | 7 |
| Loganberry | Canada | WP 500 | Foliar | 1.2-1.8 | | | 2 |
| Loquat | Turkey | WP 500 | Foliar | | 0.15 | | 3 |
| Mango | Mexico | WP 500 | Foliar | | 0.13-0.175 | | Nil |
| Mango | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Mango | Thailand | WP 500 | Foliar | 1.6-2.0 | 0.1-0.13 | | |
| Melon | Japan | WP 800 | Foliar | 2-4 | 0.13 | 5 | 14 |
| Melon | Hungary | WP 500 | Foliar | 0.4-1.0 | 0.06-0.14 | | 14 |
| Melon | Portugal | WP 830 | Foliar | | 0.15-0.2 | | 28 |
| Nectarine | Argentina | WP 800 | Foliar | | 0.12-0.14 | | 14 |
| Nectarine | Greece | WP 830 | Foliar | | 0.13 | 4 | 20 |
| Nectarine | USA | WP 500 | Foliar | 1.4-4.4 | | | Nil |
| Nectarine | Uruguay | WP 800 | Foliar | | 0.13 | | 7 |
| Oil palm | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Olive | Spain | SC 475 | Foliar | | 0.125-0.15 | | 10 |
| Onion | Argentina | WP 800 | | | 0.12 | | Ns |
| Onion | Brazil | WP 500 | Foliar | | 0.11-0.12 | | 7 |
| Onion | Costa Rica | WP 500 | Foliar | 0.5-2 | 0.12 | | 3-7 |
| Onion | Dominican Republic | WP 500 | Foliar | 1.5-4.8 | | | 3 |
| Onion | El Salvador | WP 500 | Foliar | 0.5-2 | 0.12 | | 3-7 |
| Onion | Guatemala | WP 500 | Foliar | 0.5-2 | 0.12 | | 3-7 |
| Onion | Honduras | WP 500 | Foliar | 0.5-2 | 0.12 | | 3-7 |
| Onion | Nicaragua | WP 500 | Foliar | 0.5-2 | 0.12 | | 3-7 |
| Onion | Panama | WP 500 | Foliar | 0.5-2 | 0.12 | | 3-7 |
| Onion | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Onion | Turkey | WP 830 | Foliar | | 0.15 | | 7 |
| Orange | Peru | WP 800 | Foliar | | 0.16-0.2 | | 3-7 |

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| Papaya | Venezuela | WP 500 | Foliar | 2.0-2.5 | | | 30 |
| Parsley | Argentina | WP 800 | | | 0.12 | | Ns |
| Peas | Poland | WP | Seed treatment | 0.15/100 kg seed | | | - |
| Peas | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Peas (chick) | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Peach | Argentina | WP 800 | Foliar | | 0.12-0.14 | | 14 |
| Peach | Brazil | WP 500 | Foliar | | 0.11-0.12 (1-3 l/tree) | | 1 |
| Peach | Canada | WP 500 | Foliar | 3-3.6 | | | 2 |
| Peach | Colombia | WP 500 | Foliar | | 0.11 | | |
| Peach | France | WP 830 | Foliar | | 0.25 | | 14[1] |
| Peach | Greece | WP 830 | Foliar | | 0.13 | 4 | 20 |
| Peach | Poland | WP 500 | Foliar | 1.5-2.3 | | | 7 |
| Peach | Portugal | WP 830 | Foliar | | 0.15-0.2 | | 7 |
| Peach | Romania | WP 800 | Foliar | | 0.13 | | 14 |
| Peach | Turkey | WP 500 | Foliar | | 0.1-0.15 | 2+ | 3 |
| Peach | Uruguay | WP 800 | Foliar | | 0.13 | | 7 |
| Peach | USA | WP 500 | Foliar | 2.2-4.4 | | | Nil |
| Peach | Venezuela | WP 500 | Foliar | 2.0-2.5 | | | 30 |
| Peanut | Bolivia | TS 750 | Foliar | | 0.15 | | |
| Peanut | Paraguay | TS 750 | Foliar | | 0.15 | | |
| Pear | Argentina | WP 800 | Foliar | | 0.12 | | 14 |
| Pear | Australia | WG 800 | Foliar | | 0.1 | 5 | 7 |
| Pear | Belgium | WG 800 | Foliar | | 0.1-0.12 | | 14 |
| Pear | Brazil | WP 500 | Foliar | | 0.11-0.12 (1-3 l/tree) | | 1 |
| Pear | Canada | WP 500 | Foliar | 3 | | | 7 |
| Pear | Chile | WP 800 | Foliar | | 0.14 | | (1-10)[2] |
| Pear | Colombia | WP 500 | Foliar | | 0.11 | | |
| Pear | Ecuador | WP 800 | Foliar | 0.8-1.2 | | 1-2[1] | 15 |
| Pear | Ireland | WP 800 | Foliar | 2.7 | | 12 | 14 |
| Pear | Israel | WP 500 | Foliar | 1.3-3.8 | 0.13 | | 7 |
| Pear | Italy | WP 450 | Foliar | | 0.13-0.16 | | 15 |
| Pear | Japan | WP 800 | Foliar | 2.4-8 | 0.08-0.13 | 9 | 7 |
| Pear | Mexico | WP 500 | Foliar | | 0.01-0.013 | 2 | Nil |
| Pear | Netherlands | WP 830 | Foliar | | 0.05-0.21 | | 7 (0.06%) 21 (>0.1%) |
| Pear | Poland | WP 500 | Foliar | 1.5-2.3 | | 10-12[1] | 7 |
| Pear | Portugal | WP 830 | Foliar | | 0.15-0.2 | | 21 |
| Pear | Romania | WP 800 | Foliar | | 0.13 | | 14 |
| Pear | South Africa | WP 500 | Foliar | | 0.08-0.1 | | 14 |
| Pear | Syria | WP 830 | Foliar | | 0.1 | | 7 |
| Pear | Turkey | WP 500 | Foliar | | 0.08 | 2+ | 3 |
| Pear | UK | WG 800 | Foliar | 2.7 | | 12 | 14 |
| Pear | Uruguay | WP 800 | Foliar | | 0.1-0.13 | 2 | 7 |
| Pear | USA | WP 500 | Post-H dip | | 0.15 | | Nil |
| Pepper (bell) | Argentina | WP 800 | Foliar | 1.6 | 0.12 | | 7 |
| Pepper | Ecuador | WP 800 | Foliar | 0.8-1.2 | | | 15 |
| Pepper | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Pepper | Venezuela | WP 500 | Foliar | | 0.5-1.0 | | 30 |
| Pineapple | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Plum | Argentina | WP 800 | Foliar | | 0.12 | | 14 |
| Plum/prune | Belgium | WG 800 | Foliar | | 0.12 | | 4 |
| Plum/prune | Canada | WP 500 | Foliar | 3-3.6 | | | 2 |
| Plum/prune | Colombia | WP 500 | Foliar | | 0.11 | | |
| Plum | Greece | WP 830 | Foliar | | 0.13 | 4 | 20 |
| Plum | Italy | WP 450 | Foliar | 1.1-2.3 | | | 20 |
| Plum | Japan | WP800 | Foliar | 2.4-8 | 0.08-0.13 | 5 | 14 |

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| Plum/prune | Portugal | WP 830 | Foliar | | 0.15-0.2 | | 7 |
| Plum | Romania | WP 800 | Foliar | | 0.13 | | 14 |
| Plum | Turkey | WP 500 | Foliar | | 0.1 | 2+ | 3 |
| Plum | Uruguay | WP 800 | Foliar | | 0.12 | | 7 |
| Plum/prune | USA | WP 500 | Foliar | 2.2-3.3 | | | Nil |
| Pome fruit | France | WP 830 | Foliar | 1.5[1] | 0.15 | | 14[1] |
| Pome fruit | Germany | WP 830 | Foliar | 1.5[1] | 0.1 | 13 | 21 |
| Pome fruit | Greece | WP 830 | Foliar | | 0.13 | 3+ | 15 |
| Pome fruit | Morocco | WP 500 | Foliar | | 0.15 | | |
| Pome fruit | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Potato | Argentina | WP 800 | Foliar | 0.96-1.2 | 0.5 | | 7 |
| Potato | Brazil | WP 500 | Foliar | | 0.11-0.12 | | 14 |
| Potato | Colombia | WP 500 | Foliar | | 0.11 | | |
| Potato | Dominican Republic | WP 500 | Foliar | 1.5-4.8 | 0.17 | | 3 |
| Potato | Ecuador | WP 800 | Foliar | 0.8-1.2 | | | 15 |
| Potato | El Salvador | WP 500 | Foliar | 1-2 | | | 3 |
| Potato | Greece | WP 830 | Foliar | | 0.13 | | 20 |
| Potato | Guatemala | WP 500 | Foliar | 1-2 | | | 3 |
| Potato | Honduras | WP 500 | Foliar | 1-2 | | | 3 |
| Potato | Mexico | WP 500 | Foliar | | 0.1-0.2 | | 14 |
| Potato | Morocco | WP 500 | Foliar | | 0.15 | | |
| Potato | Nicaragua | WP 500 | Foliar | 1-2 | | | 3 |
| Potato | Panama | WP 500 | Foliar | 1-2 | | | 3 |
| Potato | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Potato | Poland | WP 500 | Foliar | 1.5-1.8 | | | 7 |
| Potato | Portugal | WP 830 | Foliar | | 0.15-0.2 | | 7 |
| Potato | Spain | WP 475 | Foliar | | 0.13-0.15 | | 10 |
| Potato | Turkey | WP 500 | Foliar | | 0.18 | 2+ | 7 |
| Potato | Uruguay | WP 800 | Foliar | 0.6-1.0 | | | 7 |
| Potato | Venezuela | WP 500 | Foliar | | 0.5-1.0 | | 30 |
| Pumpkin | Colombia | WP 500 | Foliar | | 0.11 | | |
| Quince | Uruguay | WP 800 | Foliar | | 0.1-0.13 | 2 | 7 |
| Raspberry | Belgium | WG 800 | Foliar | | 0.12 | | 4 |
| Raspberry | Canada | WP 500 | Foliar | 2 | | | 2 |
| Raspberry | Netherlands | WP 830 | Foliar | | 0.13 | | 4 |
| Rhubarb | Canada | WP 500 | Foliar | 1.1-1.6 | | | 2 ( in forcing sheds) |
| Soya | Bolivia | TS 750 | Foliar | | 0.11 | | |
| Soya | Costa Rica | WP 500 | Foliar | | 0.12 | | 7 |
| Soya | El Salvador | WP 500 | Foliar | | 0.12 | | 7 |
| Soya | Guatemala | WP 500 | Foliar | | 0.12 | | 7 |
| Soya | Honduras | WP 500 | Foliar | | 0.12 | | 7 |
| Soya | Nicaragua | WP 500 | Foliar | | 0.12 | | 7 |
| Soya | Panama | WP 500 | Foliar | | 0.12 | | 7 |
| Soya | Paraguay | TS 750 | Foliar | | 0.11 | | |
| Soya | Thailand | WP 500 | Foliar | 0.47-0.63 | 0.08-0.1 | 3-4 | |
| Squash | Argentina | WP 800 | Foliar | 2.2 | 0.12 | | 7 |
| Squash | Mexico | WP 500 | Foliar | 1-1.5 | | | Nil |
| Squash | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Stone fruit except apricot | Australia | WP 800 | Foliar | | 0.1 | 5 | 7 |
| Stone fruit | Chile | WP 800 | Foliar | | 0.14 | | (0-10)[2] |
| Stone fruit | Greece | WP 830 | Foliar | | 0.13 | 4 | 20 |
| Stone fruit | Israel | WP 500 | Foliar | 1.3-3.8 | 0.13 | | 7 |
| Stone fruit | Spain | SC 475 | Foliar | | 0.13-0.15 | | 10 |
| Strawberry | Australia | WP 800 | Foliar | | 0.10-0.16 | 5 | 1 |
| Strawberry | Argentina | WP 800 | Foliar | | 0.08-0.2 | | 5 |
| Strawberry | Belgium | WG 800 | Foliar | | 0.12 | | 4 (field)<br>14 (glass) |

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| Strawberry | Canada | WP 500 | Foliar | 2.3-3.4 | | | 2 |
| Strawberry | Chile | WP 800 | Foliar | 1.6-3.2 | | 1-2[1] | (2-15)[2] |
| Strawberry | Colombia | WP 500 | Foliar | | 0.11 | | |
| Strawberry | Costa Rica | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Strawberry | Ecuador | WP 800 | Foliar | 1.2-1.6 | | | 15 |
| Strawberry | El Salvador | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Strawberry | Guatemala | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Strawberry | Honduras | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Strawberry | Israel | WP 500 | Foliar | 1.3-3.8 | 0.13 | | 7 |
| Strawberry | Japan | WP 800 | Foliar | 1.5-3 | 0.1 | 2 | 30 |
| Strawberry | Kenya | WP 800 | Foliar | 1 | | | 7 |
| Strawberry | Mexico | WP 500 | Foliar | 1.5-2 | | | nil |
| Strawberry | Netherlands | WP 830 | Foliar | | 0.13 | 2-4[1] | 4 field<br>14 glass |
| Strawberry | Nicaragua | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Strawberry | Panama | WP 500 | Foliar | 0.5 | 0.12 | | 7 |
| Strawberry | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Strawberry | Spain | SC 475 | Foliar | | 0.13-0.15 | | 21 |
| Strawberry | USA | WP 500 | Foliar | 1.6-3.3 | | | Nil |
| Strawberry | Venezuela | WP 500 | Foliar | | 0.5-0.62 | | 30 |
| Sunflower | Venezuela | WP 500 | Foliar | 1.0-1.5 | | | 30 |
| Sweet potato | Argentina | WP 800 | Foliar | | 0.24 | | 7 |
| Tomato | Argentina | WP 800 | Foliar | 2.2 | 0.12 | | 7 |
| Tomato | Brazil | WP 500 | Foliar | | 0.11-0.12 | | 1 |
| Tomato | Canada | WP 500 | Foliar | 2.3-3.4 | 0.12 | | 2 |
| Tomato | Colombia | WP 500 | Foliar | | 0.11 | | |
| Tomato | Costa Rica | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Tomato | Dominican Republic | WP 500 | Foliar | 1.5-4.8 | 0.17 | | 3 |
| Tomato | Ecuador | WP 800 | Foliar | 0.8-1.2 | | 2[1] | 15 |
| Tomato | El Salvador | WP 800 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Tomato | Guatemala | WP 800 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Tomato | Honduras | WP 800 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Tomato | Hungary | WP 500 | Foliar | 0.4-1.0 | 0.06-0.14 | | 5 |
| Tomato | Japan | WP 800 | Foliar | 1-3 | 0.07-0.1 | 5 | 1 |
| Tomato | Mexico | WP 500 | Foliar | 0.75-1.5 | | | Nil |
| Tomato | Morocco | WP 500 | Foliar | | 0.15 | | |
| Tomato | Nicaragua | WP 800 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Tomato | Panama | WP 800 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Tomato | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Tomato | Portugal | WP 830 | Foliar | | 0.15-0.2 | | 7 |
| Tomato | Romania | WP 800 | Foliar | | 0.13 | | 21 |
| Tomato | Spain | SC 475 | Foliar | | 0.13-0.15 | 4[1] | 10 |
| Tomato | Syria | WP 830 | Foliar | | 0.17 | | 7 |
| Tomato | Turkey | WP 500 | Foliar | | 0.13-0.15 | 2+ | 7 |
| Tomato | Venezuela | WP 500 | Foliar | | 0.5-1.0 | | 30 |
| Vegetables | Greece | WP 830 | Foliar | | 0.13 | | 20 if eaten cooked, otherwise last application at 1st flowering |
| Vegetables | Hungary | WP 500 | Foliar | 0.4-1.5 | 0.1-0.15 | | 14 |
| Watermelon | Argentina | WP 800 | Foliar | 2.2 | 0.12 | | 7 |
| Watermelon | Brazil | WP 500, PM 500 | Foliar | | 0.11-0.12 | | 1 |
| Watermelon | Colombia | WP 500 | Foliar | | 0.11 | | |
| Watermelon | Costa Rica | WP 500 | Foliar | 0.75 | 0.12 | | 7 |
| Watermelon | El Salvador | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Watermelon | Guatemala | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Watermelon | Honduras | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Watermelon | Mexico | WP 500 | Foliar | 1-1.5 | | | Nil |

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray concentration, kg ai/hl | No. | |
| Watermelon | Nicaragua | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Watermelon | Panama | WP 500 | Foliar | 0.75-2 | 0.12 | | 3-7 |
| Watermelon | Philippines | WP 500 | Foliar | 0.75-1.5 | 0.19-0.38 | | Nil |
| Wheat | Bolivia | TS 750 | Foliar | | 0.14 | | |
| Wheat | Paraguay | TS 750 | Foliar | | 0.14 | | |

Ns = not specified
[1] information not on label but part of GAP followed in the country
[2] PHI depends on destination for export
[3] Fruit = apple, pear, cherry, nectarine, peach, plum, strawberry

## RESIDUES RESULTING FROM SUPERVISED TRIALS

Details of the residue trials are shown in tables 19-38 and are reviewed in order of the Codex Alimentarius Classification of Foods and Feeds.

Table 19. *Citrus. Lemon,* USA. *Mandarin*, Japan. *Oranges*, Brazil, Spain, USA. *Grapefruit,* USA.
Table 20. *Apple,* Argentina, Australia, Brazil, Canada, Chile, France, Germany, Hungary, Israel, Japan, Netherlands, Portugal, South Africa, UK, USA.
Table 21. *Pear,* Australia, Chile, Germany, Italy, Japan, South Africa, UK, USA.
Table 22. *Cherry,* Belgium, Germany, Japan, USA.
Table 23. *Plum,* Chile, Greece, Japan, Portugal, Spain, USA.
Table 24. *Apricots,* USA.
Table 25. *Nectarine,* Chile, Greece, Spain, USA.
Table 26. *Peach,* Australia, Canada, Chile, Italy, Japan, Spain, USA.
Table 27. *Blueberry,* USA.
Table 28. *Grape,* Australia, Brazil, Chile, France, Germany, Japan, USA.
Table 29. *Raspberry,* USA.
Table 30. *Strawberry,* Australia, Belgium, Chile, Germany, Hungary, Israel, Netherlands, Spain, USA.
Table 31. *Melon,* Japan, USA.
Table 32. *Cucumber*, Brazil, Japan, USA.
Table 33. *Squash,* USA
Table 34. *Tomato,* Brazil, Greece, Israel, Japan, Mexico, USA.
Table 35. *Soya bean,* Thailand
Table 36. *Potato,* Brazil, Canada, Mexico, Netherlands, Poland, UK, USA.
Table 37. *Radish,* Germany.
Table 38. *Almond*, USA.
Table 39. *Chive,* Germany.

Where residues were not detected the results are reported as below the limit of quantification (LOD), e.g. <0.05 mg/kg. Residues, application rates and spray concentrations have generally been rounded to 2 significant figures. Although trials included results for untreated controls, these results are not reported in the tables unless the residues in the control samples were greater than the LOD. The prefix “c” in the tables indicates samples from control plots. Where possible, residues are recorded uncorrected for analytical recoveries. It should be noted that unless stated otherwise concurrent recoveries were acceptable and any corrections would be small.

Most trials were fully reported, the exceptions being the Belgian trial on cherries, the Chilean trials on grapes (1991), the German trials on strawberries, grapes (1974-1977), radishes and chives, the Israeli tomato trial and the Polish trials on potatoes.

Captan was applied to oranges in supervised trials in Brazil, Spain and the USA by backpack, oscillating boom sprayer and mist-blower. Plot sizes ranged from 1 to 120 trees. The interval between the last two sprays in the Brazil trials was 289-292 days. In view of the long interval the residue data may be more reflective of a single spray. Mandarin trials in Japan were reported for pulp and peel separately rather than for whole fruit. Some of the trials were conducted indoors. Mandarins normally comprise 25-30% peel and 70-75% pulp, and these figures can be used to estimate the residues on a whole-fruit basis. Application of captan to lemons in Arizona was by hand gun (4 trees/plot) in California by oscillating boom spray (25 trees/plot). Grapefruit (30 trees/plot) were sprayed with a hand gun in the USA.

Plot sizes for the apple trials ranged from 1 to 16 trees. Equipment used for the application of the foliar sprays included backpack, motorised hand-gun and mist-blower. The high residues of captan and THPI detected in control samples in the 1991 Israel trials can be explained by a captan spray applied 26 days before the start of the trial. Sampling dates were not reported in one of the 1992 French trials. Captan was applied to pears by motorised knapsack and mist-blower. Plot sizes ranged from 1 to 12 trees.

Foliar sprays were applied to stone fruit by hand gun, knapsack sprayer, high-volume sprayer and mist-blower. The plot sizes were 1-4 trees for cherries, 1 tree to a plot of 4900 $m^2$ for nectarines and peaches and 1-64 trees for plums. For apricots most plots were 1-6 trees with the largest 93 trees.

For the aerial application of captan to blueberries plot sizes were about 2 ha. Captan was applied as a foliar spray to strawberries with backpacks, motorised sprayers and mist-blowers. The Belgian and Netherlands trials were in glasshouses while plastic tunnels were used in the Israel trials. All the other trials were in the field. Methods of irrigation included both furrow and sprinkler. Supervised grape trials were located at various sites in Australia, Brazil, Chile, France, Germany, Japan (including indoors) and the USA. Application equipment included knapsack sprayers, motorised airblast sprayers and mist-blowers. Plot sizes ranged from 3 to 300 $m^2$. Plot sizes for the USA raspberry trials were 39-1500 $m^2$ with application equipment ranging from backpack sprayers to hydraulic boom sprayers.

In cucumber, melon and squash trials captan was applied by knapsack sprayers, ground sprayers, tractor mounted boom sprays and aircraft. The plots ranged in size from a single 7.6 m row to an area of 93 $m^2$ for cucumbers, 11-61 $m^2$ for ground application to melons (0.4 ha for aerial application) and 15-23 $m^2$ for squash. In trials in Japan, captan was applied to both field and glasshouse cucumbers and melons.

Supervised trials were reported on tomatoes in Brazil, Greece, Israel, Japan (outdoor and glasshouse), Mexico and the USA. Plot sizes ranged from 7 to 93 $m^2$ for ground application (334 $m^2$ for aerial spraying). Ground application was by backpack sprayer or tractor mounted boom.

Plot sizes for potato trials were 13 - 100 $m^2$. Foliar application of captan was by knapsack sprayers, hand-held boom sprayers and motorised sprayers with hand gun. Other trials were with broadcast application to open furrows and seed treatment.

Trials were reported from Germany for the pre-emergent treatment of indoor-grown radishes and chives. Only summaries were provided.

Supervised trials on almonds grown in California, USA, were reported. Plot sizes were 41-1300 $m^2$ for ground applications with hand guns and airblast sprayers, and 0.8-2.2 ha for aerial applications.

Table 19. Residues of captan and THPI in citrus fruit after foliar applications of WP formulations. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | PHI, days | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| **Lemons** | | | | | | | |
| USA (AZ), 1986 (Lisbon) | 9.0 | | 2 | 0 | 7.0<br>8.5 | 0.18<br>0.16 | TMN603A |
| USA (CA), 1986 (Eureka) | 9.0 | | 2 | 0 | 4.6<br>4.5 | 0.08<br>0.08 | TMN603A |
| **Mandarins** | | | | | | | |
| Japan, 1983 (Miyakawa) (INDOOR) Pulp | 6.7 | 1.3 | 5 | 1<br>3<br>7 | 0.76<br>0.96<br>0.60 | | TMN602A |
| Japan, 1983 (Miyakawa) (INDOOR) Peel | 6.7 | 1.3 | 5 | 1<br>3<br>7 | 11<br>17<br>20<br>c0.44 | | TMN602A |
| Japan, 1983 (Miho-Wase) (INDOOR) Pulp | 6.7 | 1.3 | 5 | 1<br>3<br>4 | 1.4<br>0.40<br>0.28<br>c0.02 | | TMN602A |
| Japan, 1983 (Miho-Wase) (INDOOR) Peel | 6.7 | 1.3 | 5 | 1<br>3<br>4 | 32<br>18<br>14<br>c0.44 | | TMN602A |
| Japan, 1983 (Miyakawa) Pulp | 6.7 | 1.3 | 5 | 1<br>3<br>7 | 0.39<br>0.16<br>0.11 | | TMN602A |
| Japan, 1983 (Miyakawa) Peel | 6.7 | 1.3 | 5 | 1<br>3<br>7 | 9.8<br>15<br>19<br>c0.19 | | TMN602A |
| Japan, 1983 (Miho-Wase) Pulp | 6.7 | 1.3 | 5 | 1<br>3<br>4 | 0.30<br>0.06<br>0.10<br>c0.006 | | TMN602A |
| Japan, 1983 (Miho-Wase) Peel | 6.7 | 1.3 | 5 | 1<br>3<br>4 | 30<br>14<br>9.2<br>c0.31 | | TMN602A |
| **Oranges** | | | | | | | |
| Brazil, 1992 (Valencia) | | 0.12 | 3 | 7<br>28 | 0.34<br>0.16 | <0.05<br><0.05 | RJ1478B |
| Brazil, 1992 (Valencia) | | 0.24 | 3 | 7<br>28 | 0.48<br>0.26 | <0.05<br><0.05 | RJ1478B |
| Brazil, 1992 (Valencia) | | 0.12 | 3 | 7<br>28 | 0.17<br>0.14 | <0.05<br><0.05 | RJ1478B |
| Brazil, 1992 (Valencia) | | 0.24 | 3 | 7<br>28 | 0.92<br>0.36 | <0.05<br><0.05 | RJ1478B |
| Brazil, 1992 (Valencia) | 0.5 | 0.12 | 3 | 7<br>28 | 0.07<br>0.10 | | BR-14-91-S-005-P |
| Brazil, 1992 (Valencia) | 1.0 | 0.24 | 3 | 7<br>28 | 0.09<br>0.10 | | BR-14-91-S-005-P |
| Brazil, 1992 (Valencia) | 0.5 | 0.12 | 3 | 7<br>28 | 0.06<br>0.02 | | BR-14-91-S-006-P |
| Brazil, 1992 (Valencia) | 1.0 | 0.24 | 3 | 7<br>28 | 0.27<br>0.11 | | BR-14-91-S-006-P |
| Spain, 1991 (Valencia) | 6.3 | 0.13 | 1 | 10<br>20<br>28 | 0.40<br>0.30<br>0.39 | <0.05<br><0.05<br><0.05 | RJ1173B |
| Spain, 1991 (Valencia) | 7.5 | 0.15 | 1 | 10<br>20<br>28 | 1.0<br>0.56<br>0.44 | <0.05<br><0.05<br><0.05 | RJ1173B |

| Country (location), year (variety) | Application | | | PHI, days | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Spain, 1991 (Valencia) | 6.3 | 0.13 | 1 | 10<br>20<br>28 | 2.1<br>1.7<br>1.5 | <0.05<br><0.05<br><0.05 | RJ1173B |
| Spain, 1991 (Valencia) | 7.5 | 0.15 | 1 | 10<br>20<br>28 | 2.7<br>1.7<br>1.7 | <0.05<br><0.05<br><0.05 | RJ1173B |
| USA (FL), 1982 (Valencia) | 6.7 | | 1 | 0 | 0.50 | 0.12 | TMN603B |
| USA (CA), 1986 (Valencia) | 2×9.0<br>1×5.6 | | 3 | 0 | 2.0, 1.5 | 0.03,<br>0.02 | TMN603B |
| USA (AZ), 1986 (Valencia) | 2×9.0<br>1×5.6 | | 3 | 0 | 2.3, 2.3 | 0.04,<br>0.03 | TMN603B |
| USA (CA), 1983 (Navel) | 12 | | 2 | 0 | 2.7, 2.4 | 1.8, 1.3 | TMN603B |
| **Grapefruit** | | | | | | | |
| USA, (AZ) 1986 (Marsh/Red Blush) | 9.0 | | 2 | 0 | 2.4<br>4.2 | 0.05<br>0.09 | TMN603C |

c: control

Table 20. Residues of captan and THPI in apples after foliar applications of various captan formulations and after post-harvest dipping. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Argentina, 1984 (Red Delicious) | WP | 7.0 | 0.16 | 2 | 14 | 0.0005,<br>0.0003,<br>0.0004 | 0.001,<br>0.0009,<br>0.0007 | R-7108 |
| Australia, 1991 (Granny Smith) | WG | | 0.12 | 7<br>6<br>5 | 7<br>14<br>27 | 3.6<br>3.7<br>1.0 | | S38990 91-50 |
| Australia, 1991 (Granny Smith) | WG | | 0.25 | 7<br>6<br>5 | 7<br>14<br>27 | 8.1<br>7.9<br>2.0 | | S38990 91-50 |
| Brazil, 1992 (Gala) | SC | | 0.06 | 11 | 1<br>7 | 0.06<br><0.05 | 0.10<br>0.06 | RJ1419B |
| Brazil, 1992 (Gala) | SC | | 0.12 | 11 | 1<br>7 | 0.44<br>0.11 | 0.11<br>0.05 | RJ1419B |
| Brazil, 1992 (Gala) | SC | | 0.24 | 11 | 1<br>7 | 0.55<br>0.13 | 0.20<br>0.10 | RJ1419B |
| Brazil, 1992 (Gala) | WP | | 0.06 | 11 | 1<br>7 | 0.15<br>0.12 | 0.11<br><0.05 | RJ1419B |
| Brazil, 1992 (Gala) | WP | | 0.12 | 11 | 1<br>7 | 0.68<br>0.38 | 0.18<br>0.13 | RJ1419B |
| Brazil, 1992 (Gala) | WP | | 0.24 | 11 | 1<br>7 | 1.2<br>0.31 | 0.20<br>0.18 | RJ1419B |
| Brazil, 1992 (Fuji) | SC | | 0.06 | 11 | 1<br>7 | 0.14<br>0.24<br>c0.08 | 0.07<br>0.05<br>c<0.05 | RJ1419B |
| Brazil, 1992 (Fuji) | SC | | 0.12 | 11 | 1<br>7 | 1.0<br>0.19 | 0.38<br>0.08 | RJ1419B |
| Brazil, 1992 (Fuji) | SC | | 0.24 | 11 | 1<br>7 | 2.1<br>0.33 | 0.53<br>0.09 | RJ1419B |
| Brazil, 1992 (Fuji) | WP | | 0.06 | 11 | 1<br>7 | 1.7<br>0.14 | 0.61<br><0.05 | RJ1419B |
| Brazil, 1992 (Fuji) | WP | | 0.12 | 11 | 1<br>7 | 2.5<br>0.13 | 0.55<br>0.06 | RJ1419B |
| Brazil, 1992 (Fuji) | WP | | 0.24 | 11 | 1<br>7 | 4.6<br>0.30 | 0.92<br>0.09 | RJ1419B |
| Brazil, 1992 (Fuji) | SC | | 0.06 | 10 | 1<br>7 | 1.0<br>0.27<br>c0.13 | 0.12<br>0.09<br>c<0.05 | RJ1419B |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Brazil, 1992 (Fuji) | SC | | 0.12 | 10 | 1<br>7 | 1.4<br>0.47 | 0.12<br><0.05 | RJ1419B |
| Brazil, 1992 (Fuji) | SC | | 0.24 | 10 | 1<br>7 | 1.7<br>0.54 | 0.15<br><0.05 | RJ1419B |
| Brazil, 1992 (Fuji) | WP | | 0.06 | 10 | 1<br>7 | 3.1<br>0.22 | 0.22<br>0.33 | RJ1419B |
| Brazil, 1992 (Fuji) | WP | | 0.12 | 10 | 1<br>7 | 4.1<br>0.30 | 0.18<br><0.05 | RJ1419B |
| Brazil, 1992 (Fuji) | WP | | 0.24 | 10 | 1<br>7 | 5.2<br>0.45 | 0.19<br>0.05 | RJ1419B |
| Canada, 1991 (Macintosh) | WP | 3.0 | 0.13 | 10 | 6<br>13<br>20 | 3.2<br>2.1<br>2.6 | 0.08<br>0.05<br><0.05 | RJ1190B |
| Canada, 1991 (Macintosh) | WP | 3.0 | 0.13 | 10 | 6<br>13<br>20 | 3.5<br>3.9<br>1.7 | 0.09<br>0.08<br><0.05 | RJ1190B |
| Canada, 1991 (Macintosh) | WP | 6.0 | 0.25 | 10 | 6<br>13<br>20 | 4.8<br>4.2<br>3.5 | 0.11<br>0.09<br>0.07 | RJ1190B |
| Canada, 1991 (Indared) | WP | 3.0 | 0.13 | 10 | 7<br>14<br>21 | 2.9<br>2.9<br>1.2 | 0.05<br><0.05<br><0.05 | RJ1190B |
| Canada, 1991 (Indared) | WP | 3.0 | 0.13 | 10 | 7<br>14<br>21 | 2.9<br>1.1<br>2.7 | <0.05<br><0.05<br><0.05 | RJ1190B |
| Canada, 1991 (Indared) | WP | 6.0 | 0.25 | 10 | 7<br>14<br>21 | 6.5<br>2.3<br>5.4 | 0.09<br>0.08<br>0.08 | RJ1190B |
| Canada, 1991 (Macintosh) | WP | 3.0 | 0.13 | 10 | 6<br>13 | 4.0<br>4.5 | 0.06<br>0.05 | RJ1190B |
| Canada, 1991 (Macintosh) | WP | 3.0 | 0.13 | 10 | 6<br>13<br>21 | 4.1<br>4.5<br>4.5 | 0.07<br><0.05<br><0.05 | RJ1190B |
| Canada, 1991 (Macintosh) | WP | 6.0 | 0.25 | 10 | 6<br>13<br>21 | 9.5<br>9.1<br>7.7 | 0.11<br>0.08<br>0.07 | RJ1190B |
| Canada, 1991 (Red Delicious) | WP | 3.0 | 0.13 | 10 | 7<br>14<br>21 | 2.8<br>1.5<br>0.99 | 0.05<br><0.05<br><0.05 | RJ1190B |
| Canada, 1991 (Red Delicious) | WP | 3.0 | 0.13 | 10 | 7<br>14<br>21 | 4.2<br>3.3<br>2.8 | 0.06<br><0.05<br><0.05 | RJ1190B |
| Canada, 1991 (Red Delicious) | WP | 6.0 | 0.25 | 10 | 7<br>7<br>14<br>14<br>21<br>21 | 6.9<br>c0.22<br>5.5<br>c0.09<br>4.8<br>c0.06 | 0.10<br>c<0.05<br>0.10<br>c<0.05<br>0.05<br>c<0.05 | RJ1190B |
| Chile, 1992 (Granny Smith) | WP | 3.6 | 0.15 | 1 | 29<br>60 | 0.44<br>0.13 | <0.05<br><0.05 | RJ1302B |
| Chile, 1992 (Star King) | WP | 3.6 | 0.15 | 1 | 28<br>59 | 0.37<br>0.05 | 0.06<br><0.05 | RJ1302B |
| Chile, 1992 (Granny Smith) | WP | 3.6 | 0.15 | 1 | 28<br>59 | 0.46<br>0.15 | <0.05<br><0.05 | RJ1302B |
| Chile, 1991 (Red King Oregon) | WP | | 0.13 | 3 | 120 | 0.01, 0.04, 0.02, <0.01 | <0.1, <0.1, <0.1, <0.1 | R-6986 |
| France, 1997 (Royal Gala) | SC | 1.5 | 0.15 | 9 | 0<br>14 | 2.6<br>0.49 | | R-9675 |
| France, 1997 (Golden) | SC | 1.5 | | 9 | 0<br>14 | 3.2<br>2.1 | | R-9675 |
| France, 1992 (Golden Delicious) | WDG | | 0.15 | 12 | 0<br>0 | 2.8, 5.5, 3.8, 4.0 | <0.02 (2)<br><0.02 (2) | R-7149 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 22<br>22<br>44<br>44 | 1.6, 1.1,<br>2.2, 3.8<br>1.2, 1.0,<br>1.1, 0.95<br>c0.10,<br>c<0.02,<br>c<0.02 | <0.02 (2)<br><0.02 (2)<br><0.02 (2)<br><0.02 (2)<br>c<0.02 (3) | |
| France, 1992 (Golden Delicious) | WDG | | 0.15 | 10 | 0<br>0<br>?<br>?<br>?<br>? | 5.9, 3.4,<br>5.3, 3.1<br>3.1, 3.3,<br>0.08, 2.7<br>1.08, 0.61,<br>1.1, 1.1<br>c<0.02,<br>c3.4, c0.04 | <0.02 (2)<br><0.02 (2)<br><0.02 (2)<br><0.02 (2)<br><0.02 (2)<br><0.02 (2)<br>c<0.02 (3) | R-7149 |
| France, 1992 (Golden Delicious) | WDG | | 0.15 | 15 | 0<br>0<br>11<br>11<br>31<br>31 | 3.4, 13, 9.7,<br>6.8<br>5.6, 5.5,<br>4.8, 3.9<br>2.8, 4.1<br>3.7, 2.8<br>c0.11,<br>c0.03,<br>c0.83 | | R-7149 |
| France, 1992 (Golden Delicious) | WDG | | 0.15 | 14 | 0<br>0<br>17<br>17<br>36<br>36 | 2.1, 4.9,<br>1.6, 3.3<br>0.71, 3.0,<br>1.5, 0.86<br>1.0, 2.1,<br>2.2, 0.67<br>c0.07,<br>c<0.02,<br>c<0.02 | | R-7149 |
| France, 1991 (Golden) | WP | 1.5 | | 16 | 30<br>39 | 3.0<br>2.4 | 0.08<br>0.07 | RJ1261B |
| France, 1991 (Ozark Gold) | WP | 1.9 | | 10 | 29<br>41 | 0.12<br>0.06 | <0.05<br><0.05 | RJ1261B |
| France, 1991 (Ozark Gold) | WP | 1×0.75<br>9×1.5 | | 10 | 31<br>40 | 0.60<br>0.37 | <0.05<br><0.05 | RJ1261B |
| Germany, 1994 (Elstar) | WP | 1.9 | 0.15 | 15 | 0<br>2<br>3<br>7<br>14 | 5.0<br>4.3<br>3.5<br>4.5<br>2.3 | <0.2<br>-<br>-<br>-<br>1.1 | R-7784 |
| Germany, 1994 (Cox's Orange) | WP | 1.9 | 0.15 | 15 | 0<br>2<br>3<br>7<br>14 | 3.1<br>3.2<br>3.0, 2.1<br>2.9, 2.8<br>2.9, 1.0 2.6 | <0.20<br>-<br>-<br>-<br>0.83 | R-7784 |
| Germany, 1994 (Royal Gala) | WP | 1.5 | 0.15 | 12 | 0<br>2<br>3<br>7<br>14 | 2.0<br>2.1<br>1.4<br>0.84<br>0.76 | <0.2<br>-<br>-<br>-<br>0.36 | R-7784 |
| Germany, 1994 (Indared) | WP | 1.5 | 0.15 | 12 | 0<br>2<br>3<br>7<br>14 | 4.5<br>4.1<br>2.0<br>2.0<br>1.3 | <0.2<br>-<br>-<br>-<br>0.43 | R-7784 |
| Germany, 1996 (Golden Delicious) | WDG | 1.1-1.3 | 0.12 | 15 | 0<br>7<br>14<br>21 | 2.6<br>1.4<br>1.6<br>1.1 | 0.79<br>-<br>0.77<br>- | R-9077 |
| Germany, 1996 (Gloster) | WDG | 1.2-1.3 | 0.12 | 15 | 0 | 2.0 | 1.1 | R-9077 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 6<br>13<br>20 | 1.2<br>1.2<br>1.0 | -<br>0.63<br>- | |
| Germany, 1996 (Golden Delicious) | WDG | 1-7-1.8 | 0.12 | 13 | 0<br>7<br>14<br>20<br>20 | 2.6, 4.3 3.3<br>1.3, 3.0 2.2<br>2.0, 2.4<br>2.7, 3.0,<br>2.2, 2.4 | 2.3<br>-<br>2.7<br>-<br>- | R-9077 |
| Germany, 1993 (Cox's Orange) | WP | 1.5 | 0.5 | 10 | 0<br>3<br>7<br>14<br>20 | 7.5, 7.0<br>11, 6.2<br>9.9, 6.7<br>2.5<br>2.3 | 0.33, 0.35<br>0.36, 0.42<br>0.40, 0.45<br>0.41<br>0.53 | RJ1592 |
| Germany, 1993 (Elstar) | WP | 1.5 | 0.5 | 10 | 0<br>0<br>3<br>3<br>7<br>13<br>20 | 6.3<br>c0.11<br>6.6<br>c0.11<br>3.2<br>3.1<br>1.8 | 0.25<br>c<0.05<br>0.27<br>c<0.05<br>0.29<br>0.28<br>0.30 | RJ1592 |
| Germany, 1993 (Indared) | WP | 1.5 | 0.5 | 10 | 0<br>3<br>6<br>13<br>20 | 1.6<br>1.4<br>1.1<br>0.82<br>0.90 | 0.07<br>0.06<br>0.07<br>0.05<br>0.09 | RJ1592 |
| Germany, 1993 (Cox's Orange) | WDG | 1.5 | 0.5 | 10 | 0<br>3<br>7<br>14<br>20 | 8.8, 11<br>10, 7.1<br>10, 7.9<br>2.8<br>2.4 | 0.31, 0.44<br>0.32, 0.37<br>0.38, 0.44<br>0.45<br>0.58 | RJ1592 |
| Germany, 1993 (Elstar) | WDG | 1.5 | 0.5 | 10 | 0<br>3<br>7<br>13<br>20 | 5.5<br>5.3<br>3.8<br>4.0<br>1.7 | 0.26<br>0.27<br>0.27<br>0.36<br>0.43 | RJ1592 |
| Germany, 1993 (Indared) | WDG | 1.5 | 0.5 | 10 | 0<br>3<br>6<br>13<br>20 | 2.7<br>2.6<br>1.7<br>1.4<br>0.99 | 0.11<br>0.08<br>0.08<br>0.09<br>0.14 | RJ1592 |
| Germany, 1991 (Red King) | WP | 1.6 | | 10 | 0<br>3<br>7<br>14<br>19 | 3.1<br>2.7<br>4.1<br>2.7<br>1.6 | 0.52<br>0.44<br>0.61<br>0.47<br>0.23 | 91JH045F |
| Germany, 1991 (Red King) | SG | 1.6 | | 10 | 0<br>3<br>7<br>14<br>19 | 3.0<br>4.8<br>4.5<br>2.9<br>2.1 | 0.53<br>0.69<br>0.74<br>0.49<br>0.32 | 91JH045F |
| Germany, 1991 (Red King) | WP | 1.6 | | 12 | 0<br>3<br>7<br>14<br>14<br>20 | 2.7<br>5.0<br>2.0<br>1.5<br>c0.03<br>1.7 | 0.30<br>0.42<br>0.14<br>0.12<br>c<0.05<br>0.17 | 91JH045F |
| Germany, 1991 (Red King) | SG | 1.6 | | 12 | 0<br>3<br>7<br>14<br>14<br>20 | 2.8<br>4.4<br>2.8<br>1.8<br>c0.03<br>1.4 | 0.31<br>0.31<br>0.17<br>0.21<br>c<0.05<br>0.11 | 91JH045F |
| Germany, 1991 (Red King) | WP | 1.6 | | 10 | 0<br>0 | 2.3<br>c0.03 | 0.24<br>c<0.05 | 91JH045F |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 3<br>7<br>14<br>22 | 2.2<br>2.0<br>1.0<br>0.95 | 0.26<br>0.53<br>0.27<br>0.21 | |
| Germany, 1991 (Red King) | SG | 1.6 | | 10 | 0<br>0<br>3<br>7<br>14<br>22 | 3.8<br>c0.03<br>3.2<br>2.2<br>1.2<br>1.5 | 0.50<br>c<0.05<br>0.50<br>0.52<br>0.34<br>0.33 | 91JH045F |
| Germany, 1996 (Golden Delicious) | WP | 1.3 | 0.15 | 12 | 0<br>7<br>14<br>21 | 2.6<br>1.4<br>1.6<br>1.1 | 0.79<br>-<br>0.77<br>- | R9784 |
| Germany, 1996 (Gloster) | WP | 1.3 | 0.15 | 12 | 0<br>7<br>14<br>21 | 2.0<br>1.2<br>1.2<br>1.0 | 1.1<br>-<br>0.63<br>- | R9784 |
| Germany, 1996 (Golden Delicious) | WP | 1.8 | 0.15 | 13 | 0<br>7<br>14<br>21 | 3.1<br>2.6<br>2.2<br>1.8 | 2.3<br>-<br>2.7<br>- | R9784 |
| Hungary, 1992 (Gloster) R-7006 | WG | 1.6 | 0.16 | 9 | 10 | 1.2, 1.2,<br>1.2, 1.5, 1.2 | | R-7006 |
| Israel, 1991 (Starking)[1] | WP | 2.5 | 0.13 | 4 | 0<br>0<br>23<br>23<br>36<br>36<br>83<br>83 | 1.6, 1.6,<br>2.4, 2.5<br>0.73, 0.85,<br>0.90, 0.24<br>0.55, 0.17,<br>0.50, 0.23<br>0.89, 0.74,<br>0.78, 0.39 | 0.25, 0.36,<br>0.39, 0.41<br>0.56, 0.13,<br>0.10,<0.0<br>2<br><0.02<br><0.1<br>0.22<br><0.02<br><0.02<br><0.02 | CT/36/92 |
| Israel, 1991 (Starking) | WP | 5 | 0.25 | 4 | 0<br>0<br>23<br>23<br>36<br>36<br>83<br>83 | 2.0, 1.9,<br>2.0, 2.2<br>0.82, 0.91,<br>0.85, 0.64<br>0.57, 0.88,<br>0.64, 0.14<br>0.64, 2.0,<br>1.8, 4.9 | 0.50, 0.20,<br>0.31, 0.87<br><0.1, <0.1<br>-, <0.02<br><0.1,<br>0.35,<br><0.02 (2)<br><0.02 (2)<br><0.02 (2) | CT/36/92 |
| Japan, 1971 (Golden) | WP | | 0.1 | 4 | 77 | 0.06 | | TMN-575A |
| Japan, 1971 (Golden) | WP | | 0.1 | 8 | 56 | 0.08 | | TMN-575A |
| Japan, 1972 (Starking) | WP | | 1.3 | 13 | 1<br>3<br>5<br>10 | 0.02<br>0.02<br>0.04<br>0.01 | | TMN-575A |
| Japan, 1992 (Fuji) | WP | 8 | 1.3 | 8 | 3<br>7<br>14<br>21 | 1.7<br>1.8<br>1.3<br>1.8 | | TMN-575A |
| Japan, 1992 (Fuji) | WP | 8 | 1.3 | 8 | 3<br>7<br>14<br>21 | 1.6<br>4.2<br>2.1<br>1.5 | | TMN-575A |
| Japan, 1991 (Fuji) | WP | 8 | 0.13 | 8 | 3<br>7 | 10<br>13 | 0.23<br>0.28 | RJ1185B |

[1] Controls for days 0, 23 and 36 contained captan at >0.4-0.49 mg/kg and THPI at <0.1 to 0.33 mg/kg. It should be noted that captan was sprayed on the plots 26 days before the trial commenced.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 14<br>21 | 4.6<br>3.5 | 0.24<br>0.14 | |
| Japan, 1991 (Fuji) | WP | 8 | 0.13 | 8 | 14<br>21 | 7.2<br>5.3 | 0.32<br>0.29 | RJ1185B |
| Japan, 1991 (Tsurgaru) | WP | 8 | 0.13 | 8 | 14<br>21 | 3.8<br>3.0<br>c0.09 | 0.32<br>0.44<br>c<0.05 | RJ1185B |
| Netherlands, 1991 (Elstar) | WDG | 0.96 | 0.5 | 16 | 0<br>0<br>0<br>7<br>7<br>14<br>14<br>21<br>21 | 2.7, 2.1<br>1.4, 2.8<br>c0.16<br>0.99, 0.59<br>0.93, 1.2<br>1.2, 0.93,<br>0.83, 0.90<br>1.0, 0.68<br>0.41, 0.38 | 0.06, 0.07<br>0.08, 0.08<br>c0.12<br>0.11, 0.13<br>0.12, 0.11<br>0.11, 0.09<br>0.13, 0.16<br>0.23, 0.13<br>0.25, 0.13 | RJ1115B |
| Netherlands, 1991 (Elstar) | WP | 1.0 | 0.5 | 16 | 0<br>0<br>0<br>7<br>7<br>14<br>14<br>21<br>21 | 1.5, 2.6<br>1.4, 2.6<br>c0.16<br>0.67, 1.7<br>1.7, 1.1<br>0.88, 1.1<br>1.8, 1.4<br>0.40, 0.54<br>0.55, 0.37 | 0.05, 0.05<br>0.05<br><0.05<br>c0.12<br>0.13, 0.22<br>0.20, 0.14<br>0.12, 0.09<br>0.16, 0.10<br>0.15, 0.15<br>0.14, 0.15 | RJ1115B |
| Netherlands, 1991 (Elstar) | DF | 1.1 | 0.5 | 16 | 0<br>0<br>0<br>7<br>7<br>14<br>14<br>21<br>21 | 2.5, 1.3<br>1.3, 4.0<br>c0.16<br>2.3, 2.4<br>1.0, 1.4<br>0.85, 1.1<br>0.86, 1.5<br>0.84, 0.56<br>0.66, 0.58 | 0.06, 0.05<br>0.05, 0.12<br>c0.12<br>0.14, 0.14<br>0.13, 0.14<br>0.12, 0.10<br>0.13, 0.16<br>0.19, 0.11<br>0.17, 0.18 | RJ1115B |
| Netherlands, 1991 (Cox) | WDG | 0.96 | 0.5 | 16 | 0<br>0<br>0<br>14<br>14<br>14<br>21<br>21<br>21 | 1.8, 1.6<br>2.2, 1.8<br>c0.19<br>0.38, 0.57<br>1.1, 0.60<br>c0.18<br>0.18, 0.12<br>0.23, 0.26<br>c0.06 | 0.13, 0.08<br>0.14, 0.13<br>c0.35<br>0.08, 0.12<br>0.12, 0.11<br>c<0.05<br>0.09, 0.06<br>0.11, 0.11<br>c0.06 | RJ1115B |
| Netherlands, 1991 (Cox) | WP | 1.0 | 0.5 | 16 | 0<br>0<br>0<br>14<br>14<br>21<br>21<br>21 | 3.1, 2.7<br>2.9, 3.0<br>c0.19<br>1.1, 1.3<br>c0.18<br>1.0, 0.34<br>0.48, 0.48<br>c0.06 | 0.13, 0.11<br>0.09, 0.11<br>c0.35<br>0.28, 0.15<br>c<0.05<br>0.11, 0.14<br>0.12, 0.13<br>c0.06 | RJ1115B |
| Netherlands, 1991 (Cox) | DF | 1.1 | 0.5 | 16 | 0<br>0<br>0<br>14<br>14<br>14<br>21<br>21<br>21 | 1.2, 1.4<br>1.3, 1.3<br>c0.19<br>0.55, 0.54<br>1.6, 1.7<br>c0.18<br>0.31, 0.63<br>0.33, 0.77<br>c0.06 | 0.12, 0.11<br>0.12, 0.09<br>c0.35<br>0.13, 0.08<br>0.15, 0.16<br>c<0.05<br>0.14, 0.15<br>0.14, 0.22<br>c0.06 | RJ1115B |
| Portugal, 1991 (Golden Delicious) | WP | 2.4 | 0.29 | 10 | 0<br>0<br>10 | 6.3, 5.1,<br>6.7, 6.7<br>2.6, 2.5, | <0.1, <0.1<br><0.1, <0.1<br><0.1, <0.1 | CT/37/92 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 10<br>21<br>21 | 4.4, 2.4<br>3.1, 2.9,<br>3.2, 2.5 | <0.1, <0.1<br><0.1, <0.1<br><0.1, <0.1 | |
| South Africa, 1992 (Starking) | SC | | 0.15 | 2 | 0<br>4<br>8<br>16<br>32<br>59 | 4.3<br>5.0<br>1.0<br>1.2<br>2.1<br>1.2 | 0.09<br>0.14<br>0.07<br>0.13<br>0.06<br>0.06 | RJ1416B |
| South Africa, 1992 (Granny Smith) | SC | | 0.08 | 2 | 4<br>8<br>16<br>59 | 2.0<br>2.0<br>2.0<br>0.15 | 0.13<br>0.09<br>0.11<br><0.05 | RJ1416B |
| South Africa, 1992 (Granny Smith) | SC | | 0.15 | 2 | 4<br>8<br>32<br>59 | 3.4<br>4.0<br>1.2<br>0.45 | 0.14<br>0.19<br><0.05<br><0.05 | RJ1416B |
| South Africa, 1992 (Starking) | SC | | 0.08 | 2 | 0<br>4<br>8<br>16<br>32<br>59 | 3.5<br>2.6<br>5.3<br>3.6<br>0.82<br>0.17 | 0.07<br>0.10<br>0.13<br>0.11<br><0.05<br><0.05 | RJ1416B |
| UK, 1986 (Cox) | WP | 2.9 | 0.29 | 3 | 0<br>14<br>21 | 1.5<br>0.5<br>0.3 | 0.2<br>0.2<br>0.1 | 197/AL/86/FF |
| UK, 1986 (Cox) | WP | 2.9 | 0.29 | 4 | 0<br>14<br>21 | 2.5<br>1.2<br>0.4 | 0.2<br>0.2<br>0.1 | 197/AL/86/FF |
| UK, 1986 (Golden Delicious) | WP | 2.9 | 0.29 | 4 | 0<br>14<br>21 | 3.8<br>1.4<br>0.4 | 0.1<br>0.1<br>0.1 | 197/AL/86/FF |
| UK, 1986 (Cox) | WP | 0.66 | 1.3 | 5 | 0<br>14<br>21 | 1.1<br>0.2<br>0.3 | 0.2<br>0.2<br>0.1 | 197/AL/86/FF |
| UK, 1991 (Cox) | WP | 2.7 | | 9 | 0<br>14<br>28<br>42<br>56 | 7.7<br>2.4<br>1.4<br>1.9<br>1.1 | 0.19<br>0.07<br>0.10<br>0.07<br>0.05 | RJ1134B |
| UK, 1991 (Cox) | WP | 2.7 | | 3 | 0<br>7<br>21<br>35 | -<br>6.6<br>4.4<br>1.6 | -<br>0.25<br>0.15<br>0.07 | RJ1134B |
| UK, 1991 (Cox) | WDG | 2.7 | | 9 | 0<br>14<br>28<br>42<br>56 | 4.9<br>2.5<br>2.6<br>0.98<br>0.31 | 0.21<br>0.14<br>0.09<br><0.05<br><0.05 | RJ1134B |
| UK, 1991 (Cox) | WP | 2.7 | | 10 | 0<br>14<br>28<br>42<br>56 | 6.3<br>3.1<br>1.4<br>0.51<br>0.38 | 0.23<br>0.14<br>0.07<br>0.05<br><0.05 | RJ1134B |
| UK, 1991 (Cox) | WP | 2.7 | | 3 | 0<br>7<br>14<br>35 | -<br>3.1<br>2.4<br>0.42 | -<br>0.11<br>0.12<br>0.05 | RJ1134B |
| UK, 1991 (Cox) | WDG | 2.7 | | 10 | 0<br>14<br>28<br>42<br>56 | 1.3<br>0.72<br>0.33<br>0.11<br>0.15 | 0.07<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1134B |
| UK, 1991 (Cox) | WP | 2.7 | | 10 | 0 | 8.3 | 0.32 | RJ1134B |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 14<br>14<br>28<br>42<br>42<br>56 | 3.9<br>c0.06<br>3.3<br>1.9<br>c0.07<br>0.81 | 0.15<br>c<0.05<br>0.17<br>0.12<br><0.05<br>0.06 | |
| UK, 1991 (Cox) | WP | 2.7 | | 3 | 0<br>7<br>14<br>14<br>35<br>42 | -<br>8.7<br>4.2<br>c0.06<br>3.0<br>c0.07 | 0<br>0.46<br>0.36<br>c<0.05<br>0.21<br>c<0.05 | RJ1134B |
| UK, 1991 (Cox) | WDG | 2.7 | | 10 | 0<br>14<br>14<br>28<br>42<br>42<br>56 | 4.6<br>0.91<br>c0.06<br>0.58<br>0.37<br>c0.07<br>0.18 | 0.33<br><0.05<br>c<0.05<br><0.05<br><0.05<br>c<0.05<br><0.05 | RJ1134B |
| UK, 1990 (Cox) | WP | 2.7 | | 9 | 7<br>21 | 4.4<br>1.3 | 0.15<br>0.08 | RJ1014B |
| UK, 1990 (Cox) | WP | 2.7 | | 8 | 12<br>26<br>39<br>53<br>67 | 0.45<br>1.0<br>0.58<br>0.24<br>0.14 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1014B |
| UK, 1990 (Cox) | WP | 2.7 | | 3 | 7 | 4.2 | 0.08 | RJ1014B |
| UK, 1990 (Cox) | WP | 1×2.8<br>7×2.7 | | 8 | 12<br>26<br>39<br>53<br>67 | 2.0<br>1.1<br>0.98<br>0.51<br>0.22 | 0.08<br><0.05<br>0.06<br><0.05<br><0.05 | RJ1014B |
| UK, 1990 (Cox) | WP | 2.7 | | 3 | 6<br>20<br>35 | 4.8<br>1.1<br>0.62 | 0.14<br>0.09<br>0.09 | RJ1014B |
| UK, 1990 (Cox) | WP | 2.7 | | 8 | 11<br>38 | 2.5<br>0.68 | 0.07<br><0.05 | RJ1014B |
| UK, 1990 (Cox) | WP | 2.7 | | 3 | 6 | 0.98 | <0.05 | RJ1014B |
| UK, 1990 (Cox) | WDG | 2.7 | | 9 | 7<br>21 | 4.3<br>1.8 | 0.13<br>0.09 | RJ1014B |
| UK, 1990 (Cox) | WDG | 2.7 | | 8 | 12<br>26<br>39<br>53<br>67 | 3.7<br>1.0<br>0.7<br>0.54<br>0.11 | 0.11<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1014B |
| UK, 1990 (Cox) | WDG | 2.7 | | 8 | 12<br>26<br>39<br>53<br>67 | 2.2<br>0.85<br>0.87<br>0.37<br>0.21 | 0.07<br>0.07<br>0.07<br><0.05<br><0.05 | RJ1014B |
| UK, 1990 (Cox) | WDG | 2.7 | | 8 | 11<br>25<br>38 | 1.6<br>0.69<br>0.29 | <0.05<br><0.05<br><0.05 | RJ1014B |
| USA (MI) 1990 (Yellow Delicious) | WP | Po 1.5 g/l for 10 sec | | | 0<br>30 | 2.2<br>2.9 | <0.05<br>0.12 | RR 91-028B Cold storage at 1°C for 30 days |
| USA (WV) 1990 (Spartan) | WP | Po 1.5 g/l for 3 min | | | 0<br>30 | 6.6<br>7.8 | 0.06<br>0.08 | RR 91-028B Cold storage at 1°C for 30 days |
| USA (MI), 1990 (Yellow Delicious) | WP | 3.4 | | 10 | 41<br>55<br>69 | 0.56<br>0.40<br>0.36 | <0.05<br><0.05<br><0.05 | RR 92-023B |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 83<br>97 | 0.15<br>0.06 | <0.05<br><0.05 | |
| USA (WV), 1990 (Spartan) | WP | 3.4 | | 10 | 14<br>21<br>28<br>42<br>55 | 1.5<br>0.87<br>0.45<br>0.09<br>0.06 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | RR 92-023B |
| USA (WA), 1986 (Winter Banana) | WP | 13 | | 8 | 0 | 9.1, 5.5 | 0.40, 0.43 | 051631-D |
| USA (WA) 1986 (Red Delicious) | WP | 4.5 | | 6<br>✈ | 0 | 0.86, 0.74, 0.42 | <0.05 (3) | 40189803 |
| USA (WA) 1986 (Red Delicious) | WP | 4.5 | | 6[1] | 0 | 2.2, 2.8, 1.9 | <0.05 (3) | 40189803 |
| USA (WA) 1986 (Red Delicious) | WP | 4.5 | | 8 | 0 | 5.5, 5.1 | 0.23, 0.21 | 056131-C |
| USA (WA) 1986 (Winter Banana) | WP | 4.5 | | 8 | 0 | 1.2, 5.2 | 0.37, 0.76 | 056131-C |
| USA (WA) 1986 (Red Delicious) | WP | 4.5 | | 7 | 7<br>14 | 5.2, 3.5<br>2.2, 3.3 | 0.21, 0.20<br>0.19, 0.28 | 056131-C |
| USA (MI) 1986 (Jonathan) | WP | 4.5 | | 8 | 0<br>7<br>14 | 3.4, 3.9<br>1.7, 1.3<br>2.2, 0.64 | <0.05 (2)<br><0.05 (2)<br><0.05 (2) | 056131-C |
| USA (NY) 1986 (McIntosh) | WP | 4.5 | | 8 | 0<br>7<br>14 | 4.7, 2.8<br>2.8, 2.5<br>2.8, 2.1 | 0.10, 0.09<br>0.12, 0.12<br>0.23, 0.10 | 056131-C |
| USA (CA), 1986 (Granny Smith) | WP | 4.5 | | 8 | 0 | 3.8, 4.9<br>c0.08 | 0.08, 0.13<br>c<0.05 | 056131-C |
| USA (NC), 1986 (Red Delicious) | WP | 4.5 | | 8 | 0 | 0.31, 1.5 | 0.06, 0.05 | 056131-C |
| USA (VA), 1986 (Golden Delicious) | WP | 4.5 | | 8 | 0 | 1.4, 0.41 | 0.07, ND | 056131-C |
| USA (WA) 1986 (Red Delicious) | WP | 8×4.5 + Po 1.5 g/l | | | 0 | 7.7, 7.0 | 0.35, 0.28 | 056131-C |
| USA (WA), 1986 (Red Delicious) | WP | 1.5 g/l Po/CT | | | 0 | 4.0, 4.0 | 0.08, 0.09 | 056131-C |
| USA (NY), 1986 (McIntosh) | WP | 8×4.5 + Po 1.5 g/l | | | 0 | 5.7, 5.9 | 0.16, 0.10 | 056131-C |
| USA (NY), 1986 (McIntosh) | WP | 1.5 g/l Po/CT | | | 0 | 2.5, 3.3 | 0.07, 0.10 | 056131-C |

✈ aerial application
[1] dilute spray

Table 21. Residues of captan and THPI in pears after foliar applications of various captan formulations and after post-harvest dipping. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Australia, 1991 (Packham's Triumph) | WG | | 0.13 | 5<br>4<br>3 | 6<br>14<br>27 | 2.5<br>1.9<br>1.4 | | S38990 91-48 |
| Australia, 1991 (Packham's Triumph) | WG | | 0.25 | 5<br>4<br>3 | 6<br>14<br>27 | 7.1<br>5.7<br>1.2 | | S38990 91-48 |
| Chile, 1992 (Berry Box) | WP | 3.6 | 0.12 | 1 | 26<br>57 | 1.1<br>0.10 | 0.06<br><0.05 | RJ1303B |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Chile, 1992 (Packams) | WP | 3.6 | 0.12 | 1 | 28<br>60 | 1.0<br>0.21 | 0.05<br><0.05 | RJ1303B |
| Chile, 1992 (Winter Nelly) | WP | 3.6 | 0.12 | 1 | 30<br>61 | 1.3<br>0.31 | <0.05<br><0.05 | RJ1303B |
| Germany, 1993 (William Christ) | WP | 1.5 | 0.5 | 1 | 0<br>3<br>7<br>14<br>20 | 3.8<br>1.4<br>1.8<br>1.3<br>0.67 | 0.08<br>0.05<br>0.05<br><0.05<br><0.05 | RJ1592 |
| Germany, 1993 (William Christ) | WG | 1.5 | 0.5 | 1 | 0<br>3<br>7<br>14<br>20 | 3.6<br>1.8<br>0.88<br>1.2<br>0.23 | 0.11<br>0.06<br><0.05<br><0.05<br><0.05 | RJ1592 |
| Italy, 1997 (Conference) | WG | 2.5-2.7 | 0.13 | 8 | 0<br>7<br>14 | 3.2<br>2.1<br>0.81 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Conference) | WG | 2.5-2.7 | 0.13 | 8 | 0<br>7<br>14 | 3.6<br>2.6<br>2.0 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Conference) | WG | 2.5-2.7 | 0.13 | 6 | 0<br>7<br>14 | 3.7<br>2.7<br>1.2 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Conference) | WP | 2.4-2.6 | 0.13 | 8 | 0<br>7<br>14 | 3.5<br>2.9<br>1.1 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Abate Fetel) | WG | 1.8-2.0 | 0.13 | 8 | 0<br>7<br>14 | 2.9<br>1.6<br>1.3 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Abate Fetel) | WG | 1.8-2.0 | 0.13 | 8 | 0<br>7<br>14 | 3.1<br>1.8<br>2.0 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Abate Fetel) | WG | 1.9-2.0 | 0.13 | 6 | 0<br>7<br>14 | 3.3<br>1.9<br>1.6 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Abate Fetel) | WP | 1.7-2.0 | 0.13 | 6 | 0<br>7<br>14 | 3.3<br>2.4<br>1.9 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Abate Fetel) | WG | 1.9-2.0 | 0.13 | 8 | 0<br>7<br>14 | 2.0<br>1.2<br>0.68 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Abate Fetel) | WG | 1.8-2.1 | 0.13 | 8 | 0<br>7<br>14 | 2.1<br>0.67<br>0.59 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Abate Fetel) | WG | 2.0-2.1 | 0.13 | 6 | 0<br>7<br>14 | 2.7<br>1.9<br>1.2 | | ERSA-DA-09.10/97 |
| Italy, 1997 (Abate Fetel) | WP | 1.9-2.0 | 0.13 | 8 | 0<br>7<br>14 | 2.5<br>1.1<br>0.72 | | ERSA-DA-09.10/97 |
| Japan, 1988 (Kousui) | WP | 6.7 | 0.13 | 5 | 3<br>7<br>14 | 0.68<br>0.50<br>0.44<br>c0.09 | | TMN-656A |
| Japan, 1988 (Kousui) | WP | 6.7 | 0.13 | 7 | 3<br>7<br>14 | 1.0<br>0.77<br>0.62<br>c0.09 | | TMN-656A |
| Japan, 1988 (Kousui) | WP | 6.7 | 0.13 | 9 | 3 | 1.3 | | TMN-656A |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 7<br>14 | 0.99<br>0.69<br>c0.09 | | |
| Japan, 1988 (Housui) | WP | 6.7 | 0.13 | 5 | 3<br>7<br>14 | 6.8<br>1.8<br>2.3<br>c0.04 | | TMN-656A |
| Japan, 1988 (Housui) | WP | 6.7 | 0.13 | 7 | 3<br>7<br>14 | 5.2<br>2.6<br>2.5<br>c0.04 | | TMN-656A |
| Japan, 1988 (Housui) | WP | 6.7 | 0.13 | 9 | 3<br>7<br>14 | 6.2<br>1.8<br>2.6<br>c0.04 | | TMN-656A |
| South Africa, 1992 (Bon Chretien) | SC | 2.1 | 0.08 | 2 | 32<br>40 | <0.05<br><0.05 | <0.05<br><0.05 | RJ1417B |
| South Africa, 1992 (Bon Chretien) | SC | 4.2 | 0.15 | 2 | 32<br>40 | 0.69<br>0.74 | <0.05<br><0.05 | RJ1417B |
| South Africa, 1992 (Packham's Triumph) | SC/WP | 2×4.2 SC, 2×4.2 WP | 0.10 | 4 | 105<br>112 | 0.15<br>0.07 | <0.05<br><0.05 | RJ1417B |
| UK, 1991 (Conference) | WDG | 2.7 | | 9 | 0<br>14<br>28<br>42<br>56 | 13<br>2.0<br>1.6<br>0.51<br>0.39 | 0.42<br>0.08<br>0.06<br><0.05<br><0.05 | RJ1171B |
| UK, 1991 (Conference) | WP | 2.7 | | 9 | 0<br>14<br>28<br>42<br>56 | 10<br>2.6<br>1.7<br>0.81<br>0.33 | 0.36<br>0.11<br>0.05<br><0.05<br><0.05 | RJ1171B |
| UK, 1991 (Conference) | WP | 2.7 | | 3 | 7<br>21<br>35 | 5.5<br>2.6<br>0.49 | 0.18<br>0.10<br><0.05 | RJ1171B |
| UK, 1991 (Comice) | WG | 2.7 | | 10 | 0<br>14<br>28<br>42<br>56 | 4.8<br>1.2<br>0.62<br>0.36<br>0.50 | 0.17<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1171B |
| UK, 1991 (Comice) | WP | 2.7 | | 10 | 0<br>14<br>28<br>42<br>56 | 6.5<br>1.9<br>1.4<br>0.89<br>0.22 | 0.18<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1171B |
| UK, 1991 (Comice) | WP | 2.7 | | 3 | 7<br>21<br>35 | 7.4<br>6.5<br>1.7 | 0.21<br>0.14<br><0.05 | RJ1171B |
| UK, 1990 (Conference) | WG | 2.7 | | 9 | 7<br>21<br>35<br>49<br>63 | 9.2<br>2.6<br>0.59<br>1.9<br>0.47 | 0.17<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1003B |
| UK, 1990 (Conference) | WP | 2.7 | | 9 | 7<br>21<br>35<br>49<br>63 | 7.3<br>3.1<br>1.4<br>0.73<br>0.18 | 0.14<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1003B |
| UK, 1990 (Conference) | WP | 2.7 | | 3 | 7<br>21<br>35 | 7.0<br>2.4<br>1.2 | 0.18<br>0.10<br><0.05 | RJ1003B |
| UK, 1990 (Conference) | WG | 2.7 | | 8 | 12<br>26<br>37<br>51 | 1.7<br>1.5<br>0.74<br>0.24 | <0.05<br><0.05<br><0.05<br><0.05 | RJ1003B |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 65 | 0.12 | <0.05 | |
| UK, 1990 (Conference) | WP | 2.7 | | 8 | 37<br>51<br>65 | 1.5<br>0.54<br>0.15 | <0.05<br><0.05<br><0.05 | RJ1003B |
| UK, 1990 (Conference) | WP | 2.7 | | 3 | 7<br>20<br>34 | 1.5<br>0.94<br>0.23 | <0.05<br><0.05<br><0.05 | RJ1003B |
| USA (WA), 1986 (Bartlett) | WP | 4.5 | | 5 | 0<br>7<br>14 | 4.3, 1.3<br>2.9, 2.9<br>1.3, 2.0 | <0.05 (2)<br><0.05 (2)<br><0.05 (2) | 056131-O |
| USA (CA), 1986 (Bartlett) | WP | 4.5 | | 5 | 0 | 0.90, 1.6 | <0.05 (2) | 056131-O |
| USA (NY), 1986 (Bartlett) | WP | 4.5 | | 5 | 0<br>7<br>14 | 2.9, 1.8<br>1.6, 1.5<br>1.5, 1.4 | <0.05 (2)<br><0.05 (2)<br>0.08, 0.11 | 056131-O |
| USA (MI), 1986 (Bartlett) | WP | 4.5 | | 5 | 0 | 5.4, 5.7 | <0.05 (2) | 056131-O |
| USA (WA), 1986 (Bartlett) | WP | 5×4.5 + Po dip 1.5 g/l | | | 0 | 11, 6.1 | <0.05 (2) | 056131-O |
| USA (CA), 1986 (Bartlett) | WP | 5×4.5 + Po dip 1.5 g/l | | | 0 | 2.6, 2.9 | <0.05 (2) | 056131-O |
| USA (WA), 1986 (Bartlett) | WP | Po dip 1.5 g/l | | | 0 | 11, 11 | 0.47, 0.34 | 056131-O |
| USA (CA), 1986 (Bartlett) | WP | Po dip 1.5 g/l | | | 0 | 4.7, 3.0 | 0.07, 0.06 | 056131-O |
| USA (WA), 1978 (Anjou) | WP | 6.7 | 0.12 | 2 | 0<br>1<br>3<br>7<br>14 | 1.6<br>1.0<br>0.5<br>0.6<br>0.3 | 2.3<br>1.4<br>0.16<br>1.3<br>0.05 | TMN-654A |
| USA (WA), 1978 (Bosc) | WP | 6.7 | 0.12 | 2 | 0<br>1<br>3<br>7<br>14 | 1.5<br>1.0<br>0.5<br>0.6<br>0.3 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | TMN-654A |

Table 22. Residues of captan and THPI in cherries after foliar applications of various captan formulations and after post-harvest dipping. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Belgium, 1991 (Gorsem-Kreik) | WP | 1.9 | 0.62 | 2 | 7 | 0.88, 0.39 | | R7025 |
| Canada, 1982 Sweet cherry (Bing) | WP | 2.4[1] | | 7 | -0<br>0<br>1<br>2<br>3<br>5<br>7<br>10<br>14 | 2.8<br>6.7<br>5.2<br>2.8<br>4.6<br>5.0<br>4.4<br>1.9<br>1.8<br>c0.01 | | Northover *et al.* 1986 |
| Canada, 1983 Sweet cherry (Bing) | WP | 4.5[1] | | 5 | -0<br>0<br>1<br>2<br>3<br>5 | 4.1<br>27<br>9.5<br>7.9<br>12<br>9.8 | | Northover *et al.* 1986 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 7<br>11<br>14 | 13<br>4.5<br>4.3<br>c0.04 | | |
| Canada, 1982 Sour cherry (Montmorency) | WP | 3.0 | 0.1 | 1 | 0<br>1<br>3<br>5<br>8<br>10<br>14 | 17<br>15<br>7.6<br>4.9<br>4.8<br>4.3<br>2.3<br>c0.18 | | Northover *et al.* 1986 |
| Canada, 1982 Sour cherry (Montmorency) | WP | 3.4[1] | | 7 | -0<br>0<br>1<br>2<br>3<br>5<br>7<br>10<br>14 | 10<br>13<br>8.1<br>6.2<br>7.0<br>9.7<br>8.6<br>5.5<br>4.8<br>c0.02 | | Northover *et al.* 1986 |
| Canada, 1983 Sour cherry (Montmorency) | WP | 3.4[1] | | 4 | -0<br>0<br>1<br>2<br>3<br>5<br>7<br>11<br>14 | 3.5<br>30<br>32<br>19<br>11<br>13<br>8.3<br>3.0<br>2.1<br>c0.01 | | Northover *et al.* 1986 |
| Germany, 1999 (Edelfinger) | WG | 0.57-0.59 | 0.12 | 3 | 0<br>3<br>5<br>7<br>14 | 2.6<br>1.9<br>0.98<br>1.1, 0.38<br>0.69 | | R11155 |
| Germany, 1999 (Schatten morelle) | WG | 0.57-0.59 | 0.12 | 3 | 7<br>15 | 0.82<br>0.08 | | R11155 |
| Germany, 1999 (Schatten morelle) | WG | 0.59-0.64 | 0.12 | 3 | 0<br>3<br>5<br>7<br>14 | 5.2<br>4.0<br>3.2<br>2.9<br>2.5 | | R11155 |
| Germany, 1999 (Johanna) | WG | 0.61-0.62 | 0.12 | 3 | 7<br>14 | 3.7<br>1.7 | | R11155 |
| Germany, 1975 (Frühe Ludwig) | WP | 2.1 | | 5 | 0<br>1<br>3<br>5 | 13<br>12<br>9.9<br>1.7 | | TMN-600B |
| Germany, 1975 (Schattenmorelle) | WP | 2.1 | | 5 | 0<br>1<br>3<br>5 | 14<br>15<br>8.4<br>8.4 | | TMN-600B |
| Germany, 1975 (Schattenmorelle) | WP | 2.1 | | 5 | 0<br>1<br>3<br>5 | 8.6<br>9.0<br>7.3<br>5.8 | | TMN-600B |
| Japan, 1990 (Takasago) | WP | 5.6 | 0.08 | 5 | 14<br>21 | 1.5<br>0.89<br>c0.005 | | TMN-601A |
| Japan, 1990 (Takasago) | WP | 7.0 | 0.1 | 4 | 14<br>21 | 1.3<br>0.71<br>c0.005 | | TMN-601A |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Japan, 1990 (Takasago) | WP | 7.0 | 0.1 | 5 | 14<br>21 | 1.7<br>0.63<br>c0.005 | | TMN-601A |
| Japan, 1990 (Satonishiki) | WP | 5.6 | 0.08 | 5 | 14<br>21 | 0.69<br>0.62<br>c0.006 | | TMN-601A |
| Japan, 1990 (Satonishiki) | WP | 7.0 | 0.1 | 4 | 14<br>21 | 0.66<br>0.54<br>c0.006 | | TMN-601A |
| Japan, 1990 (Satonishiki) | WP | 7.0 | 0.1 | 5 | 14<br>21 | 0.77<br>0.40<br>c0.006 | | TMN-601A |
| Japan, 1990 (Takasago) | WP | 5.6 | 0.08 | 5 | 14<br>21 | 2.2<br>0.99<br>c0.006 | | TMN-601A |
| Japan, 1990 (Takasago) | WP | 7.0 | 0.1 | 4 | 14<br>21 | 1.5<br>0.89<br>c0.006 | | TMN-601A |
| Japan, 1990 (Takasago) | WP | 7.0 | 0.1 | 5 | 14<br>21 | 2.3<br>1.4<br>c0.006 | | TMN-601A |
| Japan, 1990 (Satonishiki) | WP | 5.6 | 0.08 | 5 | 14<br>21 | 1.2<br>0.25<br>c0.01 | | TMN-601A |
| Japan, 1990 (Satonishiki) | WP | 7.0 | 0.1 | 4 | 14<br>21 | 0.78<br>0.64<br>c0.01 | | TMN-601A |
| Japan, 1990 (Satonishiki) | WP | 7.0 | 0.1 | 5 | 14<br>21 | 1.5<br>0.53<br>c0.01 | | TMN-601A |
| Japan, 1991 (Satonishiki) | WP | | 0.09 | 5 | 14<br>21 | 0.58<br>0.38 | <0.05<br><0.05 | RJ1409B |
| Japan, 1991 (Satonishiki) | WP | | 0.13 | 5 | 14<br>21 | 1.3<br>1.3 | <0.05<br><0.05 | RJ1409B |
| USA (WA), 1986 | WP | Po dip 1.5 g/l | | | 0 | 3.8, 14 | 0.09, 0.30 | 056131-H |
| USA (WA), 1986 | WP | Po dip 1.5 g/l | | | 10 | 7.3, 5.8 | 0.15, 0.17 | 056131-H |
| USA (WA), 1986 | WP | Po dip 1.5 g/l | | | 0 | 15, 14 | 0.23, 0.24 | 056131-H |
| USA (WA), 1986 | WP | 7×2.2 + Po dip 1.5 g/l | | | 0 | 35, 13 | 0.45, 0.20 | 056131-H |
| USA (WA), 1986 | WP | 7×2.2 + Po dip 1.5 g/l | | | 10 | 25, 9.2 | 0.44, 0.35 | 056131-H |
| USA (WA), 1986 | WP | 7×2.2 + Po dip 1.5 g/l | | | 0 | 2.1, 23 | 0.15, 0.34 | 056131-H |
| USA (WA) 1986 (Lambert) | WP | 2.2 | | 7 | 0 | 17, 19<br>c0.61, 0.21 | 0.22, 0.24<br>c<0.05 (2) | 056131-H |
| USA (WA) 1986 (Van) | WP | 2.2 | | 7 | 0 | 14, 14<br>c1.6, 5.3 | 0.15, 0.13<br>c<0.05 (2) | 056131-H |
| USA (MI) 1986 (Montmorency) | WP | 2.2 | | 7 | 0 | 9.6, 11 | 0.19, 0.18 | 056131-H |
| USA (NY) 1986 (Emperor Francis) | WP | 2.2 | | 7 | 0 | 12, 10 | 0.17, 0.15 | 056131-H |
| USA (MI), 1976 (Montmorency) | WP | 2.2 | | 6 | 0<br>1<br>3<br>7<br>12 | 13<br>20<br>15<br>5.9<br>4.3 | | TMN-600C |
| USA (MI), 1976 (Montmorency) | WP | 2.2 | | 6 | 0<br>1<br>3<br>7 | 21<br>11<br>19<br>20 | | TMN-600C |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 12 | 8.2<br>c0.1 | | |
| USA (MI), 1976 (Montmorency) | WP | 1.7 | | 6 | 0<br>1<br>3<br>7<br>12 | 12<br>14<br>8.3<br>5.5<br>1.9 | | TMN-600C |
| USA (MI), 1976 (Montmorency) | WP | 1.7 | | 6 | 0<br>1<br>3<br>7<br>12 | 20<br>16<br>9.7<br>16<br>12<br>c0.1 | | TMN-600C |
| USA (NY), 1977 (Emperor Francis/Napolean) | WP | 2.2 | | 7 | 8 | 0.92 | | TMN-600C |
| USA (IL), 1977 (Montmorency) | WP | 2.2 | | 7 | 1 | 4.3 | | TMN-600C |
| USA (MT), 1980 (Lambert) | WP | 2.2 | | 6 | 1<br>2<br>3<br>7<br>14 | 2.4<br>1.6<br>2.4<br>2.2<br>1.4 | | TMN-600C |
| USA (MT), 1980 (Lambert) | WP | 2.2 | | 6 | 0<br>1<br>3 | 5.5<br>3.4<br>4.7 | | TMN-600C |
| USA (MT), 1980 (Lambert) | WP | 2.2 | | 8 | 1<br>8 | 2.3<br>2.8 | | TMN-600C |

[1] application rate for last spray

Table 23. Residues of captan and THPI in plums after foliar applications of WP formulations. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | PHI, days | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Chile, 1992 (Roy Sum) | 2.9 | 0.14 | 1 | 26<br>49 | 0.62,<br>0.66<br><0.05<br>(2) | <0.05 (4) | RJ1299B |
| Chile, 1992 (Angelino) | 2.9 | 0.14 | 1 | 26<br>50 | 0.12,<br>0.55<br>0.06<br><0.05 | <0.05 (4) | RJ1299B |
| Chile, 1992 (Roy Sum) | 2.9 | 0.14 | 1 | 27<br>48 | 0.06,<br>0.11<br>0.08,<br>0.05 | <0.05 (4) | RJ1299B |
| Greece, 1999 (Angelino) | 2.9-3.1 | 0.13 | 4 | 7<br>14<br>21 | 0.24<br>0.23<br>0.13 | 0.082<br>0.099<br>0.070 | R9029 |
| Japan, 1988 (Kojyo) | 6.7 | 0.13 | 3 | 14<br>21 | 0.95<br>0.81<br>c0.085 | | TMN-664A |
| Japan, 1988 (Kojyo) | 6.7 | 0.13 | 5 | 14 | 3.0<br>c0.085 | | TMN-664A |
| Japan, 1988 (Sagijyuku) | 6.7 | 0.13 | 3 | 14<br>21 | 1.3<br>1.8 | | TMN-664A |
| Japan, 1988 (Sagijyuku) | 6.7 | 0.13 | 5 | 14 | 3.0 | | TMN-664A |

| Country (location), year (variety) | Application | | | PHI, days | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Portugal, 1999 (Madley) | 2.9-3.0 | 0.2 | 4 | 7<br>14<br>21 | 6.7<br>1.2<br>0.43 | 0.66<br>0.17<br>0.31 | R9029 |
| Spain, 1999 (Golden Japan) | 3.0 | 0.15 | 4 | 7<br>10<br>21 | 1.0<br>0.57<br>0.67 | 0.16<br>0.13<br>0.13 | R9029 |
| Spain, 1999 (Golden Japan) | 3.0 | 0.13 | 4 | 7<br>10<br>21 | 1.3<br>0.85<br>0.57 | 0.17<br>0.18<br>0.20 | R9029 |
| USA (CA), 1975 (Casselman) | 3.4 | 0.12 | 3 | 13 | <0.05 | | TMN663B |
| USA (NY), 1977 (Purple Plum) | 3.4 | 0.12 | 13 | 2 | 0.71 | | TMN663B |
| USA (NY), 1978 (Fellenburg) | 3.4 | 0.36 | 9 | 0<br>3<br>7<br>10 | 7.9<br>4.8<br>3.4<br>2.6 | | TMN663B |
| USA (CA) 1978 (Queen Anne) | 6.7 | | 1 | 0<br>1<br>3<br>7<br>14 | 0.64<br>0.47<br>0.31<br>0.81<br>0.54 | 0.67<br>0.34<br>0.38<br>0.60<br>0.42 | TMN663B |
| USA (CA), 1986 (Black Amber) | 3.4 | | 9 | 0 | 0.45,<br>0.14 | <0.05 (2) | 056131-P |
| USA (CA), 1986 (Queen Anne) | 3.4 | | 9 | 0 | 0.42,<br>0.60 | <0.05 (2) | 056131-P |
| USA (MI), 1986 (Stanley) | 3.4 | | 9 | 0<br>7<br>14 | 3.5, 5.6<br>3.0, 3.4<br>2.2, 1.5 | <0.05 (2)<br><0.05 (2)<br><0.05 (2) | 056131-P |
| USA (CA) 1986 (Queen Anne) | 10 | | 9 | 0 | 4.8, 3.8 | <0.05 (2) | 056131-P |

Table 24. Residues of captan and THPI in apricots after foliar applications of various captan formulations in the USA. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| CA, 1986 (Improved Flaming Gold) | WP | 2.8 | | 5 | 0 | 4.4, 4.5 | 0.18, 0.21 | 056131-E |
| CA, 1986 (Katy) | WP | 2.8 | | 5 | 0 | 6.6, 5.3 | 0.10, 0.20 | 056131-E |
| CA, 1986 (Castle Bright) | WP | 2.8 | | 5 | 0 | 5.0, 6.8 | 0.08, 0.12 | 056131-E |
| CA, 1980 (Tilton) | WP | 5.6 | | 2 | 1 | 3.3, 2.8 | 0.11, 0.11 | TMN-588A |
| CA, 1978 (Tilton) | WP | 6.7 | | 1 | 0<br>1<br>3<br>7<br>14 | 13.4<br>9.3<br>13.6<br>8.3<br>4.3 | <0.05<br><0.05<br><0.05<br>0.35<br>0.24 | TMN-588B |
| CA, 1978 (Golden Amber) | WP | 6.7 | | 1 | 0<br>1<br>3<br>7<br>14 | 6.6<br>4.0<br>6.0<br>3.6<br>2.0 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | TMN-588B |
| CA, 1978 | WP | 3.4 | | 1 | 0<br>1<br>3<br>7<br>14 | 2.0<br>2.7<br>3.3<br>2.0<br>1.7<br>c<0.05 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05<br>c0.27 | TMN-588B |
| IL, 1978 | WP | 1.1 | | 4 | 7 | 0.94 | | TMN-588B |
| CA, 1975 (Weinburger) | WP/4F | 3.4 | | 2 | 5 | 1.33 | | TMN-588B |

Table 25. Residues of captan and THPI in nectarines after foliar applications of WP formulations. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | PHI, days | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Chile, 1992 (Sun Sweet) | 2.9 | 0.14 | 1 | 31<br>53 | 0.16, 0.22<br>0.14, 0.10 | <0.05 (2)<br><0.05 (2) | RJ1362B |
| Chile, 1992 (L.316) | 2.9 | 0.14 | 1 | 31<br>52 | 0.17, 0.30<br>0.16, 0.15 | <0.05 (2)<br><0.05 (2) | RJ1362B |
| Chile, 1992 (Fiar Lane) | 2.9 | 0.14 | 1 | 31<br>52 | 0.16<br>0.11 | <0.05<br><0.05 | RJ1362B |
| Greece, 1999 (Tasty Free) | 2.9-3.0 | 0.13 | 4 | 7<br>14<br>21 | 5.6<br>4.1<br>1.5 | 0.66<br>0.51<br>0.19 | R9028 |
| Greece, 1999 (Arm King) | 2.9-3.0 | 0.13 | 4 | 7<br>14<br>21<br>7<br>14<br>21 | 3.1<br>2.3<br>0.90<br>c0.023,<br>c<0.01,<br>c<0.01 | 0.26<br>0.41<br>0.22<br>c<0.05<br>c<0.05<br>c<0.05 | R9028 |
| Spain, 1999 (Snow Queen) | 2.8-3.0 | 0.15 | 4 | 7<br>11<br>21<br>7<br>11<br>21 | 2.1<br>1.8<br>0.62<br>c0.012,<br>c<0.01,<br>c<0.01 | 0.36<br>0.21<br>0.11<br>c<0.05<br>c<0.05<br>c<0.05 | R9028 |
| Spain, 1999 (Snow Queen) | 3.0 | 0.15 | 4 | 7<br>11<br>21 | 2.4<br>1.3<br>0.43 | 0.30<br>0.17<br>0.11 | R9028 |
| Spain, 1991 (Red Globe) | 1.6 | 0.25 | 1 | 10<br>20<br>28 | 0.40<br>0.33<br>0.17 | <0.05<br><0.05<br><0.05 | RJ1172B |
| Spain, 1991 (Red Globe) | 1.9 | 0.3 | 1 | 10<br>20<br>28 | 0.77<br>0.47<br>0.18 | <0.05<br><0.05<br><0.05 | RJ1172B |
| USA (CA), 1986 (Spring Red) | 2.8 | | 6 | 0 | 2.2, 1.5 | <0.05 (2) | 056131-M |
| USA (CA), 1986 (Mike Grant) | 2.8 | | 6 | 0 | 1.3, 1.6 | 0.06,<br><0.05 | 056131-M |
| USA (CA), 1986 (Snow Queen) | 2.8 | | 6 | 0 | 3.9, 2.7 | 0.12, 0.06 | 056131-M |
| USA (CA), 1980 (LeGrande) | 6.7 | | 9 | 0<br>1<br>3<br>7<br>10 | 10<br>8.5<br>6.6<br>9.7<br>3.2<br>c0.03-0.05 | 0.28<br>0.32<br>0.21<br>0.26<br>0.20<br>c0.01 | TMN-642 |

Table 26. Residues of captan and THPI in peaches after foliar applications of various captan formulations and after post-harvest dipping. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Australia, 1991 (Golden Queen) | WP | | 0.13 | 5<br>4<br>3 | 6<br>14<br>27 | 4.7<br>2.8<br>1.5 | | S38990-51 |
| Australia, 1991 (Golden Queen) | WP | | 0.25 | 5<br>4 | 6<br>14 | 11<br>4.8 | | S38990-51 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | 3 | 27 | 3.7 | | |
| Canada, 1975 (Redskin) | WP | 3.4 | | 2 | 3 | 5.5 | | TMN-648E |
| Canada, 1975 (Redskin) | WP | 2.1 | | 2 | 1<br>3<br>7 | 6.1<br>2.6<br>2.3 | | TMN-648E |
| Canada, 1975 (Baby Gold) | WP | 2.1 | | 3 | 1<br>3<br>7 | 4.8<br>0.82<br>0.66 | | TMN-648E |
| Canada, 1975 (Red Haven) | WP | 2.1 | | 2 | 3 | 1.8 | | TMN-648E |
| Canada, 1975 (Red Haven) | WP | 2.1 | | 2 | 3 | 1.4 | | TMN-648E |
| Canada, 1981 (Red Haven) | WP | 3.0[1] | 0.1 | 3 | -0<br>0<br>1<br>3<br>5<br>7<br>10 | 3.0<br>12<br>6.5<br>5.8<br>6.6<br>6.1<br>4.8<br>c0.06 | | Northover *et al.* 1986 |
| Canada, 1981 (Garnet Beauty) | WP | 3.4[1] | | 3 | -0<br>0<br>1<br>4<br>7<br>12<br>14 | 1.7<br>9.9<br>7.3<br>2.7<br>2.2<br>2.9<br>1.7 | | Northover *et al.* 1986 |
| Canada, 1982 (Red Haven) | WP | 3.4[1] | | 3 | -0<br>0<br>1<br>2<br>3<br>5<br>7<br>10<br>14 | 1.8<br>3.4<br>2.6<br>2.2<br>3.0<br>2.6<br>1.9<br>2.9<br>3.2<br>c0.02 | | Northover *et al.* 1986 |
| Canada, 1983 (Red Haven) | WP | 3.4[1] | | 4 | -0<br>0<br>1<br>2<br>3<br>5<br>7<br>10<br>14 | 9.6<br>11<br>10<br>12<br>16<br>13<br>11<br>8.9<br>2.9<br>c0.24 | | Northover *et al.* 1986 |
| Chile, 1992 (Pomona) | WP | 2.9 | 0.14 | 1 | 27<br>50 | 2.0, 1.7<br>0.25, 0.27<br>c0.08 | 0.06, 0.05<br><0.05 (2)<br>c<0.05 | RJ1356B |
| Chile, 1992 (E. Lady) | WP | 2.9 | 0.14 | 1 | 24<br>24<br>46<br>46 | 0.44, 0.43<br>c0.07<br>0.32, 0.34<br>c0.22 | <0.05 (2)<br>c<0.05<br><0.05 (2)<br>c<0.05 | RJ1356B |
| Chile, 1992 (O'Henry) | WP | 2.9 | 0.14 | 1 | 28<br>50 | 1.0, 0.24<br>0.30, 0.31 | <0.05 (2)<br><0.05 (2) | RJ1356B |
| Italy, 1996 (Weinbercke) | WP | 3.0 + 3×1.5 | 0.25 + 3×0.13 | 4 | 0-<br>0+<br>10<br>20<br>31 | 0.08<br>0.67<br>0.26<br>0.19<br>0.14 | | ERSA-DA-07/96 |
| Italy, 1996 (Red Haven) | WP | 3.7 + 3×1.8 | 0.25 + 3×0.13 | 4 | 0-<br>0+ | 0.08<br>1.1 | | ERSA-DA-07/96 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 10<br>20<br>31<br>40 | 1.0<br>0.95<br>0.30<br>0.20 | | |
| Italy, 1995 (Suncrest) | WP | 3.3 +<br>3×1.3 | 0.25+4<br>×0.13 | 5 | 39 | 0.11 | | ERSA-DA-08/95 |
| Italy, 1995 (Michelini) | WP | 2.5 +<br>3×1.3 | 0.25+3<br>×0.13 | 4 | 0<br>10<br>20<br>30<br>40 | 1.7<br>1.5<br>1.0<br>0.52<br>0.17 | | ERSA-DA-09/95 |
| Italy, 1997 (Spring Crest) | WDG | 2.6 +<br>3×1.3 | 0.26+3<br>×0.13 | 4 | 0<br>10<br>20<br>30 | 1.0<br>0.90<br>0.05<br>0.05 | | ERSA-DA-04/97 |
| Italy, 1997 (Spring Crest) | WP | 2.5 +<br>3×1.3 | 0.25+3<br>×0.13 | 4 | 0<br>10<br>20<br>30 | 1.0<br>0.81<br>0.06<br>0.08 | | ERSA-DA-04/97 |
| Italy, 1998 (Glohaven) | WG | | 0.25+3<br>×0.13 | 4 | 0-<br>0+<br>10<br>20<br>30 | <0.01<br>2.1<br>1.0<br>0.49<br>0.23 | | ERSA-DA-04/98 |
| Italy, 1998 (Glohaven) | WP | | 0.25+3<br>×0.13 | 4 | 0-<br>0+<br>10<br>20<br>30 | <0.01<br>1.4<br>1.2<br>0.71<br>0.18 | | ERSA-DA-04/98 |
| Japan, 1975 (Okubo) | WP | 8 | 0.13 | 8 | 2<br>5<br>10 | 1.1 (pulp)<br>1.3<br>0.30<br>c0.004 | | TMN-650A |
| Japan, 1975 (Okubo) | WP | 8 | 0.13 | 8 | 2<br>5<br>10 | 364 (peel)<br>454<br>209<br>c0.25 | | TMN-650A |
| Japan, 1975 (Okubo) | WP | 4 | 0.13 | 8 | 1<br>5<br>10 | 0.89 (pulp)<br><0.004<br><0.004 | | TMN-650A |
| Japan, 1975 (Okubo) | WP | 4 | 0.13 | 8 | 1<br>5<br>10 | 82 (peel)<br>0.18<br>0.02<br>c0.14 | | TMN-650A |
| Japan, 1981 (Shimizu-Hakuto) | WP | 8 | 0.13 | 6 | 1<br>3<br>7 | 0.21 (pulp)<br>0.20<br>0.55<br>c0.005 | | TMN-650A |
| Japan, 1981 (Shimizu-Hakuto) | WP | 8 | 0.13 | 6 | 1<br>3<br>7 | 50 (peel)<br>107<br>97<br>c0.24 | | TMN-650A |
| Japan, 1981 (Hakuto) | WP | 5.3 | 0.13 | 6 | 1<br>3<br>7 | 0.35 (pulp)<br>0.27<br>0.21<br>c0.006 | | TMN-650A |
| Japan, 1981 (Hakuto) | WP | 5.3 | 0.13 | 6 | 1<br>3<br>7 | 70 (peel)<br>41<br>57<br>c0.56 | | TMN-650A |
| Spain, 1991 (Spring Crest) | WP | 4.7 | 0.25 | 3 | 10<br>20<br>28 | 2.8<br>2.7<br>1.8<br>c0.08 | <0.05<br><0.05<br><0.05<br>c<0.05 | RJ1172B |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Spain, 1991 (Spring Crest) | WP | 5.6 | 0.3 | 3 | 10<br>20<br>28 | 3.5<br>3.5<br>1.8<br>c0.08 | <0.05<br><0.05<br><0.05<br>c<0.05 | RJ1172B |
| USA (CA), 1978 (Red Haven) | WP | 6.7 | 0.12 | 1 | 0<br>1<br>3<br>7<br>14 | 7.8<br>6.8<br>3.6<br>4.0<br>2.0 | 1.0<br>0.2<br>0.6<br>0.14<br>0.08 | TMN-648D |
| USA (CA), 1986 (Fay) | WP | 4.5 | | 8 | 0 | 9.9, 14 | 0.08, 0.18 | 056131-N |
| USA (CA), 1986 (Fay Alberta) | WP | 4.5 | | 8 | 0 | 10, 2.9 | 0.29, 0.65 | 056131-N |
| USA (CA), 1986 | WP | Po 1.5 g/l | | | 0 | 39, 27 | 0.50, 0.38 | 056131-N |
| USA (CA), 1986 | WP | 8×4.5 + Po 1.5 g/l | | | 0 | 38, 48 | 0.61, 0.56 | 056131-N |
| USA (GA), 1986 (Red Skin) | WP | Po 1.5 g/l | | | 0 | 136, 144 | 1.7, 0.87 | 056131-N |
| USA (GA), 1986 (Red Skin) | WP | 8×4.5 + Po 1.5 g/l | | | 0 | 131, 135 | 2.0, 2.4 | 056131-N |
| USA (SC), 1986 (Topaz) | WP | 4.5 | | 8 | 0 | 7.4, 3.4 | <0.05 (2) | 056131-N |
| USA (GA), 1986 (Red Skin) | WP | 4.5 | | 8 | 0 | 1.4, 2.0<br>c0.42 | <0.05 (2)<br>c<0.05 | 056131-N |
| USA (MI), 1986 (Red Haven) | WP | 4.5 | | 8 | 0 | 5.7, 6.0 | <0.05 (2) | 056131-N |
| USA (NY), 1986 (Red Haven) | WP | 2.8 | | 8 | 0 | 7.7, 9.0 | 0.25, 0.42 | 056131-N |
| USA (WA), 1987 (Elberta) | WP | 4.5 | | 6 ✈ | 0 | 3.6, 2.5, 4.3 | <0.05 (3) | 40189814 |
| USA (WA), 1987 (Elberta) | WP | 4.5 | | 6 | 0 | 5.8, 3.0, 2.8 | 0.08, 0.07, 0.07 | 40189814 |
| USA (CA), 1987 | WP | 4.5 | | 8 ✈ | 0 | 4.3, 0.80, 2.6 | 0.07, <0.05 (2) | 40189814 |
| USA (CA), 1987 | WP | 4.5 | | 8[2] | 0 | 7.8, 6.9, 6.2 | 0.15, 0.16, 0.16 | 40189814 |
| USA (CA), 1987 | WP | 4.5 | | 8[3] | 0 | 11, 12, 12 | 0.50, 0.33, 0.24 | 40189814 |

✈ aerial application
[1] application rate for last spray
[2] concentrated spray
[3] dilute spray

Table 27. Residues of captan and THPI in blueberries after foliar applications of various WP formulations in the USA. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Location, year (variety) | Application | | | PHI, days | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Maine, 1987 | 2.8 | | 4 | 21 | 0.66 | 0.00 | IR-4 3458 |
| Maine, 1987 | 2.8 | | 10 | 0 | 8.4 | 0.15 | IR-4 3458 |
| WA, 1987 (Jersey) | 2.8 | | 3 | 64 | 0.67 | 0.00 | IR-4 3458 |
| WA, 1987 (Jersey) | 2.8 | | 4 | 7 | 3.4 | <0.05 | IR-4 3458 |
| WA, 1987 (Jersey) | 2.8 | | 14 | 0 | 18 | 0.17 | IR-4 3458 |
| MI, 1987 (Bluecrop + Jersey) | 2.8 | | 3 ✈ | 0<br>3<br>5<br>7<br>10 | 1.7<br>2.0<br>0.50<br>0.31<br>0.50 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | IR-4 3458 |
| MI, 1987 (Bluecrop) | 2.8 | | 3 ✈ | 7 | 3.3 | <0.05 | IR-4 3458 |

| Location, year (variety) | Application | | | PHI, days | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | 8 | 2.8 | <0.05 | |
| MI, 1987 (Bluecrop) | 2.2 | | 4 | 0<br>3<br>5<br>7<br>10 | 8.3<br>5.5<br>6.5<br>2.6<br>1.8 | 0.12<br>0.08<br>0.06<br>0.07<br><0.05 | IR-4 3458 |
| MI, 1987 (Earliblue + Bluecrop) | 4.5 | | 4 | 0<br>3<br>5<br>7<br>10 | 23<br>16<br>11<br>8.5<br>7.2 | 0.32<br>0.23<br>0.15<br>0.16<br>0.06 | IR-4 3458 |
| MI, 1987 (Earliblue + Bluecrop) | 2.8 | | 4 | 0<br>3<br>5<br>7<br>10 | 4.0<br>5.4<br>2.4<br>2.0<br>1.3 | 0.07<br>0.08<br><0.05<br><0.05<br><0.05 | IR-4 3458 |
| MI, 1987 (Earliblue + Bluecrop) | 2.8 | | 8 | 0<br>3<br>5<br>7<br>10 | 8.2<br>6.9<br>6.8<br>6.1<br>3.6 | 0.11<br>0.09<br>0.10<br>0.05<br><0.05 | IR-4 3458 |
| NJ, 1987 (Elliot) | 2.8 | | 6 ✈ | 1<br>3<br>5<br>7 | 4.8<br>4.3<br>3.5<br>1.2 | <0.05<br><0.05<br><0.05<br><0.05 | IR-4 3458 |
| NJ, 1987 (Elliot) | 2.8 | | 6 | 1<br>3<br>5<br>7 | 11<br>15<br>7.1<br>7.1 | 0.09<br>0.14<br>0.06<br>0.21 | IR-4 3458 |
| NJ, 1984 (Bluecrop) | 2.7 | | 5 ✈ | 0 | 3.7, 4.8, 2.7, 4.0, 7.1, 5.9 | <0.05 (6) | TMN-596 |
| NJ, 1984 (Rubel) | 2.7 | | 5 ✈ | 0 | 4.2 | <0.05 | TMN-596 |
| NJ, 1984 (Jersey) | 2.7 | | 5 ✈ | 0 | 4.0 | <0.05 | TMN-596 |
| NJ, 1984 (Coville) | 2.7 | | 5 ✈ | 0 | 3.2 | <0.05 | TMN-596 |
| NJ, 1987 (Rancocus) | 2.7 | | 5 ✈ | 0 | 6.9 | <0.05 | TMN-596 |
| NJ, 1984 (Bluecrop) | 2.7 | | 4 ✈ | 0 | 3.9 | <0.05 | TMN-596 |
| NJ, 1984 (Bluecrop) | 2.8 | | 3 ✈ | 0 | 6.7, 6.9, 6.3, 6.2, 6.0, 3.5, 6.3, 4.8 | <0.05 (8) | TMN-596 |
| NJ, 1984 | 2.7 | | 6 ✈ | 1 | 4.0, 6.0, 4.3 | <0.05 (3) | TMN-596 |
| OR, 1976 (Highbush) | 2.8 | | 4 | 0 | 6.5<br>c1.2 | | TMN-596 |

✈ aerial application

Table 28. Residues of captan and THPI in grapes after foliar applications of various captan formulations. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Australia, 1994 (Malbec) | WG | | 0.10 | 8 | 7<br>14<br>21 | 3.6<br>1.9<br>1.2 | 0.09<br><0.05<br><0.05 | R-11122 |
| Australia, 1994 (Malbec) | WG | | 0.21 | 8 | 7<br>14<br>21 | 8.2<br>3.5<br>2.6 | 0.22<br>0.11<br>0.10 | R-11122 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Australia, 1994 (Grenache) | WG | | 0.10 | 3 | 0<br>7<br>14<br>21 | 2.0<br>0.69<br>1.4<br>3.4 | 0.09<br>0.05<br>0.05<br>0.1 | R-11122 |
| Australia, 1994 (Grenache) | WG | | 0.21 | 3 | 0<br>7<br>14<br>21 | 5.9<br>7.5<br>4.7<br>5.0 | 0.11<br>0.16<br>0.11<br>0.08 | R-11122 |
| Brazil, 1995 (Italia) | WP | | 0.12 | 4 | 2 h<br>12 h<br>24 h | 0.73<br>0.61<br>0.78 | | D3.2.5/62 |
| Brazil, 1995 (Italia) | WP | | 0.24 | 4 | 1 | 1.19 | | D3.2.5/62 |
| Brazil, 1994 (Niágara Rosa) | WP | 0.96 | 0.12 | 5 | 1 | 2.5 | | D3.2.5/63 |
| Brazil, 1994 (Niágara Rosa) | WP | 1.9 | 0.24 | 5 | 1 | 4.6 | | D3.2.5/63 |
| Chile, 1992 (Red Seedless) | WP | 2.4 | 0.20 | 2 | 7<br>21 | 9.1<br>0.19 | 0.21<br><0.05 | RJ1374B |
| Chile, 1992 (Red Seedless) | WP | 2.4 | 0.20 | 1 | 7<br>21 | 1.6<br>6.4<br>c0.10 | 0.13<br>0.36<br>c<0.05 | RJ1374B |
| Chile, 1992 (Thompson Seedless) | WP | 2.4 | 0.20 | 1 | 7<br>21 | 2.4<br>6.1<br>c0.09 | 0.23<br>0.11<br>c0.07 | RJ1374B |
| Chile, 1992 (Thompson Seedless) | WP | | 0.12-0.13 | 4 | 15 | 18, 12, 25 | 1.5, 1.2, 1.2 | R-6987 |
| France, 1991 (Merlot Noir) | SC | 1.8 | | 14 | 0<br>0<br>11<br>11<br>20<br>20<br>33<br>33 | 5.6, 5.4, 2.5, 2.4<br>7.8, 5.5, 4.8, 2.3<br>4.2, 1.6<br>2.2, 0.96<br>2.9, 2.8<br>1.1, 0.83 | | R-6404 |
| France, 1991 (Merlot Noir) | SC | 3.5 | | 11 | 33<br>33 | 3.1, 3.0<br>4.4, 2.5 | | R-6404 |
| France, 1991 (Grenache) | SC | 1.8 | | 7 | 0<br>0<br>10<br>10<br>22<br>22<br>45<br>45 | 3.4, 1.6<br>3.3, 1.2<br>0.94, 2.1<br>1.9, 1.6<br>0.7, 1.4<br>1.3, 1.2<br>0.63, 0.53<br>1.3, 0.54 | | R-6404 |
| France, 1991 | SC | 1.8 | | 7 | 0<br>0<br>10<br>10<br>21<br>21<br>45<br>45 | 2.3, 2.4<br>2.3, 2.3<br>1.2, 1.2<br>2.4, 1.5<br>0.29, 0.41<br>0.33, 0.46<br>0.09, 0.06<br>0.06, 0.05 | | R-6404 |
| Germany, 1974 (Bacchus) | WP | | 0.08 | 6 | 0<br>28<br>47<br>77 | 2.2<br>0.31<br>0.11<br>0.12 | | BBA TR 1074 |
| Germany, 1974 (Müller- | WP | 2.0 | 0.24 | 7 | 3 | 1.8 | | BBA KH |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Thurgau) | | | | | 45 | 0.28 | | 1074 |
| Germany, 1974 (Müller-Thurgau) | WP | 1.6 | 0.33 | 7 | 0<br>28<br>42 | 7.4<br>1.8<br>1.4 | | BBA GE 1074 |
| Germany, 1974 (Müller-Thurgau) | WP | | 0.08 | 10 | 0<br>21<br>41 | 7.0<br>2.0<br>0.48 | | BBA WU 1074 |
| Germany, 1974 (Müller-Thurgau) | WP | 1.3 | 0.33 | 8 | 0<br>28<br>42<br>57 | 8.1<br>0.74<br>1.4<br>0.23 | | BBA OP 1074 |
| Germany, 1977 (Müller-Thurgau) | WP | 1.4 | 0.35 | 6 | 0<br>14<br>28<br>35<br>50 | 3.8<br>4.1<br>4.1<br>2.8<br>2.5 | | BBA 16684 |
| Germany, 1977 (Müller-Thurgau) | WP | | 0.09 | 10 | 0<br>14<br>35<br>46 | 13<br>5.3<br>4.7<br>4.3 | | BBA 13845 |
| Germany, 1977 (Müller-Thurgau) | WP | | 0.09 | 10 | 0<br>14<br>35<br>46 | 7.2<br>3.0<br>3.3<br>3.0 | | BBA 13845 |
| Germany, 1977 (Bacchus) | WP | 4×1.8<br>6×2.2 | 0.09 | 10 | 0<br>14<br>28<br>35<br>47 | 5.4<br>4.9<br>3.7<br>6.3<br>2.0 | | BBA TR 1277 |
| Germany, 1977 | WP | 1.8 | 0.09 | 8 | 0<br>14<br>28<br>35<br>55 | 4.9<br>1.7<br>3.3<br>0.79<br>0.39 | | BBA 1377 |
| Germany, 1977 (Müller-Thurgau) | WP | 4.0 | 0.5 | 7 | 3<br>45 | 14<br>2.5 | | BBA 1174 KH |
| Germany, 1974 (Müller-Thurgau) | WP | 5×2.5<br>2×3.2 | 5×0.5<br>2×0.64 | 7 | 0<br>28<br>42 | 10<br>2.2<br>3.2 | | BBA GE 1174 |
| Germany, 1974 (Bacchus) | WP | | 0.13 | 6 | 0<br>28<br>47<br>77 | 18<br>0.61<br><0.02<br>0.96 | | BBA TR 1174 |
| Germany, 1990 (Bacchus) | WP | 2×1.8, 5×2.7, 1×3.6 | | 8 | 28 | 6.5 | 0.26 | RJ1160B |
| Germany, 1990 (Bacchus) | WP | 2×1.8, 4×2.7, 1×3.6 | | 7 | 36<br>43 | 2.2<br>3.0 | 0.08<br>0.12 | RJ1160B |
| Germany, 1990 (Bacchus) | WP | 2×1.8, 3×2.7, 1×3.6 | | 6 | 55<br>50 | 1.5<br>2.4 | 0.08<br>0.14 | RJ1160B |
| Germany, 1990 (Portugieser) | WP | 2×1.8, 5×2.7, 1×3.6 | | 8 | 28 | 2.8 | 0.08 | RJ1160B |
| Germany, 1990 (Portugieser) | WP | 2×1.8, 4×2.7, 1×3.6 | | 7 | 34<br>42 | 1.5, 1.9<br>1.6 | 0.09, 0.13<br><0.05 | RJ1160B |
| Germany, 1990 (Portugieser) | WP | 2×1.8, 3×2.7, 1×3.6 | | 6 | 49<br>55 | 1.6<br>0.85 | <0.05<br>0.06 | RJ1160B |
| Germany, 1990 (Bacchus) | WP | 2×1.8, 5×2.7, 1×3.6 | | 8 | 28 | 8.3 | 0.19 | RJ1154B |
| Germany, 1990 (Bacchus) | WP | 2×1.8, 4×2.7, 1×3.6 | | 7 | 43<br>36 | 7.0<br>4.4 | 0.24<br>0.17 | RJ1154B |
| Germany, 1990 (Bacchus) | WP | 2×1.8, 3×2.7, 1×3.6 | | 6 | 55<br>50 | 2.8<br>0.93 | 0.08<br><0.05 | RJ1154B |
| Germany, 1990 (Portugieser) | WP | 2×1.8, 5×2.7, 1×3.6 | | 8 | 28 | 1.8, 1.5 | 0.14, 0.13 | RJ1154B |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Germany, 1990 (Portugieser) | WP | 2×1.8, 4×2.7, 1×3.6 | | 7 | 42<br>34 | 1.4<br>1.1 | 0.08<br>0.09 | RJ1154B |
| Germany, 1990 (Portugieser) | WP | 2×1.8, 3×2.7, 1×3.6 | | 6 | 55<br>49 | 0.93<br>1.1 | 0.08<br>0.10 | RJ1154B |
| Germany, 1990 (Riesling) | WP | 2×1.8, 5×2.7, 1×3.6 | | 8 | 28 | 15 | 0.35 | RJ1154B |
| Germany, 1990 (Riesling) | WP | 2×1.8, 4×2.7, 1×3.6 | | 7 | 43<br>36 | 7.7<br>9.8 | 0.34<br>0.32 | RJ1154B |
| Germany, 1990 (Riesling) | WP | 2×1.8, 3×2.7, 1×3.6 | | 6 | 57<br>48 | 7.1<br>7.4 | 0.22<br>0.22 | RJ1154B |
| Germany, 1990 (Weissburgunder) | WP | 2×1.8, 5×2.7, 1×3.6 | | 8 | 30 | 3.4, 3.7 | 0.20, 0.25 | RJ1154B |
| Germany, 1990 (Weissburgunder) | WP | 2×1.8, 4×2.7, 1×3.6 | | 7 | 38<br>43 | 1.7<br>1.9 | 0.15<br>0.14 | RJ1154B |
| Germany, 1990 (Weissburgunder) | WP | 2×1.8, 3×2.7, 1×3.6 | | 6 | 49<br>56 | 1.3<br>0.79 | 0.10<br><0.05 | RJ1154B |
| Germany, 1991 (Kerner) | WP | 7×2.3, 1×2.7 | | 8 | 0<br>10<br>21<br>26<br>33 | 1.5<br>1.4<br>0.65<br>0.59<br>0.42 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1176B |
| Germany, 1991 (Kerner) | WG | 7×2.3, 1×2.7 | | 8 | 0<br>10<br>21<br>26<br>33 | 2.3<br>1.1<br>0.70<br>0.54<br>0.46 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1176B |
| Germany, 1991 (Dornfelder) | WP | 7×2.3, 1×2.7 | | 8 | 0<br>10<br>21<br>29<br>35 | 6.3<br>2.7<br>2.3<br>2.2<br>2.2 | 0.10<br><0.05<br><0.05<br>0.05<br>0.06 | RJ1176B |
| Germany, 1991 (Dornfelder) | WG | 7×2.3, 1×2.7 | | 8 | 0<br>10<br>21<br>29<br>35 | 6.7<br>4.4<br>2.2<br>2.1<br>3.1 | 0.09<br>0.06<br><0.05<br><0.05<br><0.05 | RJ1176B |
| Germany, 1991 (Ortega) | WP | 7×2.3, 1×2.7 | | 8 | 0<br>10<br>20<br>27<br>33 | 2.5<br>1.6<br>2.4<br>3.0<br>1.4 | 0.07<br><0.05<br>0.05<br><0.05<br>0.06 | RJ1176B |
| Germany, 1991 (Ortega) | WG | 7×2.3, 1×2.7 | | 8 | 0<br>10<br>20<br>27<br>33 | 2.9<br>3.3<br>2.4<br>2.2<br>2.9 | 0.08<br>0.07<br>0.05<br>0.05<br>0.08 | RJ1176B |
| Germany, 1991 (Müller-Thurgau) | WP | 7×2.3, 1×2.7 | | 8 | 0<br>10<br>21<br>26<br>35 | 6.0<br>3.0<br>2.0<br>2.1<br>1.3 | 0.06<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1176B |
| Germany, 1991 (Müller-Thurgau) | WG | 7×2.3, 1×2.7 | | 8 | 0<br>10<br>21<br>26<br>35 | 2.9<br>3.1<br>2.2<br>2.6<br>1.8 | 0.07<br>0.06<br><0.05<br><0.05<br><0.05 | RJ1176B |
| Germany, 1994 | WP/ WG | 2.3 | | | 0<br>7<br>14<br>20<br>27 | 6.3<br>5.2<br>4.4<br>4.7<br>4.8 | 0.12<br>0.11<br>0.22<br>0.13<br>0.20 | AZ26686/94 |
| Germany, 1994 | WP/ WG | 2.3 | | | 0<br>7<br>14 | 5.4<br>6.8<br>3.6 | 0.20<br>0.19<br>0.13 | AZ26686/94 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 21<br>28 | 2.5<br>4.4 | 0.08<br>0.12 | |
| Germany, 1994 (White grapes) | WP/ WG | 2.3 | | | 0<br>7<br>14<br>21<br>28 | 6.0<br>5.6<br>6.9<br>4.8<br>5.6 | 0.39<br>0.59<br>0.51<br>0.28<br>0.71 | AZ27150/94 |
| Germany, 1994 (Red grapes) | WP/ WG | 2.3 | | | 0<br>7<br>14<br>21<br>28 | 10<br>7.1<br>8.2<br>8.5<br>8.4 | 0.36<br>0.53<br>0.25<br>0.30<br>0.59 | AZ27150/94 |
| Japan, 1971 (Delaware) | WP | 2.5 | 0.1 | 3 | 23 | 0.43 | | TMN-621A |
| Japan, 1971 (Delaware) | WP | 2.5 | 0.1 | 5 | 15 | 0.46 | | TMN-621A |
| Japan, 1973 (Delaware) | WP | 3 | 0.1 | 3 | 27 | 0.65 | | TMN-621A |
| Japan, 1973 (Delaware) | WP | 3 | 0.1 | 5 | 13 | 1.6 | | TMN-621A |
| Japan, 1987 (Kyoho) | WP | 3 | 0.1 | 2 | 3<br>7<br>14<br>21 | 3.9<br>1.6<br>3.5<br>2.1 | | TMN-621A |
| Japan, 1987 (Kyoho) | WP | 3 | 0.1 | 3 | 3<br>7<br>14<br>21 | 5.7<br>5.5<br>3.2<br>3.7 | | TMN-621A |
| Japan, 1987 (Kyoho) | WP | 3 | 0.1 | 5 | 3<br>7<br>14<br>21 | 4.5<br>3.6<br>3.0<br>3.7 | | TMN-621A |
| Japan, 1987 (Kyoho) | WP | 3 | 0.1 | 2 | 3<br>7<br>14<br>21 | 3.4<br>1.9<br>2.8<br>2.2<br>c0.04 | | TMN-621A |
| Japan, 1987 (Kyoho) | WP | 3 | 0.1 | 3 | 3<br>7<br>14<br>21 | 5.3<br>3.2<br>3.4<br>3.5 | | TMN-621A |
| Japan, 1987 (Kyoho) | WP | 3 | 0.1 | 5 | 3<br>7<br>14<br>21 | 4.7<br>5.3<br>4.3<br>3.0 | | TMN-621A |
| Japan, 1987 (Delaware) | WP | 2.5 | 0.1 | 2 | 3<br>7<br>14<br>21 | 3.7<br>2.5<br>2.0<br>1.7<br>c0.02 | | TMN-621A |
| Japan, 1987 (Delaware) | WP | 2.5 | 0.1 | 3 | 3<br>7<br>14<br>21 | 3.4<br>2.3<br>2.6<br>1.7 | | TMN-621A |
| Japan, 1987 (Delaware) | WP | 2.5 | 0.1 | 5 | 3<br>7<br>14<br>21 | 5.4<br>4.8<br>3.4<br>3.3 | | TMN-621A |
| Japan, 1988 (Delaware) INDOOR | WP | 3 | 0.1 | 2 | 14<br>21<br>30 | 3.9<br>2.6<br>2.4<br>c0.04 | | TMN-621A |
| Japan, 1988 (Delaware) INDOOR | WP | 3 | 0.1 | 3 | 14<br>21<br>30 | 4.4<br>6.1<br>6.3 | | TMN-621A |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Japan, 1988 (Delaware) INDOOR | WP | 3 | 0.1 | 5 | 14<br>21<br>30 | 9.2<br>11<br>7.7 | | TMN-621A |
| Japan, 1988 (Delaware) | WP | 3 | 0.1 | 2 | 14<br>21<br>30 | 5.7<br>3.7<br>2.9<br>c0.04 | | TMN-621A |
| Japan, 1988 (Delaware) | WP | 3 | 0.1 | 3 | 14<br>21<br>30 | 7.1<br>8.5<br>7.1 | | TMN-621A |
| Japan, 1988 (Delaware) | WP | 3 | 0.1 | 5 | 14<br>21<br>30 | 7.8<br>9.4<br>9.7 | | TMN-621A |
| Japan, 1988 (Black Olympia) INDOOR | WP | 3 | 0.1 | 2 | 14<br>21<br>30 | 3.8<br>2.8<br>1.8 | | TMN-621A |
| Japan, 1988 (Black Olympia) INDOOR | WP | 3 | 0.1 | 3 | 14<br>21<br>30 | 2.5<br>1.2<br>1.9 | | TMN-621A |
| Japan, 1988 (Black Olympia) INDOOR | WP | 3 | 0.1 | 5 | 14<br>21<br>30 | 3.6<br>2.7<br>2.1 | | TMN-621A |
| Japan, 1988 (Kyoho) INDOOR | WP | 3 | 0.1 | 2 | 14<br>21<br>30 | 1.0<br>0.52<br>0.64<br>c0.13 | | TMN-621A |
| Japan, 1988 (Kyoho) INDOOR | WP | 3 | 0.1 | 3 | 14<br>21<br>30 | 0.69<br>0.88<br>0.79 | | TMN-621A |
| Japan, 1988 (Kyoho) INDOOR | WP | 3 | 0.1 | 5 | 14<br>21<br>30 | 2.7<br>2.6<br>1.1 | | TMN-621A |
| Japan, 1991 (Kyohou) | WP | 3 | | 5 | 14<br>21 | 5.8<br>3.8 | 0.08<br>0.08 | RJ1177B |
| Japan, 1991 (Kyohou) | WP | 3 | | 3 | 14<br>21 | 1.1<br>3.2 | <0.05<br>0.06 | RJ1177B |
| Japan, 1991 (Kyohou) | WP | 3 | | 5 | 14<br>21 | 14<br>13<br>c0.17 | 0.27<br>0.25<br>c<0.05 | RJ1177B |
| Japan, 1991 (Kyohou) | WP | 3 | | 3 | 14<br>21 | 12<br>11<br>c0.17 | 0.24<br>0.15<br>c<0.05 | RJ1177B |
| Japan, 1991 (Kyohou) | WP | 3 | | 5 | 14 | 6.1 | 0.21 | RJ1177B |
| Japan, 1991 (Kyohou) | WP | 3 | | 4 | 21 | 2.9 | 0.08 | RJ1177B |
| Japan, 1991 (Kyohou) | WP | 3 | | 5 | 14 | 6.1 | 0.19 | RJ1177B |
| Japan, 1991 (Kyohou) | WP | 3 | | 4 | 21 | 4.7 | 0.27 | RJ1177B |
| USA (PA), 1989 (Concord) | WP | 2.2 | | 6 | 36 | 0.44 | <0.05 | RR 92-007B |
| USA (NY), 1987 (Catawba) | WP | 2.2 | | 6 | 0 | 8.0<br>8.4<br>8.0 | 0.21<br>0.22<br>0.25 | 40189811 |
| USA (NY), 1987 (Catawba) | WP | 2.2 | | 6 | 0 | 3.5<br>2.2<br>2.1 | 0.07<br><0.05<br><0.05 | 40189811 |
| USA (WA), 1989 (Concord) | WP | 2.2 | | 2<br>3<br>4 | 76<br>67<br>57 | 0.07<br>0.17<br>0.24 | <0.05<br><0.05<br><0.05 | RR 90-379B |
| USA (NY) 1989 (Catawba | WP | 2.2 | | 2<br>3<br>4 | 103<br>90<br>78 | <0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05 | RR 90-379B |
| USA (PA) 1989 (Concord | WP | 2.2 | | 2 | 90 | <0.05 | <0.05 | RR 90-379B |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | 3<br>4 | 80<br>73 | <0.05<br><0.05 | <0.05<br><0.05 | |
| USA (MI) 1989 (Concord) | WP | 2.2 | | 2<br>3<br>4 | 116<br>96<br>84 | <0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05 | RR 90-379B |
| USA (CA), 1989 (Thompson Seedless) | WP | 2.2 | | 2<br>3<br>4 | 136<br>118<br>104 | 0.17<br>0.94<br>0.10 | <0.05<br><0.05<br><0.05 | RR 90-379B |
| USA (WA), 1986 | WP | 2.2 | | 6 | 0 | 0.93, 1.3 | <0.05 (2) | 056131-K |
| USA (CA), 1986 (Thompson Seedless) | WP | 2.2 | | 6 | 0 | 11, 22 | 0.20, 0.28 | 056131-K |
| USA (CA), 1986 (Emperor) | WP | 2.2 | | 6 | 0 | 7.4, 5.8 | <0.05 (2) | 056131-K |
| USA (CA), 1986 (Emperor) | WP | 2.2 | | 6 | 0 | 3.7, 1.3, 1.3 | <0.05 (3) | 056131-K |
| USA (NY), 1986 (Aurora) | WP | 2.2 | | 6 | 0 | 7.1, 7.2, 6.4 | 0.19, 0.11, 0.14 | 056131-K |
| USA (NY), 1986 (Concord) | WP | 2.2 | | 6 | 0 | 6.4, 4.8, 4.2 | 0.14, 0.19, 0.18 | 056131-K |
| USA (MI), 1986 (Concord) | WP | 2.2 | | 6 | 0 | 11, 8.1 | 0.14, 0.12 | 056131-K |

Table 29. Residues of captan and THPI in raspberries after foliar applications of a captan WP formulation in the USA. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Location, year (variety) | Application | | | PHI, days | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| OR, 1990 (Meeker) | 1.1-1.8<br>(last 1.1) | | 9 | 0<br>7 | 5.6<br>2.1 | 0.49<br>0.16 | IR-4 3953 |
| OR, 1990 (Meeker) | 1.8 | 0.15 | 9 | 0<br>7 | 5.7<br>1.1 | 0.37<br>0.12 | IR-4 3953 |
| WA, 1990 (Meeker) | 3.3-5.5<br>(last 3.3) | 0.24 | 10 | 0<br>7 | 36<br>16 | 2.1<br>0.89 | IR-4 3953 |
| WA, 1990 (Meeker) | 5.6 | 0.24 | 10 | 0<br>7 | 38<br>23 | 1.7<br>1.2 | IR-4 3953 |
| OR, 1991 (Meeker) | 2.8-3.4<br>(last 2.8) | 0.3-0.4 | 11 | 0<br>7 | 11, 11<br>8.3, 7.8 | 0.50, 0.42<br>0.31, 0.41 | IR-4 3953b |
| OR, 1991 (Meeker) | 2.8-3.4<br>(last 2.8) | 0.3-0.4 | 10 | 3 | 6.7, 7.2 | 0.19, 0.23 | IR-4 3953b |
| WA, 1991 (Willamette) | 2.8 | 0.67 | 7 | 0<br>3<br>7 | 37, 24<br>20, 38<br>18, 18 | 1.0, 0.50<br>0.53, 0.55<br>1.0, 1.1 | IR-4 3953b |
| WA, 1992 (Willamette) | 2.3 | 0.54 | 5 | 3 | 12, 12, 13, 13 | 0.65, 0.42, 0.49, 0.54 | IR-4 A3953 |
| WA, 1992 (Willamette) | 2.8 | 0.67 | 5 | 3 | 17, 14, 16, 13 | 0.38, 0.45, 0.27, 0.37 | IR-4 A3953 |
| WA, 1995 (Meeker) | 2.3 | 0.25 | 6 | 3 | 8.3, 6.8 | 0.56, 0.42 | IR-4 B3953 |
| OR, 1995 (Meeker) | 2.3 | 0.24 | 6 | 3 | 7.7, 7.4 | 0.43, 0.44 | IR-4 B3953 |
| PA, 1995 (Titan) | 2.3 | 0.49 | 6 | 3 | 18, 13 | 0.86, 0.59 | IR-4 B3953 |

Table 30. Residues of captan and THPI in strawberries after foliar applications of various captan formulations. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Australia, 1991 (Promise) | WP | 3.0 | | 5 | 1<br>2<br>3 | 6.9<br>6.6<br>7.9 | | S38990 91-49 |
| Australia, 1991 (Promise) | WP | 6.0 | | 5 | 1<br>2<br>3 | 20<br>12<br>12 | | S38990 91-49 |
| Belgium, 1991 (Elsanta) | WP | 1.3 | 0.13 | 8 | 0<br>0<br>4<br>4<br>7<br>7 | 1.9, 1.7<br>1.8, 2.6<br>1.7, 1.9<br>2.3, 2.4<br>0.96, 1.3<br>1.1, 1.1 | | RIC1800 |
| Belgium, 1999 (Elsanta) INDOOR | WG | 1.2 | 0.12 | 2 | 14 | 0.18 | | R11136 |
| Belgium, 1999 (Elsanta) INDOOR | WG | 1.2 | 0.12 | 2 | 14 | 0.13 | | R11136 |
| Chile, 1991 (Selva) | WP | 3.2 | | 1 | 3<br>7 | 3.8, 3.3<br>3.4, 4.2 | 0.65, 0.67<br>0.22, 0.23 | RJ1367B |
| Chile, 1991 (Chandler) | WP | 3.2 | | 1 | 3<br>7 | 2.1, 3.0<br>2.9, 3.8<br>c<0.05 | 0.55, 0.73<br>0.39, 0.39<br>c0.07 | RJ1367B |
| Chile, 1991 (Pajaro) | WP | 3.2 | | 1 | 3<br>7 | 3.3, 4.8<br>1.9, 2.7<br>c0.07 | 0.46, 0.44<br>0.19, 0.25<br>c<0.05 | RJ1367B |
| Germany 1961 (Senga-Sengana) | WP | 0.75 | 0.13 | 1 | 0<br>3<br>7<br>14 | 1.8<br>1.0<br>0.7<br>0.4 | | BBA |
| Germany 1962 (Senga-Sengana) | WP | 0.75 | 0.13 | 1 | 0<br>3<br>7<br>14 | 2.2<br>2.0<br>1.1<br>0.05 | | BBA |
| Germany 1964 | WP | 1.3 | 0.13 | 2 | -10<br>-1<br>8 | 0.5<br><0.1<br><0.1 | | BBA CPT 1/1964 |
| Hungary, 1991 (Gorella) | WP | 1.3 | | 3 | -0<br>-0<br>0<br>0<br>5<br>5<br>10<br>10<br>10<br>10 | 1.1, 2.9<br>2.3, 1.8<br>7.4, 6.9<br>5.1, 8.4<br>2.2, 3.2<br>2.2, 3.3<br>0.65, 0.83<br>0.79, 0.93<br>0.70, 0.88<br>0.86 | <0.1, <0.1<br><0.1, <0.1<br><0.2, <0.2<br><0.1, <0.2<br><0.1, <0.1<br><0.1, <0.1<br><0.1, <0.1<br><0.1, <0.1<br><0.1, <0.1<br><0.1 | CT/42/92 |
| Israel, 1991 (Dorit) under plastic tunnels | WP | 1.3 | 0.18 | 3 | 0<br>0<br>17<br>17<br>24<br>24<br>31<br>31 | 3.6, 1.6<br>3.7, 2.8<br>1.5, 1.3<br>1.3, 2.4<br>0.69, 0.94<br>0.86, 1.2<br>0.48, 0.67<br>2.7, 0.69 | <0.2, <0.2<br><0.2, <0.1<br><0.1, <0.1<br><0.1, <0.1<br><0.1, <0.2<br><0.2, <0.1<br><0.1, <0.1<br><0.1, <0.1 | CT/38/92 Control samples contaminated |
| Israel, 1991 (Dorit) under plastic tunnels | WP | 2.5 | 0.18 | 3 | 0<br>0<br>17<br>17<br>24 | 8.0, 8.5<br>3.7, 4.0<br>2.4, 2.1<br>3.0, 1.6<br>22, 4.4 | <0.2, <0.2<br><0.2, <0.1<br><0.1, <0.1<br><0.1, <0.1<br><0.1, <0.2 | CT/38/92 Control samples contaminated |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 24<br>31<br>31 | 2.1, 2.6<br>1.4, 1.5<br>1.6, 3.0 | <0.2, <0.1<br><0.1, <0.1<br><0.1, <0.1 | |
| Netherlands, 1999 (Elsanta) INDOOR | WG | 1.2 | 0.12 | 2 | 14 | 0.25 | | R11136 |
| Netherlands, 1999 (Elsanta) INDOOR | WG | 1.2 | 0.12 | 2 | 14 | 0.07 | | R11136 |
| Spain, 1992 (Pájaro) | WP | | 0.15 | 4 | 0<br>0<br>12<br>12<br>21<br>21 | 0.17, 0.16<br>0.08, 0.24<br>0.01, 0.04<br>0.01 <0.01<br>≤0.01 (2)<br><0.01 (2) | 0.43, 1.5<br>1.2, 3.1<br>0.93, 0.09<br>0.15, 0.10<br>0.10 <0.01<br><0.01 0.15 | FRESA SP 92 |
| USA (FL), 1993 (Oso Grande) | WP | 3.4 | | 8 | 2 | 3.9, 5.0, 4.6 | 0.70, 0.86, 0.79 | 056131-A |
| USA (FL), 1993 (Oso Grande) | WP | 3.4 | | 8+8 | 2 | 2.0, 2.2, 2.4 | 0.43, 0.32, 0.32 | 056131-A |
| USA (FL), 1993 (Oso Grande) | WP | 3.4 | | 8+8 +8 | 2 | 2.0, 1.4, 1.6 | 0.46, 0.38, 0.54 | 056131-A |
| USA (FL), 1993 (Sweet Charlie) | WP | 3.4 | | 8 | 2 | 6.6, 6.3, 6.1 | 0.59, 0.42, 0.34 | 056131-A |
| USA (FL), 1993 (Sweet Charlie) | WP | 3.4 | | 8+8 | 2 | 3.8, 3.0, 3.4 | 0.60, 0.54, 0.57 | 056131-A |
| USA (FL), 1993 (Sweet Charlie) | WP | 3.4 | | 8+8 +8 | 2 | 2.7, 1.8, 3.0 | 0.62, 0.57, 0.65 | 056131-A |
| USA (CA), 1985 | WP | 3.4 | | 12 | 0 | 10, 7.2 | 0.34, 0.29 | TMN-684A |
| USA (OR), 1985 (Hood) | WP | 3.4 | | 12 | 0 | 8.7, 8.6 | 0.23, 0.26 | TMN-684A |
| USA (CA), 1985 | WP | 3.4 | | 12 | 0 | 12, 6.7 | 0.90, 0.64 | TMN-684A |
| USA (WA), 1986 | WP | 3.4 | | 7 | 0 | 3.6, 4.4 | 0.15, 0.22 | 056131-V |
| USA (CA), 1986 (Driscoll) | WP | 3.4 | | 8 | 0 | 12, 12, 9.5 | 1.4, 1.4, 1.2 | 056131-V |
| USA (CA), 1986 (Tuft) | WP | 3.4 | | 8 | 0 | 5.4, 4.9, 4.8 | 0.53, 0.51, 0.83 | 056131-V |
| USA (FL), 1986 (Chandler) | WP | 3.4 | | 6 | 0 | 1.6, 2.0 | 0.14, 0.15 | 056131-V |
| USA (FL), 1986 (Chandler) | WP | 3.4 | | 7 | 0 | 1.5, 2.6 | 0.08, 0.19 | 056131-V |
| USA (MI), 1986 (Holiday) | WP | 3.4 | | 8 | 0 | 3.9, 3.0 | 0.50, 0.43 | 056131-V |
| USA (NC), 1986 (Apollo) | WP | 3.4 | | 8 | 0 | 7.2, 7.7 | 0.25, 0.30 | 056131-V |
| USA (CA), 1987 (Pajaro) | WP | 4.5 | | 6 ✈ | 0 | 15, 13, 8.9 | 0.39, 0.69, 0.22 | 40189822 |
| USA (CA), 1987 (Pajaro) | WP | 4.5 | | 6[1] | 0 | 0.84, 0.60, 1.0 | 0.08, 0.08, 0.09 | 40189822 |
| USA (CA), 1987 (Pajaro) | WP | 4.5 | | 6[2] | 0 | 13, 8.1, 8.9, 6.4 | 0.61, 0.48, 0.54, 0.42 | 40189822 |

✈ aerial application
[1] concentrated spray
[2] dilute spray

Table 31. Residues of captan and THPI in melons after foliar applications of various captan formulations. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | PHI, | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | days | Captan | THPI | |
| Japan, 1989 (Muskmelon, Andes) INDOOR | 4.0 | 0.2 | 5 | 1<br>3<br>7<br>14 | <0.005<br><0.005<br><0.005<br>≤0.005 | | TMN-636A |

| Country (location), year (variety) | Application | | | PHI, | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | days | Captan | THPI | |
| Japan, 1989 (Muskmelon, Andes)<br>INDOOR | 4.0 | 0.2 | 7 | 1<br>3<br>7<br>14 | <0.005<br><0.005<br><0.005<br>≤0.005 | | TMN-636A |
| Japan, 1989 (muskmelon, Sunday-Akifuyu)<br>INDOOR | 4.0 | 0.2 | 5 | 1<br>3<br>7<br>14 | <0.005<br>0.006<br><0.005<br>≤0.005 | | TMN-636A |
| Japan, 1989 (muskmelon, Sunday-Akifuyu)<br>INDOOR | 4.0 | 0.2 | 7 | 1<br>3<br>7<br>14 | 0.006<br>0.008<br><0.005<br>≤0.005 | | TMN-636A |
| Japan, 1989 (muskmelon, Andes)<br>INDOOR | 4.0 | 0.2 | 5 | 1<br>3<br>7<br>14 | <0.005<br><0.005<br><0.005<br>≤0.005 | | TMN-636A |
| Japan, 1989 (muskmelon, Andes)<br>INDOOR | 4.0 | 0.2 | 7 | 1<br>3<br>7<br>14 | <0.005<br><0.005<br><0.005<br>≤0.005 | | TMN-636A |
| Japan, 1989 (muskmelon, Sunday-Akifuyu)<br>INDOOR | 4.0 | 0.2 | 5 | 1<br>3<br>7<br>14 | <0.005<br><0.005<br><0.005<br>≤0.005 | | TMN-636A |
| Japan, 1989 (muskmelon, Sunday-Akifuyu)<br>INDOOR | 4.0 | 0.2 | 7 | 1<br>3<br>7<br>14 | <0.005<br>0.006<br><0.005<br>≤0.005 | | TMN-636A |
| Japan, 1991 (muskmelon, Andes) | 4.0 | 0.25 | 5<br>4 | 14<br>21 | 4.6<br>3.0 | 0.35<br>0.22 | RJ1427B |
| Japan, 1991 (muskmelon, Andes) | 4.0 | 0.25 | 5<br>4 | 14<br>21 | 4.0<br>2.9 | 0.26<br>0.12 | RJ1427B |
| Japan, 1991 (muskmelon, Arseinu-Natsu II) | 4.0 | 0.25 | 5<br>4 | 14<br>21 | 3.6<br>1.8 | 0.32<br>0.23 | RJ1427B |
| Japan, 1991 (muskmelon, Arseinu-Natsu II) | 4.0 | 0.25 | 5<br>4 | 14<br>21 | 4.1<br>3.9 | 0.22<br>0.34 | RJ1427B |
| USA (CA), 1986 (cantaloupe, Imperial Valley) | 2.2 | | 7 | 0 | 1.6, 2.0 | 0.13, 0.15 | 056131-G |
| USA (CA), 1986 (cantaloupe, Burpee Hybrid) | 2.2 | | 7 | 0 | 1.7, 2.9<br>c0.08 | 0.29, 0.22<br>c<0.05 | 056131-G |
| USA (FL), 1986 (cantaloupe, Asgrow-Summet) | 2.2 | | 7 | 0 | 0.25, 0.56 | 0.27, 0.38 | 056131-G |
| USA (MI), 1986 (cantaloupe, Star Trek) | 2.2 | | 7 | 0 | 0.52, 0.30 | 0.15, 0.10 | 056131-G |
| USA (TX), 1986 (cantaloupe, Aurora) | 2.2 | | 7 | 0 | 0.29, 0.17 | 0.10, 0.06 | 056131-G |
| USA (CA), 1987 (cantaloupe, SJ45) | 2.2 | | 7 ✈ | 0 | 1.6, 1.7, 1.8 | <0.05 (3) | 056131-G |
| USA (CA), 1987 (cantaloupe, SJ45) | 2.2 | | 7[1] | 0 | 4.3, 6.7, 6.4 | 0.11, 0.16, 0.17 | 056131-G |
| USA (CA), 1985 (cantaloupe, SJ45) | 2.2 | | 6 | 0 | 0.74, 1.1 | 0.03, 0.05 | TMN-634A |
| USA (FL), 1985 (cantaloupe, Gold Star) | 2.2 | | 9 | 0 | 0.35, 0.36 | 0.07, 0.09 | TMN-634A |
| USA (FL), 1978 (watermelon/Dixie Lee) | 2.2 | | 6 | 6 | <0.01, <0.01 | | T-4619 |

✈ aerial application
[1] dilute spray

Table 32. Residues of captan and THPI in cucumbers after foliar applications of captan. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Brazil, 1995 (Caipira Safira) | WP | | 0.11 | 4 | 1 | 0.16 | | 5.214-95003719 |
| Brazil, 1995 (Caipira Safira) | WP | | 0.22 | 4 | 1 | 0.22 | | 5.214-95003719 |
| Brazil, 1994 (Safira) | WP | | 0.11 | 4 | 0.08<br>0.5<br>1 | 0.06<br>0.04<br>0.06 | | 5.214-192/95 |
| Brazil, 1994 (Safira) 5.214-192/95 | WP | | 0.22 | 4 | 1 | 0.13 | | 5.214-192/95 |
| Japan, 1969 (Ohtone-1) INDOOR | WP | 1.5 | 0.13 | 3 | 1<br>3<br>7<br>10 | 1.9<br>1.9<br>1.1<br>0.77 | | TMN-609B |
| Japan, 1969 (Ohtone-1) INDOOR | WP | 1.5 | 0.13 | 6 | 1<br>3<br>7<br>10 | 1.9<br>1.1<br>1.1<br>0.56 | | TMN-609B |
| Japan, 1969 (Yamashiro) | WP | | 0.13 | 3 | 1<br>3<br>7<br>10 | 0.24<br>0.06<br><0.01<br><0.01 | | TMN-609B |
| Japan, 1969 (Yamashiro) | WP | | 0.13 | 6 | 1<br>3<br>7<br>10 | 0.20<br>0.05<br><0.01<br><0.01 | | TMN-609B |
| Japan, 1969 (Shinko-A) | WP | 5 | 1.3 | 3 | 1<br>3<br>7<br>10 | 1.5<br>0.45<br>0.19<br>0.08 | | TMN-609B |
| Japan, 1969 (Shinko-A) | WP | 5 | 1.3 | 6 | 1<br>3<br>7<br>10 | 1.2<br>0.42<br>0.19<br>0.04 | | TMN-609B |
| USA (WA), 1986 | WP | 2.2 | | 6 | 0 | 8.7, 10 | 0.60, 0.51 | 056131-J |
| USA (CA), 1986 (Marketmore) | WP | 2.2 | | 6 | 0 | 3.8, 1.7 | 0.15, 0.18 | 056131-J |
| USA (FL), 1986 (Surecrop) | WP | 2.2 | | 6 | 0 | 0.07, 0.06 | <0.05 (2) | 056131-J |
| USA (NJ), 1986 (Lemon cucumber) | WP | 2.2 | | 6 | 0 | 7.1, 0.11 | 0.08, 0.08 | 056131-J |
| USA (TX), 1986 (Sprint) | WP | 2.2 | | 6 | 0 | 0.99, 0.82 | 0.07, 0.07 | 056131-J |
| USA (CA), 1985 | WP | 2.2 | | 6 | 0 | 0.03, 0.10 | <0.01, 0.02 | TMN-609A |
| USA (FL), 1985 (Poinsett) | WP | 2.2 | | 6 | 0 | 0.42, 0.61 | 0.03, 0.07 | TMN-609A |
| USA (NY), 1985 (Marketmore 76) | WP | 2.2 | | 7 | 0 | 1.2, 0.91 | 0.13, 0.11 | TMN-609A |

Table 33. Residues of captan and THPI in squash after six foliar applications of a WP formulation in the USA. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately.

| Location, year (variety) | Application, kg ai/ha | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|
| | | | Captan | THPI | |
| CA,1985 | 2.2 | 0 | 0.12, 0.14 | 0.11, 0.07 | TMN-683 |

| Location, year (variety) | Application, kg ai/ha | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|
| | | | Captan | THPI | |
| FL, 1985 (summer squash, Seneca zucchini) | 2.2 | 0 | 0.26, 0.68 | 0.07, 0.10 | TMN-683 |
| NY, 1985 (squash, Ambassador) | 2.2 | 0 | 1.1, 1.4 | 0.11, 0.14 | TMN-683 |
| USA, 1985 | 2.2 | 6 | <0.01 | | TMN-683 |

Table 34. Residues of captan and THPI in tomatoes after foliar applications of various WP formulations. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | PHI | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | days | Captan | THPI | |
| Brazil, 1991 (Santa Clara) | | 0.12 | 8 | 1<br>7 | 0.46<br>0.34 | 0.27<br>0.12 | RJ1435B |
| Brazil, 1991 (Santa Clara), | | 0.24 | 8 | 1<br>7 | 0.81<br>0.57 | 0.31<br>0.21 | RJ1435B |
| Brazil, 1991 (Santa Clara) | | 0.12 | 8 | 1<br>7 | 0.12<br>0.08 | 0.09<br>0.07 | RJ1435B |
| Brazil, 1991 (Santa Clara) | | 0.24 | 8 | 1<br>7 | 0.27<br>0.18 | 0.16<br>0.10 | RJ1435B |
| Brazil, 1991 (Santa Clara) | | 0.12 | 8 | 1<br>7 | 0.18<br>0.13 | 0.08<br>0.06 | RJ1435B |
| Brazil, 1991 (Santa Clara) | | 0.24 | 8 | 1<br>7 | 0.64<br>0.29 | 0.13<br>0.08 | RJ1435B |
| Brazil, 1993 (Santa Clara) | | 0.12 | 10 | 1<br>2 | <0.01<br>0.02 | | |
| Brazil, 1993 (Santa Clara) | | 0.24 | 10 | 1<br>2 | 0.05<br>0.03 | | |
| Greece, 1991 (Star Pack) | 0.5 | | 1 | 1<br>7<br>15<br>22<br>28 | 0.38, 0.26<br><0.05<br><0.05<br><0.05<br>1.9, <0.05<br>c0.59,<br>c<0.05 | 0.07, 0.07<br>0.08, 0.07<br><0.05<br><0.05<br>0.47 <0.05<br>c0.27,<br>c<0.05 | RJ1189B |
| Greece, 1991 (Rio Grande) | 0.5 | | 1 | 1<br>7<br>15<br>22<br>28 | 0.11, 0.43<br>0.07, 0.09<br><0.05<br><0.05<br>1.4, 2.2<br>c0.78, 1.6 | <0.05<br><0.05<br><0.05<br><0.05<br>0.21, 0.30<br>c0.29, 0.46 | RJ1189B |
| Greece, 1991 (Troyan) | 1.0 | | 1 | 1<br>7<br>14<br>21<br>28 | 0.61<br>0.21<br>0.17<br><0.05<br>0.13 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1189B |
| Greece, 1991 (Troyan) | 1.0 | | 1 | 1<br>7<br>14<br>21<br>28 | 0.15<br><0.05<br><0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05 | RJ1189B |
| Israel, 1991 | 1.3 | | 8 | 0<br>4<br>11 | 0.21<br>0.11<br>0.07 | | CT/40/92 |
| Israel, 1991 | 2.5 | | 8 | 0<br>4<br>11 | 0.32<br>0.20<br>0.07 | | CT/40/92 |
| Japan, 1969 (Fukujyu) INDOOR | 2.3 | 0.13 | 3 | 1<br>3<br>7<br>14 | 0.40<br>0.34<br>0.20<br>0.17 | | TMN-649A |
| Japan, 1969 (Fukujyu) INDOOR | 2.3 | 0.13 | 6 | 1 | 1.1 | | TMN-649A |

| Country (location), year (variety) | Application | | | PHI | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | days | Captan | THPI | |
| | | | | 3<br>7<br>14 | 0.81<br>0.25<br>0.20 | | |
| Japan, 1969 (Hikari) INDOOR | 3.8 | 0.13 | 3 | 1<br>3<br>7<br>14 | 0.28<br>0.45<br>0.23<br>0.19 | | TMN-649A |
| Japan, 1969 (Hikari) INDOOR | 3.8 | 0.13 | 6 | 1<br>3<br>7<br>14 | 0.78<br>0.52<br>0.53<br>0.27 | | TMN-649A |
| Japan, 1969 (Hokan-2) | | 0.13 | 3 | 1<br>3<br>7<br>14 | 0.22<br>0.13<br>0.07<br><0.01 | | TMN-649A |
| Japan, 1969 (Hokan-2) | | 0.13 | 6 | 1<br>3<br>7<br>14 | 0.29<br>0.16<br>0.07<br>0.01 | | TMN-649A |
| Japan, 1969 (Hikari) | 2.5 | 0.13 | 3 | 1<br>3<br>7<br>14 | 0.41<br>0.76<br>0.36<br>0.21 | | TMN-649A |
| Japan, 1969 (Hikari) | 2.5 | 0.13 | 6 | 1<br>3<br>7<br>14 | 0.21<br>0.79<br>0.27<br>0.31 | | TMN-649A |
| Japan, 1969 (Super Hokan) | | 0.13 | 3 | 1<br>3<br>7<br>14 | 0.28<br>0.11<br>0.02<br>0.01 | | TMN-649A |
| Japan, 1969 (Super Hokan) | | 0.13 | 6 | 1<br>3<br>7<br>14 | 0.45<br>0.13<br>0.07<br>0.03 | | TMN-649A |
| Japan, 1969 (Fukujyu) | | 0.13 | 3 | 1<br>3<br>7<br>14 | 2.3<br>1.1<br>0.28<br>0.39 | | TMN-649A |
| Japan, 1969 (Fukujyu) | | 0.13 | 6 | 1<br>3<br>7<br>14 | 1.7<br>0.87<br>0.62<br>0.49 | | TMN-649A |
| Japan, 1969 (Fukujyu) | 2.3 | 0.13 | 3 | 1<br>3<br>7<br>14 | 0.40<br>0.61<br>0.30<br>0.20 | | TMN-649A |
| Japan, 1969 (Fukujyu) | 2.3 | 0.13 | 6 | 1<br>3<br>7<br>14 | 0.50<br>0.29<br>0.22<br>0.38 | | TMN-649A |
| Japan, 1969 (Hikari) | 1.9 | 0.13 | 3 | 3<br>7<br>14 | 1.0<br>0.42<br>0.27 | | TMN-649A |
| Japan, 1969 (Hikari) | 1.9 | 0.13 | 6 | 3<br>7<br>14 | 0.66<br>0.41<br>0.21 | | TMN-649A |
| Mexico, 1992 (Peto 2) | 1.5 | | 3 | 7<br>14 | 0.37<br><0.05 | 0.18<br><0.05 | RJ1431 |
| Mexico, 1992 (Rio Grande), 92JH139, RJ1431 | 1.5 | | 3 | 7<br>14 | 0.12<br>0.13 | <0.05<br>0.13 | RJ1431 |
| USA (CA), 1986, (Peto 19) | 4.2 | | 4 | 0 | 0.55, 0.48 | 0.06, 0.13 | 056131-W |
| USA (CA), 1986, (Peto 19) | 13 | | 4 | 0 | 0.87, 1.8 | 0.12, 0.15 | 056131-W |
| USA (FL), 1986, (Better Boy) | 4.2 | | 4 | 0 | 0.28, 0.43 | 0.09, 0.07 | 056131-W |

| Country (location), year (variety) | Application | | | PHI | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | days | Captan | THPI | |
| USA (NY), 1986, (Heintz 1350) | 4.2 | | 4 | 0 | 1.5, 2.2<br>c0.40 | 0.08, 0.08<br>c<0.05 | 056131-W |
| USA (MI), 1986, (H7814) | 4.2 | | 4 | 0<br>7<br>14 | 1.4, 1.4<br>0.88, 0.52<br>0.18, 0.16 | 0.10, 0.11<br>0.09, 0.08<br>0.08, 0.07 | 056131-W |
| USA (TX), 1986, (Flori Americana) | 4.2 | | 4 | 0 | 1.4, 0.25 | 0.11, 0.10 | 056131-W |
| USA (TX), 1987, (Flori Americana) | 4.2 | | 4 ✈ | 0 | 0.38, 0.53 | 0.08, 0.11 | 40189823 |
| USA (TX), 1987, (Flori Americana) | 4.2 | | 4[1] | 0 | 2.1, 0.51 | 0.32, 0.27 | 40189823 |

✈ aerial application
[1] dilute spray

Table 35. Residues of captan and THPI in soya beans after foliar application in Thailand.

| Country, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Thailand, 1989 (Sukhothai 1) | WP | 0.5 | 0.1 | | 39 | <0.001 | | |
| | WP | 1.0 | 0.2 | | 39 | <0.001 | | |

Table 36. Residues of captan and THPI in potatoes after foliar, soil and seed applications of various captan formulations. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| Foliar application | | | | | | | | |
| Brazil, 1992 (Monalisa) | WP | | 0.12 | 8 | 14 | <u><0.01</u> | | D3.2.1/07 |
| Brazil, 1992 (Monalisa) | WP | | 0.24 | 8 | 14 | <0.01 | | D3.2.1/07 |
| Brazil, 1994 (Monalisa) | WP | | 0.12 | 7 | 7 | <u><0.05</u> | | D3.2.1/07 |
| Brazil, 1994 (Monalisa) | WP | | 0.24 | 7 | 7 | <0.05 | | D3.2.1/07 |
| Canada, 1993 (Russet Burbank) | WP | 3 | | 3 | 6 | <0.05 | <0.05 | RJ1602B |
| Canada, 1993 (Russet Burbank) | WP | 3 | | 3 | 6 | <0.05 | <0.05 | RJ1602B |
| Canada, 1993 (Russet Burbank) | WP | 6 | | 3 | 6 | <0.05 | <0.05 | RJ1602B |
| Canada, 1993 (Chieftain) | WP | 3 | | 5 | 7 | <0.05 | <0.05 | RJ1602B |
| Canada, 1993 (Chieftain) | WP | 3 | | 5 | 7 | <0.05 | <0.05 | RJ1602B |
| Canada, 1993 (Chieftain) | WP | 6 | | 5 | 7 | <0.05 | 0.06 | RJ1602B |
| Canada, 1993 (Chieftain) | WP | 3 | | 5 | 6 | <0.05 | <0.05 | RJ1602B |
| Canada, 1993 (Chieftain) | WP | 3 | | 5 | 6 | <0.05 | <0.05 | RJ1602B |
| Canada, 1993 (Chieftain) | WP | 6 | | 5 | 6 | <0.05 | 0.05 | RJ1602B |
| Canada, 1994 (Russet Burbank) | DF | 3.6 | | 7 | 5 | <0.05 | 0.11 | RJ1840B |
| Canada, 1994 (Russet Burbank) | DF | 3.6 | | 7 | 5 | <0.05 | 0.07 | RJ1840B |
| Canada, 1994 (Russet Burbank) | DF | 7.2 | | 7 | 5 | 0.06 | 0.16 | RJ1840B |
| Canada, 1994 (Russet Burbank) | DF | 3.6 | | 7 | 7 | <0.05 | <0.05 | RJ1840B |
| Canada, 1994 (Russet Burbank) | DF | 3.6 | | 7 | 7 | <0.05 | <0.05 | RJ1840B |
| Canada, 1994 (Russet Burbank) | DF | 7.2 | | 7 | 7 | <0.05 | <0.05 | RJ1840B |
| Mexico, 1991 (Alpha) | WP | 1.0 | 0.33 | 4 | 7 | <u><0.05</u> | <0.05 | RJ1432B |
| Mexico, 1991 (Alpha) | WP | 0.83 | 0.33 | 4 | 14 | <u><0.05</u> | <0.05 | RJ1432B |
| Mexico, 1991 (Alpha) | WP | 0.83 | 0.33 | 4 | 6<br>13 | <0.05<br><u><0.05</u> | <0.05<br><0.05 | RJ1432B |
| Mexico, 1991 (Alpha) | WP | 0.83 | 0.33 | 4 | 7<br>14 | <0.05<br><u><0.05</u> | <0.05<br><0.05 | RJ1432B |
| Netherlands, 1992 (Maritiema) | FL | 1.9 | | 11 | 0<br>0<br>13 | <0.02 (2)<br><0.02 (2)<br><0.02 (2) | <0.1,<br><0.1<br><0.1, | R-6596 |

| Country (location), year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Captan | THPI | |
| | | | | | 13 | <0.02 (2) | <0.1<br><0.1,<br><0.1<br><0.1,<br><0.1 | |
| Netherlands, 1992 (Maritiema) | FL | 3.8 | | 11 | 0<br>0<br>13<br>13 | <0.02, (2)<br><0.02 (2)<br><0.02 (2)<br><0.02 (2) | <0.1,<br><0.1<br><0.1,<br><0.1<br><0.1,<br><0.1<br><0.1,<br><0.1 | R-6596 |
| Poland 1994 (Bogna) | WP | 1.5 | 0.5 | 1 | 16 | <0.05 | | |
| Poland 1995 (Atos, Cisa, Tarpan) | WP | 1.8 | | 9 | 32 | <0.05 | | |
| UK, 1992 (Maris Piper) | FL | 1.2 | | 6 | 20 | <0.01<br><0.01 | <0.01<br><0.01 | R-6917 |
| UK, 1992 (Maris Piper) | FL | 2.4 | | 6 | 20 | <0.01<br><0.01 | <0.01<br><0.01 | R-6917 |
| UK, 1992 (King Edward) | FL | 1.2 | | 5 | 47 | <0.01<br><0.01 | <0.01<br><0.01 | R-6917 |
| UK, 1992 (King Edward) | FL | 2.4 | | 5 | 47 | <0.01<br><0.01 | <0.01<br><0.01 | R-6917 |
| Broadcast application to open furrow at planting | | | | | | | | |
| Mexico, 1996 (Alpha) | SC | 9.1 | | 1 | 103 | <0.01,<br><0.01 | | AA960302 |
| Mexico, 1996 (Alpha) | SC | 20 | | 1 | 103 | <0.01,<br><0.01 | | AA960302 |
| Mexico, 1996 (Alpha) | SC | 11 | | 1 | 141 | <0.01,<br><0.01 | | AA960302 |
| Mexico, 1996 (Alpha) | SC | 23 | | 1 | 141 | <0.01,<br><0.01 | | AA960302 |
| Seed treatment | | | | | | | | |
| USA (CA), 1986 (White Rose), seed Tx | WP | | 0.75/100 kg seed | 1 | 74<br>116 | <0.05<br><0.05 | <0.05<br><0.05 | 056131-R |
| USA (MA), 1986 (Superior) seed Tx | WP | | 1.4/100 kg seed | 1 | 61<br>105 | <0.05<br><0.05 | <0.05<br><0.05 | 056131-R |
| USA, 1978 (seed Tx) | WP | | 0.75-1.2/100 kg seed | 1 | 135 | 0.00 | | TMN-665A |

Table 37. Residues of captan and THPI in indoor grown radishes after pre-emergent application of a captan WP formulation in Germany.

| Year (variety) | Application | | | PHI, days | Residue, mg/kg | Ref. |
|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | | |
| 1975 (Rota) | 66[1] | 0.17 | 1 | 21 | <0.03 | BBA57/75 |
| 1976 (Cherry Belle) | 66[1] | 0.17 | 1 | 38 | <0.03 | BBA 2559 |
| 1976 (Cherry Belle) | 66[1] | 0.17 | 1 | 112 | <0.03 | BBA 2553 |
| 1975 (Hilmar Treib) | 66[1] | 0.17 | 1 | 47 | <0.03 | BBA 2709 |
| 1975 (Neckar-perle) | 66[1] | 0.17 | 1 | 47 | <0.03 | BBA 2710 |
| 1976 (Karissma GS kalibriert) | 66[1] | 0.17 | 1 | 48 | <0.03 | BBA 1482 |
| 1975 (Cherry Belle) | 66[1] | 0.17 | 1 | 97 | <0.03 | BBA 3200 |
| 1976 (Roky) | 66[1] | 0.17 | 1 | 54 | <0.03 | BBA 72/76 75/76 |

[1]watering pre-emergence at 8 g ai/m$^2$, 4 l water/m$^2$

Table 38. Residues of captan and THPI in almonds after application of a captan WP formulation in the USA. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately. Doubly underlined residues are from treatments according to GAP and were used to estimate maximum residue levels.

| Location, year (variety) | Application | | PHI, days | Sample | Residue, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | No. | | | Captan | THPI | |
| CA, 1986 (Price) | 5.0 | 4 | 130 | Nut | <0.05, 0.20<br>c0.06 | 0.09, <0.05<br>c<0.05 | 056131-B |
| | | | | Hull | 40, 27 | 0.33, 0.23 | |
| | | | | Shell | 3.0, 0.77 | 0.31 <0.05 | |
| CA, 1986 | 5.0 | 5 | 30 | Nut | 0.10, 0.09 | 0.05 <0.05 | 056131-B |
| | | | | Hull | 48, 13 | 0.21, 0.05 | |
| | | | | Shell | 1.2, 3.8 | <0.05 0.43 | |
| CA, 1986 (Non-Pareil) | 5.0 | 4 | 102 | Nut | ≤0.05 (2) | <0.05 (2) | 056131-B |
| | | | | Hull | 25, 16 | 0.30, 0.54 | |
| | | | | Shell | 1.6, 1.1 | 0.16, 0.05 | |
| CA, 1986 (Non-Pareil) | 5.0 | 5 | 30 | Nut | ≤0.05 (2) | <0.05 (2) | 056131-B |
| | | | | Hull | 13, 13 | 0.36, 0.25 | |
| | | | | Shell | 1.3, 2.2 | 0.14, 0.23 | |
| CA, 1986 (Mission) | 5.0 | 4 | 124 | Nut | ≤0.05 (2) | <0.05 (2) | 056131-B |
| | | | | Hull | 20, 16<br>c0.63 | 0.28, 0.35<br>c<0.05 | |
| | | | | Shell | 0.42, 0.54 | <0.05 (2) | |
| CA, 1986 (Non-Pareil) | 5.0 | 5 | 30 | Nut | ≤0.05 (2) | 0.15, 0.11 | 056131-B |
| | | | | Hull | 40, 54 | 2.07, 1.31 | |
| | | | | Shell | 1.1, 0.81 | 0.49, 0.27 | |
| CA, 1987 (Non-Pareil) | 5.0 | 4✈ | 142 | Nut | ≤0.05 | <0.05 | 40189802 |
| | | | | Shell | 0.15, 0.14, 0.09 | <0.05 (3) | |
| | | | | Hull | 3.8, 3.6, 4.5<br>c0.11 | 0.08, 0.11, 0.17<br>c0.07 | |
| CA, 1987 (Non-Pareil) | 5.0 | 4[1] | | Nut | ≤0.05 | <0.05 | 40189802 |
| | | | | Shell | 0.17, 0.16, 0.07 | <0.05 (3) | |
| | | | | Hull | 6.7, 4.5, 5.3<br>c0.11 | 0.15, 0.13, 0.17<br>c0.07 | |
| CA, 1987 (Non-Pareil) | 5.0 | 4[2] | | Nut | ≤0.05 | <0.05 | 40189802 |
| | | | | Shell | 0.19, 0.18, 0.07 | <0.05 (3) | |
| | | | | Hull | 6.7, 7.7, 6.8<br>c0.11 | 0.28, 0.21, 0.22<br>c0.07 | |
| CA, 1980 (Non-Pareil) | 4.5 | 2 | 195 | Kernel | ≤0.03 | <0.01 | TMN-562A |
| | | | | Hulls | 0.05 | <0.01 | |
| | | | | Shells | <0.03 | <0.01 | |
| CA, 1982 (Mission) | 4.5 | 8 | 160 | Kernel | 0.01, <0.01<br>c0.02 | <0.01, <0.01<br>c<0.01 | TMN-562A |
| | | | | Hulls | 5.6, 8.4<br>c0.13 | 0.10, 0.17<br>c0.01 | |
| | | | | Shells | 0.13, 0.44<br>c0.01 | 0.02, 0.03<br>c<0.01 | |
| CA, 1985 (Non-Pareil) | 5.6 | 4 | 128 | Kernel | 0.04, 0.03 | <0.01, <0.01 | TMN-562A |
| | | | | Hulls | 6.4, 4.3 | 0.05, 0.08 | |
| | | | | Shells | 0.09, 0.06<br>c0.01 | <0.01 (2)<br>c<0.01 | |
| CA, 1985 (Non-Pareil) | 5.6 | 4 ✈ | 152 | Kernel | ≤0.01 (2) | <0.01 (2) | TMN-562A |
| | | | | Hulls | 0.28, 0.19 | <0.01 (2) | |
| | | | | Shells | 0.01 (2) | <0.01 (2) | |

✈ aerial application
[1] concentrated spray
[2] dilute spray

Table 39. Residues of captan and THPI in indoor-grown chives after pre-emergent application of a captan WP formulation in Germany. Analyses of replicate field samples from one plot or from duplicate plots in one trial are shown separately.

| Year (variety) | Application | | | PHI, days | Residue, mg/kg | Ref. |
|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | | |
| 1976 (Fein-stengeliger) | 66[1] | 0.17 | 1 | 12 | 20, 32 | BBA 4/75 MZ |
| 1976 (Hild 68) | 66[1] | 0.17 | 1 | 22 | 1.4 | BBA 1480 |
| 1976 (Hild 68) | 66[1] | 0.17 | 1 | 15 | 0.62 | BBA 1481 |
| 1976 (Feinröhriger) | 66[1] | 0.17 | 1 | 32 | 0.13 | BBA 2708 |
| 1976 (Feinröhriger) | 66[1] | 0.17 | 1 | 26 | 0.26 | BBA 2706 |
| 1976 | 66[1] | 0.17 | 1 | 19 | 3.5 | BBA 2569 |

[1]watering at 8 g ai/$m^2$, 4 l water/$m^2$

Animal feeding studies

Groups of 4 lactating Holstein dairy cattle (487-737 kg bw) were dosed with captan at levels nominally equivalent to 0, 10, 30 and 100 ppm in the diet for 29 days (Wiebe, 1991). Doses were administered by gelatin capsule. Milk was collected from each of the cows before dosing and on days 1, 4, 7, 10, 14, 21 and 28 of dosing. Three cows from each group were slaughtered on day 29, three hours after receiving the final dose. The remaining cow from each group was placed on a captan-free diet, milked at intervals, and slaughtered 7 days after receiving the last dose. Samples of muscle, liver, kidney, fat and milk were analysed for five captan metabolites: THPI, *trans*-3-OH THPI, *trans*-5-OH THPI, *cis*-3-OH THPI and *cis*-5-OH THPI. Ground samples of tissue were prepared within 35 hours of slaughter and stored at -20°.

Neither of the *cis* isomers was detected in the milk or tissue samples. Residues of THPI and the *trans* isomers reached a plateau in milk by 1-4 days of dosing. Residue levels of the individual isomers were >400 times less than the corresponding captan feeding levels. No residues of the metabolites were detectable in milk by three days after the cessation of dosing or in tissues seven days after administration of the last dose.

Table 40. Mean residues of captan metabolites in milk of dairy cows dosed with captan at levels nominally equivalent to 10, 30 and 100 ppm in the diet for 29 days (Wiebe, 1991).

| Day | Residue, mg/kg | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 10 ppm feed level | | | 30 ppm feed level | | | 100 ppm feed level | | |
| | THPI | *trans*-3-OH THPI | *trans*-5-OH THPI | THPI | *trans*-3-OH THPI | *trans*-5-OH THPI | THPI | *trans*-3-OH THPI | *trans*-5-OH THPI |
| 1 | <0.01 | 0.02 | <0.01 | 0.03 | 0.08 | 0.01 | 0.15 | 0.31 | 0.06 |
| 4 | <0.01 | 0.02 | <0.01 | 0.02 | 0.06 | <0.01 | 0.16 | 0.25 | 0.04 |
| 7 | <0.01 | 0.02 | <0.01 | 0.03 | 0.06 | <0.01 | 0.30 | 0.28 | 0.04 |
| 10 | <0.01 | 0.02 | <0.01 | 0.02 | 0.06 | <0.01 | 0.19 | 0.18 | 0.03 |
| 14 | <0.01 | 0.02 | <0.01 | 0.03 | 0.06 | <0.01 | 0.17 | 0.20 | 0.03 |
| 21 | <0.01 | 0.02 | <0.01 | 0.03 | 0.06 | <0.01 | 0.20 | 0.21 | 0.03 |
| 28 | <0.01 | 0.02 | <0.01 | 0.03 | 0.06 | <0.01 | 0.21 | 0.23 | 0.04 |
| 30 | <0.01 | <0.01 | <0.01 | 0.01 | <0.01 | <0.01 | 0.02 | 0.10 | <0.01 |
| 32 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 |
| 35 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 | <0.01 |

Table 41. Residues of captan metabolites in tissues of dairy cows dosed with captan at levels nominally equivalent to 10, 30 and 100 ppm in the diet for 29 days (Wiebe, 1991). Maximum sampling to co-extraction intervals were 159 days for milk, 317 days for fat, 311 days for kidneys, 316 days for liver and 315 days for muscle.

| Sample | Residue, mg/kg | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | 10 ppm feed level | | | 30 ppm feed level | | | 100 ppm feed level | | |
| | THPI | *trans*-3-OH THPI | *trans*-5-OH THPI | THPI | *trans*-3-OH THPI | *trans*-5-OH THPI | THPI | *trans*-3-OH THPI | *trans*-5-OH THPI |
| Muscle | 0.02 (2), 0.01 | 0.02 (2), 0.01 | <0.01 (3) | 0.04, 0.05, 0.13 | 0.04, 0.05, 0.08 | 0.01 (3) | 0.36, 0.26, 0.10 | 0.21 (2), 0.13 | 0.04, 0.06, 0.02 |
| Liver | 0.02 (2), 0.03 | 0.01, <0.01 (2) | <0.01 (3) | 0.08 (2), 0.20 | 0.03 (2), 0.07 | <0.01 (3) | 0.49, 0.28, 0.15 | 0.13, 0.12, 0.09 | <0.01 (3) |
| Kidney | 0.02 (2), 0.01 | 0.02 (3) | <0.01 (3) | 0.06 (2), 0.15 | 0.07, 0.08, 0.13 | 0.02 (3) | 0.38, 0.27, 0.11 | 0.34, 0.30, 0.18 | 0.08, 0.10, 0.03 |
| Fat | <0.01 (3) | <0.01 (3) | <0.01 (3) | 0.01, 0.02, 0.05 | <0.01 (3) | <0.01 (3) | 0.12, 0.08, 0.03 | 0.04, 0.02, 0.01 | <0.01 (3) |

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### In processing

Processing studies on apples, cherries, citrus fruit, cucumbers, melons, grapes, plums and tomatoes were reported to the Meeting. Captan is susceptible to hydrolysis with cleavage of the N-S bond, resulting in the formation of THPI. Processing can result in increased conversion of captan to THPI. The 1994 and 1997 evaluations of captan introduced the use of processing yields for THPI.

Processing yield = (THPI residues in processed commodity) ÷ (RAC captan residues × 0.503 + RAC THPI residues).

The factor 0.503 is the ratio of the molecular weight of THPI (151.2) to that of captan (300.6).

Residues and, where practicable, processing factors for captan and processing yields for THPI are shown in Tables 42-59.

Citrus fruit. Captan was applied to oranges at 5.7-12 kg ai/ha by hand sprayers and tractor-driven sprayers in the USA. Fruit harvested on the day of the last spray were processed according to Kesterson and Braddock (1979). Oranges were washed, juice and peel separated, oil extracted from a portion of the peel, the remaining peel shredded and lime added, and the shredded peel pressed and dried.

Table 42. Captan residues in citrus processing fractions in the USA.

| Location, year (variety) | Rate, kg ai/ha | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield | Ref. |
|---|---|---|---|---|---|---|---|
| Oranges | | | | | | | |
| FL, 1981 (Hamlin) | 5.7 | Fruit | <0.03 | | <0.01 | | TMN-603B |
| | | Dried pulp | <0.03 | | <0.01 | | |
| FL, 1981 (Valencia) | 5.7 | Fruit | <0.03 | | <0.01 | | TMN-603B |
| | | Dried pulp | <0.03 | | 0.28 | 28 | |
| FL, 1982 (Valencia) | 6.7 | Fruit | 0.50 | | 0.12 | | TMN-603B |
| | | Dry pulp | <0.03 | <0.06 | 0.04 | 0.1 | |
| CA, 1983 (Navel) | 2×12 | Fruit | 2.7, 2.4 | | 1.8, 1.3 | | TMN-603B |

| Location, year (variety) | Rate, kg ai/ha | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield | Ref. |
|---|---|---|---|---|---|---|---|
| | | Juice | <0.01 | <0.01 | <0.01 | <0.01 | |
| | | Peel oil | 0.42 | 0.2 | 0.02 | <0.01 | |
| | | Dried peel | <0.01 | <0.01 | 0.10 | 0.04 | |
| CA, 1986 (Valencia) | 2×9.0 + 1×5.6 | Fruit | 2.0, 1.5 | - | 0.03, 0.02 | - | TMN-603B |
| | | Washed | 0.08 | 0.05 | 0.02 | 0.02 | |
| | | Washed peel | 0.02 | 0.01 | 0.08 | 0.09 | |
| | | Peeled fruit | <0.01 | <0.01 | 0.01 | 0.01 | |
| | | $CH_2Cl_2$ washed fruit | <0.01 | <0.01 | 0.02 | 0.02 | |
| | | Commerciall y washed fruit | 0.19 | 0.1 | <0.01 | <0.01 | |
| | | Wet peel | 0.01 | <0.01 | 0.12 | 0.1 | |
| | | Dried peel | <0.01 | <0.01 | <0.01 | <0.01 | |
| | | Dried fines of peel | <0.01 | <0.01 | 0.02 | 0.02 | |
| | | Juice | <0.01 | <0.01 | <0.01 | <0.01 | |
| | | Molasses | <0.01 | <0.01 | <0.01 | <0.01 | |
| | | Peel oil | <0.01 | <0.01 | <0.01 | <0.01 | |
| AZ, 1986 (Valencia) | 2×9.0 + 1×5.6 | Fruit | 2.3, 2.3 | | 0.03, 0.04 | - | TMN-603B |
| | | Washed peel | 0.23 | 0.1 | 0.47 | 0.4 | |
| | | Peeled fruit | 0.01 | <0.01 | 0.01 | 0.01 | |
| | | Washed fruit | 0.40 | 0.2 | 0.09 | 0.08 | |
| | | $CH_2Cl_2$ washed fruit | 0.26 | 0.1 | 0.02 | 0.02 | |
| Lemons | | | | | | | |
| AZ, 1986 (Lisbon) | 2×9.0 | Fruit | 7.0, 8.5 c0.13 | - | <0.01 | - | TMN-603A |
| | | Peeled fruit | 0.02 | 0.003 | 0.11 | 0.01 | |
| | | Peel | 0.51 | 0.07 | 1.7 | 0.2 | |
| | | Washed fruit | 1.3 | 0.2 | 0.08 | 0.01 | |
| | | Solvent washed | 0.92 | 0.1 | 0.16 | 0.02 | |
| CA, 1986 (Eureka) | 2×9.0 | Fruit | 4.6, 4.5 c0.05 | - | 0.08 (2) <0.01 | - | TMN-603A |
| | | Washed peel | 0.89 | 0.2 | 0.12 | 0.05 | |
| | | Juice | 0.11 | 0.02 | 0.08 | 0.03 | |
| | | Washed fruit | 0.26 | 0.06 | 0.05 | 0.02 | |
| | | Solvent washed | 0.86 | 0.2 | 0.10 | 0.04 | |
| Grapefruit | | | | | | | |
| AZ, 1986 (Marsh/Red Blush) | 2×9.0 | Fruit | 2.4, 4.2 c0.64 | - | 0.05, 0.09 <0.01 | 0.01 | TMN-603C |
| | | Peeled fruit | <0.01 | <0.01 | 0.01 | 0.01 | |
| | | Washed peel | 0.32 | 0.1 | 0.52 | 0.3 | |
| | | Washed fruit | 0.59 | 0.2 | 0.04 | 0.02 | |
| | | Solvent washed fruit | 0.17 | 0.05 | 0.04 | 0.02 | |
| | | Dried peel | 0.14, 0.11 | 0.04 | 0.16, 0.16 | 0.09 | |
| | | Dried fines of peel | 0.13, 0.12 | 0.04 | 0.51, 0.12 | 0.2 | |
| | | Wet peel | 0.07, 0.06 | 0.02 | 0.17, 0.13 | 0.09 | |
| | | Peel oil | 0.54, 1.1 | 0.24 | 0.16, 0.12 | 0.08 | |
| | | Molasses | <0.01 (2) | <0.01 | 0.14, 0.06 | 0.06 | |
| | | Juice | <0.01 | <0.01 | <0.01 | <0.01 | |

Apples. Processing studies on apples were carried out in Germany in 1991, 1994 and 1996 and in the USA in 1986 and 1990.

The fate of captan in processed apple commodities was studied by Specht (1992) using Golden Delicious apples that had been sprayed 12 times with captan (WP or SG formulations) at 1.6 kg ai/ha. The apples, harvested 14 days after the last application, were processed according to normal domestic procedures. Apple sauce was made by cooking apples, cut into small pieces, in water until soft and passing through a sieve. For apple juice (warm), apples were cut into small pieces and placed in a steam juice extractor. The residue after removal of the juice was pomace (juice warm). Apple juice (cold) was produced by a juice extractor. The centrifuged juice was collected and the residue taken as pomace (juice cold). Dried apples were prepared by desiccation of cored apples.

Table 43. Captan residues in Golden Delicious apples and processed apple commodities in Germany, 1991 (Specht, 1992).

| Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|
| Apples, washed | 2.2<br>c0.01 | - | 0.14<br>c0.05 | - |
| Sauce | <0.01 | <0.01 | 0.92 | 0.7 |
| Juice (warm) | <0.01 | <0.01 | 1.3 | 1.0 |
| Pomace (juice warm) | 0.03 | 0.01 | 1.5 | 1.2 |
| Juice (cold) | 0.10<br>c0.01 | 0.05 | 0.07<br>c<0.01 | 0.06 |
| Pomace (juice cold) | 3.8<br>c0.09 | 1.7 | 0.99<br>c<0.01 | 0.8 |
| Dried apples | 2.8<br>c0.02 | 1.3 | 3.2<br>c0.05 | 2.6 |
| Apples, washed | 1.8<br>c0.01 | - | 0.18<br>c0.05 | - |
| Sauce | <0.01 | <0.01 | 1.2 | 1.1 |
| Juice (warm) | <0.01 | <0.01 | 1.1 | 1.1 |
| Pomace (juice warm) | 0.01 | <0.01 | 1.2 | 1.1 |
| Juice (cold) | 0.25<br>c0.01 | 0.1 | 0.13<br>c<0.01 | 0.1 |
| Pomace (juice cold) | 5.2<br>c0.09 | 2.9 | 0.84<br>c<0.01 | 0.8 |
| Dried apples | 1.4<br>c0.02 | 0.8 | 2.7<br>c0.05 | 2.5 |
| Apples, washed | 1.1 | - | 0.21 | - |
| Sauce | <0.01 | <0.01 | 1.0 | 1.3 |
| Juice (warm) | <0.01 | <0.01 | 0.88 | 1.1 |
| Pomace (juice warm) | 0.02 | 0.02 | 0.87 | 1.1 |
| Dried apples | 2.2<br>c0.03 | 1.9 | 2.1<br>c<0.01 | 2.7 |
| Apples, washed | 1.8 | - | 0.36 | - |
| Sauce | <0.01 | <0.01 | 0.77 | 0.6 |
| Juice (warm) | <0.01 | <0.01 | 1.1 | 0.9 |
| Pomace (juice warm) | 0.03 | 0.02 | 1.1 | 0.9 |
| Dried apples | 2.0<br>c0.03 | 1.1 | 1.8<br>c<0.01 | 1.4 |

Fuchsbichler (1995) studied the effect of processing on captan residues in apples. Apple sauce and apple juice (warm and cold) were produced as described above.

Table 44. Captan residues in apples and processed apple commodities in Germany, 1994 (Fuchsbichler, 1995).

| Variety | Rate, kg ai/ha | PHI, days | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|---|---|
| Indared | 12×1.5 | 3 | Fruit | 2.0 | - | - | |
| | | 3 | Juice cold | 0.06 | 0.03 | - | |
| | | 3 | Juice heated | <0.05 | <0.03 | - | |
| | | 3 | Apple sauce | <0.05 | <0.03 | - | |
| | | 7 | Fruit | 2.0 | - | - | |
| | | 7 | Juice cold | 0.08 | 0.04 | - | |
| | | 7 | Juice heated | <0.05 | <0.03 | - | |
| | | 7 | Apple sauce | <0.05 | <0.03 | - | |
| | | 14 | Fruit | 1.3 | - | 0.43 | - |
| | | 14 | Juice cold | 0.07 | 0.05 | <0.02 | <0.02 |
| | | 14 | Juice heated | <0.05 | <0.04 | 0.58 | 0.5 |
| | | 14 | Apple sauce | <0.05 | <0.04 | 0.51 | 0.5 |
| Elstar | 12×1.9 | 3 | Fruit | 3.5 | - | - | |
| | | 3 | Juice cold | 0.51 | 0.15 | - | |
| | | 3 | Juice heated | <0.05 | <0.02 | - | |
| | | 3 | Apple sauce | <0.05 | <0.02 | - | |
| | | 14 | Fruit | 2.3 | - | 1.1 | - |
| | | 14 | Juice cold | 0.11 | 0.05 | 0.43 | 0.2 |
| | | 14 | Juice heated | <0.05 | <0.03 | 2.8 | 1.2 |
| | | 14 | Apple sauce | <0.05 | <0.03 | 1.7 | 0.8 |
| Cox's Orange | 12×1.9 | 3 | Fruit | 2.5 | - | - | |
| | | 3 | Juice cold | 0.27 | 0.1 | - | |
| | | 3 | Juice heated | <0.05 | <0.03 | - | |
| | | 3 | Apple sauce | <0.05 | <0.03 | - | |
| | | 14 | Fruit | 2.2 | - | 0.83 | - |
| | | 14 | Juice cold | 0.15 | 0.07 | 0.68 | 0.4 |
| | | 14 | Juice heated | <0.05 | <0.03 | 2.2 | 1.1 |
| | | 14 | Apple sauce | <0.05 | <0.03 | 1.4 | 0.7 |

Apples were processed by washing and pressing twice at 50-60 bar for 2 minutes to produce unclarified juice and pomace (Fuchsbichler, 1997). Pasteurized juice was prepared by heating unclarified juice at 93-95°C for 1 minute. Apple quarters were boiled (98°C) for 45 min and then sieved to give apple purée (sauce). Dried apples were prepared by drying apple slices at 60-61°C for 8.5 hours to a moisture content of about 20%.

Table 45 Captan residues in apples and processed apple commodities in Germany, 1996 (Fuchsbichler, 1997).

| Variety | Rate , kg ai/ha | PHI, days | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|---|---|
| Golden Delicious | 12×1.3 | 14 | Fruit | 1.6 | - | 0.77 | - |
| | | 14 | Washed | 0.43 | 0.3 | 0.22 | 0.1 |
| | | 14 | Unclarified juice | 0.08 | 0.05 | - | - |
| | | 14 | Pasteurized unclarified juice | <0.05 | <0.03 | - | - |
| | | 14 | Apple purée | <0.05 | <0.03 | - | - |
| | | 14 | Dried apples | <0.05 | <0.03 | <0.20 | <0.1 |
| Gloster | 12×1.3 | 13 | Fruit | 1.2 | - | 0.63 | - |
| | | 13 | Washed | 0.37 | 0.3 | <0.20 | <0.2 |
| | | 13 | Unclarified juice | 0.12 | 0.1 | - | - |
| | | 13 | Pasteurized unclarified juice | <0.05 | <0.04 | - | - |
| | | 13 | Apple purée | <0.05 | <0.04 | - | - |
| | | 13 | Dried apples | <0.05 | <0.04 | <0.20 | <0.2 |
| Golden Delicious | 13×1.8 | 14 | Fruit | 2.2 | - | 2.7 | - |
| | | 14 | Washed | 1.2 | 0.5 | 0.59 | 0.2 |
| | | 14 | Unclarified juice | 0.19 | 0.09 | - | - |
| | | 14 | Pasteurized unclarified juice | <0.05 | <0.02 | - | - |
| | | 14 | Dried apples | <0.05 | <0.02 | <0.20 | <0.05 |

Smith (1987p) treated apple trees 8 times with captan (WP formulation) with a foliar spray. Apples were harvested on the day of the last application and processed. They were graded, peeled, cored and trimmed, crushed and pulped. A sample of the pulp was collected and peels, cores and trimmings added to produce wet pomace. A sample of wet pomace was dried at 53°C to produce dry pomace. Processing of the pulp was continued by straining, enzyme clarification and filtration. The juice was heated to 88°C and canned.

Table 46. Captan residues in Red Delicious apples, pomace and juice after processing in Washington, USA, 1986 (Smith, 1987p).

| Rate, kg ai/ha | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|
| 8×4.5 | Apples | 5.5, 5.1 | - | 0.23, 0.21 | - |
| | Dry pomace | 10 | 1.9 | 12 | 4.2 |
| | Wet pomace | 2.1<br>c0.1 | 0.4 | 1.9<br>c<0.05 | 0.7 |
| | Juice | <0.05 | <0.01 | 0.10 | 0.03 |
| 8×13 | Apples | 9.1, 5.5 | - | 0.40, 0.43 | - |
| | Dry pomace | 7.9 | 1.1 | 42 | 10 |
| | Wet pomace | 7.8<br>c0.1 | 1.1 | 3.5<br>c<0.05 | 0.9 |
| | Juice | <0.05 | <0.01 | 2.2 | 0.5 |

Iwata (1992a) applied captan to apples at two different locations in the USA as 10 foliar sprays at 3.4 kg ai/ha. The spray intervals were 14 days at the Michigan site (Yellow Delicious) and 7-17 days at the West Virginia site (Spartan). Apples were harvested 14-97 days after the last spray and processed according to normal commercial practices as shown in Figure 6.

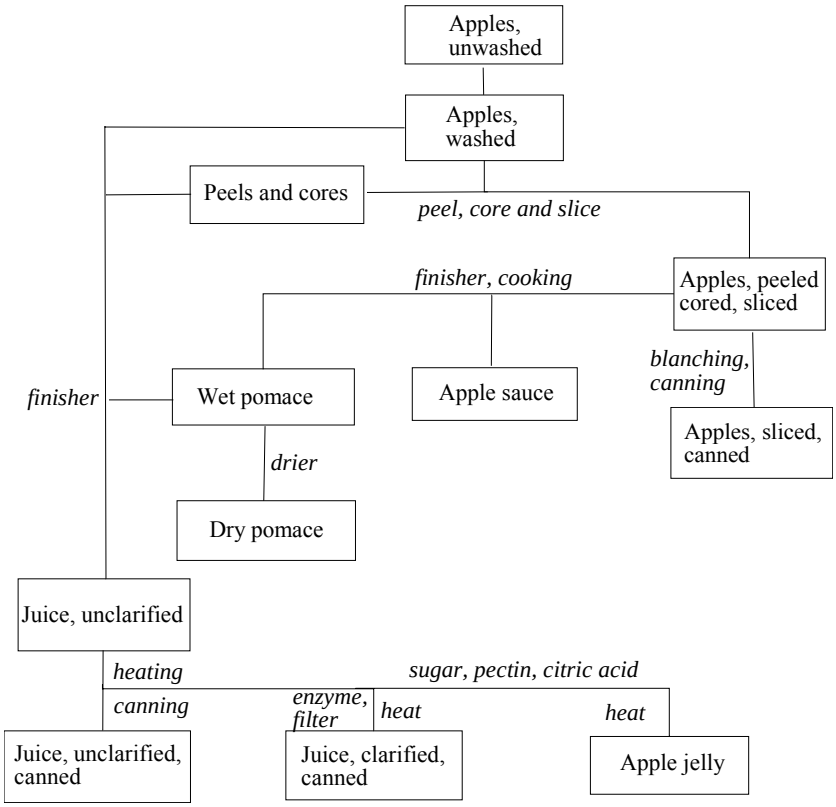



Figure 6. Apple processing (Iwata, 1992a).

Table 47. Residues of captan and THPI in apples and processed apple commodities after foliar applications of captan in Michigan, USA. Captan WP was applied to Yellow Delicious apples at 10×3.4 kg ai/ha at 14-day intervals (Iwata, 1992a).

| Commodity | Residues, mg/kg | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | PHI 97 days | | PHI 83 days | | PHI 69 days | | PHI 55 days | | PHI 41 days | |
| | Captan | THPI | Captan | THPI | Captan | THPI | Captan | THPI | Captan | THPI |
| Apples, field | 0.06 | <0.05 | 0.15 | <0.05 | 0.36 | <0.05 | 0.40 | <0.05 | 0.56 | <0.05 |
| Unwashed | 0.06 | <0.05 | 0.10 | <0.05 | 0.19 | <0.05 | 0.36 | <0.05 | 0.44 | <0.05 |
| Washed + rinsed | <0.05 | <0.05 | <0.05 | <0.05 | 0.10 | <0.05 | 0.29 | <0.05 | 0.17 | <0.05 |
| Peeled, cored, sliced | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| Apple sauce | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| Baby food apple sauce | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| Canned apple slices | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| Unclarified juice | <0.05 | <0.05 | <0.05 | <0.05 | 0.13 | 0.05 | 0.19 | 0.08 | 0.29 | 0.10 |
| Canned, unclarified juice | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | 0.11 | <0.05 | 0.18 | <0.05 | 0.27 |
| Filter cake | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | 0.10 | <0.05 | 0.13 | 0.06 | 0.19 |
| Canned clarified juice | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | 0.08 | <0.05 | 0.12 | <0.05 | 0.18 |
| Apple jelly | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | 0.08 | <0.05 | 0.11 |
| Wet pomace | <0.05 | <0.05 | <0.05 | <0.05 | 0.06 | 0.09 | <0.05 | 0.15 | 0.07 | 0.21 |
| Dry pomace | 0.05 | 0.08 | <0.05 | 0.10 | 0.11 | 0.33 | 0.13 | 0.59 | 0.13 | 0.84 |

Table 48. Residues of captan and THPI in apples and processed apple commodities after foliar applications of captan in West Virginia, USA. Captan WP was applied to Spartan apples at 10×3.4 kg ai/ha at 7-17 day intervals (Iwata, 1992a).

| Commodity | Residues, mg/kg | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | PHI, 55 days | | PHI, 42 days | | PHI, 28 days | | PHI, 21 days | | PHI, 14 days | |
| | Captan | THPI | Captan | THPI | Captan | THPI | Captan | THPI | Captan | THPI |
| Apples, field | 0.06 | <0.05 | 0.09 | <0.05 | 0.45 | <0.05 | 0.87 | <0.05 | 1.5 | <0.05 |
| Unwashed | <0.05 | <0.05 | 0.22 | <0.05 | 0.89 | <0.05 | 1.7 | <0.05 | 2.2 | <0.05 |
| Washed + rinsed | <0.05 | <0.05 | 0.12 | <0.05 | 0.55 | <0.05 | 0.81 | <0.05 | 1.5 | <0.05 |
| Peeled, cored, sliced | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | 0.09 | <0.05 | <0.05 | <0.05 |
| Apple sauce | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | 0.07 |
| Baby food apple sauce | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | 0.07 |
| Canned apple slices | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| Unclarified juice | <0.05 | <0.05 | 0.06 | 0.09 | 0.28 | 0.11 | 1.1 | 0.27 | 2.3 | 0.26 |
| Canned, unclarified juice | <0.05 | <0.05 | <0.05 | 0.13 | <0.05 | 0.36 | <0.05 | 0.98 | <0.05 | 1.6 |
| Filter cake | <0.05 | <0.05 | <0.05 | 0.09 | 0.10 | 0.27 | 0.37 | 0.70 | 0.56 | 0.97 |
| Canned clarified juice | <0.05 | <0.05 | <0.05 | 0.07 | <0.05 | 0.38 | <0.05 | 1.0 | <0.05 | 1.3 |
| Apple jelly | <0.05 | <0.05 | <0.05 | 0.05 | <0.05 | 0.15 | <0.05 | 0.42 | <0.05 | 0.58 |
| Wet pomace | <0.05 | <0.05 | 0.06 | 0.10 | 0.21 | 0.27 | 0.77 | 0.60 | 0.72 | 0.85 |
| Dry pomace | <0.05 | <0.05 | 0.12 | 0.34 | 0.41 | 1.0 | 1.4 | 2.1 | 2.0 | 2.6 |

Cherries. In three trials in Germany cherries were treated 3 times with captan at 0.12 kg ai/hl and harvested 7 days after the last application (Pollmann, 2000). Residues were measured in washed fruit and in fruit prepared for canning, simulated by adding 10 g sugar/100 g fruit and 200 ml water, then heating to 94-95°C at 3°C/min, and holding at that temperature for 5 min before cooling to room temperature and freezing.

Table 49. Captan residues in cherries processed in Germany in 1999 (Pollmann 2000).

| Variety | Rate, kg ai/hl | PHI, days | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg |
|---|---|---|---|---|---|---|
| Edelfinger | 3×0.12 | 7 | Fruit | 1.1 | - | - |
| | | 7 | Washed | 0.38 | 0.3 | - |
| | | 14 | Canned | <0.002 | <0.001 | 0.03 |
| Schatten-morelle | 3×0.12 | 7 | Fruit | 2.9 | - | |
| | | 7 | Washed | 0.89 | 0.3 | - |
| | | 7 | Canned | <0.002 | <0.001 | 0.10 |
| Johanna | 3×0.12 | 7 | Fruit | 3.7 | - | - |
| | | 7 | Washed | 1.8 | 0.5 | |
| | | 7 | Canned | <0.002 | <0.001 | 0.05 |

Northover *et al.* (1986) studied the effect of temperature on the residues of captan in stored whole cherries and the effect of washing on residues of captan in cherries and peaches. The residues of captan in whole sweet cherries (Bing variety) did not decrease on storage at 4 and 20°C for 14 days. Tumble washing sweet cherries for as little as 15 sec reduced captan residues by 70-74%. (1 kg lots were dropped into 3 l distilled water within a 34 l container and tumbled manually every 2.5 sec to resemble domestic washing for 15, 30, 60 or 120 sec. Fruit were then drained and dried on absorbent paper (without rubbing) for 1-2 h at 20°C). The addition of the surfactant Tween 20 or $NaHCO_3$ to the wash solution did not remove any more residue than tap or distilled water alone. Residues of captan in peaches were reduced by 56% when hand-washed and by 70% when washed with brushing.

Table 50. Captan residues in cherries and peaches before and after washing (Northover *et al.*, 1986).

| Country, year (variety) | Sample | Residue, mg/kg | Processing factor |
|---|---|---|---|
| Canada, 1982, Sweet cherry (Bing) | Fruit | 2.7 | - |
| | Washed distilled water 15 s | 0.8 | 0.3 |
| | Washed distilled water 30 s | 0.6 | 0.2 |
| | Washed distilled water 60 s | 0.3 | 0.1 |
| | Washed distilled water 120 s | 0.2 | 0.07 |
| Canada, 1983, Sweet cherry (Bing) | Fruit | 6.8 | - |
| | Washed distilled water 15 s | 1.8 | 0.3 |
| | Washed distilled water 30 s | 1.9 | 0.3 |
| | Washed distilled water 60 s | 0.3 | 0.04 |
| | Washed distilled water 120 s | 0.2 | 0.03 |
| | 0.1% Tween 20, 120 s, rinse | 0.2 | 0.03 |
| | 4.2 g $NaHCO_3$/l, 120 s, no rinse | 0.2 | 0.03 |
| | 4.2 g $NaHCO_3$/l, 120 s, rinse | 0.3 | 0.04 |
| | Tap water, 120 s, rinse | 0.6 | 0.09 |
| Canada, 1983, Peach (Red Haven) | Fruit | 11 | - |
| | Hand wash 5 s | 4.9 | 0.4 |
| | Hand wash 10 s | 4.6 | 0.4 |
| | Brush wash 10 s | 3.3 | 0.3 |

Prunes. Plums in the USA were sprayed 9 times with captan at 3.4 kg ai/ha with harvest on the day of the final application. Samples were washed and graded and dried in a bin dryer at 74°C for 16 hours to produce prunes (Smith, 1987q).

Table 51. Captan residues in Stanley plums and dried prunes in Michigan, USA, 1986 (Smith, 1986q).

| Rate, kg ai/ha | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|
| 9×3.4 | Fruit | 3.5, 5.6 | - | <0.05, <0.05 | - |
| | Dry prunes | 0.59 | 0.1 | 5.2 | 2.3 |

Grapes. In three trials in Germany in 1991 red and white grapes were treated with eight sprays of captan (SG and WP formulations) at 2.3 kg ai/ha and fruit harvested 26-29 days after the final application. Grapes were processed into must and wine and residues measured in must, young wine and mature wine (Jones, 1992g). The grapes were mashed, destemmed and 50 mg $SO_2$/l added. The mash was heated to 60°C, cooled, drained and pressed. The turbid must was separated, appropriate quantities of sugar and yeast added and the solution fermented in bottles for 12-15 days. Samples of young wine were collected and $SO_2$ and bentonite added. Samples of mature wine were collected 6 months later.

Table 52. Captan residues in grapes before and after processing in Germany in 1991 (Jones, 1992g).

| Variety | Form | Rate, kg ai/ha | PHI, days | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|---|---|---|
| Kerner | WP | 8×2.3 | 26 | Grapes | 0.59 | - | <0.05 | - |
| | | | 27 | Must | 0.08 | 0.1 | 0.44 | 1.3 |
| | | | 53 | Young wine | <0.05 | <0.08 | 0.43 | 1.2 |
| | | | 192 | Mature wine | <0.05 | <0.08 | 0.81 | 2.3 |
| Kerner | SG | 8×2.3 | 26 | Grapes | 0.54 | - | <0.05 | - |
| | | | 27 | Must | 0.08 | 0.1 | 0.36 | 1.1 |
| | | | 53 | Young wine | <0.05 | <0.09 | 0.44 | 1.4 |
| | | | 192 | Mature wine | <0.05 | <0.09 | 0.71 | 2.2 |
| Dornfelder | WP | 8×2.3 | 29 | Grapes | 2.2 | - | 0.05 | - |
| | | | 31 | Must | <0.05 | <0.02 | 1.4 | 1.2 |
| | | | 50 | Young wine | <0.05 | <0.02 | 1.3 | 1.1 |
| | | | 189 | Mature wine | <0.05 | <0.02 | 1.8 | 1.6 |
| Dornfelder | SG | 8×2.3 | 29 | Grapes | 2.1 | - | 0.05 | - |
| | | | 31 | Must | <0.05 | <0.02 | 1.3 | 1.2 |
| | | | 50 | Young wine | <0.05 | <0.02 | 1.5 | 1.4 |
| | | | 189 | Mature wine | <0.05 | <0.02 | 1.9 | 1.7 |

The fate of captan residues during the processing of grapes from trials in Germany in 1994 was reported by Specht (1995c). No details were provided of either the field or processing parts of the trial.

Table 53. Captan residues in grapes before and after processing in Germany, 1994 (Specht, 1995).

| Grapes | PHI, days | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|---|
| White grapes | 26 | Fruit | 5.6 | - | 0.71 | - |
| | | Must | 0.57 | 0.1 | 2.0 | 0.6 |
| | | Must unpasteurized | 0.26 | 0.05 | 1.9 | 0.5 |
| | | Young wine (unpasteurized must) | 0.03 | 0.005 | 0.75 | 0.2 |
| | | Wine (after storage) | <0.01 | <0.002 | 1.1 | 0.3 |
| Red grapes | 28 | Grapes | 8.4<br>c0.59 | - | 0.59<br>c0.06 | |
| | | Must unpasteurized | 0.47<br>c0.08 | 0.06 | 1.6<br>c0.49 | 0.3 |
| | | Must pasteurized | <0.01<br>c<0.01 | <0.001 | 5.0<br>c0.71 | 1.0 |
| | | Young wine (must after mash heating) | <0.01<br>c<0.01 | <0.001 | 3.3<br>c0.58 | 0.7 |
| | | Young wine (grapes after mash fermentation) | <0.01<br>c<0.01 | <0.001 | 2.7<br>c0.56 | 0.6 |
| | | Wine (must after mash heating) after storage | <0.01 | <0.001 | 3.5 | 0.7 |
| | | Wine (grapes after mash fermentation) after storage | <0.01 | <0.001 | 3.4 | 0.7 |

Grenache grapes in Australia 1994 were treated with 8 foliar sprays of captan at 0.1 or 0.2 kg ai/hl and harvested 7, 14 or 21 days after the last spray. The grapes were processed into juice and wine (Brown *et al.*, 1995). No details were provided.

Table 54. Captan residues in grapes before and after processing in Australia in 1994 (Brown *et al.*, 1995).

| Rate, kg ai/hl | PHI, days | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|---|
| 8×0.10 | 7 | Fruit | 0.69 | - | 0.05 | - |
| | 7 | Juice | 0.46 | 0.7 | 1.0 | 1.3 |
| | 7 | Wine | <0.05 | <0.07 | 0.69 | 0.9 |
| | 14 | Fruit | 1.4 | - | 0.05 | - |
| | 14 | Juice | 0.22 | 0.2 | 0.96 | 0.6 |
| | 14 | Wine | <0.05 | <0.04 | 0.46 | 0.3 |
| | 21 | Fruit | 3.4 | - | 0.1 | - |
| | 21 | Juice | 0.27 | 0.08 | 1.3 | 0.2 |
| | 21 | Wine | <0.05 | <0.01 | 0.38 | 0.1 |
| 8×0.21 | 7 | Fruit | 7.5 | - | 0.16 | - |
| | 7 | Juice | 0.14 | 0.02 | 1.6 | 0.2 |
| | 7 | Wine | <0.05 | <0.007 | 0.95 | 0.1 |
| | 14 | Fruit | 4.7 | - | 0.11 | - |
| | 14 | Juice | 2.5 | 0.5 | 2.2 | 0.4 |
| | 14 | Wine | <0.05 | <0.01 | 1.4 | 0.3 |
| | 21 | Fruit | 5.0 | - | 0.08 | - |
| | 21 | Juice | 1.6 | 0.3 | 1.5 | 0.3 |
| | 21 | Wine | <0.05 | <0.01 | 1.4 | 0.3 |

Iwata (1992b) studied the effect of processing on Concord grapes treated in Pennsylvania, USA, with 6 foliar applications of a WP formulation at 2.2 kg ai/ha. Grapes were harvested 36 days after the final application and processed by simulated commercial procedures (Figure 7) into juice, jelly and pomace.

Table 55. Captan residues in Concord grapes before and after processing in the USA (Iwata, 1992b).

| Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|
| Fruit (field) | 0.4 | - | <0.05 | - |
| Fruit (Processor) | 0.39 | - | 0.08 | - |
| Table grapes (home washed) | 0.30 | 0.8 | 0.07 | 0.3 |
| Destemmed crushed grapes | 0.06 | 0.2 | 0.18 | 0.7 |
| Depectinised mash | <0.05 | <0.1 | 0.17 | 0.6 |
| 185-degree juice | <0.05 | <0.1 | 0.21 | 0.8 |
| Clear juice | <0.05 | <0.1 | 0.14 | 0.5 |
| Thick juice | <0.05 | <0.1 | 0.19 | 0.7 |
| Filtered juice | <0.05 | <0.1 | 0.16 | 0.6 |
| Filter cake | <0.05 | <0.1 | 0.18 | 0.7 |
| Grape jelly | <0.05 | <0.1 | 0.08 | 0.3 |
| Canned juice | <0.05 | <0.1 | 0.20 | 0.7 |
| Wet pomace | <0.05 | <0.1 | 0.07 | 0.3 |
| Dry pomace | <0.05<br>c<0.05 | <0.1 | 0.27<br>c0.14 | 1.0 |

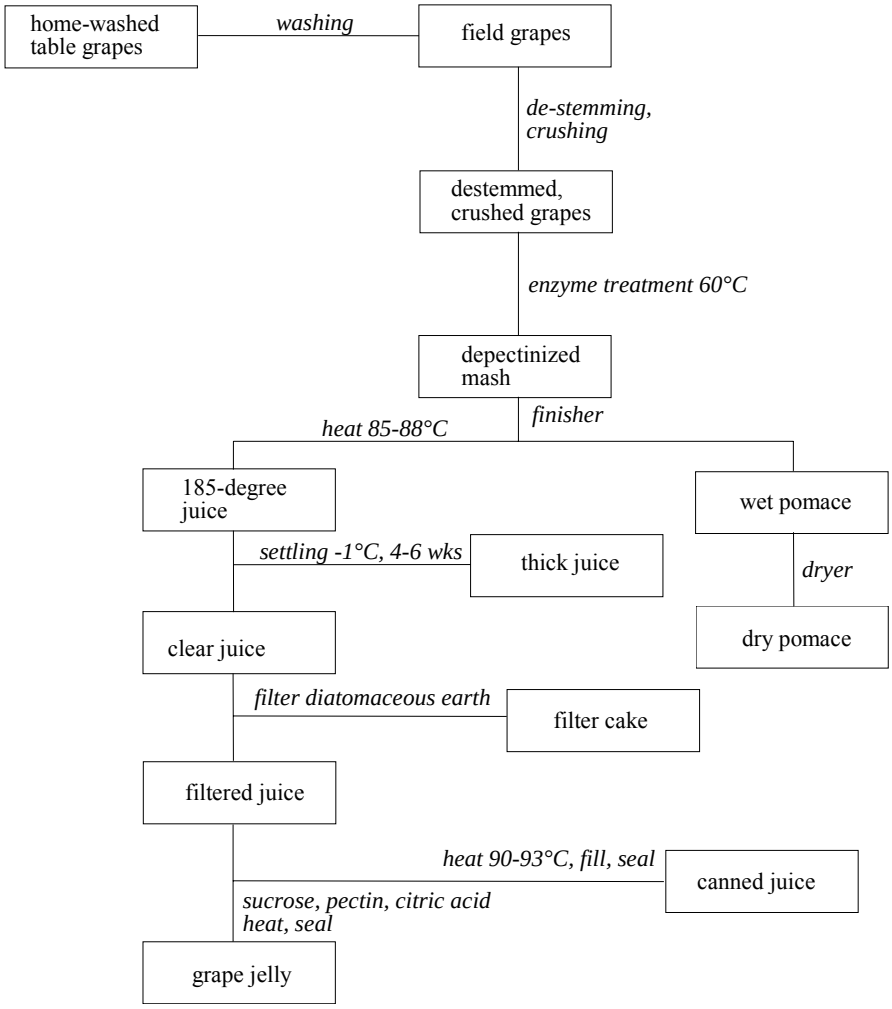



Figure 7. Grape processing (Iwata, 1992b).

Riggle (1991) studied the fate of captan residues in grapes during processing. Three field trials were conducted in the USA states California and New York. For the production of juice and pomace, grapes were mashed, the stems removed and the mashed grapes pressed to produce juice and wet pomace. The wet pomace was dried in a forced air oven at 77-88°C to produce dry pomace (less than 10% moisture content). Raisins were produced by destemming grapes and drying the whole grapes in a forced air oven at 49-63°C to achieve a moisture content of 15-18%. The process is shown in Figure 8.

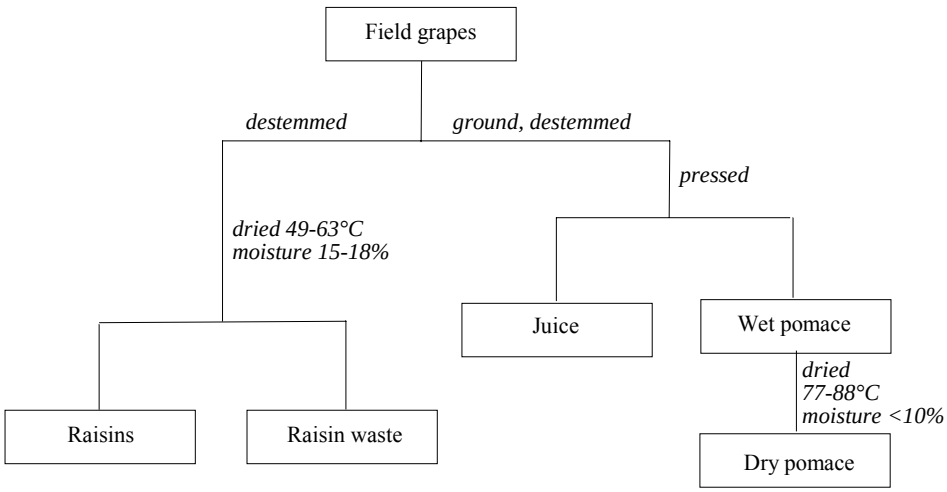


Figure 8. Grape processing (Riggle, 1991).

Table 56. Captan residues in grapes before and after processing in the USA, 1989 (Riggle, 1991).

| Location (variety) | Rate, kg ai/ha | PHI, days | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg |
|---|---|---|---|---|---|---|
| CA (Thompson Seedless) | 2×2.2 | 119 | Grapes | 0.08, 0.07 | - | <0.05, <0.05 |
| | | | Juice | <0.05 | <0.6 | <0.05 |
| | | | Wet pomace | 0.09 | 1.1 | <0.05 |
| | | | Dry pomace | 0.11 | 1.4 | <0.05 |
| | | | Raisins | 0.08 | 1 | <0.05 |
| | | | Raisin waste | 0.62 | 7.8 | <0.05 |
| CA (Thompson Seedless) | 3×2.2 | 91 | Grapes | 0.64, 0.38 | - | <0.05, <0.05 |
| | | | Juice | 0.51 | 1 | 0.12 |
| | | | Wet pomace | 0.46 | 0.9 | 0.06 |
| | | | Dry pomace | 0.22 | 0.4 | 0.12 |
| | | | Raisins | 0.61 | 1.2 | 0.06 |
| | | | Raisin waste | 13 | 26 | 0.20 |
| CA (Thompson Seedless) | 4×2.2 | 77 | Grapes | 0.17, <0.05 | - | <0.05, <0.05 |
| | | | Juice | 0.10 | 0.9 | 0.07 |
| | | | Wet pomace | 0.17 | 1.5 | <0.05 |
| | | | Dry pomace | 0.09 | 0.8 | 0.09 |
| | | | Raisins | 0.29 | 2.6 | <0.05 |
| | | | Raisin waste | 5.8 | 52 | 0.10 |
| CA (Thompson Seedless) | 2×2.2 | 118 | Grapes | <0.05, <0.05 | | <0.05, <0.05 |
| | | | Juice | <0.05 | | <0.05 |
| | | | Wet pomace | <0.05 | | <0.05 |
| | | | Dry pomace | <0.05 | | <0.05 |
| | | | Raisins | <0.05<br>c0.08 | | <0.05<br>c<0.05 |
| | | | Raisin waste | 0.09 | | <0.05 |
| CA (Thompson Seedless) | 3×2.2 | 98 | Grapes | 0.07, <0.05 | - | <0.05, <0.05 |
| | | | Juice | 0.05 | 0.8 | <0.05 |
| | | | Wet pomace | 0.09 | 1.5 | <0.05 |
| | | | Dry pomace | <0.05 | <0.8 | <0.05 |
| | | | Raisins | 0.08<br>c0.08 | 1.3 | <0.05<br>c<0.05 |
| | | | Raisin waste | 0.30 | 5 | <0.05 |
| CA (Thompson Seedless) | 4×2.2 | 84 | Grapes | 0.11, 0.22 | - | <0.05, <0.05 |
| | | | Juice | <0.05 | <0.3 | 0.09 |
| | | | Wet pomace | 0.12 | 0.7 | <0.05 |
| | | | Dry pomace | 0.09 | 0.5 | <0.05 |
| | | | Raisins | 0.19<br>c0.08 | 1.2 | <0.05<br>c<0.05 |
| | | | Raisin waste | 0.52 | 3.2 | <0.05 |
| NY (Aurora) | 2×2.2 | 85 | Grapes | <0.05, <0.05 | | <0.05, <0.05 |
| | | | Juice | <0.05 | | <0.05 |
| | | | Wet pomace | <0.05 | | <0.05 |
| | | | Dry pomace | <0.05 | | 0.07 |
| NY (Aurora) | 3×2.2 | 62 | Grapes | 0.20, 0.08 | - | <0.05, <0.05 |
| | | | Juice | <0.05 | <0.4 | <0.05 |
| | | | Wet pomace | 0.19 | 1.4 | <0.05 |
| | | | Dry pomace | 0.09 | 0.6 | 0.09 |
| NY (Aurora) | 4×2.2 | 50 | Grapes | 0.32, 0.29 | - | <0.05, <0.05 |
| | | | Juice | 0.07 | 0.2 | <0.05 |
| | | | Wet pomace | 0.31 | 1 | 0.09 |
| | | | Dry pomace | 0.29 | 1 | 0.39 |

Strawberries. Strawberries were treated with twelve foliar sprays of captan WP at 3.4 kg ai/ha and harvested on the day of the final spray. Processing was by washing and cooking the berries (TMN-684A).

Table 57. Captan residues in strawberries before and after processing in the USA, 1985 (Tomen, 1985).

| Location (variety) | Rate, kg ai/ha | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|---|
| CA | 12×3.4 | Berries | 7.2, 10 | - | 0.29, 0.34 | - |
| | | Washed | 1.2 | 0.1 | 0.42 | 0.1 |
| | | Washed + cooked | <0.01 | <0.01 | 0.54 | 0.1 |
| OR (Hood) | 12×3.4 | Berries | 8.7, 8.6 | - | 0.23, 0.26 | - |
| | | Washed | 1.1 | 0.1 | 0.04 | 0.01 |
| | | Washed + cooked | <0.01 | <0.01 | 0.55 | 0.1 |
| CA | 12×3.4 | Berries | 12, 6.7 | - | 0.90, 0.64 | - |
| | | Washed | 0.27 | 0.03 | 0.37 | 0.07 |
| | | Washed + cooked | <0.01 | <0.01 | 0.45 | 0.08 |

Tomatoes. Captan was applied as 4 foliar sprays to Peto 19 tomatoes at 4.2 or 13 kg ai/ha. Fruit were collected on the same day as the last spray and delivered overnight to the processing facility. The tomatoes were washed, sorted and trimmed. Some tomatoes were crushed and frozen as wet pomace, some crushed and dried as dry pomace. The remaining tomatoes were peeled and then subjected to hot breaking and pulping. Some of the sample was processed into purée, paste, ketchup and juice (Figure 9).

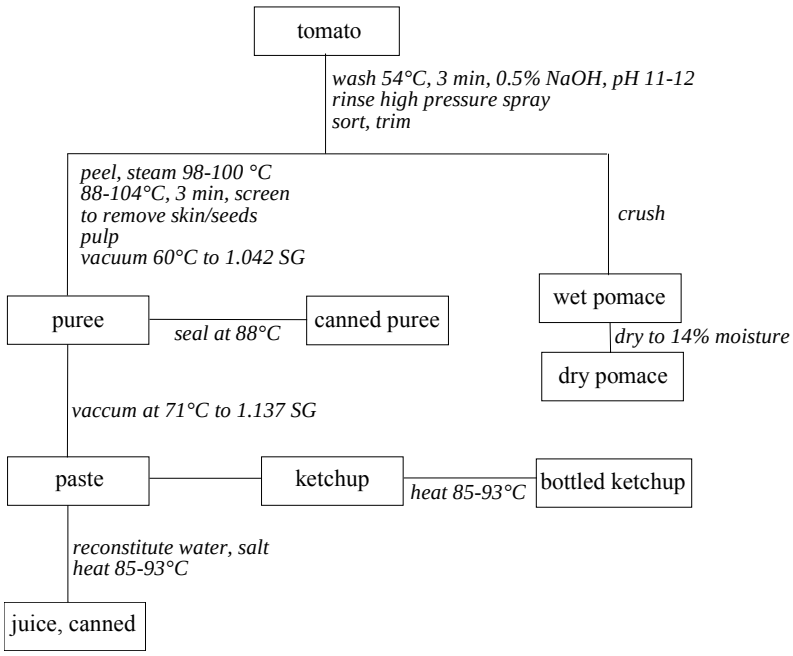



Figure 9. Tomato processing (Smith, 1987m).

Table 58. Captan residues in Peto 19 tomatoes before and after processing in California, USA, 1986 (Smith, 1987m).

| Rate, kg ai/ha | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|
| 4×4.2 | Fruit | 0.55, 0.48 | - | 0.06, 0.13 | - |
| | Wet pomace | 0.10 | 0.2 | 0.09 | 0.3 |
| | Dry pomace | 0.06 | 0.1 | 2.1 | 5.9 |
| | Purée | <0.05 | <0.1 | 0.23 | 0.6 |

| Rate, kg ai/ha | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Processing yield |
|---|---|---|---|---|---|
| | Juice | <0.05 | <0.1 | 0.09 | 0.3 |
| | Ketchup | <0.05 | <0.1 | 0.46 | 1.3 |
| 4×13 | Fruit | 0.87, 1.8 | | 0.12, 0.15 | - |
| | Wet pomace | <0.05 | <0.04 | 0.14 | 0.1 |
| | Dry pomace | 0.11 | 0.08 | 2.3 | 1.6 |
| | Purée | <0.05 | <0.04 | 0.38 | 0.3 |
| | Juice | <0.05 | <0.04 | 0.12 | 0.08 |
| | Ketchup | <0.05 | <0.04 | 0.73 | 0.5 |

Cucurbits (Cucumber, melon, squash). Cucurbits were treated with 6-9 sprays of a captan WP formulation at 2.2 kg ai/ha and harvested on the day of the final application. Fruit were processed by washing, peeling and cooking.

Table 59. Captan residues in cucurbits before and after processing in the USA, 1985.

| Location (commodity) | Rate, kg ai/ha | Sample | Captan, mg/kg | Processing factor | THPI, mg/kg | Ref. |
|---|---|---|---|---|---|---|
| CA (cucumber) | 6×2.2 | Fruit | 0.03, 0.10 | - | <0.01, 0.02 | TMN-609A |
| | | Washed fruit | 0.01 | 0.2 | 0.01 | |
| | | Washed peel | 0.02 | 0.3 | 0.05 | |
| | | Peeled fruit | <0.01 | <0.2 | <0.01 | |
| | | Washed, sliced, cooked | <0.01 | <0.2 | <0.01 | |
| NY (cucumber, Marketmore 76) | 6×2.2 | Fruit | 1.2, 0.91 | - | 0.13, 0.11 | TMN-609A |
| | | Washed fruit | 0.05 | 0.05 | 0.06 | |
| | | Washed peel | 0.15 | 0.1 | 0.57 | |
| | | Peeled fruit | <0.01 | <0.01 | 0.05 | |
| | | Washed, sliced, cooked fruit | <0.01 | <0.01 | 0.05 | |
| CA (cantaloupe, 45SJ) | 6×2.2 | Fruit | 0.74, 1.1 | - | 0.03, 0.05 | TMN-634A |
| | | Peel | 1.0 | 1.1 | 0.13 | |
| | | Peeled fruit | <0.01 | <0.01 | <0.01 | |
| FL (cantaloupe, Gold Star) | 9×2.2 | Fruit | 0.35, 0.36 | - | 0.07, 0.09 | TMN-634A |
| | | Peel | 0.16 | 0.4 | 0.30 | |
| | | Peeled fruit | 0.01 | 0.03 | 0.02 | |
| CA (squash) | 6×2.2 | Fruit | 0.12, 0.14 | - | 0.11, 0.07 | TMN-634A |
| | | Washed | 0.01 | 0.08 | 0.03 | |
| | | Washed, peeled fruit | <0.01 | <0.08 | 0.01 | |
| | | Washed peel | 0.05 | 0.4 | 0.07 | |
| | | Sliced cooked fruit | <0.01 | <0.08 | 0.02 | |
| NY (squash, Ambassador) | 6×2.2 | Fruit | 1.1, 1.4 | - | 0.11, 0.14 | TMN-634A |
| | | Washed fruit | 0.05 | 0.04 | 0.09 | |
| | | Washed, peeled fruit | <0.01 | <0.01 | 0.02 | |
| | | Washed peel | 0.12 | 0.1 | 0.20 | |
| | | Washed peeled, cooked | <0.01 | <0.01 | 0.04 | |

**Residues in the edible portions of food commodities**

No additional information. The distribution and nature of captan residues greatly influence the residues in edible portions of food commodities. In general, captan is a surface residue. Processing to products that do not include the skin generally provides significant reductions in residues. Heating, as

part of normal processing or cooking, increases the rate of hydrolysis of captan, converting most of the residue to THPI.

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The Meeting was provided with the results of a 1991 USA nationwide survey in which whole milk samples were collected from randomly selected retail stores. Residues of captan and the metabolites THPI, 3-OH THPI and 5-OH THPI were determined by a validated method with limits of quantification of 0.005 mg/kg. The residues of captan and metabolites were below the limit of quantification in all of the 224 samples analysed. Captan was determined in foods prepared for consumption in the 1997, 1998 and 1999 USA Food and Drug Administration Pesticide Program Residue Monitoring Total Diet Studies. The number of samples analysed was 1036 in 1997, 1036 in 1998 and 1040 in 1999. Although residues of captan were detected in each of the surveys the incidence of detections was less than 2% and did not trigger specific mention in the reports. Gilvydis *et al.* (1986) reported a survey of captan residues in strawberries and grapes grown in Michigan and Indiana, USA, in the 1984 growing season. Captan was applied by overhead irrigation, tractor sprayer or aerial application at rates ranging from 0.6 to 6.7 kg ai/ha. Residues of captan were found in all 28 strawberry samples analysed with residue levels ranging from less than 0.01 to 1.5 mg/kg at 2 to 38 days after the last spray. Only six of 15 grape samples (Concord variety) treated with captan contained residues. The grape samples were harvested >54 days after the last spray with residues in the range less than 0.01 to 0.08 mg/kg. The government of Poland provided summary information on the residues of captan in apples, cherries and plums

Table 60. Residues of captan in apples, cherries and plums in Poland.

| Year | No. of Samples | No. of Detections | No. of samples in residue range, mg/kg | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | <0.01[1] | >0.01-≤0.02 | >0.02-≤0.05 | >0.05-≤0.1 | >0.1-≤0.5 | >0.2-≤0.5 | >0.5-≤1 | >1-≤2 |
| Apples | | | | | | | | | | |
| 1997 | 294 | 35 | 259 | 2 | 6 | 6 | 11 | 7 | 3 | |
| 1998 | 290 | 20 | 170 | | 1 | 1 | 2 | 3 | 9 | 4 |
| Cherries | | | | | | | | | | |
| 1997 | 81 | 2 | 79 | | | 2 | | | | |
| 1998 | 114 | 14 | 100 | | 2 | 2 | 3 | 5 | | 1 |
| Plums | | | | | | | | | | |
| 1997 | 53 | 0 | | | | | | | | |
| 1998 | 61 | 0 | | | | | | | | |

[1] LOQ

## NATIONAL MAXIMUM RESIDUE LIMITS

The residue is defined as captan *per se* in Australia, the countries of the EU, and the USA, the only countries for which information was provided. The Meeting was informed that the following national MRLs had been established.

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| Argentina | Potato | 10 |
| | Tomato, pimento, aubergine, water melon, pumpkin, melon | 15 |
| Austria | Pome fruit, berries and small fruit, grapes and tomatoes | 3 |
| | Beans, broad-leaved endives, endives, leeks, stone fruit, lettuce and peas | 2 |
| | Other fruit and vegetable | 0.1 |
| Australia | Pome fruit, grape, strawberry, apple pomace, dry | 10 |
| | Stone fruit | 15 |
| | Edible offal (mammalian) | 0.05 |
| | Meat (mammalian) | 0.05 |
| | Milks | 0.01 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Treatment of seed rice and vegetable seeds, fungicidal seed dressing | No MRL required |
| Belgium | Apples/pears, small fruit, tomatoes | 3 |
| | Berries | 10 |
| | Stone fruit (cherries/prunes), beans, endive, leeks, lettuce | 2 |
| | others | 0.1[1] |
| Brazil | Garlic, apple, pear | 25 |
| | Citrus, peach, tomato | 15 |
| | Onion, cucumber | 10 |
| | Melon, watermelon, grapes | 2 |
| | Potato | 1 |
| Canada | Apples, apricots, blueberries, cranberries, cherries, grapes, peaches, pears, plums, raspberries, strawberries, tomatoes and potatoes. | 5 |
| EU (Directive 76/895/EEC) | Pome fruit, berries and small fruit, grapes and tomatoes | 3 |
| | Beans, broad-leaved endives, endives, leeks, stone fruit, lettuce and peas. | 2 |
| | Other fruits and vegetables | 0.1 |
| France | Pome fruits (apples, pears) berries, grapes, small fruits, tomatoes, stone fruits | 3 |
| | Chicory - salads, beans, leek, peas, endive | 2 |
| | Other fruits and vegetables | 0.1 |
| Germany | Pome fruit, berry fruit, tomatoes, grapes | 3 |
| | Beans, chicory, endive, peas, lettuce, leeks, stone fruit | 2 |
| | Hops, others | 0.1 |
| Greece | Pome fruit, berries, small fruit, grapes, tomatoes | 3 |
| Hungary | Paprika, tomato | 5 |
| | Melon, cucumber | 2 |
| | Other vegetables | 5 |
| | Fruit | 5 |
| | Wine/grape | 2 |
| Italy | Pome fruit, berries, grapes, tomatoes | 3 |
| | Stone fruit, leaf greens, lettuce, beans, peas, leeks | 2 |
| | Other vegetables | 0.1 |
| Japan | Tomato, eggplant, cucumber, apple, pear, grape, cherry, plum, peach, strawberry, melon | 5 |
| Kenya | Apples, cherries | 40 |
| | Pears | 30 |
| | Apricots | 20 |
| | Citrus fruits, peaches, plums, rhubarb, tomatoes | 15 |
| | Strawberries, raspberries, cranberries, cucumbers | 10 |
| | Green beans, lettuce, marrow, peppers, raisins | 5 |
| Malaysia | Leafy vegetables, non-leafy vegetables | 10 |
| | Tea, coffee | 15 |
| | Onions, potatoes | 20 |
| Mexico | Grape | 50 |
| | Apple/pear, avocado, garlic, carrots, melon, cucumber, watermelon, strawberry, tomato | 25 |
| | Mango | 50 |
| | Other crops (not listed here) | 2-100 |
| Netherlands | Berries and small fruit (including currants and strawberries), top fruit (apple, pear), tomato | 3 |
| | Leaf vegetables (including lettuce and endive), stone fruit, leeks, pulse crops | 2 |
| | Other fruit and nuts, other vegetables (including spinach), cereals | 0.1 |
| Poland | Berries and other small fruits, pome fruit, tomato | 3 |
| | Leek, legume vegetables, lettuce (head), stone fruits | 2 |
| | Other products of plant origin | 0.1 |
| Spain | Pome fruits, grapes, berries, small fruits, tomato, aubergine | 3 |
| | Stone fruit, beans, pea, lettuce, leek | 2 |
| | Citrus | 0.5 |
| | All other vegetables | 0.1 |
| Sweden | Fruit and vegetables (except those listed below) | 0.1 |
| | Apples, pear, berries, grapes, tomatoes | 3 |
| | Stone fruits, salads, beans, peas | 2 |
| Switzerland | Pome, berries, small fruit, tomatoes, aubergine | 3 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Stone fruit, salads, beans, peas, leek | 2 |
| Turkey | Peaches, apples, pears, citrus fruits | 5 |
| | Raisins, tomatoes, peppers | 3 |
| | Grape | 2 |
| | Milk | 0.05 |
| | Cucumber, lettuce | 1 |
| USA | Almond hulls, beet (greens), cherries, lettuce, plums (fresh prunes), spinach | 100 |
| | Almonds, beet (roots), broccoli, Brussels sprouts, cabbage, carrots, cauliflower, collards, sweet corn (husk removed), kale, mustard greens, peas (dry), peas (succulent), rutabagas (roots), soybeans (dry), soybeans (succulent), turnip (greens), turnip (roots) | 2 |
| | Apples, beans (dry), beans (succulent), blackberries, blueberries (huckleberries), cantaloupes, cucumbers, dewberries, eggplants, honeydew melons, muskmelons, onions (dry bulb), pears, peppers, potatoes, pumpkins, raspberries, squash (summer), squash (winter), strawberries, tomatoes, watermelons | 25 |
| | Apricots, celery, grapes, mangoes, nectarines, onions (green), peaches | 50 |
| | Cattle fat, cattle meat, cattle meat-byproducts, hog fat, hog meat, hog meat-byproducts | 0.05 |

## APPRAISAL

Captan was first evaluated in 1965. It was listed by the 1995 CCPR (ALINORM 95/24 A) for periodic re-evaluation, and the 1997 CCPR scheduled it for consideration by the FAO Panel of the 1998 JMPR (ALINORM 97/24 A). As the rights on this compound were being shifted from one company to another, a request was made that re-evaluation of captan be deferred until 2000. The Meeting received information on the physicochemical properties, metabolism, environmental fate, analytical methods, stability under storage, registered uses, residues found in supervised trials, and processing.

### *Metabolism*

Captan is susceptible to cleavage of the N-S bond to produce 1,2,3,6-tetrahydrophthalimide (THPI) and derivatives of the trichloromethylthio side-chain. The Meeting received reports of studies of the distribution and metabolism of captan in animals and plants in which captan was radiolabelled at the cyclohexene ring, the indole ring, or the carbon of the trichloromethylthio side-chain.

#### *Animals and birds*

In a material balance study, [trichloromethyl-$^{14}$C]captan was administered to lactating goats by gelatine capsule (at a dose equivalent to 55 ppm) for 2 days, and the animals were slaughtered 16 h after the last dose. Most of the radiolabel was recovered in the gastrointestinal tract (20%) and as expired $CO_2$ (43%) and most of the remainder in urine (8%), faeces (4.6%), and milk (0.2%). When a lactating goat was given [trichloromethyl-$^{14}$C]captan at a dose equivalent to 50 ppm for 7 days, 36% of the radiolabel was recovered in the excreta. The concentration of total radiolabelled residues in milk reached a plateau at 2.2 mg/kg (expressed as captan) on days 4-5. The highest concentrations were observed in kidney (4.4 mg/kg) and liver (4.7 mg/kg) (as captan). The low recovery of the administered dose is probably due to bacterial conversion of $^{14}CO_2$ to methane in the rumen.

The radiolabel in tissues at sacrifice accounted for 1.3% of a dose of [trichloromethyl-$^{14}$C]captan administered to a lactating goat orally at 1.4 mg/kg bw per day for 3 days. The highest concentrations were found in liver (2.0 mg/kg) and kidney (1.6 mg/kg) (as captan). Most of the radiolabel in tissues and milk was incorporated into natural products.

When a lactating goat was given a capsule containing [carbonyl-$^{14}$C]captan at 1.4 mg/kg bw per day three times daily (equivalent to 50 ppm), the major metabolites in urine were *cis*- or *trans*-3-hydroxy-1,2,3,6-tetrahydrophthalimide (3-OH THPI), *cis*- or *trans*-5-hydroxy-1,2,5,6-

trihydrophthalimide (5-OH THPI), and 4,5-dihydroxyhexahydrophthalimide (4,5-diOH HHPI). The major metabolites in tissues and milk were 1,2,3,6-tetrahydrophthalimide (THPI), *cis*- or *trans*-3-OH THPI), and *cis*- or *trans*-5-OH THPI. The concentrations of total radioactive residues (in rank order) were 2.3 mg/kg in kidney, 1.7 mg/kg in liver, 0.66 mg/kg in muscle, and 0.36 mg/kg in fat, as captan.

More than 88% of a dose of [trichloromethyl-$^{14}$C]captan administered to a hen by capsule for 2 days at a rate equivalent to 10 ppm was recovered in excreta and as $^{14}CO_2$. Only 2.8% of the dose was recovered in the carcase.

When hens were dosed orally with [trichloromethyl-$^{14}$C]captan at a nominal rate equivalent to 10 ppm for 10 days, the concentrations of radiolabelled residues in eggs reached a plateau by day 8 of dosing. The concentrations were highest in kidney, liver, and egg yolk. Much of the radiolabelled residue was incorporated into natural products.

When a group of laying hens was dosed orally with [cyclohexene-$^{14}$C]captan at a nominal rate equivalent to 10 ppm for 10 days, the concentrations of radiolabelled residues in eggs reached a plateau 2-4 days after the start of dosing. Most of the dose was excreted. The radiolabel in tissues and eggs represented 3.2% of the administered dose. The major metabolites identified in tissues and eggs were THPI, 3-OH THPI, 5-OH THPI, THPI expoxide, *cis*- or *trans*-6-carbamoyl-3-cyclohexene-1-carboxylic acid, and 4,5-dihydroxyhexahydrophthalimide.

The studies of metabolism show that captan is rapidly degraded in goats and hens and is not detectable in tissues, milk, or eggs. The N-S bond is cleaved to form THPI and derivatives of the trichloromethylthio side-chain. THPI undergoes a variety of oxidations and hydroxylations to yield THPI epoxide, 4,5-dihydroxyhexa-hydrophthalimide, 3-OH THPI, 5-OH THPI, and *cis*- or *trans*-6-carbamoyl-3-cyclohexene-1-carboxylic acid as the major metabolites. The tetrachloromethylthio derivatives are metabolized with incorporation of the trichloromethyl carbon into natural products, including $CO_2$ and $CH_4$.

*Plants*

When lettuce and tomato plants were treated four times with [trichloromethyl-$^{14}$C]-captan or [cyclohexene-$^{14}$C]captan at 4.5 kg ai/ha at 7-day intervals, most of the radiolabel was found in the leaves and fruit of tomatoes and the leaves of lettuce 3 h after the last spray.

When tomatoes were treated with [cyclohexene-$^{14}$C]captan, unextractable residues represented less than 9% of the total radiolabel in all components except tomato pulp, in which unextractable residues represented 42% of the total radiolabel. When tomato pulp was fractionated, 71% of the radiolabel was associated with carbohydrates, 18% with amino acids, and 3% with lignins.

With both labels, most of the residue remained on the surface of the plant or fruit as unmetabolized captan. In the plants, captan was metabolized to THPI, which undergoes further transformation.

Most the radiolabel in field-grown Golden Delicious apples on trees treated with [carbonyl-$^{14}$C]captan and harvested 3 h and 20 days after treatment was located on the surface of the fruit and was present as captan. Residues of THPI and *cis*- or *trans*-6-carbamoyl-3-cyclohexene-1-carboxylic acid represented 3.3-7.6% and 0.4-2.4% of the radioactive residue, respectively. The concentrations of residues in apple peel and pulp were low, captan representing 46 and 15%, respectively, of the radiolabel. The main metabolites in peel and pulp were THPI and *cis*- or *trans*-6-carbamoyl-3-cyclohexene-1-carboxylic acid.

In apples, tomatoes, and lettuce, most residue was present on the surface of the leaves and fruit, mainly as unchanged captan. Metabolism in these plants included cleavage of the thio-indole

bond with incorporation of the carbon of the trichloromethylthio side-chain into natural products. The other major product after cleavage, THPI, is further metabolized to *cis*- or *trans*-6-carbamoyl-3-cyclohexene-1-carboxylic acid and THPI expoxide. Captan is also oxidized to captan epoxide, which may undergo hydrolysis to form THPI epoxide.

***Environmental fate***

*Confined rotational crops*

In a study of confined crop rotation, beet, lettuce, and wheat seeds were planted in soil treated with [cyclohexene-$^{14}$C]captan or [trichloromethyl-$^{14}$C]captan 34 and 88 days after treatment and grown to maturity. Little radiolabel was found in the crops at harvest. The concentrations of radiolabelled residues in immature plants were highest in lettuce and beet. The concentrations in crops planted 88 days after application of captan to the soil were lower than those in crops planted 34 days after application. No residues of captan were detected. Most of the residue consisted of THPI and a variety of more polar metabolites, the most significant being *cis*- or *trans*-6-carbamoyl-3-cyclohexene-1-carboxylic acid and 4,5-dihydroxyhexahydrophthalimide. The Meeting concluded that the concentrations of residues of captan inadvertently introduced into rotational crops would not be significant and that the carryover of captan under field conditions would be <0.01 mg/kg, a typical lower limit of quantification (LOQ).

*Degradation in soil*

The aerobic and anaerobic degradation of [trichloromethyl-$^{14}$C]captan was studied on sandy loam soils. Under aerobic conditions, most of the radiolabel was recovered as $^{14}CO_2$. The calculated degradation half-life of [trichloromethyl-$^{14}$C]captan was 1-3 days at 25°C. Under aerobic conditions in sterile soil, 75% of the radiolabel was recovered as $^{14}CO_2$ within 90 days of incubation. When non-sterile soil was used, 100% of the radiolabel was recovered as $^{14}CO_2$ within $^{14}$ days of incubation. The radiolabel recovered as $^{14}CO_2$ after aerobic incubation at 25 °C of [carbonyl-$^{14}$C]-captan on loamy sand represented 20% by 7 days and reached 94% by 244 days of incubation. No captan was detected after 7 days of anaerobic incubation of [carbonyl-$^{14}$C]captan on loamy sand. Less than 9% of the radiolabel was recovered as $^{14}CO_2$ after 9 months of incubation. The major metabolites identified were THPI, *cis*-6-cyano-3-cyclohexenecarboxylic acid, *cis*- or *trans*-6-carbamoyl-3-cyclohexene-1-carboxylic acid, and *cis*-4-cyclohexene1,2-dicarboxylic acid. The half-life for aerobic degradation of THPI at 20°C in the dark was 5-6 days in loamy sand or sandy loam and 20 days in sand. The half-lives for aerobic degradation of *cis*-4-cyclohexene1,2-dicarboxylic acid were 4-5 days in loamy sand or sandy loam and 7 days in sand.

Captan is not susceptible to photolytic degradation, as the loss after irradiation of [trichloromethyl-$^{14}$C]captan or [cyclohexene-$^{14}$C]captan on sandy loam soil was minor when compared with hydrolysis and metabolic degradation.

Studies of the dissipation of captan in loamy sand, sand, clay, loam, and silt loam soils showed that it did not migrate below the top 15 cm of soil, except in a single sample of loamy sand (strawberry plot). The half-lives for captan in the 0-7.5-cm soil horizon were 14 days in an apple orchard, 2.5 days in a strawberry plot, 24 days in a grape plot, 4 days in a cantaloupe plot, and 3-6 days in tomato plots.

Captan is not amenable to adsorption or desorption from soil or water systems owing to its rapid hydrolysis. The degradation of captan in soil-water mixtures was dependent on pH, being most rapid at pH 7, the highest pH studied. The only degradate detected was THPI. The presence of soil in the test solutions resulted in an increased rate of degradation.

In studies of leaching in three soil types, captan was not readily leached, none being found below the 0-5-cm horizon. The degradation half-lives for captan in aged soil samples were 10-35 days.

*Fate in water and sediment systems*

The half-life of captan in two non-sterile water and sediment systems was <24 h, no captan being detected after 24 h of incubation. Captan is rapidly hydrolysed to THPI. Other products identified after the incubation were *cis*- or *trans*-6-carbamoyl-3-cyclohexene-1-carboxylic acid, *cis*-4-cyclohexene-1,2-dicarboxy-lic acid, and THPI epoxide. The products were degraded, such that none could be detected after 59 days of incubation. Negligible amounts of $^{14}CO_2$ evolved in the sterile systems. Most of the radiolabel present after 90 days of incubation was found in THPI. There was no significant volatilization of captan from soil.

***Methods of analysis***

Adequate methods have been developed for the determination of residues of captan and THPI on crops and for THPI and the hydroxylated metabolites 3-OH THPI and 5-OH THPI in animal commodities. The methods typically involve maceration of the sample with a solvent, which is usually ethyl acetate or acetone. As captan is readily hydrolysed at high pH, a small quantity of phosphoric acid is often added at the extraction step in order to lower the pH. Different procedures are required for the clean-up of captan and THPI: extracts of captan are cleaned-up on a silica column, while THPI must be partitioned with basic aqueous buffer and then with dichloromethane. The final extracts are analysed on a gas chromatograph equipped with an electron capture detector for captan and a thermionic detector for THPI. Typical LOQs are 0.01 mg/kg for captan and 0.02 mg/kg for THPI. The hydroxylated metabolites 3-OH THPI and 5-OH THPI must be silylated before determination by gas chromatography. Extensive data on recovery were presented for the most common methods.

***Stability of residues in stored analytical samples***

The possibility that captan on agricultural commodities might be hydrolysed must be considered when conducting analyses. Samples for analysis should be stored whole, and the extraction step should be completed as soon as possible after maceration. The stability of captan and THPI during frozen storage of field and fortified samples of almonds, almond nuts (whole, coarsely ground), apples, apple juice, apple sauce, beet tops, cherries, corn grain, cucumbers, dry grape pomace, lettuce, maize grain (whole, coarsely ground), melons, potato tubers, raisins, soya bean forage, soya beans, spinach (leaves, coarsely chopped, finely chopped), strawberries, sugar-beet tops, tomatoes, tomato pomace, tomato sauce, and wheat forage were determined.

The concentrations of residues of captan represented more than 70% of the initial concentration for at least 15 months in apple juice and soya bean forage; 14 months in strawberries; 13 months in apples; 12 months in cherries; 10 months in raisins; 9 months in whole almond nuts, apple sauce, dry grape pomace, potatoes, tomatoes, dry tomato pomace, and tomato sauce; and 6 months in sugar-beet tops. Generally, when captan was degraded the concentration of THPI increased concomitantly. THPI was stable for at least 14 months when stored frozen in a variety of matrices.

Captan residues were more stable when stored in whole commodities than in homogenized samples. Maceration may increase exposure of captan residues to plant enzymes and water. As the main route of decomposition appears to be hydrolysis to THPI, the finding of lower concentrations of THPI residues than of captan residues is a good indication that the residue is stable in storage. With the exception of homogenized cucumbers (and presumably other cucurbits), the stability of captan in the commodities for which maximum residue levels are recommended is acceptable. The stability of captan in cucurbits might be acceptable if the samples are stored whole.

Captan is not expected to be detected in milk, eggs, or animal tissues. THPI, 3-OH THPI, and 5-OH THPI were stable in frozen fortified bovine milk and tissue samples for 3.2-3.7 years.

***Definition of the residue***

Captan is the major component of the residue in plants but may be hydrolysed to THPI during preparation of samples for analysis, frozen storage (especially of homogenized samples), and processing of the raw agricultural commodity. A separate analysis would be required if THPI were included in the residue definition, but it usually represents only a minor part of the residue and its inclusion in the residue definition for captan would make little difference. On the basis of the metabolism of captan in plants, the conclusions of the 1995 JMPR on the toxicity of residues of captan, and the available analytical methods, the Meeting concluded that the residue for compliance with MRLs and for estimation of dietary intake should continue to be captan.

***Results of supervised trials***

Captan is registered for use as a fungicide with foliar, soil, and post-harvest applications. The results of supervised trials were reported for citrus (oranges, mandarins, lemons, grapefruit), apples, pears, cherries, peaches, nectarines, plums, apricots, blueberries, strawberries, grapes, raspberries, cucumbers, melons, tomatoes, potatoes, radishes, chives, and almonds.

Trials with mandarin were presented from Japan, but the application rates were exaggerated and did not comply with GAP; furthermore, residues in pulp and peel were analysed separately. Although data were made available for lemon and grapefruit in the USA, the data were not evaluated as there was no matching GAP.

The results of trials with orange in Brazil and Spain were available. In four trials in Brazil that complied with GAP (0.11-0.12 kg ai/hl; PHI, 7 days), the concentrations of residues were 0.06, 0.10, 0.17, and 0.34 mg/kg. THPI was not detected in the two trials in which it was measured (<0.05 mg/kg). The concentrations of residues of captan in four trials conducted in Spain according to GAP (0.15-0.25 kg ai/hl; PHI, 10 days) were 0.4, 1.0, 2.1, and 2.7 mg/kg. No residues of THPI were detected (<0.05 mg/kg). The Meeting concluded that the results of the studies in Brazil and Spain could not be combined for the purposes of estimating a maximum residue level as they represented two different populations. A trial reported from the USA was not conducted according to GAP and was not considered further. Insufficient information was available to recommend a maximum residue level for oranges.

Supervised field trials on apple were reported from Argentina, Australia, Brazil, Canada, Germany, Hungary, Japan, The Netherlands, South Africa, the UK, and the USA. Trials in Chile, France, Israel, and Portugal did not correspond to GAP in those countries and were not evaluated.

The registered use pattern in Argentina is 0.12 kg ai/hl with a 14-day PHI. The concentration of residues in apples in a single trial with a spray concentration of 0.16 kg ai/hl was 0.0005 mg/kg.

In Australia, seven sprays at 0.13 kg ai/hl were used, and apples were sampled 7 days after the last spray. GAP in Australia is sufficiently close: five applications of 0.1 kg ai/hl with a 7-day PHI. The concentration of captan was 3.7 mg/kg. THPI was not measured.

In the six trials in Brazil, apples were sprayed 10-11 times at 0.12 kg ai/hl. The GAP rate is 0.11-0.12 kg ai/hl with a 1-day PHI. The concentrations of captan residues after 1 were 0.44, 0.68, 1.0, 1.4, 2.5, and 4.1 mg/kg and those of THPI were 0.11, 0.12, 0.18, 0.18, 0.38, and 0.55 mg/kg.

Eight trials on apples in Canada, in which the conditions corresponded to GAP (3 kg ai/ha; PHI, 7 days) resulted in concentrations of captan of 2.8, 2.9, 2.9, 3.2, 3.9, 4.2, 4.5, and 4.5 mg/kg and residues of THPI of <0.05, <0.05, 0.05, 0.05, 0.05, 0.06, 0.07, and 0.08 mg/kg.

The concentrations of captan in three German trials conducted according to GAP (0.1 kg ai/hl; PHI, 21 days) were 1.0, 1.1, and 3.0 mg/kg.

In a single trial in Hungary that complied with its GAP (1-1.5 kg ai/ha or 0.1-0.15 kg ai/hl; PHI, 10 days), the concentration of residues of captan was 1.5 mg/kg.

In Japan, captan is registered for use on apples at 2-8 kg ai/ha or 0.07-0.13 kg ai/hl with harvesting 14 days after the last spray. The concentrations of residues in five trials were 1.3, 2.1, 3.8, 4.6, and 7.2 mg/kg.

The GAP in The Netherlands is 0.05-0.21 kg ai/hl with a PHI of 7 days when application is at 0.06 kg ai/hl and 21 days when the application rate exceeds 0.1 kg ai/hl. The concentrations of residues of captan in six trials were 0.26, 0.55, 0.77, 0.84, and 1.0 (2 trials) mg/kg, and those of THPI residues were 0.11 (2 trials), 0.14, 0.19, 0.22, and 0.23 mg/kg.

The application rate in two trials conducted in South Africa was sufficiently close to GAP in that country (0.08-0.1 kg ai/hl; PHI, 14 days). The concentrations of residues in apples treated twice at 0.08 kg ai/hl with a 16-day PHI were 2.0 and 3.6 mg/kg, while that of THPI was 0.11 mg/kg for both trials.

GAP in the UK is 2.7 kg ai/ha with a PHI of 14 days. In 15 trials in which apples were given 3-16 applications at 2.7-2.9 kg ai/ha with a PHI of 12-14 days, the concentrations of captan were 0.5, 0.72, 0.91, 1.0, 1.2, 1.4, 2.0, 2.2, 2.4, 2.4, 2.6, 3.1, 3.7, 3.9, and 4.2 mg/kg. Those of THPI residues were <0.05 (3 trials), 0.07 (2 trials), 0.08, 0.09, 0.10, 0.11, 0.12, 0.14, 0.15, 0.2 (2 trials), and 0.36 mg/kg.

Two trials in the USA met GAP in that country, which includes both pre-harvest application (2.2-4.5 kg ai/ha; PHI, 0 day) and post-harvest application (dipping at 1.5 g ai/l; withholding interval, 0 day). After eight foliar sprays at 4.5 kg ai/ha and post-harvest dipping at 1.5 g ai/l, the concentrations of residues of captan were 5.9 and 7.7 mg/kg, and those of THPI were 0.09 and 0.35 mg/kg. Post-harvest dipping alone resulted in concentrations of similar magnitude: 2.9, 3.3, 4.0, and 7.8 mg/kg. The concentrations of THPI residues were 0.12, 0.10, 0.09, and 0.08 mg/kg in the same trials. The residues of captan after pre-harvest application alone at 4.5 kg ai/ha in nine trials were 0.86, 1.4, 1.5, 2.8, 3.9, 4.7, 4.9, 5.2, and 5.5 mg/kg, and those of THPI were <0.05, 0.07, 0.05, <0.05, <0.05, 0.10, 0.13, 0.76, and 0.21 mg/kg 0 days after the last spray.

The Meeting concluded that the results of trials of captan in apples by foliar and post-harvest applications should not be combined for the purposes of estimating a maximum residue level or STMR value, as they represent different residue populations. Rather, the results of trials of post-harvest application, the critical use pattern, should be used. The concentrations of residues of captan in apples in the six post-harvest trials in the USA, in rank order (median in italics), were 2.9, 3.3, **4.0**, **5.9**, 7.7, and 7.8 mg/kg. The Meeting decided to combine the data on apples with that on pears (see below) to estimate a maximum residue level for pome fruit. The STMR and HR values for captan in apples were estimated to be 4.95 and 7.8 mg/kg, respectively.

Supervised trials on pear conducted according to GAP were provided from Australia, Italy, Japan, the UK, and the USA. Trials in Chile, Germany, and South Africa did not correspond to GAP in those countries and were not evaluated.

The concentration of captan residues in a single trial in Australia after five applications of 0.13 kg ai/hl was 2.5 mg/kg 6 days after the last spray. GAP in Australia is five applications of 0.1 kg ai/hl with a PHI of 7 days. THPI residues were not measured.

In 12 trials in Italy, pears were given six to eight applications of 0.13 kg ai/hl with a PHI of 14 days. This rate compares well with the Italian GAP of 0.13-0.16 kg ai/hl and a PHI of 15 days. The concentrations of residues of captan 14 days after the last spray were 0.59, 0.68, 0.72, 0.81, 1.1, 1.2, 1.2, 1.3, 1.6, 1.9, 2.0, and 2.0 mg/kg. THPI residues were not determined.

The registered application rate in Japan is seven sprays at 0.08-0.13 kg ai/hl (2.4-8 kg ai/ha) with a 7-day PHI. In six trials, pears were treated with five to nine applications of 0.13 kg ai/hl. Seven days after the last spray, the concentrations of captan were 0.50, 0.77, 0.99, 2.3, and 2.6 (2 trials) mg/kg. THPI residues were not measured.

The results of five trials were provided by the UK in which treatment comprised eight to 10 sprays at 2.7 kg ai/ha and sampling 12-14 days after the last spray. GAP in that country is 12 sprays at 2.7 kg ai/ha with a 14-day PHI. The concentrations of residues of captan were 1.2, 1.7, 1.9, 2.0, and 2.6 mg/kg, and those of THPI were <0.05, <0.05, <0.05, 0.08, and 0.11 mg/kg.

In the USA, captan is registered for post-harvest dipping at 1.5 g ai/l. The concentrations of residues of captan in pears dipped at 1.5 g ai/l were 11 and 4.7 mg/kg, and those of THPI were 0.47 and 0.07 mg/kg, respectively.

The Meeting considered that the post-harvest trials in the USA represent a different population from that in the other trials and that the results should not be combined for the purposes of estimating a maximum residue level or STMR value. However, it considered that similar residues would occur after post-harvest dipping of apples and pears and that the results for pears could be combined with those for apples to estimate a maximum residue level and STMR value for pome fruit. The concentrations of residues of captan in apples and pears in the eight trials of post-harvest treatment, in rank order, were 2.9, 3.3, 4.0, **4.7**, **5.9**, 7.7, 7.8, and 11 mg/kg. The Meeting estimated a maximum residue level of 15 mg/kg, an STMR value of 5.3 mg/kg, and a HR value for captan in pome fruit of 11 mg/kg. The estimated maximum residue level replaces the current recommendations of 20 mg/kg for apples and 10 mg/kg for pears.

Supervised trials on cherry were provided from Canada, Germany, Japan, and the USA. A trial in Belgium did not correspond to GAP in that country and was not evaluated.

In five trials in Canada approximating GAP (3-3.6 kg ai/ha; PHI, 2 days for sweet cherries and 5 days for sour cherries), the concentrations of residues of captan were 5.0 and 13 mg/kg in sweet cherries and 4.9, 9.7, and 13 mg/kg in sour cherries.

Two trials in Germany were evaluated on the basis of Belgian GAP (spray concentration, 0.12 kg ai/hl; PHI, 4 days), as details of GAP in Germany were not provided. The concentrations of residues of captan were 1.9 and 4.0 mg/kg 3 days after the last spray.

When captan was applied according to GAP in Japan (five sprays at 3-6 kg ai/ha or 0.1 kg ai/hl; PHI, 14 days), the concentrations of residues of captan in two trials were 0.58 and 1.3 mg/kg. Those in 12 trials of treatment with four to five sprays at 5.6-7 kg ai/ha (0.08-0.1 kg ai/hl) were 0.66, 0.69, 0.77, 0.78, 1.2, 1.3, 1.5 (3 trials), 1.7, 2.2, and 2.3 mg/kg at a PHI of 14 days.

In the USA, captan is registered for both pre-harvest use (1.1-2.2 kg ai/ha; PHI, 0 days) and post-harvest use (1.5 g ai/l). In two trials in which cherries were treated before harvest with seven sprays at 2.2 kg ai/ha and after harvest at 1.5 g ai/l, the concentrations of residues of captan were 23 and 35 mg/kg and those of THPI were 0.34 and 0.45 mg/kg. When captan was used as a post-harvest dip only in two trials, the concentrations of captan were 14 and 15 mg/kg and those of THPI were 0.23 and 0.30 mg/kg. Pre-harvest use of captan in 12 trials of six to seven sprays at 1.7-2.2 kg ai/ha resulted in residue concentrations of 2.4, 2.8, 4.3, 5.5, 11, 12, 14 (2 trials), 19, 20 (2 trials), and 21 mg/kg. In the trials in which THPI residues were measured, the concentrations were 0.13, 0.17, 0.18, and 0.24 mg/kg.

The Meeting concluded that the residues of captan in cherries in the post-harvest trials in the USA and in the trials in Japan (indoor and outdoor) represented different populations from those in the other trials, and the results could not be combined for the purposes of estimating a maximum residue level or STMR value. The concentrations of residues of captan in cherries in the remaining 19 trials, in rank order, were 1.9, 2.4, 2.8, 4.0, 4.3, 4.9, 5.0, 5.5, 9.7, **11**, 12, 13 (2 trials), 14 (2 trials), 19, 20 (2 trials), and 21 mg/kg. The Meeting estimated a maximum residue level of 25 mg/kg, an STMR value of 11 mg/kg, and a HR value for captan in cherries (whole fruit basis) of 21 mg/kg. The estimated maximum residue level replaces the current recommendation of 40 mg/kg for cherries.

Supervised trials on plum conducted according to GAP were provided from Greece, Japan, Portugal, Spain, and the USA. Trials from Chile did not correspond to GAP in that country and were not evaluated.

The concentration of captan in a trial conducted according GAP in Greece for stone fruit (0.13 kg ai/hl; PHI, 20 days) was 0.13 mg/kg, and that of THPI was 0.07 mg/kg.

In Japan, captan is registered for use at 2.4-8 kg ai/ha (0.08-0.13 kg ai/hl) on plums, with the last application at least 14 days before harvest. The concentrations of residues of captan in plums in four trials that complied with GAP were 0.95, 1.8, and 3.0 (2 trials) mg/kg.

The concentration of captan in a trial conducted according GAP in Portugal (0.15-0.2 kg ai/hl; PHI, 7 days) was 6.7 mg/kg, and that of THPI was 0.67 mg/kg.

In two trials conducted according to GAP in Spain for stone fruit (0.13-0.15 kg ai/ha; PHI, 10 days), the concentrations of captan were 0.67 and 0.85 mg/kg, and those of THPI were 0.13 and 0.18 mg/kg.

Captan is registered in the USA for use on plums at 2.2-3.4 kg ai/ha with a PHI of 0 days. The concentrations of captan in four trials approximating GAP were 0.45, 0.60, 5.6, and 7.9 mg/kg, and the corresponding values for THPI residues were <0.05 mg/kg, although this compound was not measured in the trial in which 7.9 mg/kg of captan were found.

The concentrations of residues of captan in plums in the 12 trials, in rank order, were 0.13, 0.45, 0.60, 0.67, 0.85, **0.95**, **1.8**, 3.0 (2 trials), 5.6, 6.7, and 7.9 mg/kg. The Meeting estimated a maximum residue level of 10 mg/kg, an STMR value of 1.4 mg/kg, and a HR value for captan in plums (whole fruit basis, including prunes) of 7.9 mg/kg. The estimated maximum residue level replaces the current recommendation of 5 mg/kg for plums including prunes.

The concentrations of captan in apricot in four trials in the USA that complied with GAP (1.7-2.7 kg ai/ha; PHI, 0 day) were 3.3, 4.5, 6.0, and 6.8 mg/kg, and those of THPI were <0.05-0.21 mg/kg. There was insufficient information to estimate a maximum residue level for apricots.

Supervised trials on nectarine conducted according to GAP were provided from Greece and Spain. Trials from Chile and the USA did not correspond to the maximum GAPs in those countries and were not evaluated.

The concentrations of captan in two trials conducted according to GAP in Greece for stone fruit (0.13 kg ai/hl; PHI 20 days) were 0.90 and 1.5 mg/kg, and those of residues of THPI were 0.19 and 0.22 mg/kg.

In two trials conducted according to GAP in Spain for stone fruit (0.13-0.15 kg ai/ha; PHI, 10 days), the concentrations of captan were 1.3 and 1.8 mg/kg and those of THPI were 0.17 and 0.21 mg/kg.

An inadequate number of trials of use of captan on nectarines was available to estimate a maximum residue level, but the Meeting agreed that the results of trials in comparable countries on peaches and nectarines treated at the same rates and harvested at the same PHI could be combined for the purposes of estimating a maximum residue level and STMR value. Application of captan to peaches in seven trials in Italy at rates that corresponded to the Spanish GAP for nectarines resulted in concentrations of captan residues in peaches of 0.26, 0.81, 0.90, 1.0 (2 trials), 1.2, and 1.5 mg/kg.

The concentrations of captan in the four trials on nectarines and seven on peaches, in rank order, were 0.26, 0.81, 0.90 (2 trials), **1.0** (2 trials), 1.2, 1.3, 1.5 (2 trials), and 1.8 mg/kg. The Meeting estimated a maximum residue level of 3 mg/kg, an STMR value of 1 mg/kg, and a HR value in nectarines (whole fruit) of 1.8 mg/kg. The estimated maximum residue level replaces the current recommendation of 5 mg/kg for nectarines.

Supervised trials on *peach* conducted according to GAP were provided from Australia, Canada, Italy, and the USA. Trials from Chile, Japan, and Spain did not correspond to GAP in those countries and were not evaluated.

The concentration of captan in a trial in Australia was 4.7 mg/kg 6 days after treatment with five sprays of 0.13 kg ai/hl. GAP for stone fruit is five applications at 0.1 kg ai/hl with a 7-day PHI.

In Canada, GAP permits application of captan to peaches as two sprays of 3.4 kg ai/ha with a 2-day PHI. In five trials, the concentrations of captan were 3.2, 5.5, 6.6, 7.3, and 16 mg/kg 1 or more days after the last spray. Residues of THPI were not measured.

As described above, seven trials in Italy were evaluated on the basis of the Spanish GAP for stone fruit (0.13-0.15 kg ai/hl; PHI, 10 days), as Italian GAP was not provided The concentrations of captan were 0.26, 0.81, 0.90, 1.0 (2 trials), 1.2, and 1.5 mg/kg. THPI residues were not measured.

In the USA, GAP for peaches is 2.2-4.5 kg ai/ha with a PHI of 0 days. The concentrations of captan in 10 trials were 2.0, 4.3 (2 trials), 5.8, 6.0, 7.4, 7.8, 10, 12, and 14 mg/kg, and those of THPI were <0.05 (4 trials), 0.07, 0.08, 0.15, 0.18, 0.29, and 0.33 mg/kg.

The concentrations of captan in the 23 trials in peaches, in rank order, were 0.26, 0.81, 0.90, 1.0, 1.0, 1.2, 1.5, 2.0, 3.2, 4.3, 4.3, ***4.7***, 5.5, 5.8, 6.0, 6.6, 7.3, 7.4, 7.8, 10, 12, 14, and 16 mg/kg. The Meeting estimated a maximum residue level of 20 mg/kg, an STMR value of 4.7 mg/kg, and a HR value for captan in peaches (whole fruit basis) of 16 mg/kg. The estimated maximum residue level replaces the current recommendation of 15 mg/kg for peaches.

In the USA, captan is registered for use on *blueberry* at a rate of 1.1-2.7 kg ai/ha with a 0-day PHI. The concentrations of captan in 16 trials that complied with GAP were 2.0-18 mg/kg, and those of THPI were <0.05-0.17 mg/kg.

The concentrations of captan in the 16 trials in blueberries, in rank order, were 2.0, 3.2, 3.9, 4.0, 4.2, 4.8, 5.4, 6.5, ***6.9*** (2 trials), 7.1, 8.2, 8.3, 8.4, 15, and 18 mg/kg. The Meeting estimated a maximum residue level of 20 mg/kg, an STMR value of 6.9 mg/kg, and a HR value for captan in blueberries of 18 mg/kg. The estimated maximum residue level confirms the current recommendation of 20 mg/kg for blueberries.

Data were available from supervised trials on *grape* conducted according to GAP in Australia, Brazil, Germany, Japan, and the USA. Trials from Chile and France did not correspond to GAP and were not evaluated.

GAP in Australia allows a maximum of five applications at 0.1 kg ai/hl with harvesting 7 days after the final spray. In two trials, the concentrations of captan were 3.6 and 3.4 mg/kg, and those of THPI were 0.09 and 0.10 mg/kg.

The concentrations of captan in grapes in two trials conducted according to GAP in Brazil (0.11-0.12 kg ai/hl; PHI, 1 day) were 0.78 and 2.5 mg/kg.

Four trials in Germany were evaluated with the GAP for Belgium (0.12 kg ai/hl; PHI, 42 days), as GAP in Germany was not provided. The concentrations of captan were 0.79, 3.3, 4.7, and 6.3 mg/kg 35 days after the last of 10 sprays at 0.09 kg ai/hl.

In Japan, captan is registered for use on grapes at a maximum of five sprays at 0.1 kg ai/hl (2-3 kg ai/ha) with a PHI of 30 days. The concentrations of captan in nine indoor trials were 0.64, 0.79, 1.1, 1.8, 1.9, 2.1, 2.2, 6.3, and 7.7 mg/kg. When captan was applied to grapes in four outdoor trials, the concentrations were 0.7, 2.9, 7.1, and 9.7 mg/kg.

In nine trials conducted according to GAP in the USA (1.1-2.2 kg ai/ha; PHI, 0 day), the concentrations of captan in grapes were 1.3, 3.5, 3.7, 6.4, 7.2, 7.4, 8.4, 11, and 22 mg/kg, and those of THPI were <0.05 (3 trials), 0.07, 0.11, 0.14 (2 trials), 0.22, and 0.28 mg/kg.

The concentrations of captan in grapes in 23 trials, in rank order, were 0.65, 0.78, 0.79, 1.3, 2.4, 2.5, 2.9, 3.3, 3.4, 3.5, 3.6, ***3.7***, 4.7, 6.3 (2 trials), 6.4, 7.1, 7.2, 7.4, 7.7, 8.4, 11, and 22 mg/kg. The Meeting estimated a maximum residue level of 25 mg/kg, an STMR value of 3.7 mg/kg, and a HR value for captan in grapes of 22 mg/kg. The estimated maximum residue level confirms the current recommendation of 25 mg/kg for grapes.

Supervised trials on *raspberry* carried out in the USA were evaluated according to Canadian GAP (2 kg ai/ha; PHI, 2 days). The Meeting considered that the decrease in residues of captan was slow and that the concentrations in raspberries 0 and 3 days after the last spray could be used to estimate a maximum residue level and an STMR value. The concentrations of captan in five trials in the USA in which raspberries were treated at 1.8-2.3 kg ai/ha and harvested 0-3 days after the last spray were, in rank order, 5.7, 7.7, ***8.3***, 13, and 18 mg/kg. The Meeting estimated a maximum residue level of 20 mg/kg, an STMR value of 8.3 mg/kg, and a HR value for captan in raspberries of 18 mg/kg.

Data were available from supervised trials on *strawberry* conducted according to GAP in Belgium, Hungary, The Netherlands, Spain, and the USA.

In Belgium and The Netherlands, GAP permits application of captan at 0.12 kg ai/hl with a 4-day PHI for field-grown strawberries and a 14-day PHI for strawberries grown in a glasshouse. The concentrations of captan were 2.4 mg/kg in field-grown strawberries after 4 days in Belgium and 0.18, 0.13, 0.25, and 0.07 mg/kg in glasshouse-grown strawberries after 14 days in Belgium and The Netherlands. Trials conducted in Germany were evaluated with the GAP for Belgium, as thar for Germany was not reported. The concentrations in two field trials were 1 and 2 mg/kg 3 days after a single spray at 0.13 kg ai/hl.

GAP in Hungary permits application of three sprays of captan at 1-1.5 kg ai/ha (0.1-0.15 kg ai/hl) with a 10-day PHI. The concentration in a single trial was 0.93 mg/kg, while that of THPI was <0.1 mg/kg.

The concentration of captan in strawberries in a single Spanish trial that complied with GAP (0.13-0.15 kg ai/hl; PHI, 21 days) was <0.01 mg/kg, and that of THPI was 0.15 mg/kg.

Use of captan on strawberries in the USA is permitted at a rate of 1.6-3.4 kg ai/ha with harvesting on the day of the last application. The concentrations of captan in 10 trials were 2.0, 2.6, 3.9, 4.4, 5.4, 7.7, 8.7, 10, and 12 (2 trials) mg/kg, and those of THPI were 0.15, 0.19, 0.22, 0.23, 0.3, 0.34, 0.5, 0.53, 0.9, and 1.4 mg/kg.

The Meeting considered that the indoor trials in Belgium and The Netherlands and the field trial in Spain represented different populations of residues and could not be used to estimate an STMR value. The concentrations of captan in strawberries in 14 trials, in rank order, were 0.93, 1.0, 2.0, 2.0, 2.4, 2.6, ***3.9***, ***4.4***, 5.4, 7.7, 8.7, 10, and 12 (2 trials) mg/kg. The Meeting estimated a maximum residue level of 15 mg/kg, an STMR value of 4.2 mg/kg, and a HR value for captan in strawberries of 12 mg/kg. The estimated maximum residue level replaces the current recommendation of 30 mg/kg for strawberries.

Data were available from supervised trials on *melon* conducted according to GAP in Japan and the USA.

In four field trials in Japan, where GAP is 0.13 kg ai/hl or 2-4 kg ai/ha with a PHI of 14 days, the concentrations of captan were 3.6, 4.0, 4.1, and 4.6 mg/kg. Values <0.005 mg/kg were found in eight indoor trials.

Trials conducted in the USA were evaluated with Mexican GAP (1-1.5 kg ai/ha, with no specification of the interval between the last spray and harvesting, implying that harvesting on the day of the last spray is permitted), as GAP was not specified for the USA. The concentrations of captan in nine trials conducted at 2.2 kg ai/ha with harvesting on the day of the last of seven applications were 0.29, 0.36, 0.52, 0.56, 1.1, 1.8, 2.0, 2.9, and 6.7 mg/kg.

The Meeting decided that the residues in the indoor trials in Japan represented a different population from the others and discounted them for the purposes of estimating the STMR value. The concentrations of captan in melons in 13 trials, in rank order, were 0.29, 0.36, 0.52, 0.56, 1.1, 1.8, ***2.0***, 2.9, 3.6, 4.0, 4.1, 4.6, and 6.7 mg/kg. For the purposes of estimating the STMR and HR values for use in assessing dietary intake, it was noted that peeling reduced the concentration of captan in cantaloupe by 98% in a processing study in the USA (TMN-634A). The Meeting estimated a maximum residue level of 10 mg/kg, an STMR value of 0.04 (2.0-0.02), and a HR value for captan in melons excluding watermelon of 0.13 (6.7-0.02) mg/kg.

Data were available from supervised trials on *cucumber* conducted according to GAP in Brazil and Japan.

Captan is registered for use on cucumbers in Brazil at a spray concentration of 0.1 kg ai/hl with a 1-day PHI. The concentrations of captan in two trials were 0.06 and 0.16 mg/kg.

In six trials conducted according to GAP in Japan (0.1-0.13 kg ai/hl or 1.5-4 kg ai/ha; PHI, 1 day), the concentrations was 1.9 mg/kg in two indoor trials and 0.20, 0.24, 1.2, and 1.5 mg/kg in four outdoor trials.

The Meeting decided that the residues in the Japanese trials conducted indoors represented a different population from the others and discounted them for the purposes of estimating the maximum residue level and STMR value. The concentrations of captan in six trials in cucumbers, in rank order, were 0.06, 0.16, 0.20, ***0.24***, 1.2, and 1.5 mg/kg. The Meeting estimated a maximum residue level of 3 mg/kg, an STMR value of 0.22 mg/kg, and a HR value for captan in cucumbers of 1.5 mg/kg.

Although four trials on *squash* were reported from the USA, they could not be evaluated because the trial conditions did not correspond to GAP. There was insufficient information to recommend a maximum residue level for squash.

Data were available from supervised trials in Brazil and Japan on *tomato* conducted according to GAP.

In four trials conducted according to GAP in Brazil (0.11-0.12 kg ai/hl; PHI, 1 day), the concentrations of captan were 0.02, 0.12, 0.18, and 0.46 mg/kg. THPI was not measured in one of the trials, but the concentrations in the other three were 0.08, 0.09, and 0.27 mg/kg.

Captan is registered for use on tomatoes in Japan at 0.007-0.1 kg ai/hl (1-3 kg ai/ha) with a 1-day PHI. The concentrations of captan in tomatoes in four indoor trials were 0.40, 0.45, 0.78, and 1.1 mg/kg, and those in 12 outdoor trials were 0.22, 0.28, 0.29, 0.45, 0.50, 0.61, 0.66, 0.76, 0.79, 1.0, 1.7, and 2.3 mg/kg. Residues of THPI were not measured.

The Meeting decided that the residues in the Brazilian trials represented a different population from the others and discounted them for the purposes of estimating the STMR value. The concentrations of captan in tomatoes in the 16 trials in Japan, in rank order, were 0.22, 0.28, 0.29, 0.40, 0.45 (2 trials), 0.50, ***0.61***, ***0.66***, 0.76, 0.78, 0.79, 1.0, 1.1, 1.7, and 2.3 mg/kg. The Meeting estimated a maximum residue level of 5 mg/kg, an STMR value of 0.64 mg/kg, and a HR value for captan in tomatoes of 2.3 mg/kg. The estimated maximum residue level replaces the current recommendation of 2 mg/kg for tomatoes.

The single trial from Thailand on *soya bean* could not be evaluated because the information on GAP in Thailand did not specify a PHI. There was insufficient information to recommend a maximum residue level for soya beans.

Data were available from supervised trials on *potato* conducted according to GAP in Brazil and Mexico.

The concentration of captan in a single trial in Brazil conducted according to GAP (0.11-0.12 kg ai/hl; PHI, 14 days) was <0.01 mg/kg; THPI was not measured. In a separate trial, with harvesting 7 days after the last application, no residues of captan were detected (<0.05 mg/kg).

In Mexico, captan is registered for application to potatoes at a spray concentration of 0.1-0.2 kg ai/hl with a 7-day PHI. The concentrations of captan and THPI were <0.05 mg/kg in four trials conducted at 1.5 times the Mexican GAP.

The concentrations of captan in potatoes in the six trials, in rank order, were <0.01 and ***<0.05*** (5 trials) mg/kg. The maximum residue level, the STMR value, and the HR value for captan in potatoes were all estimated by the Meeting to be 0.05 mg/kg. The results of many trials in which the GAP was not reported but in which the PHIs and application rates were similar to or greater than those in Brazil and Mexico support the conclusion that the concentration of residues will be <0.05 mg/kg.

Although eight trials on *radish* were reported from Germany, GAP was not provided, and they could not be evaluated. There was insufficient information to recommend a maximum residue level for radishes.

Data were available from supervised trials on *almond* conducted according to GAP in the USA. Captan is registered in the USA for use on almonds at a rate of 2.2-4.9 kg ai/ha with a 30-day PHI. The Meeting considered that captan is a surface residue but that shelling gives rise to residues in the nut. In 13 trials, the concentrations of captan were <0.01, 0.01, <0.03, 0.04, ***<0.05*** (7 trials), 0.10, and 0.20 mg/kg in the nut. The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR value of 0.05 mg/kg, and a HR value for captan in almonds of 0.2 mg/kg. The concentrations of captan in almond hulls 30 days after the last of five sprays at 5 kg ai/ha were 13, 48, and 53 mg/kg. The Meeting estimated a HR for almond hulls of 53 mg/kg.

Although six trials on *chives* were reported from Germany, GAP was not provided, and they could not be evaluated. There was insufficient information to recommend a maximum residue level for chives.

***Fate of residues during processing***

Information was provided to the Meeting on the fate of captan and THPI during the processing of lemons, oranges, grapefruit, apples, cherries, plums, grapes, strawberries, tomatoes, melons, cucumbers, and squash, and processing factors were calculated for processed commodities derived from these raw agricultural commodities. As maximum residue levels were not estimated for lemons, mandarins, grapefruit, oranges, and squash, the effect of processing is not discussed further.

Processing factors were calculated for captan only when it was the residue of concern for surveillance and estimation of dietary intake. When the concentration in the processed commodity does not exceed the LOQ, the processing factor is calculated from the LOQ and is prefixed with '<'. In all the studies of processing, heating and cooking had dramatic effects on the concentrations of captan residues.

The processing factors for apples and apple pomace (dry) were <0.3, <0.8, 0.23, 0.31, 0.33, 0.8, 0.9, 1.1, 1.3, 1.4, 1.6, and 1.9. When the two factors calculated for residues that are <LOQ are excluded, the mean processing factor is 1.0 ($\nu$ = 10). Application of the mean processing factor to the STMR and HR values for apples provides STMR-P and HR-P values for apple pomace (dry) of 4.95 and 7.8 mg/kg, respectively. The Meeting recommended withdrawal of the current recommendation (2 mg/kg) for dry apple pomace.

The processing factors for apples and apple juice (cold pressed) were <0.3, <0.8, <0.8, 0.03, 0.04, 0.05 (4 studies), 0.07, 0.09, 0.1 (3 studies), 0.15, 0.4, 0.5 (2 studies), 0.6, 0.7, 1.2, and 1.6. The mean processing factor, after exclusion of factors for residues in the processed commodity <LOQ, is 0.3 ($\nu$ = 19). Application of the mean processing factor to the STMR and HR values for apples provides STMR-P and HR-P values for juice of 1.5 and 2.3 mg/kg, respectively.

Washing apples removed approximately 50% of the captan residues, and negligible amounts remained after peeling. Captan is readily degraded on heating, such that the processing factors for pasteurized juice, apple sauce, apple jelly, and canned slices are essentially 0. The mean processing factor for dried apples, after exclusion of factors for residues in the processed commodity <LOQ, is 0.85.

A processing factor of 0.3 was obtained for washed cherries. As canning of cherries involves heating, no residues of captan were detectable. Washing peaches reduced the concentration of captan by 60%.

A processing factor of 0.1 was obtained for plums and prunes. Application of the processing factor to the STMR and HR values for plums results in STMR-P and HR-P values for dried plums (prunes) of 0.14 and 0.79 mg/kg, respectively.

The processing factors for raisins were 1, 1.2, 1.2, 1.3, and 2.6 (mean, 1.5). Application of the mean processing factor to the STMR and HR values for grapes results in an STMR-P value of 5.6 mg/kg and a HR-P value of 33 mg/kg for raisins. The Meeting estimated a maximum residue level for captan in dried grapes (currants, raisins, and sultanas) of 50 mg/kg, which confirms the current recommendation.

Captan did not concentrate in wine, pasteurized juice, depectinized juice, or grape jelly. For these commodities, the mean processing factors were <0.05. The processing factors for washed fruit, destemmed crushed grapes, cold pressed juice, wet pomace (cold pressed), and dry pomace were 0.8, 0.2, 0.4, 1, and 0.6, respectively.

A processing factor of 0.1 was obtained for washed strawberries. Cooking strawberries resulted in no detectable residues of captan.

The processing factors for both tomato juice and purée were <0.04 and <0.1. Application of a processing factor of 0.1 to the STMR and HR values for tomatoes provides STMR-P and HR-P values for juice and purée of 0.06 and 0.23 mg/kg.

Washing cucumbers reduced the concentration of captan by 80%, while peeling or cooking reduced it to <LOQ. Most of the captan residue in melons was removed by peeling, with processing factors of <0.01 and 0.3 obtained in the samples reported.

***Residues in animal commodities***

When captan is fed to animals, it is rapidly hydrolysed and metabolized to THPI, which is the residue of interest. The concentrations of THPI in milk reached a plateau after 1-4 days when dairy cattle were dosed with gelatine capsules at nominal rates equivalent to 10, 30 and 100 ppm. The plateau concentrations of THPI were <0.01, 0.03, and 0.2 mg/kg after these doses, respectively. The only other metabolite of which significant residues were detected was 3-OH THPI, for which plateau concentrations of 0.02, 0.06, and 0.2 mg/kg were found at the three doses,. After 29 days of dosing, the mean concentrations of residues were similar in liver, kidney, and muscle but were much lower in fat. There was an approximately linear trend with dose: the concentrations of the two metabolites in kidney, muscle, and liver were 0.11-0.31 mg/kg at 100 ppm, 0.04-0.12 mg/kg at 30 ppm, and 0.01-0.02 mg/kg at 10 ppm. No residues were detected in fat after administration of the lowest dose. The concentrations declined rapidly when dosing was stopped. The studies indicate that the residues of THPI resulting from feeding of captan at concentrations ≤ 10 ppm will not exceed 0.01 mg/kg in milk or 0.05 mg/kg in tissues. If it is assumed that the concentration increases linearly with dose, the residues of THPI resulting from feeding captan at concentrations ≤ 5 ppm will not exceed 0.01 mg/kg in milk or tissues, the LOQ in these media of the analytical method provided.

The dietary burden of captan residues in farm animals was estimated by the Meeting on the basis of the diets listed in Appendix IX to the FAO Manual on the Submission and Evaluation of Pesticide Residues Data for the Estimation of Maximum Residue Levels in Food and Feed (FAO, 1997). Ruminants may be fed apple pomace (dry) and potato culls. Although no information was available on residues of captan in potato waste and culls, captan is applied to potatoes as a seed and a foliar treatment. Neither use pattern would be expected to result in detectable residues, and it can be assumed that those in potato culls and processing waste are <LOQ (0.05 mg/kg).

The estimated intakes of captan by beef and dairy cattle are:

| Feed item | HR or HR-P, mg/kg | % dry matter | % of diet | | Intake, ppm of diet | |
|---|---|---|---|---|---|---|
| | | | Beef cattle | Dairy cows | Beef cattle | Dairy cows |
| Apple pomace (dry) | 7.8 | - | 20 | 40 | 1.6 | 3.1 |
| Potato culls | 0.05 | 20 | 70 | 40 | 0.2 | 0.1 |
| Almond hulls | 53 | 90 | 10 | 10 | 5.9 | 5.9 |
| Total | | | 100 | 80 | 7.7 | 9.1 |

The dietary burdens of captan in beef and dairy cattle are 7.7 and 9.1 ppm, respectively. The dietary burden of cattle is dominated by intake of residues in almond hulls. If almond hulls are not included in the calculation, the estimated dietary burden of beef and dairy cattle is <5 ppm, which would not result in residues in excess of the LOQ for THPI and its hydroxy metabolites in milk and bovine tissues. The Meeting decided that it was inappropriate to estimate maximum residue levels for animal commodities on the basis of such a minor animal feed commodity. Should future uses result in residues of captan in significant animal feeds, this study provides a good basis for setting maximum residue levels in animal commodities.

## RECOMMENDATIONS

The Meeting estimated the maximum residue and STMR levels shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: captan

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| TN 0660 | Almonds | 0.3 | - | 0.05 | 0.2 |
| FP 0226 | Apple | W[1] | 20 | - | - |
| AB 0226 | Apple pomace, dry | W | 2 | 4.95 | 7.8 |
| FB 0020 | Blueberries | 20 | 20 | 6.9 | 18 |
| FS 0013 | Cherries | 25 | 40 | 11 | 21 |
| VC 0424 | Cucumber | 3 | - | 0.22 | 1.5 |
| DF 0269 | Dried grapes (= Currants, Raisins and Sultanas) | 50 | 50 | 5.6 | 33 |
| FB 0269 | Grapes | 25 | 25 | 3.7 | 22 |
| VC 0046 | Melons, except Watermelon | 10 | - | 0.04 | 0.13 |
| FS 0245 | Nectarine | 3 | 5 | 1.0 | 1.8 |
| FS 0247 | Peach | 20 | 15 | 4.7 | 16 |
| FP 0230 | Pear | W[1] | 10 | - | - |
| FS 0014 | Plums (including Prunes) | 10 | 5 | 1.4 | 7.9 |
| FP0009 | Pome fruits | 15 Po | - | 5.3 | 11 |
| VR 0589 | Potato | 0.05 | - | 0.05 | 0.05 |
| DF 0014 | Prunes | | | 0.15 | 0.84 |
| FB 0272 | Raspberries, Red, Black | 20 | - | 8.3 | 18 |
| FB 0275 | Strawberry | 15 | 30 | 4.15 | 12 |
| VO 0448 | Tomato | 5 | 2 | 0.64 | 2.3 |
| VJ 0448 | Tomato juice | | | 0.06 | 0.23 |
| | Tomato purée | | | 0.06 | 0.23 |

[1] Now included in recommendation for Pome fruits

## Dietary risk assessment

### *Chronic intake*

The periodic review of captan resulted in recommendations for new and revised MRLs and new STMR values for raw and processed commodities. Data on consumption were available for 19 food commodities and were used in calculating dietary intake. The results are shown in Annex 3 (JMPR 2000 Report). The IEDIs for the five GEMS/Food regional diets, based on the estimated STMR values, represented 0-8% of the ADI. The Meeting concluded that long-term intake of residues of captan from uses that have been considered by the JMPR is unlikely to present a public health concern.

### *Short-term intake*

The 2000 JMPR concluded that it was unnecessary to establish an acute RfD for captan. This conclusion was based on the considerations that the pesticide is unlikely to present an acute toxicological hazard, and its residues are therefore unlikely to present an acute risk to consumers.

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# CHLORMEQUAT (015)

## EXPLANATION

Chlormequat was evaluated in the CCPR Periodic Review Programme in 1994. The Meeting estimated maximum residue levels for a number of commodities, but they were recorded as only Guideline Levels because the ADI was withdrawn. As an ADI was allocated by the 1997 JMPR, the 1994 estimates were then recommended for use as MRLs. The 1994 JMPR had requested further information on feeding studies with cows and poultry, analytical methods for animal products, processing studies on cotton seed and residue studies on mushrooms grown on straw with a residue level of 15 to 20 mg/kg.

At the 30th Session of the CCPR, it was noted that animal transfer studies on poultry and cattle would be available in 1998.

The compound was reviewed again for toxicology in 1999, when an acute reference dose was allocated and the Meeting recommended that an acute risk assessment should be carried out.

A dairy cattle and a poultry feeding study, analytical methods for the determination of chlormequat residues in water, cereals, pears and animal products as well as the results of trials on pears and cereals were reported to the present Meeting by the manufacturers (CCC Task Force). The government of the Netherlands reported the official method of analysis for chlormequat in pears. Information on national MRLs and GAP was provided by the governments of Germany, Poland and The Netherlands.

## METHODS OF RESIDUE ANALYSIS

### Analytical methods

The methods used in the past were based on semi-quantitative thin-layer chromatographic or photometric determination (JMPR, 1994). These involved a lengthy clean-up process, with poor reproducibility and high values in samples from untreated control plots. Recent methods are based on head-space gas chromatography after pyrolysis in an alkaline medium, ion-pair HPLC with conductivity detection, or LC-MS.

Cereals. Caddy and Carroll (1982) adapted the colorimetric method of Mooney and Pasarela (1967) for the determination of chlormequat in barley grain and straw. After methanolic extraction, clean-up was by chromatography on a column of alumina with an acetone/methanol mixture. The residue was determined colorimetrically as a dipicrylamine-chlorocholine chloride complex. The LOD was 0.1 mg/kg.

Chlormequat residues in grain and straw samples were determined by Byast and Tolhurst (1990, 1992), with extraction and clean-up procedures based on those of Mooney and Pasarela (1967). After extraction with methanol and clean-up on an alumina column, the sample was derivatized with sodium thiophenolate to form a volatile ether for gas chromatography with a flame photometric detector in the sulfur mode. The LOD was 0.1 mg/kg but the lowest fortification level was 1 mg/kg for wheat and barley (recoveries: grain 95%, straw 103%) and 2 mg/kg for rye and triticale (grain 73-80%, straw 73-84%).

An analytical method for the determination of chlormequat in cereal forage, straw, grain, bran, flour and oat flakes was validated by Schneider (1992, 1993). The compound was extracted with methanol, and an aliquot of the methanol extract concentrated, transferred with water to a C-18

cartridge, and the eluate cleaned up further by ion exchange. Identification was by GLC with an FID after pyrolytic decomposition of the chlormequat to acetylene by heating an alkaline medium in a closed tube for 15 min at 200°C. The recoveries are shown in Table 1. The LOD for all samples was 1 mg/kg. High values were found in the untreated control samples.

Table 1. Recoveries of chlormequat chloride from forage, grain, straw and processed cereal products (Schneider 1993).

| Sample | Fortification level, mg/kg | Recovery, mg/kg | Mean, mg/kg | SD, mg/kg | CV, % |
|---|---|---|---|---|---|
| Forage (oats) | 0 | 1.9, 1.8, 2, 1.9 | 1.9 | 0.056 | 2.9 |
| | 1 | 2.9, 2.8, 2.5, 2.8 | 2.7 (83%[1]) | 0.18 | 6.7 |
| | 10 | 8.5, 9, 11, 12 | 10 (83%[1]) | 1.6 | 16 |
| | 50 | 48, 46, 44, 42 | 45 (87%[1]) | 2.6 | 5.8 |
| Straw (wheat) | 0 | 0.38, 0.48, 0.53, 0.52 | 0.48 | 0.068 | 14 |
| | 1 | 1.4, 1.3, 1.3, 1.3 | 1.3 (82%[1]) | 0.042 | 3.2 |
| | 10 | 6.8, 8, 9.7, 8.8 | 8.3 (78%[1]) | 1.2 | 14 |
| | 50 | 41, 41, 44, 40 | 42 (82%[1]) | 1.3 | 3.1 |
| Grains (oats) | 0 | 0.25, 0.26, 0.29 | 0.27 | 0.021 | 7.8 |
| | 0.5 | 0.75, 0.74, 0.66 | 0.72 (90%[1]) | 0.049 | 6.8 |
| | 1 | 1.4, 1.3, 1.3, 1.2 | 1.29 (103%[1]) | 0.053 | 4.1 |
| | 3 | 2.5, 2.4, 2.9, 2.8 | 2.62 (79%[1]) | 0.23 | 8.8 |
| Bran (rye) | 0 | 1.1, 0.98, 0.87, 0.94 | 0.96 | 0.075 | 7.8 |
| | 1 | 2.1, 1.8, 2 | 1.95 (99%[1]) | 0.12 | 6.1 |
| | 3 | 3.1, 3.8, 3.5, 3.2 | 3.43 (82%[1]) | 0.31 | 9.0 |
| Flour (rye) | 0 | 0.54, 0.47, 0.64, 0.62 | 0.57 | 0.078 | 14 |
| | 1 | 1.36, 1.33, 1.42, 1.42 | 1.38 (81%[1]) | 0.042 | 3.0 |
| | 3 | 2.6, 2.9, 3.3, 3.2 | 2.98 (80%[1]) | 0.31 | 10 |
| Flakes (oats) | 0 | 0.36, 0.33, 0.34, 0.34 | 0.34 | 0.013 | 3.8 |
| | 1 | 1.1, 1.1, 0.96, 1.1 | 1.07 (72%[1]) | 0.076 | 7.1 |
| | 3 | 2.6, 2.8 2.3, 2.4 | 2.51 (72%[1]) | 0.21 | 8.4 |

SD: standard deviation
CV: coefficient of variation
[1]after substraction of mean blank value

Fegert (1996) validated the BASF method 314/1 for wheat and barley (forage, grain and straw). The compound was extracted with water/acetone (1:2). After liquid-liquid partition with dichlormethane/water the active ingredient was isolated as an ion-pair with sodium tetraphenyl borate as complexing reagent, extracted with hydrochloric acid and further purified by alumina column chromatography. Determination was based on ion-pair HPLC with column switching. The compound was paired with hexanesulfonic acid and chromatography was on a neutral, hydrophobic column with an aqueous/acetonitrile mobile phase and suppressed conductivity detection. (Dionex ion chromatograph; pre-column PRP-1, 150 x 4.1 mm, 10 µm; analytical column PRP-1, 250 x 4.1 mm, 10 µm). The LOD for chlormequat chloride residues was 0.5 mg/kg in straw and 0.05 mg/kg in all other samples. Control samples fortified with chlormequat chloride at 0.05, 0.5 and 5 mg/kg showed mean recoveries ranging from 71.4% ± 2.7% to 94.6% ± 3.4% (Table 2).

Table 2. Recoveries of chlormequat chloride from fortified forage, grain and straw of cereals (Fegert, 1996).

| Sample | Fortification level, mg/kg | Recovery, % | Mean, % | SD, % | CV, % |
|---|---|---|---|---|---|
| Wheat forage | 0.05 | 80, 82, 78, 75, 77 | 78 | 2.6 | 3.3 |
| | 5 | 77, 75, 75, 76, 74 | 76 | 0.9 | 1.2 |
| Wheat grain | 0.05 | 81, 79, 95, 78, 81 | 83 | 7.0 | 8.5 |
| | 5 | 74, 73, 73, 70, 67 | 71 | 2.7 | 3.8 |
| Wheat straw | 0.5 | 81, 83, 82, 84, 83 | 83 | 1.4 | 1.7 |
| | 5 | 79, 79, 79, 81, 81 | 80 | 1.3 | 1.6 |

| Sample | Fortification level, mg/kg | Recovery, % | Mean, % | SD, % | CV, % |
|---|---|---|---|---|---|
| Barley forage | 0.05 | 74, 75, 76, 76, 75 | 75 | 0.7 | 1.0 |
| | 5 | 87, 87, 88, 85, 82 | 86 | 2.2 | 2.6 |
| Barley grain | 0.05 | 85, 83, 96, 87, 85 | 87 | 4.8 | 5.5 |
| | 5 | 93, 89, 86, 89, 93 | 90 | 2.7 | 3.0 |
| Barley straw | 0.5 | 97, 97, 93, 98, 89 | 95 | 3.4 | 3.6 |
| | 5 | 93, 90, 93, 92, 93 | 92 | 1.4 | 1.6 |

BASF Method 314/1 was also validated for wheat grain by Schulz (1996a) and Kuhlmann (1977) with samples fortified at 0.05 mg/kg and 5 mg/kg. Schulz obtained a mean recovery and coefficient of variation of 81% and 6.5% respectively from 5 determinations at each level, with only small peaks in the control samples in the relevant retention time range, corresponding to <0.05 mg/kg as chlormequat chloride. Kuhlmann (1997) reported a mean recovery and coefficient of variation of 89.8% and 7.14% respectively from two analyses at each level.

Schneider (1997a) validated the GC-MS "Dr G. Krebs Analytik Method DrK120" in which chlormequat chloride is determined in wheat grain from a decomposition fragment formed in the injector of the gas chromatograph by comparison with an internal standard. Validation was at fortification levels of 0.05 and 5 mg/kg. The mean recovery was 92% ± 7.6%, with values around 0.05 mg/kg in the untreated control samples.

A study by Sasturain (1997) was designed to demonstrate that contract and official laboratories in Germany are capable of analysing residues of chlormequat in wheat grain. An inter-laboratory evaluation (ring test) using spiked samples (level 1 = 0.08 mg/kg, level 2 = 2.8 mg/kg) was carried out at four laboratories using method DrK120 and BASF method 314/1. The mean recovery and the coefficient of variation were 106.3% and 24.5% for level 1, and 98.2% and 2% for level 2, showing a correlation between the levels of chlormequat chloride and the precision of the results. There were no false positive results. The results indicate that the laboratories' findings do not depend on the analytical method but on the expertise and experience of each laboratory with the individual methods.

Pears. The official analytical method of The Netherlands for the determination of chlormequat in plant material (Anon., 1996) has been applied to pears. A methanol extract is cleaned by ion-exchange and alumina chromatography, then evaporated to dryness and heated in an alkaline medium in a closed tube at 215°C to convert chlormequat to acetylene, which is determined in the head-space by gas chromatography with flame-ionisation detection. The LOD of chlormequat in pears was 0.01 mg/kg.

A new analytical method relying on quantification by tandem liquid chromatography with mass spectrometric detection (LC-MS-MS) was validated for pears (Quirijns and van Dam, 1999; Quirijns, 1999). Chlormequat chloride was extracted from the plant material with water after homogenization. After filtration, 20 µl of the extract was injected on to a 100 x 3 mm I.D. 5 µm Spherisorb CN column with a 10 x 3 mm I.D. R2 guard column. Isocratic elution was with a mobile phase of methanol/water/1 M aqueous ammonium acetate (50:49:1) at a flow rate of 0.3 ml/min. MS/MS data were acquired by selecting the ion at m/z 122 as the precursor ion and the ion at m/z 58 as the product ion. The multiplier was run at 1200 V.

The recovery and repeatability are shown in Table 3. The limit of detection was 0.007 mg/kg. The LOD claimed by the authors was 0.1 mg/kg, but as the lowest fortification level was 0.29 mg/kg the validated LOD is 0.3 mg/kg.

Table 3. Recovery and repeatability of chlormequat determination in pears (Quirijns and van Dam, 1999).

| Chlormequat chloride added, mg/kg (N = 6) | Mean recovery, % | SD, % |
|---|---|---|
| 0.29 | 106 | 2.7 |
| 2.9 | 105 | 1.6 |
| 9.8 | 89 | 2.6 |

Animal products. Weidenauer (1999a,b) validated an ion-pair HPLC method to determine chlormequat in fortified poultry and dairy cow products. Samples of hen eggs, meat, liver and fat, and cow milk, meat, liver, kidney and fat were homogenized, extracted with a mixture of acetone and water (2:1), and passed through a cation exchange column. Chlormequat was eluted with diluted HCl and the eluant evaporated. The dry residue was re-dissolved in water and washed with dichloromethane, and the aqueous phase was evaporated to dryness. The residue was then transferred to an alumina column, eluted with a mixture of acetonitrile and methanol, and the eluate evaporated to dryness. The residue was transferred via methanol to water for analysis by ion-pair chromatography with column switching. The mobile phase was hexanesulfonic acid (2mmol/l)/$CH_3CN$ (97:3) in a gradient system. A Hamilton PRP-1, 15 cm x 4.1 mm column was used as pre-column and the analyte was transferred to the separation column by means of a motor driven switch valve during a period of about 1-1.5 min. A Hamilton PRP-1, 25 x 4.1 mm column was used for analyte determination. A Dionex CDM-2 conductivity detector with background conductivity suppression was used for detection. Under these conditions the retention time for chlormequat was about 22 min.

In the hen trial, no residues of chlormequat were found at or above the LOD of 0.05 mg/kg in any control samples of meat, liver, fat or eggs. At spike levels of 0.05-0.5 mg/kg the average recovery from tissues was 80% with a relative standard deviation of 9.5%, and from eggs 83.5% with a relative standard deviation of 16% (Table 4).

Table 4. Recoveries of chlormequat chloride from fortified hen tissues and eggs (Weidenauer, 1999a).

| Sample | Fortification level, mg/kg | Measured value, mg/kg | Recovery, % |
|---|---|---|---|
| Hen meat | 0 | n.d. [1] | |
| | 0.05 | 0.042 | 84 |
| | 0.5 | 0.38 | 75.5 |
| Hen liver | 0 | 0.035 | |
| | 0.05 | 0.08 | 92[2] |
| | 0.5 | 0.38 | 69[2] |
| Hen fat | 0 | n.d. | |
| | 0.05 | 0.04 | 80 |
| | 0.5 | 0.4 | 80 |
| Eggs | 0 | n.d. | |
| | 0.05 | 0.057 | 114 |
| | | 0.039 | 78 |
| | | 0.058 | 116 |
| | | 0.044 | 88 |
| | | 0.041 | 82 |
| | | 0.051 | 102 |
| | | 0.046 | 92 |
| | | 0.045 | 90 |
| | | 0.035 | 70 |
| | | 0.041 | 82 |
| | | 0.037 | 74 |
| | | 0.044 | 88 |
| | | 0.053 | 106 |
| | | 0.036 | 72 |
| | | 0.043 | 86 |
| | | 0.036 | 72 |
| | 0.5 | 0.44 | 88 |
| | | 0.43 | 86 |

| Sample | Fortification level, mg/kg | Measured value, mg/kg | Recovery, % |
|---|---|---|---|
| | | 0.49 | 98 |
| | | 0.35 | 70 |
| | | 0.39 | 78 |
| | | 0.40 | 80 |
| | | 0.50 | 100 |
| | | 0.40 | 80 |
| | | 0.38 | 76 |
| | | 0.35 | 70 |
| | | 0.34 | 68 |
| | | 0.35 | 70 |
| | | 0.32 | 64 |
| | | 0.41 | 82 |
| | | 0.41 | 82 |
| | | 0.43 | 86 |
| | | 0.37 | 74 |
| | | 0.38 | 76 |
| | | n (eggs) | 34 |
| | | Average recovery % (eggs) | 83.5 |
| | | SD | 13 |
| | | rel. SD, % | 16 |

[1] not detected
[2] corrected for control

No residues of chlormequat were found at or above the LOD of 0.05 mg/kg in any control sample of cow meat, liver, kidney or fat. 0.037 mg/kg was detected in one kidney control sample. The average recovery from the tissues was 86% at spike levels of 0.05 and 0.5 mg/kg with a relative standard deviation of 18.9% (Table 5).

Table 5. Recoveries of chlormequat chloride from fortified cow tissues (Weidenauer, 1999b).

| Sample | Fortification level, mg/kg | Measured value, mg/kg | Recovery, % |
|---|---|---|---|
| Meat | 0 | n.d. [1] | |
| | 0.05 | 0.042 | 84 |
| | 0.5 | 0.4 | 80 |
| Liver | 0 | n.d. | |
| | 0.05 | 0.052 | 104 |
| | 0.5 | 0.41 | 82 |
| Kidney | 0 | n.d., 0.037 | |
| | 0.05 | 0.061, 0.085 | 122, 170 (96)[2] |
| | 0.1 | 0.078 | 78 |
| Fat | 0 | n.d. | |
| | 0.05 | 0.031, 0.039 | 62, 78 |
| | 0.5 | 0.4, 0.355 | 80, 71 |

[1]not detected
[2]corrected for control

Table 6 shows the recoveries of chlormequat chloride from spiked milk, skimmed milk and cream. Only two milk and one cream control samples showed interference peaks at the retention time of chlormequat. At spike levels of 0.01-0.2 mg/kg, the average recovery of chlormequat chloride was 84.7% from milk and 88.8% from skimmed milk and cream with relative standard deviations of 14.6% and 21% respectively.

Table 6. Recoveries of chlormequat chloride from fortified cow milk, skimmed milk and cream (Weidenauer, 1999b).

| Sample | Fortification level, mg/kg | Measured value, mg/kg | Recovery, % |
|---|---|---|---|
| Milk | 0.01 | 0.013 (corresponding control sample: 0.0036) | 95[1] |
| | | 0.0089 | 89 |
| | | 0.0069 | 69 |
| | | 0.008 | 80 |
| | | 0.0092 | 92 |
| | | 0.0077 | 77 |
| | | 0.0074 | 74 |
| | | 0.012 | 120 |
| | | 0.0069 | 69 |
| | | 0.0079 | 79 |
| | | 0.0099 | 99 |
| | | 0.0082 | 82 |
| | 0.1 | 0.078 | 78 |
| | | 0.079 (corresponding control sample: 0.0036) | 76[1] |
| | | 0.071 | 71 |
| | | 0.1 | 100 |
| | | 0.097 | 97 |
| | | 0.097 | 97 |
| | | 0.08 | 80 |
| | | 0.099 | 99 |
| | | 0.078 | 78 |
| | | 0.091 (corresponding control sample: 0.0032) | 88[1] |
| | | 0.089 | 89 |
| | | 0.094 | 94 |
| | | 0.065 | 65 |
| | | 0.06 | 65 |
| | 0.2 | 0.17 | 85 |
| Skimmed milk | 0.01 | 0.0098 | 98 |
| | | 0.0087 | 87 |
| | | 0.0076 | 76 |
| | | 0.011 | 110 |
| | 0.1 | 0.071 | 71 |
| | | 0.12 | 117 |
| | | 0.063 | 63 |
| | | 0.068 | 68 |
| | 0.2 | 0.23 | 115 |
| Cream | 0.01 | 0.01 | 100 |
| | | 0.009 | 90 |
| | | 0.0071 | 71 |
| | | 0.01 | 100 |
| | 0.1 | 0.089 | 89 |
| | | 0.088 (corresponding control sample: 0.019) | 69[1] |

[1]corrected for control

Water. Mackenroth and Sasturain (1995) validated BASF method 370 for the determination of chlormequat in tap water, leachate (lysimeter) water, and water from a small stream and the German Rhine river (surface waters). A 1000 ml sample of water was extracted with dichloromethane to remove non-polar components. The ion-pairing reagent, sodium tetraphenyl borate, was then added and chlormequat was partitioned into dichloromethane, then re-extracted from the dichloromethane phase with 2 M hydrochloric acid. The HCl phase was taken to dryness, and the residue re-dissolved in acetonitrile/methanol (95:5 v/v) and cleaned up on an acid alumina column. The eluate was concentrated to dryness, dissolved in ultra-pure water and quantified by ion chromatography (Dionex ion chromatograph with suppressed conductivity detection; pre-column PRP-1, 150 x 4.1 mm, 10 µm; analytical column PRP-1, 250 x 4.1 mm, 10 µm). The mobile phase for the pre-column and analytical column consisted of a 2 mM hexanesulfonic acid solution and acetonitrile (95:5 v/v). The LOD was

0.05 µg/l, with mean recoveries at that level of 106% from tap water, 82% from lysimeter water, 77% from Rhine river water and 76% from stream water (Table 7).

Table 7. Recoveries of chlormequat chloride from water (Mackenroth and Sasturain, 1995).

| Water | Fortification level, µg/l | Recovery, % | Mean, % | SD, % | CV, % |
|---|---|---|---|---|---|
| Limburgerhof tap water | 0.05 | 106, 110, 103, 103, 107 | 106 | 2.7 | 2.6 |
| | 0.1 | 107, 94, 90, 80, 82 | 91 | 11 | 12 |
| | 5 | 90, 94, 97, 98, 97 | 95 | 3.1 | 3.3 |
| Lysimeter water | 0.05 | 89, 78, 78, 78, 89 | 82 | 5.8 | 7.0 |
| | 0.1 | 71, 68, 58, 73, 74 | 69 | 6.6 | 9.5 |
| | 5 | 95, 93, 95, 91, 91 | 93 | 2.1 | 2.3 |
| Rhine river water | 0.05 | 76, 84, 65, 81, 82 | 77 | 7.6 | 9.8 |
| | 0.1 | 83, 79, 67, 88, 76 | 79 | 7.9 | 10 |
| | 5 | 98, 96, 97, 97, 98 | 97 | 0.7 | 0.7 |
| Stream water | 0.05 | 87, 89, 87, 90, 26 | 76 | 28 | 37 |
| | 0.1 | 80, 75, 87, 85, 74 | 80 | 5.6 | 6.9 |
| | 5 | 108, 105, 105, 106, 115 | 108 | 4.0 | 3.7 |

SD: standard deviation
CV: coefficient of variation

BASF Method 370 was also validated by Schulz (1996b, 1997) for tap water and by Kuhlmann (1997) for drinking water. Five samples of tap water and 2 samples of drinking water were each fortified at 0.05 µg/l and 5 µg/l. The mean recovery and the coefficient of variation were 90.5% and 10.9% respectively for tap water and 102.7% and 7.4% respectively for drinking water. No peak was observed in the control tap water.

Schneider (1997b) validated Dr G. Krebs Analytik Methods DrK086 and DrK199 for drinking water. In DrK086 disodium ethylenediaminetetraacetate (ETDA) was added to complex the calcium ions in the water. The sample was made alkaline with sodium hydroxide beads and chlormequat was extracted with a solution of dipicrylamine in dichloromethane, then back-extracted with 1 N hydrochloric acid and taken to dryness. The residue was transferred with methanol to a thick-walled screw-top centrifuge tube, taken to dryness and 5% potassium hydroxide added. The centrifuge tube was closed and heated for 15 min at 200°C to pyrolyse the chlormequat in the alkaline solution. After cooling, 250 µl of the gas phase was injected into the GC. The acetylene produced was determined by GLC with an FID. The LOD of the method was 0.05 µg/l (Table 8).

Table 8. Recoveries of chlormequat chloride from water by method DrK086 (Schneider, 1997b).

| Addition, µg/l | Measured value, µg/l | Recovery, % |
|---|---|---|
| 0 | 0.0074 | |
| 0.05 | 0.054 | 107 |
| 0.05 | 0.053 | 106 |
| 5 | 4.9 | 99 |

In DrK119 determination was by GC-MS, the chlormequat being decomposed in the injector of the gas chromatograph as in DrK120 for wheat grain. An internal standard is used. Fortification levels were 0.05, 0.1 and 0.3 µg/l. Because the control samples contained apparent residues of about 0.025 µg/l, the results at 0.05 µg/l could not be relied upon and the LOD was 0.1 µg/l (Table 9).

Table 9. Recoveries of chlormequat chloride from water by method DrK119 (Schneider, 1997b).
F

| Addition, µg/l | Measured value, µg/l | Uncorrected recovery, % | Corrected anal. value, µg/l | Corrected recovery, % | Mean µg/l | Mean corr. recovery, % | SD, µg/l | CV, % |
|---|---|---|---|---|---|---|---|---|
| 0 | 0.028 | | | | 0.025 | | 0.0023 | 9.3 |
| 0 | 0.026 | | | | | | | |

| Addition, µg/l | Measured value, µg/l | Uncorrected recovery, % | Corrected anal. value, µg/l | Corrected recovery, % | Mean µg/l | Mean corr. recovery, % | SD, µg/l | CV, % |
|---|---|---|---|---|---|---|---|---|
| 0 | 0.024 | | | | | | | |
| 0 | 0.022 | | | | | | | |
| 0.1 | 0.098 | 98 | 0.073 | 72 | 0.072 | 71 | 0.0022 | 3.1 |
| 0.1 | 0.096 | 96 | 0.071 | 70 | | | | |
| 0.1 | 0.1 | 100 | 0.075 | 74 | | | | |
| 0.1 | 0.096 | 96 | 0.07 | 70 | | | | |
| 0.3 | 0.27 | 89 | 0.25 | 81 | 0.23 | 77 | 0.012 | 5.2 |
| 0.3 | 0.26 | 87 | 0.24 | 79 | | | | |
| 0.3 | 0.25 | 81 | 0.22 | 73 | | | | |
| 0.3 | 0.25 | 81 | 0.22 | 73 | | | | |

The inter-laboratory ring test on wheat grain described above (Sasturain, 1997) was also carried out with drinking water spiked at 0.11 and 0.25 µg/l by five laboratories using methods DrK086, DrK119 and BASF 370. The mean recoveries and the coefficients of variation were 85.5% and 5.8% at 0.11 µg/l, and 96% and 9% at 0.25 µg/l, showing satisfactory accuracy and reproducibility. There were no false positive results.

**Stability of pesticide residues in stored analytical samples**

The stability of chlormequat residues in animal products stored in freezers (milk, eggs and edible tissues) was investigated by Weidenauer (2000). Control and treated samples from the animal feeding studies A-49-97-05 (Weidenauer, 1999a) and A-49-97-06 (Weidenauer, 1999b) were used for the trial. The eggs were broken and homogenized without shells with a spatula, and the fat and liver were homogenized using a Tecator homogenizer. Aliquots of about 500 g were placed in plastic boxes and stored at ≤-18°C. Whole milk was not homogenized. The HPLC method of Weidenauer (1999a,b) described above was used. Table 10 shows the individual results and the corresponding recoveries.

Table 10. Effect of freezer storage on incurred chlormequat chloride residues in milk, eggs and edible tissues (Weidenauer, 2000).

| Com-modity | Sample no. | Sampling date | Initial analysis | | Analysis after freezer storage | | | % remaining |
|---|---|---|---|---|---|---|---|---|
| | | | Date | Residue, mg/kg | Date | Interval, months | Residue, mg/kg | |
| Milk | LA 43 | 2-Oct-97 | 7-Jan-98 | <0.01 | 14-Aug-00 | 31 | <0.01 | |
| Milk | LA 829 | 5-Oct-97 | 20-Nov-97 | 0.35 | 14-Aug –00 | 33 | 0.48 | 137 |
| Milk | LA 902 | 5-Oct-97 | 20-Nov-97 | 0.33 | 14-Aug –00 | 33 | 0.25 | 76 |
| Eggs | LA 1232 | 17-Mar-98 | 17-Sep-98 | <0.05 | 14-Aug –00 | 23 | <0.05 | |
| Eggs | LA 1170 | 16-Mar-98 | 21-Apr-98 | <0.05 | 21-Aug-00 | 28 | <0.05 | |
| Eggs | LA 1475 | 16-Mar-98 | 21-Apr-98 | 0.19 | 14-Aug –00 | 28 | 0.085 | 45 |
| Eggs | LA 1445 | 17-Mar-98 | 21-Apr-98 | 0.12 | 21-Aug –00 | 28 | 0.12 | 100 |
| Liver | LA1104 | 14-Oct-97 | 9-Jul-98 | <0.05 | 14-Aug –00 | 25 | <0.05 | |
| Liver | LA1136 | 14-Oct-97 | 9-Jul-98 | 0.4 | 14-Aug –00 | 25 | 0.33 | 82.5 |
| Liver | LA1144 | 14-Oct-97 | 9-Jul-98 | 0.5 | 14-Aug –00 | 25 | 0.3 | 60 |
| Fat | LA1106 | 14-Oct-97 | 4-Feb-98 | <0.05 | 14-Aug –00 | 30 | <0.05 | |
| Fat | LA1102 | 13-Oct-97 | 4-Feb-98 | <0.05 | 21-Aug –00 | 31 | <0.05 | |
| Fat | LA 1138 | 14-Oct-97 | 4-Feb-98 | 0.12 | 14-Aug –00 | 30 | 0.085 | 71 |
| Fat | LA 1146 | 14-Oct-97 | 4-Feb-98 | 0.09 | 21-Aug –00 | 31 | 0.081 | 90 |

## USE PATTERN

The main use of the plant growth regulator chlormequat is to consolidate the stems of cereals to prevent lodging. In pears, common uses are to inhibit vegetative growth and promote flowering in the following season. The Meeting was provided with information on currently registered uses by the governments of The Netherlands, Germany and Poland as well as by the Task Force members BASF, Ciba Speciality, Nufarm and UCB (Table 11).

Table 11. Registered uses of chlormequat in Europe at June 2000. All foliar spraying and field uses, ai expressed as chlormequat chloride.

| Crop | Country | Product, % ai | Application | | | | | PHI, days |
|---|---|---|---|---|---|---|---|---|
| | | | Rate, kg ai/ha | Spray conc., kg ai/hl | Water, l/ha | Growth stage | No. | |
| Almonds | Spain | SL 40 | 0.72 | 0.08 | 900 | 1 week before flowering | 1 | |
| Barley | Italy | SL 30.5 | 0.76 | | | | | |
| | | SL 46 | 1.8 | | | | | |
| | Spain | SL 23.6 | 0.59 | | | | | |
| Barley, summer | Belgium | SL 23 | 0.46 | | 200 | | 1 | F[2] |
| | Germany | SL 30.5 | 0.6 | | 200-400 | BBCH[1] 32-49 | 1 | 42 |
| | Ireland | SL 75 | 1.5 | | 220 | | 1 | F |
| | Netherlands | SL 30.5 | 0.61 | | | | 1 | F |
| | | SL 36 | 0.32-0.63 | | 200-600 | Beginning of shooting until stage 6-8 of Feekes scale (BBCH 30-37) | 1 | |
| | | SL 36 | 0.62-0.90 | | | | 1 | |
| | UK | SC 23 | 0.35 | | | | | |
| | | SL 64.5 | 1.6 | | | | 1-2 | F |
| Barley, winter | Belgium | SL 23 | 0.69 | | 200 | | 1 | F |
| | Germany | SL 30.5 | 0.76 | | 200-400 | BBCH 32-49 | 1 | 42 |
| | Ireland | SL 75 | 0.56-1.5 | | 220 | | 1 | F |
| | Netherlands | SL 30.5 | 0.92 | | | | 1 | F |
| | UK | SC 23 | 0.46 | | | | | |
| | | SC 34.5 | 0.69 | | | | | |
| | | SL 64.5 | 1.6 | | | | 1-2 | F |
| | | SL 72 | 1.6 | | | | 1 | F |
| | | SL 75 | 1.7 | | | | 1 | F |
| Cereals | Germany | SL 30.5 | 0.61-0.76 | | 200 | | 1 | 42 |
| | Spain | SL 40 | 1.4 | | 200-300 | From BBCH 30 and at formation of each node | 1 | 30 |
| Cereals, summer (barley, rye, triticale, wheat) | Poland | SL 46 | 0.46-0.92 | | 150-300 | Beginning of shooting (BBCH 30) | 1 | 42 |
| Cereals, winter (barley, rye, triticale, wheat) | Poland | SL 46 | 0.69-1.2 | | 150-300 | Beginning of shooting (BBCH 30) | 1 | 42 |
| | Luxembourg | SL 23 | 0.92 | | | | | |
| Cotton | Brazil | SL 10 | 0.1 | | | 70 days after emergence | 1 | |
| Grapes | Italy | SL 46 | 1.6 | | | | | |
| | Spain | SL 40 | 1.3 | 0.14 | 900 | 2-3 weeks before flowering | 1 | |
| Linseed and fibrous flax | Netherlands | SL 30.5 | 1.4 | | | | 1 | F |
| | | SL 36 | 0.90-1.4 | | 200-600 | At plant height of 30-45 cm | 1 | |
| | UK | SL 64.5 | 1.6 | | | | 1 | F |
| Maize | Belgium | SL 23 | 0.46 | | 200 | | 1 | F |
| Oats | Austria | SL 40 | 1.1-1.6 | | 200-600 | BBCH 31-39 | 1 | 42 |
| | Belgium | SL 72 | 1.4 | | 200-600 | At plant height of 40 cm | 1 | F |
| | | SL 75 | 1.4 | | | | | F |
| | | SL 75 | 1.4 | | 200-600 | At plant height of 40 cm | 1 | F |
| | Denmark | SL 46 | 1.4 | | 200-400 | BBCH 30-37 | 1 | 90 |
| | | SL 46 | 1.8 | | | | | |
| | | SL 75 | 1.1 | | 200-400 | BBCH 30-37 | 1 | 90 |
| | | SL 75 | 1.1 | | | | | |
| | Finland | SL 46 | 1.5 | | | | | F |
| | | SL 75 | 1.5 | | | | | F |
| | Germany | SL 72 | 1.4 | | 200-600 | BBCH 32-49 | 1 | 42 |
| | | SL 72 | 1.4 | | 200-400 | BBCH 32-49 | 1 | 42 |
| | Italy | SL 46 | 1.4 | | | | | |
| | Luxembourg | SL 72 | 1.4 | | 200 | | | F |
| | | SL 75 | 1.4 | | | | 4-5 | |
| | Netherlands | SL 45.7 | 1.4 | | 200-600 | Beginning of shooting until stage 6-7 of Feekes scale (BBCH 30-37) | 1 | F |
| | | SL 40 | 1.2 | | | | 1-2 | F |
| | | SL 75 | 1.4 | | | | 1 | F |
| | UK | SL 64.5 | 1.6 | | | | 1 | F |
| | | SL 72 | 1.6 | | | | 1 | F |

| Crop | Country | Product, % ai | Application | | | | | PHI, days |
|---|---|---|---|---|---|---|---|---|
| | | | Rate, kg ai/ha | Spray conc., kg ai/hl | Water, l/ha | Growth stage | No. | |
| | | SL 75 | 1.7 | | | | 1 | F |
| Peach | Spain | SL 40 | 0.9 | 0.1 | 900<br>900<br>900<br>900 | Flowering<br>May<br>June<br>Post-harvest | 2-3<br>1<br>1<br>1 | |
| Pear | Belgium | SL 72 | 1.4 | 0.24 | 600 | | 4-5 | F |
| | | SL 75 | 1.4 | 0.24 | 600 | | 4-5 | F |
| | Denmark | SL 46 | 1.8 | 0.18 | 1000 | 2 and 4 weeks after flowering | 2 | 42 |
| | | SL 75 | 0.75-1.5 | 0.075-0.15 | 1000 | 3 and 6 weeks after flowering | 2 | 42 |
| | Netherlands[3] | SL 40 | 1.3 | 0.16 | 800 | | 1-2 | 90 |
| | | SL 46 | 1.5 | 0.19 | 800 | | 1-2 | 90 |
| | | SL 45.7 | 0.93-1.8 | 0.094-0.15 | 1000-1200 | 1st treatment at end blossoming, 2nd 2-3 weeks later | 1-2 | 90 |
| | | SL 75 | 1.1-2.3 | 0.11-0.19 | 1000-1200 | | 1-2 | 90 |
| | | SL 75 | 0.75-2.3 | 0.094-0.15 | 800-1500 | | 2 | 90 |
| | Spain | SL 40 | 0.9 | 0.1 | 900<br>900<br>900<br>900 | Flowering<br>May<br>June<br>Post-harvest | 2-3<br>1<br>1<br>1 | |
| Rape | UK | SL 64.5 | 1.9 | | | | 1 | F |
| Rape, winter | Belgium | SL 23 | 0.69 | | 200 | | 1 | F |
| Rye | Austria | SL 40 | 1.5-2.4 | | 200-600 | BBCH 31-32 | 1 | 63 |
| | Belgium | SL 23 | 0.81 | | 200 | | 1 | F |
| | Denmark | SL 46 | 1.8 | | | | | |
| | | SL 46 | 1.2 | | 200-400 | BBCH 30-31 | 1 | 90 |
| | | SL 75 | 0.94 | | 200-400 | BBCH 30-31 | 1 | 90 |
| | | SL 75 | 1.1 | | | | | |
| | Finland | SL 46 | 1.4 | | | | | F |
| | | SL 75 | 2 | | | | 1 | F |
| | Italy | SL 46 | 1.4 | | | | | |
| | Spain | SL 23.6 | 0.59 | | | | | |
| | UK | SL 64.5 | 1.6 | | | | 1 | F |
| | | SL 72 | 1.6 | | | | 1 | F |
| | | SL 75 | 1.7 | | | | 1 | F |
| Rye, winter | Germany | SL 30.5 | 0.61 | | 200-400 | BBCH 32-49 | 1 | 42 |
| | | SL 72 | 1.4 | | 200-600 | BBCH 30-37 | 1 | 63 |
| | Ireland | SL 75 | 1.5-2.3 | | 220 | | 1 | F |
| | Netherlands | SL 36 | 0.32-0.63 | | 200-600 | Beginning of shooting until stage 6-7 of Feekes scale (BBCH 30-32) | 1 | |
| | | SL 30.5 | 0.61 | | | | 1 | F |
| | Sweden | SL 14 | 0.42 | | | | | F |
| Spelt wheat | Belgium | SL 72 | 0.72 | | 200-600 | BBCH 30-32 | 1 | F |
| | | SL 72 | 0.9 | | 200 | | 1 | F |
| | | SL 75 | 0.9 | | | | | F |
| | | SL 75 | 0.9 | | 200-600 | BBCH 30-32 | 1 | F |
| | Luxembourg | SL 72 | 0.9 | | 200 | | | F |
| | | SL 75 | 0.9 | | | | 4-5 | |
| Tomato | Italy | SL 46 | 0.69 | | | | | |
| Triticale | Belgium | SL 23 | 0.69 | | 200 | | 1 | F |
| | | SL 72 | 0.9 | | 200 | | 1 | F |
| | | SL 72 | 0.72 | | 200-600 | BBCH 30-32 | 1 | F |
| | | SL 75 | 0.9 | | | | | F |
| | | SL 75 | 0.75 | | 200-600 | BBCH 30-32 | 1 | |
| | Ireland | SL 75 | 1.9 | | 220 | | 1 | F |
| | Luxembourg | SL 72 | 0.9 | | 200 | | | F |
| | | SL 75 | 0.9 | | | | 4-5 | |
| | UK | SL 64.5 | 1.6 | | | | 1 | F |
| | | SL 72 | 1.6 | | | | 1 | F |
| | | SL 75 | 1.7 | | | | 1 | F |
| Wheat, winter | Austria | SL 40 | 0.19-1.6 | | 200-600 | BBCH 21-30 | 1 | 63 |
| | Belgium | SL 23 | 0.69 | | 200 | | 1 | F |
| | | SL 72 | 0.72 | | 200-600 | BBCH 30-32 | 1 | F |
| | | SL 72 | 0.9 | | 200 | | 1 | F |
| | | SL 75 | 0.9 | | | | | F |
| | | SL 75 | 0.75 | | 200-600 | BBCH 30-32 | 1 | F |
| | Denmark | SL 46 | 1.8 | | | | | |
| | | SL 46 | 0.92 | | 200-400 | BBCH 30-31 | 1 | 90 |

| Crop | Country | Product, % ai | Application | | | | | PHI, days |
|---|---|---|---|---|---|---|---|---|
| | | | Rate, kg ai/ha | Spray conc., kg ai/hl | Water, l/ha | Growth stage | No. | |
| | | SL 75 | 0.75 | | 200-400 | BBCH 30-32 | 1 | 90 |
| | | SL 75 | 1.1 | | | | | |
| | Finland | SL 46 | 1.8 | | | | 1 | F |
| | | SL 75 | 2 | | | | 1 | |
| | France | SL 34.5 | 0.69 | | | | | |
| | | SL 40 | 0.9 | | 200-300 | | 1 | F |
| | | SL 46 | 0.92 | | 200-300 | BBCH 21-30 | 1 | F |
| | | SL 75 | 0.9 | | | | | |
| | Germany | SL 30.5 | 0.76 | | 200-400 | BBCH 32-49 | 1 | 42 |
| | | SL 72 | 1.5 | | 200-600 | BBCH 21-31 | 1 | 63 |
| | Ireland | SL 75 | 1.5 | | 220 | | 1 | F |
| | | SL 75 | 1.1 | | 220 | | 2 | F |
| | Luxembourg | SL 72 | 0.9 | | 200 | | | F |
| | | SL 75 | 0.9 | | | | 4-5 | |
| | Netherlands | SL 36 | 0.63 | | 200-600 | Beginning of shooting until stage 6-7 of Feekes scale (BBCH 30-32) | 1 | |
| | | SL 30.5 | 0.61 | | | | 1 | F |
| | | SL 40 | 0.8 | | | | 1 | F |
| | | SL 45.7 | 0.38-0.92 | | 200-600 | Stage 5 of Feekes scale (BBCH 30) | 1 | F |
| | | SL 75 | 0.38-0.92 | | 200-600 | | 1 | F |
| | | SL 75 | 0.38-0.75 | | 200-600 | | 1 | F |
| | Poland | SL 46 | 0.69-1.2 | | 200-300 | Beginning of shooting (BBCH 30) | 1 | 42 |
| | | SL 46 | 0.92-1.6 | | 200-300 | | 1 | 42 |
| | | SL 67.5 | 1.2-2 | | 200-300 | | 1 | |
| | | SL 72 | 0.86-1.6 | | 200-300 | | 1 | |
| | | SL 75 | 0.90-1.7 | | 200-300 | | 1 | |
| | UK | SC 23 | 0.46 | | | | | |
| | | SC 34.5 | 0.69 | | | | | |
| | | SL 64.5 | 1.6 | | | | 1 | F |
| | | SL 72 | 1.6 | | | | 1 | F |
| | | SL 75 | 1.7 | | | | 1 | F |
| Wheat, summer | Austria | SL 40 | 0.19-1.6 | | 200-600 | BBCH 21-30 | 1 | 63 |
| | Belgium | SL 23 | 0.69 | | 200 | | 1 | F |
| | | SL 72 | 0.72 | | 200 | | 1 | F |
| | | SL 72 | 0.47-0.72 | | 200-600 | BBCH 29-30 | 1 | F |
| | | SL 75 | 0.75 | | | | | F |
| | | SL 75 | 0.45-0.75 | | 200-600 | BBCH 29-30 | 1 | F |
| | Denmark | SL 46 | 1.8 | | | | | |
| | | SL 46 | 0.69 | | 200-400 | BBCH 30-31 | 1 | 90 |
| | | SL 75 | 0.56 | | 200-400 | BBCH 30-31 | 1 | 90 |
| | | SL 75 | 1.1 | | | | | |
| | Finland | SL 46 | 1.5 | | | | 1 | F |
| | | SL 75 | 1.5 | | | | 1 | F |
| | France | SL 40 | 0.9 | | 200-300 | BBCH 21-30 | 1 | F |
| | | SL 46 | 0.92 | | 200-300 | BBCH 21-30 | 1 | F |
| | | SL 75 | 0.9 | | | | | |
| | Germany | SL 30.5 | 0.61 | | 200-400 | BBCH 32-49 | 1 | 42 |
| | | SL 72 | 0.93 | | 200-600 | BBCH 21-29 | 1 | 63 |
| | Ireland | SL 75 | 0.75 | | 220 | | 1 | F |
| | Luxembourg | SL 72 | 0.72 | | 200 | | | F |
| | | SL 75 | 0.75 | | | | 4-5 | |
| | Poland | SL 46 | 0.69 | | 200-300 | Phase of 5 leaves (BBCH 15) | 1 | 42 |
| | | SL 46 | 0.69-0.92 | | 200-300 | Beginning of shooting (BBCH 30) | 1 | |
| | | SL 67.5 | 1.2-1.6 | | 200-300 | | 1 | |
| | | SL 72 | 0.65-0.86 | | 200-300 | | 1 | |
| | | SL 75 | 0.68-0.9 | | 200-300 | | 1 | |
| | Netherlands | SL 30.5 | 0.61 | | | | 1 | F |
| | | SL 36 | 0.63 | | 200-600 | Beginning of shooting until stage 6-7 of Feekes scale (BBCH 30-32) | 1 | |
| | | SL 40 | 0.4 | | | | 1 | F |
| | | SL 45.7 | 0.38-0.46 | | 200-600 | Stage 5 of Feekes scale (BBCH 30) | 1 | F |
| | | SL 75 | 0.38-0.46 | | 200-600 | | 1 | F |
| | | SL 75 | 0.38 | | 200-600 | | 1 | F |
| | UK | SL 64.5 | 0.81-1.6 | | | | 1 | F |
| | | SL 72 | 0.79 | | | | 1 | F |

| Crop | Country | Product, % ai | Application | | | | | PHI, days |
|---|---|---|---|---|---|---|---|---|
| | | | Rate, kg ai/ha | Spray conc., kg ai/hl | Water, l/ha | Growth stage | No. | |
| | | SL 75 | 0.83 | | | | 1 | F |
| Wheat, hard | France | SL 75 | 1.6 | | | | | |
| | Italy | SL 46 | 1.6 | | | | | |
| Wheat, soft | Ireland | SL 46 | 1.9 | | | | | |
| | Italy | SL 30.5 | 0.76 | | | | | |
| | | SL 46 | 1.4 | | | | | |
| | Spain | SL 23.6 | 0.59 | | | | | |
| | | SL 46 | 1.8 | | | | | 28 |

[1]BBCH scale (Bleiholder *et al*., 1997)
[2]F: PHI fixed by approved use (growth stage at treatment)
[3]Information by the government of The Netherlands (Olthof, 2000): GAP for pears will be changed in the near future

## RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received new information on supervised residue trials on pears and cereals as well as on animal feeding studies, and the trials reported in 1994 on which the current MRL recommendations are based were re-evaluated for the estimation of STMRs and HRs. Residue data on pears, cereals (grains, forage and fodder) and rape are summarized in Tables 12-26.

Table 12. Residue trials on pears reported to the 1994 and 2000 JMPRs.
Table 13. Residue trials on cereals in Austria 1965-1970, to 2000 JMPR.
Table 14. Residue trials on summer barley in the UK 1983/84, reported to the 2000 JMPR.
Table 15. Residue trials on summer barley, reported to the 1994 JMPR.
Table 16. Residue trials on winter barley, reported to the 1994 JMPR.
Table 17. Residue trials on oats, reported to the 2000 JMPR.
Table 18. Residue trials on oats, reported to the 1994 JMPR.
Table 19. Residue trials on triticale in the UK 1989, reported to the 2000 JMPR.
Table 20. Residue trials on rye, reported to the 2000 JMPR.
Table 21. Residue trials on rye, reported to the 1994 JMPR.
Table 22. Residue trials on wheat, reported to the 2000 JMPR.
Table 23. Residue trials on summer wheat, reported to the 1994 JMPR.
Table 24. Residue trials on winter wheat, reported to the 1994 JMPR.
Table 25. Residue trials on maize, reported to the 1994 JMPR.
Table 26. Residue trials on rape seed, reported to the 1994 JMPR.

Residue levels and application rates were reported as chlormequat chloride, but the residues are generally recalculated as cation in the Appraisal. When residues were not detected they are shown as below the LOD (e.g. <0.1 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures but for residues approximating the LOD to one significant figure. HRs and STMRs from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are double underlined.

Pears (Table 12). The trials from the Netherlands on which the recommended MRL was based were not correctly evaluated by the 1994 JMPR: each trial included 4 field replicates, so only one figure from each trial should have been selected for the estimation of a maximum residue level. Further supervised trials on pears were carried out in 1998/99 in France.

Table 12. Residues of chlormequat chloride in pears.

| Reference, report no., year, country, location | Crop variety | Application rate per treatment | | | Dates of treatment | Growth stage | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Water l/ha | kg ai/hl | | | | | |
| Wit (1969)<br>CvF/PD 4-6-01, (Tox 16),1968,<br>The Netherlands | Beurre Hardy | 1.5 | | | 6/10/1968 | | 91 | 1.6<br>1.9<br>2.0<br>1.5 | JMPR 1994,<br>4 field replicates, colorimetric method<br>(Mooney and Pasarela, 1967) |
| Wit (1969)<br>CvF/PD 4-0-01<br>(Tox 16), 1968,<br>The Netherlands | Beurre Hardy | 0.74<br>0.74 | | | 6/10/1968<br>7/01/1968 | | 70 | 1.5<br>0.9<br>2.8<br>0.5 | JMPR 1994,<br>4 field replicates, colorimetric method<br>(Mooney and Pasarela, 1967) |
| Wit (1969)<br>CvF/PD 4-6-01<br>(Tox 16), 1968,<br>The Netherlands | Beurre Hardy | 1.5 | | | 5/16/1968 | | 116 | 0.3<br>0.4<br>0.4<br>0.3 | JMPR 1994,<br>4 field replicates, colorimetric method<br>(Mooney and Pasarela, 1967) |
| Wit (1969)<br>CvF/PD 4-6-01<br>(Tox 16), 1968,<br>The Netherlands | Beurre Hardy | 0.74<br>0.74 | | | 5/16/1968<br>6/10/1968 | | 91 | 0.4<br>0.8<br>0.3<br>0.6 | JMPR 1994,<br>4 field replicates, colorimetric method<br>(Mooney and Pasarela, 1967) |
| Wit (1969)<br>CvF/PD 4-6-01<br>(Tox 16), 1968,<br>The Netherlands | Doyenne du Comice | 1.2 | | | 5/08/1968 | | 142 | <0.1<br><0.1<br><0.1<br>0.2 | JMPR 1994,<br>4 field replicates, colorimetric method<br>(Mooney and Pasarela, 1967) |
| Wit (1969)<br>CvF/PD 4-6-01<br>(Tox 16), 1968,<br>The Netherlands | Doyenne du Comice | 1.2 | | | 5/08/1968 | | 142 | 0.3<br><0.1<br><0.1<br>0.6 | JMPR 1994,<br>4 field replicates, colorimetric method<br>(Mooney and Pasarela, 1967) |
| Greve and Hagedoorn (1983)<br>RIVM63760 1109A<br>1980,<br>The Netherlands, Marknesse | Doyenne du Comice | 1.6<br>1.2<br>1.2<br>1.2 | 1500<br>1500<br>1500<br>1500 | 0.11<br>0.08<br>0.08<br>0.08 | last treatment 7/02/1980 | | 90 | 0.94<br>1.6<br>1.3<br>1.5 | JMPR 1994,<br>4 field replicates, head-space GLC<br>(Greve and Hagedoorn, 1983) |
| Greve and Hagedoorn (1983)<br>RIVM63760 1109A<br>1980<br>The Netherlands, Huissen | Doyenne du Comice | 1.6<br>1.2<br>1.2<br>1.2 | 150<br>150<br>150<br>150 | 1.1<br>0.8<br>0.8<br>0.8 | last treatment 6/24.1980 | | 101 | 8.1<br>4.2<br>7.4<br>5.1 | JMPR 1994,<br>4 field replicates, head-space GLC<br>(Greve and Hagedoorn, 1983) |
| Greve and Hagedoorn (1983)<br>RIVM2104000311B<br>1983<br>The Netherlands, Kapelle | Doyenne du Comice | 1.8<br>1.8 | 1000<br>1000 | 0.18<br>0.18 | last treatment 5/24/1983 | | 124 | 3.5<br>5.3<br>3.1<br>2.4 | JMPR 1994,<br>4 field replicates, head-space GLC<br>(Greve and Hagedoorn, 1983) |

| Reference, report no., year, country, location | Crop variety | Application rate per treatment | | | Dates of treatment | Growth stage | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Water l/ha | kg ai/hl | | | | | |
| Greve and Hagedoorn (1983)<br>RIVM2104000311B<br>1983<br>The Netherlands, Geldermalsen | Doyenne du Comice | 1.8<br>1.1 | 1000<br>1000 | 0.18<br>0.11 | last treatment 02.06.83 | | 113 | 6.5<br>5.5<br>5.4<br>6.9 | JMPR 1994,<br>4 field replicates,<br>head-space GLC<br>(Greve and Hagedoorn, 1983) |
| Perny (1999)<br>R 8090 AN1, 1998<br>Northern France<br>67330-Riedheim | Williams | 1.5<br>1.5<br>1.4<br>1.5<br>1.4 | 614<br>613<br>605<br>620<br>606 | 0.24<br>0.24<br>0.24<br>0.24<br>0.24 | 6/04/1998<br>6/18/1998<br>7/02/1998<br>7/18/1998<br>7/31/1998 | 71-72<br>73<br>75<br><br>81 | <br>1<br>13<br>25<br>44 | <br>17<br>14<br>9<br>5.6 | LC-MS<br>(Quirijns and van Dam, 1999; Quirijns, 1999) |
| Perny (1999)<br>R 8090 BM1, 1998<br>Northern France<br>72800-Thoree-les-Pins | Conference | 1.5<br>1.4<br>1.4<br>1.5<br>1.4 | 610<br>595<br>601<br>612<br>603 | 0.24<br>0.24<br>0.24<br>0.24<br>0.24 | 6/12/1998<br>6/22/1998<br>7/02/1998<br>7/13/1998<br>7/29/1998 | 73<br>73-75<br>75<br>77<br>77 | 45 | 4.6 | LC-MS<br>(Quirijns and van Dam, 1999; Quirijns, 1999) |
| Perny (1999)<br>R 8090 BM2, 1998<br>Northern France<br>72800-Thoree-les-Pins | Beurre-Hardy | 1.4<br>1.4<br>1.4<br>1.4<br>1.5 | 606<br>596<br>604<br>605<br>611 | 0.24<br>0.24<br>0.24<br>0.24<br>0.24 | 6/12/1998<br>6/22/1998<br>7/02/1998<br>7/13/1998<br>7/29/1998 | 73<br>73-75<br>75<br>77<br>77 | 45 | 4.0 | LC-MS<br>(Quirijns and van Dam, 1999; Quirijns, 1999) |
| Perny (1999)<br>R 8090 BG1, 1998<br>Belgium<br>4280-Hannut-Bertree | Conference | 1.4<br>1.4<br>1.4<br>1.4<br>1.5 | 590<br>589<br>578<br>604<br>622 | 0.24<br>0.24<br>0.24<br>0.24<br>0.24 | 9/17/1998<br>6/29/1998<br>7/13/1998<br>7/27/1998<br>8/08/1998 | 74<br>74<br>77<br>79<br>79 | 44 | 7.5 | LC-MS<br>(Quirijns and van Dam, 1999; Quirijns, 1999) |
| Perny (2000)<br>R 9067 AN1, 1999<br>Northern France<br>67310-Traenheim | Conference | 1.65 | 658 | 0.25 | 6/10/1999 | 72 | 63 | 3.1 | LC-MS<br>(Quirijns and van Dam, 1999; Quirijns, 1999) |
| Perny (2000)<br>R 9067 AN2, 1999<br>Northern France<br>67330-Riedheim | Williams | 1.5 | 583 | 0.25 | 6/18/1999 | 73-74 | 55 | <0.5 | LC-MS<br>(Quirijns and van Dam, 1999; Quirijns, 1999) |
| Perny (2000)<br>R 9067 BM1, 1999<br>Northern France<br>72800-Thoree les Pins | Doyenne du Comice | 1.3 | 513 | 0.25 | 6/24/1999 | 75 | 63 | 0.57 | LC-MS<br>(Quirijns and van Dam, 1999; Quirijns, 1999) |
| Perny (2000)<br>R 9067 BM2, 1999<br>Northern France<br>72800-Thoree les Pins | Conference | 1.4 | 563 | 0.25 | 6/24/1999 | 75 | 63 | <0.5 | LC-MS<br>(Quirijns and van Dam, 1999; Quirijns, 1999) |

Cereals (Table 13). In 1965 and 1967-1970 numerous residue trials were carried out in Austria to determine chlormequat in cereals (Bayzer, 1966, 1968, 1984). Analysis was by semi-quantitative thin-layer chromatography (Dragendorff reagent) after extraction with ethanol, separation from other quartenary ammonium compounds by ion-exchange chromatography and preparative TLC. The LOD was reported as 0.1 mg/kg, but no validation was carried out. Control samples were not included. The trials were reported to the present Meeting.

Table 13. Residues of chlormequat chloride in cereals in Austria 1965-1970.

| Reference | Year, appl. rate, kg ai/ha | Commodity | No. of samples, residues in mg/kg | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | Total | <0.1 | 0.1 | 0.25 | 0.5 | 1 | 2 | 3-4 | 5-6 | 7-8 | >10 |
| Bayzer, 1966 | 1965, 1-5 | Wheat grain | 127 | 2 | 4 | 16 | 30 | 38 | 36 | | 1 | | |
| | | Wheat flour | 38 | 22 | 8 | 8 | | | | | | | |
| | | Wheat straw | 69 | 13 | | 4 | 10 | 12 | 16 | 10 | 1 | | 3 |
| Bayzer, 1966 | 1965, 6 | Barley grain | 10 | 2 | | 2 | 3 | 3 | | | | | |
| Bayzer, 1966 | 1965, 4-6 | Rye grain | 24 | 3 | | 3 | 6 | 5 | 7 | | | | |
| Bayzer, 1968 | 1967, 0.75-4 | Wheat grain | 287 | 79 | 23 | 69 | 65 | 34 | 15 | 2 | | | |
| | | Wheat flour | 10 | 4 | 3 | 2 | 1 | | | | | | |
| Bayzer, 1968 | 1967, 4-5 | Barley grain | 17 | | | 3 | 4 | 7 | 3 | | | | |
| | | Barley straw | 17 | 13 | | | | | 1 | 1 | | 2 | |
| Bayzer, 1968 | 1967, 2-5 | Oat grain | 16 | | | 2 | 3 | 5 | 6 | | | | |
| Bayzer, 1968 | 1967, 2-5 | Rye grain | 80 | 8 | | 5 | 7 | 14 | 19 | 18 | 7 | 2 | |
| Bayzer, 1984 | 1968, 1-3 | Wheat grain | 131 | 47 | 13 | 30 | 26 | 15 | | | | | |
| Bayzer, 1984 | 1968, 2-4 | Barley grain | 32 | | | | 11 | 14 | 7 | | | | |
| Bayzer, 1984 | 1968, 2-4 | Oat grain | 9 | | | 1 | 1 | | | 7 | | | |
| Bayzer, 1984 | 1968, 2-5 | Rye grain | 96 | 14 | 2 | 11 | 13 | 46 | 7 | 3 | | | |
| Bayzer, 1984 | 1969/70, appl. rate: no information | Oat grain | 92 | | | 5 | 13 | 31 | 28 | 15 | | | |

Barley. Six supervised trials carried out in 1983/84 in the UK were reported to the Meeting. No information on PHIs or analytical methods was reported. The grain and straw samples were harvested at ripening. A further trial was reported from Latvia but no information was included on application rates or analytical methods (Table 14).

The trials reported to the 1994 JMPR which complied with current GAP, and from which the present Meeting estimated a maximum residue level, are shown for summer barley in Table 15 and for winter barley in Table 16.

Table 14. Residues of chlormequat chloride in summer barley reported to the 2000 JMPR.

| Reference, report no., year, country, location | Crop variety | Application | | Sample | PHI, days | Residues, mg/kg | Analytical method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| Summer barley | | | | | | | |
| Ipatova *et al.* (1998), V/15, 1998 Latvia-Riga | | No information (“0.6 l/ha Stabilan”) | | grain<br>straw | 71<br>71 | <0.05<br><0.05 | No information |
| Denes (1991) Hungary | | No information (“3 l /ha Stabilan”) | | grain<br>straw | 61<br>61 | <0.2<br><0.3 | Semi-quantitative TLC. No reference, no validation |
| Lyttle and Baughan (1984), 1167106 UK-Tickencote | Triumph | 0.52<br>1.6 | 13<br>31-32 | grain<br>*control*<br>straw<br>*control* | no information | 3.9<br>*0.72*<br>4.3<br>*4.2* | No information |
| | | 0.52<br>1.6<br>+surfactant | 13<br>31-32 | grain<br>*control* | no information | 2.6<br>*0.72* | No information |
| Lyttle and Baughan (1984), 1167106 UK-Cranwell | Triumph | 0.52<br>1.6 | 13<br>31-32 | grain<br>*control* | no information | 2.0<br>*1.4* | No information |
| | | 0.52<br>1.6<br>+surfactant | 13<br>31-32 | grain<br>*control* | no information | 2.3<br>*1.4* | No information |

| Reference, report no., year, country, location | Crop variety | Application | | Sample | PHI, days | Residues, mg/kg | Analytical method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| Winter barley | | | | | | | |
| Lyttle and Baughan (1983),<br>1167106<br>UK-Casterton | Maris Otter | 0.52<br>0.52<br>1.6 | 13<br>29<br>31-32 | grain<br>*control* | no information | 1.5<br>*0.68* | No information |
| | | 0.52<br>0.52<br>1.6<br>+surfactant | 13<br>29<br>31-32 | grain<br>*control* | no information | 1.4<br>*0.63* | No information |
| Lyttle and Baughan (1983),<br>1167106<br>1983<br>UK-Harringworth | Igri | 0.52<br>0.52<br>1.6 | 13<br>29<br>31-32 | grain<br>*control*<br>straw<br>*control* | no information | 3.8<br>*1.6*<br>2.5<br>*1.7* | No information |
| | | 0.52<br>0.52<br>1.6<br>+surfactant | 13<br>29<br>31-32 | grain<br>*control* | no information | 2.2<br>*1.6* | No information |
| Lyttle and Baughan (1983),<br>1167106<br>1983<br>UK-Epingham | Sonja | 0.52<br>0.52<br>1.6 | 13<br>29<br>31-32 | grain<br>*control* | no information | 1.4<br>*2.0* | No information |
| | | 0.52<br>0.52<br>1.6<br>+surfactant | 13<br>29<br>31-32 | grain<br>*control* | no information | 2.2<br>*2.0* | No information |
| Lyttle and Baughan (1983),<br>1167106<br>1983<br>UK-Yaxley | Igri | 0.52<br>0.52<br>1.6 | 13<br>29<br>31-32 | grain<br>*control*<br>straw<br>*control* | no information | 1.9<br>*1.3*<br>4.3<br>*2.2* | No information |
| | | 0.52<br>0.52<br>1.6<br>+surfactant | 13<br>29<br>31-32 | grain<br>*control* | no information | 1.8<br>*1.3* | No information |

Table 15. Residues of chlormequat chloride in summer barley reported to the 1994 JMPR.

| Report no., Year, Country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 83/10206<br>1983<br>Denmark | 0.46 | forage<br>grain<br>straw | 30<br>59<br>59 | 2.5<br>0.05<br>2.7 | 78/10213<br>1978<br>Sweden | 0.23<br>0.46<br>0.92 | grain | 72<br>72<br>72 | <0.05<br>0.1<br>0.1 |
| 83/10207<br>1983<br>Denmark | 0.46 | forage<br>grain<br>straw | 29<br>70<br>70 | 0.85<br>0.3<br>1.3 | 78/10214<br>1978<br>Sweden | 0.23<br>0.46<br>0.92 | grain | 86<br>86<br>86 | <0.05<br><0.05<br><0.05 |
| 82/10190<br>1982<br>Denmark | 0.61 | grain<br>straw | 61<br>61 | <0.05<br>4.3 | 78/10215<br>1978<br>Sweden | 0.23<br>0.46<br>0.92 | grain | 112<br>112<br>112 | <0.05<br><0.05<br>0.08 |
| 82/10191<br>1982<br>Denmark | 0.61 | grain<br>straw | 77<br>77 | <0.05<br>4.4 | | | | | |
| 82/10207<br>1982<br>Germany | 0.61 | forage<br><br><br><br><br>grain<br>straw | 0<br>21<br>35<br>41<br>48<br>69<br>69 | 10<br>2.1<br>0.96<br>0.55<br>0.36<br>0.17<br>4 | 78/10216<br>1978<br>Sweden | 0.23<br>0.46<br>0.92 | grain | 75<br>75<br>75 | 0.23<br>0.5<br>0.73 |

| Report no., Year, Country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 82/10208<br>1982<br>Germany | 0.61 | forage<br><br>grain<br><br><br>straw | 0<br>20<br>34<br>41<br>48<br>34<br>41<br>48 | 7.6<br>1.5<br>0.46<br>0.5<br>0.62<br>4.4<br>3.9<br>4 | 80/10237<br>1980<br>UK | 1.6 | grain<br>straw | 97<br>97 | 0.37<br>4.9 |
| 78/10210<br>1978<br>Sweden | 0.23<br>0.46<br>0.92 | grain | 82<br>82<br>82 | 0.06<br>0.1<br>0.19 | 80/10238<br>1980<br>UK | 1.6 | straw | 104 | 1.6 |
| 78/10211<br>1978<br>Sweden | 0.23<br>0.46<br>0.92 | grain | 111<br>111<br>111 | <0.05<br><0.05<br><0.05 | 82/10186<br>1982<br>UK | 0.81 | grain | 135 | 0.18 |
| 78/10212<br>1978<br>Sweden | 0.23<br>0.46<br>0.92 | grain | 107<br>107<br>107 | <0.05<br><0.05<br><0.05 | 82/10187<br>1982<br>UK | 1.6 | grain<br>straw | 110<br>110 | 0.24<br>1.6 |

Table 16. Residues of chlormequat chloride in winter barley reported to the 1994 JMPR.

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 82/10213<br>1982<br>Denmark | 0.76 | ear<br>stalk<br>grain<br>straw | 61<br>61<br>69<br>69 | 0.1<br>0.1<br>0.05<br>0.9 | 82/10198<br>1982<br>France | 0.76<br><br><br>0.76 | grain<br>straw<br><br>grain<br>straw | 75<br>75<br><br>63<br>63 | 0.24<br>4.7<br><br>0.35<br>5.4 |
| 82/10195<br>1982<br>France | 0.76<br><br><br>0.76 | grain<br>straw<br><br>grain<br>straw | 69<br>69<br><br>56<br>56 | 0.18<br>1.8<br><br>0.16<br>11 | 83/10210<br>1983<br>France | 0.76 | grain<br>straw | 56<br>56 | 0.3<br>4.4 |
| 82/10196<br>1982<br>France | 0.76<br><br><br>0.76 | grain<br>straw<br><br>grain<br>straw | 70<br>70<br><br>57<br>57 | <0.05<br>3.1<br><br><0.05<br>8.5 | 83/10211<br>1983<br>France | 0.76 | grain<br>straw | 68<br>68 | 0.29<br>5.5 |
| 82/10197<br>1982<br>France | 0.76<br><br><br>0.76 | grain<br>straw<br><br>grain<br>straw | 77<br>77<br><br>62<br>62 | <0.05<br>0.36<br><br>0.21<br>2.4 | 83/10212<br>1983<br>France | 0.76 | grain<br>straw | 67<br>67 | 0.3<br>2.8 |
| 82/10205<br>1982<br>Germany | 0.76 | forage<br><br>grain<br><br><br>straw | 0<br>21<br>35<br>42<br>49<br>35<br>42<br>49 | 8.3<br>4.3<br>1.1<br>1.5<br>1.6<br>7.8<br>6.4<br>5.8 | 82/10206<br>1982<br>Germany | 0.76 | forage<br><br>grain<br><br><br>straw | 0<br>21<br>35<br>42<br>49<br>35<br>42<br>49 | 9.9<br>3<br>1.6<br>1.5<br>1.6<br>4.1<br>3.5<br>5.8 |
| 83/10201<br>1983<br>Germany | 0.76 | forage<br>ear<br><br>stalk<br><br><br>grain<br>straw | 0<br>21<br>35<br>21<br>35<br>42<br>49<br>49 | 9<br>7.3<br>6.5<br>7.7<br>8.8<br>12<br>2.3<br>12 | 83/10202<br>1983<br>Germany | 0.76 | forage<br><br><br><br>grain<br><br>straw | 0<br>20<br>33<br>53<br>68<br>76<br>86<br>76 | 6.4<br>1.3<br>0.89<br>1.9<br>0.18<br>0.2<br>6.2<br>3 |

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|
| 83/10203<br>1983<br>Germany | 0.76 | forage<br><br>grain<br><br>straw | 0<br>20<br>35<br>43<br>53<br>43 | 0.76<br>3.3<br>1<br>1.3<br>7.3<br>8.7 |
| 83/10205<br>1983<br>Germany | 0.76 | forage<br>ear<br><br><br>stalk<br><br><br>grain<br>straw | 0<br>21<br>35<br>42<br>21<br>35<br>42<br>49<br>49 | 10<br>7.7<br>4.7<br>2.5<br>4.9<br>7.8<br>11<br>2.1<br>9 |
| 83/10195<br>1983<br>Sweden | 0.61 | grain | 68 | 0.07<br>0.13<br>0.32<br>0.42 |
| 80/10236<br>1980<br>UK | 1.6 | grain<br>straw | 80<br>80 | 0.15<br>1 |
| 82/10189<br>1982<br>UK | 1.6 | forage<br>grain<br>straw | 51<br>115<br>115 | 0.41<br><0.05<br>2.2 |
| 83/10185<br>1983<br>UK | 0.48+1.6 | grain<br>straw | 98<br>98 | 0.05<br>8.9 |
| 84/10226<br>1984<br>UK | 0.48+1.6 | grain<br>straw | 113<br>113 | <0.05<br>2.4 |
| 83/10204<br>1983<br>Germany | 0.76 | forage<br><br><br>ear<br><br>stalk<br><br>grain<br>straw | 0<br>21<br>35<br>43<br>49<br>43<br>49<br>76<br>76 | 7.3<br>2<br>2.2<br>0.54<br>0.78<br>2.9<br>2.6<br>0.17<br>5.8 |
| 84/10231<br>1984<br>Switzerland | 0.61 | grain<br>straw | 72<br>72 | 0.23<br>4.5 |
| 84/10232<br>1984<br>Switzerland | 0.61 | grain<br>straw | 70<br>70 | 0.29<br>4.2 |
| 82/10188<br>1982<br>UK | 1.6 | forage<br>grain<br>straw | 40<br>96<br>96 | 0.97<br>0.07<br>1.1 |
| 87/10378-<br>10380<br>1987<br>UK | 1.9 | forage<br>grain<br><br>straw | 0<br>128<br><br>128 | 17,19,24<br>0.16,0.15,<br>0.36<br>1.7,2.1,2.4 |
| 83/10186<br>1983<br>UK | 0.48+1.6 | grain<br>straw | 31<br>31 | 0.24<br>0.98 |
| 87/10366<br>1987<br>UK | 0.46 | forage<br>grain<br><br>straw | 5<br>82<br><br>82 | 2, 3.3, 4.8<br>0.45, 0.5,<br>0.58<br>10,11,12 |
| 87/10366<br>1987<br>UK | 0.46 | forage<br>grain<br>straw | 0<br>75<br>75 | 9.2<br>0.43<br>16 |

Oats. The trials reported to the 2000 JMPR are shown in Table 17. High values were found in untreated control plots in four trials in Austria (1992). Semi-quantitative thin-layer chromatographic or colorimetric methods were used to analyse the samples from one trial in Germany and one in the UK which could be evaluated.

The results of trials reported to the 1994 JMPR which were used by the present Meeting for the estimation of maximum residue levels for grain, straw and forage are shown in Table 18.

Table 17. Residues of chlormequat chloride in oats reported to the 2000 JMPR.

| Reference, report no., year, country, location | Crop Variety | Application | | Sample | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| Pfarl (1993a), R92-14 / 1162, 1992 Austria-Seitenstetten | Lord | 1.4 | 39 | whole plant<br>*control* | 0 | 13.9<br>*1.6* | product: Stabilan 460 |
| | | | | plant without ear<br>*control* | 17 | 2.9<br>*2.0* | head-space GLC (Schneider, 1993) |
| | | | | ear<br>*control* | 17 | 3.8<br>*4.6* | |

| Reference, report no., year, country, location | Crop Variety | Application | | Sample | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| | | | | grain<br>*control* | 51 | 0.53<br>*0.85* | LOD 1 mg/kg |
| | | | | straw<br>*control* | 51 | 1.3<br>*0.54* | |
| Pfarl (1993a),<br>R92-14 / 1162,<br>1992<br>Austria –<br>Leonding | Lorenz | 1.4 | 39 | whole plant<br>*control* | 0 | 11.5<br>*3.4* | product:<br>Stabilan 460 |
| | | | | plant without ear<br>*control* | 13 | 3.8<br>*1.1* | head-space GLC<br>(Schneider, 1993) |
| | | | | ear<br>*control* | 13 | 3.9<br>*5.4* | |
| | | | | grain<br>*control* | 49 | 1.0<br>*0.23* | LOD 1 mg/kg |
| | | | | straw<br>*control* | 49 | 1.5<br>*0.70* | |
| Pfarl (1993b)<br>R92-15 / 1163,<br>1992<br>Austria-<br>Seitenstetten | Lord | 1.4 | 39 | whole plant<br>*control* | 0 | 10<br>*2.3* | product:<br>Stabilan 720 |
| | | | | plant without ear<br>*control* | 17 | 3.0<br>*1.9* | head-space GLC<br>(Schneider, 1993)<br>LOD 1 mg/kg |
| | | | | ear<br>*control* | 17 | 5.6<br>*3.9* | |
| | | | | grain<br>*control* | 51 | 0.80<br>*0.77* | |
| | | | | straw<br>*control* | 51 | 1.5<br>*0.61* | |
| Pfarl (1993b),<br>R92-15 /1163,<br>1992<br>Austria –<br>Leonding | Lorenz | 1.4 | 39 | whole plant<br>*control* | 0 | 13<br>*1.0* | product:<br>Stabilan 720 |
| | | | | plant without ear<br>*control* | 13 | 4.1<br>*1.6* | head-space GLC<br>(Schneider, 1993) |
| | | | | ear<br>*control* | 13 | 4.95<br>*4.1* | |
| | | | | grain<br>*control* | 49 | 1.4<br>*0.25* | LOD 1 mg/kg |
| | | | | straw<br>*control* | 49 | 1.5<br>*0.46* | |
| Brüggemann and Ocker (1988),<br>D 87/88-912, 1986<br>Germany-<br>München (Puch) | Fabian | 1.4 | 37 | whole plant | 0<br>30<br>42<br>50 | 226<br>28<br>22<br>18 | semi-quantitative TLC<br>(Brüggemann and Ocker, 1986) |
| | | | | grain<br>*control* | 74 | 3.0<br>0.3 | |
| | | | | straw | 74 | 0.7 | |
| Bayzer (1979a)<br>AE/Ni/Kl 1979 01 11, 1978,<br>UK-<br>Brant Broughton,<br>Nottinghamshire | Maris Quest | 1.6 | 32 | grain<br><br><br>straw | 65<br><br><br>65 | 0.8<br>0.8<br>0.5<br>2.0<br>3.0<br>3.0 | 3 replicates,<br>semi-quantitative colorimetric or TLC<br>(no detailed information) |

Table 18. Residues of chlormequat chloride in oats reported to the 1994 JMPR.

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 74/10197<br>1974<br>Germany | 1.2 | forage<br><br><br><br>grain<br>straw | 0<br>21<br>43<br>63<br>81<br>81 | 116<br>15<br>9.2<br>4.8<br>2.4<br>8.2 | 74/10198<br>1974<br>Germany | 1.2 | forage<br><br><br>grain<br>straw | 0<br>21<br>49<br>74<br>74 | 100<br>17<br>1.8<br>1.5<br>4.0 |
| 73/10129<br>1973<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>23<br>44<br>49<br>49 | 84<br>8.1<br>6.8<br>3.7<br>5.2 | 73/10130<br>1973<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 24<br>48<br>59<br>70<br>70 | 15<br>4.0<br>3.9<br>3.3<br>1.2 |
| 75/10184<br>1975<br>Germany | 1.2 | forage<br><br><br>grain<br>straw | 0<br>21<br>42<br>63<br>63 | 17<br>3.7<br>2.5<br>0.14<br>0.9 | 75/10185<br>1975<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>21<br>32<br>51<br>51 | 17<br>7.6<br>3.3<br>1.6<br>2.2 |
| 75/10186<br>1975<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>21<br>42<br>55<br>55 | 17<br>6.4<br>5.1<br>1.9<br>1.9 | 76/10144<br>1976<br>Germany | 1.4 | grain<br>straw | 59<br>59 | 1.8<br>1.2 |
| 78/10209<br>1978<br>Germany | 1.4 | forage<br><br><br><br>grain<br>straw | 0<br>21<br>42<br>54<br>75<br>75 | 9.9<br>3.5<br>2.3<br>3.2<br>2.4<br>1.9 | 76/10155<br>1976<br>Germany | 1.4 | forage<br><br>grain<br><br><br>straw | 0<br>21<br>42<br>57<br>63<br>42<br>57<br>63 | 11<br>1.5<br>0.05<br>1.0<br>1.1<br>1.6<br>1.6<br>1.6 |
| 76/10156<br>1976<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>21<br>42<br>73<br>60<br>73 | 20<br>1.8<br>0.36<br>0.45<br>1.3<br>0.78 | 76/10157<br>1976<br>Germany | 1.4 | forage<br><br>grain<br><br>straw | 0<br>22<br>42<br>44<br>42<br>44<br>62 | 14<br>6.9<br>1.2<br>1.5<br>12<br>9.6<br>5.3 |
| 76/10158<br>1976<br>Germany | 1.4 | forage<br><br>grain<br><br>straw | 0<br>19<br>82<br>89<br>82<br>89 | 19<br>4.3<br>2.0<br>1.9<br>4.8<br><0.1 | 80/10244<br>1980<br>Germany | 1.4 | forage<br><br>grain<br><br>straw | 0<br>32<br>82<br>91<br>82<br>91 | 12<br>2.5<br>0.86<br>1.2<br>9.9<br>3.0 |
| 80/10245<br>1980<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>21<br>30<br>91<br>91<br>91 | 6.3<br>0.69<br>1.1<br>0.09<br>0.79<br>3.0 | 80/10246<br>1980<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>21<br>50<br>57<br>57 | 3.8<br>2.9<br>2.0<br>0.51<br>9.9 |
| 80/10247<br>1980<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>20<br>42<br>62<br>62 | 6.7<br>3.1<br>2.1<br>0.9<br>6.3 | 80/10248<br>1980<br>Germany | 1.4 | forage<br><br>grain<br><br>straw | 0<br>21<br>70<br>73<br>70<br>73 | 3.8<br>1.3<br>1.7<br>1.2<br>9.9<br>8.1 |
| 74/10199<br>1974<br>UK | 1.7 | grain<br>straw | 51<br>51 | 9.2<br>25 | 76/10159<br>1976<br>UK | 1.7 | grain<br>straw | 34<br>34 | 0.63<br>0.48 |

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 77/10248<br>1977<br>UK | 1.7 | forage<br><br>grain<br>straw | 27<br>58<br>94<br>94 | 4.7<br>1.6<br>0.1<br>3.3 | | | | | |

Rye and triticale (Tables 19, 20). Four trials on triticale and two on winter rye were carried out by Byast and Tolhurst (1990). No chlormequat residues were found in any of the untreated control samples or in the treated grain. Two trials on rye were carried out in Austria in 1992 by Pfarl (1993a,b) and analysed by Schneider (1993) as described above.

The residue data reported to the 1994 JMPR which the present Meeting re-evaluated for the estimation of maximum residue levels are shown in Table 21.

Table 19. Residues of chlormequat chloride in triticale reported to the 2000 JMPR.

| Reference, report no., year, country, location | Crop variety | Application | | Sample | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| Byast and Tolhurst (1990), 52287, 1989<br>UK-Brighton | Salvo | 2.5 | 31-32 | grain<br>straw | 95<br>95 | <0.1(3)<br><0.1(3) | GLC after thiophenolate derivatization<br>(Byast and Tolhurst, 1990) |
| Byast and Tolhurst (1990), 52287, 1989<br>UK-Brighton | Salvo | 5.0 | 31-32 | grain<br>straw | 95<br>95 | <0.1(3)<br><0.1(3) | GLC after thiophenolate derivatization<br>(Byast and Tolhurst, 1990) |
| Byast and Tolhurst (1990), 52287, 1989<br>UK –Stockbridge | Lasko | 2.5 | 31-32 | grain<br>straw | 76<br>76 | <0.1(3)<br>0.24<br>0.74<br>0.59 | GLC after thiophenolate derivatization<br>(Byast and Tolhurst, 1990) |
| Byast and Tolhurst (1990), 52287, 1989<br>UK –Stockbridge | Lasko | 5.0 | 31-32 | grain<br>straw | 76<br>76 | <0.1(3)<br>0.66<br>0.13<br>0.23 | GLC after thiophenolate derivatization<br>(Byast and Tolhurst, 1990) |

Table 20. Residues of chlormequat chloride in rye reported to the 2000 JMPR.

| Reference, report no., year, country, location | Crop variety | Application | | Sample | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| Pfarl (1993a),<br>R92-14 / 1162,<br>1992<br>Austria-Linz | Eho-Kurz | 1.15 | 32 | whole plant<br>*control* | 0 | 11<br>*2.2* | product:<br>Stabilan 460 |
| | | | | plant without ear<br><br>*control* | 14<br>29<br>*14*<br>*29* | 2.4<br>2.1<br>*1.4*<br>*2.6* | head-space GLC (Schneider, 1993) |
| | | | | ear<br><br>*control* | 14<br>29<br>*14*<br>*29* | 6.1<br>5.8<br>*6.0*<br>*3.9* | |
| | | | | grain<br>*control* | 79 | 0.81<br>*0.3* | LOD 1 mg/kg |
| | | | | straw<br>*control* | 79 | 1.6<br>*0.64* | |

| Reference, report no., year, country, location | Crop variety | Application | | Sample | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| Pfarl (1993b), R92-15 /1163, 1992 Austria-Linz | Eho-Kurz | 2.16 | 32 | whole plant<br>*control* | 0 | 11.5<br>*3.4* | product: Stabilan 720 |
| | | | | plant without ear<br><br>*control* | 14<br>29<br>*14*<br>*29* | 4.3<br>2.1<br>*1.3*<br>*1.7* | head-space GLC (Schneider, 1993) |
| | | | | ear<br><br>*control* | 14<br>29<br>*14*<br>*29* | 14.7<br>6.5<br>*7.5*<br>*6.2* | |
| | | | | grain<br>*control* | 79 | 1.7<br>*0.43* | LOD 1 mg/kg |
| | | | | straw<br>*control* | 79 | 2.1<br>*0.73* | |
| Byast and Tolhurst (1990) 52287, 1989 UK-Elvedon | | 2.3 | 30 | grain<br>straw | 107<br>107 | <0.1<br><0.1 | GLC after thiophenolate derivatization (Byast and Tolhurst, 1990) |
| Byast and Tolhurst (1990) 52287, 1989 UK-Elvedon | | 4.6 | 30 | grain<br>straw | 107<br>107 | <0.1<br><0.1 | GLC after thiophenolate derivatization (Byast and Tolhurst, 1990) |
| Byast and Tolhurst (1990) 52287, 1989 UK-Isle of Wight | | 2.3 | 30 | grain<br>straw | 107<br>107 | <0.1<br><0.1 | GLC after thiophenolate derivatization (Byast and Tolhurst, 1990) |
| Byast and Tolhurst (1990) 52287, 1989 UK-Isle of Wight | | 4.6 | 30 | grain<br>straw | 107<br>107 | <0.1<br><0.1 | GLC after thiophenolate derivatization (Byast and Tolhurst, 1990) |

Table 21. Residues of chlormequat chloride in rye reported to the 1994 JMPR.

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| Summer rye | | | | | | | | | |
| 77/10249<br>1977<br>Germany | 1.1 | forage<br><br><br><br>grain<br>straw | 0<br>19<br>29<br>48<br>70<br>70 | 14<br>12<br>0.5<br>0.1<br>0.06<br>0.3 | 77/10250<br>1977<br>Germany | 1.1 | forage<br><br><br>grain<br>straw | 0<br>17<br>38<br>69<br>46<br>59<br>69 | 24<br>13<br>8.8<br>2.1<br>18<br><0.1<br>0.2 |
| 77/10251<br>1977<br>Germany | 1.1 | forage<br><br><br>grain<br>straw | 0<br>21<br>42<br>63<br>63 | 11<br>12<br>5.6<br>2.6<br>9 | 77/10252<br>1977<br>Germany | 1.1 | forage<br><br><br>grain<br>straw | 0<br>22<br>43<br>64<br>92<br>85<br>92 | 13<br>9.7<br>11<br>9.4<br>1.5<br>3.1<br>4.7 |

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| Winter rye | | | | | | | | | |
| 74/10195<br>1974<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>55<br>83<br>122<br>122 | 468<br>20<br>9.7<br>0.24<br>4.8 | 74/10194<br>1974<br>Germany | 1.4 | forage<br><br><br><br>grain<br>straw | 0<br>27<br>53<br>81<br>123<br>123 | 193<br>18<br>9<br>4.4<br>0.22<br>2.8 |
| 74/10196<br>1974<br>Germany | 1.4 | forage<br><br><br><br>grain<br>straw | 0<br>28<br>57<br>84<br>117<br>117 | 264<br>9<br>3<br>1<br>0.3<br>3.1 | 75/10188<br>1975<br>Germany | 1.4 | forage<br><br><br><br>grain<br>straw | 0<br>29<br>56<br>84<br>99<br>99 | 25<br>2.1<br>1.5<br>1.3<br>0.3<br>4.3 |
| 75/10187<br>1975<br>Germany | 1.4 | forage<br><br><br><br><br>grain<br>straw | 0<br>28<br>58<br>83<br>98<br>105<br>105 | 52<br>2.2<br>1.1<br>1.4<br>1.3<br>0.34<br>2.2 | 75/10189<br>1975<br>Germany | 1.4 | forage<br><br><br><br>grain<br>straw | 0<br>28<br>56<br>85<br>92<br>92 | 13<br>4.1<br>1.2<br>3.5<br>0.33<br>5.7 |
| 75/10190<br>1975<br>Germany | 1.4 | forage<br><br><br><br>grain<br>straw | 0<br>28<br>56<br>85<br>92<br>92 | 39<br>1.9<br>0.73<br>2.3<br><0.05<br>2.7 | 75/10197<br>1975<br>Germany | 1.4 | forage<br><br><br><br>grain<br>straw | 0<br>28<br>56<br>84<br>96<br>93<br>96 | 26<br>4.9<br>3.4<br>1.5<br>0.62<br>6.9<br>5.2 |
| 75/10198<br>1975<br>Germany | 1.4 | forage<br><br><br>grain<br>straw | 0<br>28<br>56<br>85<br>84<br>85 | 19<br>5.9<br>0.13<br>1.2<br>6.6<br>9.6 | 76/10152<br>1976<br>Germany | 1.4 | forage<br><br><br>grain<br><br><br>straw | 3<br>32<br>59<br>66<br>84<br>91<br>66<br>91 | 24<br>4.3<br>1.2<br>0.26<br>0.36<br>0.45<br>2.9<br>4.5 |
| 76/10153<br>1976<br>Germany | 1.4 | forage<br><br><br><br>grain<br>straw | 0<br>28<br>52<br>58<br>77<br>77<br>85 | 3.1<br>28<br>12<br>0.92<br>1.9<br>16<br>9.6 | 76/10154<br>1976<br>Germany | 1.4 | forage<br><br>grain<br><br>straw | 0<br>29<br>56<br>67<br>56<br>67 | 24<br>17<br>2.0<br>1.4<br>18<br>12 |
| 82/10203<br>1982<br>Germany | 0.61 | forage<br><br><br><br><br>grain<br>straw | 0<br>21<br>35<br>42<br>49<br>75<br>75 | 7.3<br>3.6<br>2.9<br>2.8<br>1.8<br>0.43<br>5.5 | 82/10204<br>1982<br>Germany | 0.61 | forage<br><br>grain<br><br><br>straw | 0<br>20<br>34<br>41<br>48<br>34<br>41<br>48 | 8.4<br>4.2<br>1.8<br>1.1<br>1.1<br>7.5<br>4.5<br>2.8 |
| 82/10193<br>1982<br>Sweden | 0.46 | grain | 77 | 0.09 | 82/10192<br>1982<br>Sweden | 0.61 | grain | 85 | <0.05 |
| 83/10191<br>1983<br>Sweden | 0.61 | grain | 80 | 0.09 | 83/10197<br>1983<br>Sweden | 0.61 | grain | 77 | 0.07 |
| 83/10193<br>1983<br>Sweden | 0.61 | grain | 86 | 0.08 | 83/10194<br>1983<br>Sweden | 0.61 | grain | 97 | 0.05 |
| 76/10149<br>1976<br>UK | 1.6 | grain<br>straw | 92<br>92 | 0.88<br>12 | 76/10150<br>1976<br>UK | 1.6 | grain<br>straw | 113<br>113 | 0.45<br>0.48 |

Wheat. Data from trials in Austria (2), Germany (3) and the UK (8) were reported to the Meeting (Table 22). High chlormequat values in the untreated control plots were found in the two trials in Austria (1992). Semi-quantitative thin-layer chromatographic or colorimetric methods were used for analysis in the trials in Germany and the UK. A further trial was reported in Latvia but no information on application rates or analytical methods was included.

The residue data reported to the 1994 JMPR which complied with current GAP and which the present Meeting used to estimate maximum residue levels are shown in Tables 23 and 24.

Table 22. Residues of chlormequat chloride in wheat reported to the 2000 JMPR.

| Reference, report no., year, country, location | Crop variety | Application | | Sample | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| Winter wheat | | | | | | | |
| Pfarl (1993a), R92-14 / 1162, 1992 Austria-Ansfelden | Ikarus | 1.4 | 32 | whole plant<br>*control* | 0<br>14<br>*0*<br>*14* | 23<br>3.4<br>*2.9*<br>*1.6* | product: Stabilan 460<br>head-space GLC (Schneider, 1993) |
| | | | | plant without ear<br>*control* | 29 | 3.5<br>*1.4* | |
| | | | | ear<br>*control* | 29 | 3.4<br>*3.9* | |
| | | | | grain<br>*control* | 79 | 0.34<br>*0.27* | LOD 1 mg/kg |
| | | | | straw<br>*control* | 79 | 1.2<br>*0.67* | |
| Pfarl (1993b), R92-15 /1163, 1992 Austria-Ansfelden | Ikarus | 1.4 | 32 | whole plant<br>*control* | 0<br>14<br>*0*<br>*14* | 24<br>4.9<br>*3.5*<br>*2.6* | product: Stabilan 720<br>head-space GLC (Schneider, 1993) |
| | | | | plant without ear<br>*control* | 29 | 2.3<br>*1.1* | |
| | | | | ear<br>*control* | 29 | 6.3<br>*5.0* | |
| | | | | grain<br>*control* | 79 | 0.41<br>*0.33* | LOD 1 mg/kg |
| | | | | straw<br>*control* | 79 | 1.2<br>*0.34* | |
| Brüggemann and Ocker (1988), UCB/D87/88-116/3, 1985 Germany-Bonn (Kessenich) | Kanzler | 1.4 | 25-29 | plant | 0<br>29<br>49<br>63 | 100<br>1.0<br>0.8<br>0.3 | semi-quantitative TLC (Brüggemann and Ocker, 1986) |
| | | | | grain<br>*control* | 94 | 0.24<br>*0.07* | |
| | | | | straw | 94 | 0.9 | |
| Brüggemann and Ocker (1988), D 87/88-03775, 1986 Germany-Hannover (Pattensen) | Kanzler | 1.4 | 22-25 | plant | 0<br>31<br>52<br>65 | 134<br>8.6<br>1.7<br>1.4 | semi-quantitative TLC (Brüggemann and Ocker, 1986) |
| | | | | grain<br>*control* | 129 | 0.2<br>*0.06* | |
| | | | | straw | 129 | 0.5 | |

| Reference, report no., year, country, location | Crop variety | Application | | Sample | PHI, days | Residues, mg/kg | Remarks, method |
|---|---|---|---|---|---|---|---|
| | | kg ai/ha | Growth stage, BBCH | | | | |
| Brüggemann and Ocker (1988), D 87/88-03788, 1986 Germany-Kiel (Rabendorf) | Kanzler | 1.4 | | grain<br>*control* | 110 | 0.2<br>*0.2* | semi-quantitative TLC (Brüggemann and Ocker, 1986) |
| Bayzer (1979a) AE/Ni/Kl 1979 01 11, 1978, UK-Farcett-Fen Huntingdonshire | Flanders | 1.6 | 6-7 leaves (BBCH 17) | grain | 112 | <0.1<br><0.1<br><0.1 | 3 replicates, semi-quantitative colorimetric or TLC (no detailed information) |
| | | | | straw | 112 | 1.0<br>1.0<br>1.5 | |
| Bayzer (1979a) AE/Ni/Kl 1979 01 11, 1978, UK-Winkburn Nottinghamshire | Maris Huntsman | 1.6 | 7-8 leaves (BBCH 18) | grain | 105 | 0.5<br>0.3<br>0.5 | 3 replicates, semi-quantitative colorimetric or TLC (no detailed information) |
| | | | | straw | 105 | 2.0<br>2.0<br>2.0 | |
| Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Newark Nottinghamshire | M. Huntsman | 1.7 | | grain<br>straw | 100<br>100 | <0.1<br>1.0 | semi-quantitative colorimetric or TLC (no detailed information) |
| Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Newark Nottinghamshire | M. Huntsman | 3.4 | | grain<br>straw | 100<br>100 | <0.1<br>2.0 | semi-quantitative colorimetric or TLC (no detailed information) |
| Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Barton Nottinghamshire | Sports-man | 3.6 | | grain<br>straw | 91<br>91 | <0.1<br>2 | semi-quantitative colorimetric or TLC (no detailed information) |
| Summer wheat | | | | | | | |
| Ipatova et al. (1998), V/15, 1998 Latvia-Riga | | no information (“0.5 l/ha Stabilan”) | | grain<br>straw | 99<br>99 | <0.05<br><0.05 | No information |
| Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Isleham Cambridgeshire | M. Dove | 0.84 | | grain<br>straw | 87<br>87 | 0.1<br>0.5 | semi-quantitative colorimetric or TLC (no detailed information) |
| Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Isleham Cambridgeshire | M. Dove | 1.7 | | grain<br>straw | 87<br>87 | 0.5<br>1.0 | semi-quantitative colorimetric or TLC (no detailed information) |
| Bayzer (1979a) AE/Ni/Kl 1979 01 11, 1978, UK-Barnby Nottinghamshire | Sappo | 0.8 | 7-8 leaves (BBCH 18) | grain | 95 | <0.1<br><0.1<br><0.1 | 3 replicates, semi-quantitative colorimetric or TLC (no detailed information) |
| | | | | straw | 95 | 1.5<br>1.5<br>1.5 | |

Table 23. Residues of chlormequat chloride in summer wheat reported to the 1994 JMPR.

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 79/10190 | 1.4 | forage | 0 | 15 | 79/10192 | 1.4 | forage | 0 | 6.1 |
| 1979 | | | 21 | 10 | 1979 | | | 20 | 3.9 |
| Germany | | | 35 | 8.9 | Germany | | | 36 | 5.4 |
| | | | 48 | 5 | | | | 55 | 3.3 |
| | | grain | 62 | 1.3 | | | grain | 86 | 0.32 |
| | | | 70 | 1.3 | | | straw | 86 | 13 |
| | | straw | 62 | 29 | | | | | |
| | | | 70 | 9.4 | | | | | |
| 79/10194 | 1.4 | forage | 0 | 16 | 79/10198 | 1.4 | forage | 0 | 7.5 |
| 1979 | | | 21 | 0.18 | 1979 | | | 21 | 7.3 |
| Germany | | | 42 | 1.6 | Germany | | | 42 | 5.3 |
| | | | 63 | 1.2 | | | | 63 | 6.7 |
| | | grain | 70 | 0.34 | | | grain | 71 | 1.2 |
| | | | 77 | 0.59 | | | straw | 71 | 17 |
| | | straw | 70 | 10 | | | | | |
| | | | 77 | 4.4 | | | | | |
| 79/10200 | 1.4 | forage | 0 | 11 | 79/10202 | 1.4 | forage | 0 | 11 |
| 1979 | | | 21 | 6.7 | 1979 | | | 20 | 5.5 |
| Germany | | | 35 | 4 | Germany | | | 36 | 3.2 |
| | | | 48 | 8.2 | | | | 55 | 5.3 |
| | | grain | 62 | 1.1 | | | grain | 86 | 0.09 |
| | | | 70 | 1.5 | | | straw | 86 | 17 |
| | | straw | 62 | 21 | | | | | |
| | | | 70 | 13 | | | | | |
| 79/10204 | 1.4 | forage | 0 | 9.7 | 79/10208 | 1.4 | forage | 0 | 8.9 |
| 1979 | | | 21 | 1.3 | 1979 | | | 21 | 9.8 |
| Germany | | | 42 | 1.7 | Germany | | | 42 | 5.1 |
| | | | 63 | 1.1 | | | | 63 | 6.5 |
| | | grain | 70 | 0.62 | | | grain | 71 | 1.3 |
| | | | 77 | 0.68 | | | straw | 71 | 18 |
| | | straw | 70 | 13 | | | | | |
| | | | 77 | 1.6 | | | | | |
| 80/10220 | 1.6 | forage | 0 | 1.2 | 80/10222 | 1.6 | forage | 0 | 1.4 |
| 1980 | | | 22 | 6 | 1980 | | | 21 | 0.95 |
| Germany | | | 43 | 7.8 | Germany | | | 57 | 0.54 |
| | | | 64 | 0.31 | | | grain | 71 | 0.52 |
| | | grain | 64 | 0.31 | | | straw | 71 | 14 |
| | | | 71 | 0.31 | | | | | |
| | | | 85 | 0.31 | | | | | |
| | | straw | 64 | 15 | | | | | |
| | | | 71 | 16 | | | | | |
| | | | 85 | 18 | | | | | |
| 80/10224 | 1.6 | forage | 0 | 9 | 80/10226 | 1.6 | forage | 0 | 10 |
| 1980 | | | 20 | 1.6 | 1980 | | | 21 | 2.9 |
| Germany | | | 42 | 0.85 | Germany | | grain | 83 | 0.25 |
| | | grain | 74 | 0.41 | | | | 87 | 0.33 |
| | | | 92 | 0.40 | | | straw | 83 | 7 |
| | | straw | 74 | 5.2 | | | | 87 | 4.6 |
| | | | 92 | 7 | | | | | |
| 80/10228 | 1.6 | forage | 0 | 8.2 | 80/10239 | 1.7 | forage | 0 | 7.3 |
| 1980 | | | 21 | 4.2 | 1980 | | | 22 | 8.5 |
| Germany | | grain | 83 | 0.30 | Germany | | | 43 | 6.3 |
| | | | 87 | 0.48 | | | grain | 64 | 0.31 |
| | | straw | 83 | 15 | | | | 71 | 0.33 |
| | | | 87 | 13 | | | | 85 | 0.39 |
| | | | | | | | straw | 64 | 20 |
| | | | | | | | | 71 | 13 |
| | | | | | | | | 85 | 18 |

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 80/10240<br>1980<br>Germany | 1.7 | forage<br><br><br>grain<br><br>straw | 0<br>20<br>42<br>74<br>92<br>74<br>92 | 9.7<br>3.6<br>1.1<br>0.56<br>0.59<br>11<br>7.3 | 80/10241<br>1980<br>Germany | 1.7 | forage<br><br>grain<br><br>straw | 0<br>21<br>83<br>87<br>83<br>87 | 12<br>3.6<br>0.44<br>0.39<br>5.8<br>4.5 |
| 80/10243<br>1980<br>Germany | 1.7 | forage<br><br>grain<br><br>straw | 0<br>21<br>83<br>87<br>83<br>87 | 7.5<br>6.6<br>0.42<br>0.44<br>6<br>12 | 82/10201<br>1982<br>Germany | 0.61 | forage<br><br><br><br>grain<br><br>straw | 0<br>21<br>35<br>42<br>48<br>69<br>48<br>69 | 8.3<br>3.2<br>2.4<br>1.7<br>0.81<br>0.77<br>6.2<br>4.3 |
| 82/10202<br>1982<br>Germany | 0.61 | forage<br><br>grain<br><br>straw | 0<br>20<br>34<br>42<br>34<br>42 | 12<br>8.2<br>1.5<br>1.4<br>13<br>12 | | | | | |

Table 24. Residues of chlormequat chloride in winter wheat reported to the 1994 JMPR.

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 82/10214<br>1982<br>Denmark | 0.61 | ear<br>stalk<br>grain<br>straw | 47<br>47<br>99<br>99 | <0.1<br>0.11<br>0.15<br>1.5 | 83/10197<br>1983<br>France | 0.61 | grain<br>straw | 96<br>96 | ≤0.05<br>2.3 |
| 83/10198<br>1983<br>France | 0.61 | grain<br>straw | 82<br>82 | ≤0.05<br>4.8 | 83/10199<br>1983<br>France | 0.61 | grain<br>straw | 84<br>84 | ≤0.05<br>2.6 |
| 80/10230<br>1980<br>Germany | 1.6 | grain<br><br>straw | 94<br>98<br>94<br>96 | 0.15<br>0.17<br>6.1<br>6 | 80/10232<br>1980<br>Germany | 1.6 | grain<br><br>straw | 94<br>98<br>94<br>96 | 0.28<br>0.17<br>3.8<br>5.1 |
| 80/10234<br>1980<br>Germany | 1.6 | grain<br><br>straw | 94<br>98<br>94<br>98 | 0.34<br>0.29<br>2.8<br>3.9 | 80/10249<br>1980<br>Germany | 1.7 | grain<br><br>straw | 94<br>98<br>94<br>98 | 0.22<br>0.23<br>7.4<br>8 |
| 80/10251<br>1980<br>Germany | 1.7 | grain<br><br>straw | 94<br>98<br>94<br>98 | 0.25<br>0.31<br>5.7<br>6.6 | 80/10253<br>1980<br>Germany | 1.7 | grain<br><br>straw | 94<br>98<br>94<br>98 | 0.33<br>0.37<br>4.4<br>4.8 |
| 82/10199<br>1982<br>Germany | 0.76 | forage[a]<br><br><br>ear<br><br>stalk<br><br>grain<br>straw | 0<br>21<br>35<br>42<br>49<br>42<br>49<br>56<br>56 | 10<br>4.4<br>2<br>0.29<br>0.84<br>2.9<br>4<br>0.28<br>7.2 | 82/10200<br>1982<br>Germany | 0.76 | forage[1]<br><br>ear<br>stalk<br>grain<br><br>straw | 0<br>21<br>35<br>35<br>42<br>49<br>42<br>49 | 8.8<br>3.3<br>2.7<br>8.3<br>0.62<br>0.53<br>15<br>15 |
| 76/10147<br>1976<br>UK | 1.6 | grain<br>straw | 93<br>93 | 0.05<br>5.4 | 77/10247<br>1977<br>UK | 1.6 | grain<br><br>straw | 51<br>131<br>131 | 1.4<br>0.3<br>0.5 |

Maize. The residue data reported to the 1994 JMPR were re-evaluated for the estimation of maximum residue levels. They are shown in Table 25.

Table 25. Residues of chlormequat chloride in maize reported to the 1994 JMPR.

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 84/10237<br>1984<br>Germany | 0.61 | forage<br><br><br>cob<br>rem[1]<br><br>grain | 0<br>26<br>34<br>98<br>98<br>111<br>111 | 4.4<br>2.7<br>4.8<br>0.34<br>2.7<br>4.1<br>0.14 | 84/10238<br>1984<br>Germany | 0.61 | forage<br><br>cob<br><br><br>rem | 0<br>17<br>35<br>86<br>113<br>35<br>86<br>113 | 22<br>6.2<br>0.88<br>1.6<br>1.7<br>8.3<br>6<br>4.3 |
| 84/10239<br>1984<br>Germany | 0.61 | forage<br><br><br>cob<br><br>rem | 0<br>21<br>32<br>68<br>109<br>68<br>109 | 9.1<br>2.4<br>1.6<br>0.82<br>1.2<br>1.2<br>2.5 | 84/10240<br>1984<br>Germany | 0.61 | forage<br><br><br>cob<br><br>rem | 0<br>22<br>34<br>83<br>106<br>83<br>106 | 26<br>1.6<br>0.69<br><0.05<br><0.05<br>0.79<br>0.68 |
| 84/10241<br>1984<br>Germany | 0.61 | forage<br><br><br>cob<br>rem<br><br>grain | 0<br>20<br>30<br>62<br>62<br>92<br>92 | 20<br>0.92<br>0.89<br>0.34<br><0.5<br>0.8<br>0.5 | 85/10309<br>1985<br>Germany | 0.61 | forage<br><br><br>cob<br>rem<br><br>grain | 0<br>13<br>36<br>71<br>71<br>90<br>90 | 4.8<br>5.0<br>1.2<br>1.2<br>3.7<br>2.4<br>0.68 |
| 85/10310<br>1985<br>Germany | 0.61 | forage<br><br><br>cob<br><br>rem | 0<br>20<br>33<br>71<br>93<br>71<br>93 | 6.3<br>0.32<br>0.39<br>0.20<br>0.23<br><0.05<br>0.36 | 85/10311<br>1985<br>Germany | 0.61 | forage<br><br>cob<br><br><br>rem | 0<br>35<br>64<br>77<br>107<br>64<br>77<br>107 | 3.1<br>3.4<br>0.4<br>0.35<br>0.44<br>2.7<br>3.9<br>5.1 |
| 85/10312<br>1985<br>Germany | 0.61 | forage<br><br><br>cob<br>rem<br><br>grain | 0<br>13<br>27<br>61<br>61<br>78<br>78 | 5.3<br>4.3<br>3.6<br>2.9<br>2.7<br>4.5<br>2.4 | | | | | |

Rape seed. The residue data reported to the 1994 JMPR on which that Meeting estimated a maximum residue level are shown in Table 26. The present Meeting re-evaluated the results to estimate an STMR>

Table 26. Residues of chlormequat chloride in rape seed reported to the 1994 JMPR.

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 85/10313<br>1985<br>Germany | 0.92 | forage<br><br>seed | 0<br>14<br>75 | 4.2<br>1.4<br>2.3 | 85/10314<br>1985<br>Germany | 0.92 | forage<br><br>seed | 0<br>14<br>70<br>87 | 2.1<br>6.1<br>1.4<br>4.3 |
| 85/10315<br>1985<br>Germany | 0.92 | forage<br><br>seed | 0<br>15<br>88 | 6.0<br>4.8<br>2.2 | 85/10316<br>1985<br>Germany | 0.92 | forage<br><br>seed | 0<br>14<br>77 | 4.1<br>1.8<br>2.6 |

| Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg | Report no., year, country | Application, kg ai/ha | Sample | PHI, days | Residues, mg/kg |
|---|---|---|---|---|---|---|---|---|---|
| 85/10317<br>1985<br>Germany | 0.92 | forage<br><br>seed | 0<br>14<br>77 | 8.9<br>6.5<br>5.8 | 86/10378<br>1986<br>Germany | 0.92 | forage<br><br>seed | 0<br>14<br>80 | 8.3<br>1.7<br>2.9 |
| 86/10379<br>1986<br>Germany | 0.92 | forage<br><br>seed | 0<br>15<br>86 | 15<br>1.4<br>2.1 | 86/10380<br>1986<br>Germany | 0.92 | forage<br><br>seed | 0<br>14<br>90 | 2.7<br>0.96<br>1.7 |
| 86/10381<br>1986<br>Germany | 0.92 | forage<br><br>seed | 0<br>14<br>77 | 9.9<br>3.0<br>2.7 | 83/10190<br>1983<br>UK | 1.9 | seed | 93 | 3.7 |

Livestock feeding trials

Hens. Four groups each of four laying Lohmann brown hens were dosed with 0, 0.72, 2.16 or 7.2 mg chlormequat chloride bird/day for 28 days, equivalent to 0, 6, 18 or 60 ppm in the feed (Weidenauer, 1999a). The hens in each group were killed after the last dose and tissue samples collected. Two additional groups of 12 hens were dosed at the highest level for 28 days and allowed to recover after the last dose for 2 or 7 days. The tissues from the birds in each group were then analysed. Eggs from the hens in each group were analysed as indicated in Table 29.

The eggs and tissues were analysed for chlormequat as described in “Analytical methods” (Weidenauer, 1999a). The LOD was 0.05 mg/kg. Table 27 shows sampling, freezing, shipping, homogenization, extraction, and analysis dates. The information on storage stability (Weidenauer, 2000) was inadequate.

Table 27. Hen feeding study dates (Weidenauer, 1999a).

| Procedure | Eggs | | Tissues | |
|---|---|---|---|---|
| | First | Last | First | Last |
| Sampling | March 2, 1998 | April 6, 1998 | March 30, 1998 | April 6, 1998 |
| Sample freezing | March 2, 1998[1] | April 6, 1998[1] | March 30, 1998[1] | April 6, 1998[1] |
| Sample receipt at lab | March 10, 1998 | April 6, 1998 | March 30, 1998 | April 6, 1998 |
| Homogenization | March 2, 1998[2] | April 6, 1998[2] | April 1, 1998 | April 7, 1998 |
| Extraction | March 13, 1998 | December 4, 1998 | May 7, 1998 | June 4, 1998 |
| Analysis | March 13, 1998 | December 7, 1998 | May 13, 1998 | June 26, 1998 |

[1]All egg and tissue samples were frozen on the day of sampling.
[2]All egg samples were homogenized on the day of sampling.

The residues of chlormequat chloride in the hen meat, liver, fat and eggs are shown in Tables 28 and 29.

Table 28. Residues of chlormequat chloride in hen tissues (Weidenauer, 1999a).

| Group no.[1] | Residues, mg/kg | | | |
|---|---|---|---|---|
| | Feeding level | Meat | Liver | Fat |
| 4 | 6 ppm | <0.05 | 0.09 | <0.05 |
| 5 | | <0.05 | <0.05[2] | <0.05 |
| 6 | | <0.05 | <0.05[2] | <0.05 |
| Mean | | <0.05 | 0.05 | <0.05 |
| 7 | 18 ppm | <0.05 | <0.05[2] | <0.05 |
| 8 | | <0.05 | 0.1 | <0.05 |
| 9 | | <0.05 | 0.09 | <0.05 |
| Mean | | <0.05 | 0.07 | <0.05 |
| 10 | 60 ppm | <0.05 | 0.12 | <0.05 |
| 11 | | <0.05 | 0.1 | <0.05 |
| 12 | | <0.05 | 0.33 | <0.05 |

| Group no.[1] | Residues, mg/kg | | | |
|---|---|---|---|---|
| | Feeding level | Meat | Liver | Fat |
| Mean | | <0.05 | 0.18 | <0.05 |
| 13 | 60 ppm | <0.05 | 0.12 | <0.05 |
| 13 | | <0.05 | <0.05[2] | <0.05 |
| 13 | | <0.05 | <0.05[2] | <0.05 |
| Mean | | <0.05 | 0.06 | <0.05 |
| 14 | 60 ppm | <0.05 | <0.05[2] | <0.05 |
| 14 | | <0.05 | 0.08 | <0.05 |
| 14 | | <0.05 | <0.05[2] | <0.05 |
| Mean | | <0.05 | <0.05[2] | <0.05 |

[1] Groups 4-12 were each of 4 hens. Groups 13 and 14 were each of 12 hens and the 3 samples were each composites of 4 hens.
[2] Half of the LOD (0.025 mg/kg) used for mean calculation

Table 29. Residues of chlormequat chloride in hen eggs (Weidenauer, 1999a).

| Group no.[1] | Feeding level | Day 0/1, mg/kg | Day 1/2, mg/kg | Day 3/4, mg/kg | Day 5/6, mg/kg | Day 7/8, mg/kg | Day 10/11, mg/kg | Day 12/13, mg/kg | Day 14/15, mg/kg | Day 17/18, mg/kg | Day 20/21, mg/kg | Day 23/24, mg/kg | Day 25/26, mg/kg |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 6 ppm | <0.05 | <0.05 | <0.05 | <0.05 | 0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 5 | | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | 0.05 | <0.05 | <0.05 | <0.05 |
| 6 | | <0.05 | <0.05 | 0.06 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| Mean | | <0.05 | <0.05 | 0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05 |
| 7 | 18 ppm | <0.05 | <0.05 | 0.06 | <0.05 | 0.09 | 0.06 | <0.05 | <0.05[2] | <0.05 | <0.05 | <0.05 | <0.05 |
| 8 | | <0.05 | <0.05 | <0.05 | <0.05 | 0.12 | 0.1 | 0.07 | 0.09 | <0.05 | <0.05 | <0.05 | <0.05 |
| 9 | | <0.05 | <0.05 | <0.05 | <0.05 | 0.1 | 0.07 | <0.05 | 0.06 | 0.05 | <0.05 | <0.05 | 0.06 |
| Mean | | <0.05 | <0.05 | <0.05 | <0.05 | 0.1 | 0.08 | <0.05 | 0.06 | <0.05 | <0.05 | <0.05 | <0.05 |
| 10 | 60 ppm | <0.05 | <0.05 | 0.1 | <0.05[2] | 0.13 | 0.08 | 0.08 | 0.12 | 0.07 | 0.07 | 0.09 | 0.06 |
| 11 | | <0.05 | <0.05 | <0.05[2] | 0.08 | 0.08 | 0.07 | 0.08 | 0.19 | 0.14 | <0.05[2] | 0.08 | 0.15 |
| 12 | | <0.05 | <0.05 | 0.07 | 0.16 | 0.08 | 0.11 | 0.1 | 0.16 | <0.05[2] | <0.05[2] | 0.05 | 0.07 |
| 13 | | <0.05 | <0.05 | 0.06 | 0.18 | 0.17 | 0.09 | 0.07 | <0.05[2] | 0.08 | 0.05 | 0.06 | 0.08 |
| 14 | | <0.05 | <0.05 | <0.05[2] | 0.11 | 0.08 | 0.13 | <0.05[2] | 0.07 | 0.06 | 0.06 | 0.06 | 0.07 |
| Mean | | <0.05 | <0.05 | 0.06 | 0.11 | 0.11 | 0.1 | 0.07 | 0.11 | 0.08 | 0.05 | 0.07 | 0.09 |

NA: not applicable
[1] Groups 4-12 were each of 4 hens. Groups 13 and 14 were each of 12 hens and the 3 samples were each composites of 4 hens.
[2]half of the LOD (0.025 mg/kg) used for calculation of mean

Cows. A feeding study was carried out on lactating cows by Weidenauer (1999b). Groups of three Holstein dairy cows were dosed with chlormequat chloride for 28 consecutive days at 0, 240, 720 or 2400 mg/animal/day, or 0, 0.4, 1.3 or 4 mg/kg bw/day, equivalent to 0, 12, 36 or 120 ppm in the diet on a dry weight basis. Two extra cows were treated at the high dose level for 28 days and slaughtered 2 or 7 days after their last dose. The doses were equivalent to 0, 0.31, 1.01 and 3.1 mg/kg bw/day calculated as chlormequat cation.

Milk was collected from each cow throughout the study. After the final dose, three cows in each group were slaughtered and tissue samples collected. Milk and tissue samples were analysed for chlormequat chloride as described above ("Analytical methods", Weidenauer, 1999b). The LOD of the ion-pair chromatography method was 0.01 mg/kg for milk and 0.05 mg/kg for tissues.

Table 30 shows the sampling, freezing, shipping, sample preparation, extraction, and analysis dates. The information on storage stability (Weidenauer, 2000) was inadequate.

Table 30. Cow feeding study dates (Weidenauer, 1999b).

| Procedure | Milk | | Tissues | |
|---|---|---|---|---|
| | First | Last | First | Last |
| Sampling | September 8, 1997 | October 20, 1997 | October 13, 1997 | October 20, 1997 |
| Sample freezing | September 8, 1997[1] | October 20, 1997[1] | October 13, 1997[1] | October 20, 1997[1] |
| Sample receipt at lab | September 17, 1997 | October 20, 1997 | October 14, 1997 | October 20, 1997 |
| Sample preparation | September 17, 1997[2] | October 16, 1997[2] | October 17, 1997 | October 24, 1997 |
| Extraction | November 14, 1997 | October 9, 1998 | February 2, 1998 | October 7, 1998 |
| Analysis | November 18, 1997 | October 15, 1998 | February 4, 1998 | October 15, 1998 |

[1] All milk and tissue samples frozen on day of sampling except samples for preparation of skimmed milk and cream, which were stored at +4°C and frozen after separation (1 or 2 days after sampling).
[2] Preparation of skimmed milk and cream; no preparation of whole milk samples required.

The residues of chlormequat chloride in cow meat, liver, kidney, fat, milk, skimmed milk and cream are shown in Tables 31, 32 and 33. The cream and fat content of the milk samples is given in Table 34.

Table 31. Residues of chlormequat chloride in cow tissues (Weidenauer, 1999b).

| Cow no. | Dose | Residue, mg/kg | | | |
|---|---|---|---|---|---|
| | | Meat | Liver | Kidney | Fat |
| 4 | 12 ppm | <0.05 | 0.08 | 0.30 | <0.05 |
| 5 | | <0.05 | 0.10 | 0.07 | <0.05 |
| 6 | | <0.05 | 0.06 | 0.12 | <0.05 |
| Mean | | <0.05 | 0.08 | 0.16 | <0.05 |
| 7 | 36 ppm | <0.05 | 0.09 | 0.46 | 0.05 |
| 8 | | 0.11 | 0.09 | 0.44 | <0.05 |
| 9 | | <0.05 | 0.05 | 0.31 | <0.05 |
| Mean | | <0.05 | 0.08 | 0.40 | <0.05 |
| 10 | 120 ppm | <0.05 | 0.04 | 0.95 | 0.10 |
| 11 | | <0.05 | 0.24 | 0.27 | 0.05 |
| 12 | | 0.07 | 0.50 | 1.06 | 0.10 |
| Mean | | <0.05 | 0.38 | 0.76 | 0.08 |
| 13 | 120 ppm recovery | <0.05 | <0.05 | 0.16 | <0.05 |
| 14 | | <0.05 | <0.05 | 0.09 | <0.05 |

Table 32. Residues of chlormequat chloride in skimmed milk and cream samples (Weidenauer, 1999b).

| Cow no. | Dose | Residue, mg/kg | | | | | |
|---|---|---|---|---|---|---|---|
| | | Skimmed milk Day 1 | Skimmed milk Day 14 | Skimmed milk Day 28 | Cream Day 1 | Cream Day 14 | Cream Day 28 |
| 4 | 12 ppm | 0.04 | 0.10 | 0.02 | <0.01 | 0.02 | 0.02 |
| 5 | | 0.02 | 0.04 | 0.03 | <0.01 | 0.03 | 0.02 |
| 6 | | 0.02 | 0.01 | 0.02 | <0.01 | 0.03 | 0.03 |
| Mean | | 0.03 | 0.05 | 0.02 | <0.01 | 0.03 | 0.02 |
| 7 | 36 ppm | 0.04 | 0.14 | 0.22 | 0.02 | 0.04 | 0.04 |
| 8 | | 0.03 | 0.02 | 0.15 | <0.01 | 0.04 | 0.07 |
| 9 | | 0.02 | 0.10 | 0.04 | 0.01 | 0.05 | 0.04 |
| Mean | | 0.03 | 0.09 | 0.14 | 0.01 | 0.04 | 0.05 |
| 10 | 120 ppm | 0.09 | 0.06 | 0.11 | 0.07 | 0.07 | 0.02 |
| 11 | | 0.09 | 0.38 | 0.16 | 0.07 | 0.11 | 0.09 |
| 12 | | 0.05 | 0.31 | 0.07 | 0.09 | 0.05 | 0.06 |
| 13 | | 0.03 | 0.02 | 0.13 | 0.09 | 0.10 | 0.04 |
| 14 | | 0.06 | 0.36 | 0.11 | 0.11 | 0.09 | 0.10 |
| Mean | | 0.06 | 0.23 | 0.12 | 0.09 | 0.08 | 0.06 |

Table 33. Residues of chlormequat chloride in cow milk (Weidenauer, 1999b).

| Animal No. | Dose | Day 0/1, mg/kg | Day 1/2, mg/kg | Day 3/4, mg/kg | Day 5/6, mg/kg | Day 7/8, mg/kg | Day 10/11, mg/kg | Day 12/13, mg/kg | Day 14/15, mg/kg | Day 17/18, mg/kg | Day 20/21, mg/kg | Day 23/24, mg/kg | Day 25/26, mg/kg |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 4 | 12 ppm | 0.02 | 0.02 | 0.02 | 0.01 | 0.01 | 0.01 | 0.05 | 0.04 | 0.02 | 0.03 | 0.05 | 0.02 |
| 5 | | <0.01 | <0.01 | 0.05 | 0.05 | 0.02 | 0.05 | 0.02 | 0.08 | <0.01 | 0.03 | <0.01 | 0.04 |
| 6 | | <0.01 | 0.01 | 0.01 | 0.05 | <0.01 | 0.05 | <0.01 | 0.04 | <0.01 | 0.03 | <0.01 | 0.05 |
| Mean | | <0.01 | 0.01 | 0.03 | 0.04 | 0.01 | 0.04 | 0.02 | 0.05 | 0.01 | 0.03 | 0.02 | 0.04 |
| 7 | 32 ppm | <0.01 | 0.04 | 0.14 | 0.17 | 0.11 | 0.17 | 0.10 | 0.26 | 0.07 | 0.09 | <0.01 | 0.09 |
| 8 | | <0.01 | 0.06 | 0.03 | 0.10 | 0.09 | 0.19 | 0.07 | 0.21 | 0.07 | 0.08 | 0.24 | 0.12 |
| 9 | | <0.01 | 0.01 | 0.05 | 0.07 | 0.08 | 0.13 | 0.06 | 0.09 | 0.02 | 0.06 | 0.13 | 0.12 |
| Mean | | <0.01 | 0.04 | 0.07 | 0.11 | 0.09 | 0.16 | 0.08 | 0.19 | 0.05 | 0.08 | 0.12 | 0.11 |
| 10 | 120 ppm | <0.01 | 0.07 | 0.47 | 0.06 | 0.28 | 0.23 | 0.29 | 0.13 | 0.09 | 0.26 | 0.16 | 0.30 |
| 11 | | <0.01 | 0.07 | 0.21 | 0.40 | 0.23 | 0.14 | 0.31 | 0.65 | 0.13 | 0.23 | 0.29 | 0.21 |
| 12 | | <0.01 | 0.20 | 0.16 | 0.10 | 0.25 | 0.21 | 0.11 | 0.07 | 0.32 | 0.35 | 0.33 | 0.13 |
| 13 | | <0.01 | 0.14 | 0.32 | 0.35 | 0.18 | 0.11 | 0.19 | 0.20 | 0.20 | 0.33 | 0.16 | 0.16 |
| 14 | | <0.01 | 0.07 | 0.56 | 0.33 | 0.2 | 0.29 | 0.35 | 0.07 | 0.21 | 0.05 | 0.19 | 0.21 |
| Mean | | <0.01 | 0.11 | 0.34 | 0.25 | 0.23 | 0.20 | 0.25 | 0.22 | 0.19 | 0.24 | 0.23 | 0.20 |

[1]NA: not applicable

Table 34. Cream and fat content of the milk samples (Weidenauer, 1999b).

| Cow no. | Dose | Fat, % | | | Cream, % | | |
|---|---|---|---|---|---|---|---|
| | | Day 1 | Day 14 | Day 28 | Day 1 | Day 14 | Day 28 |
| 4 | 12 ppm | 4.95 | 4.40 | 4.35 | 3.88 | 4.30 | 5.03 |
| 5 | | 5.45 | 5.05 | 5.00 | 4.58 | 4.48 | 4.57 |
| 6 | | 4.85 | 4.50 | 4.70 | 4.03 | 5.23 | 3.70 |
| 7 | 33 ppm | 3.85 | 3.85 | 3.90 | 3.09 | 2.37 | 2.48 |
| 8 | | 5.30 | 4.75 | 4.95 | 4.74 | 5.63 | 5.84 |
| 9 | | 3.80 | 4.95 | 5.20 | 2.10 | 4.10 | 5.44 |
| 10 | 120 ppm | 4.50 | 4.20 | 3.40 | 5.14 | 4.95 | 3.65 |
| 11 | | 3.75 | 3.95 | 3.55 | 1.92 | 3.81 | 1.34 |
| 12 | | 3.83 | 4.35 | 4.25 | 3.41 | 3.87 | 3.03 |
| 13 | | 4.30 | 4.05 | 3.80 | 3.11 | 3.34 | 3.18 |
| 14 | | 5.13 | 4.85 | 4.10 | 4.45 | 4.83 | 5.99 |

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### In storage

No information.

### In processing

Rye and oat grain from supervised trials (Pfarl, 1993b) were processed to rye bran and flour and oat flakes by Schneider (1993) but no details of the processing were reported. Brüggemann and Ocker (1988) prepared oat flakes and wheat flour according to commercial practice.

Table 35. Residues of chlormequat chloride in cereal grains and their processed products.

| Reference, report no., year, country, location | Application rate, kg ai/ha | PHI, days | Sample | Residues, mg/kg | Processing factor |
|---|---|---|---|---|---|
| Schneider (1993),<br>Pfarl (1993b),<br>R 92-15 A /1163, 1992<br>Austria-Linz | 1 x 2.2 | 79 | Rye grain<br>Rye bran<br>Rye flour | 1.7 *(control 0.43)*<br>1.9 *(control 0.97)*<br>1.3 *(control 0.57)* | <br>1.1<br>0.76 |
| Schneider (1993),<br>Pfarl (1993b),<br>R 92-15 /1163,1992,<br>Austria-Leonding | 1 x 1.4 | 49 | Oat grain<br>Oat flakes | 1.4 *(control 0.25)*<br>1.8 *(control 0.35)* | <br>1.3 |
| Brüggemann and Ocker (1988), D 87/88-912, 1986, Germany-München (Puch) | 1 x 1.4 | 74 | Oat grain<br>Chaff<br>Unchaffed grain<br>Oat flakes | 3.0<br>4.2<br><br>1.9<br>0.8 | <br>1.4<br><br>0.63<br>0.27 |
| Brüggemann and Ocker (1988), UCB/D 87/88-116/3, 1985,<br>Germany-Bonn (Kessenich) | 1 x 1.4 | 94 | Wheat grain<br>Wholemeal<br>Wheat bran<br>Wholemeal bread | 0.24<br>0.24<br>0.61<br>0.15 | <br>1<br>2.5<br>0.63 |

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Olthof (2000) reported results of the national food monitoring programme in The Netherlands for pears from 1994 to 1996. They are shown in Table 36.

Table 36. Residues of chlormequat in commercial pears, The Netherlands 1994-1996 (Olthof, 2000).

| Samples analysed | Samples without residues (<LOQ of 0.05 mg/kg) | Samples with residues < MRL | Samples with residues >MRL | Mean[1], mg/kg | MRL, mg/kg |
|---|---|---|---|---|---|
| 478 | 455 | 23 | - | 0.1 | 3 |

[1]For samples with residues <LOD a residue of 0.025 mg/kg was taken to calculate the mean

## NATIONAL MAXIMUM RESIDUE LIMITS

The national MRLs shown below were reported.

| Country | Residue definition | Commodity | MRL, mg/kg |
|---|---|---|---|
| Germany | Chlormequat, expressed as chlormequat cation | Rape seed | 10 |
| | | Cultivated mushrooms | 10 |
| | | Oats | 5 |
| | | Maize | 5 |
| | | Apples | 3 |
| | | Pears | 3 |
| | | Barley, rye, triticale, wheat | 2 |
| | | Grapes | 1 |
| | | Hops | 0.1 |
| | | Olives | 0.1 |
| | | Other oil seeds | 0.1 |
| | | Tree nuts | 0.1 |
| | | Tea | 0.1 |
| | | Other commodities of plant origin | 0.05 |
| The Netherlands | Chlormequat, expressed as chlormequat cation | Nuts | 0.1* |
| | | Pears | 3 |
| | | Table and wine grapes | 1 |
| | | Olives | 0.1* |
| | | Beans (with pods) | 0.05 |
| | | Beans (without pods) | 0.05 |
| | | Peas (with pods) | 0.05 |
| | | Peas (without pods) | 0.05 |
| | | Oil seeds | 0.1* |
| | | Tea | 0.1* |
| | | Hops | 5 |
| | | Oats | 2 |
| | | Wheat, rye, triticale, barley | 0.05* |
| | | Other food commodities | 0.05* |
| Poland | Chlormequat | Cereal grains | 3 |
| | | Pear | 3 |
| | | Tomato | 0.2 |
| | | Other products of plant origin | 0.05 |

* At or about the LOQ

## APPRAISAL

Chlormequat was evaluated within the CCPR periodic review programme in 1994. The Meeting estimated maximum residue levels for a number of commodities, which were recorded as guideline levels only, since the ADI was withdrawn. The 1994 JMPR noted that feeding studies in farm animals and analytical methods for residues in animal products would be desirable. As an ADI was allocated by the 1997 JMPR, the estimates made in 1994 were recommended for use as MRLs in 1997.

The CCPR at its thirtieth session noted that animal transfer studies in poultry and cattle would be available in 1998.

The compound was reviewed toxicologically again in 1999, when an acute RfD was allocated. The 1999 JMPR recommended that an evaluation of residues should be scheduled shortly so that an acute risk assessment could be concluded.

Analytical methods for the determination of residues of chlormequat in water, cereals, pears, and animal products, data on stability in storage of animal products, data on residues in pears and cereals, and the results of a feeding study in dairy cattle and poultry were made available to the Meeting by the manufacturers. The Netherlands submitted its official method of analysis for chlormequat in pears. Information on national MRLs and GAP was provided by the governments of Germany, The Netherlands, and Poland .

***Methods of analysis***

Chlormequat is difficult to analyse because of its chemical nature and because the residue must be separated from native quarternary ammonium compounds in plant material. Older methods involve lengthy clean-up, liquid-liquid partition or column chromatography (ion-exchange, alumina), and semi-quantitative thin-layer chromatographic or photometric detection, but these methods allow only poor reproducibility. More recent methods are based on head-space gas chromatography after pyrolysis of chlormequat to acetylene in an alkaline medium, HPLC by ion-pair chromatography with conductivity detection, or liquid chromatography with mass spectrometric detection.

For cereal grains, the LOQ was 0.05 mg/kg with ion-pair chromatography and 1 mg/kg with head-space gas chromatography with flame ionization detection. The latter method resulted in high values in samples from untreated control plots. The liquid chromatography-mass spectrometric method was used to determine chlormequat residues in pears (LOQ, 0.3 mg/kg).

The ion chromatographic method was validated for animal products, resulting in LOQs of 0.05 mg/kg for eggs and tissues and 0.01 mg/kg for milk. The ion chromatographic technique was also used to analyse chlormequat in water, with an LOQ of 0.05 mg/l.

***Stability of residues in stored analytical samples***

Two samples each of milk, eggs, liver, and fat from farm animals fed chlormequat were stored for 25-33 months at -18 °C. The remaining compound represented 76-140% of the initial concentration in milk, 45-100% in eggs, 60-82% in liver, and 71-90% in fat, with great variation. The Meeting was not able to decide whether chlormequat is stable in enzyme-containing matrices and noted that the study was inadequate.

***Results of supervised trials***

The present Meeting received the results of new supervised trials on pears and cereals. These data and those reported by the 1994 JMPR on which the recommended MRLs for numerous commodities are based were re-evaluated in the view of current GAP and to estimate STMR and HR values.

Chlormequat is registered for use on *pear* in Belgium (at four to five applications of 1.4 kg ai/ha, 0.24 kg ai/hl, 600 l water/ha), Denmark (at two applications of 0.75-1.8 kg ai/ha, 0.075-0.18 kg ai/hl, 1000 l water/ha), The Netherlands (at one or two applications of 0.75-2.3 kg ai/ha, 0.094-0.15 kg ai/hl, 800-1500 l water/ha), and Spain (at five to six applications of 0.9 kg ai/ha, 0.1 kg ai/hl, 900 l water/ha). The PHIs range from 42 days in Denmark to 90 days in The Netherlands, or treatment is fixed at a certain growth stage (Belgium, Spain).

Six trials carried out in The Netherlands in 1968 were not included in the assessment as no information on the spray concentration was received, but the application rates used in two trials conducted in 1983 (two applications of 1.1-1.8 kg ai/ha, 0.11-0.18 kg ai/hl, 1000 l water/ha) were

acceptable in respect of GAP in The Netherlands. The concentrations of residues were 5.3 and 6.9 mg/kg (calculated as chlormequat chloride) 124 and 113 days after the last treatment, respectively. Two further trials (four applications of 1.2-1.6 kg ai/ha, 0.2 kg ai/hl, 600 l water/ha; PHI, 90 and 101 days) complied with the Belgian GAP. The concentrations of residues were 1.6 and 8.1 mg/kg, calculated as chlormequat chloride.

Eight supervised trials were conducted in France in 1998 and 1999. Those carried out in 1998 were in accordance with the Belgian GAP (five applications of 1.4-1.5 kg ai/ha, 0.24 kg ai/hl, 600 l water/ha). The concentrations 44 or 45 days after the last treatment were 4, 4.6, 5.6, and 7.5 mg/kg, calculated as chlormequat chloride. In 1999, only one application was given. The rates used in these trials were not compatible with a currently registered GAP.

The concentrations of residues found in the trials conducted according to GAP were, in rank order (median in italics), 1.6, 4, 4.6, ***5.3***, ***5.6***, 6.9, 7.5, and 8.1 mg/kg calculated as chlormequat chloride or 1.2, 3.1, 3.6, ***4.1***, ***4.3***, 5.3, 5.8, 6.3 mg/kg calculated as chlormequat cation. The Meeting estimated a maximum residue level of 10 mg/kg, confirming the previous recommendation, an STMR value of 4.2 mg/kg, and a HR value of 6.3 mg/kg for pears, calculated as chlormequat cation.

The Meeting received the results of numerous supervised trials on *barley* carried out in the UK, but these data could not be evaluated as high values were determined in samples from untreated plots and no information was submitted about the analytical method used. Trial carried out in Latvia in 1998 and in Hungary in 1991 provided no information on application rates or the analytical method used.

The supervised trials reported by the 1994 JMPR that are in accordance with current GAP were re-evaluated:

| Country | No. of trials | Concentration of residues, calculated as chlormequat chloride, mg/kg | In accordance with GAP of |
|---|---|---|---|
| *Summer barley* | | | |
| Denmark | 4 | <0.05 (2 trials), 0.05, 0.3 | Netherlands, Belgium |
| Germany | 2 | 0.17, 0.62 | Netherlands, Belgium |
| Sweden | 7 | <0.05 (4 trials), 0.1, 0.19, 0.73 | Netherlands |
| UK | 3 | 0.18, 0.24, 0.37 | UK |
| *Winter barley* | | | |
| Denmark | 1 | 0.05 | Netherlands |
| France | 11 | <0.05 (3 trials), 0.16, 0.18, 0.21, 0.24, 0.29, 0.3 (2 trials), 0.35 | Netherlands, Belgium |
| Germany | 5 | 1.3, 1.6, 1.6, 2.1, 2.3 | Germany |
| Germany | 2 | 0.17, 0.18 | Netherlands, Belgium |
| Sweden | 1 | 0.42 | Netherlands, Belgium |
| Switzerland | 2 | 0.23, 0.29 | Netherlands, Belgium |
| UK | 9 | <0.05 (2 trials), 0.05, 0.07, 0.15, 0.24, 0.36, 0.43, 0.58 | UK |

The 47 values for residues, in rank order, were <0.05 (11 trials), 0.05 (3 trials), 0.07, 0.1, 0.15, 0.16, 0.17 (2 trials), 0.18 (3 trials), ***0.19***, 0.21, 0.23, 0.24 (3 trials), 0.29 (2 trials), 0.3 (3 trials), 0.35, 0.36, 0.37, 0.42, 0.43, 0.58, 0.62, 0.73, 1.3, 1.6 (2 trials), 2.1, and 2.3 mg/kg calculated as chlormequat chloride, or <0.04 (11 trials), 0.04 (3 trials), 0.05, 0.08, 0.12 (2 trials), 0.13 (2 trials), 0.14 (3 trials), ***0.15***, 0.16, 0.18, 0.19 (3 trials), 0.22 (2 trials), 0.23 (3 trials), 0.27, 0.28, 0.29, 0.33 (2 trials), 0.45, 0.48, 0.57, 1.0, 1.2 (2 trials), 1.6, and 1.8 mg/kg calculated as chlormequat cation.

The Meeting estimated a maximum residue level of 2 mg/kg to replace the previous recommendation of the 1994 JMPR (0.5 mg/kg), an STMR value of 0.15 mg/kg, and a HR value of 1.8 mg/kg for barley, calculated as chlormequat cation.

The Meeting received the results of four supervised trials on *oats* carried out in 1993 in Austria, but the data could not be evaluated as high values were determined in samples from untreated plots. One trial in Germany and one in the UK were conducted in accordance with Belgian and British GAP, respectively. The concentrations were 3 and 0.8 mg/kg in oat grains, calculated as chlormequat cation.

The trials carried out in accordance with current GAP and summarized by the 1994 JMPR were re-evaluated:

| Country | No. of trials | Concentration of residues, calculated as chlormequat chloride, mg/kg | In accordance with GAP of |
|---|---|---|---|
| Germany | 16 | 0.09, 0.14, 0.45, 0.51, 0.9, 1.1, 1.2, | Belgium, Finland |
| | | 1.5, 1.6, 1.7, 1.8, 1.9, 2.0, 2.4, 2.4, 3.3 | Netherlands |
| Germany | 2 | 1.5, 3.7 | Germany |
| UK | 3 | 0.1, 0.63, 9.2 | UK |

The 23 values (21 from submissions in 1994, two from submissions in 2000), in rank order, were 0.09, 0.1, 0.14, 0.45, 0.51, 0.63, 0.8, 0.9, 1.1, 1.2, ***1.5*** (2 trials), 1.6, 1.7, 1.8, 1.9, 2.0, 2.4, 2.4, 3, 3.3, 3.7, and 9.2 mg/kg calculated as chlormequat chloride, or 0.07, 0.08, 0.11, 0.35, 0.39, 0.49, 0.62, 0.7, 0.85, 0.93, ***1.2*** (3 trials), 1.3, 1.4, 1.5, 1.6, 1.9 (2 trials), 2.3, 2.6, 2.9, and 7.1 mg/kg calculated as chlormequat cation.

The Meeting estimated a maximum residue level of 10 mg/kg, confirming the previous recommendation, an STMR value of 1.2 mg/kg, and a HR value of 7.1 mg/kg for oats, calculated as chlormequat cation.

*Triticale, rye and wheat* have comparable use patterns in GAP of Belgium, Denmark, Germany, and the UK. The Meeting received the results of four trials each on triticale and on rye (2.5-5 kg ai/ha) in the UK, which were not in accordance with GAP (maximum, 1.7 kg ai/ha). Two Austrian trials each on rye and wheat could not be evaluated as high values were determined in samples from untreated plots. Eight trials on wheat in the UK could not be evaluated as information on the analytical method used was not submitted. One trial of wheat was carried out in Latvia in 1998, but no information on the application rate or the analytical method used was received. In two trials conducted in Germany in 1988, the application rates were acceptable with regard to British, Italian, and Finnish GAP (1.5-1.7 kg ai/ha); the concentrations were 0.2 and 0.24 mg/kg, calculated as chlormequat chloride.

The supervised trials in accordance with current GAP and summarized by the 1994 JMPR were re-evaluated:

| Country | No. of trials | Concentration of residues, calculated as chlormequat chloride, mg/kg | In accordance with GAP of |
|---|---|---|---|
| *Summer rye* | | | |
| Germany | 4 | 0.06, 1.5, 2.1, 2.6 | Belgium |
| Winter rye | | | |
| Germany | 13 | <0.05, 0.22, 0.24, 0.3 (2 trials), 0.33, 0.34, | UK |
| | | 0.45, 0.62, 0.88, 1.2, 1.4, 1.9 | |
| Germany | 2 | 0.45, 1.8 | Germany |
| Germany | 7 | <0.05, 0.05, 0.07, 0.08, 0.09 (2 trials), 0.43 | Netherlands |
| *Summer wheat* | | | |
| Germany | 9 | 0.31, 0.39, 0.52, 0.59, 0.68, 1.2, 1.3 (2 trials), 1.5 | Austria, Germany, Finland, UK |
| Germany | 8 | 0.09, 0.32, 0.33, 0.41, 0.44 (2 trials), 0.48, 0.59 | Finland, UK |
| Germany | 2 | 0.81, 1.5 | Netherlands |
| *Winter wheat* | | | |
| Denmark | 1 | 0.15 | Belgium |
| France | 3 | <0.05 (3 trials) | Belgium |
| Germany | 6 | 0.17, 0.23, 0.28, 0.31, 0.34, 0.37 | Denmark, UK |

| Country | No. of trials | Concentration of residues, calculated as chlormequat chloride, mg/kg | In accordance with GAP of |
|---|---|---|---|
| Germany | 2 | 0.28, 0.62 | Belgium, Germany, Netherlands |
| UK | 2 | 0.05, 1.4 | UK |

The concentrations of residues in 26 trials in *rye grain*, in rank order, were <0.05 (2 trials), 0.05, 0.06, 0.07, 0.08, 0.09 (2 trials), 0.22, 0.24, 0.3 (2 trials), ***0.33***, ***0.34***, 0.43, 0.45 (2 trials), 0.62, 0.88, 1.2, 1.4, 1.5, 1.8, 1.9, 2.1, and 2.6 mg/kg calculated as chlormequat chloride, or <0.04 (2 trials), 0.04, 0.05 (2 trials), 0.06, 0.07 (2 trials), 0.17, 0.19, 0.23 (2 trials), ***0.26*** (2 trials), 0.33, 0.35 (2 trials), 0.48, 0.68, 0.93, 1.1, 1.2, 1.4, 1.5, 1.6, and 2 mg/kg calculated as chlormequat cation.

The concentrations of residue in 35 trials in *wheat grain* (33 from 1994, 2 from 2000) were <0.05 (3 trials), 0.05, 0.09, 0.15, 0.17, 0.2, 0.23, 0.24, 0.28 (2 trials), 0.31 (2 trials), 0.32, 0.33, 0.34, ***0.37***, 0.39, 0.41, 0.44 (2 trials), 0.48, 0.52, 0.59 (2 trials), 0.62, 0.68, 0.81, 1.2, 1.3 (2 trials), 1.4, and 1.5 (2 trials) mg/kg calculated as chlormequat chloride, or <0.04 (3 trials), 0.04, 0.07, 0.12, 0.13, 0.16, 0.18, 0.19, 0.22 (2 trials), 0.24 (2 trials), 0.25, 0.26 (2 trials), ***0.29***, 0.3, 0.32, 0.34 (2 trials), 0.37, 0.4, 0.46 (2 trials), 0.48, 0.53, 0.63, 0.93, 1 (2 trials), 1.1, and 1.2 (2 trials) mg/kg calculated as chlormequat cation.

As the use patterns are comparable and the STMR values are close, the two data sets were combined: <0.04 (5 trials), 0.04 (2 trials), 0.05 (2 trials), 0.06, 0.07 (3 trials), 0.12, 0.13, 0.16, 0.17, 0.18, 0.19 (2 trials), 0.22 (2 trials), 0.23 (2 trials), 0.24 (2 trials), 0.25, ***0.26*** (4 trials), 0.29, 0.3, 0.32, 0.33, 0.34 (2 trials), 0.35 (2 trials), 0.37, 0.4, 0.46 (2 trials), 0.48 (2 trials), 0.53, 0.63, 0.68, 0.93 (2 trials), 1 (2 trials), 1.1 (2 trials), 1.2 (3 trials), 1.4, 1.5, 1.6, and 2 mg/kg calculated as chlormequat cation.

The Meeting estimated a maximum residue level of 3 mg/kg, an STMR value of 0.26 mg/kg, and a HR value of 2 mg/kg, calculated as chlormequat cation, for rye and wheat, and recommended that these values be extrapolated to triticale. The previous MRL recommended by the 1994 JMPR for rye (3 mg/kg) was confirmed, whereas that for wheat (2 mg/kg) was replaced.

Chlormequat is registered for use on *rape seed* in Belgium (at 0.69 kg ai/ha) and in the UK (at 1.9 kg ai/ha). No new GAP and no data on residues were submitted.

The 1994 JMPR estimated a maximum residue level of 5 mg/kg for rape seed on the basis of one British and nine German trials. The concentrations of residues, in rank order, were 1.7, 2.1, 2.2, 2.3, ***2.6***, *2.7*, 2.9, 3,7, 4.3, and 5.8 mg/kg calculated as chlormequat chloride or 1.3, 1.6, 1.7, 1.8, *2*, ***2.1***, 2.2, 2.8, 3.3, 4.5 mg/kg calculated as chlormequat cation.

The Meeting estimated an STMR value of 2.05 mg/kg for rape seed.

The 1994 JMPR estimated a maximum residue level of 20 mg/kg (fresh weight) for *dry straw and fodder of barley, oats, rye, and wheat*. The 2000 JMPR considered the results of all the available supervised trials conducted according to current GAP:

| Country | No. of trials | Concentration of residues, calculated as chlormequat chloride, mg/kg | In accordance with GAP of |
|---|---|---|---|
| *Summer barley straw* | | | |
| Denmark | 4 | 1.3, 2.7, 4.3, 4.4 | Netherlands, Belgium |
| Germany | 2 | 4, 4.4 | Netherlands, Belgium |
| UK | 3 | 1.6, 1.6, 4.9 | UK |
| *Winter barley straw* | | | |
| Denmark | 1 | 0.9 | Netherlands |
| France | 11 | 0.36, 1.8, 2.4, 2.8, 3.1, 4.4, 4.7, 5.4, 5.5, 8.5, 11 | Netherlands, Spain |
| Germany | 5 | 5.8, 6.2, 6.4, 8.7, 12 | Germany |

| Country | No. of trials | Concentration of residues, calculated as chlormequat chloride, mg/kg | In accordance with GAP of |
|---|---|---|---|
| Germany | 2 | 5.8, 9 | Netherlands, Belgium |
| Switzerland | 2 | 4.2, 4.5 | Netherlands, Belgium |
| UK | 9 | 0.98, 1, 1.1, 2.2, 2.4 (2 trials), 8.9, 12, 16 | UK |
| *Oat straw* | | | |
| Germany | 16 | 0.9, 1.2 (2 trials), 1.3, 1.6, 1.9 (2 trials), 2.2, | Belgium, Finland, |
| | | 3.0, 4.0, 4.8, 6.3, 8.2, 9.9 (3 trials) | Netherlands |
| Germany | 2 | 5.2, 12 | Germany |
| UK | 3 | 0.48, 3.3, 25 | UK |
| *Summer rye straw* | | | |
| Germany | 4 | 0.2, 0.3, 4.7, 9 | Belgium |
| *Winter rye* | | | |
| Germany | 12 | 2.2, 2.7, 2.8, 3.1, 4.3, 4.5, 4.8, 5.7, 6.9, 9.6 (2 trials), 12 | UK |
| Germany | 1 | 7.5 | Germany |
| Germany | 1 | 5.5 | Netherlands |
| UK | 2 | 0.48, 12 | UK |
| *Summer wheat straw* | | | |
| Germany | 9 | 10, 13, 14, 17, 18 (2 trials), 20, 21, 29 | Austria, Germany, |
| | | | Finland, UK |
| Germany | 8 | 5.8, 7, 7, 11, 12, 13, 15, 17 | Finland, UK |
| Germany | 2 | 6.2, 13 | Netherlands |
| *Winter wheat straw* | | | |
| Denmark | 1 | 1.5 | Belgium |
| France | 3 | 2.3, 2.6, 4.8 | Belgium |
| Germany | 6 | 3.9, 4.8, 5.1, 6.1, 6.6, 8.0 | Denmark, UK |
| Germany | 2 | 7.2, 15 | Belgium, Germany, Netherlands |
| UK | 2 | 0.5, 5.4 | UK |

Two further trials each on oats and wheat were submitted to the current JMPR. The residues of chlormequat chloride were 0.7 and 3 mg/kg in oat straw and 0.5 and 0.9 mg/kg in wheat straw (fresh weight).

The concentrations of residues (fresh weight) in 39 trials with *barley straw*, in rank order, were 0.36, 0.9, 0.98, 1, 1.1, 1.3, 1.6 (2 trials), 1.8, 2.2, 2.4 (3 trials), 2.7, 2.8, 3.1, 4, 4.2, 4.3, ***4.4*** (3 trials), 4.5, 4.7, 4.9, 5.4, 5.5, 5.8 (2 trials), 6.2, 6.4, 8.5, 8.7, 8.9, 9, 11, 12 (2 trials), and 16 mg/kg calculated as chlormequat chloride or 0.28, 0.7, 0.76, 0.78, 0.85, 1, 1.2 (2 trials), 1.4, 1.7, 1.9 (3 trials), 2.1, 2.2, 2.4, 3.1, 3.3 (2 trials), ***3.4*** (3 trials), 3.5, 3.7, 3.8, 4.2, 4.3, 4.5 (2 trials), 4.8, 5, 6.6, 6.8, 6.9, 7, 8.5, 9.3, 9.3, and 12 mg/kg calculated as chlormequat cation.

The concentrations in 23 trials with *oat straw*, were 0.48, 0.7, 0.9, 1.2 (2 trials), 1.3, 1.6, 1.9 (2 trials), 2.2, *3* (2 trials), 3.3, 4, 4.8, 5.2, 6.3, 8.2, 9.9 (3 trials), 12, and 25 mg/kg calculated as chlormequat chloride or 0.37, 0.54, 0.7, 0.93 (2 trials), 1, 1.2, 1.5 (2 trials), 1.7, ***2.3*** (2 trials), 2.6, 3.1, 3.7, 4, 4.9, 6.4, 7.7 (3 trials), 9.3, and 19 mg/kg calculated as chlormequat cation.

The values in 20 trials with *rye straw* were 0.2, 0.3, 0.48, 2.2, 2.7, 2.8, 3.1, 4.3, 4.5, ***4.7***, ***4.8***, 5.5, 5.7, 6.9, 7.5, 9, 9.6 (2 trials), and 12 (2 trials) mg/kg calculated as chlormequat chloride or 0.16, 0.23, 0.37, 1.7, 2.1, 2.2, 2.4, 3.3, 3.5, ***3.7*** (2 trials), 4.3, 4.4, 5.4, 5.8, 7, 7.4 (2 trials), and 9.3 (2 trials) mg/kg calculated as chlormequat cation.

The concentrations in 35 trials with *wheat straw* were 0.5 (2 trials), 0.9, 1.5, 2.3, 2.6, 3.9, 4.8 (2 trials), 5.1, 5.4, 5.8, 6.1, 6.2, 6.6, 7 (2 trials), ***7.2***, 8, 10, 11, 12, 13 (3 trials), 14, 15 (2 trials), 17 (2 trials), 18 (2 trials), 20, 21, and 29 mg/kg calculated as chlormequat chloride, or 0.39 (2 trials), 0.7, 1.2, 1.8, 2, 3, 3.7 (2 trials), 4, 4.2, 4.5, 4.7, 4.8, 5.1, 5.4 (2 trials), ***5.6***, 6.2, 7.8, 8.5, 9.3, 10 (3 trials), 11, 12 (2 trials), 13 (2 trials), 14 (2 trials), 16 (2 trials), and 22 mg/kg calculated as chlormequat cation.

The 117 values available for *straw* (fresh weight), in rank order, are: 0.16, 0.23, 0.28, 0.37 (2 trials), 0.39 (2 trials), 0.54, 0.7 (3 trials), 0.76, 0.78, 0.85, 0.93 (2 trials), 1, 1, 1.2 (4 trials), 1.4, 1.5 (2 trials), 1.7 (3 trials), 1.8, 1.9 (3 trials), 2, 2.1 (2 trials), 2.2 (2 trials), 2.3 (2 trials), 2.4 (2 trials), 2.6, 3, 3.1 (2 trials), 3.3 (3 trials), 3.4 (3 trials), 3.5 (2 trials), ***3.7*** (6 trials), 3.8, 4, 4.2 (2 trials), 4.3 (2 trials), 4.4, 4.5 (3 trials), 4.7, 4.8 (2 trials), 4.9, 5, 5.1, 5.4 (3 trials), 5.6, 5.8, 6.2, 6.4, 6.6, 6.8, 6.9, 7 (2 trials), 7.4 (2 trials), 7.7 (3 trials), 7.8, 8.5 (2 trials), 9.3 (6 trials), 10 (3 trials), 12 (3 trials), 13 (2 trials), 14 (2 trials), 16 (2 trials), 19, and 22 mg/kg calculated as chlormequat cation.

Allowing for the standard 89% of dry matter (FAO, 1997) in cereal straw (barley, 89%; oats, 90%; rye, 88%; wheat, 88%), the Meeting estimated a maximum residue level and an STMR value for dry straw and fodder of cereal grains of 30 mg/kg and 4.2 mg/kg (3.7/0.89), respectively, calculated as chlormequat cation. The previously recommended MRL of 20 mg/kg (fresh weight) for dry straw and fodder of barley, oats, rye, and wheat is withdrawn.

The 1994 JMPR estimated a maximum residue level of 20 mg/kg for *oat and rye forage* (*green*) on a fresh weight basis. The current Meeting considered the supervised trials that had been conducted according to current use patterns:

| Country | No. of trials | Concentration of residues, calculated as chlormequat chloride, mg/kg | In accordance with GAP of |
|---|---|---|---|
| *Oats* | | | |
| Germany | 17 | 1.1, 1.3, 1.5, 1.8, 2.5, 2.9, 3.1, 3.5, 3.7, 4.3, 6.4, 6.9, 7.6, 8.1, 15, 15, 17 | Belgium, Germany, Finland, Netherlands |
| UK | 1 | 4.7 | UK |
| *Summer rye* | | | |
| Germany | 4 | 11, 12, 12, 13 | |
| Belgium | | | |
| *Winter rye* | | | |
| Germany | 14 | 1.9, 2.1, 2.2, 3.6, 4.1, 4.2, 4.3, 4.9, 5.9, 9, 17, 18, 20, 28 | Germany, UK, Netherlands |

A further trial on oats was submitted for consideration by the 2000 JMPR, in which the concentration of residue was 28 mg/kg, expressed as chlormequat chloride, in the whole green plant 30 days after treatment.

The concentrations found in all 37 trials on oat and rye forage (fresh weight) were, in rank order, 1.1, 1.3, 1.5, 1.8, 1.9, 2.1, 2.2, 2.5, 2.9, 3.1, 3.5, 3.6, 3.7, 4.1, 4.2, 4.3 (2 trials), 4.7, ***4.9***, 5.9, 6.4, 6.9, 7.6, 8.1, 9, 11, 12 (2 trials), 13, 15 (2 trials), 17 (2 trials), 18, 20, and 28 (2 trials) mg/kg calculated as chlormequat chloride, or 0.85, 1, 1.2, 1.4, 1.5, 1.6, 1.7, 1.9, 2.2, 2.4, 2.7, 2.8, 2.9, 3.2, 3.3 (3 trials), 3.6, ***3.8***, 4.6, 5, 5.3, 5.9, 6.3, 7, 8.5, 9.3 (2 trials), 10, 12 (2 trials), 13 (2 trials), 14, 16, and 22 (2 trials) mg/kg calculated as chlormequat cation.

Allowing for the standard 30% of dry matter in cereal forage (FAO, 1997), the Meeting estimated a maximum residue level of 100 mg/kg and an STMR value of 13 mg/kg (3.8/0.3), calculated as chlormequat cation (dry weight), for oat and rye forage. The previous MRL recommendation (20 mg/kg, fresh weight) is withdrawn.

No new data on residues or GAP for maize were submitted. The 1994 JMPR had received the results of nine supervised trials conducted in Germany at rates within the range of Belgium GAP. For green maize plants, including cobs, the following concentrations (fresh weight) were reported (PHI, 13-35 days): 0.39, 0.92, 1.6, 2.4, ***3.4***, 4.3, 4.8, 5.0, and 6.2 mg/kg calculated as chlormequat chloride or 0.3, 0.71, 1.2, 1.9, ***2.6***, 3.3, 3.7, 3.9, and 4.8 mg/kg calculated as chlormequat cation.

Allowing for the standard 40% of dry matter in maize forage (FAO, 1997), the Meeting estimated a maximum residue level of 15 mg/kg and an STMR value of 6.5 mg/kg (2.6/0.4), calculated as chlormequat cation (dry weight).

For maize fodder, the following concentrations (fresh weight) were reported (PHI, 78-113 days): 0.36, 0.68, 0.8, 2.4, *2.5*, 4.1, 4.3, 4.5, 5.1 mg/kg calculated as chlormequat chloride, or 0.28, 0.53, 0.62, ***1.9*** (2 trials), 3.2, 3.3, 3.5, and 4 mg/kg calculated as chlormequat cation.

Allowing for the standard 83% of dry matter in maize stover (FAO, 1997), the Meeting estimated a maximum residue level of 7 mg/kg and an STMR value of 2.3 mg/kg (1.9/0.83), calculated as chlormequat cation (dry weight).

***Residues in animal and poultry commodities***

The Meeting estimated the dietary burden of chlormequat residues in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual. Calculation from the MRLs yields maximum concentrations of residues in feed suitable for estimating MRLs for animal commodities. Calculation from the STMR values for feed allows estimation of STMR values for animal commodities.

| Commodity | MRL, mg/kg | Group | % dry matter | MRL/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |
| Barley grain | 2 | GC | 88 | 2.3 | | | | | | |
| Barley straw | 30 | AS | 100 | 30 | | | | | | |
| Oat grain | 10 | GC | 89 | 11 | 35 | 40 | 80 | 3.9 | 4.5 | 9.0 |
| Oat forage | 100 | AF | 100 | 100 | 25 | 60 | | 25 | 60 | |
| Oat straw | 30 | AS | 100 | 30 | | | | | | |
| Maize forage | 15 | AF | 100 | 15 | 40 | | | 6 | | |
| Maize fodder | 7 | AS | 100 | 7 | | | | | | |
| Rye grain | 3 | GC | 88 | 3.4 | | | | | | |
| Rye forage | 100 | AF | 100 | 100 | | | | | | |
| Rye straw | 30 | AS | 100 | 30 | | | | | | |
| Wheat grain | 3 | GC | 89 | 3.4 | | | 20 | | | 0.67 |
| Wheat straw | 30 | AS | 100 | 30 | | | | | | |
| Sum | | | | | 100 | 100 | 100 | 35 | 65 | 9.6 |

| Commodity | STMR, mg/kg | Group | % dry matter | STMR/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |
| Barley grain | 0.15 | GC | 88 | 0.17 | | | | | | |
| Barley straw | 4.2 | AS | 100 | 4.2 | | | | | | |
| Oat grain | 1.2 | GC | 89 | 1.4 | 35 | 40 | 80 | 0.47 | 0.54 | 1.1 |
| Oat forage | 12.7 | AF | 100 | 13 | 25 | 60 | | 3.2 | 7.6 | |
| Oat straw | 4.2 | AS | 100 | 4.2 | | | | | | |
| Maize forage | 6.5 | AF | 100 | 6.5 | 40 | | | 2.6 | | |
| Maize fodder | 2.3 | AS | 100 | 2.3 | | | | | | |
| Rye grain | 0.26 | GC | 88 | 0.30 | | | 20 | | | 0.06 |
| Rye forage | 12.7 | AF | 100 | 13 | | | | | | |
| Rye straw | 4.2 | AS | 100 | 4.2 | | | | | | |
| Wheat grain | 0.26 | GC | 89 | 0.29 | | | | | | |
| Wheat straw | 4.2 | AS | 100 | 4.2 | | | | | | |
| Sum | | | | | 100 | 100 | 100 | 6.3 | 8.1 | 1.1 |

*Cows*

Groups of three lactating cows were given chlormequat chloride in the diet twice daily at a dose of 240, 720, or 2400 mg/animal per day, equivalent to 0.4, 1.3, and 4 mg/kg bw per day or 12, 36, and 120 ppm on a dry weight basis, for 28 consecutive days. Two additional animals were treated at the

high dose for 28 days and slaughtered 2 or 7 days after the last dose. The doses were equivalent to 0.31, 1, and 3.1 mg/kg bw per day (or 9.3, 28, and 93 ppm), calculated as chlormequat cation. At the lowest dose, the average concentrations of chlormequat chloride residues were 0.029 mg/kg in milk, 0.1 mg/kg in liver, and 0.2 mg/kg in kidney. No residues were found in meat or fat. At the medium and high doses, the plateau concentrations of chlormequat chloride residue in milk were 0.1 and 0.2 mg/kg. Concentrations up to 0.11 mg/kg were determined in some meat and fat samples. The concentrations were 0.1 and 0.4 mg/kg in liver and 0.4 and 0.8 mg/kg in kidney at the two doses, respectively, indicating that the values in kidney were at least twice as high as in liver. The concentrations of chlormequat chloride in skimmed milk were similar to those in whole milk.

The concentration of chlormequat residues in milk reached a plateau 10-11 days after the first treatment with the medium dose, but after 3-4 days with the low and high doses. The residues were cleared rapidly from meat, fat, and liver, and none could be determined in these tissues 2 days after the end of dosing. The concentrations in milk and kidney fell to about 20% of their plateau values. After 7 days, the values for milk were below the LOQ of 0.01 mg/kg, but 0.09 mg/kg remained in kidney. Although milk and tissue samples were frozen on the day of sampling, they were analysed in part 1 year later, and no adequate information on stability was received.

According to the recommendation of the 1997 JMPR, the maximum residue level and the STMR value for milk were calculated on the basis of dietary burdens of 65 and 8.1 mg/kg, respectively, for dairy cattle. The maximum residue levels and the STMR values for meat, liver, and kidney were derived from dietary burdens of 35 or 6.3 mg/kg, respectively, for beef cattle. The following table shows the highest and the mean actual and extrapolated concentrations of residues for estimation of MRLs and STMR values for chlormequat.

| Dose, ppm | Concentration of residues, mg/kg, calculated as chlormequat cation | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Milk | | Liver | | Kidney | | Muscle | | Fat | |
| | High | Mean[1] | High | Mean | High | Mean | High | Mean | High | Mean |
| MRL for beef cattle | | | | | | | | | | |
| Extrapolated: 35 | | | 0.088 | 0.078 | 0.35 | 0.3 | 0.11 | <0.04 | 0.05 | <0.04 |
| Actual: 28 | | | 0.07 | 0.062 | 0.28 | 0.24 | 0.085 | 0.04 | | |
| MRL for dairy cows | | | | | | | | | | |
| Extrapolated: 65 | 0.35 | 0.13 | | | | | | | | |
| Actual: 93 | 0.5 | 0.18 | | | | | | | | |
| STMR for beef cattle | | | | | | | | | | |
| Extrapolated: 6.3 | | | 0.053 | 0.042 | 0.16 | 0.084 | <0.04 | <0.04 | <0.04 | <0.04 |
| Actual: 9.3 | | | 0.078 | 0.062 | | 0.23 | 0.124 | | | |
| STMR for dairy cows | | | | | | | | | | |
| Extrapolated: 8.1 | 0.05 | 0.018 | | | | | | | | |
| Actual: 9.3 | 0.06 | 0.021 | | | | | | | | |

[1] The mean concentration in milk was calculated from samples taken on days 3-28.

The Meeting estimated maximum residue levels of 0.5 mg/kg for milk, 0.1 mg/kg for liver, 0.5 mg/kg for kidney, and 0.2 mg/kg for meat and recommended that the HR values be 0.35 mg/kg for milk, 0.088 mg/kg for liver, 0.35 mg/kg for kidney, and 0.11 mg/kg for meat. The estimated STMR values are 0.018 mg/kg for milk, 0.042 mg/kg for liver, 0.084 for kidney, and 0.04 mg/kg for meat. No maximum residue level was recommended for fat.

*Chickens*

Three groups of four hens were given capsules containing chlormequat chloride at a dose of 0.72, 2.1, or 7.2 mg/bird per day, equal to 6, 18, and 60 ppm on a dry weight basis, for 28 consecutive days. Two additional groups of 12 hens were treated with the high dose for 28 days and slaughtered 2 or 7

days after the last dose. The doses were equivalent to 4.6, 14, and 46 ppm when calculated as chlormequat cation.

The lowest dose resulted in concentrations of chlormequat chloride residues in eggs at or above the LOQ of 0.05 mg/kg, while 0.05 mg/kg was found in liver and none in meat or fat. Plateau concentrations of 0.06 and 0.1 mg/kg were found in eggs of hens treated with the two higher doses after 1 week of dosing. The concentrations in meat and fat samples were below the LOQ of 0.05 mg/kg, while those in liver were 0.07 mg/kg at the medium dose and 0.18 mg/kg at the high dose.

The residues were cleared rapidly from meat, fat, and liver. No chlormequat chloride was determined in meat or fat. The concentrations in liver had fallen to 0.05 mg/kg 2 days after the end of dosing and to below the LOQ after 7 days. After 2 and 7 days, the residues in eggs had fallen to values below the LOQ of 0.05 mg/kg.

Egg and tissue samples were frozen on the day of sampling but were analysed in part 3 months (tissues) or 10 months (eggs) later. No adequate information on stability was received.

According to the recommendation of the 1997 JMPR, the maximum residue level and the STMR values for eggs and poultry tissues were calculated on the basis of dietary burdens of 9.6 and 1.1 mg/kg, respectively. The following table shows the highest and the mean actual and extrapolated concentrations of residues for estimation of MRLs and STMR values for chlormequat.

| Dose, ppm | Concentrations of residues, mg/kg, calculated as chlormequat cation | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Eggs | | Meat | | Liver | | Fat | |
| | Highest | Mean[1] | Highest | Mean | Highest | Mean | Highest | Mean |
| MRL | | | | | | | | |
| Extrapolated: 9.6 | 0.064 | 0.032 | <0.04 | <0.04 | 0.053 | 0.037 | <0.04 | <0.04 |
| Actual: 14 | 0.093 | 0.046 | | | 0.077 | 0.054 | | |
| STMR | | | | | | | | |
| Extrapolated: 1.1 | 0.011 | <0.04 | <0.04 | <0.04 | 0.017 | 0.0096 | <0.04 | <0.04 |
| Actual: 4.6 | 0.047 | | | | 0.07 | 0.04 | | |

[1] The mean concentration in eggs was calculated from samples taken on days 3-28.

The Meeting recommended an MRL of 0.1 mg/kg for eggs and offal, and 0.04* mg/kg for meat; no MRL was recommended for fat. The estimated STMR values were 0.04 for eggs, 0.0096 for liver, and 0 for meat. HR values of 0.064 mg/kg for eggs, 0.053 mg/kg for offal, and 0 for meat were estimated.

### *Fate of residues during processing*

In three studies of the processing of *rape seed* reported by the 1994 JMPR, the mean processing factor for crude rape seed oil was <0.018. On the basis of the STMR value of 2.0 mg/kg for rape seed, an STMR-P value of 0.037 mg/kg was estimated for crude rape seed oil.

One study on the processing of *barley* to barley pearls submitted to the 1994 JMPR indicates a processing factor of 0.06. On the basis of the STMR value of 0.15 mg/kg for barley grain, an STMR-P value of 0.009 mg/kg was estimated for barley pearl. Another study indicated processing factors of 0.69 for malt and 0.015 for beer. On the basis of the STMR value of 0.15 mg/kg for barley, STMR-P values of 0.1 mg/kg and 0.0023 mg/kg were estimated for malt and beer, respectively.

Two studies on the processing of *oats* to oat flakes were submitted to the 2000 JMPR, but only one could be used for evaluation (processing factor, 0.27) because high values were found in samples from untreated plots in the second study. Two further studies were reported by the 1994

JMPR (processing factors, 0.1 and 0.25). On the basis of an STMR value of 1.2 mg/kg for oat grains and a mean processing factor of 0.21, an STMR-P value of 0.25 mg/kg was estimated for oat flakes.

One study on the processing of *rye* was submitted to the 2000 JMPR but could not be used for evaluation because high values were found in samples from untreated plots. In a study reported by the 1994 JMPR, the processing factors were 3.2 for bran, 0.99 for flour, 1.3 for wholemeal, and 0.95 for wholemeal bread. On the basis of the MRL of 3 mg/kg for rye, the following maximum residue levels were estimated: 10 mg/kg for rye bran, 3 mg/kg for rye flour, and 4 mg/kg for rye wholemeal. On the basis of the STMR value of 0.26 mg/kg, STMR-P values were estimated as 0.83 mg/kg for rye bran, 0.26 mg/kg for rye flour, 0.34 mg/kg for rye wholemeal, and 0.25 mg/kg for rye wholemeal bread.

One study on the processing of *wheat* submitted to the 2000 JMPR showed processing factors of 2.5 for wheat bran, 1 for wholemeal, and 0.63 for wholemeal bread. In a study reported by the 1994 JMPR, processing factors of 4.6 for bran, 0.41 for flour, 1.4 for wholemeal, and 0.79 for wholemeal bread were estimated. The following processing factors were estimated: bran, 3.6; flour, 0.41; wholemeal, 1.2; and wholemeal bread, 0.71. On the basis of the MRL of 3 mg/kg for wheat grain, the following maximum residue levels were estimated: 10 mg/kg for wheat bran, 2 mg/kg for wheat flour, and 5 mg/kg for wheat wholemeal. On the basis of the STMR value of 0.26 mg/kg for wheat grain, STMR-P values of 0.94 mg/kg for wheat bran, 0.11 mg/kg for wheat flour, 0.31 mg/kg for wheat wholemeal, and 0.18 mg/kg for wheat wholemeal bread were estimated.

## RECOMMENDATIONS

The Meeting estimated the maximum residue and STMR levels shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: chlormequat cation.

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| GC 0640 | Barley | 2 | 0.5 | 0.15 | 1.8 |
| | Barley beer | | | 0.0023 | |
| | Barley malt | | | 0.1 | |
| | Barley pearl | | | 0.009 | |
| AS 0640 | Barley straw and fodder, dry | W[1] | 20 | | |
| PE 0112 | Eggs | 0.1 | - | 0.04 | 0.064 |
| MM 0814 | Goat meat | 0.2 | - | 0.04 | 0.11 |
| MO 0098 | Kidney of cattle, goats, pigs and sheep | 0.5 | - | 0.084 | 0.35 |
| MO 0099 | Liver of cattle, goats, pigs and sheep | 0.1 | - | 0.042 | 0.88 |
| AS 0645 | Maize fodder | 7 (dry wt.) | - | 2.3 (dry wt.) | |
| AF 0645 | Maize forage | 15 (dry wt.) | - | 6.5 (dry wt.) | |
| MM 0097 | Meat of cattle, pigs and sheep | 0.2 | - | 0.04 | 0.11 |
| ML 0107 | Milk of cattle, goats and sheep | 0.5 | - | 0.018 | 0.35 |
| GC 0647 | Oats | 10 | 10 | 1.2 | 7.1 |
| | Oat flakes | | | 0.25 | |
| AF 0647 | Oat forage (green) | 100 (dry wt.) | 20 | 12.7 (dry wt.) | |
| AS 0647 | Oat straw and fodder, dry[1] | W | 20 | | |
| FP 0230 | Pear[2] | 10 | 10 | 4.2 | 6.3 |
| PM 0110 | Poultry meat | 0.04* | - | 0 | 0 |
| PO 0111 | Poultry, Edible offal of | 0.1 | - | 0.0096 | 0.053 |

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| SO 0495 | Rape seed | | | 2.05 | |
| OC 0495 | Rape seed oil, crude | | | 0.037 | |
| GC 0650 | Rye | 3 | 3 | 0.26 | 2 |
| CM 0650 | Rye bran, unprocessed | 10 | 10 | 0.83 | |
| CF 1250 | Rye flour | 3 | - | 0.26 | |
| AF 0650 | Rye forage (green) | 100 (dry wt.) | 20 | 12.7 (dry wt.) | |
| AF 0650 | Rye straw and fodder, dry | W[1] | 20 | | |
| CF 1251 | Rye wholemeal | 4 | 3 | 0.34 | |
| | Rye wholemeal bread | | | 0.25 | |
| AS 0081 | Straw and fodder (dry) of cereal grains[3] | 30 (dry wt.) | - | 4.2 (dry wt.) | |
| GC 0653 | Triticale | 3 | - | 0.26 | 2 |
| GC 0654 | Wheat | 3 | 2 | 0.26 | 2 |
| CM 0654 | Wheat bran, unprocessed | 10 | 5 | 0.94 | |
| CF 1211 | Wheat flour | 2 | 0.5 | 0.11 | |
| AS 0654 | Wheat straw and fodder, dry | W[1] | 20 | | |
| CF 1212 | Wheat wholemeal | 5 | 2 | 0.31 | |
| | Wheat wholemeal bread | | | 0.18 | |

[1] Now included in recommendation for Straw and fodder (dry) of cereal grains
[2] The information provided to the JMPR precludes an estimate that the dietary intake would be below the acute RfD of 0.05 mg/kg bw
[3] Except Maize fodder

## Further work or information

*Desirable*

Analytical study of stability in frozen storage of samples of animal products fortified with chlormequat.

## Dietary risk assessment

### *Chronic intake*

STMR or STMR-P values were estimated by the present Meeting for 27 raw and processed food commodities. When data on consumption were available, these values were used to estimate dietary intake. The results are shown in Annex 3.

The IEDIs for the five GEMS/Food regional diets, based on the estimated STMR values, represented 0-3 % of the ADI. The Meeting concluded that long-term intake of residues of chlormequat from uses that have been considered by the JMPR is unlikely to present a public health concern.

### *Short-term intake*

The IESTI for chlormequat was calculated for the food commodities (and their processing fractions) for which maximum residue levels and STMR values were estimated and for which data on consumption were available. The results are shown in Annex 4. Pears were the only commodity for

which the IESTI exceeded the acute RfD, with values of 240% for the general population and 700% for children.

The Meeting concluded that short-term intake of residues of chlormequat when used, other than on pears, in ways that have been considered by the JMPR is unlikely to present a public health concern.

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# CHLORPYRIFOS (017)

## EXPLANATION

Chlorpyrifos was originally evaluated by the JMPR in 1972 with several subsequent evaluations for residues, most recently in 1995. The 1982 JMPR allocated an ADI of 0.01 mg/kg bw. At the 25th Session of the CCPR in 1993 (ALINORM 93/24A para. 251) chlorpyrifos was identified as a candidate for periodic review. The 29th Session in 1997 scheduled periodic reviews for toxicology in 1999 and for residue chemistry in 2000. The 1999 toxicology review confirmed the ADI of 0.01 mg/kg bw and also established an acute reference dose (acute RfD) of 0.1 mg/kg bw.

Information was supplied by the manufacturer on the identity and physical properties of the active ingredient and technical material, metabolism in plants and animals, environmental fate, storage stability, animal feeding studies, field trials, GAP (national labels) and fate of residues in processing. The governments of Australia, Germany, The Netherlands, Poland, Thailand and the USA provided additional information.

## IDENTITY

ISO common name: chlorpyrifos

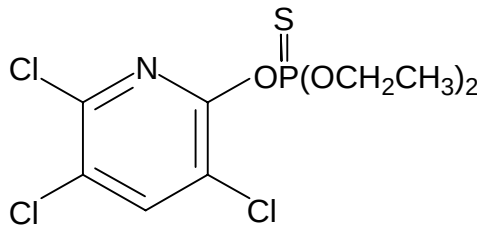
Chemical name:
- IUPAC: *O,O*-diethyl *O*-3,5,6-trichloro-2-pyridyl phosphorothioate
- CA: *O,O*-diethyl *O*-(3,5,6-trichloro-2-pyridinyl) phosphorothioate

CAS No.: 2921-88-2

CIPAC No.: 221

Synonyms: Dowco* 179, ENT 27311, OMS 971, Lorsban, Dursban

Structural formula:




Molecular formula: $C_9H_{11}Cl_3NO_3PS$

Molecular weight: 350.6

Physical and chemical properties

Pure active ingredient

Vapour pressure: $2.0 \times 10^{-5}$ mm Hg or $2.67 \times 10^{-6}$ mPa at 25°C (Chakrabarti and Gennrial, 1987)

Melting point: 42°C

Octanol/water coefficient: $P_{OW}$= 50125 (range 41335-58173) or log $P_{OW}$ = 4.7001, at 20°C (McDonald, *et al.*, 1985)

Solubility (g/100 g solvent at 25°C) in:

| | |
|---|---|
| water: | 1 x $10^{-4}$ |
| methanol: | 45 |
| ethanol: | 63 |
| xylene: | 400 |
| 1,1,1-trichloroethane: | 400 |
| diethyl ether: | 510 |
| carbon disulfide: | 590 |
| chloroform: | 630 |
| acetone: | 650 |
| methylene chloride | 714 |
| benzene: | 790 |

(Dow Chemical Co., 1987)

Specific gravity: 1.51

Hydrolysis: rate of hydrolysis is independent of pH below pH 7, with a half-life of 72 days at 25°C in sterile buffered water. The hydrolysis is base-catalysed under alkaline conditions, with a half-life of 16 days at pH 9 (McCall, 1986, GH-C 1791).

Photolysis: photolysis of chlorpyrifos in aqueous systems occurs fairly rapidly with 3,5,6-trichloro-2-pyridinol (TCP) as the main product. The average aqueous photolysis half-life of chlorpyrifos under mid-summer conditions at 40°N is about 30 days. TCP has a predicted photolysis half-life of 15 minutes based on a quantum yield study (Racke, 1993; Batzer *et al.*, 1990, GH-C 2417).

Technical material:

Appearance: white granular solid

Purity: minimum 97% (w/w) chlorpyrifos

Melting point: 41.5-43.5°C

Hydrolysis: 63-day half-life at pH 5, 23-day half-life at pH 8, phosphate buffer, 25°C). Hydrolysis rate increases approximately 3-fold for every 12°C rise. Natural waters and water containing copper or other chelating metals cause more rapid degradation than phosphate buffer, with half-lives from 1.5 days to 35 days in other media (Dow Chemical Co., 1987)

**Formulations**

Emulsifiable concentrate (EC) formulations containing 120, 225, 278 or 480 g chlorpyrifos/l

Wettable powder (WP) formulations containing 250 or 500 g chlorpyrifos/kg

Granular formulations (G) containing 50, 100 or 150 g chlorpyrifos/kg

Water-Dispersible Granule (WG) formulations containing 750 g chlorpyrifos/kg

Lorsban IPE (impregnated polyethylene), containing 1% chlorpyrifos, is a polyethylene resin impregnated with chlorpyrifos and extruded to form plastic bags for use as shrouds for bananas bunches.

## METABOLISM AND ENVIRONMENTAL FATE

### Animal metabolism

Metabolism studies on rats, hens and goats were reported. The rat study (Nolan *et al.*, 1987) was previously submitted to the WHO for review by the 1999. The main route of elimination was in the urine, 84-92% of the administered dose. No chlorpyrifos was detected in the urine, and the main metabolites were 3,5,6-trichloro-2-pyridinol (TCP), its glucuronide conjugate and possibly its sulfate conjugate.

In the poultry study (Bauriedel, 1986), 16 acclimatized White Leghorn laying hens were divided into 4 groups of 4 hens each. The hens in three of the groups each received a daily oral dose of 2.26 mg of [$^{14}$C]chlorpyrifos, labelled on the ring C adjacent to the ester bond (C-2) and on C-6. The specific activity was 16,085 dpm/µg. This corresponded to an average dietary level of 20 ppm, based on actual feed consumption during the test period of ten consecutive days. Feed consumption in the three treated groups ranged from 112 to 121 g/bird/day. Eggs and excreta were collected each day and pooled by group. About 12 hours after the final dose, the birds were killed and samples of tissues were pooled by group and stored frozen (-15$^{o}$C).

Tissues, excreta and eggs were combusted and analysed for radioactive carbon dioxide by liquid scintillation counting (LSC). The radiochemical procedures were validated by the analysis of fortified control tissue and excreta samples. At fortifications of 7500 dpm (1800 dpm for egg yolk and fat), recoveries were >90%, except from muscle at 86%. The $^{14}$C concentrations in the combined treated group tissues, expressed as chlorpyrifos, were kidney 0.154 mg/kg, liver 0.054 mg/kg, muscle 0.10 mg/kg, fat 0.198 mg/kg, skin 0.126 mg/kg, gizzard 0.024 mg/kg, and heart 0.068 mg/kg. The concentration in the gastrointestinal tract contents ranged from 0.224 to 0.393 mg/kg in the three treated groups. No radioactivity was detected in the control tissues (<0.006 to <0.015 mg/kg).

Eggs were separated into yolks and whites and combined by group and day. The radioactivity concentration in the whites reached a plateau of about 0.026 mg/kg on day 7. That in the yolks appeared to be reaching a plateau of 0.15 mg/kg on day 9 or 10. The excreta accounted for 88-94% of the total administered dose.

Tissue samples were extracted with acetone and the extracts evaporated to dryness under nitrogen, redissolved in hexane and partitioned with acetonitrile. The acetonitrile fractions from egg yolks were subjected to silica gel column chromatography. Solids from the acetone extraction of liver were extracted with methanol/6 N HCl and then with methanol. Separate liver samples were hydrolysed with 0.6 N NaOH for 3 hours at 70$^{o}$C. The hydrolysate was adjusted to pH 1 and extracted with benzene/acetonitrile (1:1).

The extracts and residual solids were radioanalysed by LSC, and combustion followed by LSC respectively. However, the study did not reveal the $^{14}$C in terms of chlorpyrifos found in the original samples and the individual extracts and residual solids, so the efficiencies of the processes could not be independently ascertained. The percentages of the TRR (total radioactive residue) in the various sample extracts were provided in flowcharts. The recoveries of the radioactive residues, based on these summary values, are shown in Table 1.

Table 1. Distribution of $^{14}C$ in extracts and solid residues from the tissues of hens dosed orally with 2.26 mg [$^{14}C$]chlorpyrifos for 10 days; 164 µCi per hen.

| Sample, and (mg/kg as chlorpyrifos) | Fraction | % of TRR |
|---|---|---|
| Liver (0.054) | Acetone | 53 |
| | Acidic methanol | 14 |
| | Methanol | 1 |
| | Solid residue | 12 |
| | Total extracted | 68 |
| | Total recovered | 80 |
| Liver, base hydrolysate | Benzene/acetonitrile | 75 |
| | Aqueous | 6 |
| | Solid residue | 12 |
| | Total extracted | 81 |
| | Total recovered | 93 |
| Egg yolk (0.15) | Acidic acetone | 99 |
| Kidney (0.15) | Acidic acetone | 85 |
| | Solid residue | 8 |
| | Total extracted | 85 |
| | Total recovered | 93 |
| Skin (0.13) | Acidic acetone | 92 |
| | Solid residue | 7 |
| | Total extracted | 92 |
| | Total recovered | 99 |
| Fat (0.20) | Acidic acetone/hexane | 105 |
| | Solid residue | 0 |

Extracts were analysed by TLC and HPLC and compounds identified by co-chromatography with authentic standards of chlorpyrifos, 3,5,6-trichloro-2-pyridinol (3,5,6-TCP), diethyl 3,5,6-trichloro-2-pyridyl phosphate (chlorpyrifos oxon) and sodium *O*-ethyl *O*-(3,5,6, trichloro-2-pyridyl) phosphorothioate. HPLC was on a C-18 column connected to a radiochemical detector, a UV detector (293 nm) and a fraction collector. TLC was on silica gel 60 F-254 plates. A TLC linear analyser was used to locate regions of radioactivity, which were scraped and analysed by LSC.

GC-MS was used only to confirm [$^{14}C$]chlorpyrifos. The compound was isolated from appropriate extracts by HPLC, gel permeation chromatography and TLC.

The compounds identified in the extracts are shown in Table 2.

Table 2. Labelled compounds identified in hen tissues and eggs.

| Sample | % of $^{14}C$ in sample | | | |
|---|---|---|---|---|
| | Chlorpyrifos (CP) | 3,5,6-trichloro pyridinol (TCP) | Unknown D | Unknown E |
| Kidney | 1 | 71 | | |
| Egg yolk | 32 | 49 | | |
| Liver | <1 | <1 | 10 | 7 |
| Liver (hydrolysed) | <1 | 64 | <1 | <1 |
| Skin | 70 | 13 | | |
| Fat | 88 | <1 | | |

In the goat metabolism study (Glas, 1981a,b; Wilkes *et al.*, 1980) two female goats were dosed orally with gelatin capsules containing 400 µl of a dosing solution containing 0.32 mCi/ml benzene (0.256 mCi/goat/day) twice a day for 10 days with [$^{14}C$]chlorpyrifos (2.99 mCi/mmole) labelled in the C-2 and C-6 positions. There was no control goat. Feed consumption was monitored.

The first goat consumed 2000 g and the second 700-2000 g each day (average 1560 ± 462 g per day). The dietary exposures were 15 ppm and 19 ppm respectively.

Milk and urine samples were collected twice daily, faeces each morning, and all were stored frozen. Within 24 hours of the final dose, the goats were slaughtered and tissues collected. Aliquots were oxidized and quantified by liquid scintillation counting. The urine and faeces contained 79-89% of the total administered dose, and about 2% was found in the milk and tissues. The residue in the milk reached a maximum on day 8 and decreased slightly thereafter. The results are shown in Table 3.

Table 3. Residues in the tissues and milk of goats dosed orally for 10 days with [$^{14}$C]chlorpyrifos at 15 and 19 ppm.

| Sample | [$^{14}$C]chlorpyrifos equivalents, mg/kg | |
|---|---|---|
| | Goat 1 (15 ppm) | Goat 2 (19 ppm) |
| Fat | 0.10 | 0.22 |
| Liver | 0.18 | 0.27 |
| Kidney | 0.26 | 0.35 |
| Muscle | 0.03 | 0.03 |
| Skin | 0.11 | 0.18 |
| Milk | 0.024 (day 8, PM) | 0.047 (day 8, PM) |

Fat, liver, kidney and milk were extracted with solvents to identify the compounds. The fat was extracted with hexane, the extract partitioned with acetonitrile, and the residual solids hydrolysed with 0.5 N potassium hydroxide for one hour at 70$^{o}$C. The hydrolysate was extracted with ether, evaporated to dryness and partitioned with hexane/acetonitrile.

Liver and kidney were each extracted with aqueous methanol (20/80), then the extracts were concentrated and extracted with diethyl ether/hexane (50/50). The residual solids were hydrolysed in the same manner as the fat solids.

The percentages of radioactivity recovered from the tissues by the solvent extractions were not reported. Muscle was not extracted because of the low radioactivity.

Four different extraction methods were used for the milk: (1) benzene from pH >12 solution; (2) basic hydrolysis (1 N potassium hydroxide, 60$^{o}$C, 3 h) followed by acidification and benzene extraction; (3) hexane extraction, partition of hexane with acetonitrile; (4) mixture with water and dilute hydrochloric acid followed by extraction with benzene. The percentages of the total radioactivity recovered by the procedures were (1) 65-72%, (2) 96-97%, (3) 55-63%, (4) 83-87%. Mild basic hydrolysis (2) released almost all of the radioactivity.

Tissue samples were hydrolysed with 0.6 N potassium hydroxide (1 h, 70$^{o}$C), and the mixture acidified and extracted with diethyl ether. The ether was evaporated to dryness and the residual oils partitioned with hexane/acetonitrile. Base hydrolysis released more than 94% of the total residue from all tissues.

Tissue and milk extracts were analysed by HPLC on a reverse-phase C-18 column. Fractions collected at one-minute intervals were measured by liquid scintillation counting and standards analysed under the same chromatographic conditions with a UV detector (293 nm).

Urine was acidified and extracted with diethyl ether. Preparative HPLC (ODS-2 reverse-phase) was used to isolate the labelled compounds.

Tentative identifications were confirmed by GC-MS in the CI mode. A splitter at the end of the GC column diverted a fraction of the column effluent to a radioactivity monitor. Purified extracts were also analysed by direct probe MS in the CI or EI mode.

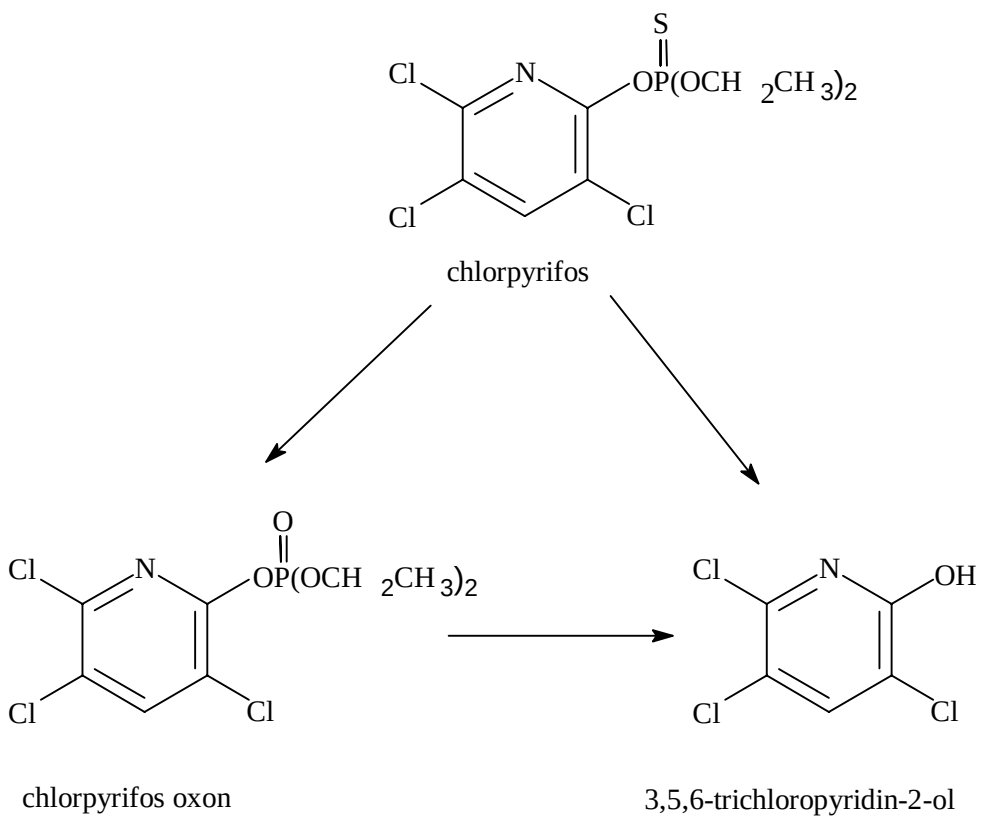
The results are shown in Table 4.

Table 4. Residues in the urine, tissues and milk of goats dosed orally for 10 days with [$^{14}$C]chlorpyrifos, expressed as % of total $^{14}$C in sample and mg/kg as chlorpyrifos..

| Sample | Compound or fraction | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Chlorpyrifos | | 3,5,6-trichloro-2-pyridinol (TCP) | | ∃-glucuronide conjugate of TCP | | *S*-ethyl, *O*-(3,5,6-trichloro-2-pyridyl) phosphorothioic acid | | Ether extract of base hydrolysate of solvent-extracted residue | | Un-extractable | | Total directly hydrolyseable to TCP | |
| | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg | % | mg/kg[1] | % of TRR |
| Urine | | 0<br>0 | | | | 90<br>80 | | 8<br>19 | | | | | | 91<br>94 |
| Milk (day 7) | 0.015<br>0.021 | 66<br>74 | 0.002<br>0.002 | 15<br>13 | | | | | | | 0.004<br>0.004 | 17<br>13 | 0.021<br>0.027 | >92<br>>94 |
| Fat | 0.07<br>0.17 | 74<br>79 | 0.01<br>0.02 | 23<br>19 | | | | | | | <0.01<br><0.01 | 2<br>2 | 0.098<br>0.22 | >98<br>>98 |
| Liver[2] | <0.01<br><0.01 | 3.5<br>0.2 | 0.09<br>0.12 | 85<br>82 | | | | | <0.01<br>0.01 | 5<br>4 | <0.01<br>0.01 | 4<br>5 | 0.17<br>0.26 | >95<br>>95 |
| Kidney[3] | <0.01<br><0.01 | 0.7<br>1.1 | 0.13<br>0.13 | 92<br>92 | | | | | <0.01<br><0.01 | 2<br>1 | 0.01<br>0.02 | 5<br>5 | 0.26<br>0.35 | >94<br>>94 |

[1] As chlorpyrifos equivalents. To convert to TCP, multiply by 0.56.
[2] Liver also contained 9.6% or 0.03 mg/kg unidentified (goat 1) and 2% or 0.01 mg/kg (goat 2).
[3] Kidney contained ≤ 0.5% (<0.01 mg/kg) unidentified.

Figure 1. Primary metabolic pathways of chlorpyrifos in livestock.



**Plant metabolism**

The metabolism of [$^{14}$C]chlorpyrifos, labelled on ring carbons 2 and 6, was studied on the leaves of maize, soya bean and sugar beet (Bauriedel, 1986). Seeds of the three crops were grown in 20 cm pots in a glasshouse. Twenty four maize plants were in a chamber which enabled volatile products to be collected. Labelled chlorpyrifos, specific activity 1.9 Ci/mole, was applied to the upper leaf surfaces as 1:l drops. The treated area was about 50 cm$^2$ on the third corn leaf, second trifoliate soya bean leaf, and fourth sugar beet leaf. Zero time samples were taken and analysed to determine the application rate (DPM). Typically, 200 :g of [$^{14}$C]chlorpyrifos was applied to each plant, and the treated leaf areas were excised, rinsed with methanol and radioanalysed after combustion or homogenized in 75% acetone at intervals to extract metabolites. The untreated plant parts were radioanalysed to ascertain the extent of translocation.

Extracts were analysed by HPLC, using a reverse-phase C-18 column, a variable wavelength UV detector and a fraction collector. One-minute fractions of eluates were collected and analysed by LSC. Standards included chlorpyrifos, chlorpyrifos oxon (diethyl 3,5,6-trichloro-2-pyridinyl phosphate), desethyl acid (*O*-ethyl *O*-3,5,6-trichloro-2-pyridinyl phosphorothioic acid, sodium salt), 3,5,6-TCP and the methoxy analogue 2,3,5-trichloro-6-methoxy pyridine ("the methoxypyridine" or TMP).

The tentative HPLC identification of 3,5,6-TCP was confirmed by GC-MS in the CI mode. Extracts were derivatized with *N*,*O*-bis(trimethylsilyl)acetamide, or BSA.

In the closed chamber experiment with corn plants, 99% of the applied radioactivity (90 :g/plant) was removed by a solvent rinse at time 0. By day 4 this had decreased to 1% and the volatile radioactivity had increased from 0 to 84%. Radioactivity in the treated leaf segment was highest 12 hours after application, 29% of that applied, and had decreased to 11% by day 4. Translocated radioactivity increased to 0.8% by day 4. The total recovery was 96%.

Plants treated with radiolabelled chlorpyrifos were analysed 8 and 16 days after application. The combined surface rinses and leaf extracts of maize, soya bean and sugar beet at day 8 contained 7.0%, 9.8% and 6.4% of the applied radioactivity, and at day 16, 5.5%, 6.9% and 6.2% respectively. Chlorpyrifos concentrations ranged from 0.1% to 4% of the applied radioactivity. HPLC analysis indicated the absence of the methoxypyridine, the oxon and the TCP. The metabolites were polar.

The polar metabolites in the extracts were separated from chlorpyrifos by evaporating the acetone solvent to dryness and partitioning the residue between ether and water, followed by acid and alkaline hydrolysis of the aqueous phase (1 N, 135$^o$C, 1 h) and enzyme hydrolysis with ∃-glucosidase (pH 5.0, 37$^o$C, 4 h). The results are shown in Table 5. No other aglycone was produced by the hydrolyses. More severe hydrolytic conditions did not produce additional 3,5,6-TCP.

Table 5. Percentage of radioactive residue in the aqueous fractions from leaf surfaces 16 days after treatment converted to 3,5,6-TCP by various treatments, as determined by HPLC.

| Treatment | % of TRR present as TCP | | |
|---|---|---|---|
| | Maize | Soya bean | Sugar beet |
| Untreated aqueous extract | 6.5 | 0.0 | 0.0 |
| Heat | 45 | 49 | 54 |
| Acid hydrolysis | 48 | 58 | 58 |
| Base hydrolysis[1] | 46 | 55 | 54 |
| Enzyme hydrolysis[2] | 38 | 30 | 25 |

[1] 2,3,5-TCP confirmed by GC-MS.
[2] ∃-glucosidase. Controls (no enzyme) generated 3.7-4.3%.

The percentage of applied radioactivity that could not be removed from the treated leaves by extraction with 75% acetone increased with time after treatment, but the insoluble portion never exceeded 3% of the applied radioactivity. The results are shown in Table 6.

Table 6. Insoluble radioactive residues.

| Days after treatment | Insoluble $^{14}C$, % of leaf content | Insoluble $^{14}C$, % of applied |
|---|---|---|
| Maize | | |
| 4 | 5.5 | 0.5 |
| 8 | 10 | 1.0 |
| 16 | 19 | 1.3 |
| 28 | 26 | 1.6 |
| Soya bean | | |
| 16 | 29 | 3.1 |
| Sugar beet | | |
| 16 | 17 | 1.4 |

The metabolism of [$^{14}C$]chlorpyrifos, labelled on ring carbons 2 and 6, was studied in field corn (Bauriedel and Miller, 1986a). The stock solution contained 0.117 g [$^{14}C$]chlorpyrifos/ml methylene chloride at a specific activity of 3.0 Ci per mole or 18996 dpm per :g. A granular formulation (3.48 ml stock solution per 8 g granules) for application to the ground and an emulsifiable concentrate formulation (8.56 ml of stock to 2 ml “Lorsban” blank) for foliar application were prepared.

The test site, a sandy loam field in Midland, Michigan, USA, consisted of four 180 cm rows with 76 cm spacing. Three of the four rows were treated at planting by T-band application. The granular formulation was sprinkled by hand into the band and furrow and immediately covered with soil previously removed from the 15 cm band. The application was equivalent to 1 lb of a 150 g/kg granular formulation per 1000 ft. row or 223 mg ai/m of row. The fourth row was planted without treatment and all four rows were surrounded by rows of untreated corn.

When the plants were about 50 cm high with 10 leaves, the untreated row and two of the treated rows were foliar-sprayed at a rate equivalent to 1.68 kg chlorpyrifos ai/ha.

At 96 days after soil application or 49 days after foliar application, green forage (3 plants) was taken from each plot. The grain was full and starting to dent. The plants were cut 7.5 cm above the soil line, shredded and homogenized, air-dried, ground to a fine powder and stored frozen.

153 days after planting, the mature crop was harvested and the fodder, without the grain, was chopped, air-dried and ground. The grain was homogenized. All samples were stored frozen.

Plant samples were extracted with acetone or diethyl ether in a Soxhlet extractor or with 75% aqueous acetone in a blender. Samples and/or extracts were hydrolysed with 1 N sodium hydroxide (1 h at 135$^{o}$C). The mixtures were neutralized or acidified and extracted with diethyl ether.

The total radiocarbon content of the plant samples was measured by combustion and LSC, and of the extracts by LSC. Extracts were analysed by HPLC, on a reverse-phase C-18 column, with a variable wavelength UV detector (280 nm) and a fraction collector. Two linear-gradient solvent systems were used. One-minute fractions were collected and analysed by LSC. Reference compounds were chlorpyrifos, the oxon, the desethyl acid, 3,5,6-TCP and the methoxypyridine. The identification of 3,5,6-TCP was confirmed by GC-MS in the CI mode.

The concentrations of $^{14}C$ (as chlorpyrifos) are shown in Table 7.

Table 7. $^{14}C$ residues in field corn samples after the application of [$^{14}C$]chlorpyrifos to the ground at 0.223 g ai/m at planting and/or to the foliage at 1.68 kg ai/ha 49 days after planting.

| Application | $^{14}C$, mg/kg as chlorpyrifos | | |
|---|---|---|---|
| | Green forage (96 days post-plant) | Dry fodder (153 days post-plant) | Grain (153 days post-plant) |
| Soil (T-Band) | 0.08 | 0.46 | 0.09 |
| Soil + Foliar | 1.62 | 4.16 | 0.13 |
| Soil + Foliar | 1.61 | 4.12 | 0.13 |
| Foliar | 2.40 | 4.04 | 0.04 |

Green forage that received both treatments was extracted with 75% acetone, releasing 28% of the TRR, about 3% of which (0.05 mg/kg as chlorpyrifos) was identified as chlorpyrifos (by HPLC) and 1% (0.02 mg/kg) as 3,5,6-TCP. About 11% of the TRR was very polar, eluting in the dead volume, and about 25% of the polar material (6% of the TRR) was base-hydrolysed to 3,5,6-TCP. Direct base hydrolysis of the green forage solubilized 91% of the TRR and 30% of the TRR (0.48 mg/kg) was identified and confirmed by GC-MS as 3,5,6-TCP.

Dry fodder that had received both treatments was extracted with 75% acetone, which released 18% of the TRR. Chlorpyrifos and 3,5,6-TCP accounted for 2% (0.08 mg/kg) and 3% (0.12 mg/kg) of the TRR respectively. Acetone extractions of a separate dry fodder sample showed 1% chlorpyrifos, 8% 3,5,6-TCP and 3% (0.12 mg/kg) of the methoxypyridine but the last was not confirmed by GC-MS. Direct base hydrolysis (1 N sodium hydroxide, 100$^{o}$C, 4 h) released 89% of the TRR; 16% of the TRR (0.67 mg/kg) was identified as 3,5,6-TCP.

Grain that received both treatments was hydrolysed (1 N sodium hydroxide, steam bath, 4 h) and then acidified and extracted with diethyl ether. Only 1.6% of the TRR was in the extract. Grain samples were sequentially extracted with ether, 75% aqueous acetone and 0.1 N sodium hydroxide. The radioactive residual solid was physically separated on 50 and 200 mesh screens. This may be indicative of incorporation of the radiolabel into protein (4% of total $^{14}C$), cellulose (14%), gluten (8%) and starch (34%).

Corn forage samples stored frozen for about 3 years were further analysed in a supplementary study (Lewer, 1990). Forage samples were extracted sequentially with acetone, acetone/water (50/50), 0.17 M sodium chloride, 0.02 M EDTA, 1.25 M NaOH, 0.1 M $NaClO_2$ and 6 M NaOH. The final residue was freeze-dried. The procedure was designed to isolate chlorpyrifos and all low molecular weight metabolites, and to solubilize high molecular weight polysaccharides, hemicellulose and lignin. Each fraction was analysed by LSC or combustion and LSC, and by HPLC. The distribution of the residues is shown in Table 8.

Table 8. Distribution of the labelled residue from the sequential extraction of corn forage (20.87 g, 1.61 mg/kg as chlorpyrifos).

| Fraction | Characterization | % of TRR | mg/kg as chlorpyrifos |
|---|---|---|---|
| Acetone | Chlorpyrifos and unconjugated metabolites | 30 | 0.48 |
| 50% aqueous acetone | Conjugated metabolites | 10 | 0.16 |
| 0.17 M sodium chloride, reflux | Polysaccharide | 10 | 0.16 |
| 0.2 M EDTA, reflux | Polysaccharide | 7 | 0.11 |
| 1.25 M sodium hydroxide | Hemicellulose | 9 | 0.15 |
| 0.1 M sodium chlorite, 80$^{o}$C | Lignin | 26 | 0.42 |
| 6 M sodium hydroxide | Hemicellulose | 1 | 0.02 |
| Residue | | 0.3 | <0.01 |
| Total $^{14}C$ recovered | | 93 | |

The extracts were analysed by HPLC but all showed similar $^{14}C$ profiles, a broad envelope of radioactivity over a 14 min range with varying amounts of 3,5,6-TCP and other compounds superimposed. The required separation was not achieved. Acidification of the sodium chlorite extract

precipitated 7.0% of the TRR, possibly in lignin. From the HPLC analyses, the amount of free 3,5,6-TCP was estimated as 3% of the TRR, and bound 3,5,6-TCP as 3.4% (from the sodium chloride and EDTA extractions). The unknown superimposed compounds were estimated to each be <0.1 mg/kg and all were more polar than 3,5,6-TCP.

In a second extraction procedure, homogenized forage was refluxed with 1 M sodium hydroxide for 2.5 h. The supernatant liquid was removed and the procedure was repeated twice on the residual solid. About 83% of the TRR was solubilized. HPLC analysis showed total 3,5,6-TCP (free and conjugated) to be 24% of the TRR, 0.39 mg/kg.

The metabolism of [$^{14}$C]chlorpyrifos, labelled on ring carbons 2 and 6, in sugar beet was studied by Bauriedel and Miller (1986b). A stock solution containing 0.117 g [$^{14}$C]chlorpyrifos per ml methylene chloride at a specific activity of 3.0 Ci per mole or 18996 dpm per :g was used to prepare a granular formulation (2.0 ml stock solution per 5 g granules) for application to the ground and an emulsifiable concentrate formulation (8.56 ml of stock to 2 ml “Lorsban” blank) for foliar application.

The trial was conducted in a field of sandy loam soil in Midland, Michigan, USA. The plot consisted of 4 1.8 m rows with 75 cm row spacing. The rows were surrounded by additional sugar beet plants. Three rows were treated at planting by T-band application. The granular formulation was sprinkled into the band and furrow at 1.0 lb formulation per 1000 feet of row or 1.126 g ai/m.

Fifty-five days after planting, chlorpyrifos was applied to the foliage of the untreated row and to two of the three treated rows at a rate equivalent to 2 pints of formulation per acre or 1.12 g ai/m. The plants nearly filled the rows.

Foliage samples were taken 38 days after planting and the remainder of the crop was harvested at maturity, 163 days after planting. Tops and beets were separated. The tops were chopped, blended and stored frozen, and the beets were washed, diced and frozen. Subsamples were radioassayed by combustion and LSC.

Samples were extracted or hydrolysed as in the corn study, and analyses were with the same HPLC system. The $^{14}$C concentrations, expressed as chlorpyrifos, are shown in Table 9.

Table 9. $^{14}$C residues in sugar beet samples after the application of [$^{14}$C]chlorpyrifos at 0.126 g ai/m at planting and/or to the foliage at 1.12 kg ai/ha 56 days after planting.

| Application | $^{14}$C as chlorpyrifos, mg/kg | | |
|---|---|---|---|
| | Green foliage (38 days post planting) | Mature tops (163 days post planting) | Mature beets (163 days post planting) |
| Soil (T-Band) | 0.61 | 0.02 | 0.11 |
| Soil + Foliar | 0.67 | 0.06 | 0.21 |
| Soil + Foliar | 0.81 | 0.04 | 0.23 |
| Foliar (56 days post plant) | - | 0.04 | 0.11 |

Extraction of green foliage with 75% acetone released about 90% of the radioactivity. HPLC analysis with two solvent systems revealed traces of 3,5,6-TCP and chlorpyrifos, and substantial amounts of polar compounds (87% of the TRR), and alkaline hydrolysis yielded 3,5,6-TCP as a major product (57% of the TRR).

Mature tops that had had both treatments were extracted sequentially with acetone and 75% acetone. The former removed chlorophyll and carotenes but only 4% of the $^{14}$C. The aqueous acetone extracted about 45% of the TRR. Solid phase extractions and HPLC analysis revealed no chlorpyrifos, traces of 3,5,6-TCP and a mixture of polar compounds. Alkaline hydrolysis (1 N sodium hydroxide)

released 65% of the TRR. HPLC analysis demonstrated that 3,5,6-TCP accounted for 29% of the TRR.

Freeze drying of the beet samples yielded 2-4% of the radioactivity in the cold trap. HPLC, confirmed by GC-MS, identified the methoxypyridine.

Methanol extraction of the beets released 85% of the TRR. Upon cooling, sucrose crystallized (with 10 g of dry beet yielding 8 g sucrose), and was recrystallized and analysed for radioactivity. Alkaline hydrolysis of the sucrose did not produce an aglycone fraction. The highest ratio of sucrose to beet radioactivity, 0.51, was in the samples which received soil treatment only, and the lowest, 0.16, was in those receiving only foliar treatment. This suggests incorporation of radioactivity from the soil. Sucrose accounted for about 40% of the TRR in beets receiving both soil and foliar treatments.

HPLC analysis of the residual liquid from the sucrose crystallization showed the presence of the methoxypyridine (7% of the TRR, including the freeze-dried portion), 3,5,6-TCP (36% of the TRR) and chlorpyrifos (<0.5%). A separate beet sample was extracted with acetone to ascertain whether the methanol solvent converted the pyridinol to the methoxypyridine and similar results were obtained.

A beet sample that had received both treatments was hydrolysed with sodium hydroxide, yielding only 3,5,6-TCP. The proportion of the label released by the hydrolysis was not reported.

A Golden Delicious apple tree (1.8 x 1.5 m) was sprayed nine times during the 1980 season with a WP formulation at 0.1 kg ai/hl chlorpyrifos (Bauriedel and Miller, 1980). The 8th and 9th applications, made 10 days apart, were with [$^{14}$C]chlorpyrifos labelled on C-2 and C-6 of the pyridinyl ring. The treatment solution contained 180 mg of the labelled chlorpyrifos mixed with 180 mg of blank formulation suspended in 300 ml water. Fourteen days after the final application 155 apples were picked and stored in a refrigerator. The total radioactivity in the apples is shown in Table 10.

Table 10. $^{14}$C residues in apples picked 14 days after two treatments with [$^{14}$C]chlorpyrifos.

| No. of apples | $^{14}$C, mg/kg as chlorpyrifos | | |
|---|---|---|---|
| | Peel | Pulp and seed | Whole apple |
| 14 | 0.72 | 0.005 | 0.09[1] |
| 12 | 0.86 | 0.005 | 0.09[1] |
| 25 | - | - | 0.14 |
| 4 individual apples | 1.1±0.6 | 0.004±0.002 | - |

[1] Calculated from residues in peel and pulp/seed.

Duplicate samples of apple peel (142 g and 110 g) were sequentially extracted with acetone and 50% aqueous methanol. The acetone extract was concentrated and extracted with ethyl ether. The ether phase contained 48-50% of the total $^{14}$C activity in the peel. The residual aqueous phase contained 8-10%. The methanol did not extract any more activity. The methanol-peel mixture was refluxed for 2 hours with 0.4 N or 1.0 N sodium hydroxide, centrifuged and the insoluble material washed with methanol and dried in a vacuum. The alkaline supernatant was concentrated, made acidic (pH 2) and extracted with diethyl ether. This ether fraction contained 35-37% of the total radioactivity in the peel. The residual aqueous fraction contained 1.4-1.5% and the residual solid contained 3.3-4.5%.

The extracts were analysed by HPLC on a C-18 reverse-phase column with a linear gradient of aqueous methanol containing 0.01 M ammonium acetate. One-minute eluate fractions were collected and analysed by LSC. Identifications of chlorpyrifos and 3,5,6-TCP were confirmed by GC-MS in the CI mode. The identifications and characterizations are shown in Table 11.

Table 11. Identification and characterization of the labelled residue isolated from the peel of apples treated with [$^{14}$C]chlorpyrifos.

| Metabolite | % of $^{14}$C in peel | $^{14}$C, mg/kg as chlorpyrifos, in peel (total 0.79 mg/kg) | Characterization |
|---|---|---|---|
| Chlorpyrifos | 36 | 0.28 | |
| 3,5,6-TCP (free) | 5.3 | 0.04 | |
| 3,5,6-TCP (conjugated) | 1.2 | <0.01 | |
| Unknown A-1 | 4.2 | 0.03 | Metabolite with 2 Cl, polar |
| Unknown A-2 | 4.2 | 0.03 | Metabolite with 2 Cl, polar |
| Unknown B | 5.2 | 0.04 | Found in the aqueous fraction from the acetone extraction. Converted to 3,5,6-TCP by refluxing with 1 N NaOH for 2 h |
| Unknown C | 5.4 | 0.04 | Formed from the base hydrolysis of the post-extraction methanol/peel mixture. Natural incorporation postulated because of lack of 3,5,6-TCP in mixture. |
| Unknown D | 4.3 | 0.03 | |
| Unknown E | 5.6 | 0.04 | |

Soya beans were sprayed mid-season 1980 with an EC formulation containing [$^{14}$C]chlorpyrifos labelled on C-2 and C-6 of the pyridinyl ring at 1.12 kg ai/ha (Bauriedel and Miller, 1981). The specific activity of the labelled chlorpyrifos was 2.5 Ci per mole and 520.5 mg plus 1 ml of emulsifiable concentrate blank and 120 ml of water was applied to 4.6 m$^2$ of crop. A forage sample was taken 14 days after treatment and beans and field trash were collected at normal harvest 52 days after treatment. Trash was defined as stems, branches, husks and a few leaves and petioles. Subsamples were dried, combusted and assayed by LSC. The remaining samples were stored frozen. The results are shown in Table 12.

Table 12. Total $^{14}$C as chlorpyrifos in soya beans after mid-season foliar application at 1.12 kg ai/ha.

| Sample | $^{14}$C, mg/kg, as harvested | $^{14}$C, mg/kg, dry basis |
|---|---|---|
| Forage homogenate | 5.09 | 25.9 |
| Forage leaves, top | | 112 |
| Forage leaves, middle | | 45.8 |
| Forage leaves, bottom | | 24.8 |
| Forage seeds-pods | | 4.4 |
| Forage stem-petiole | | 11 |
| Beans | | 0.50 |
| Field trash | | 4.15 |

Beans were extracted sequentially with n-hexane, methanol and 50% aqueous methanol and the residual solid was assayed for radioactivity. Forage and field trash were extracted sequentially with acetone and 50% aqueous methanol, and the residual solids and aqueous fractions after solvent extraction were radioassayed. The residual solids were all hydrolysed for 2 hours in 1 N sodium hydroxide in 80% aqueous methanol. The filtrates were concentrated, acidified to pH 2 and extracted with diethyl ether. The aqueous layers from these extractions were also base-hydrolysed.

Non-polar metabolite fractions were cleaned up by sublimation or steam distillation. Aqueous (polar) fractions were prepared for HPLC analysis by vacuum evaporation and re-solution in 30% aqueous methanol. Polar fractions from bean samples required C-18 SPE clean-up.

Extracts were analysed by HPLC and TLC, and GC-MS (CI mode) was used to confirm the identities of chlorpyrifos and 3,5,6-TCP. HPLC separations were on a reverse-phase C-18 column with a linear gradient of aqueous methanol containing 0.01 M ammonium acetate. Eluates were collected at one minute intervals and assayed by LSC. Non-polar fractions were also analysed by TLC.

A portion of the soya bean oil from the beans in the hexane fraction was hydrolysed and extracted to isolate glycerol and fatty acids. A portion of the soya bean insolubles after the solvent extractions was treated with sodium hydroxide to isolate protein (globulin). The purified natural products were radioanalysed. The $^{14}C$ concentration in the glycerol and fatty acids was about the same as in the oil, suggesting natural incorporation.

The distribution of the $^{14}C$ activity in the extracts and hydrolysates is shown in Table 13. The data were inadequate to determine the overall recovery.

Table 13. Distribution of $^{14}C$ in the extracts of soya bean samples.

| Fraction | $^{14}C$, % of TRR[1] | $^{14}C$, mg/kg as chlorpyrifos |
|---|---|---|
| Soya bean | | |
| Hexane | 19.7 | 0.10 |
| Acetone | 20.1 | 0.10 |
| 50% methanol | 21.2 | 0.11 |
| Alkaline hydrolysis | 31.0 | 0.16 |
| Residual solid | 8.0 | 0.04 |
| Forage | | |
| Acetone | 64.2 | 3.3 |
| 50% methanol | 12.4 | 0.63 |
| Alkaline hydrolysis | 18.4 | 0.94 |
| Residual solid | 5.0 | 0.25 |
| Field trash | | |
| Acetone | 32.7 | 1.4 |
| 50% methanol | 45.3 | 1.9 |
| Residual solid | 10.2 | 0.43 |

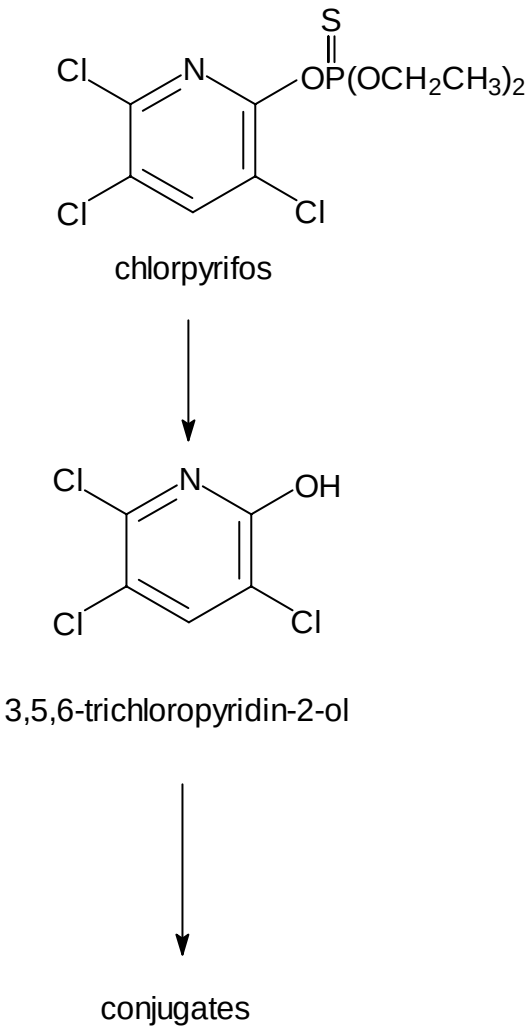
[1]Reported values appear to have been normalized to 100% total recovery.

Table 14 shows the distribution of $^{14}C$ in the soya bean samples.

Table 14. Identification and characterization of $^{14}C$ residues from the foliar mid-season application of [$^{14}C$]chlorpyrifos to soya beans at 1.12 kg ai/ha.

| Compound or fraction | $^{14}C$, % of TRR and mg/kg as chlorpyrifos | | | | | |
|---|---|---|---|---|---|---|
| | Bean (0.50 mg/kg) | | Forage (5.09 mg/kg) | | Field trash (4.15 mg/kg) | |
| | % of TRR | mg/kg | % of TRR | mg/kg | % of TRR | mg/kg |
| Chlorpyrifos | 2.6 | 0.013 | 36 | 1.8 | 29 | 1.3 |
| 3,5,6-TCP (free) | 8.8 | 0.044 | 5.7 | 0.29 | 6.0 | 0.25 |
| 3,5,6-TCP (conjugated) | 0.0 | 0.0 | 18 | 0.92 | 25 | 1.1 |
| Most abundant unknown | 6.8 | 0.034 | 6.6 | 0.34 | 4.6 | 0.19 |
| Incorporated $^{14}C$-activity (protein) | 66.0 | 0.33 | - | - | - | - |

Figure 2. Proposed metabolic pathway of chlorpyrifos in plants.




**Environmental fate in soil**

A laboratory study compared the degradation of chlorpyrifos in different soils under aerobic, aerobic/anaerobic and anaerobic conditions (Bidlack, 1979). For the aerobic experiments, 50 g soil samples were treated with [$^{14}$C]chlorpyrifos at 7.6 kg/ha and incubated at 75% of water capacity at 0.33 bar at 25°C in the dark. For the aerobic/anaerobic experiments, Commerce loam or Stockton Clay adobe soil (100 g) fortified with [$^{14}$C]chlorpyrifos at 7.6 kg/ha was incubated aerobically for 30 days; 1 g of dry alfalfa was then added to each soil, which was covered with 100 ml of water. The soil containers were purged with nitrogen and incubated. For the anaerobic experiments, the same soils (100 g) were mixed with alfalfa (1 g) and flooded with 100 ml water. The flasks were purged with nitrogen, sealed and incubated at 25$^{o}$C in the dark until anaerobic gases evolved when [$^{14}$C]chlorpyrifos was added at 7.6 kg/ha, the flasks were re-purged with nitrogen and sealed and incubation was resumed. In all cases $CO_2$ production was monitored by sodium hydroxide solution traps on the incubation flasks.

Soils and sodium hydroxide solutions were assayed for radioactivity by combustion and LSC, and by LSC respectively. The soils were extracted sequentially with 1.5 M phosphoric acid/diethyl ether and 6.5 N sodium hydroxide/diethyl ether, and the extracts analysed by TLC.

The half-life of chlorpyrifos under aerobic conditions varied from 11 days in Commerce loam to 141 days in German 2:3 standard soil (sandy loam). The half-life in Stockton clay adobe was 107

days. The average for the seven soil types was 63 days. The half-lives under aerobic/anaerobic conditions were 58 days for Stockton clay adobe and 15 days for Commerce loam, and under anaerobic conditions 51 days and 39 days. Degradation did not follow first-order kinetics. A two-compartment model was applied.

The distribution of the radioactive compounds in the Commerce and Stockton soils at various times under aerobic and anaerobic conditions is shown in Tables 15-17.

Table 15. Identification of the residue after application of [$^{14}$C]chlorpyrifos to Commerce loam and Stockton clay soil, aerobic conditions.[1]

| Compound | Commerce loam/Stockton clay, % of applied radioactivity | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | Days after treatment | | | | | | | |
| | 0 | 7 | 14 | 30 | 60 | 120 | 270 | 360 |
| Chlorpyrifos | 99/95 | 65/89 | 45/85 | 20/77 | 11/66 | 6/46 | 2/30 | 2/24 |
| Pyridinol (3,5,6-TCP) | 3.2/1.1 | 29/5.1 | 38/7.8 | 11/12 | 4.3/15 | 2.5/18 | 1.5/20 | 0.9/22 |
| Methoxypyridine (TMP) | 0/0 | 0/0 | 0/0 | 1.6/<0.1 | 1.5/1.3 | 1.6/2.9 | 0.5/4.5 | <0.1/4.6 |
| $CO_2$ | 0/0 | 2/0.6 | 10/1.5 | 57/3.9 | 74/8.1 | 82/14 | 83/26 | 88/27 |
| Unidentified[2] | 0.7/2.5 | 3.9/4.5 | 4.6/5.6 | 6.8/5.7 | 6.6/6.7 | 5.7/9.7 | 3.7/8.0 | 7.0/17 |
| Unextracted | 0.9/2.8 | 3.0/3.9 | 3.1/4.5 | 4.0/4.9 | 3.7/5.4 | 3.2/10 | 3.7/12 | 4.0/11 |
| Total | 104/101 | 103/103 | 101/104 | 100/104 | 102/101 | 101/101 | 94/100 | 102/106 |

[1] The other five soils generally yielded results between the loam and clay. Barnes loam and Catlin silty clay loam contained more methoxypyridine, 11% and 6% maximum at 120 days respectively.
[2] Includes radioactivity found on TLC plates at $R_f$ 0.11, 0.40 and at the point of application, and radioactivity extracted by cold sodium hydroxide solution but not extractable into diethyl ether.

Table 16. Identification of the residue from the application of [$^{14}$C]chlorpyrifos to Commerce loam and Stockton clay soil, anaerobic conditions.

| Compound | Commerce loam/Stockton clay, % of applied radioactivity | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Days after treatment | | | | | | | | |
| | 0 | 7 | 14 | 30 | 60 | 90 | 180 | 270 | 360 |
| Chlorpyrifos | 91/92 | 79/80 | 71/74 | 65/73 | 36/48 | 22/37 | 12/22 | 4.6/16 | 2.1/12 |
| Pyridinol (3,5,6-TCP) | 1.1/2.2 | 12/ 9.2 | 20/14 | 26/20 | 56/40 | 72/51 | 78/57 | 93/51 | 92/64 |
| Methoxypyridine (TMP) | 0/0 | 0/0 | 0.7/0 | 0/0 | 0/0 | 0/0 | 0/0 | 0/0 | 0/0 |
| $CO_2$ | 0/0 | 0.1/0.1 | 0.1/0.1 | 0/0.2 | 0.1/0.2 | 0.4/0.3 | 0.4/0.8 | 0.6/0.8 | 0.6/1.1 |
| Unidentified[1] | 0.8/0 | 2.3/6.3 | 2.9/1.2 | 1.1/4.4 | 3.2/2.3 | 2.0/4.3 | 2.8/6.2 | 0.6/9.9 | 7.5/6.1 |

| Compound | Commerce loam/Stockton clay, % of applied radioactivity | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | Days after treatment | | | | | | | | |
| | 0 | 7 | 14 | 30 | 60 | 90 | 180 | 270 | 360 |
| Unextracted | 0.8/2.4 | 1.8/4.8 | 1.4/2.3 | 2.4/10 | 0.5/4.5 | 0.5/2.6 | 0.5/6.7 | 0.9/5.0 | 0.8/11 |
| Total | 94/97 | 95/100 | 95/92 | 94/108 | 96/95 | 97/95 | 94/93 | 100/83 | 101/82 |

[1] Includes radioactivity found on TLC plates at $R_f$ 0.11, Rf 0.40 and at the point of application, and radioactivity extracted by cold sodium hydroxide solution but not extractable into diethyl ether.

Table 17. Identification of residue from the incubation of [$^{14}$C]chlorpyrifos to Commerce loam and Stockton clay soil, aerobically for 30 days, then anaerobically for 330 days.

| Compound | Commerce loam/Stockton clay, % of applied radioactivity | | | | | |
|---|---|---|---|---|---|---|
| | Days after treatment | | | | | |
| | 30 | 60 | 90 | 180 | 270 | 360 |
| Chlorpyrifos | 29/46 | 19/47 | 12/39 | 9.9/13 | 3.0/22 | 2.8/13 |
| Pyridinol (3,5,6-TCP) | 24/29 | 36/30 | 42/13 | 48/22 | 35/54 | 55/49 |
| Methoxypyridine (TMP) | 0.2/0 | 0/0.8 | 0/0 | 0.1/0.2 | 0.6/0.1 | 0/0.1 |
| $CO_2$ | 32/4.5 | 32/4.8 | 33/5.8 | 31/5.9 | 25/6.2 | 30/6.1 |
| Unidentified[1] | 4.6/5.1 | 5.7/3.1 | 4.6/4.8 | 3.3/6.9 | 9.9/8.3 | 7.3/13 |
| Unextracted | 4.1/2.8 | 1.9/4.7 | 1.8/3.5 | 4.0/5.9 | 4.3/12 | 4.1/16 |
| Total | 94/87 | 95/90 | 93/66 | 96/54 | 78/103 | 99/97 |

[1] Includes radioactivity found on TLC plates at Rf 0.11, Rf 0.40 and at the point of application, and radioactivity extracted by cold sodium hydroxide solution but not extractable into diethyl ether

Numerous other studies have been conducted on the degradation of chlorpyrifos in soil (Ware, 1993) with calculated half-lives of chlorpyrifos ranging from 1.9 to 1600 days, depending on soil type and environmental conditions.

The degradation of the metabolite TCP in soil has been studied by Ware (1993). Summary information states that the half-life of TCP in 15 different soils at a concentration of 1 mg/kg ranged from 10 to 325 days, with an average of 73 days. During the 100-day incubation, mineralization to carbon dioxide accounted for 8-77% of the applied material. Another study (Ware, 1993) considered TCP mineralization in 29 different soils. Between 2.4 and 45% of the applied 1 mg/kg TCP was mineralized to carbon dioxide during 21 days of incubation. There was no reliable correlation with pH, percentage of organic carbon, cation-exchange capacity, texture, moisture-holding capacity, or sorptive capacity. A microbiological factor was strongly indicated.

The volatility of [$^{14}$C]chlorpyrifos from soil has been reported by McCall, *et al.* (1985). The [$^{14}$C]chlorpyrifos labelled on C-2 and C-6 of the pyridinyl ring had a specific activity of 1.99 mCi/mole and was mixed with unlabelled material in acetone to a concentration of 3.67 mg/ml. There was no formulation blank. Ninety µl of the acetone solution (330:l) was applied to the surface of 55 g of soil 2.5 cm deep, adjusted to 100% of 1/3 bar moisture, to simulate an application rate of 1.12 kg

ai/ha. The sealed apparatus was swept with air at 1.0 km/h and polyurethane plugs were used to capture the volatilized chlorpyrifos. The plugs were extracted with acetone and the extract was radioassayed.

The volatility of chlorpyrifos in various soils is shown in Table 18. The data fit first-order kinetics. Half-lives ranged from 45 to 163 hours, equivalent to 80, 290 and 260 g volatilized/ha/day from German, Kawkawlin and Commerce soils respectively.

Table 18. Volatility of [$^{14}$C]chlorpyrifos applied at 1.12 kg/ha.

| % chlorpyrifos remaining in soil | | | | | |
|---|---|---|---|---|---|
| Time (h) | Mississippi Commerce loam | Time (h) | German sandy loam | Time (h) | Michigan Kawkawlin sandy clay loam |
| 0.0 | 100 | 0.0 | 100 | 0.0 | 100 |
| 2.5 | 98 | 3.0 | 99 | 2.0 | 98 |
| 7.0 | 94 | 6.0 | 98 | 4.0 | 95 |
| 10.5 | 89 | 8.0 | 97 | 8.5 | 89 |
| 24.0 | 71 | 23 | 90 | 24 | 70 |
| 36.0 | 62 | 29 | 89 | 32 | 62 |

In a confined rotational crop trial (Hamburg, 1994, GH-C 3284) [$^{14}$C]chlorpyrifos, labelled on C-2 and C-6 of the pyridinyl ring, with a specific activity of 2.45 mCi/mmol, or 15522 dpm/:g was diluted in acetone and the 12 test plots were treated with 366 mg. Each plot consisted of plywood boxes lined with polyethylene and filled with sandy loam soil (75 cm x 90 cm x 5 cm). Three plots were untreated controls. The remaining plots were situated 76 m downwind of the controls and were sprayed with the test solution (110 ml per plot).

Each of the treated plots was planted with a single crop of carrots, lettuce or wheat 30 or 132 days after treatment (DAT). Before the plantings, the soil in each plot was tilled to a depth of 7.5 cm and sampled. Lettuce and carrot seeds were planted in five planting lines to depths of 0.5-0.6 cm and 1.0 cm respectively. Wheat seed was broadcast and pushed to a depth of 3.2 cm. The plots were maintained in secured greenhouses in Watsonville, California. Immature (tiller stage) and mature wheat, 50% mature and mature carrots and mature lettuce were harvested from both plantings with control samples, and frozen until processed.

The total radioactivity in the soil at various intervals, in immature carrot roots, mature carrot roots and tops, lettuce, wheat forage (immature), grain and straw/chaff was determined by combustion and LSC. The radioactive residues were extracted with 50% aqueous acetone and water, and the extracts cleaned up by SPE. The SPE eluates and the residual solids from the acetone/water and water extractions were refluxed with 1.0 N sodium hydroxide for 4 hours, neutralized and extracted with ethyl acetate The total $^{14}$C residues and their distribution in the extracts are shown in Table 19.

Table 19. Total residues in carrots, wheat and lettuce planted 30 and 132 days after treating the soil with [$^{14}$C]chlorpyrifos at 5.3 kg ai/ha.[1]

| Crop | DAT/ DAA[2] | TRR, mg/kg as chlor-pyrifos | % of TRR in acetone /water | % of TRR in water | % of TRR in residual solid 1 | % of TRR in hydrolysate of residual solid 1 in ethyl acetate/in water | % of TRR residual solid 2 after hydrolysis | % of TRR extracted |
|---|---|---|---|---|---|---|---|---|
| Immature carrot root | 30/77 | 0.54 | 50 | 3.3 | 38 | 12/19 | 15 | 84 |
| | 132/209 | 0.38 | 70 | 3.3 | 30 | - | - | 73 |
| Carrot root | 30/138 | 0.19 | 76 | 4.7 | 25 | 5.2/10 | 7.1 | 96 |
| | 132/259 | 0.28 | 72 | 3.8 | 19 | 4.0/12 | 3.1 | 92 |
| Carrot tops | 30/138 | 0.61 | 35 | 8.4 | 50 | 14/17 | 19 | 74 |
| | 132/259 | 0.38 | 52 | 7.4 | 35 | 10/14 | 7.8 | 83 |
| Lettuce | 30/75 | 0.23 | 51 | 5.6 | 52 | 12/26 | - | 95 |
| | 132/187 | 0.083 | 34 | 6.3 | 57 | - | - | 40 |

| Crop | DAT/ DAA[2] | TRR, mg/kg as chlor-pyrifos | % of TRR in acetone /water | % of TRR in water | % of TRR in residual solid 1 | % of TRR in hydrolysate of residual solid 1 in ethyl acetate/in water | % of TRR residual solid 2 after hydrolysis | % of TRR extracted |
|---|---|---|---|---|---|---|---|---|
| Immature wheat (forage) | 30/60 | 0.66 | 52 | 6.4 | 26 | 9.9/6.2 | 7.5 | 74 |
| | 132/172 | 0.26 | 43 | 6.5 | 43 | - | - | 50 |
| Wheat grain | 30/138 | 0.30 | 22 | 17 | 76 | 6.6/54 | 14 | 100 |
| | 132/270 | 0.43 | 19 | 2.9 | 80 | - | - | 22 |
| Wheat straw/chaff | 30/138 | 0.80 | 35 | 8.0 | 48[3] | 11/11 | 21[4] | 65 |
| | 132/270 | 1.3 | 40 | 11 | 46 | - | - | 51 |

[1] Rate is 5.3 times the seasonal direct application rate for wheat in the USA. No GAP for carrot or lettuce.
[2] DAT: interval from application of chlorpyrifos to the soil to planting. DAA: interval from application of chlorpyrifos to harvest of the crop.
[3] Further characterized as 7-13% cellulose and 7.3- 17% lignin.
[4] Further characterized as 9.3-11% cellulose and 6.9% lignin.

The solid residue from the base hydrolysis of some samples was further analysed for cellulose. Buffered potassium permanganate and *tert*-butanol were used to oxidize and dissolve the non-cellulose components of the pellets. The cellulose residues were demineralized and washed in oxalic acid and hydrochloric acid in ethanol.

Cellulose and lignin were isolated from the residual solids from the acetone/water and water extractions. The solids were autoclaved with 2.5 M sodium hydroxide, filtered, and the filter cake (cellulose) washed with boiling 2.5 M sodium hydroxide and water. The lignin in the filtrate was precipitated with concentrated hydrochloric acid.

Cellulose and lignin were also isolated by extraction with 2 M hydrochloric acid/1,4-dioxane (1:9) by shaking for 5 hours at 70°C. The residue was collected as cellulose. The supernatant was concentrated and added to diethyl ether and the residue collected as lignin.

Starch was isolated from the residue from the neutral solvent extraction of wheat grain by extraction with dimethylsulfoxide:water (90:10). The addition of ethanol to the extract precipitated the starch.

Cellulose and starch samples were broken down by treatment with acid and then derivatized with phenylhydrazine to produce osazones suitable for HPLC and GC-MS analyses.

HPLC, one-dimensional TLC and GC-MS were used to analyse the extracts and derivatives. Reversed-phase or amino-bonded columns were used for HPLC and fractions were collected at one-minute intervals and analysed by LSC. TLC was used for co-chromatography of suspected TCP with an authentic TCP standard, and GC-MS to confirm the identity of TMP and TCP.

The identification or characterization of the labelled residue in the rotational crops is shown in Table 20.

Table 20. Identification of labelled residues in crops planted in soil 30 or 132 days after treatment with [$^{14}$C]chlorpyrifos.

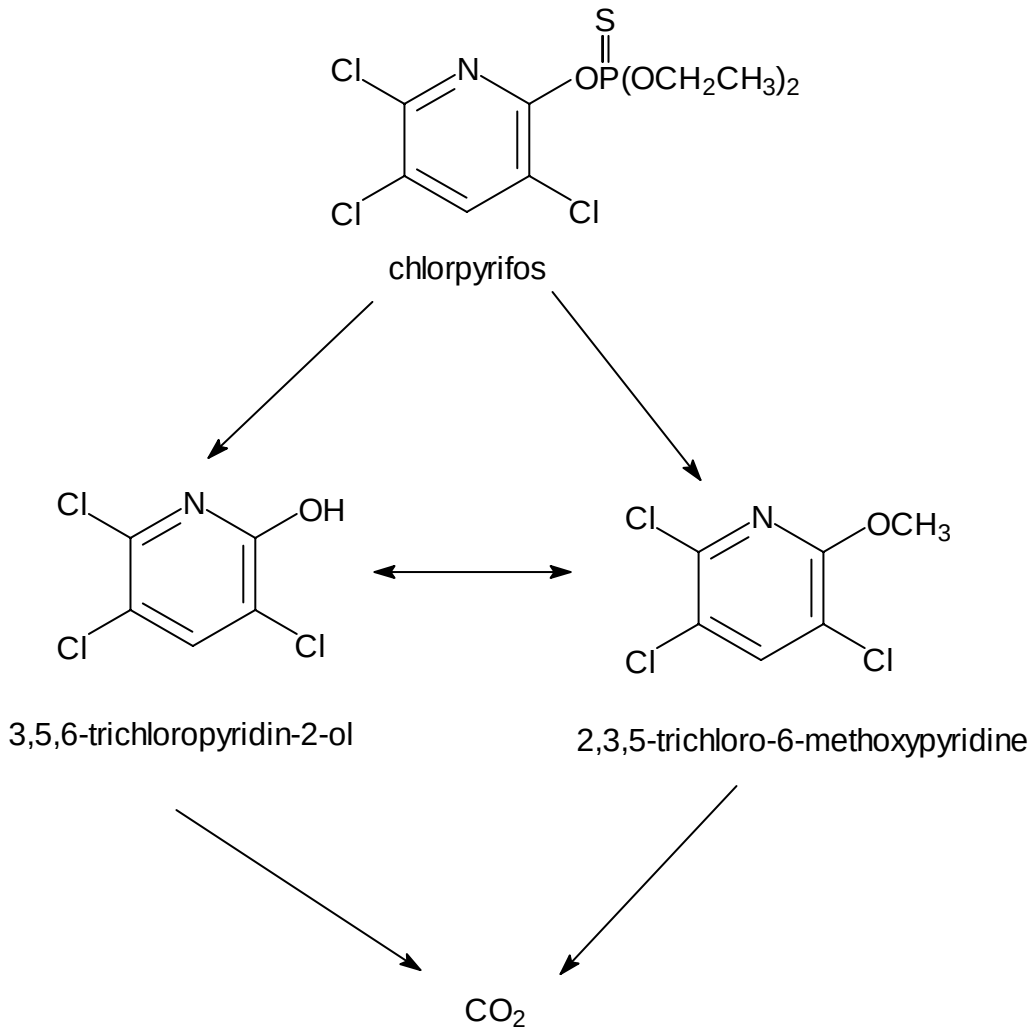
| Compound or fraction | $^{14}$C, % of TRR/mg/kg as chlorpyrifos, in | | | | | |
|---|---|---|---|---|---|---|
| | 30 DAT Carrot root | 132 DAT Carrot root | 30 DAT Lettuce | 30 DAT Wheat forage | 30 DAT Wheat straw | 30 DAT Wheat grain |
| Chlorpyrifos | 2.0/0.004 | 0 | 0 | 0 | 0 | 0 |
| TCP | 10/0.019 | 7.3/0 .020 | 5.3/0.012 | 8.1/0.054 | 4.3/0.035 | 0.3/0.001 |
| TMP | 26/0.050 | 36/0.10[2] | | | | 0 |
| Cellulose | | | | 4.6/0.030 | 9.3-13/ 0.059-0.11 | 8.5/0.025 |

| Compound or fraction | $^{14}C$, % of TRR/mg/kg as chlorpyrifos, in | | | | | |
|---|---|---|---|---|---|---|
| | 30 DAT Carrot root | 132 DAT Carrot root | 30 DAT Lettuce | 30 DAT Wheat forage | 30 DAT Wheat straw | 30 DAT Wheat grain |
| Lignin | | | | | 6.9-17/ 0.055-0.14 | |
| Starch | | | | | | 46/0.14 |
| Glucose[1] | 21/0.040 | 28/0.08 | 32/0.075 | | 21/0.16 | 49/0.14 |
| TOTAL | 59% | 71% | 37% | 13% | 42-55% | 104% |

[1]Glucose identification is tentative, based on extract fraction and HPLC retention time.
[2]Concentration of TMP in carrot peel was 54% of TRR or 0.14 mg/kg, and in peeled carrots 13% of TRR or 0.022 mg/kg.

The proposed pathways of degradation of chlorpyrifos in soil are shown in Figure 3. The data show that chlorpyrifos is degraded in large part by hydrolysis to TCP, which can then be further degraded to $CO_2$ or methylated to form TMP, which itself may be reversibly changed to the pyridinol or possibly degraded to $CO_2$.

Figure 3. Proposed pathways of degradation of chlorpyrifos in soil.




**Environmental fate in water/sediment systems**

The manufacturer did not provide any original studies, but submitted a review by Ware (1993) on which the following information on the environmental fate in water/sediment systems is based.

Owing to its non-polar nature, chlorpyrifos has a low solubility in water and great tendency to partition from aqueous into organic phases in the environment. It has a strong affinity for soil, as shown by an average soil and sediment adsorption coefficient ($K_{OC}$) of 8498 ml/g, based on 28 laboratory studies by the batch equilibrium method. The coefficient ranged from 973 to 31000. TCP has only a moderate affinity for soil, with values of $K_{OC}$ of 18.5-389 ml/g, average 159 ml/g.

A typical leaching trial of several in the laboratory and in the field was with Commerce loam (0.68% organic carbon), Tracy sandy loam (1.12% organic carbon) and Catlin silty clay loam (2.01% organic carbon) treated with [$^{14}$C]chlorpyrifos (0.5 kg/ha) and eluted with 51 cm of water through 30 cm glass columns packed with the same soils. 95-99% of the chlorpyrifos remained in the top 2 cm of the column, with none below the top 5 cm. A maximum 1.3% of the applied radioactivity was in the leachates. In field leaching studies under natural rainfall and irrigation conditions at 1.1-2.2 kg ai/ha, chlorpyrifos remained in the top 20 cm layer of soil throughout the growing season. One field trial indicated that TCP has at least a moderate tendency to be leached. In a citrus grove trial chlorpyrifos was applied three times at 1.12 kg ai/ha during the growing season with rainfall and irrigation of 110 and 48 cm respectively. Chlorpyrifos was confined to the upper 15 cm of soil, but TCP was found at depths of 46 cm.

## METHODS OF RESIDUE ANALYSIS

### Analytical methods

Methods for the determination of chlorpyrifos in plant and animal tissues, water and soil have been developed. Chlorpyrifos is determined by gas chromatography, generally with flame photometric detection. Extraction and clean-up methods may vary, depending on the sample. The limit of quantification (LOQ) is usually 0.01 mg/kg. The methods used in the determination of residues of chlorpyrifos in supervised field trials are described below and summarized in Table 21. All analyses are by GLC with flame photometric detection, except in methods ACR 71.1, ERC 87.05 and BRC 93.1 where an ECD is used.

ACR 71.1-Animal tissues, including milk and cream
Chlorpyrifos is extracted from the sample with hexane in the presence of sodium chloride, and partitioned into acetonitrile. Materials interfering with gas chromatographic analysis are removed by clean-up on a silica gel column. A second silicic acid column is required for complete clean-up of cream samples. Residues are determined by electron-capture gas chromatography.

ACR 71.14-Peaches
The sample is blended with acetone. The extract is filtered and the filtrate partitioned with an equal volume of methylene chloride. The acetone-methylene chloride layer is percolated through anhydrous sodium sulfate and then evaporated to dryness with a jet of dry air. The residue is taken up in hexane and partitioned into acetonitrile. The combined acetonitrile extracts are evaporated to dryness, the residue is redissolved in acetone for analysis.

ACR 72.1-Pork muscle, liver, kidney and fat
ACR 72.3-Poultry tissues and eggs
Chlorpyrifos is extracted by blending with acetone or hexane. The extracts are filtered. The hexane filtrate is evaporated with a jet of dry air and the acetone filtrate partitioned with an equal volume of methylene chloride, percolated through anhydrous sodium sulfate and evaporated to dryness with a jet of dry air. The residues are taken up in hexane and cleaned up by acetonitrile partition and a silica gel column. The solvent is evaporated and the analysis completed as in ACR 71.4.

ACR 72.9-Sweet corn forage, kernels and husk
The sample is blended with acetone. The extract is filtered and the filtrate is concentrated by evaporation with a jet of dry air. The acetone filtrate is transferred to an aqueous sodium sulfate solution and the chlorpyrifos is partitioned into hexane. The hexane layer is percolated through a column of anhydrous sodium sulfate and then concentrated by evaporation with a jet of dry air. The analysis is completed as in ACR 72.1.

ACR 72.15-Beans, including forage, sugar beet
Samples are blended with acetone and an aliquot of the extract is evaporated to near dryness under a Snyder column. After the addition of sodium sulfate, the residue is dissolved in hexane and cleaned up on a Florisil column before analysis.

ACR 72.15.S1-Kidney and field beans, peas and vines
After extraction, evaporation and sodium sulfate addition as in ACR 72.15, the residue is dissolved in hexane, which is flashed through a Florisil column carrying the chlorpyrifos with it. The hexane is evaporated and the residue rechromatographed on a second Florisil column in benzene. The benzene is evaporated and the sample taken up in acetone for analysis

ACR 73.5-Sugar beet, sorghum and maize
The sample is blended with acetone. The extract is filtered, the acetone evaporated, and the residue dissolved in hexane. Hexane-acetonitrile partition and clean-up on a 10% silica gel column follow. The eluting solvent is evaporated to dryness. The residues are redissolved in acetone and an aliquot is analysed.

ACR 73.5.S1-Cotton seed, wheat and processed fractions, maize, sunflower and processed fractions, apples and processed fractions, citrus and processed fractions
The sample is homogenized on a “Polytron” with acetone. The procedure is then the same as ACR 73.5 except that a 20% silica gel column is used for clean-up.

ACR 73.5.S2-Juices, especially citrus
The method is then the same as ACR 73.5 except that methanol is used for extraction.

ACR 73.6-Sugar beet juices, citrus molasses
The sample is blended with methanol and the extract filtered, and an aliquot of the filtrate is mixed with a 5% aqueous sodium sulfate solution and partitioned with hexane. The hexane layer is dried by percolating through a column of anhydrous sodium sulfate and then concentrated by evaporation. After a silica gel column clean-up the hexane eluate is evaporated to dryness. The residue is redissolved in acetone and an aliquot analysed.

ACR 74.4-Cotton and processed fractions
The sample is blended with acetone. An aliquot of the extract is evaporated to near dryness under a Snyder column, then azeotroped with hexane to a small volume. The residue is redissolved in methanol and chromatographed on an acidic alumina column. The eluate is mixed with water and partitioned with hexane. The hexane layer is further cleaned up by partitioning with acetonitrile and chromatographing on Florisil before analysis.

ACR 75.1-Oils
The sample is shaken with methanol to extract the chlorpyrifos, then filtered and diluted to a known volume. An aliquot is partitioned with hexane in the presence of water. The hexane phase is dried, evaporated to a small volume and added to a Florisil column from which the compound is eluted with benzene. The benzene is evaporated and the residue dissolved in acetone for analysis.

ACR 75.4-Citrus oil
The sample is blended with acetone, the extract filtered, and the acetone evaporated. The residue is taken up in hexane and the hexane is partitioned with acetonitrile. The acetonitrile is evaporated and the residue redissolved in hexane. The sample is chromatographed on a Florisil column and the eluate is evaporated to dryness. The residue is redissolved in acetone for analysis.

ACR 76.3-Sorghum silage, grain and processed fractions, citrus pulp
The sample is blended with acetone. The extract is filtered and the filtrate is concentrated on a hot plate under a Snyder evaporation column. The concentrate is added to a 5% aqueous sodium sulfate solution and the chlorpyrifos extracted with hexane. The hexane layer is percolated through a column of anhydrous sodium sulfate, concentrated, partitioned with acetonitrile and cleaned up on a silica gel column. The eluate is concentrated on a hot plate under a Snyder column, then taken to dryness with a jet of dry air. The residue is redissolved in acetone for analysis.

ACR 76.3.S1-Peanuts and processed fractions, sorghum and processed fractions, tomatoes and processed fractions, sunflower and processed fractions, coffee beans, apples, cotton seed
The sample is blended with acetone on a “Polytron”. The extract is filtered and an aliquot is concentrated on a hot plate under a Snyder column. Sodium sulfate is added to absorb the residual water and the chlorpyrifos is extracted with hexane. A hexane-acetonitrile partition is used for clean up. The acetonitrile is concentrated and taken to dryness as before and the residue redissolved in acetone for analysis.

ACR 76.9-Tomatoes
Samples are blended with acetone/hexane to extract the chlorpyrifos and evaporated to near dryness under a Snyder column. The remaining acetone/hexane is removed by evaporating to near dryness with benzene. The residue is dissolved in acetone/benzene and cleaned up by gel permeation chromatography. The benzene eluate is reduced to a known volume for analysis.

ACR 78.10-Alfalfa forage and hay
The sample is blended with acetone. The extract is centrifuged and a portion diluted as before and analysed directly.

ACR 79.8-Citrus fractions
Chlorpyrifos is extracted with hexane. The hexane extract is evaporated to a small volume and analysed directly.

ACR 84.4-Head and leaf lettuce, citrus, peppers, tomatoes, pears, cereal grains and forage, berries, onions, strawberries, kernels and hulls, alfalfa seed, sweet corn, sorghum forage, fodder and grain, peanuts
ACR 84.4.S1-Alfalfa forage and hay
An acetone extract is evaporated and the residue taken up in water for clean-up on a C-18 Sep-Pak eluted with methanol. The eluate is diluted with water and extracted with a known volume of hexane for analysis.

ACR 84.4.S3-Alfalfa forage and hay, field corn and processed fractions, sorghum and processed fractions, sugar beet, sunflower, apples, grapes
This method is the same as ACR 84.4 except that a capillary column is used.

ACR 90.2-Sunflower seeds and hulls, corn oil
The sample is homogenized with acetone, centrifuged, and an aliquot evaporated. The chlorpyrifos is partitioned from the co-extracted water into hexane, which is partitioned with acetonitrile. The acetonitrile is evaporated to dryness and the residue taken up in water and passed through a C-18

"Sep-Pak" which is eluted with methanol. The eluate is taken up in phosphoric acid, which is partitioned into a known volume of hexane for analysis.

ACR 90.5-Apples, pears
A methanol extract is centrifuged and an aliquot taken up in dilute phosphoric acid. This is partitioned with hexane for analysis.

ERC 87.05-Sugar beet
An acetone macerate is diluted with water and partitioned with hexane. An acetonitrile partition and Florisil mini-column clean-up are followed by gas chromatography with electron capture detection.

ERC 90.13-Bananas, sugar beet, soil
ERC 92.26 –Peaches, cauliflower
ERC 92.28- Onions
ERC 92.30- Peas
ERC 92.31- Beans
ERC 92.38-Citrus
ERC 93.13-Cabbage, Chinese cabbage
ERC 94.1-Grapes
ERC 96.04-Carrots
Chlorpyrifos is extracted by macerating with acetone/water, additional water is added and chlorpyrifos partitioned into hexane. The hexane solution is analysed on a capillary column with flame photometric detection.

BRC 93.1-Cotton seed
Ground samples are extracted with acetone and an aliquot evaporated. A 5% aqueous sodium chloride solution is added and the mixture partitioned with hexane, then the hexane with acetonitrile. Water is added and the solution partitioned with hexane which is evaporated to dryness. The residue is reconstituted in hexane and passed through a "Bond-Elut" silica column, the column washed with hexane and the chlorpyrifos eluted with an excess of hexane, and the eluate evaporated to dryness. The residue is taken up in hexane and the chlorpyrifos determined by gas chromatography with an electron capture detector.

RAM 1312-Almond kernels and hulls
Residues are extracted by blending with hexane, which is filtered. An aliquot is partitioned with acetonitrile and the acetonitrile evaporated to dryness. The sample is cleaned up on a deactivated silica gel column. The eluate is evaporated to dryness and the residue taken up in hexane for analysis.

**Enforcement and multiresidue methods**

The Government of The Netherlands submitted a multiresidue methods for the determination of chlorpyrifos in a variety of crops and products (Netherlands, 1996). The extraction methods for non-fatty samples included ethyl acetate for fruits and vegetables, and acetone followed by partition for fruits, vegetables and potatoes, nuts, cereals, pulses, oil seeds, tropical seeds and dried fruits, garlic, herbs and spices. Non-fatty ($\leq$5%) animal products are minced, mixed with sodium sulfate, and extracted with acetone/acetonitrile (1:10). Fatty animal meat is rendered at 65$^{o}$C for 8 h and the fat dissolved in light petroleum. Cheese is extracted with light petroleum. Butter is mixed with sodium sulfate and shaken with n-pentane. Avocado is extracted overnight with acetone:methylene chloride (50/50). Oils seeds are ground and extracted in a Soxhlet with light petroleum ether for 8 h. Milk is extracted either by light petroleum/acetone (50:50) or by a modified AOAC procedure.

Extracts are cleaned up by gel permeation chromatography. The eluent is acetone/cyclohexane (2:1), and the GPC column (450 mm x 10 mm) is packed with Bio-Beads SX-3.

Analyses are by GLC on a capillary column with an FPD or NPD, with confirmation with an ion-trap detector (ITD) or by GC-MS. The relative retention times on various columns are DB-1 1.00, DB-5 0.98, DB-1701 0.91, and DB-wax 0.72. The m/z fragments are 197-201, 258-262 and 314-318. With an ITD, the recoveries of chlorpyrifos from lettuce fortified at 0.03 mg/kg were 103 ± 8.5%, n = 10, and at 0.17 mg/kg 111 ± 3.5%, n = 10.

Adequate methods for the enforcement of tolerances in plant and animal commodities are described in United States Food and Drug Administration Pesticide Analytical Manual (PAM), Volume 2. For plant commodities, there are three GLC methods (Methods I, II and VI) with flame photometric detection (FPD) and a stated limit of detection of chlorpyrifos of 0.01 mg/kg. The recoveries of chlorpyrifos from fortified bananas (0.01-5.0 mg/kg), peaches (0.01-1.0 mg/kg) and cotton seed (0.01-0.1 mg/kg) were 91 ± 4%, 80 ± 2% and 94 ± 10% respectively. In a confirmatory procedure residues are hydrolysed to TCP, which is determined by GLC or GC-MS. For animal commodities, PAM Vol. 2 describes Method IV, with determination by GLC with electron capture detection. The stated limit of detection of free TCP in meat is 0.01 mg/kg. The recovery of chlorpyrifos from beef fat fortified at 0.1 mg/kg was 84%.

The US FDA PESTDATA database (PAM Vol. 1, Appendix II, 1993) indicates that chlorpyrifos is completely recovered (>80%) by FDA multiresidue method protocols D (Section 232.4) and E (Section 212.1/232.1, non-fatty matrices) and partially recovered (50-80%) by multiresidue method protocol E (Section 211.1/232.1, fatty matrices).

Table 21. Summary of methods for determination of residues of chlorpyrifos. All determinations are by GLC.

| Method no. | Date | Sample | Detector | Fortification, mg/kg | Mean recovery | LOQ, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| **Fruit samples** | | | | | | | |
| Pome fruits | | | | | | | |
| ACR 76.3.S1 | 1976 | Apples | FPD | 0.05-2.0 | 92 ± 6 | 0.05 | GH-C 1485 |
| ACR 73.5.S1 | 1973 | Whole apples | FPD | 0.01-10.0 | 96 ± 3 | 0.01 | GH-C 1107 |
| ACR 90.5 | 1990 | Whole apples | FPD | 0.01-0.50 | 96 ± 1 | 0.01 | GH-C 2397 |
| ACR 90.5 | 1990 | Whole apples | FPD | 0.01-1.0 | 98 ± 12 | 0.01 | GH-C 2449 |
| ACR 84.4 | 1984 | Whole pears | FPD | 0.01-2.0 | 90 ± 3 | 0.01 | GH-C 1789 |
| ACR 90.5 | 1990 | Whole pears | FPD | 0.01-0.30 | 98 ± 11 | 0.01 | GH-C 2449 |
| Citrus fruits | | | | | | | |
| ACR 73.5.S1 | 1973 | Grapefruit, whole | FPD | 0.01-1.0 | 86 ± 6 | 0.01 | GH-C 1441 |
| | | Peel frits | FPD | 1.0 | 71 | 1.0 | GH-C 1441 |
| | | Finisher pulp | FPD | 1.0 | 75 | 1.0 | GH-C 1441 |
| | | Chopped peel | FPD | 1.0 | 73 | 1.0 | GH-C 1441 |
| ACR 73.5.S1 | 1973 | Whole lemons | FPD | 0.01-1.0 | 89 ± 6 | 0.01 | GH-C 1441 |
| | | Peel frits | FPD | 0.01-0.10 | 74 +/-6 | 0.01 | GH-C 1441 |
| | | Finisher pulp | FPD | 0.01-0.10 | 74 ± 2 | 0.01 | GH-C 1441 |
| | | Chopped peel | FPD | 0.01-0.10 | 71 ± 0 | 0.01 | GH-C 1441 |
| ACR 73.5.S1 | 1973 | Whole oranges | FPD | 0.01-1.0 | 88 ± 4 | 0.01 | GH-C 1441 |
| | | Peel frits | FPD | 1.0 | 75 | 1.0 | GH-C 1441 |
| | | Finisher pulp | FPD | 1.0 | 74 | 1.0 | GH-C 1441 |
| | | Chopped peel | FPD | 1.0 | 74 | 1.0 | GH-C 1441 |
| ACR 73.5.S1 | 1973 | Whole tangelos | FPD | 0.01-1.0 | 86 ± 5 | 0.01 | GH-C 1441 |
| | | Peel frits | FPD | 0.02-0.10 | 76 ± 9 | 0.02 | GH-C 1441 |
| | | Finisher pulp | FPD | 0.01-0.10 | 77 ± 0 | 0.01 | GH-C 1441 |
| | | Chopped peel | FPD | 0.01-0.10 | 74 ± 5 | 0.01 | GH-C 1441 |
| ACR 73.5.S2 | 1973 | Grapefruit juice | FPD | 0.10 | 75 | 0.10 | GH-C 1441 |

| Method no. | Date | Sample | Detector | Fortification, mg/kg | Mean recovery | LOQ, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| | | Press liquor | FPD | 0.10 | 78 | 0.10 | GH-C 1441 |
| | | Emulsion water | FPD | 0.10 | 63 | 0.10 | GH-C 1441 |
| ACR 73.5.S2 | 1973 | Lemon juice | FPD | 0.01-0.10 | 72 ± 4 | 0.01 | GH-C 1441 |
| ACR 73.5.S2 | 1973 | Press liquor | FPD | 0.01-0.10 | 76 ± 2 | 0.01 | GH-C 1441 |
| | | Emulsion water | FPD | 0.01-0.10 | 77 ± 1 | 0.01 | GH-C 1441 |
| | | Orange juice | FPD | 0.10 | 78 | 0.10 | GH-C 1441 |
| ACR 73.5.S2 | 1973 | Press liquor | FPD | 0.10 | 74 | 0.10 | GH-C 1441 |
| ACR 73.5.S2 | 1973 | Emulsion water | FPD | 0.10 | 70 | 0.10 | GH-C 1441 |
| | | Tangelo juice | FPD | 0.01-0.10 | 81 ± 1 | 0.01 | GH-C 1441 |
| | | Press liquor | FPD | 0.01-0.10 | 77 ± 1 | 0.01 | GH-C 1441 |
| ACR 73.5.S2 | 1973 | Emulsion water | FPD | 0.01-0.10 | 72 ± 2 | 0.01 | GH-C 1441 |
| ACR 73.6 | 1973 | G. fruit molasses | FPD | 0.01-0.10 | 93 ± 15 | 0.01 | GH-C 1441 |
| | | Lemon molasses | FPD | 0.01-0.10 | 82 ± 14 | 0.01 | GH-C 1441 |
| | | Orange molasses | FPD | 0.01-0.10 | 84 ± 16 | 0.01 | GH-C 1441 |
| | | Tangelo molasses | FPD | 0.01-0.10 | 84 ± 11 | 0.01 | GH-C 1441 |
| ACR 75.4 | 1975 | Grapefruit oil | FPD | 0.10-5.0 | 60 ± 6 | 0.10 | GH-C 1441 |
| | | Lemon oil | FPD | 0.10-1.0 | 71 ± 7 | 0.10 | GH-C 1441 |
| | | Orange oil | FPD | 0.10-3.0 | 78 ± 1 | 0.10 | GH-C 1441 |
| | | Tangelo oil | FPD | 0.10-5.0 | 86 ± 23 | 0.10 | GH-C 1441 |
| ACR 76.3 | 1976 | Grapefruit pulp | FPD | 1.0 | 68 | 1.0 | GH-C 1441 |
| | | Lemon pulp | FPD | 1.0 | 75 | 1.0 | GH-C 1441 |
| | | Orange pulp | FPD | 1.0 | 69 | 1.0 | GH-C 1441 |
| | | Tangelo pulp | FPD | 0.05-0.10 | 71 ± 1 | 0.05 | GH-C 1441 |
| ACR 79.8 | 1979 | G.fruit rinse water | FPD | 0.01 | 113 | 0.01 | GH-C 1441 |
| | | Lemon rinse water | FPD | 0.01 | 116 | 0.01 | GH-C 1441 |
| | | Orange rinse water | FPD | 0.01 | 111 | 0.01 | GH-C 1441 |
| ACR 84.4 | 1984 | Whole oranges | FPD | 0.01-2.0 | 88 ± 5 | 0.02 | GH-C 1724 |
| ACR 84.4 | 1984 | Oranges | FPD | 0.01-1.0 | 89 ± 4 | 0.01 | GH-C 2554 |
| ERC 92.38 | 1992 | Lemon peel | FPD | 0.01-5.0 | 82 ± 4 | 0.01 | GHE-P 3228 |
| | 1992 | Lemon pulp | FPD | 0.01-5.0 | 90 ± 4 | 0.01 | GHE-P 3228 |
| | 1992 | Mandarin peel | FPD | 0.01-5.0 | 85 ± 8 | 0.01 | GHE-P 3213 |
| | 1992 | Mandarin pulp | FPD | 0.01-5.0 | 97 ± 6 | 0.01 | GHE-P 3213 |
| Stone fruits | | | | | | | |
| ACR 71.14 | 1971 | Peaches | FPD | 0.01-1.0 | 80 ± 5 | 0.01 | GH-C 479 |
| ERC 92.26 | 1992 | Peaches | FPD | 0.01-1.0 | 100 +/ 4 | 0.01 | GHE-P 3139 |
| Small fruits and berries | | | | | | | |
| ACR 84.4.S3 | 1984 | Grapes | FPD | 0.01-1.0 | 88 ± 9 | 0.01 | GH-C 3272 |
| ERC 94.1 | 1994 | Grapes | FPD | 0.01 -1.0 | 94 +/-7 | 0.01 | GHE-P 4968 |
| ACR 84.4 | 1984 | Blueberries | FPD | 0.01-2.0 | 90 ± 8 | 0.01 | GH-C 1832 |
| ACR 84.4 | 1984 | Strawberries | FPD | 0.01-10.0 | 86 ± 4 | 0.01 | GH-C 1871 |
| ACR 84.4 | 1984 | Black raspberries | FPD | 0.01-2.0 | 85 ± 7 | 0.01 | GH-C 1903 |
| | | Blackberries | FPD | 0.01-2.0 | 89 ± 7 | 0.01 | GH-C 1903 |
| | | Red Raspberries | FPD | 0.01-2.0 | 89 ± 4 | 0.01 | GH-C 1903 |
| | | Boysenberries | FPD | 0.01-2.0 | 97 ± 6 | 0.01 | GH-C 1903 |
| Tropical fruits | | | | | | | |
| ERC 90.13 | 1990 | Bananas, whole | FPD | 0.01-2.0 | 90 ± 14 | 0.01 | GHE-P 3349 |
| | | Bananas, pulp | FPD | 0.01-2.0 | 92 ± 8 | 0.01 | |
| ERC 90.13 | 1990 | Bananas, peel | FPD | 0.01-2.0 | 90 ± 17 | 0.01 | |
| **Vegetable samples** | | | | | | | |
| Brassica vegetables | | | | | | | |

| Method no. | Date | Sample | Detector | Fortification, mg/kg | Mean recovery | LOQ, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| ERC 93.13 | 1993 | Cabbage | FPD | 0.01-1.0 | 103+/-6 | 0.01 | GHE-P 3634 |
| ERC 92.26 | 1992 | Cauliflower -plant | FPD | 0.01-1.0 | 97 +/-3 | 0.01 | GHE-P 3212 |
| | | Rest of plant | FPD | 0.01-1.0 | 85 +/-7 | 0.01 | GHE-P 3212 |
| | | Curd | FPD | 0.01-1.0 | 97 +/-8 | 0.01 | GHE-P 3633 |
| Legume vegetables | | | | | | | |
| ACR 72.15.S1 | 1972 | Kidney beans | FPD | 0.05-1.0 | 98 ± 2 | 0.01 | GH-C 1157 |
| | | Kidney pods | FPD | 0.05-1.0 | 98 ± 2 | 0.01 | GH-C 1157 |
| | | Kidney whole plant | FPD | 0.05-1.0 | 91 ± 3 | 0.05 | GH-C 1157 |
| | | Kidney vines | FPD | 0.05-1.0 | 91 ± 3 | 0.05 | GH-C 1157 |
| | | Field bean vines | FPD | 0.05-1.0 | 88 ± 6 | 0.05 | GH-C 1157 |
| | | Field beans | FPD | 0.05-1.0 | 89 ± 4 | 0.05 | GH-C 1157 |
| ACR 72.15.S1 | 1972 | Peas plus pod | FPD | 0.05-1.0 | 81 +/-6 | 0.05 | GH-C 1158 |
| | | Pea vines | FPD | 0.05-1.0 | 82 ± 5 | 0.05 | GH-C 1158 |
| | | Pea whole plant | FPD | 0.05-1.0 | 82 ± 5 | 0.05 | GH-C 1158 |
| ACR 72.15.S1 | 1972 | Snapbean forage | FPD | 0.01-2.0 | 92 ± 2 | 0.01 | GH-C 660 |
| | | Snapbeans | FPD | 0.01-0.50 | 87 ± 4 | 0.01 | GH-C 660 |
| ERC 92.31 | 1992 | Beans-whole plant | FPD | 0.01-1.0 | 94 ± 6 | 0.01 | GHE-P 3231 |
| | | Rest of plant | FPD | 0.01-1.0 | 94 ± 8 | 0.01 | GHE-P 3231 |
| | | Beans | FPD | 0.01-1.0 | 94 ± 5 | 0.01 | GHE-P 3231 |
| ERC 92.30 | 1992 | Peas-whole plant | FPD | 0.01-1.0 | 95 ± 7 | 0.01 | GHE-P 3232 |
| | | Peas-rest of plant | FPD | 0.01-1.0 | 96 ± 6 | 0.01 | GHE-P 3232 |
| | | Peas | FPD | 0.01-1.0 | 98 ± 7 | 0.01 | GHE-P 3232 |
| Leafy vegetables | | | | | | | |
| ACR 84.4 | 1984 | Leaf lettuce | FPD | 0.01-5.0 | 88 ± 12 | 0.02 | GH-C 1696 |
| | | Head lettuce | FPD | 0.01-10.0 | 94 ± 10 | 0.01 | GH-C 1696 |
| Fruiting vegetables | | | | | | | |
| ACR 76.3.S1 | 1976 | Whole tomatoes | FPD | 0.01-10.0 | 87 ± 4 | 0.02 | GH-C 1282 |
| | | Juice | FPD | 0.01-0.10 | 100 ± 18 | 0.01 | GH-C 1282 |
| | | Tomato less peel | FPD | 0.01-0.50 | 94 ± 8 | 0.01 | GH-C 1282 |
| | | Purée | FPD | 0.01-0.10 | 92 ± 15 | 0.01 | GH-C 1282 |
| | | Seeds/peelings | FPD | 0.01-1.5 | 90 ± 9 | 0.01 | GH-C 1282 |
| ACR 76.3.S1 | 1976 | Tomatoes | FPD | 0.01-0.50 | 88 ± 6 | 0.01 | GH-C 1372 |
| ACR 76.3.S1 | 1976 | Tomatoes | FPD | 0.01-5.0 | 89 ± 5 | 0.02 | GH-C 1641 |
| ACR 76.9 | 1976 | Tomatoes | FPD | 0.01-0.50 | 94 ± 4 | 0.01 | GH-C 952 |
| ACR 84.4 | 1984 | Peppers | FPD | 0.01-2.0 | 86 ± 9 | 0.01 | GH-C 1757 |
| | | Tomatoes | FPD | 0.01-0.50 | 94 ± 12 | 0.01 | GH-C 1757 |
| Bulb vegetables | | | | | | | |
| ACR 84.4 | 1984 | Onions | FPD | 0.01-0.04 | 87 ± 5 | 0.01 | GH-C 1848 |
| ERC 92.28 | 1992 | Onions | FPD | 0.01-1.0 | 100 +/-6 | 0.01 | GHE-P 3466 |
| Root crops | | | | | | | |
| ACR 72.15 | 1972 | Sugar beet leaves | FPD | 0.01-1.0 | 89 ± 6 | 0.01 | GH-C 729 |
| ACR 73.5 | 1973 | Sugar beet roots | FPD | 0.01-1.0 | 87 ± 4 | 0.01 | GH-C 729 |
| | | Wet pulp | FPD | 0.01-1.0 | 90 ± 6 | 0.01 | GH-C 729 |
| | | Dry pulp | FPD | 0.01-1.0 | 100 ± 4 | 0.01 | GH-C 729 |
| | | Lime cake | FPD | 0.01-1.0 | 99 ± 4 | 0.01 | GH-C 729 |
| ACR 73.6 | 1973 | Diffusion juice | FPD | 0.01-1.0 | 103 ± 5 | 0.01 | GH-C 729 |
| | | Thin juice | FPD | 0.01-1.0 | 100 ± 2 | 0.01 | GH-C 729 |
| | | Thick juice | FPD | 0.01-1.0 | 106 ± 6 | 0.01 | GH-C 729 |
| ACR 84.4.S3 | 1984 | Sugar beet roots | FPD | 0.01-1.0 | 71 ± 4 | 0.01 | GH-C 4223 |
| | | Sugar beet tops | FPD | 0.01-10.0 | 80 ± 8 | 0.01 | GH-C 4223 |
| ERC 90.13 | 1990 | Sugar beet leaves | FPD | 0.01-10.0 | 92 +/-7 | 0.01 | GHE-P 2467 |

| Method no. | Date | Sample | Detector | Fortification, mg/kg | Mean recovery | LOQ, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| | | Sugar beet roots | FPD | 0.01-10.0 | 100 +/-7 | 0.01 | GHE-P 2467 |
| ERC 87.05 | 1987 | Sugar beet leaves | ECD | 0.01-0.5 | 70 ± 4 | 0.01 | GHE-P 1709 |
| | | Sugar beet roots | ECD | 0.01-0.5 | 73 +/-8 | 0.01 | GHE-P 1709 |
| ERC 96.04 | 1996 | Carrots | FPD | 0.01-10.0 | 88 ± 6 | 0.01 | GHE-P 5473 |
| **Cereal grains and animal feeds** | | | | | | | |
| Alfalfa | | | | | | | |
| ACR 78.10 | 1978 | Alfalfa forage | FPD | 0.50-2.0 | 95 ± 4 | 0.50 | GH-C 1610 |
| | | Alfalfa hay | FPD | 0.50-2.0 | 94 ± 4 | 0.50 | GH-C 1610 |
| ACR 78.10 | 1978 | Alfalfa forage | FPD | 0.50-5.0 | 96 ± 4 | 0.50 | GH-C 2288 |
| ACR 78.10 | 1978 | Alfalfa forage | FPD | 0.50-5.0 | 94 ± 6 | 1.0 | GH-C 2294 |
| ACR 78.10 | 1978 | Alfalfa forage | FPD | 0.5-100.0 | 97 ± 6 | 1.0 | GH-C 2334 |
| | | Alfalfa hay | FPD | 0.5-100.0 | 107 ± 7 | 1.0 | GH-C 2334 |
| ACR 84.4 | 1984 | Alfalfa seed | FPD | 0.10-0.50 | 96 ± 7 | 0.02 | GH-C 1803 |
| ACR 84.4S1 | 1984 | Alfalfa forage | FPD | 0.01-10.0 | 84 ± 10 | 0.01 | GH-C 4198 |
| | | Alfalfa hay | FPD | 0.01-20.0 | 84 ± 10 | 0.01 | GH-C 4198 |
| ACR 84.4.S3 | 1984 | Alfalfa forage | FPD | 0.01-20.0 | 84 ± 7 | 0.01 | GH-C 2752 |
| | | Alfalfa hay | FPD | 0.01-50.0 | 89 ± 11 | 0.01 | GH-C 2752 |
| Sweet corn and maize (field corn) | | | | | | | |
| ACR 71.18 | 1971 | Field corn forage | FPD | 0.01-1.0 | 92 ± 5 | 0.01 | GH-C 530 |
| | | Field corn grain | FPD | 0.01-1.0 | 83 ± 6 | 0.01 | GH-C 530 |
| | | Field corn stover | FPD | 0.01-1.0 | 93 ± 5 | 0.01 | GH-C 530 |
| ACR 72.9 | 1972 | Sweet corn forage | FPD | 0.01-1.0 | 97 ± 6 | 0.01 | GH-C 664 |
| | | Kernels | FPD | 0.01-1.0 | 94 ± 5 | 0.01 | GH-C 664 |
| | | Kernels plus cob | FPD | 0.01-1.0 | 95 ± 5 | 0.01 | GH-C 664 |
| | | Cobs plus husks | FPD | 0.01-1.0 | 101 ± 4 | 0.01 | GH-C 664 |
| | | Husks | FPD | 0.01-1.0 | 82 ± 7 | 0.01 | GH-C 664 |
| ACR 73.5.S1 | 1973 | Field corn forage | FPD | 0.05-1.0 | 81 ± 3 | 0.05 | GH-C 1284 |
| | | Fodder | FPD | 0.05-1.0 | 84 ± 2 | 0.01 | GH-C 1284 |
| | | Grain | FPD | 0.01-1.0 | 93 ± 5 | 0.01 | GH-C 1284 |
| ACR 73.5.S1 | 1973 | Field corn forage | FPD | 0.01-.10 | 84 ± 5 | 0.01 | GH-C 1068 |
| | | Field corn fodder | FPD | 0.01-0.10 | 83 ± 6 | 0.01 | GH-C 1068 |
| | | Field corn grain | FPD | 0.01-0.10 | 85 ± 4 | 0.01 | GH-C 1068 |
| ACR 73.5.S1 | 1973 | Field corn forage | FPD | 0.50-50.0 | 89 ± 8 | 1.0 | GH-C 1440 |
| | | Fodder | FPD | 0.50-10.0 | 91 ± 5 | 1.0 | GH-C 1440 |
| | | Grain | FPD | 0.01-1.0 | 82 ± 6 | 0.01 | GH-C 1440 |
| ACR 84.4 | 1984 | Sweet corn forage | FPD | 0.01-2.0 | 91 ± 9 | 0.01 | GH-C 2569 |
| | | Sweet corn ears | FPD | 0.01-0.10 | 92 ± 11 | 0.01 | GH-C 2569 |
| ACR 84.4 | 1984 | Sweet corn forage | FPD | 0.01-10.0 | 91 ± 4 | 0.01 | GH-C 1797 |
| ACR 84.4.S3 | 1984 | Field corn grain | FPD | 0.01 | 88 ± 20 | 0.01 | GH-C 2878 |
| | | Grits | FPD | 0.01-0.10 | 77 ± 14 | 0.01 | GH-C 2878 |
| | | Meal | FPD | 0.01-1.0 | 83 ± 8 | 0.01 | GH-C 2878 |
| | | Flour | FPD | 0.01-0.10 | 84 ± 9 | 0.01 | GH-C 2878 |
| | | Gluten | FPD | 0.01-0.50 | 97 ± 6 | 0.01 | GH-C 2878 |
| | | Starch | FPD | 0.01-0.05 | 84 ± 10 | 0.01 | GH-C 2878 |
| | | Screenings | FPD | 0.10-2.0 | 107 ± 12 | 0.10 | GH-C 2878 |
| ACR 90.2 | 1990 | Field corn oil | FPD | 0.01-0.10 | 71 ± 13 | 0.02 | GH-C 2878 |
| Sorghum | | | | | | | |
| ACR 73.5 | 1973 | Sorghum forage | FPD | 0.01-5.0 | 93 ± 4 | 0.01 | GH-C 900 |
| | | Sorghum silage | FPD | 0.01-1.0 | 94 ± 8 | 0.01 | GH-C 900 |
| ACR 76.3 | 1976 | Sorghum silage | FPD | 0.01-1.0 | 78 ± 6 | 0.01 | GH-C 900 |
| | | Sorghum grain | FPD | 0.01-1.0 | 84 ± 3 | 0.01 | GH-C 900 |

| Method no. | Date | Sample | Detector | Fortification, mg/kg | Mean recovery | LOQ, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| ACR 76.3 | 1976 | Sorghum grain | FPD | 0.01-0.50 | 84 +/-3 | 0.01 | GH-C 998 |
| | | Sorghum stover | FPD | 0.01-3.0 | 78 ± 4 | 0.01 | GH-C 998 |
| ACR 76.3 | 1976 | Sorghum grain | FPD | 0.02-0.50 | 85 ± 6 | 0.02 | GH-C 1109 |
| | | Flour | FPD | 0.02-0.10 | 79 ± 13 | 0.02 | GH-C 1109 |
| | | Shorts | FPD | 0.02-0.10 | 92 ± 4 | 0.02 | GH-C 1109 |
| | | Middlings | FPD | 0.02-0.10 | 88 ± 16 | 0.02 | GH-C 1109 |
| | | Screenings | FPD | 0.02-0.20 | 82 ± 10 | 0.02 | GH-C 1109 |
| ACR 76.3.S1 | 1976 | Sorghum bran | FPD | 0.02-0.50 | 90 ± 14 | 0.02 | GH-C 1109 |
| | | Sorghum germ | FPD | 0.05-0.40 | 77 ± 1 | 0.05 | GH-C 1109 |
| ACR 84.4 | 1984 | Sorghum forage | FPD | 0.05-5.0 | 85 ± 11 | 0.05 | GH-C 1813 |
| | | Sorghum fodder | FPD | 0.05-1.0 | 87 ± 10 | 0.05 | GH-C 1813 |
| | | Sorghum grain | FPD | 0.01-0.05 | 89 ± 13 | 0.01 | GH-C 1813 |
| ACR 84.4 | 1984 | Sorghum grain | FPD | 0.01-0.50 | 92 ± 17 | 0.02 | GH-C 2555 |
| | | Sorghum fodder | FPD | 0.01-1.0 | 100 ± 11 | 0.02 | GH-C 2555 |
| ACR 84.4.S3 | 1984 | Sorghum grain | FPD | 0.01-0.50 | 83 ± 7 | 0.01 | GH-C 3226 |
| | | Sorghum forage | FPD | 0.01-0.50 | 82 ± 4 | 0.01 | GH-C 3226 |
| Wheat | | | | | | | |
| ACR 73.5.S1 | 1973 | Wheat grain | FPD | 0.01-1.0 | 92 ± 8 | 0.02 | GH-C 1346 |
| | | Wheat straw | FPD | 0.01-2.0 | 90 ± 4 | 0.01 | GH-C 1346 |
| | | Wheat bran | FPD | 0.05-2.0 | 86 ± 1 | 0.05 | GH-C 1346 |
| | | Wheat flour | FPD | 0.05-2.0 | 76 ± 13 | 0.05 | GH-C 1346 |
| | | Break shorts | FPD | 0.05-2.0 | 86 ± 1 | 0.05 | GH-C 1346 |
| ACR 73.5.S1 | 1973 | Reduction shorts | FPD | 0.05-2.0 | 84 ± 6 | 0.05 | GH-C 1346 |
| | | Red dog | FPD | 0.05-2.0 | 86 ± 2 | 0.05 | GH-C 1346 |
| | | Bread | FPD | 0.05-2.0 | 84 ± 1 | 0.05 | GH-C 1346 |
| ACR 73.5.S1 | 1973 | Wheat grain | FPD | 0.01-1.0 | 98 ± 9 | 0.02 | GH-C 1639 |
| | | Wheat straw | FPD | 0.05-5.0 | 97 ± 6 | 0.02 | GH-C 1639 |
| | | Wheat forage | FPD | 0.05-20.0 | 93 ± 9 | 0.04 | GH-C 1639 |
| ACR 84.4 | 1984 | Wheat grain | FPD | 0.01-0.10 | 90 ± 0 | 0.01 | GH-C 1790 |
| | | Wheat straw | FPD | 0.01-5.0 | 88 ± 7 | 0.01 | GH-C 1790 |
| | | Wheat forage | FPD | 0.01-2.0 | 91 +/-5 | 0.01 | GH-C 1790 |
| ACR 84.4 | 1984 | Wheat grain | FPD | 0.01-1.0 | 95 ± 12 | 0.01 | GH-C 1804 |
| ERC 90.13 | 1990 | Wheat grain | FPD | 0.01-1.0 | 92 +/-9 | 0.01 | GHE-P 3720 |
| | | Wheat straw | FPD | 0.01-2.0 | 92 +/-6 | 0.01 | GHE-P 3720 |
| **Nuts and seeds** | | | | | | | |
| Almonds | | | | | | | |
| RAM 1312 | 1987 | Kernels | FPD | 0.01-8.0 | 86 ± 4 | 0.01 | GH-C 2180 |
| | | Hulls | FPD | 0.01-4.0 | 84 ± 6 | 0.01 | GH-C 2180 |
| Cotton seed | | | | | | | |
| ACR 73.5.S1 | 1973 | Cotton seed | FPD | 0.01-1.0 | 86 ± 6 | 0.01 | GH-C 1893 |
| ACR 74.4 | 1974 | Cotton seed | FPD | 0.01-0.10 | 93 ± 10 | 0.01 | GH-C 840 |
| | | Gin trash | FPD | 0.01-1.0 | 88 ± 18 | 0.03 | GH-C 840 |
| | | Hulls | FPD | 0.10 | 66 ± 9 | 0.10 | GH-C 840 |
| | | Linters | FPD | 0.01 | 85 ± 15 | 0.02 | GH-C 840 |
| | | Meal | FPD | 0.01-0.10 | 97 ± 7 | 0.01 | GH-C 840 |
| ACR 75.1 | 1975 | Oil | FPD | 0.01-0.20 | 78+/- 8 | 0.01 | GH-C 840 |
| ACR 76.3.S1 | 1976 | Cotton seed | FPD | 0.01-0.50 | 87 ± 8 | 0.01 | GH-C 1658 |
| ACR 76.3.S1 | 1976 | Cotton seed | FPD | 0.01-0.10 | 87 ± 5 | 0.01 | GH-C 1879 |
| BRC 93.1 | 1993 | Cotton seed | ECD | 0.01-1.0 | 81 ± 3 | 0.01 | GHB-P 195 |
| Coffee | | | | | | | |
| ACR 76.3.S1 | 1976 | Coffee beans | FPD | 0.01-0.10 | 88 ± 5 | 0.01 | GH-C 1462 |

| Method no. | Date | Sample | Detector | Fortification, mg/kg | Mean recovery | LOQ, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| Peanuts | | | | | | | |
| ACR 76.3.S1 | 1976 | Peanut forage | FPD | 0.05-100.0 | 79 ± 3 | 0.05 | GH-C 1071 |
| | | Kernels | FPD | 0.01-0.50 | 84 ± 3 | 0.01 | GH-C 1071 |
| | | Hulls | FPD | 0.01-0.10 | 87 ± 6 | 0.01 | GH-C 1071 |
| | | Hay | FPD | 0.05-5.0 | 89 ± 10 | 0.05 | GH-C 1071 |
| ACR 76.3.S1 | 1976 | Nut meats | FPD | 0.02 | 90 (2) | 0.02 | GH-C 1199 |
| | | Press cake | FPD | 0.05 | 98 (2) | 0.05 | GH-C 1199 |
| | | Pressed oil | FPD | 0.05 | 86 (2) | 0.05 | GH-C 1199 |
| ACR 76.3.S1 | 1976 | Peanut oil | FPD | 0.01-0.25 | 91 ± 9 | 0.01 | GH-C 1278 |
| | | Soapstock | FPD | 0.01-.020 | 91 ± 6 | 0.02 | GH-C 1278 |
| ACR 84.4 | 1984 | Peanut kernels | FPD | 0.01-0.50 | 79 ± 8 | 0.01 | GH-C 2665 |
| | | Hulls | FPD | 0.01-2.0 | 106 ± 28 | 0.03 | GH-C 2665 |
| Sunflower | | | | | | | |
| ACR 73.5.S1 | 1973 | Sunflower seed | | 0.01-1.0 | 92 ± 8 | 0.01 | GH-C 1180 |
| ACR 76.3.S1 | 1976 | Sunflower forage | FPD | 0.01-4.0 | 87 ± 6 | 0.02 | GH-C 1371 |
| | | Seed | FPD | 0.01-0.50 | 92 ± 5 | 0.01 | GH-C 1371 |
| ACR 76.3.S1 | 1976 | Sunflower seed | FPD | 0.01-0.05 | 91 ± 9 | 0.01 | GH-C 1468 |
| | | Meal | FPD | 0.01-0.05 | 77 ± 5 | 0.01 | GH-C 1468 |
| | | Hulls | FPD | 0.01-0.05 | 66 ± 34 | 0.01 | GH-C 1468 |
| | | Oil | FPD | 0.01-0.05 | 64 ± 14 | 0.01 | GH-C 1468 |
| | | Soapstock | FPD | 0.01-0.05 | 81 ± 19 | 0.01 | GH-C 1468 |
| ACR 90.2 | 1990 | Sunflower seed | FPD | 0.01-0.10 | 78 ± 6 | 0.01 | GH-C 2683 |
| ACR 90.2 | 1990 | Sunflower seed | FPD | 0.01-0.50 | 86 ± 9 | 0.01 | GH-C 3239 |
| | | Sunflower hulls | FPD | 0.01-0.50 | 97 ± 12 | 0.01 | GH-C 3239 |
| **Animal products** | | | | | | | |
| ACR 71.1 | 1971 | Milk | FPD | 0.01-0.10 | 86 ± 4 | 0.01 | GH-C 533 |
| | | Cream | FPD | 0.01-0.10 | 87 ± 16 | 0.01 | GH-C 533 |
| Claborn & Ivey | 1971 | Bovine muscle | FPD | 0.01-0.10 | 86 ± 2 | 0.01 | GH-C 566 |
| | | Liver | FPD | 0.01-0.08 | 78 ± 5 | 0.01 | GH-C 566 |
| | | Kidney | FPD | 0.01-0.03 | 74 ± 3 | 0.01 | GH-C 566 |
| | | Omental fat | FPD | 0.01-0.10 | 88 ± 5 | 0.01 | GH-C 566 |
| | | Renal fat | FPD | 0.01-0.05 | 90 ± 4 | 0.01 | GH-C 566 |
| | | Subcutaneous fat | FPD | 0.01 | 87 ± 7 | 0.01 | GH-C 566 |
| ACR 72.1 | 1972 | Pig muscle | FPD | 0.01-1.0 | 88 ± 5 | 0.01 | GH-C 549 |
| | | Liver | FPD | 0.01-1.0 | 90 +/-9 | 0.01 | GH-C 549 |
| | | Kidney | FPD | 0.01-1.0 | 88 ± 5 | 0.01 | GH-C 549 |
| | | Fat | FPD | 0.01-1.0 | 83 ± 4 | 0.01 | GH-C 549 |
| ACR 72.3 | 1972 | Chicken muscle | FPD | 0.01-1.0 | 98 ± 7 | 0.01 | GH-C 555 |
| | | Liver | FPD | 0.01-1.0 | 90 ± 7 | 0.01 | GH-C 555 |
| | | Kidney | FPD | 0.01-1.0 | 91 ± 6 | 0.01 | GH-C 555 |
| | | Fat | FPD | 0.01-1.0 | 83 ± 6 | 0.01 | GH-C 555 |
| | | Eggs | FPD | 0.01-1.0 | 94 ± 5 | 0.01 | GH-C 555 |
| **Soil and water samples** | | | | | | | |
| ACR 77.7 | 1977 | Soil | FPD | 0.01-10.0 | 96 +/-3 | 0.01 | |
| ERC 90.13 | 1990 | Soil | FPD | 0.01-1.0 | 98 +/-9 | 0.01 | |
| ACR 71.21 | 1971 | Water | FPD | 0.001-1.0 | 92 +/-5 | 0.001 ppm | |
| ACR 71.21.S1 | 1971 | Water | FPD | 1 ppb | 98 +/-4 | 1.0 ppb | |
| ERC 78.3 | 1978 | Water | FPD | 0.01-5.0 ppb | 92 +/-6 | 0.01 ppb | |

| Method no. | Date | Sample | Detector | Fortification, mg/kg | Mean recovery | LOQ, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| ACR 79.8 | 1979 | Water | FPD | 1.0-10.0 ppb | 98 +/-2 | 1.0 ppb | |
| ACR 86.5 | 1986 | Water | FPD | 0.25-25 ppb | 94 +/-9 | 0.25 ppb | |

**Stability of pesticide residues in stored analytical samples**

Stability trials were conducted with various crop samples fortified with 0.1 and 1.0 mg/kg chlorpyrifos and stored frozen. The samples were analysed periodically by a gas chromatographic method with flame photometric detection with a validated LOQ of 0.01 mg/kg. The results are shown in Table 22. Some samples were heated with alcoholic sodium hydroxide before extraction, which converted chlorpyrifos to the pyridinol and allowed determination of the total residue. In these cases, TCP was determined by GLC with EC detection to a validated level of 0.05 mg/kg (Wetters, 1990a).

Table 22. Stability of chlorpyrifos in various substrates stored at -18°C.

| Sample | Container | Storage period, days | Fortification, mg/kg | % remaining |
|---|---|---|---|---|
| Alfalfa forage | glass | 327<br>340 | 1.0<br>0.97 | 100<br>97 |
| Alfalfa hay | glass | 327<br>340 | 1.0<br>1.0 | 110<br>100 |
| Almond hulls | glass | 258 | 0.10 | 57 [1] |
| Almond kernels | glass | 258 | 0.10 | 82 [1] |
| Apple | glass | 172<br>271 | 0.10<br>0.10 | 93<br>90 |
| Apple | polyethylene | 1351<br>1533 | 1.0<br>1.0 | 90<br>80 |
| Apple | glass | 258 | 0.10<br>0.10 | 93<br>90 |
| Apricots | glass | 258 | 0.10 | 84 [1] |
| Cherries | glass | 260<br>272 | 0.10 | 102 [1] |
| Maize, cobs | glass | 30<br>150 | 0.10<br>0.10 | 91<br>74 |
| Maize, grain | glass | 30<br>150<br>810<br>810 | 0.10<br>0.10<br>0.10<br>1.0 | 81<br>82<br>85<br>70 |
| Maize, green plant | glass | 30<br>30<br>150<br>150<br>810<br>810 | 0.10<br>0.10<br>0.10<br>0.10<br>0.10<br>1.0 | 82<br>89<br>83<br>84<br>81<br>73 |
| Maize, stalks | glass | 30<br>150<br>810<br>810 | 0.10<br>0.10<br>0.10<br>1.0 | 86<br>85<br>104<br>76 |
| Orange juice | glass | 162 | 0.10 | 79 |
| Orange peel + pulp | glass | 162 | 0.10 | 103 |
| Oranges | glass | 162<br>172 | 0.10<br>0.10 | 78<br>79 |
| Peaches | glass | 258 | 0.10 | 73 [1] |
| Pears | glass | 258 | 0.10 | 75 [1] |
| Plums | glass | 258 | 0.10 | 98 [1] |
| Sorghum, dry plant | glass | 61 | 1.0 | 83 |
| Sorghum, fodder | polyethylene | 1679 | 1.0 | 92 |
| Sorghum, grain | glass | 65 | 1.0 | 77 |
| Sorghum, grain | polyethylene | 1679 | 1.0 | 76 |
| Sorghum green | glass | 65 | 1.0 | 77 |

| Sample | Container | Storage period, days | Fortification, mg/kg | % remaining |
|---|---|---|---|---|
| plant (silage stage) | | | | |
| Sorghum, green plant | glass | 61 | 1.0 | 88 |
| Sugar beet roots | polyethylene | 1369 | 1.0 | 82 [1] |
| Sugar beet lime cake | glass | 68<br>75 | 1.0<br>1.0 | 53<br>38 |
| Sugar beet tops | polyethylene | 1369 | 1.0 | 91 [1] |
| Sugar beet dry pulp | glass | 68 | 1.0 | 88 |
| Sugar beet wet pulp | glass | 68 | 1.0 | 91 |
| Sugar beet leaves | glass | 48<br>147<br>151<br>151 | 0.10<br>0.10<br>0.10<br>1.0 | 88<br>90<br>69<br>85 |
| Sugar beet roots | glass | 48<br>147<br>151<br>151 | 0.10<br>0.10<br>0.10<br>1.0 | 74<br>89<br>73<br>63 |
| Sugar beet thin juice | glass | 69 | 1.0 | 92 |
| Sugar beet diffusion juice | glass | 69 | 1.0 | 93 |
| Sugar beet thick juice | glass | 69 | 1.0 | 90 |
| Sweet corn, kernels | glass | 30<br>150 | 0.10<br>0.10 | 97<br>84 |
| Sweet corn, green plant | glass | 30<br>30<br>150<br>150 | 0.10<br>0.10<br>0.10<br>0.10 | 96<br>90<br>84<br>76 |
| Sweet corn, kernels + cobs | glass | 30<br>150 | 0.10<br>0.10 | 96<br>80 |
| Sweet corn, husks | glass | 30<br>150 | 0.10<br>0.10 | 93<br>79 |
| Sweet corn, cobs + husks | glass | 30<br>150 | 0.10<br>0.10 | 95<br>80 |
| Sweet potatoes | glass | 104 | 0.10 | 72 |
| Tomatoes | glass | 51<br>175<br>175 | 0.10<br>0.10<br>0.10 | 70<br>90<br>91 |
| Walnuts | glass | 258 | 0.10 | 77 [1] |

[1] Determined as TCP after alkaline hydrolysis

In a follow-up study in 1995, sets of fourteen 10-g replicate samples of bananas, beans, cauliflower, peaches and onions were weighed into polyethylene sample containers, fortified with 0.5 mg/kg chlorpyrifos and stored at -18°C until analysed. The results are shown in Table 23 (Khoshab and Bolton, 1995).

Table 23. Stability of chlorpyrifos in fortified substrates stored at -18°C in polythene containers.

| Sample | Storage period, days | Chlorpyrifos | |
|---|---|---|---|
| | | Fortification, mg/kg | % remaining |
| Banana peel | 34<br>126<br>247<br>370 | 0.5<br>0.5<br>0.5<br>0.5 | 92<br>92<br>83<br>96 |
| Banana pulp | 34<br>126<br>247<br>370 | 0.5<br>0.5<br>0.5<br>0.5 | 92<br>126<br>93<br>93 |
| Onion | 31<br>123<br>245<br>367 | 0.5<br>0.5<br>0.5<br>0.5 | 101<br>88<br>96<br>99 |
| Peach | 31<br>123 | 0.5<br>0.5 | 95<br>82 |

| | | | |
|---|---|---|---|
| | 245<br>367 | 0.5<br>0.5 | 95<br>99 |
| Cauliflower curd | 31<br>123<br>245<br>367 | 0.5<br>0.5<br>0.5<br>0.5 | 102<br>96<br>102<br>99 |
| Field beans | 28<br>120<br>242<br>364 | 0.5<br>0.5<br>0.5<br>0.5 | 94<br>101<br>101<br>97 |

In a study of the stability of incurred chlorpyrifos residues in animal commodities (McCollister, 1973) samples of subcutaneous fat, kidney, liver and muscle from an animal feeding study were stored at -18°C for 41, 36, 37 and 38 months respectively, and the remaining residue was determined. The percentages remaining were liver 100-150, kidney 33-130, muscle 70-193 and fat 60-86. Milk was fortified with 0.01, 0.10 and 1.0 mg/kg chlorpyrifos and stored frozen for 49 months, and the remaining residues were 69%, 74% and 74% respectively. No experimental details were reported.

**Definition of the residue**

Studies on plant and animal metabolism as well as on environmental fate indicate that the use of chlorpyrifos could leave residues of the parent compound and the main metabolite 3,5,6-trichloro-2-pyridinol (TCP) in plant and animal commodities. The 1999 JMPR considered the TCP metabolite during its deliberations, but established an ADI and an acute RfD only for the parent compound. Recent regulatory evaluations of chlorpyrifos in the USA, Australia and the EU have been consistent in the recognition of chlorpyrifos as the sole component of the residue. Analytical methods for enforcement purposes are available for the determination of chlorpyrifos residues in plant and livestock commodities, soil and water. The Meeting concluded that the residue should be defined as chlorpyrifos both for compliance with MRLs and for the estimation of dietary intake.

The octanol/water partition coefficient of chlorpyrifos, log $P_{ow}$ = 4.7, indicates that chlorpyrifos is fat-soluble. This is confirmed by the results of studies on goats and poultry, in which the concentration of labelled material in fat was up to 10 times that in muscle. The Meeting concluded that the residue should be described as fat-soluble.

## USE PATTERN

Chlorpyrifos is an insecticide used as a soil treatment (pre-plant and at planting), a seed treatment, and as a foliar spray, directed spray and dormant spray.

Registered uses of chlorpyrifos are shown in Table 24. The manufacturer submitted product labels for many countries (with English translations in most instances), but only summarized information without labels for Belgium, England, Germany, France and Portugal. Such entries are noted as "Summary only". The governments of Australia, Germany, The Netherlands, Poland, the USA and Thailand provided information on GAP or labels. The government of The Netherlands reported that it has not authorized the use of the compound on agricultural crops since 1-12-1999, and the government of the USA that the active ingredient is not authorized for use on tomatoes, and that use on apples is limited to pre-bloom dormant stages. Values in parentheses are (maximum rates) calculated from the available information.

Table 24. Registered uses of chlorpyrifos.

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Agricultural crops | France | Bait | 20 g/kg | Broadcast | Pre-plant | 1 | | | 1.0 | | Summary only |
| Agricultural crops (general treatment) | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 5.0 | | Summary only |
| Alfalfa | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 0.40 l/ha (0.19) | 7 | 20 day grazing restriction |
| Alfalfa | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 1.0 l/ha (0.48) | 21 | Max rate is 0.7 l/ha if alfalfa is to be cut for green forage immediately after 21 days. |
| Alfalfa | Spain | EC | 480 g/l | Foliar spray | | | 0.096 | | | 21 | |
| Alfalfa | Spain | WP | 250 g/kg | Foliar high volume | Early stage | 1 | 0.10 | 600 | | 21 | Summary only |
| Alfalfa | Spain | EC | 480 g/l | Foliar spray high volume | Early stages | 1 | 0.096 | 600 | | 21 | Summary only. |
| Alfalfa | Uruguay | EC | 500 g/l | At plant | | | | 80 ground 25 aerial | 0.3 l/ha (0.15) | | Formulation includes cypermethrin (50 g/l) |
| Alfalfa | USA | EC | 4 lb/gal (480 g/l) | Foliar spray, broadcast | | 1 per cutting ; 4 /yr | | 2 aerial 20 ground | 2 pt/a (1.1) | 21 @ >1 pt 14 @ 1 pt 7 @ 1/2 pt | May be applied through irrigation systems |
| Almond | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.16 l/100 l water (0.08) | 80-100 | | 21 | |
| Almond | Chile | WP | 500 g/kg | Spray | | | 0.12 l/hl | | | 14 | |
| Almond | USA | EC | 4 lb/gal (480 g/l) | Spray | Dormant | 1 | 1 pt/100 gal (0.06) | 200 gal (760) 250 gal Cali-fornia | 4 pt/a (2.2) | | Grazing restriction |
| Almond | USA | EC | 4 lb/gal (480 g/l) | Foliar spray | | 3 | | | 4 pt/a (2.2) | 14 | Grazing restriction. Use dilute or concentrate spray. |
| Almond | USA | EC | 4 lb/gal (480 g/l) | Orchard floor spray | | | | 25 gal/a (230) | 8 pt/a (4.5) | 14 | Do not exceed 16 pt/a/season. Foliar applications may also be made. |
| Almond | USA | WP | 500 g/kg | Spray | | 4 | 4 lb/100 gal (0.24) | | 8 lb/a (4.5) | 14 | One dormant + 3 foliar |
| Apple | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.15 l/ 100 l water (0.07) | 80-100 | | 30 | |
| Apple | Argentina | WP | 500 g/kg | Spray to cover total foliage | | | 0.12 kg/ 100 l water (0.06) | | | 30 | |
| Apple | Australia | WP | 500 g/kg | Spray, thorough coverage | Up to late pink (balloon ) and at end of flowering | | 0.05 | | | 7 | Do not apply for a minimum of 3 days before bees are actively foraging. |
| Apple | Australia | WP | 500 g/kg | Spray, thorough coverage | After petal fall | Two week interva ls | 0.025 | | | 7 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Apple | Australia | EC | 500 g/l | Spray. thorough coverage of branches, foliage, fruit | mid-late November and later | | 0.05 | 10-50 aerial | | 14 | |
| Apple | Belgium | EC | 480 g/l | Foliar spray | Before flower | 1 | 0.038 alt label | | 0.72 | 35 | Summary only. |
| Apple | Belgium | WP | 250 g/kg | Foliar spray | Before flower | 1 | 0.15 | | | 35 | Summary only. |
| Apple | Chile | WP | 500 g/kg | Foliar spray | November and January | | 0.12 l/hl (0.06) | | | 28 | |
| Apple | France | EC | 278 g/l | Low volume spray | BCCH 51-79 | 2 | 0.0348 | 400 | | 30 | If spray volume is less than 1000 l/ha, adjust concentration to ensure 0.35 g ai/ha |
| Apple | Italy | EC | 480 g/l 225 g/l | Foliar spray | | | 0.11 l/hl (0.053) | 1500 | (0.84) | 30 | |
| Apple | India | EC | 200 g/kg | | | | 0.05% (w/w) | | (0.010 kg/tree) | | Apply 10 -20 l of a 0.05% solution per tree. 11 day PHI pending |
| Apple | Japan | WP | 250 g/kg | Foliar spray | | 2 | 0.025 | | | 14 | |
| Apple | Korea | WP | 250 g/kg | Foliar spray | Before bloom and 20 days after petal fall (late June-early Aug) | <6 | 0.025 | | | 3 | |
| Apple | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | 1 | 2 l/hl (0.96) | | | 30 | |
| Apple | Mexico | WP | 500 g/kg | Foliar spray | | 8 | 0.18 | 1000 | | 28 | |
| Apple | New Zealand | WP | 500 g/kg | Foliar spray | At bud movement and at 2 week intervals at petal fall | Repeat | 0.025-0.038 | | 1 minimum | 14 | |
| Apple | New Zealand | EC | 400 g/l | Foliar spray | At bud movement and 10-12 days later | | 0.1 l/hl (0.04) | | 3 l/ha minimum (1.2) | | Do not use after flowering starts. |
| Apple | Portugal | EC | 480 g/l | Foliar spray high volume | fruiting | 5 | 0.096 | 1000 | | 14 | Summary only |
| Apple | South Africa | EC | 480 g/l | Spray | 3 weeks before bud swell; bud swell green tip | 2 | 0.048 | High volume | | 40 | |
| Apple | Uruguay | WP | 500 g/kg | Foliar spray | | 20-25 day repeat interval | 0.06 | | | 28 | |
| Apple | Uruguay | EC | 480 g/l | Foliar spray | | | 0.058 | 35 ground 25 aerial | | 15 | |
| Apple | UK | EC | 480 g/l | Foliar spray | April to August | 5 | | 250 | 0.96 | 14 | Summary only |
| Apple | USA | EC | 4 lb/a (480 g/l) | Spray | Dormant/ Delayed dormant | 1 | 1 pt/100 gal (0.06) | 200 - 600 gal/a (760-2300) 250 | 6 pt/a (3.4) | 70-200 variety dependent | Grazing restriction. Ground equipment only. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | | | | gal/a California | | | |
| Apricot | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.16 l/ 100 l water (0.08) | 80-100 | | 21 | |
| Apricot | South Africa | EC | 480 g/l | Spray | Dormant. Before and after pruning. | | 0.036 | High volume | | | |
| Artichokes | Spain | GR | 50 g/kg | Row or broadcast incorporated | Pre-plant At planting | 1 | | | 0.75 row 4 broadcast | | Summary only. |
| Artichokes | Spain | WP | 250 g/kg | Foliar high volume | Early stage | | 0.10 | 600 | | 21 | Summary only. |
| Artichokes | Spain | EC | 480 g/l | Foliar spray high volume | Early stages and close to harvest | | 0.096 | 500 | | 21 | Summary only. |
| Asparagus | Italy | EC | 225 g/l | Broadcast | | 1 | | 800 | 0.56 | 15 | Summary only |
| Asparagus | Spain | EC WP | 480 g/l 250 g/kg | Foliar spray, medium /high volume | | | 0.10 | 300 | | 21 | |
| Asparagus | Spain | GR | 50 g/kg | Broadcast or row, incorporated | Pre-plant At planting | 1 | | | 4.0 broadcast 0.75 row | | Summary only |
| Asparagus | USA | EC | 4 lb/gal (480 g/l) | Foliar spray | | 1 pre-harvest, 2 post-harvest in fern stage | | | 1 pt/a (1.1) | 1 | Use limited to Midwest and Pacific Northwest |
| Avocado | Australia | WP | 500 g/kg | Spray strip, applied low on tree, avoid contacting fruit. | | Every 7 days | 0.2 | 50-100 ml of water mixture per tree | | 7 | Applied as a mixture with yeast hydrolysate |
| Avocado | Australia | EC | 500 g/l | Spray | | Repeat as needed | 0.05 | | 2 l/ha (1) | 7 | Tank mix with dichlorvos |
| Avocado | New Zealand | EC | 480 g/l | Foliar spray | Pre-blossom, complete petal fall, then 21 day intervals | Repeat | 0.038 high volume to run-off | | 1 aerial | 14 | |
| Avocado | New Zealand | EC | 400 g/l | Foliar spray to run-off | Pre-blossom, at complete petal fall and then at 21 day intervals. | Repeat | 0.09 high volume | | 2 l/ha aerial (0.80) | 14 | |
| Banana | Australia | WP | 500 g/kg | Foliar spray | From flowers to first fingers | | 0.1 | 500-1000 l (air blast) | 1 (air blast) | 7 | Air blast or knapsack |
| Banana | Australia | WP | 500 g/kg | Soil around base | Spring | 1 | 0.25 | 0.6 per plant in 30 cm band | | | May also be applied as a mixture with sand (0.25 kg ai/4 kg), 30 g per plant |
| Banana | Australia | EC | 500 g/l | Foliar spray | From first flower bell to finger | Repeat as needed | 0.10 | 500 min 10 | 2 l/ha (1) | 14 | Ground only in NSW. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | exposure | | | aerial | | | |
| Banana | Australia | EC | 500 g/l | Butt and soil spray | Sept-Nov (1) Feb-Apr (2) | 2 | 0.9 (1) 0.5 (2) | | | 14 | Apply 700 ml of spray to the lower 30 cm of butt and to the surrounding soil (30 cm radius). |
| Banana | Columbia | IPE | 10 g/kg poly-ethylene | Impregnated polyethylene shrouds | After fruit bunch formation | 1 | | | | | Remove bags at normal harvest and destroy |
| Banana | Philippines | IPE | 10 g/kg poly-ethylene | Impregnated polyethylene shrouds | After fruit bunch formation | 1 | | | | | Remove bags at normal harvest and destroy |
| Banana | Spain | EC | 480 g/l | Foliar spray | | | 0.12 l/hl (0.06) 0.1 alt label | 1000 alt label | | 21 | |
| Banana | Spain | WP | 250 g/kg | Foliar spray | | | 0.10 | 500 1000 alt label | | 21 | Summary only. |
| Banana | Spain | EC | 480 g/l | Foliar spray high volume | Fruiting | 1 | 0.096 | 500 | | 21 | Summary only. |
| Banana | South Africa | EC | 480 g/l | Foliar spray, high volume | July-Feb | 4 week interval repeat | 0.036 | | | 28 | |
| Barley | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.60 l/ha (0.77) | 30 | 1.60 is for early post-emergence. 0.90 lt/ha otherwise |
| Barley | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Barley | Brazil | EC | 480 g/l | Foliar spray | | 2 | | | 0.7 l/ha (0.34) | 14 | |
| Barley | Canada | EC | 480 g/l | Foliar spray | | 5 | | 50-200 (ground) 10-30 (aerial) | 1.2 l/ha (0.58) | | Max seasonal rate is 5.275 l/ha. One application may be to the soil. |
| Barley | India | EC | 200 g/kg | Seed treatment | | | | | 1.2 g/kg seed | | |
| Barley | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.375 | | 30 day PHI pending |
| Barley | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 14 | Summary only |
| Barley | Uruguay | EC | 480 g/l | Foliar spray | | Repeat interval 15-20 days | | 35 ground 25 aerial | 0.4 l/ha (0.19) | 30 | 20 day grazing restriction |
| Beans | Australia | EC | 500 g/l | Band over plants and adjacent row | Young plants | | | | 0.8 l/ha (0.40) | | |
| Beans | Australia | EC | 500 g/l | Foliar spray | | | 0.025 | | 0.5 l/ha (0.25) | | |
| Beans | Australia | EC | 500 g/l | Soil spray | Pre-emergence | 1 | | | 0.3 l/ha (0.15) | | |
| Beans | Brazil | EC | 480 g/l | Foliar spray | | 2 | | | 1.25 l/ha (0.6) | 21 | |
| Beans | Chile | EC | 500 g/l | foliar spray | | | | | 0.6 l/ha (0.3) | 14 | Formulation is a mix with cypermethrin (50 g/l). |
| Beans | Chile | G | 150 g/kg | Broadcast or band (15-18 cm) | Pre-plant/ At planting | | | | 3 | | |
| Beans | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 4.0 | | Summary only |
| Beans | Italy | EC | 480 g/l 225 g/l | Foliar spray | | | | 600 | 1.1 l/ha (0.53) | 15 | |
| Beans | Italy | G | 75 g/kg | Row localized. Broadcast | At planting At-transplant | 1 | | | 1.2; 3 broadcast | 15 | Summary only. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | At earthing up | | | | | | |
| Beans | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.60 | | 30 day PHI pending |
| Beans | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 1.75 l/ha (0.84) | | |
| Beans | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.12 l/hl (0.06) | 200 | 0.25 l/ha (0.12) | 21 | Formulation with cypermethrin (50 g/l). |
| Beans | Portugal | GR | 50 g/kg | Broadcast incorporated | Pre-plant | 1 | | | 4.0 | | Summary only. |
| Beans | Spain | WP | 250 g/l | Foliar spray, medium/ high volume | | | 0.10 | 300 | | 21 | Summary only. See legumes. |
| Beans | Spain | GR | 50 g/kg | Broadcast row | At planting | 1 | | | 3 (4 alt label) 0.75 row | - | Summary only. |
| Beans | USA | WP (SL) | 50 g/ 100 g | Seed treatment | | 1 | | | 2 oz (1 oz ai) per 100 lb seed (0.062 kg/ 100 kg seed) | | Do not graze or feed hay from treated seed. Field, green, snap, kidney, lima, navy, string, wax. |
| Beans, green | Columbia | EC | 400 g/l | | | | | | 1.0 l/ha (0.4) | 15 | |
| Beet | Belgium | EC | 480 g/l | Foliar spray | Up to 10 leaves | 1 | | | 0.72 | | Type of beet not specified. Summary only. |
| Beetroot | Australia | EC | 500 g/l | Band over plants and adjacent row | Young plants | | | | 0.8 l/ha (0.40) | | |
| Beetroot | Australia | EC | 500 g/l | Foliar spray | | | 0.025 | | 0.5 l/ha (0.25) | | |
| Beetroot | Greece | GR | 50 g/kg | Broadcast or in-line furrows or planting holes | | | | | 0.5 furrow 1.25 broadcast | 20 | Summary only |
| Brassica (cole) | Australia | EC | 500 g/lg | Foliar spray | | 10-14 day interval | 0.2 l/hl (0.1) | | 1.0 | | Also a boom spray after planting (cabbage and cauliflower only) in NSW. |
| Brassica, head | Greece | EC WP | 480 g/l 250 g/kg | Soil band, or broadcast incorporated | Pre-plant | 1 | | 1000 broadcast | 0.96 band 2.5 broadcast | 20 | Summary only |
| Brassica, head | Greece | GR | 50 g/kg | Broadcast or in line furrows, or planting holes | Pre-plant At planting | 1 | | | 0.5 furrow 1.25 broadcast | 20 | Summary only. |
| Brassica, head | Greece | EC WP | 480 g/l 250 g/kg | Foliar spray, high volume | | 2 | 0.12 0.09 WP | 1000 | 1.2 0.9 WP | 20 | Summary only. |
| Brassica, head | Greece | EC WP | 480 g/l 250 g/kg | Bait | | 1 | | | 0.96 0.5 WP | 20 | Bait = 30 kg bran + 10 l water/ha. Summary only. |
| Brassica, flowering | Greece | EC WP | 480 g/l 250 g/kg | Foliar spray Before flowering | | 2 | 0.12 | 1000 | 1.2 | 20 | Summary only |
| Brassica, flowering | Greece | GR | 50 g/kg | Broadcast or in lines, furrows, or planting holes | Pre-plant At planting | 1 | | | 0.5 furrow 1.25 broadcast | 20 | Summary only. |
| Brassica, flowering | Greece | EC WP | 480 g/l 250 g/kg | Broadcast to soil or band, incorporated | Pre-plant | 1 | | | 1.25 band (1 WP) 2.5 broadcast | 20 | Summary only. |
| Brassica, flowering | Greece | EC | 480 g/l | Bait | | 1 | | | 0.48 | 20 | Bait = 30 kg bran + 10 l water/ha. Summary only. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Brassica | Spain | WP | 250 g/kg | Foliar high volume | Early stage | 1 | 0.10 | 600 | | 21 | Summary only. |
| Brassica | Spain | EC | 480 g/l | Foliar spray high volume | Early stages | 1 | 0.096 | 500 | | 21 | Summary only. |
| Brassica | UK | EC | 480 g/l | Plant or module drench | Transplant At planting | 1 | 0.048 transplant 0.19 module | | | 21 | 70 ml mix to base of each transplant. 1 hl mix to each 20,000 modules before planting. Summary only. |
| Brassica | UK | EC | 480 g/l | Foliar spray | | 1 | | 600 | 0.72 | 21 | Summary only |
| Brassica (cole) | USA | G | 15 g/100 g | T-band | At planting | 1 | | | 9.2 oz per 1000 ft row. 15 lb/a (2.5) for 20 in row, 7.5 lb/a for 40 in row, etc | | Do not use rutabaga tops for food or feed. Specifies: bok choy, broccoli, broccoli raab, Brussels sprouts, cabbage, cauliflower, Chinese broccoli, Chinese cabbage, collards, kale, kohlrabi, rutabaga, turnips |
| Broad bean | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Broccoli | Canada | G | 15% | In-furrow | At planting | 1 | | | 1.4 | | For 105 cm row spacing. Rate is 150 g ai/1000 m row |
| Broccoli | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 32 | |
| Broccoli | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 32 | |
| Broccoli | Canada | EC | 480 g/l | Soil spray, drench | 7-10 days and 28 days post-seed or 3 and 21 days post-transplant | 2 | 1.68 l/10 hl (0.08) | 1000 | 210 ml of EC /1000 m row (0.10 g ai/1000 m row) 7.0 l/ha (3.36) for 30 cm row space | 32 | Apply 12.5 l of solution per 100 m of row on soil, 10 cm on each side of the plant. Do not apply to harvestable portions. |
| Broccoli | Canada | WP | 50% | Spray | 2-5 leaf seedling | | | 400 | 1.125 | 32 | |
| Broccoli | South Africa | EC | 480 g/l | Foliar spray | | As needed | 0.02 | 500 | | 7 | |
| Broccoli | USA | EC | 4 lb/gal (480 g/l) | Spray band 4 in incorporated for direct seeded or spray directed to the base of new transplants. | At planting | 1 | | | 4.5 pt/a (2.5) 2.75 fl oz/1000 linear ft of row for 20 inch row spacing, 0.267 ml ai/m. | | Do not exceed 2.6 pt/a. for 40 in; 20 in, 4.5 pt/a. Do not apply to foliage. CA only. |
| Broccoli | USA | EC | 4 lb/gal (480 g/l) | Inject as sidedress on each side of row | Established plants | 1 | | 15 gal/a (140) | 1.2 fl oz/1000 linear feet of row (0.116 ml ai/m). 1.1 kg ai/ha | 30 | Double application rate for double row plantings, or 2.2 kg ai/ha. |
| Broccoli | USA | WP | 500 g/kg | Foliar | | 6 | | | 1.1 | 21 | CA and AZ only. |
| Brussels sprouts | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 32 | |
| Brussels sprouts | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 32 | |
| Brussels sprouts | Canada | EC | 480 g/l | Soil spray, | 7-10 and | 3 | 1.68 | 1000 | 210 | 32 | Apply 12.5 l solution per |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | drench | 28 days post-seed or 3 and 21 days post-transplant. | | l/10 hl (0.08) | | ml/1000 m row (0.10 g ai/1000 m row) 7.0 l/ha (3.36) for 30cm row space | | 100 m row on soil, 10 cm on each side of plant. Do not apply to harvestable portions. Do not make first application if G applied At planting. |
| Brussels sprouts | Canada | WP | 50% | Spray | 2-5 leaf seedling | 1 | | 400 | 1.125 | 32 | |
| Brussels sprouts | Germany | G | 10 g/kg | Spreading with incorpo-ration | Pre-plant | 1 | | | 1 | | |
| Brussels sprouts | Germany | G | 10 g/kg | Spreading at root collar | Post-plant | 1 | | | 0.005 g/plant | | Single plant treatment |
| Brussels sprouts | South Africa | EC | 480 g/l | Foliar spray | | As needed | 0.024 | 500 | | 7 | |
| Brussels sprouts | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.48 | 21 | |
| Brussels sprouts | USA | EC | 4 lb/a (480 g/l) | Spray band 4 in incorporated for direct seeded or spray directed to the base of new transplants. | | 1 | | | 4.5 pt/a (2.5) 2.75 fl oz/1000 linear ft of row | | Do not exceed 2.6 pt/a.; 20 in, 4.5 pt. Do not apply to foliage. |
| Brussels sprouts | USA | EC | 4lb/a (480 g/l) | Foliar spray | | 6 | | 20 gal/a (190) | 2 pt/a (1.1) | 21 | |
| Cabbage | Australia | EC | 500 g/l | Boom spray, 10 cm band | At or immedi-ately after planting | | | 500-1000 | 2 l/ha (1) | 5 | |
| Cabbage | Australia | EC | 500 g/l | Drench to plant base | | | 0.15 | | | 5 | 100 ml per plant |
| Cabbage | Belgium | EC | 480 g/l | Drench to plant base | At or just after plant | 1 | 0.096 | | | | 100 ml per plant |
| Cabbage | Brazil | EC | 480 | Foliar spray | | 2 | 60 ml/hl (0.03) | 1000 | | 21 | |
| Cabbage | Canada | G | 15% | In-furrow | At planting | 1 | | | 1.4 | | For 105 cm row spacing. Rate is 150 g ai per 1000 m row. |
| Cabbage | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 32 | |
| Cabbage | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 32 | |
| Cabbage | Canada | EC | 480 g/l | Soil spray, drench | 7-10 days and 28 days post-seed or 3 and 21 days post-transplant. | 2 | 1.68 l/10 hl (0.08) | 1000 | 210 ml/1000 m row (0.10 g ai/1000 m row) 7.0 l/ha (3.36) for 30 cm row space | 32 | Apply 12.5 l solution per 100 m row on soil, 10 cm on each side of plant. Do not apply to harvestable portions. |
| Cabbage | Canada | WP | 50% | Spray | 2-5 leaf seedling | 1 | | 400 | 1.125 | 32 | |
| Cabbage | Canada | WP | 50% | Water treatment | Transplant | 1 | 0.0162 | 0.2 l mix/plant | | 32 | Mix 65g (32.5 g ai) in 200 l of water; apply 200 ml with each plant. Do not use lindane or starter fertilizer. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Cabbage | Germany | G | 10 g/kg | Spreading with incorpo-ration | Pre-plant | 1 | | | 1 | | Savoy, white, red. |
| Cabbage | Germany | G | 10 g/kg | Spreading at root collar | Post-plant | 1 | | | 0.005 g/plant | | Single plant treatment. Savoy, white, red. |
| Cabbage | Italy | EC | 480 g/l 225 g/l | Foliar spray | | | | 600 | 1.1 l/ha (0.53) | 30 | |
| Cabbage | India | EC | 200 g/kg | | | | | 500 | 0.20 | | 7 day PHI pending. |
| Cabbage | Italy | G | 75 g/kg | Row localized. broadcast | At planting At-transplant At earthing up | 1 | | | 1.2; 3 broadcast | 30 | Summary only. |
| Cabbage | Korea | WP | 250 g/kg | Foliar spray | | <4 | 0.025 | | | 8 | |
| Cabbage | Poland | EC | 480 g/l | Foliar spray | | 2 | 0.36 l/hl (0.17) | 200 | 0.72 l/ha (0.34) | 21 | |
| Cabbage | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.15 l/hl (0.08) | 200 | 0.31 l/ha (0.016) | 21 | Formulation with cypermethrin |
| Cabbage | South Africa | EC | 480 g/l | Foliar spray | | As needed | 0.024 | 500 | | 7 | |
| Cabbage | Spain | EC WP | 480 g/l 250 g/kg | Foliar spray, medium/ high volume | | | 0.10 | 300 | | 21 | |
| Cabbage | Spain | GR | 50 g/kg | Row or broadcast, incorporated | Pre-plant At planting | 1 | | | 0.75 row 4 broadcast | | Summary only |
| Cabbage | UK | EC | 480 g/l | Overall volume spray | | | (0.12) | 600 | 0.72 | 21 | Summary only |
| Cabbage | USA | EC | 4 lb/gal (480 g/l) | Spray band 4 in incorporated for direct seeded or spray directed to the base of new transplants. | At planting | 1 | | | 4.5 pt/a (2.5) 2.75 fl oz/1000 linear ft of row | | Do not exceed 2.6 pt/a. for 40 in rows; 20 in, 4.5 pt. Do not apply to foliage |
| Cabbage | USA | EC | 4 lb/a (480 g/l) | Inject as sidedress on each side of row | Establi-shed plants | 1 | | 15 gal/a (140) | 1.2 fl oz/1000 linear feet of row (0.116 ml/m; 1.1 kg ai/ha) | 30 | Double rate for double row planting (2.2 kg ai/ha). |
| Cabbage | USA | WP | 500 g/kg | Broadcast foliar | | 6 | | | 1.1 | 21 | |
| Canola | Australia | EC | 500 g/l | Spray to soil, broadcast or in-furrow | Pre-plant At planting | | | 30 (in-furrow) | 1.5 l/ha for 1 m row spacing (0.75) | | |
| Canola | Canada | EC | 480 g/l | Foliar spray | | 4 | | 50-200 (ground) 10-40 (aerial) | 1.5 l/ha (0.72) | 21 | One application may be to the soil. |
| Carrots | Australia | EC | 500 g/l | Foliar spray | | | | | 0.35 | - | |
| Carrots | Australia | EC | 500 g/l | Band over plants and adjacent row | Young plants | | | | 0.8 l/ha (0.40) | | |
| Carrots | Canada | EC | 480 g/l | Soil spray | Pre-plant | 1 | | 400 | 4.8 l/ha (2.3) | 60 | |
| Carrots | Canada | WP | 50% | Spray | Seedling | 1 | | 400 | 2.25 | 60 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Carrots | Germany | G | 10 g/kg | Spreading with incorpo-ration | Pre-plant | 1 | | | 0.75 | | |
| Carrot | Italy | EC | 480 g/l | Foliar spray | | | | 600 | 1.0 l/ha (0.48) | 21 | |
| Carrots | Italy | G | 75 g/kg | Broadcast | At planting At earthing up | 1 | | | 3 | 21 | Summary only. |
| Carrots | Poland | EC | 480 g/l | Foliar spray | | 2 | 0.36 l/hl (0.17) | 3200 | 0.72 l/ha (0.34) | 14 | |
| Carrots | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.12 l/hl (0.06) | 200 | 0.25 l/ha (0.12) | 14 | Formulation with cypermethrin (50 g/kg) |
| Carrots | South Africa | EC | 480 g/l | Spray | At emergence of plants | 2-3 week repeat | (0.096) | 500 | 0.48 | 21 | |
| Carrots | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.96 | 14 | Summary only |
| Cassava | Australia | EC | 500 g/l | Foliar spray | seedlings | | | | 0.35 | - | |
| Cauliflower | Australia | EC | 500 g/l | Drench to plant base | | | 0.15 | | | 5 | 100 ml per plant |
| Cauliflower | Canada | G | 15% | In-furrow | At planting | 1 | | | 1.4 | | For 105 cm row spacing. Rates is 150 g ai per 1000 m row. |
| Cauliflower | Canada | EC | 480 g/l | Soil spray, drench | 7-10 days and 28 post-seed or 3 and 21 days post-transplant. | 2 | 1.68 l/10 hl (0.08) | 1000 | 210 ml EC /1000 m row (0.10 g ai/1000 m row). 7.0 l/ha (3.36) for 30 cm row space | 32 | Apply 12.5 l solution per 100 m row on soil, 10 cm on each side of plant. Do not apply to harvestable portion |
| Cauliflower | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 32 | |
| Cauliflower | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 32 | |
| Cauliflower | Canada | WP | 50% | Spray | 2-5 leaf seedling | 1 | | 400 | 1.125 | 32 | |
| Cauliflower | Germany | G | 10 g/kg | Spreading with incorpo-ration | Pre-plant | 1 | | | 1 | | |
| Cauliflower | Germany | G | 10 g/kg | Spreading at root collar | Post-plant | 1 | | | 0.005 g/plant | | Single plant treatment |
| Cauliflower | India | EC | 200 g/l | Foliar spray | | | | 500 | 0.4 | | 7 day PHI pending. |
| Cauliflower | Spain | EC WP | 480 g/l 250 g/kg | Foliar spray, medium/ high volume | | | 0.10 | 300 | | 21 | Summary only |
| Cauliflower | Spain | GR | 50 g/kg | Broadcast or row, incorporated | Pre-plant At planting | | | | 0.75 row 4 broadcast | | Summary only |
| Cauliflower | South Africa | EC | 480 g/l | Foliar spray | | As needed | 0.024 | 500 | | 7 | |
| Cauliflower | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.48 | 21 | |
| Cauliflower | USA | EC | 4 lb/a (480 g/l) | Band 4 in incorporated for direct seeding or spray directed to | At planting | 1 | | | 2.4 fl oz/1000 linear feet or row. 4 pt/a (1.9) | | Do not apply to foliage. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | base of new transplants | | | | | | | |
| Celery | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 70 | |
| Celery | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 70 | |
| Celery | Canada | WP | 50% | Spray | 2-5 leaf seedling | 1 | | 400 | 1.125 | 70 | |
| Celery | Spain | EC WP | 480 g/l 250 g/kg | Foliar spray, medium/ high volume | | | 0.10 | 300 | | 21 | Summary only |
| Celery | Spain | GR | 50 g/kg | Row or broadcast, incorporated | Pr-plant At planting | 1 | | | 0.75 row 4 broadcast | | Summary only. |
| Cereal grains | Argentina | EC | 480 g/l | Soil treatment; incorpo-rated 5-10 cm | Pre-plant | | | 100-150 | 6.0 l/ha (2.9) | | 4.0 l/ha for winter grains, incorporated 3-4 cm. |
| Cereals | Australia | EC | 500 g/l | Foliar spray | | | | | 1.5 l/ha (0.75) | 10 | 2 day gazing restriction. |
| Cereals | Australia | EC | 500 g/l | Seed treatment | | | | | 120 ml (0.06 kg ai)/100 kg seed | | Apply to seed just before sowing. |
| Cereals | Chile | G | 150 g/kg | Broadcast or band (15-18 cm) | Pre-plant/ At planting | | | | 3 | | |
| Cereals | Chile | D | 120 g/kg | Mix with fertilizer | | | | | 0.36 | | |
| Cereals | Chile | WP | 250 g/kg | In-furrow or broadcast | At planting | | | | 2.3 | | |
| Cereals | Portugal | GR | 50 g/kg | Row or broadcast, incorporate | Pre-plant At planting | 1 | | | 0.4 row 3.0 broadcast | | Summary only. |
| Cereals | Spain | DP | 30 g/kg | Dusting | Earing | 1 | | | 0.9 | 15 | Summary only. |
| Cereals | Spain | GR | 50 g/kg | Row incorporated Broadcast incorporated | At planting | 1 | | | 0.75 row 4.0 broadcast | - | Summary only |
| Cereals | UK | EC | 480 g/l | Foliar spray | | 3 | | 200 | 0.34 0.48 0.72 | 14 | Summary only. Information incomplete. Specified whet, barley, oats |
| Cherries | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.16 l/ 100 l water (0.08) | 80-100 | | 21 | |
| Cherries | Chile | WP | 500 g/kg | Spray | | | 0.12 l/hl | | | 14 | |
| Cherries | Greece | EC WP | 480 g/l 250 g/kg | Foliar spray, high volume | Flowering or fruit setting | 2 | 0.075 | 1500 | 1.9 2.1 WP | 20 | Summary only. |
| Cherries | USA | EC | 4 lb/gal (480 g/l) | Coarse, low pressure spray to trunk and lower branches | | 2 prehar-vest 1 post-harvest | 3 qt/100 gal (0.36) | | | 6 | Avoid contact with foliage in sweet cherries. Grazing prohibited. |
| Cherries, sour | USA | WP | 500 g/kg | Foliar spray | | 8 | 3 lb/100 gal (0.18) | | 8 lb/a (4.5) | 14 | |
| Cherries, sweet | USA | WP | 500 g/kg | Spray to trunk and lower limbs | | 3 | 2 lb/a00 gal (0.12) | | 8 lb/a (4.5) | 6 | Do not contact foliage |
| Chick peas | Australia | EC | 500 g/l | Soil spray | Pre- | | | | 0.3 l/ha | 10 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | emergence | | | | (0.15) | | |
| Chick peas (Bengal gram) | India | EC | 200 g/l | Foliar spry | | | | 500 | 0.6 | | 30 day PHI pending |
| Chinese cabbage | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 15 | |
| Chinese cabbage | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 15 | |
| Chinese cabbage | Canada | EC | 480 g/l | Soil spray, drench | 7-10 days post-seed or 3 days post-transplant. | 2 | 1.68 l/10 hl (0.08) | 1000 | 210 ml/1000 m row (0.10 g ai/1000 m row) 7.0 l/ha (3.36) for 30 cm row space | 15 | Apply 12.5 l solution per 100 m row on soil, 10 cm on each side of the plant. Do not apply to harvestable portions. |
| Chinese cabbage | Canada | WP | 50% | Spray | 2-5 leaf seedling | 1 | | 400 | 1.125 | 32 | |
| Chinese cabbage | Korea | WP | 250 g/kg | Foliar spray | | <4 | 0.025 | | | 8 | |
| Chinese cabbage | USA | EC | 4 lb/a (480 g/l) | Spray band 4 in incorporated for direct seeded or spray directed to the base of new transplants. | At planting | 1 | | | 4.5 pt/a (2.2) 2.75 fl oz/1000 linear ft of row | | Do not exceed 2.6 pt/a.; 20 in, 4.5 pt. Do not apply to foliage. |
| Citrus | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.12 l/100 l water (0.06) | 80-100 | | 21 | |
| Citrus | Australia | WP | 500 g/kg | Spray strip low on tree. Avoid fruit contact. | | Every 7-10 days | 0.2 | | 50-100 ml per tree (0.0002 kg ai per tree) | 7 | |
| Citrus | Australia | EC | 500 g/kg | High volume foliar spray to run-off | Nov-Mar | 2 | 0.05 | | | 14 | |
| Citrus | Australia | EC | 500 g/kg | Ground and butt spray | | 2 | 0.2 l/hl (0.1) | | 2.0 l/ha (1) | 14 | |
| Citrus | Brazil | EC | 480 | Foliar spray | | 3 | 0.2 l /hl (0.1) | 500 | | 21 | |
| Citrus | Italy | EC | 480 g/l 225 g/l | Foliar spray | | | 0.22 l/hl (0.11) | 1000 | (1.1) | 60 | 225 g/l summary only states soil application. |
| Citrus | Italy | EC | 225 g/l | Foliar spray | | 2 | 0.056 | 1500 | 0.84 | 60 | Summary only |
| Citrus | India | EC | 200 g/kg | Foliar spray | | | 0.02 % solution (w/w) | | 0.004 kg/tree | | Apply 20 l of a 0.02% solution per tree. |
| Citrus | Korea | WP | 250 g/kg | Foliar spray | | <3 | 0.031 | | | 15 | |
| Citrus | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | 0.001 l/ha (0.0005) | | | 34 | |
| Citrus | New Zealand | WP | 500 g/kg | Foliar spray, to run-off. | Pre-flower, monthly intervals from petal fall. | Repeat | 0.038 | | 0.75 minimum | 14 | If used alone, add a nonionic surfactant |
| Citrus | Portugal | EC | 480 g/l | Foliar spray, | Fruiting | 2 | 0.096 | 2500 | (2.4) | 28 | Summary only. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | high volume | | | | (1000 alt label) | | | |
| Citrus | Spain | WP | 240 g/kg | Foliar spray high volume | Fruiting | 1 | 0.072 | 2500 | | 21 | Formulation with carbonyl (375 g/kg). Summary only. |
| Citrus | Spain | EC | 260 g/l | Foliar spray high volume | Fruiting | 1 | 0.072 | 2500 | | 21 | Formulation with cypermethrin (50 g/kg). Summary only. |
| Citrus | Spain | WP | 250 g/kg | Foliar spray | Fruiting | 1 | 0.10 | 2500 (3000 alt label) | | 21 | |
| Citrus | Spain | EC | 480 g/l | Foliar spray high volume | Fruiting | 1 | 0.096 | 2500 (3000 alt label) | | 21 | Summary only. |
| Citrus | South Africa | EC | 480 g/l | Foliar spray, full cover | 80-100% pedal fall; 5-8 weeks later | 2 | 0.048 | | | 60 | |
| Citrus | South Africa | EC | 480 g/l | Foliar spray, light cover | | As needed | 0.029 | | | 60 | |
| Citrus | Thailand | EC | 200 g/l | Foliar spray, high volume | | | 0.06 | 1400 | 0.84 | 7 | |
| Citrus | Uruguay | EC | 480 g/l | Foliar spray | | | 0.048 | 35 ground 25 aerial | | 21 | |
| Citrus | USA | EC | 4 lb/gal (480 g/l) | Foliar spray | | 2 | | 100 gal/a (935) ground 15 gal/a (140) @ 7 pt/a in California and Arizona- aerial. 20 gal/a (187) @ 7 pt/a in Florida -aerial | 12 pt/a (6.7) | 21 @ 7 pints 35 @>7 pts/ a | 30 day retreatment interval; 7.5 lb ai/a/yr max (8.4). Worker reentry 2 days. Grazing prohibited. Minimum concentration is 0.5 pt per 100 gal of water per acre |
| Citrus | USA | EC | 4 lb/gal (480 g/l) | Ground spray | | Multiple | 0.5 | 25 gal/a (230) | 1 qt/a (1.1) | 28 14 @ ≤ 3qt/a/ season (3.40) | Maximum of 10 qt/a/season (11.2), except Florida, 3 qt/a/season (3.4). May be applied with sprinkler irrigation systems. Foliar applications of EC may also be made. |
| Citrus | USA | WP | 500 g/kg | Foliar spray | | 2 | 4 lb/100 gal (0.24) | | 8 lb/a (4.5) | | Do not apply to flowering trees. 30 day minimum retreatment interval |
| Citrus | USA | G | 15 g/100g | Broadcast to ground | | | | | 6.7 lb/a (1.1) | 28 | 67 lb/a/season (11 kg ai/ha/season). Grazing restriction. |
| Clover | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Coconut | India | EC | 200 g/kg | Soil drench | | | 1 g/tree | | | | 30 day PHI pending. |
| Coffee | Brazil | EC | 480 g/l | Foliar spray | | 2 | | | 1.5 l/ha (0.72) | 21 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Coffee | Columbia | EC | 400 g/l | | | | | | 3.5 l/ha (1.4) | 21 | Summary of label only. |
| Coffee | Australia | EC | 500 g/l | Spray to ground and base | Non-bearing seedlings | 0.1 | | | | | |
| Coffee | Tanzania | EC | 480 g/l | Foliar spray | | 0.96 | | | | 7 | |
| Cole crops | Australia | EC | 500 g/l | Foliar spray | | 10-14 day interval | 0.1 | 2 | | 5 | Includes cabbage, cauliflower, Brussels sprouts, broccoli |
| Collards | USA | EC | 4 lb/a (480 g/l) | Spray band 4 in incorporated for direct seeded or spray directed to the base of new transplants. | At planting | 1 | | | 4.5 pt/a (2.5) 2.75 fl oz/1000 linear ft of row | | Do not exceed 2.6 pt/a.; 20 in, 4.5 pt. Do not apply to foliage. |
| Cotton | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 2.00 l/ha (0.96) | 21 | |
| Cotton | Australia | EC | 500 g/l | Foliar spray | | Repeat as needed | | | 1.5 l/ha (075) | | Also used in-furrow, row spacing 1 m (QLD, NSW) |
| Cotton | Brazil | EC | 480 g/l | Foliar spray | | 3 | | | 2 l/ha (0.96) | 21 | |
| Cotton | Columbia | EC | 400 g/l | | | | | | 2.0 l/ha (0.8) | 14 | Summary of label only |
| Cotton | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.45 | | 12 day PHI pending |
| Cotton | India | EC | 200 g/kg | Soil spray | | | | 1000 | 0.75 | | 12 day pending PHI |
| Cotton | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | 0.35 l/hl (0.17) | | 2.0 l/ha (0.96) | 21 | |
| Cotton | Spain | EC | 480 g/l | Foliar spray | | | 0.096 | | | 21 | |
| Cotton | Spain | WP | 250 g/kg | Foliar high volume | Early stage | 1 | 0.10 | 600 (300 alt label) | | 21 | Summary only. |
| Cotton | Spain | EC | 480 g/l | Foliar spray high volume | Flowering | 1 | 0.096 | 600 | | 21 | Summary only. |
| Cotton | Spain | EC | 260 g/l | Foliar spray high volume | Flowering | 1 | 0.072 | 500 | | 21 | Formulation with cypermethrin (50 g/kg). Summary only. |
| Cotton | Spain | DP | 30 g/kg | Dusting | Flowering | 1 | | | 0.9 | - | Summary only. |
| Cotton | Spain | GR | 50 g/kg | Row Broadcast and incorporated | At planting | 1 | | | 0.75 row 4.0 broadcast | - | Summary only |
| Cotton | USA | EC | 4 lb/gal (480 g/l) | Foliar spray | | 6 | | 1 gal/a (9.4) aerial | 2 pt/a (1.1) | 14 | Grazing restriction. Do not feed gin trash. May be applied through irrigation sprinklers. |
| Courgette | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 1.0 | | |
| Cranberries | USA | EC | 4 lb/gal (480 g/l) | Foliar spray broadcast | Flower develop-ment. After 100% bloom | 2 | | 15 gal/a (140) ground 5 gal /a (47) aerial | 3 pt/a (1.7) | 60 | No applications when bog flooded. May be applied via irrigation systems. |
| Cruciferae | Belgium | EC | 480 g/l | Spray | Post-plant | 1 | | | 0.72 | 42 | Summary only. |
| Cruciferae | Belgium | GR | 50 g/kg | Spray | Post-plant | 1 | | | 0.72 | - | Summary only. |
| Cucumber | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 60 | |
| Cucumber | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 60 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Cucumber | Canada | WP | 50% | Spray | Seedling 2-5 leaf | 1 | | 400 | 1.125 | 60 | |
| Cucumber | Italy | G | 75 g/kg | Row localized. Broadcast | At planting At-transplant At earthing up | 1 | | | 1.2; 3 broadcast | 15 | Summary only. |
| Cucumber | Mexico | EC | 445 g/kg or 480 g/l | Foliar spray | | | | | 2 l/ha (0.96) | 7 | |
| Cucumber | USA | WP (SL) | 50 g/ 100 g | Seed treatment | | 1 | | | 2 oz per 100 lb seed (0.062 kg/ 100 kg seed) | | |
| Cucurbits | Australia | EC | 500 g/l | Foliar spray | | 10-14 day interval | 0.05 | | | 5 | Includes chokos, cucumbers, gherkin, marrow, melon, pumpkin, squash. |
| Currants | UK | EC | 480 g/l | Foliar spray | Flowering | 3 | | 1000 | 0.72 | 14 | Summary only. Black, red and white currants |
| Custard apple | Australia | EC | 500 g/l | Ground and trunk spray | | 2 | 2 l/hl (1) | | 10 | 14 | |
| Egg plant | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.2 | | 3 day PHI pending |
| Egg plant | Australia | EC | 500 g/l | Band over and adjacent soil | Young plants | | 0.40 | | | | |
| Egg plant | Australia | EC | 500 g/l | Foliar spray | | | 0.025 | | 0.25 | | |
| Egg plant (Aubergine) | Italy | EC | 225 g/l | Foliar broadcast | Swelling | 1 | | 800 | 0.56 | 15 | Summary only |
| Egg plant (Aubergine) | Italy | G | 75 g/kg | Ground | Transplant or earthing up | 1 | | | 1.2 | 15 | Summary only |
| Field peas | Australia | EC | 500 g/l | Soil spray | Pre-Emer-gence | | | | 0.3 l/ha (0.15) | 10 | |
| Figs | USA | EC | 4 lb/gal (480 g/l) | Soil spray incorporated 3 inc. | Dormant | 1 | | | 2 qt/a (1.9) | 7 mo | California only |
| Filberts (Hazelnuts) | Spain | WP | 250 g/kg | Foliar spray high volume | Fruiting | | 0.10 | 1500 | | 21 | Summary only. |
| Filberts (Hazelnuts) | Spain | DP | 30 g/kg | Dusting | Fruiting | 1 | | | 0.9 | 15 | Summary only. |
| Filberts (Hazelnuts) | USA | WP | 500 g/kg | Foliar spray | | 3 | 4 lb/100 gal (0.24) | | 8 lb/a (4.5) | 14 | |
| Filberts (Hazelnuts) | USA | EC | 4 lb/gal (480 g/l) | Foliar spray | | 3 | | | 4 pt/a (2.2) | 14 | Grazing restriction. Use dilute or concentrate spray. |
| Flax | Argentina | EC | 480 g/l | Soil treatment, incorporated | pre-plant | | | 100-150 | 6 l/ha (2.9) | | 30 day grazing restriction |
| Flax | Canada | EC | 480 g/l | Foliar spray | | 2 | | 50-200 (ground) 10-30 (aerial) | 1.2 l/ha (0.58) | 21 | One application may be to the soil. |
| Fodder beet | Poland | EC | 278 g/kg | Foliar high volume | | 1 | 0.28 l/hl (0.08) | 150 | 0.42 l/ha (0.12) | 30 | Formulation with dimethoate (22.2%) |
| Fodder beet | Poland | EC | 480 g/l | Spray to ground, high volume | 3-4 days pre-plant | 1 | 0.80 l/hl (0.38) | 150 | 1.20 l/ha (0.58) | 30 | |
| Fodder beet | Poland | EC | 480 g/l | Foliar high volume | After sprouting | 1 | 0.48 l/hl (0.23) | 150 | 0.72 l/ha (0.34) | 30 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Fodder beet | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.23 l/hl (0.12) | 150 | 0.35 l/ha (0.18) | 30 | Formulation with cypermethrin (50 g/l). |
| Forage crops | Australia | EC | 500 g/l | Foliar spray | | | | | 0.9 l/ha (0.45) | 10 | 2 day grazing restriction |
| Fruit | Chile | EC | 480 g/l | Tree spray | Dormant; end of winter | | 0.1 l/ hl (0.048) | | | - | |
| Fruit (trees) | Poland | EC | 278 g/kg | Foliar high volume | | 2 | 0.081 l/hl (0.022) | 500 | 0.42 (0.12 l/ha) | 30 | Formulation with dimethoate (22.2%) |
| Fruit, small | Poland | EC | 278 g/kg | Foliar high volume | 14 days before blooming and after harvest | 2 | 0.06 l/hl (0.02) | 750 | 0.42 l/ha (0.12) | 30 | Formulation with dimethoate (22.2%) |
| Fruit (trees); Fruit, small | Poland | EC | 480 g/l | Spray to ground | Before planting | 1 | 0.32 l/hl ((0.15) | 750 | 2.4 l/ha (1.2) | - | |
| Fruit (trees) | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.15 l/hl (0.08) | 500 | 0.75 l/ha (0.38) | 30 | Formulation with cypermethrin (50 g/l) |
| Fruit, small | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.10 l/hl (0.05) | 750 | 0.75 l/ha (0.38) | 30 | Formulation with cypermethrin (50 g/l) |
| Garlic | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.50 l/ha (0.72) | 21 | |
| Garlic | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | 0.16 l/100 l water (0.08) | 80-100 10 aerial | | 21 | Mix with cyper-methrin |
| Garlic | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 50 | |
| Garlic | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 50 | |
| Garlic | Canada | EC | 480 g/l | Ground drench over the row | | 1 | | 1000 | 3.5 l/ha (1.69) | 50 | |
| Gentian | Japan | WP | 250 g/kg | Foliar spray | | 2 | 0.025 | | | 2 | |
| Gherkin | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 1.0 | | |
| Ginger | Australia | WP | 500 g/kg | Spray | First shoot or first leaf. | | | | 0.45 | | |
| Ginger | Australia | EC | 500 g/l | Spray | First shoot or first leaf | | | | 0.45 | | |
| Gooseberries | UK | EC | 480 g/l | Foliar spray | | 2 | | 1000 | 0.72 | 14 | Summary only |
| Grapes | Australia | WP | 500 g/kg | Foliar spray | berry set and later | | 0.025 | | 0.25 | 7 | |
| Grapes | Australia | EC | 500 g/kg | Foliar spray | Berry set and later | | 0.025 | | 0.25 | 14 | |
| Grapes | Australia | EC | 500 g/kg | Spray | Dormant, post-pruning | | 0.05 | | | | |
| Grapes, table | Chile | WP | 500 g/kg | Foliar spray | | | 0.12 l/ha | | | 20 | |
| Grapes | France | EC | 228 g/l | Low volume row | BBCH 41-85 | | | 200 | 0.34 | 21 | |
| Grapes | Italy | EC | 480 g/l; 225 g/l | Foliar spray | Cluster closing | 2 | 0.11 l/ha (0.05) | 1000 | | 30 | Label designates "vines" as the crop. |
| Grapes | India | EC | 200 g/kg | Soil drench | | | 2 g/plant | | | | Drench at base of plant. 11 day PHI pending. |
| Grapes | Japan | EC | 400 g/l | Spray | Dormant | 1 | 0.4 | | | | |
| Grapes | New Zealand | WP | 500 g/kg | Foliar spray | At bud swell, at pre- | Repeat | 0.038 | | 0.5 minimum | 14 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | flower, at post-flower and 14 days later and then at 3 week intervals | | | | | | |
| Grapes | New Zealand | EC | 400 g/l | Foliar spray | At bud swell, pre-flower, post-flower and 14 days later, then at 3 week intervals | Repeat | 0.036 | | 1.2 l/ha minimum (0.48) | 14 | Not table grapes |
| Grapes | Portugal | EC | 480 g/l | Foliar spray, high volume | Fruiting | 2 | 0.096 | 500 (1000 alt label) | | 56 | Summary only |
| Grapes (wine) | South Africa | EC | 480 g/l | Spray | Dormant. Before bud burst | 2 | 0.096 | High volume | | | |
| Grapes (wine) | South Africa | EC | 480 g/l | Foliar spray | Four weeks after budding, 21 day repeat | Multiple | 0.036 | High volume | | 28 | |
| Grapes | Spain | EC | 480 g/l | Foliar spray | | | 0.096 | | | 21 | |
| Grapes | Spain | WP | 250 g/l | Foliar spray | Fruiting | 1 | 0.10 | 600 | | 21 | Summary only. |
| Grapes | Spain | WP | 240 g/kg | Foliar spray high volume | Fruiting | 1 | 0.048 | 500 | | 21 | Formulation with carbaryl (375 g/kg). Summary only. |
| Grapes | Spain | EC | 480 g/l | Foliar spray high volume | Fruiting | 1 | 0.096 | 500 | | 21 | Summary only. |
| Grapes | Spain | DP | 30 g/kg | Dusting | fruiting | 1 | | | 0.9 | 15 | Summary only |
| Grapes (table) | South Africa | EC | 480 g/l | Spray | Dormant. Before any signs of green material | 1 or 2 | 2X0.096 or 1X0.19 | High volume | | | Minimum of 4 l of spray mixture per vine |
| Grapes | USA | EC | 4 lb/gal (480 g/l) | Soil spray, 15 sq ft about base of each vine | | 1 | 4.5 pt/100 gal (0.27) | | 2 qt of mixture per vine (0.005 kg/vine or 15 sq ft; $7X10^{-7}$) | 35 | Do not contact fruit or foliage with spray. States East of the Rocky Mountains only. |
| Hops | | EC | 500 g/l | Foliar spray | | 1 | 0.05 l/hl (0.025) | 1500 | 1.5 l/ha (0.75) | 30 | Formulation with cypermethrin (50 g/l). |
| Hops | South Africa | EC | 480 g/l | Row spray | At plant emergence | | 0.036 | 500 | | | |
| Hops | Australia | EC | 500 g/l | Foliar spray | | | 0.05 | | | | |
| Horseradish | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.15 l/hl (0.08) | 200 | 0.31 l/ha (0.16) | 14 | Formulation with cypermethrin (50 g/l) |
| Horticultural crops | Argentina | EC | 480 g/l | Broadcast | | | 0.16 l/ 100 l water (0.08) | 80-100 | | 21 | Mix with cyper-methrin |
| Horticultural crops | Argentina | EC | 480 g/l | Soil treatment; incorp-orated | Pre-plant | | | 100-150 | 6.0 l/ha (2.9) | | |
| Kale | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 21 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Kale | USA | EC | 4 lb/a (480 g/l) | Spray band 4 in incorporated for direct seeded or spray directed to the base of new transplants. | At planting | 1 | | | 4.5 pt/a (2.2) 2.75 fl oz/1000 linear ft of row | | Do not exceed 2.6 pt/a.; 20 in, 4.5 pt. Do not apply to foliage. |
| Kiwifruit | Australia | WP | 500 g/kg | Foliar spray | Green-tip, pre-blossom or post-blossom and later | 14 day, then 21- 28 day intervals | 0.025 | | 0.5 | 7 | |
| Kiwifruit | Australia | EC | 500 g/l | Foliar spray | Green-tip and pre-blossom. Do NOT apply post-blossom | | 0.025 | | 0.5 | 14 | |
| Kiwifruit | Chile | WP | 500 g/kg | Spray | | | 0.12 l/hl | | | 14 | |
| Kiwifruit | New Zealand | WP | 500 g/kg | Foliar spray | Pre-blossom at green tip and before bloom. Post-blossom at 21-28 day intervals from 14 Feb or 60 days after complete petal fall | Repeat | 0.025 | | 0.5 | 14 | |
| Kiwifruit | New Zealand | EC | 400 g/l | Foliar spray | Pre-blossom in Oct and 7 days before introduction; post-blossom after Mar 1, 2 applications | Repeat | 0.06 l/hl (0.024) | | 1.2 l/ha minimum (0.48) | 14 | |
| Kohlrabi | USA | EC | 4 lb/a (480 g/l) | Spray band 4 in incorporated for direct seeded or spray directed to the base of new transplants. | At planting | 1 | | | 4.5 pt/a (2.5) 2.75 fl oz/1000 linear ft of row | | Do not exceed 2.6 pt/a.; 20 in, 4.5 pt. Do not apply to foliage. |
| Kohlrabi | Chile | D | 120 g/kg | Mix with fertilizer | | | | | 0.12 | | |
| Kohlrabi | Germany | G | 10 g/kg | Spreading with incorpo-ration | Pre-plant | 1 | | | 1 | | |
| Kohlrabi | Germany | G | 10 g/kg | Spreading at root collar | Post-plant | 1 | | | 0.005 g/plant | | Single plant treatment. |
| | | | | | | | | | | | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Leafy Crucifers | Australia | EC | 500 g/l | Foliar spray | | | | | 0.15 | | Includes chou moullier, kale, mustard, canola |
| Leafy Crucifers | Australia | EC | 500 g/l | Spray over plants and adjacent row | Young plants | | | | 0.8 l/.ha (0.40) | | |
| Leeks | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 3.0 | | Summary only |
| Leeks | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.12 l/ha (0.06) | 200 | 0.25 l/ha (0.12) | 21 | Formulation with cypermethrin (50 g/l). |
| Legumes | Greece | EC WP | 480 g/l 250 g/l | Foliar spray, high volume | | 2 | | | 1.2 0.9 WP | 20 | Summary only. |
| Legumes | Greece | EC | 480 g/l | Broadcast to soil or band, incorporate | Pre-plant | 1 | | | 2.5 broadcast 0.96 band | 20 | Summary only. |
| Legumes | Greece | GR | 50 g/kg | Broadcast or in line furrows or planting holes, incorporated | Pre-plant | 1 | | | 1.25 broadcast 0.5 furrow | 20 | Summary only. |
| Legumes | Greece | EC | 480 g/l | Bait | | 1 | | | 0.45 | 20 | Bait = 30 kg bran + 10 l water/ha. Summary only. |
| Legumes | Spain | EC | 480 g/l | Foliar spray high volume | | 1 | 0.096 | 500 | | 21 | Summary only. |
| Lettuce | Australia | EC | 500 g/l | Foliar spray | | | | | 0.3 l/ha (0.15) | 7 | |
| Lettuce | Australia | EC | 500 g/l | Band over plants and adjacent soil | Young plants | | | | 0.8 l/ha (0.40) | | |
| Lettuce | Greece | EC WP | 480 g/l 250 g/kg | Foliar spray, high volume | | 2 | | 600 EC | 1.2 0.9 WP | 20 | Summary only. |
| Lettuce | Greece | EC | 480 g/l | Broadcast to soil or band, incorporate | Pre-plant | 1 | | | 2.5 broadcast 0.96 band | 20 | Summary only |
| Lettuce | Greece | GR | 50 g/kg | Broadcast, incorporate | Pre-plant | 1 | | | 1.25 | 20 | Summary only. |
| Lettuce | South Africa | EC | 480 g/l | Spray | At emergence of plants | 7-14 day repeat | 0.48 | 500 | | 21 | Western Cape only. |
| Lettuce | Spain | EC WP | 480 g/l 250 g/kg | Foliar spray, medium/ high volume | | | 0.10 | 300 | | 21 | Summary only. |
| Lettuce | Spain | GR | 50 g/kg | Broadcast or row, incorporated | Pre-plant At planting | 1 | | | 0.75 row 4 broadcast | | Summary only. |
| Lentils | Canada | EC | 480 g/l | Foliar spray (boom) | Seedling stage; Flowering to early podding | 2 | | 200 (ground) 20 or 30 (aerial) | 1.2 l/ha (0.58) | 60 | 20 day PHI if rate not greater than 875 ml/ha |
| Lucerne | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Lucerne | Australia | EC | 500 g/l | Foliar spray | | | | 100 | 0.9 l/ha (0.45) | 2 | 2 day grazing restriction |
| Lupin | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Maize | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.60 l/ha (0.77) | 30 | 1.60 for early post-emergence only. Otherwise, 0.35 l/ha |
| Maize | Argentina | EC | 480 g/l | Soil treatment, incorporated | Pre-plant | | | 100-150 | 4.0 l/ha (1.9) | | |
| Maize | Australia | EC | 500 g/l | Band spray to soil | At sowing | 1 | | | 2 l/ha (1) for 1 m row spacing or 20 ml/100 m of row | | |
| Maize | Australia | EC | 500 g/l | In-furrow | At | 1 | | | 1.5 l/ha | | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | planting | | | | (0.75) for 1 m row spacing | | |
| Maize | Australia | EC | 500 g/l | Bait | At planting | 1 | | | 0.1 l per 2.5 kg bait/ha | | Bait is sorghum or wheat |
| Maize | Brazil | EC | 480 | Foliar spray | Sprouting to 35 cm or 30 days | 2 | | | 1 l/ha (0.48) | 21 | |
| Maize | Brazil | EC | 480 | Foliar spray | Sprouting to 60 days | 3 | | | 0.6 l/ha (0.29) | 21 | |
| Maize | Canada | G | 15% | Band (row), incorporated, 2.5 cm | At planting | 1 | | | 1.5 | | For 76 cm row spacing. All spacings use 15 grams (2.25 g ai) per 100 m of row. |
| Maize | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 70 | |
| Maize | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 70 | |
| Maize | Canada | WP | 50% | Spray | Seedling 2-5 leaf | 1 | | 400 | 1.125 | 70 | |
| Maize | Chile | EC | 480 g/l | Ground spray | Pre-plant, incorpo-rated 10 cm | 1 | | | 5 l/ha (2.4) | 30 | |
| Maize | Chile | EC | 480 g/l | Spray, directed to base of plants | | | | | 2 l/ha (0.96) | 30 | |
| Maize | Chile | G | 150 g/kg | Broadcast or band (15-18 cm) | Pre-plant/ At planting | | | | 3 | | |
| Maize | Chile | WP | 250 g/kg | In-furrow or broadcast | At planting | | | | 1.1 | | |
| Maize | Chile | D | 120 g/kg | Mix with fertilizer | | | | | 0.12 | | |
| Maize | Columbia | EC | 400 g/l | | | | | | 1.0 l/ha (0.4) | 14 | Summary of label only |
| Maize | France | G | 15 g/kg | Broadcast, aerial | 51. Panicle appea-rance in shaft | 2 | | | 0.375 | | Summary only. |
| Maize | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 0.5 | | Summary only |
| Maize | France | EC | 300 g/l | Low volume broadcast, incorporate | Pre-plant | | 1 | 150 | 1.5 | | |
| Maize | Italy | EC | 480 g/l | Foliar spray | 60-100 cm ht (1) end of Jul-Aug (2) | 2 | | 600 | 1.2 l/ha (0.58) (1) 1.7 l/ha (0.82) (2) | 30 | |
| Maize | Italy | EC | 225 g/l | Foliar spray | Flowering | 2 | | 600 1000 | 0.45 (1) 0.56 (2) | 30 | Summary only |
| Maize | Italy | G | 75 g/kg | Row localized. broadcast | At planting At-transplant At earthing up | 1 | | | 1.2; 3 broadcast | 30 | Summary only. |
| Maize | Mexico | G | 50 g/kg | Band treatment, soil | At planting | | | | 1.0 | | |
| Maize | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 1.5 l/ha (0.72) | 21 | |
| Maize | Portugal | WP | 50 g/kg | Row or broadcast | Pre-plant At | 1 | | | 0.5 row 5.0 | | Summary only. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | incorporate | planting | | | | broadcast | | |
| Maize | South Africa | EC | 480 g/l | Row, incorporated | At planting | 1 | (0.16) | | | | 10 ml/100 m row in 3 l water |
| Maize | South Africa | EC | 480 g/l | Broadcast to soil, incorporated (100 mm) | At planting | 1 | | 200 | 1 l/ha (0.48) | | |
| Maize | South Africa | EC | 480 g/l | Broadcast | Post planting, 30-35 after and 44-49 days after | 2 | (0.055) | | | 32 | 3.5 ml/100 m row in 3 l water |
| Maize | Spain | EC | 480 g/l | Foliar spray | | | 0.096 | 500 | (0.45) | 21 | |
| Maize | Spain | GR | 50 g/kg | Row | At planting | 1 | | | 0.75 | - | Summary only |
| Maize | Spain | WP | 250 g/kg | Foliar high volume | Early stage | 1 | 0.10 | 600 | | 21 | Summary only |
| Maize | Spain | EC | 480 g/l | Foliar spray high volume | Early stages to 2 m high | 1 | 0.096 | 600 | | 21 | Summary only. |
| Maize | Spain | GR | 50 g/kg 15 g/kg | Row | 50 cm high | 1 | | | 0.45 | | Summary only. |
| Maize | Uruguay | EC | 480 g/l | Foliar spray | | 15-20 day repeat interval | | 35 ground 25 aerial | 1.2 l/ha (0.58) | 30 | 20 day grazing restriction |
| Maize | UK | EC | 480 g/l | Foliar spray | Emergence to 2 leaves | 1 | | 200 | 0.72 | 21 | Summary only |
| Maize | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 14 | Summary only |
| Maize | USA | EC | 4 lb/gal (480 g/l) | Broadcast spray to the ground | Pre-plant, incoropo-rate 2-4 inches | 1 | | 10 gal/a (94) | 6 pt/a (3.4) | | Total use not to exceed 15 pt/a/season. |
| Maize | USA | EC | 4 lb/gal (480 g/l) | Conser-vation Tillage: T-band in open seed furrow. Broadcast to surface trash and exposed soil. | At planting Pre-plant | | | 20 gal/a (190) 5 gal/a (47) for T-band | 2 pt/a (1.12) broadcast or for 40 in row spacing. 2.6 pt/a for 30 in row spacing | | Total use not to exceed 15 pt/a/season. |
| Maize | USA | EC | 4 lb/gal (480 g/l) | Both sides of row basal treatment, ahead of cultivator | Cultivation time | | | | 2 pt/a (1.12) | | Total use not to exceed 15 pt/a/season. Cultivation application may be made in addition to use of 15G formulation |
| Maize | USA | EC | 4 lb/gal (480 g/l) | Foliar spray or band over row (6 in ht) or drop nozzles directed to base of plants | | Multiple | | 2 gal/a (19) aerial 20 gal/a (190) ground | 3 pt/a (1.7) | 35 | May be applied through sprinkler irrigation. Total use not to exceed 15 pt/a/season. 14 day grazing restriction. 35 day fodder restriction. |
| Maize | USA | G | 15 g/100g | Banded or broadcast | Pre-plant At planting Postplant at cultivation Postplant broadcast | | | | 12 oz /1000 ft row (0.17 kg ai/km) or 13.1 lb/a (2.2) for 30 in row spacing | 35 | For soil insect control, do not exceed 13.5 lb/a/season (2.3 kg ai/ha/season) or 16 oz per 1000 feet of row. For foliar insect control, do not exceed 13 lb/a/season (2.2 kg ai/ha/season) or 16 oz per |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | | | | | (banded At planting or post-plant) 13.5 lb/acre (2.3) (pre-plant broadcast) 6.5 lb/acre (1.1) (aerial or ground broadcast, postplant) | | 1000 feet of row. 14 day grazing restriction. |
| Maize | USA | WP (SL) | 50 g/ 100 g | Seed treatment | | 1 | | | 2 oz per 100 lb seed (0.062 kg/ 100 kg seed) | | Summary only |
| Mango | Australia | EC | 500 g/l | Foliar spray, thorough coverage of all branches, foliage and fruit | | | 0.05 | | | 21 | |
| Melon | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.1 | | |
| Milo | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.60 l/ha (0.77) | 30 | 1.60 for early post-emergence only. Otherwise, 0.35 l/ha |
| Milo | Uruguay | EC | 480 g/l | Foliar spray | | 15-20 day repeat interval | | 35 ground 25 aerial | 0.35 (0.14) | 20 | |
| Mint | USA | EC | 4 lb/gal (480 g/l) | Foliar spray, broadcast | | 2 | | | 4 pt/a (2.2) | 90 | One application is post-harvest Only one application during growing season. |
| Mung bean (Green gram) | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.60 | | 30 day PHI pending |
| Mustard | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.10 | | 15 day PHI pending |
| Nectarine | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.16 l/100 l water (0.08) | | | 21 | |
| Nectarine | Chile | WP | 500 g/kg | Spray | Oct-Nov | | 0.12 l/hl | | | 45 | |
| Nectarine | Uruguay | WP | 500 g/kg | Foliar spray | | 20-25 day repeat interval | 0.06 | | | | |
| Nectarine | USA | EC | 4 lb/gal (480 g/l) | Coarse spray to trunk | | 1 | 3 qt/100 gal (0.36) | | | 14 | Grazing restriction. Cover bark from ground level to scaffold limbs. |
| Nectarine | USA | EC | 4 lb/a (480 g/l) | Spray | Dormant | 1 | 1 pt/100 gal (0.06) | 200 gal (760) 250 gal California | 4 pt/a (2.2) | | Grazing restriction |
| Nectarine | USA | WP | 500 g/kg | Spray to trunk | | 1 | | | 8 lb/a (4.5) | 14 | Do not contact fruit with spray |
| Oats | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.60 l/ha (0.77) | 30 | 1.6 early post-emergence only, 0.90 otherwise |
| Oats | Australia | EC | 500 g/l | Soil spray | Pre- | | | | 0.3 l/ha | 10 | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | emergence | | | | (0.15) | | |
| Oats | Canada | EC | 480 g/l | Foliar spray | | 5 | | | 1.2 l/ha (0.58) | | Max seasonal rate is 5.275 l/ha. One application may be to the soil. |
| Oats | Chile | EC | 400 g/l | Spray | | | | | 0.4 l/ha (0.19) | 30 | |
| Oats | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 14 | Summary only |
| Oats | Uruguay | EC | 480 g/l | Foliar spray | | | | 35 ground 25 aerial | 0.4 l/ha (0.19) | 30 | 20 day grazing restriction |
| Oil seeds | Australia | EC | 500 g/l | Foliar spray | Young plants | As needed | 0.9 l/ha (0.45) | | | 10 | Grazing restriction 2 days. Specifies: cotton, mustard, linseed, peanut, poppy, canola, safflower, sunflower |
| Olive | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.12 l/100 l water (0.06) | 80-100 | | 21 | |
| Olive | Greece | EC WP | 480 g/l 250 g/kg | Foliar spray, high volume | End of July | 1 | 0.06 | 1500 | 1.8 | | Summary only |
| Olive | Spain | DP | 30 g/kg | Dusting | Flowering | 1 | | | 0.9 | - | Summary only. |
| Onions | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.50 l/ha (0.72) | 21 | |
| Onions | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | 0.16 l/100 l water. (0.08) | 80-100 10 aerial | | 21 | 160 cc/100 l water in a mix with cyper-methrin |
| Onions | Australia | EC | 500 g/l | Foliar spray, band | | | | | 0.4 | | |
| Onions | Canada | G | 15% | In-furrow | At planting | 1 | | 35 ground 25 aerial | 2.4 | 97 (picklin g) 109 (dry) | For 10 cm furrow width, 64 g (9.6 g ai) per 100 m. For 5 cm furrow width, 32 g per 100 m, or 1.2 kg ai/ha. |
| Onions (bulb) | Canada | EC | 480 g/l | Soil treatment, spray | Pre-plant | 1 | | 400 | 4.8 l/ha (2.3) | 60 | |
| Onions | Canada | WP | 50% | Spray | Seedling | 1 | | 400 | 2.25 | 60 | Do NOT use on bunching onions. |
| Onions | Columbia | EC | 400 g/l | | | | | | 1.0 l/ha (0.4) | 15 | Summary of label only. |
| Onions | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 3.0 | | Summary only |
| Onions | Germany | G | 10 g/kg | Spreading as drill treatment with soil covering | At sowing | 1 | | | 0.01 g/m row | | |
| Onions | Greece | EC | 480 g/l | Foliar spray, high volume | | 3 | | 400 | 2.5 l/ha (1.2) | 7 (20 alt label) | |
| Onions | Greece | EC | 480 g/l | Broadcast to soil or band | | 1 | | | 2.5 broadcast 0.96 band | 20 | Summary only |
| Onions | Greece | G | 50 g/kg | Broadcast to soil | | | | | 3.0 | | |
| Onions | Greece | G | 50 g/kg | In-furrow | At planting | | | | 0.5 | 21 | |
| Onions | Greece | WP | 250 g/kg | Foliar spray | At infestation | 2 week repeat | | 400 | 0.88 | 20 | |
| Onions | Greece | WP | 250 g/kg | Dust or spray to ground; broadcast or | Pre-plant | 1 | | | 2.5 broadcast 1.25 in-furrow | | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | in furrow. Soil incorpo-rate, 5-10 cm | | | | | | | |
| Onions | Greece | WP | 250 g/kg | Spray or dust | At planting; Post-planting | | | | 0.75 At planting 1.0 post-planting (0.5 in line) | | |
| Onions | Greece | G | 50 g/kg | Broadcast or in lines, furrows, or planting holes | Pre-plant At planting | | | | 0.5 furrow 1.25 broadcast | 20 | Summary only. |
| Onions | Greece | WP | 250 g/kg | Bait | | | | | 0.5 | 20 | 2 kg WP + 30 kg bran + 10 l water, per ha. |
| Onions | Italy | EC | 480 g/l 225 g/l | Foliar spray | | | | 600 | 1.1 l/ha (0.53) | 21 | |
| Onions | Italy | G | 75 g/kg | Broadcast | At planting At earthing up | 1 | | | 3 | 21 | Summary only. |
| Onions | India | EC | 200 g/kg | Foliar spray | | | | 1000 | 1.0 | | 35 day PHI pending |
| Onions | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.12 l/ha (0.06) | 200 | 0.25 l/ha (0.12) | 21 | Formulation with cypermethrin (50 g/l). |
| Onions | Portugal | G | 50 g/kg | broadcast, incorporate | Pre-plant | 1 | | | 3.0 | | Summary only. |
| Onions | Spain | WP | 250 g/kg | Foliar spray | Early and later | 1 | 0.10 | 500 | | 21 | Summary only. |
| Onions | Spain | EC | 480 g/l | Foliar spray high volume | Early stages and later | 1 | 0.096 | 500 | | 21 | Summary only. |
| Onions | Spain | G | 50 g/kg | Broadcast | At planting | 1 | | | 3 (4 alt label) | - | Summary only |
| Onions | Uruguay | EC | 480 g/l | Foliar spray | | | | | 0.1 l/ha (0.048) | 15 | |
| Onions, dry bulb | USA | G | 15 g/100 g | In-furrow | At planting | 1 | | | 3.7 oz/1000 feet row, 6.7 lb/a (1.1) | | |
| Orange, Mandarin | Japan | EC | 400 g/l | Foliar spray | | 3 | 0.04 | | | 30 | |
| Orange, Mandarin | Japan | EC | 400 g/l | Spray to trunk | | 3 | 0.4 | | | 30 | |
| Pak-choi | Canada | EC | 480 g/l | Ground spray, drench | 7-10 an 28 days post-seed or 3 and 21 days post-transplant. | 2 | 1.68 l/10 hl (0.08) | 1000 | 210 ml/1000 m row (0.10 g ai/1000 m row). 7.0 l/ha (3.36) for 30 cm row space. | 15 | Apply 12.5 l solution per 100 m row on soil, 10 cm on each side of plant. Do not apply to harvestable portion. |
| Parsnip | Australia | EC | 500 g/l | Band over plants and adjacent soil | Young plants | | | | 0.8 l/ha (0.40) | | |
| Passion fruit | Australia | WP | 500 g/kg | Spray strip at bottom of vines. Avoid fruit contact. | | Repeat 7-10 day interva l | 0.034 | 30 | (1.02) | 7 | Apply as a mixture with yeast hydrolysate. Fruit fly control. |
| Pasture | Australia | EC | 500 g/l | Foliar spray | | | | | 0.9 l/ha (0.45) | | 2 day grazing restriction |
| Pasture | Belgium | EC | 480 g/l | Foliar spray | | 1 | | | 0.72 | 21 | Summary only. |
| Pasture | Brazil | EC | 480 g/l | Foliar spray | | 2 | | | 1 l/ha (0.48) | | 7 day grazing restriction |
| Pasture | Chile | EC | 480 | Spray | | | | | 1.2 l/ha | | 3 day grazing restriction |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | | | | | (0.58) | | |
| Pasture | Columbia | EC | 400 g/l | | | | | | 3.5 l/ha (1.4) | | 15 day grazing restriction. Summary of label only. |
| Pasture | Germany | EC | 480 g/l | Spray broadcast | | 1 | 0.12 | 400 | 0.48 | 28 | |
| Pasture | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 1.5 l/ha (0.72) | | 21 day grazing restriction |
| Pasture | South Africa | EC | 480 g/l | Spray | | | | 200 | 0.25 l/ha (0.12) | | 14 day grazing restriction. Summary only. |
| Pasture | Spain | WP | 250 g/kg | Foliar spray high volume | Early | 1 | 0.10 | 40 | | | Summary only. |
| Pasture (grassland) | UK | EC | 480 g/l | Foliar spray | | 1 | | 200 | 0.72 | 14 (hay) | 14 day grazing restriction |
| Peach | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.16 l/100 l water (0.08) | 80-100 | | 21 | |
| Peach | Chile | WP | 500 g/kg | Spray | Oct-Nov | | 0.06 | | | 45 | |
| Peach | France | EC | 228 g/l | Low volume foliar | BBCH 69-79 | 3 | 0.05 | 400 | | 30 | If spray volume is less than 1000 l/ha, the conc. Is adjusted to ensure 0.5 g ai/ha. Summary only. |
| Peach | Greece | EC WP | 480 g/l 250 g/kg | Foliar spray, high volume | Flowering; green stage | 2 | 0.075 | 1500 | 1.9 2.1 WP | 20 | Summary only. |
| Peach | Italy | EC | 480 g/l 225 g/l | Foliar spray | Swelling | 2 | 0.11 l/hl (0.053) | 1500 | (0.80) | 30 | 225 g/l was summary only. |
| Peach | Japan | WP | 250 g/kg | Foliar spray | | 5 | 0.025 | | | 14 | |
| Peach | Korea | WP | 250 g/kg | Foliar spray | From early June | <5 | 0.041 | | | 14 | |
| Peach | South Africa | EC | 480 g/l | Spray | Dormant, before and after pruning | 2 | 0.03 | High volume | | | |
| Peach | Uruguay | WP | 500 g/kg | Foliar spray | | 20-25 day repeat interval | 0.06 | | | 45 | |
| Peach | Uruguay | Foliar spray | 480 g/l | Foliar spray | | | 0.058 | 35 ground 25 aerial | | 15 | |
| Peach | USA | EC | 4 lb/gal (480 g/l) | Coarse spray to trunk | | 1 | 3 qt/100 gal (0.36) | | | 14 | Grazing restriction. Cover bark from ground level to scaffold limbs. |
| Peach | USA | EC | 4 lb/a (480 g/l) | Spray | Dormant | 1 | 1 pt/100 gal (0.06) | 200 gal (760) 250 gal Cali-fornia | 4 pt/a (2.2) | | Grazing restriction |
| Peach | USA | WP | 500 g/kg | Spray to trunk | | 1 | | | 8 lb/a (4.5) | 14 | Do not contact fruit with spray |
| Peanut (Groundnut) | India | EC | 200 g/l | Foliar spray | | | | 500 | 0.20 | | 30 day PHI pending. |
| Peanut (Groundnut) | India | EC | 200 g/kg | Soil spray | | | | 1000 | 0.225 | | 30 day PHI pending. |
| Peanut | Thailand | EC | 200 g/l | Soil spray between plants | 30-35 days post-plant | 2 | | | 0.94 | | |
| Peanut | USA | EC | 4 lb/gal (480 g/l) | Soil broadcast spray, incorporate 3-4 in. | pre-plant | 1 | | 10 gal/a (94) | 4 pt/a (2.2) | 21 | Do not feed treated peanut forage or hay to dairy or meat animals. Combined total use of chlorpyrifos (EC and G) |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | | | | | | | not to exceed 4 lbs ai/a/season |
| Peanut | USA | EC | 1 lb/gal (120 g/l) | Directed Foliar spray | | 1 | | | 2 lb ai/a (2.2) | 21 | |
| Peanut | USA | G | 15g/100g | Band Or aerial broadcast | Pre-plant At planting Post-plant | Early flower-ing to peggin g for band postpla nt Broad-cast at peggin g | | | 15 oz/1000 feet row (banded at plant or postplant). 13.3 lb/a (2.2) broadcast | 21 | Do not apply more than 30 oz/1000 ft row/season or 26.6 lb/a/season (4.5). Total of pre-plant and postplant applications not to exceed 26.6 (4 lb ai)lbs/a/season (4.5). |
| Pear | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.15 l/100 l water (0.07) | | | 30 | |
| Pear | Argentina | WP | 500 g/kg | Spray to cover total foliage | | | 0.12 g/100 l water (0.06 g ai/100 l water) | | | 30 | |
| Pear | Australia | WP | 500 g/kg | Foliar spray | After pedal fall | Two week repeat interva l | 0.025 | | | 7 | |
| Pear | Australia | EC | 500 g/l | Spray, thorough coverage of branches, foliage, fruit. | mid-late November and later | | 0.05 | 10-50 aerial | | 14 | |
| Pear | Belgium | EC | 480 g/l | Foliar spray | Before flower | 1 | 0.038 alt label | | 0.72 | 35 | Summary only. |
| Pear | Belgium | WP | 250 g/kg | Foliar spray | Before flower. | 1 | 0.15 | | | 35 | Summary only. |
| Pear | Chile | WP | 500 g/kg | Spray | | | 0.12 l/ha | | | 45 | |
| Pear | Italy | EC | 480 g/l 225 g/l | Foliar spray | Swelling | 2 | 0.11 l/ha (0.053) | 1500 | | 30 | |
| Pear, Japanese | Japan | WP | 250 g/kg | Foliar spray | | 3 | 0.025 | | | 21 | |
| Pear | New Zealand | WP | 500 g/kg | Foliar spray | At bud movement and then at 2 week intervals from petal fall | Repeat | 0.038 | | 1.5 minimum | 14 | |
| Pear | Korea | WP | 250 g/kg | Foliar spray | | <3 | 0.025 | | | 21 | |
| Pear | Portugal | EC | 480 g/l | Foliar spray, high volume | | 5 | 0.096 | | | 14 | Summary only. |
| Pear | New Zealand | EC | 400 g/l | Foliar spray | At bud movement and 10-12 days later | Repeat | 0.1 | | 3 l/ha minimum (1.2) | | Do not use after flowering starts. |
| Pear | South Africa | EC | 480 g/l | Foliar | 3 weeks before bud swell; bud swell green tip | 2 | 0.048 | High volume | | | |
| Pear | UK | EC | 480 g/l | Foliar spray | April to August | 5 | | 250 | 0.96 | 14 | Summary only |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Pear | USA | EC | 4 lb/a (480 g/l) | Spray | Dormant | 1 | 1 pt/100 gal (0.06) | 200 gal (760) 250 gal California | 4 pt/a (2.2) | | Grazing restriction |
| Pear | Uruguay | WP | 500 g/kg | Foliar spray | | 20-25 day repeat interval | 0.06 | | | 45 | |
| Pear | Uruguay | EC | 480 g/l | Foliar spray | | | 0.058 | 35 ground 25 aerial | | 15 | |
| Peas | Australia | EC | 500 g/l | Band over plants and adjacent row | Young plants | | | | 0.8 l/ha (0.40) | | |
| Peas | Australia | EC | 500 g/l | Foliar spray | | | 0.025 | | 0.5 l/ha (0.25) | | |
| Peas | Australia | EC | 500 g/l | Soil spray | Pre-emergence | 1 | | | 0.3 l/ha (0.15) | | |
| Peas | Italy | EC | 480 g/l 225 g/l | Foliar spray | | | | 600 | 1.1 l/ha (0.53) | 15 | |
| Peas | Italy | G | 75 g/kg | Row localized. Broadcast | At planting At-transplant At earthing up | 1 | | | 1.2; 3 broadcast | 15 | Summary only. |
| Peas | UK | EC | 480 g/l | Spray | Preemer-gence Early post-emergence | 2 | | 200 | 0.72 | 21 | Summary only |
| Peas | USA | EC | 1 lb/gal (120 g/l) | Foliar spray | | 5 | 0.12 | | 1.12 | 28 | Grazing restriction. |
| Pecan | USA | EC | 4 lb/gal (480 g/l) | Foliar spray | | 5 | | 100 gal/a (940) ground 5 gal/a (47) aerial | 4 pt/a (2.2) | 28 | Do not exceed 20 pt/a/season. Grazing restriction. |
| Pecan | USA | WP (SL) | 500 g/kg | Seed treatment slurry | Seed | 1 | 1 oz ai/cwt | | | | Blackeyed, field, garden. |
| Pecan | USA | WP | 500 g/kg | Foliar spray | | 5 | 2 lb/100 gal (0.12) | | 8 lb/a (4.5) | 28 | |
| Pepper, Bell | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.50 l/ha (0.72) | 21 | |
| Pepper, Bell (capsicum) | Australia | EC | 500 g/l | Spray to soil on either side of plant row | | As needed | 0.035 | | 0.35 | | |
| Pepper, Bell (capsicum) | Australia | EC | 500 g/l | Foliar spray | | | 0.025 | | 0.50 | | |
| Pepper, Bell | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 40 | |
| Pepper, Bell | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 40 | |
| Pepper, Bell | Canada | WP | 50% | Spray | Seedling 2-5 leaf | 1 | | 400 | 1.125 | 40 | |
| Pepper | Italy | EC | 225 g/l | Broadcast | | 1 | | 800 | 0.56 | 15 | Summary only |
| Pepper | Italy | G | 75 g/kg | Row | At | 1 | | | 1.2; 3 | 21 | Summary only. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | localized. Broadcast | planting At-transplant At earthing up | | | | broadcast | | |
| Pepper, Bell | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 1.5 l/ha (0.72) | 7 | |
| Pepper, Bell | Mexico | WP | 500 g/kg | Foliar spray | | | | | 1.0 | 7 | |
| Pepper | Spain | WP | 250 g/kg | Foliar spray high volume | Fruiting | 2 | 0.10 | 600-1200 | | 7 | Summary only. |
| Pepper | Spain | EC | 260 g/l | Foliar spray high volume | Fruiting | 3 | 0.072 | 500 | | 21 | Formulation with cypermethrin (50 g/kg). Summary only. |
| Pepper | Spain | EC | 480 g/l | Foliar spray high volume | Fruiting and early stages | 3 | 0.096 | 500-1200 | (1.15) | 7 | Summary only. |
| Pepper | Spain | GR | 50 g/kg | Broadcast or row incorporated | Pre-plant At planting | 1 | | | 0.75 row 4 broadcast | | |
| Pepper | Spain | DP | 30 g/kg | Dusting | Fruiting | 2 | | | 0.9 | 15 | Summary only. |
| Pepper | USA | WP | 500 g/kg | Foliar spray broadcast | | 8 | | 50 gal/a (470) | 2 lb/a (1.1) | 7 | Do not apply within 10 days of transplanting. Do not apply through any type of irrigation system. |
| Pineapple | Australia | EC | 500 g/l | Foliar spray | | 90 day interval | 0.05 | | 3000 l spray/ha (1.5) | 14 | |
| Pineapple | Australia | EC | 500 g/l | Pre-plant to soil, incorporated 10 cm. | | 1 | | | 5 l/ha (2.5) | | |
| Pineapple | Columbia | EC | 400 g/l | | | | | | 2.0 l/ha (0.8) | 21 | |
| Plums | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | 0.16 l/100 l water (0.08) | 80-100 | | 21 | |
| Plums | Chile | WP | 500 g/kg | Spray | Oct-Nov | | 0.12 l/hl (0.06) | | | 45 | |
| Plums, Japanese | Japan | WP | 250 g/kg | Foliar spray | | 2 | 0.025 | | | 2 | |
| Plums | South Africa | EC | 480 g/l | Spray | Dormant; before and after pruning | 2 | 0.036 | High volume | | | |
| Plums | Uruguay | WP | 500 g/kg | | | 20-25 day repeat interval | | | | | |
| Plums | UK | EC | 480 g/l | Foliar spray | April-August | 5 | | 250 | 0.96 | 14 | Summary only |
| Plums | USA | EC | 4 lb/a (480 g/l) | Spray | Dormant | 1 | 1 pt/100 gal (0.06) | 200 gal (760) 250 gal California | 4 pt/a (2.2) | | Grazing restriction. Includes prune plums. |
| Pome fruit | Australia | EC | 500 g/l | Ground spray | | | 0.025 | 0.5 | | 14 | Spray areas infested with grasshoppers. |
| Pome fruit | Australia | EC | 500 g/l | Directed spray | | | 0.2 | | 0.06 or 100 ml mix per tree in a strip or patch low on tree. | 14 | Do not contact fruit. |
| Pome fruit | Germany | EC | 480 g/l | Broadcast | Preflower | 4 | 0.048 | 1500 | 0.723 | 14-28 | Summary only. Apple |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | foliar | Post flower | | | | | | PHI is 28 days. |
| Pome fruit | Italy | EC | 420 g/l | Overall spray, high volume | | 1 | 0.052 | | | 30 | Summary only. |
| Pome fruit | Spain | WP | 250 g/kg | Foliar spray high volume | Fruiting | 1 | 0.10 | 1500 (1000 alt label) | | 21 | Summary only |
| Popcorn | USA | EC | 4 lb/gal (480 g/l) | Broadcast spray to the ground | Pre-plant, incorpo-rate 2-4 inches | 1 | | 10 gal/a (94) | 6 pt/a (3.4) | | Total use not to exceed 15 pt/a/season. |
| Popcorn | USA | EC | 4 lb/gal (480 g/l) | Conser-vation Tillage: T-band in open seed furrow. Broadcast to surface trash and exposed soil. | At planting<br>Pre-plant | | | 20 gal/a (190) 5 gal/a (47) for T-band | 2 pt/a (1.1) broadcast or for 40 in row spacing. 2.6 pt/a for 30 in row spacing | | Total use not to exceed 15 pt/a/season. |
| Popcorn | USA | EC | 4 lb/gal (480 g/l) | Both sides of row basal treatment, ahead of cultivator | Cultivatio n time | | | | 2 pt/a (1.1) | | Total use not to exceed 15 pt/a/season. Cultivation application may be made in addition to use of 15G formulation |
| Popcorn | USA | EC | 4 lb/gal (480 g/l) | Foliar spray or band over row (6 in ht) or drop nozzles directed to base of plants | | Multipl e | | 2 gal/a (19) aerial 20 gal/a (190) ground | 3 pt/a (1.7) | 35 | May be applied through sprinkler irrigation. Total use not to exceed 15 pt/a/season. 14 day grazing restriction. 35 day fodder restriction. |
| Popcorn | USA | G | 15 g/100g | Banded or broadcast | Pre-plant At planting Postplant at cultivation Postplant broadcast | | | | 12 oz /1000 ft row (0.17 kg ai/km) (banded At planting or post-plant) 13.5 lb/acre (2.3) (pre-plant broadcast) 6.5 lb/acre (1.1) (aerial or ground broadcast, postplant) | 35 | For soil insect control, do not exceed 13.5 lb/a/season (2.3 kg ai/ha/season) or 16 oz per 1000 feet of row. For foliar insect control, do not exceed 13 lb/a/season (2.2 kg ai/ha/season) or 16 oz per 1000 feet of row. 14 day grazing restriction. |
| Potatoes | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.50 l/ha (0.72) | 21 | |
| Potatoes | Argentina | EC | 480 g/l | Soil treatment, incorporated | Pre-plant | | | 100-150 | 6 l/ha (2.9) | | |
| Potatoes | Argentina | EC | 480 g/l | Soil treatment, incorpo-rated | Preplant and immediate -ly after hilling up | 2 | | 100-150 | 4 l/ha (1.9) 2 l/ha (1.0) | | |
| Potatoes | Australia | EC | 500 g/l | Soil treatment | Pre-plant, incorpo-rated; at hilling-up | 2 | | | 6 l/ha (3); 1 l/ha (0.5) | | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Potatoes | Australia | EC | 500 g/l | Foliar spray | | | 0.025 | | 0.5 l/ha (0.25) | | |
| Potatoes | Brazil | EC | 480 | Foliar spray | | 2 | | | 1.5 l/ha (0.72) | 21 | |
| Potatoes | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 7 | |
| Potatoes | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 7 | |
| Potatoes | Canada | EC | 480 g/l | Foliar spray | | 9 | | 400-800 | 1.0 l/h (0.48) | 7 | |
| Potatoes | Canada | WP | 50% | Spray | Seedling 2-5 leaf | 1 | | 400 | 1.125 | 7 | |
| Potatoes | Chile | EC | 500 g/l | Foliar spray | | | | | 0.4 l/ha (0.2) | 14 | Formulation is a mix with cypermethrin (50 g/l). |
| Potatoes | Chile | G | 150 g/kg | Broadcast or band (15-18 cm) | Pre-plant/ At planting | | | | 3 | | |
| Potatoes | Chile | D | 120 g/kg | Mix with fertilizer | | | | | 0.12 | | |
| Potatoes | Columbia | EC | 400 g/kg | | | | | | 4.0 l/ha (1.6) | 15 | Summary of label only. |
| Potatoes | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 1.25 | | Summary only |
| Potatoes | Italy | EC | 480 g/l | Foliar spray | | Repeat as needed | | 600 | 1.6 l/ha (0.77) | 15 | |
| Potatoes | Italy | EC | 225 g/l | Foliar spray | Post-flowering | 2 | | 600 | 0.56 | 30 | Summary only |
| Potatoes | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.50 | | 35 day PHI pending. |
| Potatoes | Poland | EC | 268 g/kg | Foliar, high volume | | 2 | 0.28 l/hl (0.075) | 150 | 0.42 l/ha (0.11) | 30 | Formulation with dimethoate (22.2%) |
| Potatoes | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.20 l/hl (0.1) | 150 | 0.3 l/ha (0.15) | 30 | Formulation with cypermethrin (50 g/kg). |
| Potatoes | Portugal | EC | 480 g/l | Foliar spray, high volume | Fruiting | 2 | 0.096 | 500 (1000 alt label) | | 14 | Summary only. |
| Potatoes | Portugal | GR | 50 g/kg | Broadcast or in-row | At planting | 1 | | 5.0 broadcast 1.25 in-row | | | Summary only. |
| Potatoes | South Africa | EC | 480 g/l | Spray, with good ground coverage | Pre-plant; Post-plant | 1 pre; multiple post at 2-3 week intervals | (0.24)pre-plant | 500 post | (0.72) pre-plant for 1 m row spacing. 0.5 l/ha post plant (0.24) | 7 | Apply pre-plant in a 100 mm band just before closing furrows, 15 ml/100 m row length in 3 l water. |
| Potatoes | South Africa | EC | 480 g/l | Spray | Immediate-ly before tuber initiation and at 2 week intervals | Multiple | | 500 increase with crop density | 1l/ha (0.48) | 7 | |
| Potatoes | Spain | EC | 480 g/l | Foliar spray | | | 0.096 | 300 | | 21 | |
| Potatoes | Spain | EC | 260 g/l | Foliar spray high volume | 40 cm high | 1 | 0.072 | 500 | | 21 | Formulation with cypermethrin (50 g/kg). Summary only. |
| Potatoes | Spain | WP | 250 g/kg | Broadcast spray, high volume | | | 0.10 | 300 | | 21 | Summary only. |
| Potatoes | Spain | WP | 240 g/kg | Foliar high volume | 40 cm high | 1 | 0.048 | 750 | | 21 | Formulation with carbaryl (375 g/kg). Summary. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Potatoes | Spain | EC | 480 g/l | Foliar spray high volume | 20-40 cm high | 1 | 0.096 | 500 | | 21 | Summary only. |
| Potatoes | Spain | GR | 50 g/kg | Row Broadcast | At planting | 1 | | | 1.5 row 5 broadcast | - | Summary only. |
| Potatoes | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 21 | Summary only. |
| Potatoes | Uruguay | EC | 480 g/l | Soil treatment, band/furrow | At planting | | | | 1.5 l/ha (0.72) | | |
| Potatoes | Uruguay | EC | 480 g/l | Broadcast soil treatment, incorporate 10 cm | Pre-plant | | | | 3.5 l/ha (1.7) | | |
| Potatoes | Uruguay | EC | 480 g/l | Foliar spray | | 15-20 day repeat interval | | | 1.5 l/ha (0.72) | | |
| Pumpkin | USA | WP (SL) | 50 g/ 100 g | Seed treatment | | 1 | | | 2 oz per 100 lb seed (0.062 kg/ 100 kg seed) | | |
| Quince | Uruguay | WP | 500 g/kg | Foliar spray | | 20-25 day repeat interval | 0.06 | | | | |
| Quince | Uruguay | EC | 480 g/l | Foliar spray | | 15-20 day repeat interval | 0.058 | 35 ground 25 aerial | | 15 | |
| Radish | Australia | EC | 500 g/l | Foliar spray | | | 0.025 | | 0.5 l/ha (0.25) | | |
| Radish | Australia | EC | 500 g/l | Band over plants and adjacent soil | Young plants | | | | 0.8 l/ha (0.40) | | |
| Radish | USA | EC | 4 lb/gal (480 g/l) | Soil treatment drench in the seed furrow | At planting | 1 | | 40 gal/a (370) | 5.5 pt/a (3.1). 1.0 fl oz /1000 ft of row | | |
| Radish | USA | G | 15 g/100 g | In-furrow | At planting | 1 | | | 3.3 oz per 1000 ft row, 18.3 lb/a (3.1) | | |
| Rape seed | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Rape seed | Australia | EC | 500 g/l | Broadcast spray | | | | | 1.5 l/ha (0.75) | | Label is unclear. Implies soil application. |
| Rape | Poland | EC | 500 g/l | Foliar spray | 2 | | 0.20 l/hl (0.10) | 150 | 0.31 l/ha (0.03) | 21 | Formulation with cypermethrin (50 g/l). |
| Rape seed (oilseed rape) | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | | |
| Raspberries | UK | EC | 480 g/l | Foliar spray | May-June | 2 | | 500 | 0.72 | 7 | Summary only |
| Rhubarb | UK | EC | 480 g/l | Foliar spray | Before stem extension | 1 | | 1000 | 0.96 | 21 | Summary only |
| Rice | Australia | EC | 500 g/l | Foliar spray | | | | | 1.5 l/ha (0.75) | 10 | Maximum rate when water >15 cm or high amount of decaying material. |
| Rice | Columbia | EC | 400 g/l | | | | | | 2.0 l/ha (0.8) | 15 | Summary of label only |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| Rice | India | EC | 200 g/kg | Foliar application | | | | 500 | 0.375 | | 30 day PHI pending |
| Rice | India | EC | 200 g/kg | Seedling dip | | | 0.02% solution | | | | |
| Rice | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | 250 ground 50 aerial | 1.25 l/ha (0.6) | | 21 day grazing restriction |
| Rice | Philippines | EC | 300 g/l | Foliar | | 3 | | 160-192 | 0.3 | 7 | |
| Rice | Thailand | EC | 200 g/l | Foliar spray, high volume | | | 0.08 | 500 | 0.4 | 7 | |
| Rutabaga | Canada | G | 15% | In-furrow | At planting | 1 | | | 1.4 | | For 105 cm row spacing. Rate is 150 g ai/1000 m row. |
| Rutabaga | Canada | EC | 480 g/l | Ground spray, no incorporation | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 30 | |
| Rutabaga | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 30 | |
| Rutabaga | Canada | EC | 480 g/l | Soil spray, drench | 7-10, 28, 49 and 70 days post-seed. | 4 | 1.68 l/10 hl (0.08) | 1000 | 210 ml/1000 m row (0.10 g ai/1000 m row) 7.0 l/ha (3.36) for 30 cm row space | 30 | Apply 12.5 l solution per 100 m row on soil, 10 cm on each side of plant. Do not apply to harvestable portions. Do not use first treatment if G used at planting. |
| Rutabaga | Canada | WP | 50% | Spray | Seedling 2-5 leaf | 1 | | 400 | 1.125 | 30 | |
| Rutabaga | USA | EC | 4 lb/a (480 g/l) | Band 4 in spray over row, shallow incorporation | At planting | 1 | | 40 gal/a (370) | 4.5 pt/a (2.5). 3.3 fl oz/1000 linear ft of row | | Do not use rutabaga tops for food or feed. |
| Rye | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.60 l/ha (0.77) | 30 | 1.6 early post-emergence only, otherwise 0.90 l/ha. |
| Rye | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Rye | Uruguay | EC | 480 g/l | Foliar spray | | 15-20 day repeat interval | | 35 ground 25 aerial | 0.4 l/ha (0.19) | 30 | 20 day grazing restriction |
| Safflower | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Safflower | Australia | EC | 500 g/l | Spray to soil, broadcast or in-furrow | Pre-plant At planting | | | 30 (in-furrow) | 1.5 l/ha for 1 m row spacing (0.75) | | |
| Shallot | Australia | EC | 500 g/l | Foliar spray, band | | | | | 0.4 | | |
| Shallot | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 3.0 | | Summary only |
| Silver beet | Australia | EC | 500 g/l | Foliar spray | | | | | 0.3 l/ha (0.15) | | |
| Sorghum | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.60 l/ha (0.77) | | 1.6 for early post-emergence only. Otherwise, 0.35 l/ha |
| Sorghum | Argentina | EC | 480 g/l | Soil treatment, incorporated | Pre-plant | | | 100-150 | 4.0 l/ha (1.9) | 30 | |
| Sorghum | Australia | EC | 500 g/l | Foliar spray | | | | | 1.5 l/ha (0.75) | 2 | Do NOT use on Sugar Drip or Alpha sorghum |
| Sorghum | Australia | EC | 500 g/l | Soil, in- | At | | | 30 | 1.5 l/ha | | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | furrow | planting | | | | (0.75) or 15 ml per 100 m row, 1 m row spacing | | |
| Sorghum | Australia | EC | 500 g/l | Bait | At plant | | | | 100 ml/2.5 kg bait/ha | | Bait is wheat or sorghum |
| Sorghum | Brazil | EC | 480 g/l | Foliar spray | Sprouting to 70 days | 2 | | | 0.75 l/ha (0.36) | 21 | |
| Sorghum | Mexico | G | 50 g/kg | Band treatment, soil. | At planting | | | | 1.0 | | |
| Sorghum | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 1.5 l/ha (0.72) | | |
| Sorghum, grain | South Africa | EC | 480 g/l | Row or overall, incorporate (100 mm) | Pre-plant | 1 | 0.16 row | 200 overall | 1l/ha (0.48) | 32 | For row application, 10 ml/100 m row in 3 l water |
| Sorghum | Spain | EC WP | 480 g/l 250 g/kg | Broadcast spray, high volume | | | 0.10 | 500 | | 21 | Summary only. |
| Sorghum | Spain | GR | 1.5 k/kg | Row | | | | | 0.45 | 21 | Summary only. |
| Sorghum, grain | USA | EC | 4 lb/gal (480 g/l) | Foliar spray, broadcast or band (8-12 in). | | | | | 2 pt/a (1.1) | 30 @ 1pt/a 60 @ >1pt/a | Do not exceed 3 pints/a/season. Do not treat sweet varieties of sorghum. Do not reduce dosage for banded or directed application. |
| Sorghum (Milo) | USA | G | 15 g/100 g | Band incorporate | At planting | 1 | | | 12 oz per 1000 feet row, or 13.1 lb/a (2.0) for 30 in row spacing | | |
| Soya beans | Australia | EC | 500 g/l | Bait | At planting | | | | 0.1 l per 2.5 kg bait/ha | | Bait is sorghum or wheat |
| Soya beans | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 2.0 l/ha (0.96) | 45 | |
| Soya beans | Brazil | EC | 480 g/l | Foliar spray | | 2 | | | 1.0 l/ha (0.48) | 21 | |
| Soya beans | France | EC | 300 g/l | Low volume broadcast, incorporate | Pre-plant | 1 | | 150 | 1.5 | | Summary only |
| Soya beans | Italy | EC | 480 g/l 225 g/l | Foliar spray | Milky ripe | | | 800 | 1.2 l/ha (0.58) | 120 | |
| Soya beans | Italy | G | 75 g/kg | Row localized. Broadcast | At planting At-transplant At earthing up | 1 | | | 1.2; 3 broadcast | 120 | Summary only. |
| Soya beans | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 1.5 l/ha (0.72) | 21 | |
| Soya beans | Thailand | EC | 200 g/l; 400 g/l | Foliar spray, high volume | | 3 | 0.12 | 625 | 0.75 | 7 | 10 day retreatment interval. 14 day PHI for undefined conditions. |
| Soya beans | Uruguay | EC | 500 g/l | Foliar spray | | 15-20 day repeat interval | | 80 ground 25 aerial | 0.75 l/ha (0.38) | 45 | Formulation includes cypermethrin (50 g/l) |
| Soya beans | USA | EC | 4 lb/gal | Soil spray. | Pre-plant | 1 | | 10 | 2 pt/a | | Do not apply in-furrow. |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | (480 g/l) | Band 4-6 in At planting; 9-12 in post-emergence. | through post-emergence | | | gal/a (94) | (1.1) | | 36 in row spacing requires 8.8 oz of spray per 100 feet or row. |
| Soya beans | USA | EC | 4 lb/a (480 g/l) | Foliar spray, broadcast | | | | | 2 pt/a (1.1) | 28 | Last two treatments must be a minimum of 14 days apart. Do not exceed 6 pt (3 lb ai) per acre per season. Grazing /feeding restriction. Only 1 application after pod set on determinate soya. May be applied with sprinkler irrigation. |
| Soya beans | USA | G | 15 g/100 g | Band incorporate | At planting Post-plant | 1 | | | 8 oz per 1000 ft row, 8.7 lb/a (1.5) for 30 in row spacing. | | Do not apply as an in-furrow treatment. |
| Spinach | France | G | 50 g/kg | Broadcast | Pre-plant | 1 | | | 1.0 | | |
| Stone fruit | Australia | WP | 500 g/kg | Foliar spray | After petal fall | Two week repeat interval | 0.025 | | | 14 | |
| Stone fruit | Australia | WP | 500 g/kg | Spray strip low on tree. Avoid fruit contact. | | 7 day repeat interval | 0.2 | | 0.05-0.1 0 per tree | 14 | Applied as a mixture with yeast hydrolysate |
| Stone fruit | Australia | EC | 500 g/l | | | | 100 ml/hl (0.05) | 2000 | 2 l/ha (1) | 14 | |
| Stone fruit | Australia | EC | 500 g/l | Bait | Spring | | 200 ml (0.20 kg ai) + 250 ml sunflower oil per 5 kg cracked bait | | | 14 | Wheat or sorghum used as bait. |
| Stone fruit | Australia | EC | 500 g/l | Foliar spray, with thorough coverage of branches, foliage and fruit | | Repeat | 0.1 l/hl (0.05) | | | 14 | |
| Stone fruit | New Zealand | EC | 480 g/l | Foliar spray | Petal fall and 2 week intervals | Repeat | 0.038 | | 1 minimum | 28 | |
| Stone fruit | New Zealand | EC | 400 g/l | Foliar spray | Early bud movement | | 0.1 | | 3 l/ha minimum (1.2) | | Do not use after flowering starts. |
| Strawberries | Australia | EC | 500 g/l | Bait, broadcast to plant bases and inter-rows. | Recently ratooned or newly planted runners | | 100 ml per 10 kg bran bait | | | | |
| Strawberries | Belgium | GR | 50 g/kg | Post-plant | 1 | | | | 2 | 42 | Summary only. |
| Strawberries | Canada | EC | 480 g/l | Foliar spray | Jun 1-June 15 | 1 | | 2000 | 1.2 l/ha (0.58) | 20 | |
| Strawberries | Canada | WP | 50% | Foliar spray | June 1-June 15 | 1 | | 2000 | 0.56 | 20 | |
| Strawberries | Poland | EC | 480 g/l | Foliar spray | After harvest | | 0.19 l/hl | 750 | 1.44 l/ha (0.69) | - | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | | | (0.09) | | | | |
| Strawberries | Spain | EC | 480 g/l | Foliar spray | | | 0.096 | | | 21 | |
| Strawberries | Spain | WP | 250 g/kg | Foliar spray | Fruiting | 1 | 0.10 | 600 | | 21 | Summary only |
| Strawberries | Spain | EC | 480 g/l | Foliar spray high volume | Fruiting | 1 | 0.096 | 500 | | 21 | Summary only. |
| Strawberries | UK | EC | 480 g/l | Foliar spray | Mar-May | 3 | | 1000 | 0.72 | 7 | Summary only |
| Strawberries | UK | EC | 480 g/l | Foliar spray | Post-harvest to vines | 1 | 0.096 | | | | |
| Strawberries | USA | EC | 4 lb/gal (480 g/l) | Foliar spray broadcast | Budding | 2 | | 40 gal/a (370) | 1 qt/a (1.1) | 21 | Pre-bloom use only. Do not use when berries are forming or present. |
| Sugar beet | Canada | EC | 480 g/l | Soil OR foliar spray | | 1 | | 200 | 2.4 l/ha (1.2) | 90 | |
| Sugar beet | Chile | EC | 480 g/l | Ground spray | Pre-plant, incorpo-rated to 10 cm | 1 | | | 5 l/ha (2.4) | 30 | |
| Sugar beet | Chile | E C | 480 g/l | Spray | | | | | 2 l/ha (0.96) | 30 | |
| Sugar beet | Chile | EC | 150 g/kg | Broadcast or band (15-18 cm) | Pre-plant/ At planting | | | | 3 | | |
| Sugar beet | Chile | D | 120 g/kg | Mix with fertilizer | | | | | 0.12 | | |
| Sugar beet | Chile | WP | 250 g/kg | In-furrow or broadcast | At planting | | | | 1.1 | | |
| Sugar beet | France | EC | 300 g/l | Low volume broadcast, incorporate | Pre-plant | 1 | | 150 | 1.5 | | Also fodder beet. Summary only. |
| Sugar beet | Germany | EC | 480 g/l | Spray to soil, incorporate | Pre-plant | 1 | (0.24) | 400 | 0.96 | | |
| Sugar beet | Italy | EC | 480 g/l 225 g/l | Foliar spray | | | | 600 | 1.1 l/ha (0.53) | 60 | 225 g/l summary only stated 120 day PHI |
| Sugar beet | Italy | G | 75 g/kg | Row localized. Broadcast | At planting At-transplant At earthing up | 1 | | | 1.2; 3 broadcast | 60 | Summary only |
| Sugar beet | Poland | EC | 278 g/l | Foliar, high volume | | 1 | 0.28 l/hl (0.078) | 150 | 0.42 l/ha (0.12) | 30 | Formulation with dimethoate (22.2%). |
| Sugar beet | Poland | EC | 480 g/l | Spray to ground, high volume | 3-4 days pre-plant | 1 | 0.80 l/hl (0.38) | 150 | 1.20 l/ha (0.58) | 30 | |
| Sugar beet | Poland | EC | 480 g/l | Foliar high volume | After sprouting | 1 | 0.48 l/hl(0.23) | 150 | 0.72 l/ha (0.34) | 30 | |
| Sugar beet | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.23 l/hl (0.12) | 150 | 0.35 l/ha (0.18) | 30 | Formulation with cypermethrin (50 g/l). |
| Sugar beet | Spain | WP | 250 g/kg | Foliar spray high volume | Early stage and later. | 1 | 0.10 | 400 | | 21 | Summary only |
| Sugar beet | Spain | WP | 240 g/kg | Foliar spray high volume | 6 leaves | 1 | 0.048 | 400 | | 21 | Summary only. Formulation with carbaryl (375 g/kg). |
| Sugar beet | Spain | EC | 480 g/l | Foliar spray high volume | Several leaves and later | | 0.096 | 400 | | 21 | Summary only. |
| Sugar beet | Spain | GR | 50 g/kg | Row, broadcast and incorporated | At planting | 1 | | | 0.75 row 4.0 broadcast | - | Summary only |
| Sugar beet | Uruguay | EC | 480 g/l | Soil | Pre-plant | | | | 3.5 l/ha | | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | treatment, incorporate 2.5-5.0 cm | | | | | (1.7) | | |
| Sugar beet | Uruguay | EC | 480 g/l | Foliar spray | | 15-20 day repeat interval | | 35 ground 25 aerial | 1.2 l/ha (0.58) | | |
| Sugar Beet | UK | EC | 480 g/l | Foliar spray | 1st true leaves to late July | 2 | | 200 | 0.72 | 21 | Summary only |
| Sugar beet | USA | EC | 4 lb/gal (480 g/l) | Spray to soil Incorporated 1-2 in | Pre-plant or At planting | 1 | | | 1 pt/a (0.56) centered on a 10 in band centered on rows for furrows 30 in apart | | Do not apply as in-furrow treatment |
| Sugar beet | USA | EC | 4 lb/gal (480 g/l) | Foliar spray broadcast or band over rows | | 4 | | 2 gal/a (19) aerial 10 gal/a (94) ground 6.5 gal/a (61) band | 2 pt/a (1.1) | 30 | 8 pt/a/season maximum. |
| Sugar beet | USA | G | 15 g/100g | Band incorporate | At planting Post-emergence | 1 | | | 9 oz/1000 ft row, or 13.5 lb/a (2.3) for a 22 in row spacing | | |
| Sugar cane | Australia | EC | 500 g/l | Foliar spray | | | | 60 | 2.0 l/ha (1.0) | 7 | Grazing restriction 2 days |
| Sugar cane | Australia | EC | 500 g/l | Low pressure spray | At planting | 1 or 2 | | | 2 l/ha (1) if 1 1.5 l/ha (0.75) if 2 | | Apply onto the plant set and adjacent to soil. Repeat at 12 weeks. |
| Sugar cane | India | EC | 200 g/kg | In-furrow | | | | 1000 | 1.2 | | 90 day PHI pending |
| Sugar cane | India | EC | 200 g//kg | Foliar spray | | | | 500 | 0.30 | | 90 day PHI pending |
| Sugar cane | Mexico | EC | 480 g/l | Foliar spray | | | | | 1.75 l/ha (0.84) | 60 | |
| Sunflower | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 0.90 l/ha (0.43 kg/ha) | 25 | . |
| Sunflower | Argentina | EC | 480 g/l | Soil treatment, incorp-orated | Pre-plant | | | 100-150 | 4.0 l/ha (1.9) | 25 | |
| Sunflower | Australia | EC | 500 g/l | Bait | At planting | | | | 0.1 l/2.5 kg bait/ha | | Bait is wheat or sorghum |
| Sunflower | Australia | EC | 500 g/l | Spray to soil, broadcast or in-furrow | Pre-plant At planting | | | 30 (in-furrow) | 1.5 l/ha for 1 m row spacing (0.75) | | |
| Sunflower | Canada | EC | 480 g/l | Foliar or ground spray | | 2 | | 2; 200 (ground) | 1.2 l/ha (0.58) | 42 | One application may be to the soil. |
| Sunflower | France | EC | 300 g/l | Low volume broadcast, | Pre-plant | 1 | | 150 | 1.5 | | Summary only |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | incorporate | | | | | | | |
| Sunflower | Italy | EC | 225 g/l | Broadcast | | 1 | | 800 | 0.56 | 120 | Summary only |
| Sunflower | Italy | G | 75 g/kg | Row localized. Broadcast | At planting At-transplant At earthing up | 1 | | | 1.2; 3 broadcast | 120 | Summary only. |
| Sunflower | Spain | GR | 50 g/kg | Row | At planting | 1 | | | 0.75 | - | Summary only |
| Sunflower | Spain | EC WP | 480 g/l 250 g/kg | Foliar spray, high volume | | 0.10 | 300 | | | 21 | Summary only. |
| Sunflower | Spain | GR | 50 g/kg | Broadcast or row, incorporated | Pre-plant At planting | | | | 4.0 broadcast 0.75 row | | Summary only. |
| Sunflower | Uruguay | EC | 500 g/l | Foliar spray | | 15-20 day repeat interva l | | 80 ground 25 aerial | 0.45 l/ha (0.22) | | Formulation includes cypermethrin (50 g/l) |
| Sunflower | USA | EC | 4 lb/gal (480 g/l) | Spray to soil incorporate 2-4 in. | Pre-plant | 1 | | 10 gal/a (94) | 4 pt/a (2.2) | | |
| Sunflower | USA | EC | 4 lb/gal (480 g/l) | Foliar spray broadcast | Post-emergence | 3 | | | 3 pt/a (1.7) | 42 | Do not apply more than 9 pt/a/season (5.0 kg ai/ha/season). Grazing restriction. |
| Sunflower | USA | G | 15 g/100 g | Band incorporate | At planting | 1 | | | 8 oz/1000 ft row, or 14.5 lb/a (2.4) for 18 in row spacing. | | |
| Swede | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 21 | |
| Sweet corn | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.50 l/ha (0.72) | 21 | |
| Sweet corn | Canada | G | 15% | Band (row), incorporated 2.5cm | At planting | 1 | | | 1.5 | | For 76 cm row spacing. Rate is 15 g (2.25 g ai) per 100 m of row for all spacings. |
| Sweet corn | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | 70 | |
| Sweet corn | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | 70 | |
| Sweet corn | Canada | WP | 50% | Spray | Seedling 2-5 leaf | 1 | | 400 | 1.15 | 70 | |
| Sweet corn | UK | EC | 480 g/l | Foliar spray | Emergenc e to 2 leaves | 1 | | 200 | 0.72 | 21 | Summary only |
| Sweet corn | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 14 | Summary only |
| Sweet corn | USA | EC | 4 lb/gal (480 g/l) | Broadcast spray to the ground | Pre-plant, incorpo-rate 2-4 inches | 1 | | 10 gal/a (94) | 6 pt/a (3.4) | | Total use not to exceed 15 pt/a/season. |
| Sweet corn | USA | EC | 4 lb/gal (480 g/l) | Conser-vation Tillage: T-band in open seed furrow. Broadcast to surface trash | At planting Pre-plant | | | 20 gal/a (190) 5 gal/a (47) for T-band | 2 pt/a (1.1) broadcast or for 40 in row spacing. 2.6 pt/a for 30 in | | Total use not to exceed 15 pt/a/season (8.4 kg ai/ha/season) |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | and exposed soil. | | | | | row spacing | | |
| Sweet corn | USA | EC | 4 lb/gal (480 g/l) | Both sides of row basal treatment, ahead of cultivator | Cultivation time | | | | 2 pt/a (1.1) | | Total use not to exceed 15 pt/a/season. Cultivation application may be made in addition to use of 15G formulation |
| Sweet corn | USA | EC | 4 lb/gal (480 g/l) | Foliar spray or band over row (6 in ht) or drop nozzles directed to base of plants | | Multiple | | 2 gal/a (19) aerial 20 gal/a (190) ground | 3 pt/a (1.7) | 35 | May be applied through sprinkler irrigation. Total use not to exceed 15 pt/a/season. 14 day grazing restriction. 35 day fodder restriction. |
| Sweet corn | USA (Florida and Georgia only) | EC | 4 lb/gal (480 g/l) | Broadcast foliar spray | | 11 | | 2 gal/a (19) aerial | 1 pt/a (1.1) | 21 | May be applied through sprinkler irrigation. Do not use in conjunction with postplant application of G. |
| Sweet corn | United State (Delaware only) | EC | 4 lb/gal (480 g/l) | Broadcast foliar spray | | 13 | | | 0.5 | 7 | |
| Sweet corn | USA | G | 15 g/100g | Banded or broadcast | Pre-plant At planting Postplant at cultivation Postplant broadcast | | | | 12 oz /1000 ft row (0.17 kg ai/km) (banded At planting or post-plant) 13.5 lb/acre (2.3) (pre-plant broadcast) 6.5 lb/acre (1.1) (aerial or ground broadcast, postplant) | 35 | For soil insect control, do not exceed 13.5 lb/a/season (2.3 kg ai/ha/season) or 16 oz per 1000 feet of row. For foliar insect control, do not exceed 13 lb/a/season (2.2 kg ai/ha/season) or 16 oz per 1000 feet of row. 14 day grazing restriction. |
| Sweet corn | USA | WP | 500 g/kg | Seed treatment | | 1 | 1oz ai/cwt (0.062 kg ai/100 kg seed) | | | | |
| Sweet potato | Australia | EC | 500 g/l | Foliar spray | | | 0.015 | | 0.3 l/ha (0.15) | | |
| Sweet potato | Australia | EC | 500 g/l | Band over plants and adjacent soil | Young plants | | | | 0.8 l/ha (0.40) | | |
| Sweet potato | Thailand | EC | 200 g/l | Foliar spray, high volume | | | 0.075 | 1000 | 0.75 | 7 | |
| Sweet potato | USA | EC | 4 lb/gal (480 g/l) | Broadcast to soil, incorporate 4-6 in | pre-plant | 1 | | | 4 pt/a (2.2) | 125 | Plant crop within 14 days of treatment |
| Sweet potato | USA | G | 15 g/ 100g | Broadcast to soil, incorporate | Pre-plant | 1 | ` | | 13.5 lb/a (2.3) | 125 | |
| Tea | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.15 | | 7 day PHI pending. |
| Tea | Japan | EC | 400 g/l | Foliar spray | | 2 | 0.04 | | | 21 | |
| Tobacco | Argentina | EC | 480 | Soil treatment, incorp- | Pre-plant | | | 100-150 | 6.0 l/ha (2.9) | | |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | orated | | | | | | | |
| Tobacco | Argentina | EC | 300 g/l | spray | | 2 | 0.4% (0.04 l/10 l water) | 250 | 1 l/ha (0.3) | | |
| Tobacco | Australia | EC | 500 g/l | Spray to soil | Pre-plant | | | | 3 l/ha (1.5) | | |
| Tobacco | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.35 | | 30 day PHI pending. |
| Tobacco | Japan | EC | 400 g/l | Foliar spray | | 2 | 0.04 | | | | |
| Tobacco | South Africa | EC | 480 g/l | Spray to stem and soil | | Repeat | 0.096 | | | | 30 ml per plant |
| Tobacco | Spain | GR | 50 g/kg | Row | At planting | 1 | | | 0.75 | - | Summary only |
| Tobacco | USA | EC | 4 lb/a (480 g/l) | Broadcast spray to soil, incorporate 4 in | Pre-transplant | 1 | | | 5 qt/a (5.6) | | |
| Tobacco | USA | G | 15 g/ 100g | Broadcast to soil, incorporate | Pre-plant | 1 | | | 20 lb/a (3.4) | | |
| Tomatoes | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80-100 10 aerial | 1.50 l/ha (0.72) | 21 | |
| Tomatoes | Argentina | EC | 480 g/l | Spray to run-off; high volume | | | | 80-100 | 0.16 l/ 100 l water (0.08) | 21 | Mix with cyper-methrin |
| Tomatoes | Australia | EC | 500 g/l | Foliar spray | At flowering and later | 7 to 10 day interva l | 0.2 l/hl (0.1) | | 2 l/ha (1) | 3 | |
| Tomatoes | Australia | EC | 500 g/l | Band spray to open furrow | At planting | | 5 l/ha (2.5) | | | | |
| Tomatoes | Australia | EC | 500 g/l | Boom spray; Drench | At planting; Not specified | | 0.3 l/hl (0.15) | 1000 0.1 l to base of each plant | 2 l/ha (1) | | |
| Tomatoes | Brazil | EC | 480 g/l | Foliar spray | Small fruit | 7 | | | 1.5 l/ha (0.72) | 21 | |
| Tomatoes | Canada | EC | 480 g/l | Ground spray, no incorpo-ration | Pre-transplant | 1 | | 200 | 2.4 l/ha (1.15) | | |
| Tomatoes | Canada | EC | 480 g/l | Ground spray | 2-5 leaf | 1 | | 400 | 2.4 l/ha (1.15) | | Formulation is a mix with cypermethrin (50 g/l) |
| Tomatoes | Canada | WP | 50% | Spray | Seedling 2-5 leaf | 1 | | 400 | 1.125 | 70 | |
| Tomatoes | Chile | EC | 500 g/l | Foliar spray | | | | | 0.4 l/ha (0.2) | 14 | |
| Tomatoes | Columbia | EC | 400 g/l | | | | | | 1.0 l/ha (0.4) | 21 | Summary of label only. |
| Tomatoes | Italy | EC | 480 g/l 225 g/l | Foliar spray | | | | 600 | 1.1 l/ha (0.53) | 21 | |
| Tomatoes | Italy | G | 75 g/kg | Row localized. Broadcast | At planting At-transplant At earthing up | 1 | | | 1.2; 3 broadcast | 21 | Summary only. |
| Tomatoes | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 2.0 l/ha (0.96) | 1 | |
| Tomatoes | Mexico | WP | 500 g/kg | Foliar spray | | | | | 1.0 | 1 | |
| Tomatoes | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.12 l/hl | 200 | 0.25 l/ha (0.12) | 21 | Formulation with cypermethrin (0.50 g/l). |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | | | (0.06) | | | | |
| Tomatoes | Portugal | EC | 480 g/l | Foliar spray high volume | Fruiting | 2 | 0.096 | 500-1000 | | 14 | Summary only. |
| Tomatoes | Portugal | GR | 50 g/kg | Broadcast or in row | Pre-plant | 1 | | | 5.0 broadcast 1.25 in row | | Summary only. |
| Tomatoes | South Africa | EC | 480 g/l | Foliar spray, full cover | | Multiple, 7 day interval | 0.096 | | | 4 | |
| Tomatoes | Spain | WP | 250 g/kg | Foliar spray High volume | fruiting | 2 | 0.10 | 600-1200 | | 7 | Summary only. |
| Tomatoes | Spain | EC WP | 480 g/l 250 g/l | Foliar spray high volume | Early stages and fruiting | 3 | 0.096 | 500-1200 | | 7 | Summary only. |
| Tomatoes | Spain | EC | 260 g/l | Foliar spray high volume | Fruiting | 3 | 0.072 | 500-1200 | | 21 | Formulation with cypermethrin (50 g/kg). Summary only. |
| Tomatoes | Spain | DP | 30 g/kg | Dusting | Fruiting | 2 | | | 0.9 | 15 | Summary only. |
| Tomatoes | Spain | GR | 50 g/kg | Row broadcast | At planting | 1 | | | 0.5 row (0.75 alt label) 5 broadcast (4 alt label) | - | Summary only. |
| Tomatoes | Uruguay | EC | 500 g/l | Foliar spray | | 15-20 day repeat interval | 0.075 | 80 ground 25 aerial | | 14 | |
| Turnips | Australia | EC | 500 g/l | Foliar spray | | | 0.015 | | 0.3 l/ha (0.15) | | |
| Turnips | Australia | EC | 500 g/l | Band over plants and adjacent soil | Young plants | | | | 0.8 l/ha (0.40) | | |
| Turnips | Italy | EC | 225 g/l | Broadcast | | 1 | | 600 | 0.56 | 21 | Summary only |
| Turnips | Italy | G | 75 g/kg | Broadcast | At planting At earthing up | 1 | | | 3 | 21 | Summary only. |
| Turnips | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 21 | Summary only |
| Turnips | USA | EC | 4 lb/a (480 g/l) | Spray band 4 in incorporated for direct seeded or spray directed to the base of new transplants. | | 1 | | | 4.5 pt/a (2.5) 2.75 fl oz/1000 linear ft of row | | Do not exceed 2.6 pt/a.; 20 in, 4.5 pt. Do not apply to foliage. |
| Urd bean (Black gram) | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.60 | | 30 day PHI pending |
| Vegetables | Australia | EC | 500 g/l | Foliar spray | | | | | 0.8 l/ha (0.4) | 3 tomato 5 cole 14 asparagus | Includes asparagus, beans beetroot, broccoli, Brussels sprouts, cabbage, cauliflower, pepper, carrot, celery, egg plant, onion, peas, potato, radish, rhubarb, shallot, sweet potato, tomato turnip |
| Vegetables | Belgium | GR | 50 g/kg | Post-plant | 1 | | | | 2 | 90 | Summary only. |
| Vegetables | Belgium | EC | 480 g/l | Foliar | | | | | 0.72 | 42 | |
| Vegetables | Chile | EC | 500 g/l | Foliar spray | | | | | 0.8 l/ha | 14 | Formulation is a mix |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| (garden) | | | | | | | | | (0.4) | | with cypermethrin (50 g/l). |
| Vegetables (garden) | Chile | G | 150 g/kg | Broadcast or band (15-18 cm) | Pre-plant/ At planting | | | | 3 | | |
| Vegetables (garden) | Germany | G | 2 g/100g | Dusting drench | 1 | | | | | | Summary only |
| Vegetables (garden) | Germany | G | 1 g/100 g | Soil to row | Pre-plant Pre-emergence | 1 | | | 1.0 | | Summary only |
| Vegetables (various species) | Poland | EC | 500 g/l | Foliar spray | | 2 | 0.12 l/hl (0.06) | 200 | 0.25 l/ha (0.12) | 21 | Formulation with cypermethrin (0.50 g/l). |
| Vegetables (garden) | Spain | EC | 480 g/l | Foliar spray | | | 0.096 | | | 21<br>7 tomato<br>7 pepper | |
| Vegetables | Spain | GR | 50 g/kg | Row Broadcast | At planting | 1 | | | 1 row 4 broadcast | - | Summary only |
| Vegetables | UK | EC | 480 g/l | Foliar spray | June- July | 2 (1 lettuce) | | 600 | 0.96 | 21 (14 carrots) | Summary only. Potatoes, beet root, lettuce, leeks, onions, parsnips, celery, brassica, carrots. |
| Vegetables, stalk and stem | Australia | EC | 500 g/l | Band over plants and adjacent row | Young plants | | | | 0.8 l/ha (0.40) | 14 asparagus | Includes asparagus, celery and rhubarb |
| Vegetables, stalk and stem | Australia | EC | 500 g/l | Foliar spray | | | 0.025 | | 0.5 l/HA (0.25) | | Includes asparagus, celery and rhubarb. |
| Vegetables, stem | Greece | EC<br>WP | 480 g/l<br>250 g/l | Foliar spray, high volume | | 2 | 0.12<br>0.09 WP | 1000 | 1.2 0.9 WP | 20 | Summary only. Includes celery, .fennel, asparagus, glove artichokes, leeks. |
| Vegetables, stem | Greece | EC<br>WP | 480 g/l<br>250 g/kg | Broadcast to soil or band incorporated | Pre-plant | 1 | | | 2.5 broadcast<br>0.96 band | 20 | Summary only. Includes celery, .fennel, asparagus, glove artichokes, leeks |
| Vegetables, stem | Greece | GR | 50 g/kg | Broadcast to soil or in-line furrows or planting holes | Pre-plant At planting | 1 | | | 0.5 furrow<br>1.5 broadcast | 20 | Summary only. Includes celery, .fennel, asparagus, glove artichokes, leeks |
| Vegetables, stem | Greece | EC<br>WP | 480 g/l<br>250 g/kg | Bait | | 1 | | 10 | 0.45 | 20 | Bait = 30 kg bran + 10 l water/ha. Summary only. Includes celery, .fennel, asparagus, glove artichokes, leeks |
| Vegetables, root and tuber | Greece | EC<br>WP | 480 g/l<br>250 g/kg | Foliar spray, high volume | | 2 | | 500 | 1.2 0.9 WP | 20 | Summary only. Includes beetroot and carrots. |
| Vegetables, root and tuber | Greece | EC<br>WP | 480 g/l<br>250 g/kg | Soil band or broadcast, incorporate | Pre-plant | 1 | | | 2.5 broadcast<br>0.96 band | 20 | Summary only. Includes beetroot and carrots. |
| Walnut | Chile | WP | 500 g/kg | Spray | | | 0.12 l/hl | | | 14 | |
| Walnut | USA | WP | 500 g/kg | Foliar spray | | 3 | 4 lb/100 gal (0.24) | | 2lb ai/a (2.24) | 14 | 1 dormant + 2 foliar |
| Walnut | USA | EC | 4 lb/gal (480 g/l) | Foliar spray | | 2 | | | 4 pt/a (2.2) | 14 | Grazing restriction. Use dilute or concentrate spray. |
| Wheat | Argentina | EC | 480 g/l | Broadcast | Post-emergence | | | 80 - 100<br>10 aerial | 1.60 l/ha (0.77) | 30 | 1.6 early post-emergence only, otherwise 0.90. |
| Wheat | Australia | EC | 500 g/l | Soil spray | Pre-emergence | | | | 0.3 l/ha (0.15) | 10 | |
| Wheat | Brazil | EC | 480 g/l | Foliar spray | | 2 | | | 1.5 l/ha (0.72) | 21 | |
| Wheat | Canada | EC | 480 g/l | Foliar spray | | 5 | | | 1.2 l/ha (0.58) | 60 | Max seasonal rate is 5.275 l/ha. One application may be to the |

| Crop | Country | Formulation | | Application | | | | | | PHI, days | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Type | Conc. of ai | Method | Growth stage | No. | kg ai/hl | Water l/ha | kg ai/ha | | |
| | | | | | | | | | | | soil. |
| Wheat | Chile | EC | 480 g/l | Spray | | | | | 0.4 l/ha (0.19) | 30 | |
| Wheat | India | EC | 200 g/kg | Seed treatment | | | | | 0.8 g / kg seed | | |
| Wheat | India | EC | 200 g/kg | Foliar spray | | | | 500 | 0.6 | | 30 day PHI pending |
| Wheat | Mexico | EC | 480 g/l or 445 g/kg | Foliar spray | | | | | 1.0 l/ha (0.48) | 21 | |
| Wheat | South Africa | EC | 480 g/l | Foliar spray | | Multiple | | 200 ground 30 aerial | 2.0 | 32 | |
| Wheat (winter) | UK | EC | 480 g/l | Overall volume spray | Pre-emergence | 1 | | 200 | 1.92 | | Summary only |
| Wheat (winter) | UK | EC | 480 g/l | Overall volume spray | | 3 | | | 0.72 | 14 | Summary only. 2.16 kg ai/ha/season |
| Wheat (spring) | UK | EC | 480 g/l | Overall volume spray | | | | 200 | 0.72 | 14 | Summary only |
| Wheat | Uruguay | EC | 480 g/l | Foliar spray | | 15-20 day repeat interval | | 35 ground 25 aerial | 0.4 l/ha (0.19) | 30 | 20 day grazing restriction |
| Wheat | USA | EC | 4 lb/gal (480 g/l) | Foliar spray | | 2 | | 2 gal/a (19) | 1 pt/a (0.56) | 28 | 14 day grazing restriction. Ground, aerial, or sprinkler irrigation application. |
| Wheat | USA | EC (E-SG) | 4 lb/gal (480 g/l) | Foliar (aerial, ground, sprinkler irrigation) | | 2 | | 2 gal/a (19) | 0.5 lb/a (0.56) | 28 | 14 day grazing restriction. 28 day feeding (straw) |

## RESIDUES RESULTING FROM SUPERVISED TRIALS

Residue trials were reported by the manufacturer and by the governments of Germany, Poland and Thailand. The results of the trials are tabulated according to the CCPR Classification as follows.

| Commodity | Table no. |
|---|---|
| Mandarin orange | 25 |
| Orange | 26 |
| Grapefruit | 27 |
| Lemon | 28 |
| Apple (Chile) | 29 |
| Apple (Italy) | 30 |
| Apple (New Zealand, Germany, UK) | 31 |
| Apple (USA) | 32 |
| Apple (Brazil) | 33 |
| Pear | 34 |
| Peach (Chile) | 35 |
| Peach (Greece, Spain, Italy) | 36 |
| Peach (USA) | 37 |
| Plum | 38 |
| Blueberry & Caneberry | 39 |
| Raspberry & Gooseberry | 40 |
| Strawberry (UK) | 41 |
| Strawberry (USA) | 42 |
| Grape (France) | 43 |
| Grape (Italy, Greece, South Africa) | 44 |
| Grape (USA) | 45 |
| Banana (Australia, South Africa) | 46 |
| Banana (Spain) | 47 |
| Banana (Ecuador, Costa Rica, Honduras, Philippines) | 48 |
| Kiwifruit | 49 |
| Onions (Canada, USA) | 50 |
| Onions (Greece) | 51 |
| Onions (UK) | 52 |
| Broccoli | 53 |
| Brussels sprouts | 54 |
| Cabbage (Brazil) | 55 |
| Cabbage (South Africa) | 56 |
| Cabbage (UK) | 57 |
| Cabbage USA) | 58 |
| Chinese Cabbage | 59 |
| Cauliflower | 60 |
| Pepper (Spain) | 61 |
| Pepper (USA) | 62 |
| Tomato (Brazil, Mexico, Spain) | 63 |
| Tomato (Australia, South Africa) | 64 |
| Tomato (USA) | 65 |
| Egg plant | 66 |
| Lettuce (Spain) | 67 |
| Lettuce (USA) | 68 |
| Lettuce (USA) | 69 |
| Common bean (Italy) | 70 |
| Common bean (USA) | 71 |
| Pea (USA) | 72 |
| Pea (UK) | 73 |
| Soya bean (USA) | 74 |
| Soya bean (Thailand) | 75 |
| Carrots | 76 |
| Potato (Brazil) | 77 |
| Potato (Brazil) | 78 |
| Potato (Australia, Columbia, Poland) | 79 |

| Commodity | Table no. |
|---|---|
| Potato (Canada, South Africa, UK) | 80 |
| Sugar beet (France, Germany, UK) | 81 |
| Sugar beet (Japan) | 82 |
| Sugar beet (Canada, USA) | 83 |
| Maize (Brazil) | 84 |
| Maize (Brazil) | 85 |
| Maize (USA) | 86 |
| Sweet corn (Canada, USA) | 87 |
| Sweet corn (USA) | 88 |
| Rice (Australia, Columbia) | 89 |
| Rice (Philippines, Vietnam, Thailand) | 90 |
| Rice (India) | 91 |
| Sorghum | 92 |
| Wheat (Brazil) | 93 |
| Wheat (Germany, UK) | 94 |
| Wheat (Canada, USA) | 95 |
| Alfalfa (USA) | 96 |
| Alfalfa (USA) | 97 |
| Almond | 98 |
| Pecan | 99 |
| Walnut | 100 |
| Cotton seed | 101 |
| Peanut | 102 |
| Sunflower | 103 |
| Coffee | 104 |

The results are not corrected for recoveries. Double underlined results are from trials according to maximum GAP (±30%) appropriate for the estimation of STMRs and maximum residue levels. Application rates in parentheses are calculated values.

Citrus fruits. Field trials were reported on mandarins/clementines (Italy, Spain), lemons (Spain, USA), oranges/tangelos (South Africa, USA) and grapefruit (USA).

In thirteen supervised trials in Spain from 1992 to 1995 (Khoshab *et al.*, 1993a, Khoshab and Berryman, 1994a, Portwood, and Williams, 1995a-c) mandarin oranges in the maturing stage were treated with EC formulations containing 480 g ai/l and in some cases also with WG 750 g ai/l, at 1.6-3.8 kg ai/ha. In the decline trials, samples were taken from one to 116 days after the last treatment. Whole mandarins were analysed at intervals, and pulp and peel from some samples at harvest. Six trials were conducted in Italy in 1995 (Portwood and Williams, 1996d-f). The results are shown in Table 25.

Table 25. Residues of chlorpyrifos and 3,5,6-trichloropyridinol in mandarin oranges from supervised trials in Spain and Italy.

| Location, (variety), year | Application | | | | PHI, days | Residues, mg/kg | | Analytical method/ recovery at mg/kg | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Chlorpyrifos | TCP | | |
| Spain GAP | EC<br>WP | (3.0)<br>(3.0) | 0.10<br>0.10 | 1<br>1 | 21<br>21 | | | | |
| Martinez (Clemenules) 1992 | EC | (2.88) | 0.096 | 1 | 1<br>27<br>56<br>89<br>116 | 1.4<br>0.4<br>0.27<br>0.17<br>0.14 (0.43 peel, <0.1 pulp) | 1.1<br>0.4<br>0.25<br>0.20<br>0.12 (0.36 peel, <0.25 pulp) | ERC 92.38.<br>CP: 92% @ 0.1<br>TCP: 96% @ 0.25 | GHE-P 3213 |

| Location, (variety), year | Application | | | | PHI, days | Residues, mg/kg | | Analytical method/ recovery at mg/kg | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Chlorpyrifos | TCP | | |
| Puzol, Valencia (Hernandina) 1993 | EC | (2.9) | 0.096 | 1 | 22 | 0.99<br>0.89<br>0.75<br>0.81 | - | ERC 92.38 CP: 91% @ 0.01 | GHE-P 3733 |
| Puzol, Valencia (Clemenules) 1993 | EC | (2.9) | 0.096 | 1 | 22 | 1.19 | - | | |
| Librilla, Murcia (Nova) 1993 | EC | (3.8) | 0.096 | 1 | 22 | 0.55 | - | | |
| Carcagentes, Valencia (Clementina fina) 1994 | WG | 1.6 | 0.094 | 1 | 0<br>3<br>7<br>14<br>21 | 2.4<br>1.9<br>1.2<br>1.0<br>0.47 | - | ERC 92.38 CP: 96±4% @ 0.01 | GHE-P-4516 |
| | WG | 0.80 | 0.047 | 1 | 0<br>3<br>7<br>14<br>21 | 1.5<br>1.2<br>0.84<br>0.41<br>0.33 | - | | |
| | EC | 1.6 | 0.096 | 1 | 0<br>3<br>7<br>14<br>21 | 2.1<br>2.4<br>1.2<br>0.82<br>0.70 | - | | |
| Piscasent, Valencia (Marisol) 1995 | WG | 2.8 | 0.065 | 1 | 0<br>5<br>10<br>15<br>20 | 1.4<br>0.33<br>0.31<br>0.26<br>0.15 | - | ERC 92.38<br><br>78% @ 0.10 | GHE-P-4808 |
| | EC | 2.8 | 0.065 | 1 | 0<br>5<br>10<br>15<br>20 | 1.6<br>0.52<br>0.38<br>0.37<br>0.33 | - | | |
| Benifayo, Valencia (Marisol) 1995 | WG | 1.7 | 0.065 | 1 | 20 | 0.16 | | ERC 92.38 70% @ 0.10 | GHE-P-4809 |
| | EC | 1.7 | 0.065 | 1 | 20 | 0.23 | | | |
| Sevilla (Navelina) 1995 | WG | 1.6 | 0.065 | 1 | 0<br>6<br>11<br>16<br>21 | 0.48<br>(0.18)<br>0.18<br>0.15<br>0.07 | - | ERC 92.38 104% @ 0.1 | GHE-P-4816 |
| | EC | 1.6 | 0.065 | 1 | 0<br>6<br>11<br>16<br>21 | 0.44<br>0.28<br>0.22<br>0.12<br>0.16 | | | |
| Italy GAP | EC | 0.84 | 0.056 | 2 | 60 | | | | |
| | WG | 1.1 | 0.11 | | | | | | |

| Location, (variety), year | Application | | | | PHI, days | Residues, mg/kg | | Analytical method/ recovery at mg/kg | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | No. | | Chlorpyrifos | TCP | | |
| Palagonia, Catania (Monreal Clementine) 1995 | WG | 1.9 | 0.065 | 3 | 0<br>5<br>11<br>16<br>21 | 2.7<br>0.99<br>0.92<br>0.84<br>0.60 | - | ERC<br>92.38<br><br>89% @ 0.10 | GHE-P-4970R |
| | EC | 1.9 | 0.065 | 3 | 0<br>5<br>11<br>16<br>21 | 2.8<br>1.5<br>1.4<br>1.1<br>0.86 | - | | |
| Catania (Navelina Nucellare) 1995 | WG | 1.9 | 0.065 | 1 | 0<br>6<br>11<br>16<br>21 | 0.94<br>0.24<br>0.31<br>0.35<br>0.25 | - | ERC<br>92.38<br><br>92±16% @ 0.01 | GHE-P-4969R |
| | EC | 1.9 | 0.065 | 1 | 0<br>6<br>11<br>16<br>21 | 1.0<br>0.49<br>0.50<br>0.47<br>0.43 | - | | |
| Catania, C. da Coccumella (Navelina) | WG | 1.9 | 0.065 | 3 | 21 | 0.11 | - | ERC<br>92.38<br>100% @0.10 | GHE-P-5426R |
| | EC | 1.9 | 0.065 | 3 | 21 | 0.24 | - | | |

Field trials on oranges were reported from the USA. Chlorpyrifos as a 4 lbs/gal EC formulation was applied once or twice in high volume (dilute) and low volume (concentrated sprays at 1.4 to 14 kg ai/ha in 16 trials on oranges, grapefruit, lemons and tangelos in California, Texas and Florida (Wetters, 1981). The high volume sprays contained 0.03-0.06 kg ai/hl at a volume of 2340 to 23400 l/ha and the low 0.20 to 1.5 kg ai/hl at a volume of 700 to 940 l/ha. Whole fruit samples were collected 14-19 and 21-26 days after the last treatment and analysed by gas chromatography with flame photometric detection (ACR 73.5.S1). Recoveries were 91 ± 4.2% at 0.01 mg/kg. Processed fractions were also analysed.

In a single separate trial in California (Robb, 1991a), Valencia oranges were treated by airblast equipment with three foliar sprays an EC formulation at 6.7, 2.2 and 6.7 kg ai/ha, diluted in 940 l/water/ha 325, 80 and 35 days before harvest, and two orchard floor applications with a hydraulic boom sprayer at 5.6 kg ai/ha, totalling 11.2 kg ai/ha, 240 l/water/ha, 88 and 28 days before harvest. Two plots of 4 replicates (each one tree with all quadrants sampled for a total of 30 mature fruit) were treated. Samples of oranges were collected 28 days after the last application and analysed by method ACR 84.4. The mean recovery was 89 ± 4.7% at 0.01 mg/kg.

In 1975 (Wetters, 1977) three trials were carried out in California. In two an EC formulation of chlorpyrifos was applied to duplicated plots as a single high or low volume foliar spray. In the third only a single high volume application was made. The high volume sprays contained 0.09 kg ai/hl applied at 13400 to 18700 l/ha, and the low 1.3 to 1.8 kg ai/hl in 940 l total volume/ha. The calculated application rate was 17 kg ai/ha maximum for both methods. Whole oranges were collected 14 days after treatment and analysed by method ACR 73.5.S1 for chlorpyrifos and ACR 71.19R for 3,5,6-TCP. Recoveries of chlorpyrifos were 90 ± 10% at 0.01 mg/kg and of 3,5,6-TCP 104% at 0.05 mg/kg.

In a trial at an exaggerated application rate in California in 1978 (Wetters, 1978) an EC formulation of 0.06 kg ai/hl was applied with an oscillating boom citrus sprayer at 8.0 or 11 kg ai/ha. The total volume applied was 13400 or 18600 l/ha. Samples were collected 14 and 21 days after treatment and analysed by method ACR 73.5.S1.

In 1984 in California side-by-side trials of low and high volume applications of chlorpyrifos were conducted in two locations (Wetters, 1985). Whole oranges were analysed by method ACR 84.4 for chlorpyrifos and 3,5,6-TCP. Recoveries of chlorpyrifos from whole fruit fortified at 0.01 to 2.0 mg/kg were 88 ± 5%, n = 11, and of 3,5,6-TCP at 0.05 to 2.0 mg/kg, 95 ±4%, n = 11.

In eight supervised trials in South Africa single high-volume treatments with an EC formulation were applied to Valencia and navel orange trees at 0.05 to 0.10 kg ai/hl (Hollick and Sandenskog, 1976). Each tree was treated with 30 or 60 l of spray solution and samples were taken at intervals of 7 days until about 90 days after application (normal harvest). The oranges were peeled, the weight of pulp and peel recorded and the pulp and peel analysed separately. By method ERC 76.1 Recoveries from pulp fortified at 0.01 mg/kg were 101 ± 9.2% and from peel at 0.10 mg/kg, 86%. The level of chlorpyrifos in the whole fruit was calculated from these analyses and the relative weights. No residue (<0.01 mg/kg) was found in any pulp sample.

The results of the orange trials are shown in Table 26.

Table 26. Residues of chlorpyrifos and 3,5,6-TCP in whole oranges from the application (foliar except as noted) of a chlorpyrifos EC formulation.

| Location, (variety), Year | Application | | | PHI, days | Residues, mg/kg | | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | CP | TCP | |
| South Africa GAP | | 0.048<br>0.029 | 2 as needed | 60<br>60 | | | EC 480 g/l |
| Nelspruit, SA (Navel) 1975 | 0.015 kg ai/tree | 0.05 | 1 | 7<br>33<br>62<br>91 | 0.21<br>0.10<br>0.05<br>0.03 | | GHE-P-413 |
| Transval, SA (navel) 1975 | 0.03 kg ai/tree | 0.05 | 1 | 7<br>31<br>59<br>92 | 0.45<br>0.209<br>0.12<br>0.05 | | |
| | 0.06 kg ai/tree | 0.10 | 1 | 7<br>31<br>59<br>92 | 0.59<br>0.29<br>0.17<br>0.12 | | |
| Transval, SA (Valencia) 1975 | 0.03 kg ai/tree | 0.05 | 1 | 7<br>31<br>59<br>92 | 0.56<br>0.32<br>0.14<br>0.06 | | |
| | 0.06 kg ai/tree | 0.10 | 1 | 7<br>31<br>59<br>92 | 0.72<br>0.45<br>0.27<br>0.17 | | |
| Citrusdal, SA (Valencia) 1975 | | 0.05 | 1 | 7<br>30<br>58<br>86 | 0.37<br>0.25<br>0.21<br>0.13 | | |
| | | 0.10 | 1 | 7<br>30<br>58<br>86 | 0.62<br>0.54<br>0.55<br>0.29 | | |

| Location, (variety), Year | Application | | | PHI, days | Residues, mg/kg | | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | CP | TCP | |
| E. Transval, SA (Valencia) 1975 | | 0.05 | 2 | 31<br>61<br>91<br>190 | 0.21<br>0.19<br>0.13<br>0.07 | | |
| US GAP | 6.7<br>1.1 | 0.7<br>0.5 | 2 foliar<br>10 to ground | 35<br>28 | | | |
| Riverside, California (Valencia) 1978 | 8.4 | 0.06<br>(1500 gal/A) | 1 | 14<br><br><br>21 | 0.19<br>0.20<br>0.15<br>0.12<br>0.12<br>0.09 | 0.13<br>0.13<br>0.14<br>0.09<br>0.11<br>0.09 | GH-C 1441 |
| | 8.4 | 0.9<br>(100 gal/A) | 1 | 14<br><br><br>21 | 0.65<br>0.57<br>0.45<br>0.34<br>0.46<br>0.35 | 0.43<br>0.35<br>0.59<br>0.29<br>0.32<br>0.33 | |
| | 14 | 0.06<br>(2500 gal/A) | 1 | 14<br><br><br>21 | 0.29<br>0.30<br>0.29<br>0.16<br>0.18<br>0.19 | 0.23<br>0.22<br>0.24<br>0.15<br>0.20<br>0.15 | |
| | 14 | 1.5<br>(100 gal/A) | 1 | 14<br><br><br>21 | 2.3<br>2.1<br>2.2<br>1.7<br>1.9<br>1.0 | 1.7<br>1.7<br>1.6<br>0.92<br>1.5<br>0.84 | |
| Davis, California (Navel) 1984 | 6.7 | 0.05 | 1 | 35 | 0.38<br>0.41<br>0.39 | 0.30<br>0.24<br>0.27 | GH-C 1724 |
| | 6.7 | 0.7 | 1 | 21 | 1.3<br>2.2 | 1.1<br>1.6 | |
| Solomon, California (Valencia) 1983 | 6.7 | 0.05 | 1 | 36 | 0.26<br>0.093<br>0.11 | 0.23<br>0.13<br>0.16 | |
| | 6.7 | 0.7 | 1 | 21 | 0.38<br>0.36 | 0.28<br>0.28 | |
| Lake Alfred, Florida (Valencia) 1979 | 4.4 | 0.03<br>(20-22 gal/tree; 1540 gal/A) | 2 | 14 | 0.30<br>0.34<br>0.29<br>0.31 | 0.23<br>0.28<br>0.21<br>0.21 | |
| | 8.6 | 0.06<br>(20-22 gal/tree; 1540 gal/A) | 2 | 14 | 0.58<br>0.65<br>0.77<br>0.84 | 0.45<br>0.58<br>0.43<br>0.55 | |
| Lake Alfred, Florida (Orlando tangelo) 1980 | 4.4 | 0.03<br>(20-22 gal/tree; 1540 gal/A) | 2 | 14 | 0.43<br>0.50<br>0.44 | 0.37<br>0.35<br>0.31 | |
| | 8.6 | 0.06<br>(20-22 gal/tree; 1540 gal/A) | 2 | 14 | 0.68<br>0.74<br>0.60 | 0.44<br>0.40<br>0.40 | |
| Lake Alfred, Florida (Valencia) | 4.4 | 0.03 | 2 | 15 | 0.27 | 0.20 | |

| Location, (variety), Year | Application | | | PHI, days | Residues, mg/kg | | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | CP | TCP | |
| 1979 | | | | | | | |
| | 8.6 | 0.06 | 2 | 15 | 0.51 | 0.44 | |
| Auburndale, Florida (Orlando tangelo) 1978 | 4.4 | 0.03 (800 gal/A) | 2 | 15 | 0.45 | 0.28 | |
| | 8.6 | 0.06 (800 gal/A) | 2 | 15 | 0.59 | 0.36 | |
| Weslaco, Texas (Valencia) 1978 | 1.4 | 0.06 (250 gal/A) | 1 | 13<br><br><br><br><br><br>21 | 0.06<br>0.05<br>0.07<br>0.04<br>0.05<br>0.05<br>0.1<br>0.05<br>0.06<br>0.05<br>0.06<br>0.05 | 0.05<br>0.04<br>0.06<br>0.03<br>0.04<br>0.04<br>0.05<br>0.04<br>0.04<br>0.04<br>0.03<br>0.04 | |
| | 1.4 | 0.20 (75 gal.A) | 1 | 13<br><br><br>21 | 0.34<br>0.47<br>0.45<br>0.38<br>0.49<br>0.34 | 0.25<br>0.24<br>0.32<br>0.20<br>0.31<br>0.21 | |
| Porterville, California (Valencia) 1989-1990 | 6.7 foliar<br>5.6 ground<br>2.2 foliar<br>6.7 foliar<br>5.6 ground | 0.7<br>2.4<br>0.7<br>0.7<br>2.4 | 5 | 35 foliar<br>28 ground | 0.28<br>0.33<br>0.48<br>0.54<br><br>0.66<br>0.48<br>0.30<br>0.38 | | GH-C 2554/ Each result is from a separate tree. Duplicate plots. |
| Riverside, California (Valencia) 1975 | 12 | 0.09 | 1 | 14 | 0.40<br>0.55<br>0.49<br>0.36 | 0.31<br>0.31<br>0.30<br>0.21 | GH-C 1041 |
| | 12 | 1.3 | 1 | 14 | 1.2<br>1.0<br>1.3<br>1.1 | 1.2<br>0.84<br>0.73<br>0.89 | |
| Riverside, California (Valencia) 1978 | 8.0 | 0.06 | 1 | 14 | 0.30<br>0.28<br>0.32<br>0.33<br><br>0.34<br>0.44<br>0.35<br>0.26 | 0.18<br>0.20<br>0.17<br>0.19<br><br>0.16<br>0.20<br>0.17<br>0.15 | GH-C 1141/ 4 replicate samples from each of duplicate plots |
| | 11 | 0.06 | 1 | 14 | 0.59<br>0.56<br>0.58<br>0.46<br><br>0.47 | 0.31<br>0.26<br>0.38<br>0.26<br><br>0.24 | |

| Location, (variety), Year | Application | | | PHI, days | Residues, mg/kg | | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | CP | TCP | |
| | | | | | 0.44<br>0.44<br>0.52 | 0.27<br>0.26<br>0.30 | |

A series of supervised trials and processing studies in the USA on lemons and grapefruits (Wetters, 1981) was reported. Whole grapefruit and lemons were analysed by method ACR 73.5.S1. In a residue decline study in Spain on lemons (Khoshab *et al.*, 1993b) the fruits were separated into peel and pulp and each was analysed by method ERC 92.38. At a fortification level of 0.10 mg/kg, the mean recoveries of chlorpyrifos were 93% (n = 2) from pulp and 94% (n = 2) from peel. The corresponding recoveries of TCP were 100% and 106%. The results are shown in Tables 27 and 28.

Table 27. Residues of chlorpyrifos and TCP in grapefruit from the foliar application of a chlorpyrifos EC formulation in the USA.

| Location, (Variety) Year | Application | | | PHI, days | Residues, mg/kg | | Reference |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | CP | TCP | |
| USA GAP | 6.7<br>1.1 | 0.7<br>0.5 | 2 foliar<br>10 ground | 35<br>28 | | | |
| Lake Alfred, Florida (Marsh) 1980 | 2.9 | 0.03 | 2 | 14 | 0.38<br>0.31<br>0.32 | 0.21<br>0.22<br>0.21 | GH-C 1441 |
| | 5.9 | 0.06 | 2 | 14 | 0.52<br>0.45<br>0.57 | 0.32<br>0.35<br>0.42 | |
| Lake Alfred, Florida (March) 1979 | 2.9 | 0.03 | 2 | 15 | 0.20 | 0.18 | |
| | 5.9 | 0.06 | 2 | 15 | 0.36 | 0.29 | |
| Weslaco, Texas (Ruby red) 1978 | 1.4 | 0.06 | 1 | 13<br><br><br>21 | 0.032<br>0.033<br>0.035<br>0.067<br>0.032<br>0.050 | 0.038<br>0.053<br>0.037<br>0.053<br>0.043<br>0.051 | |
| | 1.4 | 0.20 | 1 | 13<br><br><br>21 | 0.23<br>0.38<br>0.27<br>0.23<br>0.31<br>0.31 | 0.29<br>0.28<br>0.30<br>0.15<br>0.36<br>0.26 | |
| Weslaco, Texas (Webb Redblush) 1978 | 1.4 | 0.06 | 1 | 13<br><br><br>21 | 0.036<br>0.048<br>0.043<br>0.040<br>0.039<br>0.047 | 0.047<br>0.050<br>0.037<br>0.038<br>0.029<br>0.030 | |
| | 1.4 | 0.20 | 1 | 13<br><br><br>21 | 0.26<br>0.36<br>0.21<br>0.34<br>0.20<br>0.15 | 0.22<br>0.24<br>0.19<br>0.23<br>0.19<br>0.13 | |

Table 28. Residues of chlorpyrifos and TCP in lemons from the foliar application of a chlorpyrifos EC formulation in trials in Spain and the USA.

| Location, (Variety) Year | Application | | | PHI, days | Residue, mg/kg | | Reference |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | CP | TCP | |
| Spain GAP | 2.4 | 0.096 | 1 | 21 | | | |
| Alhama, Murcia (Fino) 1992 | 2.9 | 0.1 | 1 | 0<br>25<br>53<br>77<br>100 | 1.2<br>0.10<br>0.05<br>0.05<br>0.09 | 0.94<br>0.14<br>0.10<br>0.10<br>0.18 | GHE P 3228 |
| USA GAP | 6.7<br>1.1 | 0.7<br>0.5 | 2 foliar<br>10 ground | 35<br>28 | | | |
| Riverside, California (Lupe) 1978 | 6.7 | 0.06 | 1 | 19<br><br><br>26 | 0.18<br>0.094<br>0.13<br>0.12<br>0.12<br>0.10 | 0.15<br>0.10<br>0.15<br>0.14<br>0.15<br>0.11 | GH-C 1441 |
| | 6.7 | 0.7 | 1 | 19<br><br><br>26 | 0.035<br>0.059<br>0.062<br>0.025<br>0.055<br>0.039 | 0.049<br>0.065<br>0.077<br>0.052<br>0.085<br>0.065 | |
| | 8.4 | 0.06 | 1 | 14<br><br><br>21 | 0.27<br>0.30<br>0.31<br>0.20<br>0.21<br>0.22 | 0.23<br>0.21<br>0.22<br>0.18<br>0.21<br>0.20 | |
| | 8.4 | 0.7 | 1 | 14<br><br><br>21 | 0.16<br>0.16<br>0.18<br>0.14<br>0.16<br>0.14 | 0.13<br>0.14<br>0.14<br>0.13<br>0.15<br>0.14 | |
| Indiantown, Florida (Bearss) 1980 | 5.8 | 0.03 | 2 | 14 | 0.27<br>0.28<br>0.19 | 0.29<br>0.26<br>0.26 | |
| | 12 | 0.06 | 2 | 14 | 0.39<br>0.31<br>0.49 | 0.40<br>0.36<br>0.34 | |
| Indiantown, Florida (Bearss) 1978 | 5.8 | 0.03 | 2 | 14 | 0.38 | 0.29 | |
| | 12 | 0.06 | 2 | 14 | 0.20 | 0.19 | |

Pome Fruits

Apples. Supervised trials on apples in Chile, Italy, Japan, New Zealand, the UK, the USA, Brazil and Canada were reported. The government of Germany provided summary information on trials in 1983. The results are shown in Tables 29-33.

In Chile, after two applications of “Lorsban” 50WP at 1.5 and 1.6 kg ai/ha during fruit development, samples were collected at intervals up to 28 days later and analysed (Pompeu-Braga, 1982).

Table 29. Residues of chlorpyrifos in apples from supervised trials in Chile.

| Year | Application | | | | PHI, days[1,2] | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Chile | 50WP | | 0.06 | | 28 | | |
| | | | | | | | |
| Chile, 1982 | WP | 1.5+1.6 | 0.05 | 2 | 7 | 0.07, 0.12, 0.07, 0.13, 0.09 | GHB-P 008 |
| | | | | | 14 | 0.14, 0.12, 0.24, 0.14, 0.10 | |
| | | | | | 21 | 0.06, 0.08, 0.07, 0.16, 0.07 | |
| | | | | | 28 | 0.09, 0.05, 0.07, 0.06 | |

[1] LOQ 0.01 mg/kg

In four trials in Italy using EC formulation 480 g/l of chlorpyrifos apple trees were treated two or three times at 0.77 or 1.2 kg ai/ha, spray volume 15 l/ha, and samples collected at regular intervals from 0 to 87 days later (Teasdale, 1988a). The results are shown in Table 30.

Table 30. Residues of chlorpyrifos in apples in supervised trials in Italy.

| Year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Italy | EC | (0.84) | 0.053 | | 30 | | |
| 1987 | EC | 0.77 | | 2 | 0 | 1.30 | GHE-P 1872 |
| | | | | | 17 | 0.28 | |
| | | | | | 28 | 0.17 | |
| | | | | | 37 | 0.14 | |
| | | | | | 51 | 0.10 | |
| | | | | | 58 | 0.09 | |
| | | | | | 87 | 0.03 | |
| Italy, 1987 | EC | 1.2 | | 2 | 0 | 2.5 | GHE-P 1872 |
| | | | | | 17 | 0.60 | |
| | | | | | 28 | 0.48 | |
| | | | | | 37 | 0.22 | |
| | | | | | 51 | 0.14 | |
| | | | | | 58 | 0.12 | |
| | | | | | 87 | 0.07 | |
| Italy, 1987 | EC | 0.77 | | 3 | 0 | 0.84 | GHE-P 1872 |
| | | | | | 9 | 0.66 | |
| Italy, 1987 | EC | 0.77 | -- | 3 | 23 | 0.28 | GHE-P 1872 |
| | | | | | 30 | 0.19 | |
| | | | | | 59 | 0.12 | |
| Italy, 1987 | EC | 1.2 | -- | 3 | 0 | 1.8 | GHE-P 1872 |
| | | | | | 9 | 1.2 | |
| | | | | | 23 | 0.04 | |
| | | | | | 30 | 0.48 | |
| | | | | | 59 | 0.36 | |

[1] LOQ 0.01 mg/kg

Results of supervised trials in New Zealand, Germany and the UK are shown in Table 32. Trials in New Zealand were at a rate of 0.025 kg ai/hl, applied 3 or 7 times at 2-4 week intervals. Samples were collected at regular intervals from 0 to 28 days after treatment (Upritchard *et al.*, 1982).

In trials in Germany plots at seven different locations were treated 4 times with a foliar spray of a WP formulation (250 g/kg) at 0.75 kg ai/ha or 0.05 kg/hl. Fruits were collected at intervals of 0 to 28 days after the last application (growth stage 79) (Federal Biological Research Center for Agriculture and Forestry, Germany, 1999).

In the UK Cox's Orange pippin and Worcester Pearmain apples were treated with chlorpyrifos EC insecticide once at 0.48 kg ai/ha, then three times at 0.96 kg ai/ha. Samples were collected from four trees at random from the treated plots 16 days after the last application (Hollick and Gilmour, 1974).

Table 31. Residues of chlorpyrifos in apples from supervised trials in New Zealand, Germany and the UK.

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-New Zealand | 50WP | 1 minimum | 0.025-0.038 | | 14 | | |
| New Zealand | 50WP | | 0.025 | 7 | 1 | 0.59, 0.59 | GHF-P 230 |
| 1972 | | | | | 2 | 0.45, 0.40 | |
| | | | | | 7 | 0.32, 0.32 | |
| | | | | | 14 | 0.14, 0.19 | |
| | | | | | 21 | <0.05,<0.05 | |
| New Zealand | 50WP | | 0.025 | 3 | 1 | 0.22, 0.19 | GHF-P 230 |
| 1980 | | | | | 2 | 0.36, 0.22 | |
| | | | | | 4 | 0.37, 0.10 | |
| | | | | | 7 | 0.09, 0.08 | |
| | | | | | 14 | 0.09, 0.07, 0.16 | |
| | | | | | 23 | 0.19, 0.28 | |
| | | | | | 28 | 0.14, 0.14 | |
| GAP-Germany | | | | | | | |
| NONE (See UK) | | | | | | | |
| Frankfurt, Germany 1983 (Ontario) | 250WP | 0.75 | 0.05 | 4 | 0<br>7<br>14<br>21<br>28 | 0.72<br>0.06<br>0.08<br>0.05<br>0.05 | Federal Biological Research Center for Agriculture and Forestry, Germany, 1999. Only summary information. |
| Münster, Germany 1983 (Jonathan) | 250WP | 0.75 | 0.05 | 4 | 0<br>7<br>14<br>21<br>28 | 1.6<br>0.72<br>0.53<br>0.29<br>0.41 | |
| Dossenheim, Germany 1983 (Cox Orange) | 250WP | 0.75 | 0.05 | 4 | 0<br>7<br>14<br>21<br>28 | 1.2<br>0.30<br>0.17<br>0.08<br>0.08 | |
| Heidesheim, Germany 1983 (Golden Delicious) | 250WP | 0.75 | 0.05 | 4 | 0<br>7<br>21<br>28 | 0.38<br>0.11<br>0.06<br>0.06 | |
| Lanförden Germany 1983 | 250WP | 0.75 | 0.05 | 4 | 0<br>7 | 0.88<br>0.58 | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| (James Grieve) | | | | | 14<br>21<br>28 | 0.43<br>0.28<br>0.21 | |
| Jork, Germany 1983 (Gloster) | 250WP | 0.75 | 0.05 | 4 | 0<br>7<br>13 | 1.1<br>0.83<br>0.94 | |
| Saarwellingen, Germany 1983 (Golden Delicious) | 250WP | 0.75 | 0.05 | 4 | 0<br>7<br>14<br>21<br>28 | 0.41<br>0.32<br>0.11<br>0.13<br>0.12 | |
| GAP-UK | EC | 0.96 | (0.38) | 5 | 14 | | |
| UK, 1974 (Worcester Pearmain) | EC | 0.48(1x) + 0.96(3x) | | 4 | 16 | 0.17 | GHE-P 195 |
| UK, 1974 | | | | | | | |
| (Cox's Orange Pippin) | EC | 0.48(1x) + 0.96(3x) | | 4 | 16 | 0.18 | GHE-P 195 |

[1] LOQ 0.01 mg/kg for New Zealand and the UK, 0.02 mg/kg for Germany.

In California and Washington, USA, chlorpyrifos 50W insecticide was cover-sprayed four times post-petal fall at 1.68 kg ai/ha or a total of 6.7 kg ai/ha per season. In Michigan, New York and Pennsylvania a total of 9.6 kg ai/ha was applied in eight applications during the season, from petal fall to seventh cover. Samples were collected 14, 21 and 28 days after the last application (Wetters and Ervick, 1990a).

In trials in New York, USA, in 1980 seven foliar applications of chlorpyrifos 50W insecticide as a dilute spray of 0.15 kg ai/hl or as a tenfold concentrate were made during the season at 2.24 kg ai/ha, and samples collected 0, 7, 14, 21 and 28 days after the last treatment (Miller, 1980a).

In trials in seven US States, 1973-1976, the trees were foliar-sprayed with chlorpyrifos 25W or 50W formulations at the standard dilution of 0.06 kg ai/hl or at 4-20 times this concentration, and samples were collected 0 to 35 days after treatment (Wetters and Ervick, 1978). The residues found up to 28 days after treatment are shown in Table 32.

In apple trials in 1987 in Ontario and Quebec, Canada, Lorsban 50W was applied five times at 1.7 kg ai/ha during the season at 20-day intervals, beginning approximately 108 days before harvest, with the last application 27 days before harvest, and samples were collected at harvest (Dixon-White, 1991). The results are shown in Table 32.

Table 32. Residues of chlorpyrifos in apples from supervised trials in the USA and Canada.

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-USA | 4lb/gal EC | 3.4 | 0.06 | | 70-200 [2] | | Limited to pre-bloom, dormant use. |
| CA, 1988 | 50W | 1.68 | 0.047 | 4 | 14 | 0.34, 0.21, 0.24, 0.35 | GH-C 2397 |
| | | | | | 21 | 0.18, 0.20, 0.46, 0.18 | |
| | | | | | 28 | 0.17, 0.30, 0.22, 0.34 | |
| CA, 1988 | 50W | 1.68 | 0.47 | 4 | 14 | 0.52, 0.66, 0.17, 0.53 | GH-C 2397 |
| | | | | | 21 | 0.45, 0.37, 0.19, 0.23 | |

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 28 | 0.49, 0.50, 0.21, 0.19 | |
| WA, 1988 | 50W | 1.68 | 0.047 | 4 | 14 | 0.21, 0.17, 0.13, 0.14 | GH-C 2397 |
| | | | | | 21 | 0.13, 0.18, 0.11, 0.13 | |
| | | | | | 28 | 0.16, 0.13, 0.08, 0.20 | |
| WA, 1988 | 50W | 1.68 | 0.47 | 4 | 14 | 0.18, 0.21, 0.33, 0.19 | GH-C 2397 |
| | | | | | 21 | 0.13, 0.14, 0.17, 0.13 | |
| | | | | | 28 | 0.27, 0.27, 0.18, 0.26 | |
| MI, 1988 | 50W | 1.6(x4)+0.8(x4) | 0.022-0.047 | 8 | 14 | 0.13, 0.05, 0.11, 0.07 | GH-C 2397 |
| MI, 1988 | 50W | 1.6(x4)+0.8(x4) | 0.022-0.047 | 8 | 21 | 0.01, 0.01, 0.02 | GH-C 2397 |
| | | | | | 28 | 0.01 | |
| MI, 1988 | 50W | 1.6(x4)+0.8(x4) | 0.22-0.47 | 8 | 14 | 0.15, 0.17, 0.17, 0.12 | GH-C 2397 |
| | | | | | 21 | 0.04, 0.04, 0.11, 0.15, | |
| | | | | | 28 | 0.05, 0.03, 0.04, 0.19 | |
| MI, 1988 | 50W | 1.6(x4)+0.8(x4) | 0.022-0.047 | 8 | 14 | 0.08, 0.06, 0.20, 0.08 | GH-C 2397 |
| | | | | | 21 | 0.07, 0.05, 0.1, 0.11 | |
| | | | | | 28 | 0.04, 0.07, 0.05, 0.13 | |
| MI, 1988 | 50W | 1.6(x4)+0.8(x4) | 0.22-0.47 | 8 | 14 | 0.18, 0.13, 0.26, 0.19 | GH-C 2397 |
| | | | | | 21 | 0.10, 0.12, 0.09, 0.09 | |
| | | | | | 28 | 0.04, 0.05, 0.19, 0.13 | |
| NY, 1988 | 50W | 1.6(x4)+0.8(x4) | 0.022-0.047 | 8 | 14 | 0.30, 0.74, 0.36, 0.30 | GH-C 2397 |
| | | | | | 21 | 0.29, 0.37, 0.33, 0.33 | |
| | | | | | 28 | 0.57, 0.25, 0.34, 0.38 | |
| NY, 1999 | 50W | 1.6(x4)+0.8(x4) | 0.22-0.47 | 8 | 14 | 0.23, 0.10, 0.20, 0.09 | GH-C 2397 |
| | | | | | 21 | 0.14, 0.17, 0.14, 0.06 | |
| | | | | | 28 | 0.15, 0.10, 0.07, 0.1 | |
| PA, 1988 | 50W | 1.6(x4)+0.8(x4) | 0.022-0.047 | 8 | 14 | 0.23, 0.20, 0.19, 0.17 | GH-C 2397 |
| | | | | | 21 | 0.14, 0.12, 0.11, 0.28 | |
| | | | | | 28 | 0.17, 0.09, 0.28, 0.26 | |
| PA, 1988 | 50W | 1.6(x4)+0.8(x4) | 0.22-0.47 | 8 | 14 | 0.09, 0.05, 0.10, 0.06 | GH-C 2397 |
| | | | | | 21 | 0.06, 0.10, 0.08, 0.12 | |
| | | | | | 28 | 0.04, 0.05, 0.07, 0.06 | |
| NY, 1980 | 50W | 2.24 | 0.15 | 7 | 0 | 1.3, 1.4, 0.95, 1.62 | GH-C 1485 |
| | | | | | 7 | 0.58, 0.38, 0.72, 0.68 | |
| | | | | | 14 | 0.48, 0.42, 0.48, 0.37 | |
| | | | | | 21 | 0.28, 0.21, 0.43, 0.44 | |
| | | | | | 28 | 0.30, 0.29, 0.39, 0.35 | |
| NY, 1980 | 50W | 2.24 | 1.5 | 7 | 0 | 1.23, 1.32, 1.13, 1.5 | GH-C 1485 |
| | | | | | 7 | 0.35, 0.48, 0.52, 0.57 | |
| | | | | | 14 | 0.27, 0.23, 0.25, 0.29 | |
| | | | | | 21 | 0.21, 0.31, 0.33, 0.27 | |
| | | | | | 28 | 0.22, 0.20, 0.36, 0.33 | |
| CA, 1976 | | | | | | | |
| | 50W | 4.48 | 0.06 | 4 | 28 | 0.39, 0.45, 0.27, 0.30 | GH-C 1107 |
| | | 4.48 | 1.2 | 4 | 28 | 0.49, 0.40, 0.34, 0.33 | |
| WA, 1976 | 50W | 3.36 | 0.06 | 5 | 28 | 0.52, 0.48, 0.36, 0.19 | GH-C 1107 |
| MI, 1976 | 50W | 2.24 | 0.06 | 7 | 28 | 0.25, 0.52, 0.24, 0.43 | GH-C 1107 |

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| MI, 1973 | 25 WP | 0.7 | 0.06 | 9 | 28 | 0.11, 0.17, 0.11, 0.19 | GH-C 1107 |
| NC, 1976 | 50W | 2.24 | 0.06 | 9 | 28 | 1.1 | GH-C 1107 |
| WI, 1976 | 50W | 2.24 | 0.06 | 10 | 28 | 0.14, 0.13, 0.16, 0.07 | GH-C 1107 |
| GAP- Canada NONE | | | | | | | |
| Canada, 1987 | | | | | | | |
| (Red Delicious) | 50 WP | 1.7 | 0.043 | 5 | 27 | 0.21 | GH-C 2449 |
| Canada, 1987 | 50 WP | 1.7 | | 5 | 27 | 0.30 | GH-C 2449 |
| (Macintosh) | | | | | | | |
| Canada, 1987 | 50 WP | 1.7 | 0.06 | 5 | 27 | 0.36 | GH-C 2449 |
| (Macspur) | | | | | | | |
| Canada, 1987 (Red Delicious) | 50 WP | 1.7 | 0.06 | 5 | 27 | 0.31 | GH-C 2449 |

[1] LOQ 0.01 mg/kg
[2] Variety-dependent.

In a trial in Brazil chlorpyrifos was applied 3 times at different rates at the developmental stage of the fruit (Pinheiro *et al*., 1999), and samples taken 0, 3, 7, 10 and 14 days after the last application. The results are shown in Table 33.

Table 33. Residues of chlorpyrifos in apples from supervised trials in Brazil.

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Brazil NONE GAP-Argentina | WP 500 g/kg | | 0.06 | | 30 | | |
| Brazil, 1999 | 75 WG | 0.48 | 0.048 | 3 | 3 | 0.12, 0.18, 0.11 | GHB-P 415 |
| | | | | | 7 | 0.13, 0.12, 0.10 | |
| | | | | | 10 | 0.14, 0.15, 0.08 | |
| Brazil, 1999 | 75 WG | 0.75 | 0.075 | 3 | 3 | 0.15, 0.16, 0.19 | GHB-P 415 |
| | | | | | 7 | 0.14, 0.10, 0.17 | |
| | | | | | 10 | 0.11, 0.13, 0.13 | |
| | | | | | 14 | 0.08, 0.10, 0.11 | |
| Brazil, 1999 | 75 WG | 0.96 | 0.096 | 3 | 3 | 0.78, 0.85, 0.33 | GHB-P 415 |
| | | | | | 7 | 0.17, 0.16, 0.20 | |
| | | | | | 10 | 0.14, 0.20, 0.33 | |
| | | | | | 14 | 0.16, 0.16, 0.23 | |
| Brazil, 1999 | 75 WG | 1.5 | 0.15 | 3 | 3 | 0.93, 0.54, 0.66 | GHB-P 415 |
| | | | | | 7 | 0.66, 0.68, 0.33 | |
| | | | | | 10 | 0.20, 0.43, 0.20 | |
| | | | | | 14 | 0.28, 0.35, 0.35 | |

[1] LOQ 0.01 mg/kg

Pears. In eight trials in California, Michigan, New York, Oregon, Pennsylvania and Washington, (the largest US areas of pear production) Lorsban 50W was applied to the trees at 1.6 kg ai/ha as a concentrate or dilute spray. The first applications were made at blossom pink stage and at petal fall, and six more applications at approximately 14-day intervals and samples taken 14 and 28 days after the last application (Miller and McKellar, 1986a).

In trials in Canada five applications were made to the trees during the season at 20-day intervals beginning approximately 108 days before harvest, at 1.7 kg ai/ha (Dixon-White, 1991).

In UK trials on Conference and Comice pears, the trees were treated with Dursban 4E at 0.48 kg ai/ha, followed by three applications at 0.96 kg ai/ha. Samples were collected from four trees at random from the treated plots, 27 days after the last application (Hollick and Gilmour, 1974).

The results are shown in Table 34.

Table 34. Residues of chlorpyrifos in pears from supervised trials in Canada, the USA and the UK.

| Location, year (Variety) | Application | | | | PHI, days | Residues, mg/kg[1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP- USA NONE | | | | | | | |
| CA, 1983 | 50W | 1.6 | 0.06 | 8 | 14 | 0.23 | GH-C 1789 |
| (Bartlett) | | | | | 28 | 0.07 | |
| | | 1.6 | 0.51 | 8 | 14 | 0.59 | |
| | | | | | 28 | 0.59 | |
| OR, 1983 | 50W | 1.6 | 0.05 | 8 | 14 | 0.31 | GH-C 1789 |
| (Bartlett) | | | | | 28 | 0.26 [2] | |
| CA, 1983 | 50W | 1.6 | 0.43 | 8 | 14 | 0.19 | GH-C 1789 |
| (Bartlett) | | | | | 28 | 0.034 | |
| | | 1.6 | 0.08 | 8 | 14 | 0.14 | |
| | | | | | 28 | 0.04 | |
| MI, 1983 | 50W | 1.6 | 0.34 | 8 | 14 | 0.91 | GH-C 1789 |
| (Bartlett) | | | | | 28 | 0.43 | |
| | | 1.6 | 0.08 | 8 | 14 | 0.72 [3] | |
| | | | | | 28 | 0.16 | |
| MI, 1983 | 50W | 1.6 | 0.34 | 8 | 14 | 1.8 | GH-C 1789 |
| (Bartlett) | | | | | 28 | 1.4 | |
| | | 1.6 | 0.08 | 8 | 14 | 0.73 | |
| | | | | | 28 | 0.37 | |
| NY, 1984 | 50W | 1.6 | 0.34 | 8 | 14 | 0.53 | GH-C 1789 |
| (Bartlett) | | | | | 28 | 0.17 | |
| | | 1.6 | 0.06 | 8 | 14 | 0.54 | |
| | | | | | 28 | 0.25 | |
| PA, 1984 | 50W | 1.6 | 0.34 | 8 | 14 | 0.35 | GH-C 1789 |
| (Bartlett) | | | | | 27 | 0.06 | |
| | | 1.6 | 0.08 | 8 | 14 | 0.24 | |
| | | | | | 27 | 0.06 | |
| WA, 1983 | 50W | 1.6 | 0.34 | 8 | 14 | 0.63 | GH-C 1789 |
| (Bartlett) | | | | | 28 | 0.22 | |
| | | 1.6 | 0.03 | 8 | 14 | 0.18 | |
| | | | | | 28 | 0.13 | |
| GAP- Canada NONE | | | | | | | |

| Location, year (Variety) | Application | | | | PHI, days | Residues, mg/kg[1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| Canada, 1987 | 50W | 1.7 | 0.04 | 5 | 29 | 0.16 [3] | GH-C 2449 |
| (Bosc) | | | | | | | |
| Canada, 1987 | 50W | 1.7 | 0.04 | 5 | 28 | 0.07[2] | GH-C 2449 |
| (Moonglow) | | | | | | | |
| UK GAP | EC 480g/l | 0.96 | (0.38) | 5 | 14 | | |
| UK, 1974 (Comice) | EC | 0.48(1x) + 0.96(3x) | | 5 | 27 | 0.12 | GHE-P 195 |
| UK, 1974 (Conference) | EC | 0.48(1x) + 0.96(3x) | | 5 | 27 | 0.10 | GHE-P 195 |

[1] LOQ 0.01 mg/kg
[2] Average of duplicate determinations.
[3] Average of triplicate determinations.

## Stone Fruits

Peaches. In three trials in Chile in support of a USA import tolerance a WP formulation was applied to fruit-bearing peach trees and fruits collected 45 days after the treatment. The fruits were washed, brushed and waxed and then frozen pending analysis (Catta-Preta and Rampazzo, 1994).

In another trial in Padre Hurtado, central Chile, a WP formulation was applied to fruit-bearing trees at 120 g/hl and samples taken 0, 14, 25 and 45 days after treatment, stored frozen and analysed (Catta-Preta and Rampazzo, 1995).

In ten further trials in Chile during the 1998-9 season chlorpyrifos was applied once at 1.2 kg ai/ha to five different varieties of tree. Five trials were with the 75 G (water-dispersible granule) formulation containing 750 g ai/kg, and five with the 50WP. Samples were collected at intervals up to 52 days (Do Amaral *et al.*, 1999a,b). The results are shown in Table 35.

Table 35. Residues of chlorpyrifos in peaches in Chile from the foliar application of chlorpyrifos.

| Location, Year, (Variety) | Application | | | PHI, days | Residue, mg/kg | Reference/ comment |
|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Chlorpyrifos | |
| Chile GAP | | 0.06 | 3 | 45 | | WP |
| Buin, Chile (Elegant Lady)<br>1993 | (1.4) | 0.06 | 1 | 45 | 0.023 | GHB-P 212.<br>WP |
| Padre Hurtado, (O'Henry)<br>1993 | (1.2) | 0.06 | 1 | 45 | 0.045 | GHB-P 212.<br>WP |
| Lampa, Chile (O'Henry)<br>1993 | (1.3) | 0.06 | 1 | 45 | 0.017 | GHB-P 212.<br>WP |
| Padre Hurtado, Chile<br>(O'Henry)<br>1993-1994 | (1.2) | 0.06 | 1 | 0<br>14<br>25<br>45 | 1.3<br>0.43<br>0.28<br>0.04 | GHB-P 254<br>WP |
| Chile, 1998<br>(Royal Glory) | (1.2) | 0.08 | 1 | 0<br>17<br>34<br>45<br>52 | 5.4, 3.5<br>2.8, 1.2<br>0.42, 0.17<br>0.25, 0.05<br>0.13 | GHB-P 422<br>WG |

| Location, Year, (Variety) | Application | | | PHI, days | Residue, mg/kg | Reference/ comment |
|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Chlorpyrifos | |
| Chile, 1998<br>(Iris Rosso) | (1.2) | 0.08 | 1 | 0<br>17<br>34<br>45 | 10<br>1.6<br>0.34<br>0.09 | GHB-P 422 WG |
| Chile, 1998<br>(Rich May) | (1.2) | 0.08 | 1 | 0<br>17<br>34<br>45 | 2.8<br>0.65<br>0.10<br>0.05 | GHB-P 422 WG |
| Chile,<br>1998<br>(Flavourcrest) | (1.2) | 0.8 | 1 | 0<br>17<br>34<br>45<br>52 | 2.4<br>0.67<br>0.20<br>0.08<br>0.03, 0.08 | GHB-P 422 WG |
| Chile, 1998<br>(Red Top) | (1.2) | 0.8 | 1 | 0<br>17<br>34<br>45<br>52 | 3.0<br>0.58<br>0.20<br>0.04<br>0.02 | GHB-P 422 WG |
| Chile, 1998<br>(Royal Glory) | (1.2) | 0.12 | 1 | 0<br>17<br>34<br>45<br>52 | 3.6, 2.1<br>3.1, 0.56<br>0.20, 0.15<br>0.13, 0.03<br>0.02 | GHB-P 423 WP |
| Chile, 1998<br>(Iris Rosso) | (1.2) | 0.12 | 1 | 0<br>17<br>34<br>45 | 0.47<br>0.85<br>0.19<br>0.07 | GHB-P 423 WP |
| Chile, 1998<br>(Rich May) | (1.2) | 0.12 | 1 | 0<br>17<br>34<br>45 | 2.0<br>0.53<br>0.13<br>0.03 | GHB-P 423 WP |
| Chile, 1998<br>(Flavourcrest) | (1.2) | 0.12 | 1 | 0<br>17<br>34<br>45<br>52 | 4.2<br>0.79<br>0.17<br>0.04<br>0.01, 0.03 | GHB-P 423 WP |
| Chile, 1998<br>(Red Top) | (1.2) | 0.12 | 1 | 0<br>17<br>34<br>45<br>52 | 3.7<br>0.63<br>0.09<br>0.04<br><0.01 | GHB-P 423 WP |

The results of supervised trials on peaches in Greece, Italy and Spain are shown in Table 36. In some trials in Greece in 1992, a single application of chlorpyrifos EC (480 g ai/l) diluted to 0.096 kg ai/hl was applied to peach trees when the fruits were 3 cm in diameter at 1.44 kg ai/ha and samples were taken at intervals from 0 to 39 days after treatment (Khoshab, 1993d).

In another trial in Greece, a single application of chlorpyrifos WG formulation (750 g ai/l) diluted to 0.05 kg ai/hl was applied at 0.63 kg ai/ha to peach trees with fruit 4-6 cm in diameter and samples were taken at intervals from 0 to 22 days after application (Portwood and Williams, 1996h).

In a trial in Spain a single application of chlorpyrifos formulation containing 480 g ai/l diluted to 0.096 kg ai/hl was applied at 1.44 kg ai/ha to peach trees with fruit about 3 cm in diameter. Samples were taken at intervals from the day of application to 28 days later (Khoshab, 1993c).

In two trials in Italy in 1992, a single application of chlorpyrifos EC (480 g ai/l) at 0.08 kg ai/hl was applied at 1.2 kg ai/ha to peach trees with fruit 3-4 cm in diameter. Peaches were harvested at intervals from 0 to 40-42 days after treatment (Khoshab, 1993e). In similar trials in 1994, single applications of the WG or EC formulation, diluted to 0.056 kg ai/hl, at 0.85 kg ai/ha were made with fruit 4-5 cm in diameter. A further application of the WG formulation at 0.028 kg ai/hl was made at 0.43 kg ai/ha. Fruits were sampled from 0 to 39 days after treatment (Portwood and Williams, 1995b).

Table 36. Residues of chlorpyrifos in whole peaches from supervised trials in Greece, Spain and Italy.

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Greece | 480 EC | (1.9) | 0.08 | 2 | 20 | | |
| | 250 WP | 2.1 | 0.08 | 2 | 20 | | |
| Greece, 1992 | EC | 1.44 | 0.096 | 1 | 0 | 2.5 | GHE-P 3139. Plot = 4 trees. |
| | | | | | 9 | 1.0 | |
| | | | | | 19 | 0.33 | |
| | | | | | 29 | 0.17 | |
| | | | | | 39 | 0.10 | |
| Greece, 1995 | WG | 0.63 | 0.05 | 1 | 0 | 0.70 | GHE-P 4806 |
| | | | | | 5 | 0.32 | |
| | | | | | 10 | 0.19 | |
| | | | | | 15 | 0.12 | |
| | | | | | 22 | 0.07 | |
| GAP Spain NONE. GAP Greece. | 480 EC | ( 1.9) | 0.08 | 2 | 20 | | |
| Spain, 1992 | EC | 1.44 | 0.096 | 1 | 0 | 2.5 | GHE-P 3138. Plot = 4 trees. |
| | | | | | 7 | 0.78 | |
| | | | | | 14 | 0.55 | |
| | | | | | 20 | 0.04 | |
| | | | | | 28 | 0.04 | |
| GAP-Italy | 480 EC | 0.80 | 0.053 | 2 | 30 | | |
| | 225 EC | 0.80 | 0.056 | 2 | 30 | | |
| Ferrara, Italy, 1992 | 480 EC | 1.2 | 0.08 | 1 | 0 | 2.2 | GHE-P 3142. Plot = 6 trees. |
| | | | | | 9 | 1.0 | |
| | | | | | 20 | 0.35 | |
| | | | | | 30 | 0.07 | |
| | | | | | 40 | 0.05 | |
| Verona, Italy, 1992 | 480 EC | 1.2 | 0.08 | 1 | 0 | 1.2 | GHE-P 3142. Plot = 6 trees |
| | | | | | 10 | 0.72 | |
| | | | | | 20 | 0.43 | |
| | | | | | 30 | 0.19 | |
| | | | | | 42 | 0.04 | |
| Italy, 1994 | 480 EC | 0.85 | 0.056 | 1 | 0 | 0.87 | GHE-P 4229 |
| | | | | | 10 | 0.42 | |
| | | | | | 20 | 0.17 | |
| | | | | | 31 | 0.04 | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 39 | 0.04 | |
| Italy, 1994 | WG | 0.85 | 0.056 | 1 | 0 | 0.75 | GHE-P 4229 |
| | | | | | 10 | 0.24 | |
| | | | | | 20 | 0.09 | |
| | | | | | 31 | 0.05 | |
| | | | | | 39 | 0.03 | |
| Italy, 1994 | WG | 0.43 | 0.028 | 1 | 0 | 0.35 | GHE-P 4229 |
| | | | | | 10 | 0.13 | |
| | | | | | 20 | 0.05 | |
| | | | | | 31 | 0.03 | |
| | | | | | 39 | <0.01 | |

[1] Average of duplicate determinations

The US label only allows for trunk spray or dormant season (winter) applications to peach trees. In trials in Georgia, Arkansas and Mississippi applications of chlorpyrifos EC diluted to 0.36-0.72 kg ai/hl were sprayed on peach tree trunks, wetting the trunk thoroughly. Peaches collected 0 to 30 days after treatment were analysed (McKellar, 1971).

Table 37. Residues of chlorpyrifos in peaches from supervised trials in the USA (trunk spray).

| Location, year | Application | | | PHI, days | Residues, mg/kg [1,2] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/hl | No. | | | |
| GAP-USA | 480 EC | 0.36 | 1 | 14 | | |
| Georgia, 1969 | EC | 0.36 | 1 | 0 | <0.01, <0.01, <0.01 | GH-C 479 |
| Trunk | | | | 7 | <0.01, <0.01, <0.01, <0.01 | |
| | | | | 14 | <0.01, <0.01, <0.01, ≤0.01 | |
| | | | | 28 | <0.01,<0.01, <0.01, <0.01 | |
| Arkansas, 1969 | EC | 0.36 | 1 | 0 | 0.02, 0.01, 0.04, 0.03, 0.02 | GH-C 479 |
| Trunk | | | | | 0.01, <0.01, 0.011, 0.01 | |
| | | | | 7 | <0.01, <0.01, <0.01, <0.01 | |
| | | | | 14 | <0.01, <0.01, <0.01, ≤0.01 | |
| | | | | 28 | <0.01,<0.01, <0.01 | |
| Arkansas, 1969 | EC | 0.36 | 1 | 0 | 0.01, 0.03, 0.01, 0.01 | GH-C 479 |
| Trunk | | | | 7 | 0.01,0.01, <0.01, <0.01 | |
| | | | | 14 | <0.01, <0.01, <0.01, <0.01 | |
| | | | | 30 | 0.01, 0.01,<0.01, 0.01, 0.01 | |
| Arkansas, 1969 | EC | 0.72 | 1 | 0 | 0.06, 0.05, 0.02, 0.01, 0.01 | GH-C 479 |
| Trunk | | | | 7 | <0.01, <0.01, <0.01, <0.01 | |
| | | | | 14 | <0.01, <0.01, 0.01, <0.01 | |
| | | | | 30 | 0.01, 0.011, 0.021,0.01 | |
| Mississippi, 1969 | EC | 0.36 | 1 | 0 | 0.01, 0.02, 0.12, 0.03 | GH-C 479 |
| Trunk | | | | 7 | <0.01, 0.01, <0.01, <0.01 | |
| | | | | 14 | <0.01, <0.01, <0.01, 0.01 | |
| | | | | 28 | <0.01, <0.01, <0.01, <0.01 | |
| Mississippi, 1969 | EC | 0.72 | 1 | 0 | 0.05, 0.09, 0.04, 0.04 | |

| Location, year | Application | | | PHI, days | Residues, | |
|---|---|---|---|---|---|---|
| | Form. | kg ai/hl | No. | | mg/kg [1,2] | Reference |
| Trunk | | | | 7 | 0.01, 0.01, 0.01, 0.01 | GH-C 479 |
| | | | | 14 | <0.01, <0.01, <0.01, <0.01 | GH-C 479 |
| | | | | 28 | <0.01,<0.01, <0.01, <0.01 | |

[1] LOQ 0.01 mg/kg.
[2] Analyses were also conducted for the oxygen analogue and TCP. The former was not found in any sample. TCP was found in two samples, <0.05 mg/kg.

Plums. Results of supervised trials conducted on plums/prunes in Chile, Japan and Germany are shown in Table 38.

Three field trials were conducted in Colina, Padre Hurtado and Buin, representative fruit growing regions in central Chile, to support a US import tolerance. Lorsban 50W was applied to fruiting plum trees at 0.06 kg ai/hl. 45 days after treatment control and treated samples were picked, washed, brushed and waxed by simulated commercial practices (Catta-Preta, 1994).

In trials in Japan the 25 WP formulation at 0.025 kg ai/hl was applied twice at a 7-day interval at 1.0 kg ai/ha, and samples were taken 14, 21 and 30 days after application (Ishikura, 1993).

Trials in Germany were approximately according to UK GAP, because GAP was not then established in Germany. The 25 WP formulation was applied to plums 4 times at three different rates, and the plums were harvested at intervals up to 21 days after the last treatment (Osborne, 1989a).

Table 38. Residues of chlorpyrifos in plums from supervised trials in Chile, Japan and Germany.

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP Chile | 500 WP | | 0.06 | | 45 | | |
| Chile, 1993 | WP | (0.77) | 0.06 | 1 | 45 | 0.004, 0.005 [2] | GHB-P 213 |
| Chile, 1993 | WP | (1.1) | 0.06 | 1 | 45 | <0.002, <0.002 [2] | GHB-P 213 |
| Chile, 1993 | WP | (1.0) | 0.06 | 1 | 45 | <0.002, <0.002 [2] | GHB-P 213 |
| GAP-Japan | 250 WP | | 0.025 | 2 | 14 | | |
| Japan | WP | 1 | 0.025 | 2 | 14 | 0.05 [3] | GHF-P 1328 |
| | | | | | 21 | 0.02 [3] | |
| | | | | | 30 | 0.01 [3] | |
| Japan | WP | 1 | 0.025 | 2 | 14 | 0.03 [3] | GHF-P 1328 |
| | | | | | 21 | 0.04 [3] | |
| | | | | | 30 | 0.01 [3] | |
| GAP Germany NONE | | | | | | | |
| GAP- UK | 480 EC | 0.96 | 0.38 | 5 | 14 | | |
| Germany, 1987 | 25 WP | 0.75 | 0.1 | 4 | 0 | 0.29 | GHE-P 1911 |
| | | | | | 7 | 0.12 | |
| | | | | | 10 | 0.14 | |
| | | | | | 14 | 0.10 | |
| Germany, 1987 | WP | 0.93 | 0.05 | 4 | 0 | 0.22 | GHE-P 1911 |
| | | | | | 7 | 0.14 | |
| | | | | | 10 | 0.13 | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 14 | 0.08 | |
| | | | | | 21 | 0.08 | |
| Germany, 1987 | WP | 0.83 | 0.15 | 4 | 0 | 0.52 | GHE-P 1911 |
| | | | | | 7 | 0.28 | |
| | | | | | 10 | 0.26 | |
| | | | | | 14 | 0.20 | |
| | | | | | 10 | 0.13 | |
| | | | | | 14 | 0.08 | |
| | | | | | 21 | 0.08 | |
| Germany, 1987 | WP | 0.83 | 0.15 | 4 | 0 | 0.52 | |
| | | | | | 7 | 0.28 | |
| | | | | | 10 | 0.26 | |
| | | | | | 14 | 0.20 | |
| | | | | | 21 | 0.14 | |
| Germany, 1987 | 25 WP | 0.75 | 0.05 | 4 | 0 | 0.32 | GHE-P 1911 |
| | | | | | 7 | 0.20 | |
| | | | | | 10 | 0.10 | |
| | | | | | 14 | 0.04 | |

[1] Average of duplicate determinations. LOQ 0.02 mg/kg. Recoveries averaged 83%.
[2] LOQ 0.002 mg/kg. Recoveries averaged 91 (± 4)%.
[3] LOQ 0.005 mg/kg (not validated). Recoveries averaged 96%.

## Berries and other small fruits

Berries. In caneberry trials in the USA (Wetters and Markle, 1987) a foliar spray of a WP formulation was applied during the growing season, with the last application 14 days before harvest. In seven field trials on blueberries in six States, representing the main blueberry growing regions of the USA, a WP formulation was sprayed on the leaves at 3 x 1.7 kg ai/ha at about 14 day intervals with a 14-day PHI (Wetters, 1986). The conditions and results are shown in Table 39.

Table 39. Residues of chlorpyrifos and TCP in blueberries and caneberries from application of a WP formulation in the USA.

| Country, year | Application | | PHI, days | Chlorpyrifos, mg/kg | Reference |
|---|---|---|---|---|---|
| | kg ai/ha | No. | | | |
| No GAP in USA or NAFTA[1] country | | | | | |
| Blueberries | | | | | |
| Michigan 1986 | 1.7 | 3 | 15 | 0.17 | GH-C 1832 |
| | | | 29 | 0.01 | |
| Michigan, 1986 | 1.7 | 3 | 14 | 0.09 | GH-C 1832 |
| | | | 28 | 0.04, 0.04, 0.04, 0.05 | |
| New Jersey, 1986 | 1.7 | 3 | 1 | 1.7 | GH-C 1832 |
| | | | 3 | 1.7 | |
| | | | 7 | 0.26 | |
| | | | 14 | 0.99 | |
| | | | 21 | 0.03 | |

| Country, year | Application | | PHI, days | Chlorpyrifos, mg/kg | Reference |
|---|---|---|---|---|---|
| | kg ai/ha | No. | | | |
| New York, 1986 | 1.7 | 3 | 14 | 0.02 | GH-C 1832 |
| | | | 28 | 0.02 | |
| Oregon, 1986 | 1.7 | 3 | 15 | 1.8 | GH-C 1832 |
| | | | 29 | 0.51 | |
| Pennsylvania, 1986 | 1.7 | 3 | 14 | 0.36 | GH-C 1832 |
| | | | 21 | 0.08 | |
| | | | 28 | 0.11 | |
| Washington, 1986 | 1.7 | 3 | 14 | 0.89 | GH-C 1832 |
| | | | 28 | 0.34 | |
| Blackberries | | | | | |
| Maryland, 1984 | 1.7 | 3 | 14 | 0.08 | GH-C 1903 |
| | 1.7 | 4 | 14 | 0.16 | |
| Michigan, 1983 | 1.7 | 3 | 14 | 0.33 | GH-C 1903 |
| | | | 28 | 0.19 | |
| New York, 1984 | 1.7 | 3 | 14 | 0.14 | GH-C 1903 |
| | | | 14 | 0.25 | |
| Oregon, 1983 | 1.7 | 3 | 15 | 1.9 | GH-C 1903 |
| | | | 29 | 0.57 | |
| Boysenberries | | | | | |
| Oregon, 1983 | 1.7 | 3 | 15 | 1.3 | GH-C 1903 |
| | | | 29 | 0.33 | |
| Black raspberries | | | | | |
| Pennsylvania, 1984 | 1.7 | 3 | 14 | 0.34 | GH-C 1903 |
| | | | 21 | 0.30 | |
| | | | 28 | 0.13 | |
| Red raspberries | | | | | |
| Maryland, 1984 | 1.7 | 3 | 14 | 0.12 | GH-C 1903 |
| | | | 14 | 0.14 | |
| Michigan, 1984 | 1.7 | 3 | 14<br>28 | 0.58<br>0.32 | GH-C 1903 |
| New York, 1984 | 1.7 | 3 | 14<br>28 | 0.45<br>0.07 | GH-C 1903 |
| Oregon, 1983 | 1.7 | 3 | 12<br>26 | 1`.8<br>0.46 | GH-C 1903 |
| Washington, 1983 | 1.7 | 3 | 14<br>28 | 0.38<br>0.16 | GH-C 1903 |

[1] North American Free Trade Agreement

In residue trials on raspberries and gooseberries in the UK during 1974 to 1977 a single application of an EC formulation of chlorpyrifos was made at 0.72 kg ai/ha. Samples were collected from 3 to 28 days after treatment and analysed by a gas-chromatographic method sensitive to 0.01 mg/kg (Hollick and Walker, 1976a; Freeman, 1978a). The results are shown in Table 40.

Table 40. Residues of chlorpyrifos in raspberries and gooseberries from supervised trials in the UK.

| Country, year | Application | | | | PHI, days | Residue, mg/kg [1] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-UK | 480 EC | 0.72 | (0.14)<br>(0.07) | 2 | 7<br>14 | | Raspberry<br>Gooseberry |
| 1974 | EC | 0.71 | | 1 | 13 | 0.11 | GHE-P 435 |
| Raspberries | | | | | 13 | 0.07 | |
| | | | | | 14 | 0.15 | |

| Country, year | Application | | | | PHI, days | Residue, mg/kg [1] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| 1976 | EC | 0.72 | | 1 | 7 | 0.52 | GHE-P 435 |
| Raspberries | | | | | 14 | 0.18 | |
| 1974 | EC | 0.71 | | 1 | 28 | 0.13 | GHE-P 435 |
| Gooseberries | | | | | 26 | 0.16 | |
| 1977 | EC | 0.71 | | 1 | 3 | 0.37 | GHE-P 575 |
| Raspberries | | | | | 7 | 0.25 | |
| | | | | | 13 | 0.21 | |
| | | | | | 14 | 0.09 | |
| 1977 Gooseberries | EC | 0.72 | | 1 | 14 | 0.05 | GHE-P 575 |

[1] Average of two determinations. LOQ 0.01 mg/kg

Strawberries. Supervised trials on strawberries were conducted in the UK in 1977, 1992 and 1995. In the 1977 trial a single application of chlorpyrifos EC at 0.71-0.72 kg ai/ha was made and samples were taken 3-14 days after treatment (Freeman, 1978a).

In the later trials multiple applications of chlorpyrifos WG or EC formulations were made at approximately 2-week intervals until harvest. In one 1995 trial a single application was made to each of six separate plots and a seventh plot received 6 applications. A similar trial was conducted in 1992. In two further trials in 1992 there was one application to 5 separate plots and a sixth plot received 5 applications. The formulations were diluted to 0.072 kg ai/hl and applied at 0.72 kg ai/ha. Fruits were sampled at commercial harvest, 7 days after the final application, and at other intervals up to 75 days (Portwood, 1996a).

In two other trials chlorpyrifos WG diluted to 0.072 kg ai/hl and applied at a rate of 0.72 kg ai/ha was applied at 2-week intervals until harvest. In one trial one plot received 6 applications and in the other the plot received 5 applications. Fruits were sampled at commercial harvest, 7 days after final application (Portwood, 1996b).

The results are shown in Table 41.

Table 41. Residues of chlorpyrifos in strawberries from supervised trials in the UK.

| Year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-UK | 480 EC | 0.72 | 0.072 | 3 | 7 | | |
| 1977 | EC | 0.72 | | 1 | 3 | 0.08 | GHE-P 575 |
| | | | | | 7 | 0.09 | |
| | | | | | 14 | 0.02 | |
| 1977 | EC | 0.71 | | 1 | 3 | 0.08 | GHE-P 575 |
| | | | | | 7 | 0.04 | |
| | | | | | 13 | 0.03 | |
| | | | | | 14 | 0.04 | |
| 1992 | WG | 0.72 | 0.072 | 6 | 7 | 0.09 | GHE-P 5492R |
| | | 0.72 | 0.072 | 1 | 7 | 0.06 | |
| | | | | 1 | 21 | 0.03 | |
| | | | | 1 | 35 | 0.03 | |
| | | | | 1 | 46 | 0.02 | |

| Year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | 1 | 61 | <0.01 | |
| | | | | 1 | 75 | <0.01 | |
| 1992 | EC | 0.72 | 0.072 | 5 | 7 | 0.10 | GHE-P 5492R |
| | | 0.72 | 0.072 | 1 | 7 | 0.05 | |
| | | | | 1 | 22 | 0.02 | |
| | | | | 1 | 38 | <0.01 | |
| | | | | 1 | 52 | <0.01 | |
| | | | | 1 | 69 | <0.01 | |
| 1992 | WG | 0.72 | 0.072 | 5 | 7 | 0.10 | GHE-P 5492R |
| | | 0.72 | 0.072 | 1 | 7 | 0.09 | |
| | | | | 1 | 22 | 0.02 | |
| | | | | 1 | 38 | <0.01 | |
| | | | | 1 | 52 | <0.01 | |
| | | | | 1 | 69 | <0.01 | |
| 1995 | WG | 0.72 | 0.072 | 6 | 7 | 0.15 | GHE-P 5493R |
| 1995 | WG | 0.72 | 0.072 | 5 | 7 | 0.12 | GHE-P 5493R |
| 1995 | EC | 0.72 | 0.072 | 6 | 7 | 0.14 | GHE-P 5492R |
| | EC | 0.72 | 0.072 | 1 | 7 | 0.07 | |
| | | | | 1 | 21 | 0.02 | |
| | | | | 1 | 35 | 0.03 | |
| | | | | 1 | 46 | <0.01 | |
| | | | | 1 | 61 | <0.01 | |
| | | | | 1 | 75 | <0.01 | |

[1] LOQ 0.01 mg/kg

In trials in Idaho and Oregon, USA, plots of strawberries were treated pre-plant in 1985 with Lorsban 4E insecticide at 2.24 kg ai/ha, and by two foliar sprays the following year at 1.12 kg ai/ha. In a similar trial in California, the plots received only the two foliar sprays. Strawberries were harvested from each trial 21 days after the last application. The results are shown in Table 42 (Wetters, 1987a).

Table 42. Residues of chlorpyrifos in strawberries from supervised trials in the USA.

| Location, Year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP USA | 480 g/l | 1.12 | 0.30 | 2 | 21 | | Pre-boom only |
| CA, 1986 | EC | 1.12 | 0.28 | 2 | 21 | 0.06, 0.07, 0.03, 0.04 | GH-C 1871 |
| ID, 1986 | EC | 1.12 | 0.27 | 2 | 21 | 0.01, 0.01, 0.02, 0.02 | GH-C 1871 |
| OR, 1986 | EC | 1.12 | 0.27 | 2 | 21 | 0.03, 0.04, 0.03, 0.04 | GH-C 1871 |

[1] LOQ 0.01 mg/kg

Grapes. Supervised trials were conducted in France, Greece, Italy, South Africa and the USA from 1980 to 1995. The results are shown in Tables 43 to 45.

In two trials in Northern France in 1993 (4 replicate plots analysed separately) single applications of chlorpyrifos in an EC formulation contained either 228 g ai/l at 1.5 l/ha or 480 g/l at 0.71 l/ha, both giving rates of 0.34 kg ai/ha. Samples were taken at harvest, 21 days after treatment (Khoshab and Berryman, 1994b).

In two further trials in 1994 in Northern France, single applications of the 228 EC or 480 EC formulations of chlorpyrifos both diluted to 0.16 kg ai/hl were applied at 0.34 kg ai/ha. Grapes were sampled at intervals from 0 to 28 days (Khoshab *et al.*, 1995b).

In two trials in 1995, one in the north and one in the south of France, single applications of either chlorpyrifos WG or 480 EC formulations diluted to 0.065 kg ai/hl at 0.34 kg ai/ha were applied to the vines 28 and 27 days before harvest respectively. Fruits were sampled at intervals from days 0 to 27 (Portwood and Williams, 1996i).

In a trial with four replicate plots, sampled separately, of 12 vines for each treatment, in Southern France in 1994 single applications of either the 228 EC or 480 EC formulation of chlorpyrifos diluted to 0.166 kg ai/hl at 0.34 kg ai/ha were applied and the grapes harvested 24 days after treatment (Khoshab *et al.*, 1995c).

Table 43. Residues of chlorpyrifos in grapes from supervised trials in France.

| Year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-France | EC | 0.34 | (0.17) | | 21 | | |
| 1993 | 228 EC | 0.34 | 0.134 | 1 | 21 | 0.06, 0.04, 0.04, 0.04 | GHE-P 3636 |
| 1993 | 480 EC | 0.34 | 0.134 | 1 | 21 | 0.02, 0.02, 0.01, 0.01 | GHE-P 3636 |
| 1994 | 228 EC | 0.34 | 0.16 | 1 | 0 | 0.28 | GHE-P 4080 |
| | | | | | 7 | 0.16 | |
| | | | | | 15 | 0.09 | |
| | | | | | 22 | 0.06 | |
| | | | | | 28 | 0.07 | |
| 1994 | 480 EC | 0.34 | 0.16 | 1 | 0 | 0.34 | GHE-P 4080 |
| | | | | | 7 | 0.14 | |
| | | | | | 15 | 0.10 | |
| | | | | | 22 | 0.04 | |
| | | | | | 28 | 0.06 | |
| 1994 | 228 EC | 0.34 | 0.166 | 1 | 24 | 0.06, 0.08, 0.08, 0.08 | GHE-P 4082 |
| 1994 | 480 EC | 0.34 | 0.166 | 1 | 24 | 0.07, 0.08, 0.06, 0.08 | GHE-P 4082 |
| 1995 | WG | 0.34 | 0.065 | 1 | 0 | 0.56 | GHE-P 4968R |
| | | | | | 7 | 0.21 | |
| | | | | | 14 | 0.19 | |
| | | | | | 22 | 0.14 | |
| | | | | | 28 | 0.08 | |
| 1995 | 480 EC | 0.34 | 0.065 | 1 | 0 | 0.39 | GHE-P 4968R |
| | | | | | 7 | 0.16 | |
| | | | | | 14 | 0.07 | |
| | | | | | 22 | 0.1 | |
| | | | | | 28 | 0.03 | |

| Year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| 1995 | WG | 0.34 | 0.065 | 1 | 0 | 1.3 | GHE-P 4968R |
| | | | | | 7 | 0.36 | |
| | | | | | 14 | 0.15 | |
| | | | | | 20 | 0.10 | |
| | | | | | 27 | 0.12 | |
| 1995 | 480 EC | 0.34 | 0.065 | 1 | 0 | 1.0 | GHE-P 4968R |
| | | | | | 7 | 0.17 | |
| | | | | | 14 | 0.13 | |
| | | | | | 20 | 0.15 | |
| | | | | | 27 | 0.11 | |

[1] LOQ 0.01 mg/kg

In trials in South Africa in 1980 two applications of the EC formulation diluted to 0.036 or 0.072 kg ai/hl were made, with samples taken at intervals from 0 to 35 days (Freeman *et al.*, 1980). In trials at three locations in Italy in 1986, single applications of the EC formulation were made to plots of grape vines at either 0.69 or 0.77 kg ai/ha, and in two other trials two applications at 0.77 kg ai/ha. Samples were taken between 7 and 69 days (Teasdale, 1988b).

In a trial in Greece according to GAP in Italy (Portwood and Williams, 1996j) single applications of either chlorpyrifos WG or 480 EC formulations, diluted to 0.065 kg ai/hl, were made at 0.83 kg ai/ha 28 days before harvest.

The results of all the trials are shown in Table 44.

Table 44. Residues of chlorpyrifos in grapes from supervised trials in Italy, Greece and South Africa.

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Italy | EC | (0.5) | 0.05 | 2 | 30 | | |
| Italy, 1986 | EC | 0.69 | | 1 | 7 | 0.17 | GHE-P 1818R |
| | | | | | 14 | 0.09 | |
| | | | | | 33 | 0.04 | |
| | | | | | 49 | 0.06 | |
| Italy, 1986 | EC | 0.69 | | 1 | 7 | 0.50 | GHE-P 1818R |
| | | | | | 14 | 0.19 | |
| | | | | | 33 | 0.02 | |
| | | | | | 49 | 0.03 | |
| Italy, 1986 | EC | 0.77 | | 1 | 12 | 0.69 | GHE-P 1818R |
| | | | | | 19 | 0.28 | |
| | | | | | 38 | 0.10 | |
| | | | | | 69 | 0.05 | |
| Italy, 1986 | EC | 0.77 | | 2 | 13 | 0.50 | GHE-P 1818R |
| Italy, 1986 | EC | 0.77 | | 2 | 44 | 0.26 | GHE-P 1818R |
| GAP-Greece NONE | | | | | | | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Italy | | | | | | | |
| Greece, 1995 | WG | 0.54 | 0.065 | 1 | 28 | 0.09 | GHE-P 4967 |
| | | | | | | | |
| | EC | 0.54 | 0.065 | 1 | 28 | 0.32 | |
| GAP-SA | 480 EC | | 0.036 | | 28 | | High volume |
| South Africa, | EC | | 0.036 | 2 | 0 | 1.1 | GHE-P 792 |
| 1980 | | | | | 7 | 0.92 | |
| | | | | | 14 | 0.49 | |
| | | | | | 21 | 0.17 | |
| | | | | | 28 | 0.13 | |
| | | | | | 35 | 0.09 | |
| South Africa, | EC | | 0.072 | 2 | 7 | 0.93 | GHE-P 792 |
| 1980 | | | | | 14 | 0.14 | |
| | | | | | 21 | 0.20 | |
| | | | | | 28 | 0.28 | |
| | | | | | 35 | 0.17 | |
| South Africa, | EC | | 0.036 | 2 | 0 | 2.2 | GHE-P 792 |
| 1980 | | | | | 7 | 0.71 | |
| | | | | | 14 | 0.26 | |
| | | | | | 21 | 0.24 | |
| | | | | | 28 | 0.17 | |
| | | | | | 35 | 0.15 | |
| South Africa, | EC | | 0.072 | 2 | 0 | 3.9 | GHE-P 792 |
| 1980 | | | | | 7 | 0.97 | |
| | | | | | 14 | 0.42 | |
| | | | | | 21 | 0.29 | |
| | | | | | 28 | 0.20 | |
| | | | | | 35 | 0.24 | |

[1] LOQ 0.01 mg/kg

In a trial at 12 locations in the USA foliar sprays of both chlorpyrifos 480 EC and 50W were applied to grapes at a total rate of 6.7 kg ai/ha. 480 EC was applied at bud swell, 2.24 kg ai/ha, and 50W approximately 51, 41, 31 and 21 days before normal harvest at 1.12 kg ai/ha. Grapes were sampled 14, 21 and 30 days after the last application (McCormick *et al.*, 1994). The results are shown in Table 45.

Table 45. Residues of chlorpyrifos in grapes from supervised trials in the USA.

| Location, year, (variety) | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-USA<br>No foliar. soil spray only about vines. No NAFTA[2] country GAP. | EC | | 0.27 | 1 | 35 | | |
| NY, 1993 | EC + WP(x4) | 2.24 + 1.12(4x) | 0.48 + 0.24 | 1 + 4 | 14 | 0.33 | GH-C 3272 |

| Location, year, (variety) | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| (Concord) | | | | | 21 | 0.31 | |
| | | | | | 30 | 0.37 | |
| NY, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.09 + 0.05 | 1 + 4 | 14 | 0.41 | GH-C 3272 |
| (Concord) | | | | | 21 | 0.38 | |
| | | | | | 30 | 0.40 | |
| WA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.61 | GH-C 3272 |
| (Concord) | | | | | 21 | 0.71 | |
| | | | | | 30 | 0.54 | |
| WA, 1993 | EC + WP(x4) | 2.24 + 1,12(x4) | 0.09 + 0.05 | 1 + 4 | 14 | 0.78 | GH-C 3272 |
| (Concord) | | | | | 21 | 0.59 | |
| | | | | | 30 | 0.65 | |
| NY, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.61 | GH-C 3272 |
| (Catawba) | | | | | 21 | 0.44 | |
| | | | | | 30 | 0.43 | |
| WA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.71 | GH-C 3272 |
| (Chardonnay) | | | | | 21 | 0.55 | |
| | | | | | 30 | 0.58 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.19 | GH-C 3272 |
| (Thompson) | | | | | 21 | 0.08 | |
| | | | | | 30 | 0.10 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.20 | GH-C 3272 |
| (Thompson) | | | | | 21 | 0.15 | |
| | | | | | 30 | 0.05 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.14 | GH-C 3272 |
| (Flame seedless) | | | | | 21 | 0.12 | |
| | | | | | 30 | 0.05 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.09 + 0.05 | 1 + 4 | 14 | 0.25 | GH-C 3272 |
| (Flame seedless) | | | | | 21 | 0.15 | |
| | | | | | 30 | 0.11 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.45 | GH-C 3272 |
| (Chenin blanc) | | | | | 21 | 0.26 | |
| | | | | | 30 | 0.28 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.09 + 0.05 | 1 + 4 | 14 | 0.31 | GH-C 3272 |
| (Chenin blanc) | | | | | 21 | 0.22 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.09 + 0.05 | 1 + 4 | 30 | 0.19 | GH-C 3272 |
| (Chenin blanc) | | | | | | | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.30 | GH-C 3272 |
| (Carignane) | | | | | 21 | 0.32 | |
| | | | | | 30 | 0.36 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.25 | GH-C 3272 |
| (Zinfandel) | | | | | 21 | 0.17 | |
| | | | | | 30 | 0.14 | |
| CA, 1993 | EC + WP(x4) | 2.24 + 1.12(x4) | 0.48 + 0.24 | 1 + 4 | 14 | 0.35 | GH-C 3272 |
| (Riesling) | | | | | 21 | 0.17 | |

| Location, year, (variety) | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 30 | 0.14 | |
| CA, 1993 | EC + WP(x4) | 2.24+ 1.12(x4) | 0.48 +0.24 | 1 + 4 | 14 | 0.12 | GH-C 3272 |
| (Riesling) | | | | | 21 | 0.08 | |
| | | | | | 30 | 0.08 | |

[1] LOQ 0.01 mg/kg
[2] North American Free Trade Agreement

Assorted tropical and sub-tropical fruits, inedible peel

Banana. Chlorpyrifos is applied to banana plants as a direct foliar spray, with an EC or WP formulation. Supervised trials were conducted in Australia, South Africa and Spain from 1977 to 1994. The results are shown in Tables 46 and 47.

In Australia, the 500 EC formulation was applied aerially to plants at 0.8 kg ai/ha, and samples analysed 24 hours and 14 days later (Vella, 1981).

In residue trials in South Africa during 1977-1979 bananas were treated with chlorpyrifos at rates of 0.036 and 0.075 kg ai/hl, and samples taken at regular intervals up to 21 days after treatment (Hollick and Walker, 1980).

Table 46. Residues of chlorpyrifos in bananas from foliar application in Australia and South Africa.

| Country, year | Application | | | | PHI, days | Residues, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Australia | 500 EC | 1.0 | 0.1 | | 14 | | |
| | 500 WP | 1.0 | 0.1 | | 14 | | |
| Australia, aerial 1981 | | | | | | | |
| Peel | EC | 0.8 | | 1 | 0 | 0.03, 0.04, 0.08, 0.05 [1] | PAU 3183042 |
| | | | | | 14 | 0.02, 0.03, 0.03, 0.03 [1] | |
| Pulp | | | | | 0 | <0.02, <0.02, <0.02, <0.02 [1] | |
| | | | | | 14 | <0.02, <0.02, <0.02, <0.02 [1] | |
| GAP-South Africa | 480 EC | | 0.036 | 8 | 28 | | |
| South Africa, 1978 | | | | | | | |
| Peel | EC | | 0.036 | 1 | 0 | 0.73 [2] | GHE-P 722 |
| | | | | | 1 | 0.71 [2] | |
| | | | | | 3 | 0.44 [2] | |
| | | | | | 7 | 0.29 [2] | |
| | | | | | 11 | 0.37 [2] | |
| | | | | | 21 | 0.33 [2] | |
| Pulp | | | | | 0 | <0.01 [2] | GHE-P 722 |
| | | | | | 1 | <0.01 [2] | |
| | | | | | 3 | <0.01 [2] | |
| | | | | | 7 | <0.01 [2] | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 11 | <0.01 [2] | |
| | | | | | 21 | <0.01 [2] | |
| South Africa, 1978 | 480 EC | | 0.075 | 3 | 0 | <0.01 | GHE-P 722 |
| Pulp | | | | | 3 | <0.01 | |
| | | | | | 5 | <0.01 | |
| | | | | | 7 | <0.01 | |
| | | | | | 12 | <0.01 | |

[1]LOQ 0.02 mg/kg
[2] Recoveries averaged 93% (±4%) for banana peel and 97% (±4%) for banana pulp. LOQ 0.01 mg/kg.

In three trials at two locations in Spain during 1992 single applications of chlorpyrifos diluted to 0.096 kg ai/hl were applied at 0.43 kg ai/ha to the plants when the bananas were mature. Fruit were sampled at intervals from one day to 21 days after treatment. The recoveries of chlorpyrifos from whole bananas in two of the trials were 88±11%, n = 9 at 0.01 to 2.0 mg/kg. The results of the third trial were considered invalid owing to unexplained contamination of untreated samples (Khoshab, *et al.*, 1994c).

In trials in four different glasshouses near La Nuncia, Spain, in 1994, single applications of chlorpyrifos EC formulation were made to 4 trees approximately 21 days before harvest. The formulation was diluted to 0.096 kg ai/hl and applied to run-off at a rate of 6.4 or 4.3 kg ai/ha (Trials A and B) and 2.7 kg ai/ha (Trials C and D). The recovery of chlorpyrifos from whole bananas was 88% at 0.1 mg/kg and 94% at 0.25 mg/kg fortification. The fruit were harvested 21 or 22 days after the application. In trial D, each of the four treated trees was sampled and analysed separately (Khoshab and Clements, 1995a).

In a separate glasshouse trial in Spain in 1994, one application of the EC formulation diluted to 0.096 kg ai/hl was made at 2.7 kg ai/ha, and samples were taken at intervals from 0 to 21 days after treatment (Khoshab and Clements, 1995b). The analytical recovery from whole bananas was 95% at 0.10 mg/kg.

Table 47. Residues of chlorpyrifos in bananas from supervised trials in Spain.

| Year | Application | | | No. | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | | | | |
| GAP-Spain | 480 EC | | 0.096 | 1 | 21 | | |
| | 250 WP | | 0.1 | 1 | 21 | | |
| 1992 | EC | 0.43 | 0.096 | 1 | 1 | 1.5 | GHE-P 3349 |
| whole fruit | | | | | 3 | 1.2 | |
| whole fruit | | | | | 7 | 0.73 | |
| whole fruit | | | | | 14 | 0.80 | |
| peel | | | | | 14 | 2.0 | |
| pulp | | | | | 14 | 0.01 | |
| whole fruit | | | | | 21 | 0.48 | |
| peel | | | | | 21 | 1.3 | |
| pulp | | | | | 21 | 0.01 | |
| 1992 | EC | 0.43 | 0.096 | 1 | 1 | 1.6 | GHE-P 3349 |

| Year | Application | | | No. | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | | | | |
| whole fruit | | | | | 3 | 1.1 | |
| whole fruit | | | | | 7 | 0.57 | |
| whole fruit | | | | | 14 | 0.59 | |
| peel | | | | | 14 | 1.4 | |
| pulp | | | | | 14 | <0.01 | |
| whole fruit | | | | | 21 | 0.37 | |
| peel | | | | | 21 | 0.83 | |
| pulp | | | | | 21 | <0.01 | |
| 1994 (Glasshouse) trial A | EC | 6.4 | 0.096 | 1 | 21 | 0.75 whole fruit | GHE-P 4518 |
| (Glasshouse) trial B | EC | 4.3 | 0.096 | 1 | 21 | 1.1 whole fruit | GHE-P 4518 |
| (Glasshouse) trial C | EC | 2.7 | 0.096 | 1 | 22 | 1.1 whole fruit | GHE-P 4518 |
| (Glasshouse) trial D | EC | 2.7 | 0.096 | 1 | 22 | 1.6; 1.5; 1.6; 1.6 | GHE-P 4518 |
| (Glasshouse) | EC | 2.7 | 0.096 | 1 | 0 | 1.7 whole fruit | GHE-P 4519 |
| | | | | | 3 | 1.2 | |
| | | | | | 7 | 0.99 | |
| | | | | | 14 | 0.83 | |
| | | | | | 21 | 1.6 | |

[1] LOQ 0.01 mg/kg.

1% chlorpyrifos is also impregnated into plastic resins which are extruded into shrouds to cover the banana bunches. In Latin America trials were conducted in five locations in 1972 in Ecuador, Honduras and Costa Rica. Chlorpyrifos-impregnated bags were prepared by either spraying of pre-formed bags, or extrusion of chlorpyrifos-impregnated polyethylene pellets into roll film which was cut into bags. In the first method polyethylene bags were suspended by clamps from a wire and the exposed surface was sprayed with an airbrush with a solution of chlorpyrifos. The bags were then turned inside out so that the treated area was inside the bag. In the second method chlorpyrifos was blended with low-density polyethylene pellets in a shell blender at 90°C for half an hour. Pellets were tumble-blended with base resin to give a polymer mixture containing 1 and 2% (w/w) chlorpyrifos. The resin samples were then fabricated into a film, with an extruder.

Treated bags were placed over young banana bunches according to conventional practice until harvest (about 1 to 3 months), when samples of fingers selected at random from the top, middle and bottom sections of the shrouded stem were collected (Herman and Dishburger, 1972).

Using similar methods, in a trial in the Philippines to compare slow-release chlorpyrifos IPE with the standard chlorpyrifos IPE, bananas were shrouded weekly with the “Lorsban”-impregnated bags until harvest at 12 weeks (Ling, 1986).

The results are shown in Table 48.

Table 48. Residues of chlorpyrifos in bananas from trials using impregnated IPE bags.

| Country, year | Application | | | PHI, weeks | Residues, mg/kg | Reference |
|---|---|---|---|---|---|---|
| | Form. | g ai/bag | No. | | | |
| GAP-Columbia | 1% in IPE | | 1 bag/season | 12-13 | | |

| Country, year | Application | | | PHI, weeks | Residues, mg/kg | Reference |
|---|---|---|---|---|---|---|
| | Form. | g ai/bag | No. | | | |
| Ecuador, 1972 | 1% in IPE | 0.33 | 1 bag/season | 12-13 | | |
| Whole | | | | | 0.05, 0.06, 0.04 | GH-C 603 |
| Peel | | | | | 0.17, 0.06, 0.04 | |
| Pulp | | | | | <0.01, <0.01, ≤0.01 | |
| Costa Rica, 1972 | 1% in IPE | 0.33 | 1 bag/season | 12-13 | | GH-C 603 |
| Whole | | | | | 0.04, 0.01, 0.03 | |
| Peel | | | | | 0.19, 0.02, 0.08 | |
| Pulp | | | | | <0.01, <0.01, 0.01 | |
| Costa Rica, 1972 | 1% in IPE | 0.33 | 1 bag/season | 12-13 | | |
| Whole | | | | | 0.01, 0.01, 0.01 | GH-C 603 |
| Peel | | | | | 0.12, 0.04, 0.06 | |
| Pulp | | | | | <0.01, <0.01, ≤0.01 | |
| Costa Rica, 1972 | 1% in IPE | 0.66 | 1 bag/season | 12-13 | | GH-C 603 |
| Whole | | | | | 0.05, 0.04, 0.04 | |
| Peel | | | | | 0.14, 0.11, 0.06 | |
| Pulp | | | | | <0.01, <0.01, ≤0.01 | |
| Honduras, 1972 | 1% in IPE | 0.25 | 1 bag/season | 12-13 | | GH-C 603 |
| Whole | | | | | 0.01, <0.01, 0.01 | |
| Peel | | | | | 0.02, 0.05, 0.02 | |
| Pulp | | | | | <0.01, <0.01, ≤0.01 | |
| Honduras, 1972 | 1% in IPE | 0.5 | 1 bag/season | 12-13 | | GH-C 603 |
| Whole | | | | | 0.01, 0.01, <0.01 | |
| Peel | | | | | 0.03, 0.02, <0.01 | |
| Pulp | | | | | <0.01,<0.01, ≤0.01 | |
| Costa Rica, 1972 | 1% in IPE | 0.25 | 1 bag/season | 12-13 | | GH-C 603 |
| Whole | | | | | 0.01, 0.02, 0.02 | |
| Peel | | | | | 0.03, 0.02, 0.05 | |
| Pulp | | | | | <0.01, <0.01, ≤0.01 | |
| Costa Rica, 1972 | 1% in IPE | 0.5 | 1 bag/season | 12-13 | | GH-C 603 |
| Whole | | | | | 0.08, 0.07, 0.13 | |
| Peel | | | | | 0.13, 0.06, 0.08 | |
| Pulp | | | | | <0.01, <0.01, ≤0.01 | |
| GAP-Philippines | 1% in IPE | | 1 bag/season | 12-13 | | |
| Philippines, 1983 | 1% in IPE | 0.4 | 1 bag/season | | | |
| Whole | | | | 10 | 0.10, 0.07, 0.11 | PM-86-010 |
| Peel | | | | | 0.08, 0.05, 0.07 | |
| Pulp | | | | | 0.02, 0.02, 0.04 | |
| Whole | | | | 11 | 0.12, 0.08, 0.08 | |
| Peel | | | | | 0.08, 0.06, 0.01 | |
| Pulp | | | | | 0.04, 0.02, 0.07 | |
| Whole | | | | 12 | 0.06, 0.06, 0.13 | |
| Peel | | | | | 0.01, <0.01, 0.11 | |
| Pulp | | | | | 0.05, 0.05, 0.02 | |
| Philippines, 1983 | 1% in IPE | 0.4 | | | | |
| Whole | | | | 10 | 0.07, 0.05, 0.07 | PM-86-010 |

| Country, year | Application | | | PHI, weeks | Residues, mg/kg | Reference |
|---|---|---|---|---|---|---|
| | Form. | g ai/bag | No. | | | |
| Peel | | | | | 0.06, <0.01, 0.03 | |
| Pulp | | | | | 0.01, 0.05, 0.04 | |
| Whole | | | | 11 | 0.02, 0.04, 0.03 | |
| Peel | | | | | <0.01, <0.01, 0.02 | |
| Pulp | | | | | 0.02, 0.02, 0.03 | |
| Whole | | | | 12 | 0.04, 0.21, 0.07 | |
| Peel | | | | | <0.01, 0.19, 0.05 | |
| Pulp | | | | | 0.04, 0.02, 0.02 | |

Kiwifruit. In New Zealand in a supervised trial in 1978 chlorpyrifos was applied at 0.025 kg ai/hl and fruits were collected at intervals up to 21 days after treatment (MacDairmid and Mercer, 1978). In another trial in 1979, chlorpyrifos was applied at 0.025 kg ai/hl in EC and WP formulations. Three and 9 applications were made and samples taken at intervals from the day after the last application to 21 days (Upritchard, 1980).

In trials in 1985 in New Zealand chlorpyrifos 50W was applied to kiwifruit at 1 kg ai/ha and the mature fruits were harvested 10-39 days after the last application, but necessary details were not reported (Wilson, 1986).

The results of the trials are shown in Table 49.

Table 49. Residues of chlorpyrifos in whole kiwifruit from supervised trials in New Zealand.

| Year | Application | | | | PHI, days | Residues, mg/kg[1] | | Reference |
|---|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | Whole | Pulp | |
| GAP-New Zealand | 500 WP | 0.50 | 0.025 | | 14 | | | |
| | 400 EC | 0.48 | 0.024 | | 14 | | | |
| 1985[2] | WP | 1.0 | | 4 | 16 | 0.29 | | GHF-P 544 |
| | | | | | 20 | 0.14 | | |
| | | | | | 39 | 0.09 | | |
| 1985[2] | WP | 1.0 | | 5 | 10 | 0.91 | | |
| | | | | | 12 | 0.27, 0.37 | | GHF-P 544 |
| | | | | | 13 | 1.6 | | |
| | | | | | 16 | 0.30 | | |
| | | | | | 21 | 0.37 | | |
| | | | | | 24 | 0.67 | | |
| 1985[2] | WP | 1.0 | | 6 | 11 | 0.55 | | GHF-P 544 |
| | | | | | 13 | 0.54 | | |
| | | | | | 16 | 0.66 | | |
| | | | | | 17 | 0.62 | | |
| | | | | | 25 | 0.26, 0.37 | | |
| | | 1.0 | - | 7 | 11 | 0.37 | | GHF-P 544 |
| 1977 | WP | | 0.025 or 0.09 | 1 | 0 | 0.25 | <0.05 | GHF-P 086. Conflicting rates reported. Whole fruit calculated. |
| . | | | | | 1 | 0.08 | <0.05[3] | Whole fruit calculated. |
| | | | | | 2 | 0.08 | <0.05 | |
| | | | | | 4 | 0.08 | <0.05 | |

| Year | Application | | | | PHI, days | Residues, mg/kg[1] | | Reference |
|---|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | Whole | Pulp | |
| | | | | | 7 | 0.07 | <0.05 | |
| | | | | | 14 | <0.05 | <0.05 | |
| | | | | | 21 | <0.05 | <0.05 | |
| 1979 | WP | 1.0 | 0.025 | 4 | 1 | 0.96 | <0.05 | GHE-P 147 |
| | | | | | 2 | 0.79 | | |
| | | | | | 4 | 0.51 | | |
| | | | | | 7 | 0.49 | | |
| | | | | | 14 | 0.26 | <0.05 | |
| | | | | | 21 | 0.40 | | |
| 1979 | WP | 1.0 | 0.025 | 9 | 1 | 1.5 | 0.10[4] | GHE-P 147. Whole fruit calculated. |
| | | | | | 2 | 1.1 | 0.08 | |
| | | | | | 4 | 1.7 | 0.13 | |
| | | | | | 7 | 1.2 | 0.07 | |
| | | | | | 14 | 1.9 | 0.08 | |
| | | | | | 21 | 0.48 | 0.08 | |
| 1979 | EC | 1.0 | 0.025 | 4 | 1 | 0.83 | <0.05 | GHE-P 147 |
| . | | | | | 2 | 0.93 | | |
| | | | | | 4 | 1.0 | | |
| | | | | | 7 | 0.85 | | |
| | | | | | 14 | 0.75 | <0.05 | |
| | | | | | 21 | 0.59 | | |
| 1979 | EC | 1.0 | 0.025 | 9 | 1 | 2.2 | 0.05 | GHE-P 147. Whole fruit calculated. |
| . | | | | | 2 | 2.2 | 0.09 | |
| | | | | | 4 | 1.6 | 0.07 | |
| | | | | | 7 | 1.4 | 0.11 | |
| | | | | | 14 | 1.0 | 0.14 | |
| | | | | | 21 | 0.71 | 0.15 | |

[1] LOQ 0.01 mg/kg
[2] Each result is from a different field.
[3] Recovery from pulp was 94% at 0.05 mg/kg fortification of control samples.
[4] Recovery from pulp was 97% at 0.1 mg/kg fortification of control samples.

## Bulb Vegetables

Onions. An at-plant treatment was reported from the USA (Doom, 1986). G and EC formulations were tested in separate plots, at the rate of 1.12 kg ai/ha. They were applied to onions as a drench and 4.5 kg samples were collected at the regular harvest time of 182 or 210 days after application. Additional trials were reported from Canada, where a G formulation was applied with onion seeds in the seed furrow at the rate of 1.1 kg ai/ha. Samples were collected 80, 109 and 121 days after treatment and analysed (Plaumann *et al.*, 1982). The findings are shown in Table 50.

Table 50. Residues of chlorpyrifos in onions from the at-planting application of G or EC formulations.

| Location, (Variety) Year | Application | | PHI, days | Residue, mg/kg | | Reference/Comment |
|---|---|---|---|---|---|---|
| | kg ai/ha | No. | | Chlorpyrifos | TCP | |
| USA GAP | 1.1 | 1 | | | | G. In-furrow |

| Location, (Variety) Year | Application | | PHI, days | Residue, mg/kg | | Reference/Comment |
|---|---|---|---|---|---|---|
| | kg ai/ha | No. | | Chlorpyrifos | TCP | |
| Canada GAP | 2.4 | 1 | | | | G. In-furrow. For 5 cm furrow width, 1.12 kg ai/ha |
| Selema, California USA (Stockton Early Yellow) 1985-1986 | 15.7 g ai/1000ft 1.12 | 1 | 210 | 0.019, 0.017, 0.016, 0.012 | 0.053, 0.012, 0.025, 0.008 | GH-C 1848 G |
| | | | | 0.024, 0.031, 0.016, 0.007 | 0.062, 0.054, 0.034, <0.01 | EC. 374 l/ha |
| Imperial Valley, California, USA (Colossal) 1985-1986 | 15.7 g ai/1000 ft 1.12 | 1 | 182 | ≤0.01 | <0.01 | G |
| | | | | ≤0.01 | <0.01 | EC. 355 l/ha |
| Canada 1982 | 1.1 | 1 | 80 109 121 | 0.14, 0.12, 0.10, 0.11 0.04, 0.05, 0.04, 0.05 0.03, 0.05, 0.06, 0.05 | | GHS-C 43 |

Seven supervised trials on onions were carried out in Greece in 1993-1995. In the 1993 trials two foliar applications of chlorpyrifos EC at 0.96 kg ai/ha were made to the plants at the 6-7 leaf growth stage 41 and 21 days before harvest. Whole onions were sampled at harvest and all replicates analysed separately (Khoshab and Koliopanos, 1994).

In the two trials in 1994 single applications of EC formulation at a spray concentration of 0.24 kg ai/hl was sprayed on bulb onion plants at 0.96 kg ai/ha 40 days before normal harvest. Whole plant samples were taken at intervals from the day of application to harvest (Khoshab, 1996a). Similar trials were conducted in 1995 using either the EC or WG (750 g ai/l) formulation (Portwood and Williams, 1996k).

The results are shown in Table 51.

Table 51. Residues of chlorpyrifos in onions from supervised trials in Greece (foliar application).

| Year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Greece | 480 EC | 1.20 2.5 (0.96 band) | 0.3 | 3 1 | 7, 20 | | Foliar Broadcast soil/band Labels have conflicting PHIs |
| | 250 WP | 2.5 1.25 0.88 | | 1-2 | 20 | | Pre-plant At planting Post-planting foliar |
| | 50 G | 3.0 | | | | | |
| 1993 | EC | 0.96 | | 2 | 20 | 0.03, 0.02, 0.02, 0.01 | GHE-P 3466 |
| 1994 | EC | 0.96 | 0.24 | 1 | 0 | 9.1 | GHE-P 4514 |
| | | | | | 10 | 0.03 | |
| | | | | | 20 | ≤0.01 | |
| | | | | | 30 | <0.01 | |
| | | | | | 40 | ND [3] | |
| 1994 | EC | 0.96 | 0.24 | 1 | 0 | 11.0 | GHE-P 4514 |
| | | | | | 10 | 0.03 | |
| | | | | | 20 | ≤0.01 | |
| | | | | | 30 | <0.01 | |

| Year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 40 | ND [4] | |
| 1995 | EC | 0.96 | 0.24 | 2 | 0 | 6.1 | GHE-P 4807 |
| | | | | | 5 | 0.24 | |
| | | | | | 10 | 0.13 | |
| | | | | | 15 | 0.03 | |
| | | | | | 20 | 0.05 | |
| 1995 | WP | 0.96 | 0.24 | 2 | 0 | 7.9 | GHE-P 4807 |
| | | | | | 5 | 0.17 | |
| | | | | | 10 | 0.07 | |
| | | | | | 15 | 0.04 | |
| | | | | | 20 | 0.02 | |
| 1995 | EC | 0.96 | 0.24 | 2 | 0 | 5.8 | GHE-P 4807 |
| | | | | | 5 | 0.09 | |
| | | | | | 10 | 0.04 | |
| | | | | | 15 | 0.03 | |
| 1995 | EC | 0.96 | 0.24 | 2 | 20 | 0.02 | GHE-P 4807 |
| 1995 | WP | 0.96 | 0.24 | 2 | 0 | 9.2 | GHE-P 4807 |
| | | | | | 5 | 0.33 | |
| | | | | | 10 | 0.16 | |
| | | | | | 15 | 0.06 | |
| | | | | | 20 | 0.05 | |

[1] All results averages of duplicate determinations
[2] LOQ 0.01 mg/kg
[3] ND = less than 20% of LOQ

In four trials at different locations in the UK in 1993 single applications of EC were applied to the plants at 0.96 kg ai/ha. Whole onions were sampled at harvest after 21-23 days (Khoshab *et al.*, 1994d). The results are shown in Table 52.

Table 52. Residues of chlorpyrifos in onions from supervised trials in the UK (foliar application).

| Year | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-UK | 480 EC | 0.96 | 0.16 | 1-2 | 21 | | Specific commodity under Vegetables on label |
| 1993 | EC | 0.96 | 0.16 | 1 | 23 | 0.04 | GHE-P 3488 |
| 1993 | EC | 0.96 | 0.16 | 1 | 22 | 0.05, 0.03, 0.08, 0.08 | GHE-P 3488 |
| 1993 | EC | 0.96 | 0.16 | 1 | 21 | 0.06 | GHE-P 3488 |
| 1993 | EC | 0.96 | 0.16 | 1 | 23 | 0.07 | GHE-P 3488 |

[1] LOQ, 0.01 mg/kg.

Brassica vegetables

Broccoli. In nine supervised trials in main US broccoli growing areas in 1983–1984 multiple applications of chlorpyrifos 50W were made at 1.12 kg ai/ha, alone or following application at transplant of the EC formulation at 0.8 to 2.2 kg ai/ha. Sample of heads with 2-3 inches of stems were collected at random 14-15 days and 21 days after the last application (Miller *et al.*, 1986a).

In a trial in California in 1986 EC was applied to broccoli twice during the season at 1.68 kg ai/ha, at planting and 30 days later. Samples were collected at normal harvest (McKellar and Ordiway, 1986a). The results are shown in Table 53.

Table 53. Residues of chlorpyrifos in broccoli from supervised trials in the USA.

| Country, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA | 480 EC | 2.5 at planting | 1 | | | Total EC use not to exceed 2.9 kg ai/ha/season. At planting use limited to CA |
| | | 1.1 directed post plant | 1 | 30 | | |
| | 50% WP | 1.1 foliar | 6 | 21 | | AZ and CA only |
| CA, 1983 | WP | 2.2 | 9 | 7 | 0.28 | GH-C 1788 |
| | | | | 14 | <0.01 | |
| | | | | 21 | <0.01 | |
| FL, 1983 | EC + WP | 2.1 + 1.1(x7) | 8 | 7 | 0.03 | GH-C 1788 |
| | | | | 14 | <0.01 | |
| | | | | 21 | <0.01 | |
| FL, 1984 | EC + WP | 1.5 + 1.1(x7) | 8 | 7 | 2.17 | GH-C 1788 |
| | | | | 14 | 0.65 | |
| | | | | 21 | 0.07 | |
| IL, 1983 | EC + WP | 1.5 + 1.1(x6) | 7 | 7 | 0.08 | GH-C 1788 |
| | | | | 14 | <0.01 | |
| | | | | 21 | <0.01 | |
| MA, 1983 | EC + WP | 0.8 + 1.1(x4) | 5 | 7 | 0.16 | GH-C 1788 |
| | | | | 14 | 0.05 | |
| | | | | 21 | 0.01 | |
| MI, 1983 | EC + WP | 1.2 + 1.1(x9) | 10 | 7 | 0.66 | GH-C 1788 |
| | | | | 14 | 0.9 | |
| | | | | 21 | 0.05 | |
| MI, 1983 | EC + WP | 1.7 + 1.1(x6) | 7 | 7 | 0.19 | GH-C 1788 |
| | | | | 15 | 0.05 | |
| | | | | 21 | <0.01 | |
| NJ, 1983 | EC + WP | 0.8 + 1.1(10) | 11 | 7 | 4.5 | GH-C 1788 |
| | | | | 14 | 1.7 | |
| | | | | 21 | 1.4 | |
| OR, 1983 | EC + WP | 1.5 + 1.1(8) | 9 | 7 | 0.86 | GH-C 1788 |
| | | | | 14 | 0.07 | |
| | | | | 21 | 0.03 | |
| CA, 1986 | EC | 1.68 | 2 | 103 | <0.01, <0.01, <0.01, <0.01 | GH-C 1802 |

[1] LOQ 0.01 mg/kg

Brussels sprouts. In a similar trial to the last, also in California in 1986, chlorpyrifos EC was applied to Brussels sprouts twice during the season at a rate of 1.68 kg ai/ha, at planting and 30 days later. Samples were collected at normal harvest (McKellar and Ordiway, 1986a). The results are shown in Table 54.

Table 54. Residues of chlorpyrifos in Brussels sprouts from supervised trials in the USA.

| Year | Application | | | | PHI, Days | Residues, mg/kg [1, 2] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-USA:<br>Brussels sprouts | EC | 2.5 at planting<br>1.1 foliar spray | <br>(0.58) | 1<br>6 | <br>21 | | |
| CA, 1986<br>Brussels sprouts | EC | 1.68 | | 2 | 139 | <0.01, <0.01, <0.01, <0.01 | GH-C 1802 |

[1] LOQ 0.01 mg/kg

Cabbage. In Brazil an EC formulation was applied three times to cabbages at 0.72 or 1.4 kg ai/ha near maturity (Balderrama and Matos, 1994a,b).

Table 55. Residues of chlorpyrifos from the application of an EC formulation to cabbages in Brazil.

| Location, (Variety) Year | Application | | | PHI, days | Residue | Reference/ Comment |
|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Chlorpyrifos, mg/kg | |
| Brazil GAP | (0.3) | 0.03 | 2 | 21 | | Manufacturer applied GAP for Germany. |
| Mogi-Mirim<br>(Georgia Agroc)<br>1992 | 0.72 | 0.24<br>0.24<br>0.08 | 3 | 3<br>7<br>14<br>21 | 2.9<br>0.92<br>0.06<br>0.01 | GHB-P 192 |
| | 1.4 | | 3 | 3<br>7<br>14<br>21 | 7.6<br>2.0<br>0.32<br>0.05 | GHB-P 192 |
| Mori-Mirim, (Chato de Quintal Ag, Savoy)<br>1992 | 0.72 | 0.24<br>0.24<br>0.08 | 3 | 3<br>7<br>14<br>21 | 0.02<br>0.02<br><0.01<br><0.01 | GHB-P 193. |
| | 1.4 | | 3 | 3<br>7<br>14<br>21 | 0.08<br>0.05<br>0.01<br><0.01 | GHB-P 193 |

In trials in South Africa in 1977 single spray applications of an EC formulation were made at 0.08 to 0.22 kg ai/ha, 0.01 to 0.024 kg ai/hl. Samples were collected at intervals from 0 to 35 days (Freeman, 1978b). The results are shown in Table 56.

Table 56. Residues of chlorpyrifos in head cabbages from supervised trials in South Africa.

| Year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-SA | 480 EC | (0.12) | 0.024 | | 7 | | |
| 1977 | EC | 0.17 | 0.019 | 1 | 1 | 1.2 | GHE-P 585 |
| | | | | | 3 | 0.75 | |
| | | | | | 7 | 0.21 | |
| | | | | | 14 | 0.08 | |

| Year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 21 | 0.01 | |
| | | | | | 28 | <0.01 | |
| 1977 | EC | 0.22 | 0.024 | 1 | 1 | 1.4 | GHE-P 585 |
| | | | | | 3 | 0.56 | |
| | | | | | 7 | 0.22 | |
| | | | | | 14 | 0.09 | |
| | | | | | 21 | 0.01 | |
| | | | | | 28 | 0.02 | |
| 1977 | EC | 0.08 | 0.01 | 1 | 2 | 0.01 | GHE-P 585 |
| | | | | | 4 | 0.01 | |
| | | | | | 7 | 0.02 | |
| | | | | | 14 | 0.01 | |
| | | | | | 21 | <0.01 | |
| | | | | | 28 | <0.01 | |
| | | | | | 35 | <0.01 | |
| 1977 | EC | 0.19 | 0.024 | 1 | 2 | 0.01 | GHE-P 585 |
| | | | | | 4 | 0.02 | |
| | | | | | 7 | 0.01 | |
| | | | | | 14 | <0.01 | |
| | | | | | 21 | <0.01 | |
| | | | | | 28 | <0.01 | |
| | | | | | 35 | <0.01 | |

[1] Average of duplicate analyses
[2] LOQ 0.01 mg/kg

In two trials in the UK in 1976 an EC formulation was applied either as a single foliar application at 0.72 kg ai/ha with samples taken after 20 days, or as a soil drench with 2 treatments at 0.024 kg ai/hl and samples taken after 20 and 59 days (Hollick and Walker, 1976b).

In 1993 immature cabbage plants were treated with an EC formulation at 0.96 and 0.48 kg ai/ha at the 2-4 leaf and 10 leaf growth stages, and samples taken at intervals up to 28 days after the second application (Khoshab and Hastings, 1994a).

In another trial in 1993 cabbage plants were treated at the 2-leaf and heart-forming growth stages with an EC formulation at 0.96 and 0.48 kg ai/ha respectively. Samples were taken at harvest 21 days after the second application (Khoshab and Hastings, 1994b).

In trials in 1994 two applications of EC or WG formulations were made to cabbages at 0.96 kg ai/ha and 0.08 kg ai/hl (1st application) and 0.72 kg ai/ha and 0.06 kg ai/hl (2nd). In a third trial the WG formulation was applied twice, firstly at 0.48 kg ai/ha and 0.08 kg ai/hl, then at 0.36 kg ai/ha and 0.06 kg ai/hl. Samples were collected at intervals up to 21 days after the last application (Portwood and Williams, 1995c).

In a further trial in the UK in 1994 two applications (0.96 and 0.72 kg ai/ha) of chlorpyrifos EC were made at the 6-leaf growth stage and 21 days before harvest and samples taken at harvest (Khoshab,

1995a). In another trial under similar conditions samples were taken at intervals up to 21 days after application (Khoshab, 1995b).

The results are shown in Table 57.

Table 57. Residues of chlorpyrifos in cabbages from supervised trials in the UK.

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP- UK | EC | 0.72 | (0.12) | | 21 | | |
| 1976 | EC | 0.72 | | 1 | 20 | 0.01 | GHE-P 438 |
| | | | | | | | Foliar |
| 1976 | EC | | 0.024 | 2 | 20 | <0.01 | GHE-P 438 |
| | | | | | 59 | <0.01 | Soil drench |
| 1993 | EC | 0.96 + 0.48 | 0.08 + 0.16 | 2 | 0 | 17 | GHE-P 3634 |
| | | | | | 7 | 0.62 | Early foliar |
| | | | | | 14 | 0.04 | |
| | | | | | 21 | 0.03 | |
| | | | | | 28 | 0.01 | |
| 1993 | EC | 0.96 + 0.48 | 0.16 + 0.08 | 2 | 21 | 0.05, 0.05, 0.05, 0.07 | GHE-P 3635 |
| | | | | | | | Early foliar |
| 1994 | EC | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 1.5 | GHE-P 4355 |
| | | | | | 6 | 0.72 | Foliar |
| | | | | | 9 | 0.41 | |
| | | | | | 15 | 0.21 | |
| | | | | | 21 | 0.10 | |
| 1994 | WG | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 2.4 | GHE-P 4355 |
| | | | | | 6 | 0.75 | Foliar |
| | | | | | 9 | 0.45 | |
| | | | | | 15 | 0.25 | |
| | | | | | 21 | 0.15 | |
| 1994 | WG | 0.48 + 0.36 | 0.08 + 0.06 | 2 | 0 | 1.11 | GHE-P 4355 |
| | | | | | 6 | 0.39 | Foliar |
| | | | | | 9 | 0.20 | |
| | | | | | 15 | 0.12 | |
| | | | | | 21 | 0.05 | |
| 1994 | EC | 0.96 + 0.72 | 0.12 + 0.16 | 2 | 21 | 0.26 | GHE-P 4513 |
| | | | | | | | Foliar |
| 1994 | EC | 0.96 + 0.72 | 0.12 + 0.16 | 2 | 6 | 0.32 | GHE-P 4512 |
| | | | | | 12 | 0.22 | Foliar |
| | | | | | 15 | 0.28 | |
| | | | | | 21 | 0.02 | |

[1] Average of duplicate analyses
[2] LOQ 0.01 mg/kg

In supervised trials on cabbages in the USA in 1983 and 1984 multiple applications of chlorpyrifos 50WP were made at 1.12 kg ai/ha, alone or after EC applications of 0.84–2.1 kg ai/ha at planting. Samples were collected at random 14-15 and 21 days after the last application, and quartered

cabbage heads including the wrapper leaves analysed (Miller, *et al.*, 1986a). The results are shown in Table 58.

Table 58. Residues of chlorpyrifos in cabbages from supervised trials in the USA (foliar or at planting plus foliar applications).

| Location, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA | 480 EC | 2.5 | 1 | 30 | | At-seeding, incorporated; sidedress after transplant |
| | 500WP | 1.12 | 6 | 21 | | Broadcast foliar. |
| CA, 1983 | WP | 1.12 | 9 | 7 | 0.9 | GH-C 1788 |
| | | | | 14 | 0.05 | |
| | | | | 21 | <0.01 | |
| TX, 1983 | WP | 1.12 | 10 | 7 | 0.12 | GH-C 1788 |
| | | | | 14 | 0.4 | |
| | | | | 21 | 0.4 | |
| FL, 1984 | EC + WP | 1.5 + 1.1(x8) | 9 | 7 | 1.4 | GH-C 1788 |
| | | | | 14 | 0.51 | |
| | | | | 21 | 0.50 | |
| IL, 1983 | EC + WP | 1.5 + 1.1(x6) | 7 | 7 | 0.02 | GH-C 1788 |
| | | | | 14 | 0.04 | |
| | | | | 21 | <0.01 | |
| MA, 1983 | EC + WP | 0.8 + 1.1(x5) | 6 | 7 | 0.86 | GH-C 1788 |
| | | | | 14 | 0.33 | |
| | | | | 21 | 0.15 | |
| MI, 1983 | EC + WP | 1.5+ 1.1(x7) | 8 | 7 | 0.04 | GH-C 1788 |
| | | | | 14 | 0.07 | |
| | | | | 21 | <0.01 | |
| NJ, 1983 | EC + WP | 0.8 + 1.1(8) | 9 | 7 | 2.1 | GH-C 1788 |
| | | | | 14 | 2.8 | |
| | | | | 21 | 0.94 | |
| | | | | | | |
| FL, 1983 | EC + WP | 2.1 + 1.1(7) | 8 | 7 | 1.4 | GH-C 1788 |
| | | | | 14 | 0.23 | |
| | | | | 21 | 0.22 | |
| | | | | 7 | 0.12 | |
| | | | | 14 | 0.02 | |
| | | | | 21 | 0.03 | |
| | | | | 7 | 0.29 | |
| | | | | 14 | 0.01 | |
| | | | | 21 | 0.03 | |
| MI, 1983 | EC + WP | 1.2 + 1.1(9) | 10 | 7 | 1.4 | GH-C 1788 |
| | | | | 14 | 0.12 | |
| | | | | 21 | 0.26 | |
| | | | | 7 | 0.23 | |
| | | | | 14 | 0.02 | |
| | | | | 21 | 0.01 | |
| | | | | 7 | 0.22 | |

| Location, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| | | | | 14 | 0.03 | |
| | | | | 21 | 0.03 | |
| IL, 1983 | EC + WP | 1.7 + 1.1(9) | 10 | 7 | 1.0 | GH-C 1788 |
| | | | | 14 | 0.21 | |
| | | | | 21 | 0.22 | |
| | | | | 7 | 4.2 | |
| | | | | 14 | 0.88 | |
| | | | | 21 | 0.71 | |

[1] LOQ 0.01 mg/kg

Chinese cabbage. Three residue trials were conducted at different locations in the UK during 1993. A single spray of EC containing 480 g ai/l was applied at 0.16 kg ai/hl and 0.96 kg ai/ha to plants at growth stages between 5 and 18 leaves. The plants were harvested from 0 to 37 days after application. In two of the trials replicate samples were bulked in the field, and in the third analysed separately (Khoshab *et al.*, 1993f). The results are shown in Table 59.

Table 59. Residues of chlorpyrifos in Chinese cabbage from supervised trials in the UK.

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP- UK | EC | 0.96 | 0.16 | 2 | 21 | | Specified on label as brassica |
| Bray, Berkshire, 1993 | EC | 0.96 | 0.16 | 1 | 0 | 35, 39, 3, 34, 30 | GHE-P 3252 |
| | | | | | 8 | 2.6, 3.0, 3.3, 2.9 | |
| | | | | | 16 | 0.71, 0.50, 0.75, 0.56 | |
| | | | | | 24 | 0.19, 0.15, 0.15, 0.11 | |
| | | | | | 37 | ND, ND, ND, ND | |
| Bray, Berkshire, 1993 | EC | 0.96 | 0.16 | 1 | 0 | 31, 31, 36, 27 | GHE-P 3252<br>Replicate plot |
| | | | | | 8 | 1.0, 2.5, 2.6, 2.2 | |
| | | | | | 16 | 0.70, 0.47, 0.56, 0.52 | |
| | | | | | 24 | 0.17, 0.10, 0.10, 0.16 | |
| | | | | | 37 | <0.01, <0.01, <0.01, <0.01 | |
| Methwold Hythe, Thetford, 1993 | EC | 0.96 | 0.16 | 1 | 0 | 35 | GHE-P 3252 |
| | | | | | 6 | 5.4 | |
| | | | | | 12 | 1.8 | |
| | | | | | 18 | 0.60 | |
| | | | | | 24 | 0.18 | |
| Methwold Hythe, Thetford, 1993 | EC | 0.96 | 0.16 | 1 | 0 | 28 | GHE-P 3252<br>Replicate plot |
| | | | | | 6 | 4.2 | |
| | | | | | 12 | 1.4 | |
| | | | | | 18 | 0.34 | |
| | | | | | 24 | <0.01 | |
| Derbyshire, 1993 | EC | 0.96 | 0.16 | 1 | 0 | 64 | GHE-P 3252 |

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 7 | 0.87 | |
| | | | | | 14 | 0.12 | |
| | | | | | 21 | 0.04 | |
| | | | | | 28 | <0.01 | |
| Derbyshire, 1993 | EC | 0.96 | 0.16 | 1 | 0 | 67 | GHE-P 3252 Replicate plot |
| | | | | | 7 | 0.90 | |
| | | | | | 14 | 0.16 | |
| | | | | | 21 | 0.04 | |
| | | | | | 28 | <0.01 | |

[1] Average of two analyses
[2] LOQ 0.01 mg/kg

Cauliflower. All the ten supervised trials were carried out in the UK from 1992 to 1995. In two in 1992 single applications of EC formulation were sprayed on plants at the 10-15 cm or 20-30 cm height stages at 0.16 kg ai/hl and 0.96 kg ai/ha. The plants were harvested from 0 to 28 days after treatment (normal harvest). Whole plants taken up to 21 days after treatment were analysed, but the curd and the rest of plant were analysed separately in the samples taken at harvest (Khoshab *et al.*, 1993g).

In a trial in 1993 a single application of EC formulation was sprayed at growth stage 12-20 true leaves at 0.16 kg ai/hl, 0.96 kg ai/ha. Samples were harvested 21 and 34 days after application (Khoshab and Berryman, 1994d).

In another two trials in 1993, two applications of EC formulation were made at 0.89 and 0.48 kg ai/ha or 0.96 and 0.48 kg ai/ha at the 4-leaf and small curd growth stages. Samples were taken at harvest, 21 days after the last application (Khoshab and Berryman, 1994c).

In a further trial in 1994 two applications of EC or WG formulations were made to cabbages at 0.96 kg ai/ha and 0.08 kg ai/hl (1st application) and 0.72 kg ai/ha and 0.06 kg ai/hl (2nd). In a third trial the WG formulation was applied twice, firstly at 0.48 kg ai/ha and 0.08 kg ai/hl, then at 0.36 kg ai/ha and 0.06 kg ai/hl.

In trials in 1994 two applications of WG or EC formulations were made to plants at the 50-cm stage (no hearts visible) at 0.16 and 0.12 kg ai/hl, applied at 0.96 and 0.72 kg ai/ha respectively. In a third trial the WG formulation was applied at 0.08 kg ai/hl and 0.48 kg ai/ha (1st application) and 0.36 kg ai/ha 0.06 kg ai/hl (2nd) for the WG formulation. The plants were sampled at intervals up to harvest 22 days after treatment. On the day of harvest, leaves and curd were also analysed (Portwood and Williams, 1995d).

In another trial in 1994 two applications of EC were made to cauliflower plants post-emergence and 21 days before harvest, firstly at 0.96 kg ai/ha and then at 0.72 kg ai/ha. Whole plants were sampled at intervals up to 15 days, and curd and leaves at harvest 21 days after the second application (Khoshab, 1995c).

In 1995 trials two applications of chlorpyrifos EC or WG formulations were made to cauliflowers at crop growth stages BBCH 103 and 41 at 0.16 kg ai/hl and 0.12 kg ai/hl and applied at 0.96 kg and 0.72 kg ai/ha respectively. Plants were sampled at intervals from the day of final application to 36 days after treatment (normal harvest), when curd was analysed (Portwood and Williams, 1996l).

In a further trial in 1995 two applications of chlorpyrifos WG were made to cauliflower plants when hearts were 1 inch across at 0.16 and 0.12 kg ai/hl, applied at 0.96 kg ai/ha and 0.72 kg ai/ha respectively. The curds were sampled at harvest, 21 days after treatment (Portwood, 1996e).

The results are shown in Table 60.

Table 60. Residues of chlorpyrifos in cauliflowers from supervised trials in the UK (foliar applications).

| Year, sample | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-UK | 480 EC | 0.48 | (0. 24) | | 21 | | Conflicting information. |
| | | 0.72 | (0.12) | 1 | 21 | | |
| | | 0.96 | (0.16) | 2 | 21 | | Specified as brassica on label. |
| 1992 | 480 EC | 0.96 | 0.16 | 1 | 0 | 31 | GHE-P 3212 |
| Whole plant | | | | | 7 | 1.5 | |
| | | | | | 14 | 0.53 | |
| | | | | | 21 | 0.11 | |
| Plant except curd | | | | | 28 | 0.03 | |
| Curd | | | | | 28 | <0.01 | |
| 1992 | 480 EC | 0.96 | 0.16 | 1 | 0 | 54 | GHE-P 3212 |
| Whole plant | | | | | 7 | 0.82 | |
| | | | | | 14 | 0.04 | |
| | | | | | 22 | 0.01 | |
| 1993 | 480 EC | 0.96 | 0.16 | 1 | 21 | ≤0.01 | GHE-P 3633 |
| Curd | | | | | 34 | <0.01, <0.01, <0.01, <0.01 | |
| 1993 Curd | 480EC | 0.89 + 0.48 | 0.15 + 0.09 | 2 | 21 | <0.01, <0.01, <0.01, <0.01 | GHE-P 3506 |
| 1993 Curd | 480 EC | 0.96 + 0.48 | 0.16 + 0.09 | 2 | 21 | <0.01, <0.01, <0.01, <0.01 | GHE-P 3506 |
| 1994 Whole plant | 480 EC | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 6.1 | GHE-P 4354 |
| | | | | | 5 | 1.6 | |
| | | | | | 12 | 0.66 | |
| | | | | | 15 | 0.31 | |
| Leaves | | | | | 22 | 0.25 | |
| Curd | | | | | 22 | ≤0.01 | |
| 1994 Whole plant | WG | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 7.0 | GHE-P 4354 |
| | | | | | 5 | 1.3 | |
| | | | | | 12 | 0.62 | |
| | | | | | 15 | 0.31 | |
| Leaves | | | | | 22 | 0.17 | |
| Curd | | | | | 22 | ≤0.01 | |
| 1994 Whole plant | WG | 0.48 + 0.36 | 0.08 + 0.06 | 2 | 0 | 2.7 | GHE-P 4354 |
| | | | | | 5 | 0.67 | |
| | | | | | 12 | 0.21 | |
| | | | | | 15 | 0.14 | |
| Leaves | | | | | 22 | 0.10 | |
| Curd | | | | | 22 | <0.01 | |

| Year, sample | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| 1994 Whole plant | EC | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 13 | GHE-P 4511 |
| | | | | | 6 | 1.5 | |
| | | | | | 12 | 0.47 | |
| | | | | | 15 | 0.27 | |
| Leaves | | | | | 21 | 0.33 | |
| Curd | | | | | 21 | 0.01 | |
| 1995 Whole plant | EC | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 12 | GHE-P 5450 |
| | | | | | 3 | 5.5 | |
| | | | | | 9 | 1.1 | |
| | | | | | 16 | 0.23 | |
| Curd | | | | | 36 | <0.01 | |
| 1995 Whole plant | WG | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 10 | GHE-P 5450 |
| | | | | | 3 | 4.8 | |
| | | | | | 9 | 2.1 | |
| | | | | | 16 | 0.24 | |
| Curd | | | | | 36 | <0.01 | |
| 1995 Whole plant | WG | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 12 | GHE-P 5450 |
| | | | | | 3 | 5.5 | |
| | | | | | 9 | 1.1 | |
| | | | | | 16 | 0.42 | |
| Curd | | | | | 36 | <0.01 | |
| 1995 Whole plant | WG | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 14 | GHE-P 5450 |
| | | | | | 3 | 5.7 | |
| | | | | | 9 | 1.7 | |
| | | | | | 16 | 0.28 | |
| Curd | | | | | 36 | <0.01 | |
| 1995 Whole plant | WG | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 14 | GHE-P 5450 |
| | | | | | 3 | 5.5 | |
| | | | | | 9 | 1.43 | |
| | | | | | 16 | 0.31 | |
| Curd | | | | | 36 | <0.01 | |
| 1995 Whole plant | WG | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 0 | 17 | GHE-P 5450 |
| | | | | | 3 | 2.3 | |
| | | | | | 9 | 0.37 | |
| | | | | | 16 | 0.18 | |
| Curd | | | | | 36 | <0.01 | |
| 1995 Curd | WG | 0.96 + 0.72 | 0.16 + 0.12 | 2 | 21 | 0.02 | GHE-P 5451 |

[1] Average of two determinations
[2] LOQ 0.01 mg/kg

Fruiting vegetables, other than cucurbits

Peppers. In three trials in Spain in 1988 EC (480 g ai/l) was applied twice, at 1.15 kg ai/ha and 1.44 kg ai/ha at a concentration of 0.096 kg ai/hl. Samples were harvested at intervals up to 28 days after the final application. Bulk samples were prepared by combining replicate samples (Osborne, 1989b).

The results are shown in Table 61.

Table 61. Residues of chlorpyrifos in peppers from supervised trials in Spain.

| Year | Application | | | | PHI, days | Residues, mg/kg [1] | |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | Reference |
| GAP-Spain | 480 EC | (1.15) | 0.096 | 3 | 7 | | |
| | 250 WP | (1.2) | 0.1 | 2 | 7 | | |
| 1988 | EC | 1.15 + 1.44 | 0.096 | 2 | 4 | 0.28 [2] | GHE-P 1990 |
| | | | | | 7 | 0.37 [2] | |
| | | | | | 14 | 0.30 [2] | |
| | | | | | 21 | 0.36 [2] | |
| | | | | | 28 | 0.30 [2] | |
| 1988 | EC | 1.06 + 1.25 | 0.096 | 2 | 4 | 0.52, 0.53, 0.32, 0.26 | GHE-P 1990 |
| | | | | | 7 | 0.37, 0.47 | |
| | | | | | 14 | 0.22, 0.19, 0.11, 0.13 | |
| | | | | | 22 | 0.37, 0.33, 0.20, 0.14 | |
| | | | | | 28 | 0.35, 0.24 | |
| 1988 | EC | 1.15 + 1.34 | 0.096 | 2 | 7 | 0.45 [2] | GHE-P 1990 |
| | | | | | 14 | 0.19 [2] | |
| | | | | | 21 | 0.12 [2] | |
| | | | | | 28 | 0.06 [2] | |

[1] LOQ 0.01 mg/kg
[2] Average of two analyses

Several supervised trials on bell peppers were conducted in seven US States during 1982-1984, with the WP formulation applied as a multiple foliar spray at 10 -14 day intervals, at 1.12 kg ai/ha. Samples were collected 7, 14 and 21 days after the final application (Miller *et al.*, 1985b). The results are shown in Table 62.

Table 62. Residues of chlorpyrifos in bell peppers from supervised trials in the USA.

| Location, year (Variety) | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-US | WP | 1.12 | (0.23) | 8 | 7 | | |
| CA, 1983 | WP | 1.12 | 0.17 | 7 | 7 | 0.01 | GH-C 1757 |
| (Serrano) | | | | | 14 | 0.04 | |
| | | | | | 21 | 0.01 | |
| FL, 1983 | WP | 1.12 | 0.16 | 5 | 7 | 0.27 | GH-C 1757 |
| (Hungarian Wax) | | | | | 14 | 0.01 | |
| | | | | | 21 | 0.01 | |
| FL, 1983 | WP | 1.12 | | 8 | 7 | 0.60 | GH-C 1757 |
| (Jalapeño) | | | | | 14 | 0.13 | |
| | | | | | 21 | 0.02 | |
| MI, 1983 | WP | 1.12 | | 7 | 7 | 0.39 | GH-C 1757 |
| (Small fry) | | | | | 14 | 0.15 | |
| | | | | | 21 | 0.02 | |
| MS, 1983 | WP | 1.12 | | 10 | 7 | 1.4 | GH-C 1757 |

| Location, year (Variety) | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| (Red Cayenne)) | | | | | 14 | 0.90 | |
| | | | | | 21 | 0.27 | |
| NJ, 1983 | WP | 1.12 | | 8 | 7 | 0.40 | GH-C 1757 |
| (Vineland Special) | | | | | 14 | 0.14 | |
| | | | | | 21 | 0.16 | |
| NY, 1983 | WP | 1.12 | | 8 | 7 | 0.52 | GH-C 1757 |
| (Hungarian Wax) | | | | | 14 | 0.16 | |
| | | | | | 21 | 0.13 | |
| OR, 1983 | WP | 1.12 | | 8 | 7 | 0.13 | GH-C 1757 |
| (Anaheim) | | | | | 14 | 0.14 | |
| | | | | | 21 | 0.04 | |
| CA, 1983 | WP | 1.12 | | 10 | 7 | 0.23, 0.27 | GH-C 1757 |
| (Sweet Pimiento) | | | | | 14 | 0.22, 0.18 | |
| | | | | | 21 | 0.10, 0.09 | |
| CA, 1983 | WP | 1.12 | | 7 | 7 | 0.10 | GH-C 1757 |
| (Bell) | | | | | 14 | 0.05 | |
| | | | | | 21 | 0.01 | |
| FL, 1983 | WP | 1.12 | | 10 | 0 | 0.27, 0.13, 0.12, 0.10 | GH-C 1757 |
| (Early CA Wonder) | | | | | 7 | 0.13, 0.07, 0.10, 0.08 | |
| | | | | | 14 | 0.13, 0.08, 0.06, 0.03 | |
| | | | | | 21 | 0.04, 0.02, 0.04, 0.02 | |
| Fl, 1983 | WP | 1.12 | | 10 | 0 | 0.89, 0.78, 0.90, 0.63 | GH-C 1757 |
| (Early CA Wonder) | | | | | 7 | 0.47, 0.52, 0.60, 0.35 | |
| | | | | | 14 | 0.33, 0.47, 0.33, 0.33 | |
| | | | | | 21 | 0.38, 0.32, 0.19, 0.10 | |
| MS, 1984 | WP | 1.12 | | 10 | 7 | 0.06 | GH-C 1757 |
| (Yolo Wonder) | | | | | 14 | 0.02 | |
| | | | | | 21 | 0.01 | |
| NJ, 1983 | WP | 1.12 | | 10 | 7 | 0.81 | GH-C 1757 |
| (Yolo Wonder L) | | | | | 14 | 0.43 | |
| | | | | | 21 | 0.18 | |
| NY, 1982 | WP | 1.12 | | 8 | 8 | 0.30 | GH-C 1757 |
| (Skipper) | | | | | 15 | 0.12 | |
| | | | | | 22 | 0.10 | |
| OR, 1983 | WP | 1.12 | | 8 | 7 | 0.48 | GH-C 1757 |
| (California Wonder) | | | | | 14 | 0.39 | |
| | | | | | 21 | 0.16 | |
| CA, Davis, 1983 | WP | 1.12 | | 7 | 7 | 0.04, 0.10 | GH-C 1757 |
| (Yolo Wonder) | | | | | 14 | 0.02, 0.07 | |
| | | | | | 21 | 0.01, 0.01 | |

[1] LOQ 0.01 mg/kg

Tomatoes. Supervised trials were carried out in Australia, Brazil, Mexico, Spain and the USA from 1975 to 1995.

In a trial in Brazil, tomatoes were treated 6 times at 0.72 or 1.4 kg ai/ha with an EC formulation and harvested 3-21 days after the final application (Pinto and Gagnotto, 1994).

Tomatoes in Mexico were treated three times with EC at 0.96 kg ai/ha at weekly intervals by ground and aerial applications and samples were taken one day after each application and 3, 7 and 14 days after the last application (Miller and Ervick, 1976).

In trials in Spain in 1995 the WG 750 or EC 480 formulation was applied once to tomatoes 7 days before normal harvest at 0.065 kg ai/hl and 0.695 kg ai/ha and samples taken 0 to 7 days after treatment (Portwood, 1996c).

Table 63. Residues of chlorpyrifos in tomatoes after foliar application of an EC Formulation in Brazil, Mexico and Spain.

| Location, (Variety) Year | Application | | | PHI, days | Chlorpyrifos, mg/kg[1] | Reference/comment |
|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | | |
| GAP- Brazil | 0.72 | | 7 | 21 | | 480 g/l EC |
| Mogi Mirim (Peixe) 1992 | 0.72 | | 6 | 3<br>7<br>14<br>21 | 0.18, 0.30, 0.24<br>0.12, 0.09, 0.18<br>0.03, 0.03, 0.02<br>0.02, 0.03, 0.01 | GHB-P 159. 300l/ha for first two applications; 900 l/ha for last 4 applications. |
| | 1.4 | | 6 | 21 | 0.03, 0.03, 0.06 | |
| GAP- Mexico | 1.0<br>0.96 | | | 1<br>1 | | 500 WP<br>480 g/l EC |
| Mexico, 1976 | 0.96 | | 1<br>2<br>3<br>3<br>3<br>3 | 1<br>1<br>1<br>3<br>7<br>14 | 0.01, 0.01, 0.01, <0.01<br>0.02, 0.01, 0.01, 0.01<br>0.02, 0.06, 0.04, 0.01<br>0.01, 0.02, 0.01, 0.01<br>0.01, 0.02, 0.01, 0.01<br>0.01, 0.01, 0.01, <0.01 | GH-C 952<br>EC |
| Mexico, 1976 | 0.96 | | 1<br>2<br>3<br>3<br>3<br>3 | 1<br>1<br>1<br>3<br>8<br>15 | 0.07, 0.19, 0.16, 0.13<br>0.12, 0.08, 0.15, 0.19<br>0.17, 0.11, 0.03, 0.13<br>0.07, 0.07, 0.07, 0.06<br>0.04, 0.02, 0.03.0.04<br>0.05, 0.03, 0.04, 0.02 | GH-C 952<br>EC |
| Mexico, 1976 | 0.96 | | 1<br>2<br>3<br>3<br>3<br>3 | 1<br>1<br>1<br>3<br>8<br>15 | 0.13, 0.12, 0.08, 0.22<br>0.13, 0.14, 0.33, 0.13<br>0.24, 0.08, 0.20, 0.07<br>0.10, 0.16, 0.12, 0.15<br>0.06, 0.17, 0.03, 0.03<br>0.23, 0.04, 0.03, 0.02 | GH-C 952<br>EC |
| GAP- Spain | (1.2)<br>(1.2) | 0.10<br>0.096 | 2<br>3 | 7<br>7 | | WP 250g/kg<br>EC 480 g/l |
| Spain, 1995 | | 0.065 | 1 | 0<br>3<br>5<br>7 | 0.27[2]<br>0.11[2]<br>0.09[2]<br>0.06[2] | GHE-P 5495<br>WG |
| Spain, 1995 | | 0.065 | 1 | 0<br>3<br>5<br>7 | 0.27[2]<br>0.12[2]<br>0.12[2]<br>0.08[2] | GHE-P 5495<br>EC |

[1] LOQ 0.01 mg/kg in all trials. Recoveries averaged 94% from the Mexican trials, 92% from the Brazilian trials and 103% from the Spanish trials.
[2] Average of duplicate analyses

.

Two trials in Australia (1975) were at 0.05 kg ai/hl and 0.10 kg ai/hl, each in triplicate. The plants received seven treatments. Tomato samples were taken at intervals up to 21 days after the last treatment (Tucker, 1975).

Four decline trials were carried out during 1978 in South Africa, with single applications at rates of 0.048, 0.072, 0.096 and 0.192 kg ai/hl and samples taken at intervals up to 29 days after treatment (Iosson, 1979).

Table 64. Residues of chlorpyrifos in tomatoes from supervised trials in Australia and South Africa.

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Australia | 500 EC | (1.0) | 0.10 | 7-10 | 3 | | Foliar |
| Australia, 1975 | EC | | 0.05 | 7 | 0 | 0.20, 0.23, 0.26 | GHF-P 030 |
| | | | | | 1 | 0.07, 0.10, 0.05 | |
| | | | | | 3 | 0.01, 0.02, 0.01 | |
| | | | | | 10 | <0.01, <0.01, <0.01 | |
| | | | | | 14 | <0.01, <0.01, <0.01 | |
| | | | | | 21 | <0.01, <0.01, <0.01 | |
| Australia, 1975 | EC | | 0.10 | 7 | 0 | 0.25, 0.37, 0.27 | GHF-P 030 |
| | | | | | 1 | 0.13, 0.24, 0.26 | |
| | | | | | 3 | 0.12, 0.13, 0.06 | |
| | | | | | 10 | 0.02, 0.02, 0.01 | |
| | | | | | 14 | <0.01, <0.01, <0.01 | |
| | | | | | 21 | <0.01, <0.01, <0.01 | |
| GAP-S. Africa | EC | | 0.096 | multiple | 4 | | |
| South Africa, | EC | | 0.048 | 1 | 0 | 0.12 [3] | GHE-P 664 |
| 1978 | | | | | 1 | 0.10 [3] | |
| | | | | | 4 | 0.12 [3] | |
| | | | | | 7 | 0.06 [3] | |
| | | | | | 14 | 0.05 [3] | |
| | | | | | 20 | 0.03 [3] | |
| | | | | | 29 | <0.01 [3] | |
| South Africa, | EC | | 0.072 | 1 | 0 | 0.22 [3] | GHE-P 664 |
| 1978 | | | | | 1 | 0.18 [3] | |
| | | | | | 4 | 0.19 [3] | |
| | | | | | 7 | 0.23 [3] | |
| | | | | | 14 | 0.07 [3] | |
| | | | | | 20 | 0.01 [3] | |
| | | | | | 29 | 0.01 [3] | |
| South Africa, | EC | | 0.096 | 1 | 0 | 0.16 [3] | GHE-P 664 |
| 1978 | | | | | 1 | 0.24 [3] | |
| | | | | | 4 | 0.19 [3] | |
| | | | | | 7 | 0.23 [3] | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 14 | 0.10 [3] | |
| | | | | | 20 | 0.03 [3] | |
| | | | | | 29 | 0.02 [3] | |
| South Africa, | EC | | 0.192 | 1 | 0 | 0.75 [3] | GHE-P 664 |
| 1978 | | | | | 1 | 1.1 [3] | |
| | | | | | 4 | 0.89 [3] | |
| | | | | | 7 | 0.59 [3] | |
| | | | | | 14 | 0.26 [3] | |
| | | | | | 20 | 0.06 [3] | |
| | | | | | 29 | 0.05 [3] | |

[1] LOQ 0.01 mg/kg
[2] Recoveries averaged 96% from the Australian trials and 97% from the South African trials
[3] Average of duplicate analyses

Chlorpyrifos EC was applied as a directed ground application in two trials in the USA (1979) at 1.12 kg ai/ha. Six or seven applications were made following a weekly or biweekly schedule during the fruiting season and samples were collected 3, 7 and 14 days after the last application (Miller, 1980b).

In another US trial in 1982 at 12 locations ten weekly applications of EC formulation were made to tomatoes during the vegetative and reproductive stages at 1.12 kg ai/ha and samples taken 0, 7, 14 and 21 days after the last application (Miller, 1983a).

In thirteen trials in seven US States in 1983 the 50W or 4E formulation was applied to the tomatoes at 1.12 kg ai/ha. The first application was made after transplant and the subsequent 7-12 sprays were applied at 10-14 day intervals with samples taken 7, 14 and 21 days after the last application (Miller, 1985b).

Table 65. Residues of chlorpyrifos in tomatoes from supervised trials in the USA.

| Location, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA<br>NONE | | | | | | |
| GAP-Mexico[2] | EC 480 g/l<br>WP 500 g/kg | 0.96<br>1.0 | | 1<br>1 | | |
| GA, 1979 | EC | 1.12 | 7 | 0 | 0.11, 0.22, 0.20, 0.15 | GH-C 1372 |
| | | | | 7 | 0.10, 0.06, 0.09, 0.10 | Directed ground |
| | | | | 14 | 0.03, 0.02, 0.03, 0.02 | |
| OH, 1979 | EC | 1.12 | 6 | 0 | 0.14, 0.23,, | GH-C 1372 |
| | | | | 7 | 0.12, 0.17, 0.21, 0.13 | Directed ground |
| | | | | 14 | 0.14, 0.13, 0.14, 0.09 | |
| FL, 1982 | EC | 1.12 | 10 | 0 | 0.08 | GH-C 1641 |
| Floradale) | | | | 7 | 0.24 | Foliar |
| | | | | 14 | 0.03 | |
| | | | | 21 | 0.03 | |
| FL, 1982 | EC | 1.12 | 9 | 0 | 0.17 | GH-C 1641 |

| Location, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| (Burgis) | | | | 7 | 0.3 | Foliar |
| | | | | 14 | 0.02 | |
| | | | | 21 | 0.04 | |
| FL, 1982 | EC | 1.12 | 10 | 0 | 0.10 | GH-C 1641 |
| (Floradale) | | | | 7 | 0.19 | Foliar |
| | | | | 14 | 0.02 | |
| | | | | 21 | 0.05 | |
| MI, 1982 | EC | 1.12 | 10 | 0 | 0.14 | GH-C 1641 |
| (G-465) | | | | 7 | 0.09 | Foliar |
| | | | | 14 | 0.11 | |
| | | | | 21 | 0.02 | |
| SC, 1982 | EC | 1.12 | 10 | 0 | 0.43 | GH-C 1641 |
| (Floradale) | | | | 7 | 0.11 | Foliar |
| | | | | 14 | 0.10 | |
| | | | | 21 | 0.18 | |
| IL, 1982 | EC | 1.12 | 10 | 0 | 0.41 | GH-C 1641 |
| (Campbell C-28) | | | | 7 | 0.24 | Foliar |
| | | | | 14 | 0.31 | |
| | | | | 21 | 0.22 | |
| IN, 1982 | EC | 1.12 | 10 | 0 | 0.24 | GH-C 1641 |
| (Campbell C-28) | | | | 7 | 0.14 | Foliar |
| | | | | 14 | 0.06 | |
| | | | | 21 | 0.09 | |
| CA, 1982 | EC | 1.12 | 10 | 0 | 0.49 | GH-C 1641 |
| (Ace) | | | | 7 | 0.07 | Foliar |
| | | | | 14 | 0.06 | |
| | | | | 21 | 0.02 | |
| NY, 1982 | EC | 1.12 | 10 | 0 | 0.70 | GH-C 1641 |
| (Jet Star) | | | | 7 | 0.81 | Foliar |
| | | | | 14 | 0.56 | |
| | | | | 21 | 0.36 | |
| PA, 1982 | EC | 1.12 | 10 | 0 | 1.5 | GH-C 1641 |
| (Floradale) | | | | 7 | 0.82 | Foliar |
| | | | | 14 | 0.84 | |
| | | | | 21 | 0.30 | |
| GA, 1982 | EC | 1.12 | 10 | 0 | 0.57 | GH-C 1641 |
| (Walter) | | | | 7 | 0.08 | Foliar |
| | | | | 14 | 0.25 | |
| MA, 1982 | EC | 1.12 | 10 | 0 | 0.28 | GH-C 1641 |
| (Jet Star) | | | | 1 | 0.12 | Foliar |
| | | | | 7 | 0.04 | |
| | | | | 14 | 0.03 | |
| | | | | 21 | 0.02 | |
| FL, 1983 | WP | 1.12 | 8 | 7 | 0.12 | GH-C 1757 |

| Location, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| (Floradale) | | | | 14 | 0.06 | Foliar |
| | | | | 21 | 0.08 | |
| IL, 1983 | WP | 1.12 | 10 | 7 | 0.02 | GH-C 1757 |
| (Golden Boy) | | | | 14 | 0.01 | Foliar |
| | | | | 21 | 0.02 | |
| MI, 1983 | WP | 1.12 | 9 | 7 | 0.01 | GH-C 1757 |
| (Glamour) | | | | 14 | 0.01 | Foliar |
| | | | | 21 | <0.01 | |
| MS, 1984 | WP | 1.12 | 10 | 7 | 0.05 | GH-C 1757 |
| (Floradale) | | | | 14 | 0.04 | Foliar |
| | | | | 21 | 0.01 | |
| NJ, 1983 | WP | 1.12 | 10 | 7 | 0.32 | GH-C 1757 |
| (1327) | | | | 14 | 0.05 | Foliar |
| | | | | 21 | 0.02 | |
| OR, 1983 | WP | 1.12 | 10 | 7 | 0.05 | GH-C 1757 |
| (Willamette) | | | | 14 | 0.07 | Foliar |
| | | | | 21 | 0.01 | |
| CA, 1983 | WP | 1.12 | 10 | 7 | 0.02 | GH-C 1757 |
| (Ace 55) | | | | 14 | 0.02 | Foliar |
| | | | | 21 | 0.01 | |
| CA, 1983 | EC (4E) | 1.12 | 10 | 7 | 0.10 | GH-C 1757 |
| (Ace 55) | | | | 14 | 0.10 | Foliar |
| | | | | 21 | 0.06 | |
| CA, 1983 | EC (1E) | 1.12 | 10 | 7 | 0.11 | GH-C 1757 |
| (Ace 55) | | | | 14 | 0.04 | Foliar |
| | | | | 21 | 0.01 | |
| FL, 1983 | WP | 1.12 | 9 | 7 | 0.12 | GH-C 1757 |
| (Sunny) | | | | 14 | 0.03 | Foliar |
| | | | | 21 | 0.03 | |
| FL, 1983 | EC (1E) | 1.12 | 9 | 7 | 0.11 | GH-C 1757 |
| (Sunny) | | | | 14 | 0.05 | foliar |
| | | | | 21 | 0.02 | |
| MI, 1983 | WP | 1.12 | 7 | 7 | 0.04 | GH-C 1757 |
| (Pik Red) | | | | 14 | 0.10 | Foliar |
| | | | | 21 | 0.02 | |
| MI, 1983 | EC(1E) | 1.12 | 7 | 7 | 0.39 | GH-C 1757 |
| (Pik Red) | | | | 14 | 0.14 | Foliar |
| | | | | 21 | 0.01 | |

[1] LOQ 0.01 mg/kg
[2] None of the trials were in geographic zones corresponding to those of Mexico

Egg plant. In a trial in 1972 in Turkey chlorpyrifos was applied as a directed spray to egg plants and other fruiting vegetables at 0.96 kg ai/ha. Samples were collected 0, 7 and 14 days after application. The results for egg plants are shown in Table 66 (Hollick and Collison, 1972).

Table 66. Residues of chlorpyrifos in egg plant from supervised trials in Turkey.

| Year | Application | | | | PHI, days | Residues, mg/kg | References |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Turkey NONE | | | | | | | |
| 1972 | EC | 0.96 | | 3 | 0 | 0.33 | GHE-P 101 |
| | | | | | 7 | 0.03 | |
| | | | | | 14 | 0.02 | |

Leafy vegetables

Lettuce. Two trials were reported from Spain. Immature head lettuce at the 6-8 leaf stage were treated with an EC formulation (Butcher and Teasdale, 1990).

Table 67. Residues of chlorpyrifos and TCP in head lettuce from the foliar application of an EC formulation in Spain.

| Location, (Variety) Year | Application | | | PHI, days | Residue, mg/kg | | Reference |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | CP | TCP | |
| GAP- Spain | (0.3) | 0.10 | | 21 | | | |
| S Jaume D'enveja, Spain (Eugenia) 1989 | 0.48 | | 2 | 19<br>25 | 0.10<br><0.02 | 0.25<br><0.05 | GHE-P 2194 |
| Amposta, Spain (Ruver) 1989 | 0.48 | | 2 | 19<br>25 | 0.23<br>0.08 | 0.22<br>0.13 | |

Several supervised trials in the USA were carried out to establish MRLs for head and leaf lettuce. Proposed GAP is eight applications as a directed spray at 1.12 kg ai/ha and a PHI of 21 days, the first application to be made within three weeks of planting and subsequent applications at 4- to 14-day intervals (Woods, 1984).

The results are shown in Tables 68 and 69.

Table 68. Residues of chlorpyrifos in head lettuce from supervised trials in the USA.

| Location, year (Variety) | Application | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA NONE | | | | | | |
| CA, 1983 | WP | 1.12 | 7 | 7 | 1.8 | GH-C 1696 |
| | | | | 14 | 0.20 | |
| | | | | 21 | 0.02 | |
| CA, 1983 | EC (4E) | 1.12 | 8 | 7 | 0.63 | GH-C 1696 |
| (Great Lakes) | | | | 14 | 0.42 | |
| | | | | 21 | 0.27 | |
| CA, 1983 | WP | 1.12 | 8 | 7 | 0.53 | GH-C 1696 |

| Location, year (Variety) | Application | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| (Great lakes) | | | | 14 | 0.77 | |
| | | | | 21 | 0.33 | |
| CA, 1983 | WP | 1.12 | 6 | 7 | 0.12, 0.09, 0.10, 0.17 | GH-C 1696 |
| (Iceberg) | | | | 13 | 0.03, 0.08, 0.05, 0.01 | |
| | | | | 21 | 0.03, 0.01, 0.01, 0.06 | |
| CA, 1982 | WP | 1.12 | 10 | 7 | <0.01, 0.01, 0.03, 0.05 | GH-C 1696 |
| (Iceberg) | | | | 14 | 0.02, 0.04, 0.03, 0.03 | |
| | | | | 21 | <0.01, 0.01, 0.01, 0.02 | |
| FL, 1982 | WP | 1.12 | 4 | 7 | 2.6, 1.2, 1.6, 3.7 | GH-C 1696 |
| (Great Lakes) | | | | 14 | 0.03, 0.05, 0.40, 0.07 | |
| | | | | 21 | 0.01, 0.01, 0.01, 0.01 | |
| FL, 1983 | EC (4E) | 1.12 | 7 | 7 | 0.35 | GH-C 1696 |
| (Mesa 654) | | | | 14 | 0.25 | |
| | | | | 21 | 0.13 | |
| FL, 1983 | WP | 1.12 | 7 | 7 | 0.66 | GH-C 1696 |
| (Mesa 654) | | | | 14 | 0.32 | |
| | | | | 21 | 0.15 | |
| FL, 1983 | 1 E | 1.12 | 7 | 7 | 0.53 | GH-C 1696 |
| (Mesa 654) | | | | 14 | 0.13 | |
| | | | | 21 | 0.12 | |
| FL, 1983 | WP | 1.12 | 5 | 7 | 0.55 | GH-C 1696 |
| (Iceberg) | | | | 14 | 0.02 | |
| | | | | 21 | <0.01 | |
| IL, 1983 | WP | 1.12 | 5 | 7 | 0.01 | GH-C 1696 |
| (Iceberg) | | | | 14 | 0.01 | |
| | | | | 21 | 0.01 | |
| MI, 1983 | WP | 1.12 | 5 | 7 | 0.49 | GH-C 1696 |
| | | | | 14 | 0.36 | |
| | | | | 22 | 0.03 | |
| MI, 1983 | EC (4E) | 1.12 | 7 | 7 | 0.12 | GH-C 1696 |
| (Ithaca) | | | | 14 | 0.05 | |
| | | | | 21 | 0.04 | |
| MI, 1983 | WP | 1.12 | 7 | 7 | 0.14 | GH-C 1696 |
| | | | | 14 | 0.04 | |
| | | | | 21 | 0.06 | |
| MI, 1983 | 1 E | 1.12 | 7 | 7 | 0.15 | GH-C 1696 |
| | | | | 14 | 0.03 | |
| | | | | 21 | 0.06 | |
| OR, 1983 | WP | 1.12 | 8 | 7 | 0.17 | GH-C 1696 |
| (Great Lakes) | | | | 14 | 0.33 | |
| | | | | 21 | 0.09 | |

[1] LOQ 0.01 mg/kg

Table 69. Residues of chlorpyrifos in leaf lettuce from supervised trials in the USA.

| Location, year (Variety) | Application | | | PHI, days | Residues, mg/kg[1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |

| Location, year (Variety) | Application | | | PHI, days | Residues, mg/kg[1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA NONE | | | | | | |
| CA, 1983 | WP | 1.12 | 7 | 7 | 0.57 | GH-C 1696 |
| | | | | 14 | 0.06 | |
| | | | | 21 | 0.07 | |
| CA, 1983 | EC | 1.12 | 8 | 7 | 3.6 | GH-C 1696 |
| (Royal Oak Leaf) | | | | 14 | 0.32 | |
| | | | | 21 | 0.84 | |
| CA, 1983 | WP | 1.12 | 8 | 7 | 2.7 | GH-C 1696 |
| (Royal Oak Leaf) | | | | 14 | 0.81 | |
| | | | | 21 | 0.64 | |
| CA, 1983 | WP | 1.12 | 5 | 9 | 0.43 | GH-C 1696 |
| (Paris Island) | | | | 14 | 0.19 | |
| | | | | 21 | 0.03 | |
| FL, 1983 | WP | 1.12 | 5 | 7 | 0.95 | GH-C 1696 |
| (Valmaine) | | | | 14 | 0.35 | |
| | | | | 21 | 0.07 | |
| FL, 1983 | WP | 1.12 | 5 | 7 | 5.6 | GH-C 1696 |
| (Boston) | | | | 14 | 0.29 | |
| | | | | 21 | 0.09 | |
| IL, 1983 | WP | 1.12 | 5 | 7 | 1.8 | GH-C 1696 |
| (Black Seeded Simpson) | | | | 14 | 0.05 | |
| | | | | 21 | 0.02 | |
| MA, 1983 | WP | 1.12 | 4 | 7 | 7.0 | GH-C 1696 |
| (Salad Bowl) | | | | 14 | 0.01 | |
| | | | | 21 | 0.07 | |
| MI, 1983 | WP | 1.12 | 5 | 7 | 1.1 | GH-C 1696 |
| Leaf Lettuce | | | | 14 | 0.17 | |
| | | | | 21 | 0.04 | |
| MI, 1983 | WP | 1.12 | 5 | 7 | 0.08 | GH-C 1696 |
| (Grand Rapids) | | | | 14 | 0.17 | |
| | | | | 22 | 0.01 | |
| OR, 1983 | WP | 1.12 | 6 | 7 | 2.2 | GH-C 1696 |
| (Chicken) | | | | 14 | 0.06 | |
| | | | | 21 | 0.02 | |
| TX, 1983 | WP | 1.12 | 9 | 7 | 0.42 | GH-C 1696 |
| (Black Seeded Simpson) | | | | 14 | 0.11 | |
| | | | | 21 | 0.05 | |

[1] LOQ 0.01 mg/kg

Legumes

Common beans. Supervised trials were conducted in Italy from 1992 to 1994 and in the USA from 1969 to 1972. The results are shown in Tables 70 and 71 respectively.

In three residue trials at different locations in Italy single applications of EC (228 g ai/l) was made to field bean plants at the flowering stage at a spray concentration of 0.14 kg ai/hl, 0.57 kg ai/ha.

Whole plant samples were taken at intervals for 9 days. Beans with pods and the rest of the plant were sampled separately at normal harvest after 15 days (Khoshab *et al.*, 1993h).

In a trial in Italy in 1994 single applications of EC (480 g ai/l) were sprayed on field bean plants at flowering stage at 0.57 kg ai/ha, 0.14 kg ai/hl. Whole plants were sampled 0, 5, 10 and 14 days after application, and pods and the rest of the plant separately at normal harvest 20 days after application (Khoshab, 1995d).

Table 70. Residues of chlorpyrifos in common beans from supervised trials in Italy.

| Year, sample | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Italy | EC | 0. 53 | (0.09) | | 15 | | |
| 1992 | EC | 0.57 | 0.14 | 1 | 0 | 23 | GHE-P 3231 |
| whole plant | | | | | 5 | 2.8 | |
| whole plant | | | | | 9 | 1.3 | |
| beans with pods | | | | | 15 | 0.05 | |
| rest of plant | | | | | 15 | 0.53 | |
| 1992 | EC | 0.57 | 0.14 | 1 | 0 | 45 | GHE-P 3231 |
| whole plant | | | | | 6 | 1.6 | |
| whole plant | | | | | 10 | 0.52 | |
| beans with pods | | | | | 15 | ≤0.01 | |
| rest of plant | | | | | 15 | 0.09 | |
| 1992 | EC | 0.57 | 0.14 | 1 | 0 | 35 | GHE-P 3231 |
| whole plant | | | | | 4 | 5.4 | |
| whole plant | | | | | 10 | 0.78 | |
| beans with pods | | | | | 15 | ≤0.01 | |
| rest of plant | | | | | 15 | 0.14 | |
| 1994 | EC | 0.57 | 0.14 | 1 | 0 | 20 | GHE-P 4515 |
| whole plant | | | | | 5 | 2.3 | |
| whole plant | | | | | 10 | 0.59 | |
| whole plant | | | | | 14 | 0.15 | |
| pods | | | | | 20 | <0.01 | |
| rest of plant | | | | | 20 | 0.04 | |

[1] Average of duplicate analyses
[2] LOQ 0.01 mg/kg

In trials in several US States in 1969-1972, snap bean seeds were treated before planting with WP slurries containing 25% or 50% ai, at 0.63 to 1.9 g ai/kg seeds. Green plant samples were collected at various stages of growth, and beans at harvest (McKellar and Dishburger, 1973).

Similar trials were carried out on field and kidney beans during 1972-1976. The seeds were treated with chlorpyrifos at rates up to 2.5 g ai/kg of seeds before planting. Beans, pods and vines were collected at normal harvest time, and whole plants 4 and 6 weeks after planting (Norton, 1978).

The results are shown in Table 71.

Table 71. Residues of chlorpyrifos from seed treatment of common beans in supervised trials in the USA.

| Location, Year, Sample | Application | | | PHI, days | Residues, mg/kg [1,2] | Reference/ Comments |
|---|---|---|---|---|---|---|
| | Form. | g ai/ kg | No. | | | |
| GAP-USA | WP (SL) | 0.62 | 1 | NA | | Seed treatment |
| MS, 1972 | | | | | | |
| Kidney beans, shelled | WP | 1.9 | 1 | 93 | <0.01, 0.01, 0.01, <0.01 | GH-C 1157 |
| Kidney bean pods | WP | 1.9 | 1 | 93 | 0.01, 0.01, 0.02, 0.01 | |
| Kidney bean vines | WP | 1.9 | 1 | 93 | 0.02, 0.02, 0.02, 0.01 | |
| IL, 1973 | | | | | | |
| Kidney beans & pods | WP | 1.9 | 1 | 117 | 0.01, 0.01, 0.01 | GH-C 1157 |
| Kidney bean vines | WP | 1.9 | 1 | 117 | 0.01, 0.01, 0.01 | |
| MS, 1973 | | | | | | |
| Kidney beans & pods | WP | 1.9 | 1 | 106 | 0.03, 0.02, 0.08 | GH-C 1157 |
| Kidney bean vines | WP | 1.9 | 1 | 106 | 0.07, 0.04, 0.06 | |
| NY, 1973 | | | | | | |
| Kidney beans & pods | WP | 1.9 | 1 | 75 | <0.01, 0.01, <0,01 | GH-C 1157 |
| Kidney bean vines | WP | 1.9 | 1 | 75 | <0.01, <0.01, <0.01 | |
| NY, 1974 | | | | | | |
| Kidney beans & pods | WP | 1.2 | 1 | 105 | 0.01, 0.01, 0.01, 0.01 | GH-C 1157 |
| Whole plant | WP | 1.2 | 1 | 28 | 0.05, 0.09,0.03, 0.03 | |
| Whole plant | WP | 1.2 | 1 | 41 | 0.09, 0.18, 0.03, 0.07 | |
| Kidney bean vines | WP | 1.2 | 1 | 105 | 0.26, 0.03, 0.03, 0.02 | |
| CA, 1976 | | | | | | |
| Kidney beans | WP | 1.2 | 1 | 161 | 0.01 | GH-C 1157 |
| Kidney bean vines | WP | 1.2 | 1 | 161 | 0.01, 0.14 | |
| MI, 1972 | | | | | | |
| Field beans | WP | 0.63 | 1 | 114 | 0.01 | GH-C 1157 |
| | | 1.2 | 1 | 114 | 0.01 | |
| | | 2.5 | 1 | 114 | 0.02 | |
| Field bean vines | WP | 0.63 | 1 | 114 | 0.05 | |
| | | 1.2 | 1 | 114 | 0.02 | |
| | | 2.5 | 1 | 114 | 0.86 | |
| WI, 1971 | | | | | | |
| Snap bean plant | WP | 0.63 | 1 | 16 | <0.01 | GH-C 660 |
| | | 0.63 | 1 | 28 | <0.01 | |
| | | 0.63 | 1 | 58 | <0.01 | |
| Snap bean plants | WP | 1.9 | 1 | 16 | 0.01 | GH-C 660 |
| | | 1.9 | 1 | 28 | 0.01 | |
| | | 1.9 | 1 | 58 | <0.01 | |
| Beans (with pod) | WP | 0.63 | 1 | 58 | <0.01 | |
| | | 1.9 | 1 | 58 | <0.01 | |
| NY, 1971 | WP | 0.63 | 1 | 31 | <0.01 | GH-C 660 |
| Snap bean plant | | 0.63 | 1 | 31 | 0.03 | |
| | | 0.63 | 1 | 46 | <0.01 | |
| | | 0.63 | 1 | 58 | 0.01 | |
| Snap bean plant | WP | 1.9 | 1 | 31 | 0.22 | |
| | | 1.9 | 1 | 46 | <0.01 | |

| Location, Year, Sample | Application | | | PHI, days | Residues, mg/kg [1,2] | Reference/ Comments |
|---|---|---|---|---|---|---|
| | Form. | g ai/ kg | No. | | | |
| | | 1.9 | 1 | 58 | <0.01 | |
| Beans (with pod) | WP | 0.63 | 1 | 58 | <0.01 | |
| | | 1.9 | 1 | 58 | <0.01 | |
| MS, 1971 | WP | 0.63 | 1 | 28 | 0.04, 0.05, 0.06, 0.06 | GH-C 660 |
| Green plant | | 0.63 | 1 | 43 | 0.01, 0.01, <0.01, <0.01 | |
| | | 0.63 | 1 | 59 | 0.01, 0.01, 0.04, 0.03 | |
| Green plant | WP | 1.9 | 1 | 28 | 0.28, 0.07, 0.16, 0.41 | |
| | | 1.9 | 1 | 43 | 0.01, 0.01, 0.02, 0.01 | |
| | | 1.9 | 1 | 59 | 0.14, 0.18, 0.17, 0.05 | |
| Beans (with pod) | WP | 0.63 | 1 | 58 | <0.01, <0.01, <0.01,<0.01 | |
| | | 1.9 | 1 | 58 | <0.01, <0.01, <0.01, 0.01 | |
| FL, 1972 | WP | 0.63 | 1 | 28 | 0.01, 0.01, 0.01, 0.01 | GH-C 660 |
| Green Plant | | 0.63 | 1 | 41 | 0.01, <0.01, 0.01, <0.01 | |
| | | 0.63 | 1 | 57 | <0.01, <0.01, <0.01, 0.01 | |
| Green Plant | WP | 1.9 | 1 | 28 | 0.03, 0.04, 0.02, 0.02 | |
| | WP | 1.9 | 1 | 41 | 0.01, 0.02, 0.02, 0.02 | |
| | WP | 1.9 | 1 | 57 | 0.01, 0.01, 0.01, 0.02 | |
| Beans (with pod) | WP | 0.63 | 1 | 57 | <0.01, <0.01, <0.01, 0.01 | GH-C 660 |
| | WP | 1.9 | 1 | 57 | <0.01, <0.01, <0.01, 0.01 | |
| OR, 1971 | WP | 0.63 | 1 | 48 | 0.24, 0.59, 0.45, 0.29 | GH-C 660 |
| Green Plant | WP | 0.63 | 1 | 61 | <0.01, <0.01, <0.01, <0.01 | |
| | WP | 1.9 | 1 | 48 | 1.4, 1.4, 1.1, 1.3 | |
| | WP | 1.9 | 1 | 61 | 0.01, <0.01, 0.01, 0.07 | |
| IL, 1969 | WP | 1.2 | 1 | 55 | <0.01 | GH-C 660 |
| Green plant | | | | 57 | <0.01 | |
| | | | | 59 | <0.01, <0.01 | |
| | | | | 60 | <0.01, <0.01 | |
| Beans (with pod) | WP | 1.2 | 1 | 55 | <0.01 | GH-C 660 |
| | | | | 57 | <0.01 | |
| | | | | 59 | <0.01, <0.01 | |
| | | | | 60 | <0.01, <0.01 | |
| | | | | 61 | <0.01, <0.01, <0.01 | |

[1] LOQ 0.01 mg/kg
[2] Recoveries averaged 92% from snap beans, 98% from kidney beans and pods, 91% from kidney bean vines and whole plants, 88% from field beans and 89% from field beans and vines

Peas. Supervised trials on peas were carried out in the USA, 1972-1976, and in the UK in 1975 and 1992-1993. The results are shown in Tables 72 and 73.

In the US trials seeds were treated before planting with a slurry of chlorpyrifos (25% or 50% WP) at 0.63 to 1.9 g ai/ kg seeds. Samples of peas, pods and vines were collected at normal harvest, and whole plants from one location four and six weeks after planting (Norton, 1979).

Table 72. Residues of chlorpyrifos from seed treatment of peas in supervised trials in the USA.

| Location, Year | Application | | | PHI, days | Residues, mg/kg [1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | g ai/kg | No. | | | |
| GAP-USA | WP (SL) | 0.62 | 1 | NA | | Seed treatment |
| IL, 1972 | | | | | | |
| peas + pods | WP | 0.63 | 1 | 61 | 0.01 | GH-C 1158 |
| | | | | 64 | 0.01 | |
| | | | | 68 | 0.01 | |
| | | | | 70 | <0.01 | |
| | | | | 77 | 0.01, 0.01 | |
| vines | WP | 0.63 | 1 | 61 | 0.01 | |
| | | | | 68 | 0.01 | |
| MS, 1972 | | | | | | |
| peas (shelled) | WP | 1.9 | 1 | 46 | 0.15 | GH-C 1158 |
| vines | | | | 46 | 1.1 | |
| MS, 1973 | | | | | | |
| peas (shelled) | WP | 1.9 | 1 | 52 | 0.01 | GH-C 1158 |
| IL, 1974 | | | | | | |
| peas + pods | WP | 0.63 | 1 | 58 | <0.01, <0.01, <0.01 | GH-C 1158 |
| vines | WP | 0.63 | 1 | 58 | 0.02, 0.01, 0.01 | |
| IL, 1975 | | | | | | |
| peas + pods | WP | 1.2 | 1 | 59 | <0.01, <0.01, 0.01, <0.01 | GH-C 1158 |
| vines | WP | 1.2 | 1 | 59 | 0.01, 0.01, 0.01, 0.03 | |
| WA, 1975 | | | | | | |
| peas + pods | WP | 0.63 | 1 | 73 | 0.01, 0.01, 0.01, 0.01 | GH-C 1158 |
| vines | WP | 0.63 | 1 | 73 | 0.03, 0.04, 0.05, 0.10 | |
| NY, 1976 | | | | | | |
| peas + pods | WP | 0.63 | 1 | 68 | 0.01, 0.01, 0.01, 0.01 | GH-C 1158 |
| vines | WP | 0.63 | 1 | 68 | 0.09, 0.09, 0.09, 0.17 | |

[1] LOQ was 0.05 mg/kg with recoveries averaging 81% from peas and pods and 82% from plants and vines.

In three trials (two on vining peas and one on edible podded peas) at different locations in the UK in 1992 single applications of EC was sprayed onto the plants at 0.36 kg ai/hl, 0.72 kg ai/ha. Whole plant samples were taken at intervals until harvest when peas were sampled separately (Khoshab *et al.*, 1993i).

Two other trials in 1993 were similar to the above. Samples of peas were taken at harvest (69 or 75 days after application). Each sample consisted of four replicates which were analysed separately (Khoshab and Berryman, 1994e).

In 1975 in two trials in the UK, peas were treated at 0.75 kg ai/ha and sampled approximately 30 days later (Hollick and Walker, 1976c).

The results of trials in the UK are shown in Table 73.

Table 73. Residues of chlorpyrifos in peas from supervised trials in the UK (foliar application).

| Year, Sample | Application | | | | PHI, days | Residues, mg/kg[1,2] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | g ai/ha | kg ai/ hl | No. | | | |

| Year, Sample | Application | | | | PHI, days | Residues, mg/kg[1,2] | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | Form. | g ai/ha | kg ai/ hl | No. | | | |
| GAP-UK | 480 EC | 0.72 | (0.36) | 2 | 21 | | Foliar treatment |
| 1992 | | | | | | | |
| whole plant | EC | 0.72 | 0.36 | 1 | 0 | 22 | GHE-P 3232 |
| | | | | | 21 | 0.07 | |
| | | | | | 48 | 0.03 | |
| | | | | | 57 | 0.03 | |
| rest of plant | | | | | 78 | <0.01, <0.01 | |
| pods + peas | | | | | 78 | <0.01, <0.01 | |
| 1992 | | | | | | | |
| whole plant | 480 EC | 0.72 | 0.36 | 1 | 0 | 39 | GHE-P 3232 |
| | | | | | 21 | 0.04 | |
| | | | | | 48 | <0.01 | |
| rest of plant | | | | | 59 | <0.01, <0.01 | |
| pods + peas | | | | | 59 | <0.01, <0.01 | |
| 1992 | | | | | | | GHE-P 3232 |
| whole plant | 480 EC | 0.72 | 0.36 | 1 | 0 | 47 | |
| | | | | | 21 | 0.02 | |
| | | | | | 48 | <0.01 | |
| rest of plant | | | | | 64 | 0.01 | |
| peas | | | | | 64 | <0.01 | |
| 1993 | | | | | | | |
| peas | 480 EC | 0.72 | 0.36 | 1 | 75 | <0.01, <0.01, | GHE-P 3487 |
| | | | | | | <0.01, <0.01 | |
| 1993 | | | | | | | |
| peas | 480 EC | 0.72 | 0.36 | 1 | 69 | <0.01 | GHE-P 3487 |
| 1975 peas | 480 EC | 0.75 | 0.13 | 1 | 33 | <0.01 | GHE-P 437 |
| 1975 peas | 480 EC | 0.75 | 0.13 | 1 | 30 | <0.01 | GHE-P 437 |

[1] Average of duplicate analyses
[2] LOQ 0.01 mg/kg

Soya beans. Supervised trials on soya beans in 1975 and 1976 in seven locations in the midwest, south and southwestern States of the USA were reported. EC was applied as a broadcast directed spray at emergence followed by 3 or 4 foliar applications during the growing season at rates ranging from 0.56 to 2.2 kg ai/ha at emergence and 0.56 to 1.1 kg ai/ha at each foliar treatment and total rates of 3.36 to 5.5 kg ai/ha. Samples of green forage were taken at intervals from 0 to 22 days after the last application and straw and beans at normal harvest (28-51 days after the last treatment) as shown in Table 74 (Miller, 1979a).

Table 74. Residues of chlorpyrifos in soya beans from supervised trials in the USA (foliar application).

| Location, year | Application | | | PHI, days | Residues, mg/kg[1,2] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA | EC | 1.1 | | NA | | Pre-plant through post-emergence |
| | EC | 1.1 | 3 | 28 | | Foliar broadcast |
| | G | 1.5 (30 inch row space) | 1 | NA | | At planting, post-plant, incorporated. |
| IL, 1975 | | | | | | |
| Green forage | EC | 2.2 + 0.56 (x2) + 1.1 | 4 | 1 | 39, 21, 45 | GH-C 1224 |
| | | | | 7 | 7.8, 6.4, 6.3 | |
| | | | | 14 | 5.5, 3.4, 3.5 | |
| | | | | 22 | 3.6, 1.4, 1.7 | |
| Straw | EC | 2.2 + 0.56 (x2) + 1.1 | 4 | 51 | 12, 6.7, 2.9 | |
| Soya beans | EC | 2.2 + 0.56 (x2) + 1.1 | 4 | 51 | 0.02, 0.01, 0.01 | |
| MS, 1975 | | | | | | |
| Green forage | EC | 0.56 (x2) + 1.12 (x2) | 4 | 0 | 35, 52, 62 | GH-C 1224 |
| | | | | 7 | 11, 12, 8.9 | |
| | | | | 14 | 2, 1.4, 1.4 | |
| | | | | 21 | 0.38, 0.01, 0.20 | |
| Straw | EC | 0.56 (x2) + 1.12 (x2) | 4 | 28 | 1.5, 7.6, 1.4 | |
| MS, 1975 | | | | | | |
| Soya beans | EC | 0.56 (x2) + 1.12 (x2) | 4 | 28 | <0.01, <0.01, <0.01 | GH-C 1224 |
| GA, 1975 | | | | | | |
| Straw | EC | 2.2 + 0.56 (x2) + 1.1 | 4 | 30 | 1.6, 1.6, 1.8 | GH-C 1224 |
| Soya beans | EC | 2.2 + 0.56 (x2) + 1.1 | 4 | 30 | <0.01, <0.01, <0.01 | |
| IL, 1976 | | | | | | |
| Straw | EC | 2.2 + 0.56 (x2) + 1.1 (x2) | 5 | 28 | 0.87, 0.27, 0.45 | GH-C 1224 |
| Soya beans | EC | 2.2 + 0.56 (x2) + 1.1 (x2) | 5 | 28 | 0.01, 0.01, <0.01 | |
| IA, 1975 | | | | | | |
| Straw | EC | 2.2 + 0.56 (x2) + 1.1 (x2) | 5 | 30 | 1.1, 0.70, 0.70 | GH-C 1224 |
| Soya beans | EC | 2.2 + 0.56 (x2) + 1.1 (x2) | 5 | 30 | 0.02, 0.05, 0.02 | |
| NE, 1975 | | | | | | |
| Straw | EC | 2.2 + 0.56 (x2) + 1.1 (x2) | 5 | 31 | 0.47, 0.51, 0.45 | GH-C 1224 |
| Soya beans | EC | 2.2 + 0.56 (x2) + 1.1 (x2) | 5 | 31 | 0.01, 0.01, 0.01 | |
| NC, 1975 | | | | | | |
| Straw | EC | 2.2 + 0.56 (x2) + 1.1 (x2) | 5 | 38 | 2.1, 2.2, 1.6 | GH-C 1224 |
| Soya beans | EC | 2.2 + 0.56 (x2) + 1.1 (x2) | 5 | 38 | 0.01, 0.24, 0.14 | |

[1] LOQ 0.01 mg/kg
[2] Recoveries averaged 89% from green forage, 85% from straw and 88% from soya beans

The Government of Thailand reported four field trials conducted in 1988, 1995 and 1996. In the first two trials an EC formulation was applied 9 times at 0.94 or 1.9 kg ai/ha. In the 1995 trial an EC formulation was applied 4 times at 0.12 or 0.24 kg ai/ha and samples taken 7, 14 and 21 days after the final application. In the 1996 trial, an EC formulation was applied 4 times at 0.12 or 0.24 kg ai/ha and dry seeds were sampled 8, 15 and 22 days after the last application. The pods were dried in the sun before removing the seeds. Samples were analysed by The Netherlands multi-residue method. No recoveries were reported (Thai Industrial Standards Institute, Bangkok, Thailand). The conditions and findings are shown in Table 75.

Table 75. Residues of chlorpyrifos in soya beans from foliar applications of an EC Formulation in field trials in Thailand.

| Location, Year | Application | | | PHI, days | Residue, mg/kg[1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | | |
| GAP- Thailand | 0.75 | 0.12 | | 7 | | EC 200 g/l |
| Nakronsawan Field Crop Research Center, 1988 | 0.94 | 0.12 | 9 | 15 | <0.01 | Govt of Thailand<br>EC 200 g/l<br>4 replicate plots, each 5 x 8 m. |
| | 1.9 | 0.24 | 9 | 15 | <0.01 | |
| Nakronsawan Crop Research Center, 1988 | 0.44 | 0.12 | 9 | 14 | <0.01 | Govt of Thailand<br>EC 200 g/l<br>4 replicate plots, each 5 x 8 m. |
| | 1.9 | 0.24 | 9 | 14 | <0.01 | |
| Saraburi Province, 1995 | 0.75 | 0.12 | 4 | 7<br>14<br>21 | 0.23<br>0.06<br>0.02 | Govt of Thailand<br>EC 400 g/l<br>4 replicate plots, each 6 x 12 m. |
| | 1.5 | 0.24 | 4 | 7<br>14<br>21 | 0.32<br>0.06<br>0.11 | |
| Chainat Province, 1996 | 0.75 | 0.12 | 4 | 8<br>15<br>22 | 1.6<br>0.97<br>0.09 | Govt of Thailand<br>EC 400 g/l<br>4 replicate plots, each 8 x 8 m |
| | 1.5 | 0.24 | 4 | 8<br>15<br>22 | 2.9<br>1.4<br>0.82 | |

[1] Reported limit of detection 0.001 mg/kg.

Root and tuber vegetables

Carrots. Supervised trials were conducted in South Africa in 1976, in the UK in 1976-1977 and 1995 and in The Netherlands in 1995. The results are shown in Table 77.

In two trials in South Africa carrots were treated four times at approximately monthly intervals with an EC formulation at 0.48 kg ai/ha (GAP) and 0.72 kg ai/ha. Samples from both trials were taken 1, 3, 7, 21 and 28 days after the last application (Hollick and Walker, 1978a).

Three trials were carried out in the UK in 1975-1976. In one carrots were treated at 1.44 kg ai/ha with samples taken 49 days after treatment, and in the other two at 1.2 and 0.96 kg ai/ha with samples taken 15, 24 and 30 days after application (Hollick and Walker, 1977).

In two late-season trials in the UK in 1995 two applications of chlorpyrifos WG (750 g ai/kg) formulation were sprayed on carrots at 0.96 kg ai/ha and 0.16 kg ai/hl. The carrots were sampled at harvest 14 days after the last application (Portwood and Williams, 1996n).

In two trials at different locations in The Netherlands in 1995 chlorpyrifos was sprayed twice at 21-day intervals at 0.96 kg ai/ha and 0.48 kg ai/hl, and samples were taken from 0 to 14 days after the final application (Portwood, 1996d).

Table 76. Residues of chlorpyrifos in carrots from supervised trials in the UK, The Netherlands and South Africa (foliar application).

| Country, Year | Application | | | | PHI, days | Residues, mg/kg [1,2,3] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-South Africa | 480 EC | 0.48 | (0.096) | | 21 | | |
| South Africa, 1976 | EC | 0.48 | | 4 | 1 | 0.08 | GHE-P 542 |
| | | | | | 3 | 0.09 | |
| | | | | | 7 | 0.08 | |
| | | | | | 21 | 0.05 | |
| | | | | | 28 | 0.05 | |
| South Africa, 1976 | EC | 0.72 | | 4 | 1 | 0.31 | GHE-P 542 |
| | | | | | 3 | 0.10 | |
| | | | | | 7 | 0.14 | |
| | | | | | 21 | 0.09 | |
| | | | | | 28 | 0.07 | |
| South Africa, 1976 | EC | 0.72 | | 4 | 1 | 0.31 | GHE-P 542 |
| | | | | | 3 | 0.10 | |
| | | | | | 7 | 0.14 | |
| | | | | | 21 | 0.09 | |
| | | | | | 28 | 0.07 | |
| UK | 480 EC | 0.96 | 0.48 | | 14 | | |
| UK, 1976 | EC | 0.96 | | 2 | 15 | 0.02 | GHE-P 439 |
| | | | | | 24 | 0.03 | |
| | | | | | 30 | 0.02 | |
| UK, 1976 | EC | 1.2 | | 2 | 15 | 0.03 | GHE-P 439 |
| | | | | | 24 | 0.03 | |
| | | | | | 30 | 0.03 | |
| UK, 1975 | EC | 1.44 | | 2 | 49 | 0.03 | GHE-P 439 |
| UK, 1995 | WG | 0.96 | 0.16 | 2 | 14 | <0.01, <0.01 | GHE-P 5473 |
| GAP -Netherlands NONE. See UK. | | | | | | | |
| Netherlands, 1995 | WG | 0.94 + 0.97 | 0.48 | 2 | 0 | 13 | GHE-P 5491 |
| | | | | | 4 | 2.18 | |
| | | | | | 9 | 1.03 | |
| | | | | | 14 | 0.01 | |
| Netherlands, 1995 | WG | 0.98 + 0.95 | 0.48 | 2 | 0 | 17.5 | GHE-P 5491 |
| | | | | | 4 | 2.97 | |
| | | | | | 9 | 0.56 | |
| | | | | | 14 | 0.03 | |

[1] Average of duplicate analyses
[2] LOQ 0.01 mg/kg
[3] Recoveries 88% from the 1995 trials and 101% from those in 1975-1977

Potatoes. Two residue field trials were reported from Brazil, where a granular formulation was applied to the furrow at planting. Potato samples were taken at normal harvest, about 100 days after planting (Balderrama and Matos, 1994c). In separate studies at two sites 3-4 applications were made with EC formulations (Balderrama and Matos, 1994d,e). During the 1994-95 season two trials were carried out in Brazil with the EC formulation applied once at planting in furrow according to GAP for the 10G formulation. Samples were collected 103 to 105 days after treatment (Do Amaral, 1999).

Table 77. Residues of chlorpyrifos in potato tubers from the at-planting application to potato fields of a granular formulation.

| Location, (Variety) Year | Application | | PHI, days | Residue, mg/kg[1] | Reference/ Comment |
|---|---|---|---|---|---|
| | kg ai/ha | No. | | | |
| GAP- Brazil<br>NONE<br>GAP- Chile<br>GAP-Argentina | <br><br>3<br>2.9 | <br><br>1<br>1 | | | <br><br>G<br>EC |
| Mogi-Mirim, Brazil (Achat) 1993 | 1.5 | 4 | 124 | 0.20;0.03;0.19 | GHB-P 218 |
| | 3.0 | 4 | 124 | 0.29;0.10;0.14 | |
| | 6.0 | 4 | 124 | 0.29;0.08;0.05 | |
| Ouro Fino, Brazil | 1.5 | 4 | 105 | 0.17;0.13;0.07 | |
| | 3.0 | 4 | 105 | 0.12;0.51;0.23 | |
| | 6.0 | 4 | 105 | 0.96;0.66;0.46 | |
| Mogi-Mirim, Brazil (Achat) 1994 | 0.90 | 3 | 21 | <0.01 | GHB-P 224. Spray volume 400 l/ha |
| | 1.8 | 3 | 21 | <0.01 | |
| Ouro Fino, Brazil (Chatti) 1994 | 0.90 | 4 | 21 | <0.01 | GHB-P 224. Spray volume 400 l/ha. |
| | 1.8 | 4 | 21 | <0.01 | |
| Brazil, 1994 | 2.9 | 1 | 103 | 0.03, 0.13, 0.08 | GHB-P 349<br>EC formulation |
| | 5.9 | 1 | 103 | 0.27, 0.18, 0.22 | |
| Brazil, 1994 | 2.9 | 1 | 105 | 0.02, <0.01, 0.02 | GHB-P 349<br>EC formulation |
| | 5.9 | 1 | 105 | 0.03, 0.06, 0.01 | |

[1] LOQ 0.01 mg/kg.

Table 78. Residues of chlorpyrifos in potato tubers from the foliar application of an EC formulation in Brazil.

| Location, (Variety) Year | Application | | PHI, days | Residue, mg/kg | Reference |
|---|---|---|---|---|---|
| | kg ai/ha | No. | | | |
| Brazil GAP | 0.72 | 2 | 21 | | |
| Mogi-Mirim, Brazil (Achat) 1992 | 0.72 | 4 | 21 | 0.01 | GHB-P 194 |
| | 1.4 | 4 | | 0.02 | |
| Guarapuava, Brazil (Bintje)1993 | 0.72 | 4 | 36 | 0.02 | |

| Location, (Variety) Year | Application | | PHI, days | Residue, mg/kg | Reference |
|---|---|---|---|---|---|
| | kg ai/ha | No. | | | |
| | 1.4 | 4 | 36 | 0.03 | |

In two trials in Australia in 1982 an EC formulation was applied at 3.0 or 1.5 kg ai/ha pre-planting and 0.45 kg ai/ha pre-emergence, and samples taken at harvest 165 days after the pre-emergence application (Vella, 1983).

In trials in Columbia in 1982, potatoes were treated two or three times during the season with the 480 EC formulation at 1.44 and 1.92 kg ai/ha. The tubers were collected from all plots 127 days after the last application (Hollick, 1983).

The government of Poland submitted summary information on two 1994 trials. A foliar application of an EC formulation was made to vines at 0.42 kg ai/ha (Institute of Plant Protection, Poland, 1999).

Table 79: Residues of chlorpyrifos in potatoes from supervised trials in Australia, Columbia and Poland.

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2,3] | Reference/ comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Australia | 500 EC | 3.0 pre-plant<br>0.5 hilling up | | 2 | | | Soil treatment |
| Australia, 1982 | EC | 3 + 0.45 | | 2 | 165 | <0.01, <0.01, <0.01, <0.01 | PAU 3183 068 |
| Australia, 1982 | EC | 1.5 + 0.45 | | 2 | 165 | <0.01, <0.01, <0.01, <0.01 | PAU 3183 068 |
| GAP-Columbia | 480 EC | 1.6 | | 2-4 | 15 | | |
| Columbia, 1982 | 480 EC | 1.44 | | 2 | 127 | 0.02 | GHB-P 013 |
| | | | | 3 | 127 | 0.03 | |
| Columbia, 1982 | 480 EC | 1.92 | | 2 | 127 | 0.02 | GHB-P 013 |
| Columbia, 1982 | | | | 3 | 127 | 0.04 | |
| GAP- Poland | 268 EC | 0.42 | 0.28 | 2 | 30 | | |
| Bonin, Poland 1994 (Aster) | 278 g/l EC | 0.42 | | 1 | 3<br>7<br>14 | <0.02<br><0.02<br><0.02 | Institute of Plant Protection, Poland, 1999 |
| Bonin, Poland 1994 (Bogna) | 278 g/l EC | 0.42 | | 1 | 49 | <0.02 | |

[1] Average of duplicate analyses
[2] LOQ 0.01 mg/kg for Australia and Columbia, 0.02 mg./kg for Poland.
[3] Recoveries 88-101%

In a trial in South Africa in 1976, foliar applications were made to potatoes three times at 0.72 kg ai/ha, and samples taken at intervals of 1, 3, 7, 14, 21 and 28 days after the last treatment (Freeman, 1976).

In two trials in the UK in 1977, potatoes were treated once or twice with the 480 EC formulation at 0.96 kg ai/ha, and samples taken at normal harvest 34 or 60 days later (Hollick and Walker, 1978b).

Potatoes in Ontario, Canada, were treated with the 480 EC formulation at 0.5 kg ai/ha in nine successive weekly applications, with samples taken 7, 14 and 20 days after the last (Fairbairn and McKellar, 1980).

Table 80. Residues of chlorpyrifos in potatoes from supervised trials in South Africa, the UK and Canada (foliar application).

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP- South Africa | 480 EC | 0.72<br>0.48 | (0.096) | 1 | 7 | | Pre-plant 1 m row space. Foliar |
| South Africa, 1976 | 480 EC | 0.72 | | 3 | 1 | <0.01 | GHE-P 560 |
| | | | | | 3 | <0.01 | |
| | | | | | 7 | 0.01 | |
| | | | | | 14 | 0.01 | |
| | | | | | 21 | 0.02 | |
| | | | | | 28 | <0.01 | |
| GAP-UK | 480 EC | 0.72 | (0.36) | | 21 | | |
| UK, 1977 | 480 EC | 0.96 | | 2 | 34 | <0.01 | GHE-P 572 |
| UK, 1977 | 480 EC | 0.96 | | 1 | 60 | <0.01 | GHE-P 572 |
| GAP-Canada | 500 WP | 1.1 | (0.28) | 1 | 7 | | |
| | 480 EC | 0.48 | (0.12) | 9 | 7 | | |
| Canada, 1977 | EC | 0.5 | | 9 | 7 | 0.01, <0.01, <0.01 | GHS-C-12 |
| | | | | | 14 | <0.01, <0.01, <0.01 | |
| | | | | | 20 | <0.01, <0.01, <0.01 | |

[1] All results averages of duplicate analyses.
[2] LOQ 0.01 mg/kg.

Sugar beet. Supervised trials on sugar beet were conducted in the UK, France and Germany in 1975, 1980 and 1990 respectively. The trials in the UK were with an EC formulation at 1.44 kg ai/ha and samples were taken approximately 120 days after application (Hollick and Walker, 1977).

In a trial in France during 1986 a latex suspension concentrate containing 300 g/l chlorpyrifos was applied pre-sowing to some plots. Others were treated with a liquid form containing 300 g chlorpyrifos + 160 g lindane/l for comparison. Samples of sugar beet were taken at harvest and frozen before the roots and tops were analysed separately (Day, 1987a).

In trials in Germany in 1990, the 480 EC formulation was applied to plots of immature sugar beet at 0.72 kg ai/ha. Samples of the whole plant were taken 1 day after application, roots and leaves were then sampled separately at various growth stages up to and including normal harvest (Khoshab *et al.*, 1991).

The results are shown in Table 81.

Table 81. Residues of chlorpyrifos in sugar beet from supervised trials in the UK, France and Germany (pre-plant or foliar applications).

| Country, year | Application | | | | PHI, days | Residues, mg/kg[1,2,3] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-UK | EC | 0.72 | (0.36) | 2 | 21 | | |
| UK, 1975 | | | | | | | GHE-P 439 |
| roots | EC | 1.44 | | 1 | 117 | <0.01 | Foliar |
| tops | EC | 1.44 | | 1 | 117 | <0.01 | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg[1,2,3] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| UK, 1975 | | | | | | | GHE-P 439 |
| roots | EC | 1.44 | | 1 | 123 | <0.01 | |
| tops | EC | 1.44 | | 1 | 123 | <0.01 | |
| GAP-France | 300 SC | 1.5 | (1) | 1 | | | Pre-plant |
| France, 1986 | | | | | | | |
| tops | 300 SC | 3 | | 1 | | <0.01, <0.01, <0.01, <0.01 | GHE-P 1709 |
| | Liquid mix | 1.5 | | 1 | | <0.01, <0.01, <0.01, ≤0.01 | Pre-plant |
| roots | 300 SC | 3 | | 1 | | <0.01, <0.01, <0.01, <0.01 | |
| | Liquid mix | 1.5 | | 1 | | <0.01, <0.01, <0.01, ≤0.01 | |
| GAP-Germany | 480 EC | 0.96 | (0.24) | 1 | | | Pre-plant |
| Germany, 1990 | | | | | | | |
| tops | EC | 0.72 | | 1 | 16 | 0.26 | GHE-P 2467 |
| | | | | | 37 | 0.01 | Foliar |
| | | | | | 74 | <0.01 | |
| | | | | | 126 | <0.01 | |
| roots | EC | 0.72 | | 1 | 16 | 0.04 | |
| | | | | | 37 | <0.01 | |
| | | | | | 74 | <0.01 | |
| | | | | | 126 | <0.01 | |
| Germany, 1990 | | | | | | | |
| tops | EC | 0.72 | | 1 | 20 | 0.16 | GHE-P 2467 |
| | | | | | 56 | <0.01 | |
| | | | | | 90 | <0.01 | |
| | | | | | 138 | <0.01 | |
| roots | EC | 0.72 | | 1 | 20 | 0.04 | |
| | | | | | 56 | <0.01 | |
| | | | | | 90 | <0.01 | |
| | | | | | 138 | <0.01 | |
| Germany, 1990 | | | | | | | GHE-P 2467 |
| tops | EC | 0.72 | | 1 | 7 | 2.6 | |
| | | | | | 48 | 0.01 | |
| | | | | | 79 | <0.01 | |
| | | | | | 122 | <0.01 | |
| | | | | | 150 | <0.01 | |
| roots | EC | 0.72 | | 1 | 7 | 0.06 | |
| | | | | | 48 | <0.01 | |
| | | | | | 79 | <0.01 | |
| | | | | | 122 | <0.01 | |
| | | | | | 150 | <0.01 | |
| Germany, 1990 | | | | | | | GHE-P 2467 |
| tops | EC | 0.72 | | 1 | 9 | 1.8 | |
| | | | | | 35 | <0.01 | |
| | | | | | 71 | <0.01 | |
| | | | | | 118 | <0.01 | |
| roots | EC | 0.72 | | 1 | 9 | 0.04 | |
| | | | | | 35 | 0.02 | |
| | | | | | 71 | <0.01 | |
| | | | | | 118 | <0.01 | |

.
[1] Average of duplicate analyses
[2] LOQ 0.01 mg/kg
[3] Recoveries 85% to 101% from the roots and from 84% to 92% from the tops

In supervised trials in Japan the EC formulation containing 400 g ai/l was applied twice at 0.32 kg ai/ha at 10-day intervals. Samples were taken 30, 45 and 60 days after the last application and the roots analysed (Ishikura, 1996). The results are shown in Table 82.

Table 82. Residues of chlorpyrifos in sugar beet from supervised trials in Japan.

| Country, year | Application | | | | PHI, days | Residues, mg/kg[1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Japan NONE | | | | | | | |
| 1995 | EC | 0.32 | 0.027 | 2 | 30 | 0.05 | GHF-P 1491 |
| | | | | 2 | 45 | 0.02 | |
| | | | | 2 | 60 | <0.01 | |
| 1995 | EC | 0.32 | 0.027 | 2 | 30 | <0.01 | GHF-P 1491 |
| | | | | 2 | 45 | <0.01 | |
| | | | | 2 | 60 | <0.01 | |
| 1995 | EC | 0.32 | 0.027 | 2 | 30 | <0.01 | GHF-P 1491 |
| | | | | 2 | 45 | 0.01 | |
| | | | | 2 | 60 | 0.01 | |
| 1995 | EC | 0.32 | 0.027 | 2 | 30 | 0.01 | GHF-P 1491 |
| | | | | 2 | 45 | <0.01 | |
| | | | | 2 | 60 | 0.013 | |
| 1995 | EC | 0.32 | 0.027 | 2 | 30 | 0.01 | GHF-P 1491 |
| | | | | 2 | 45 | <0.01 | |
| | | | | 2 | 60 | 0.01 | |

[1]LOQ 0.01 mg/kg. Recoveries averaged 87%.

In a Canadian trial a single foliar application of the 480 EC formulation was made at the 5-leaf growth stage at 1.12 kg ai/ha, and samples of roots and tops were taken after 87 days at normal harvest (Wetters and Fairbairn, 1980).

In field trials on sugar beet in four US States the WG formulation of chlorpyrifos was compared with the commercial EC formulation. Four applications of 1.12 kg ai/ha each were made at approximately 60, 40, 35 and 30 days before harvest, the crop was divided into roots and tops and analysed for residues (McCormick and Bormett, 1996a).

The results are shown in Table 83.

Table 83. Residues of chlorpyrifos in sugar beet from supervised trials in Canada and the USA (foliar application).

| Country, year | Application | | | | PHI, days | Residues mg/kg [1,2] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Canada | 480 EC | 1.2 | 0.6 | 1 | 90 | | Soil or foliar |
| Canada, 1977 | | | | | | | |
| roots | EC | 1.12 | | 1 | 87 | <0.01, <0.01, <u>≤0.01,</u> <0.01 | GHS-C 10 |
| tops | EC | 1.12 | | 1 | 87 | <0.01, <0.01, <u>≤0.01</u>, <0.01 | |

| Country, year | Application | | | | PHI, days | Residues mg/kg [1,2] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-USA | EC | 1.1 | 1.2 | 4 | 30 | | |
| | G | 2.3 | | 1 | NA | | At planting |
| CA, 1995 | | | | | | | |
| roots | EC | 1.18 | 0.4 | 4 | 30 | 0.02, 0.02 | GH-C 4223 |
| tops | | | | | | 0.26, 0.68 | |
| roots | WG | 1.15 | 0.4 | 4 | 30 | 0.02, 0.02 | |
| tops | | | | | | 0.15, 0.15 | |
| ND, 1995 | | | | | | | |
| roots | EC | 1.12 | 0.5 | 4 | 29 | 0.03, 0.01 | GH-C 4223 |
| tops | | | | | | 6.6, 4.6 | |
| roots | WG | 1.16 | 0.6 | 4 | 29 | 0.02, 0.02 | |
| tops | | | | | | 3.1, 2.8 | |
| TX, 1995 | | | | | | | |
| roots | EC | 1.11 | 0.6 | 4 | 30 | <0.01, 0.01 | GH-C 4223 |
| tops | | | | | | 0.24, 0.44 | |
| roots | WG | 1.12 | 0.5 | 4 | 30 | 0.01, 0.01 | |
| tops | | | | | | 0.21, 0.42 | |
| WA, 1995 | | | | | | | |
| roots | EC | 1.12 | 0.5 | 4 | 30 | 0.01, 0.01 | GH-C 4223 |
| tops | | | | | | 1.3, 1.2 | |
| roots | WG | 1.12 | 0.5 | 4 | 30 | 0.01, 0.01 | |
| tops | | | | | | 0.82, 1.4 | |

[1] In the US trials, the limit of detection was 0.003 mg/kg and the LOQ 0.01 mg/kg. Recoveries averaged 80% from tops and 71% from roots.
[2] In the Canadian trials, the LOQ was 0.01 mg/kg. Recoveries averaged 88%

## Cereal grains and animal feed commodities

Maize (field corn). The results of residue trials with a G formulation (at planting) and an EC formulation (foliar) applied to maize were reported from Brazil (Balderrama and Matos, 1994f,g). The results are shown in Tables 84 and 85.

Table 84. Residues of chlorpyrifos from single at-planting applications of a G formulation to maize.

| Location, Year | Application kg ai/ha | PHI, days | chlorpyrifos, mg/kg | Reference/Comment |
|---|---|---|---|---|
| Chile GAP<br>Argentina-GAP | 3 (G) broadcast<br>1.9 (EC) soil incorp | | | No GAP for Brazil |
| Londrina, Brazil (AG-122) 1993-1994 | 1.1 | 123 | <0.01 | GHB-P 217 |
| | 2.2 | 123 | <0.01 | |
| Rio Verde, Brazil (BR-201) 1993-1994 | 1.1 | 125 | <0.01 | |
| | 2.2 | 125 | <0.01 | |

Table 85. Residues of chlorpyrifos from the foliar application of an EC formulation to maize in Brazil.

| Location, Year | Application | | PHI, days | chlorpyrifos, mg/kg | Reference |
|---|---|---|---|---|---|
| | kg ai/ha | No. | | | |
| Brazil GAP | 0.48 | 2-3 | 21 | | |
| Mogi-Mirim, (Dina 50) 1992-1993 | 0.29 | 3 | 20 | <0.01 | GHB-P 185. 300 l/ha. |
| | 0.58 | 3 | 20 | ≤0.01 | |
| Cascavel, (Cargil 606) 1992-1993 | 0.29 | 3 | 115 | <0.01 | GHB-P 185. 300 l/ha. |
| | 0.58 | 3 | 115 | <0.01 | |

In a set of US trials in four States, surface band or seed furrow applications of chlorpyrifos were applied at planting. A granular formulation was used for the surface band applications at 1.12 to 3.36 kg ai/ha and a ULV EC formulation at 1.12 kg ai/ha for the seed furrow directed sprays. Green forage samples were collected either at monthly intervals or the silage stage, and grain and stover samples at normal harvest. Samples were twenty immature or 10 mature plants taken at random from the centre in each replicate, and chopped, mixed and sub-sampled. 1.4 to 2.3 kg of grain were collected. All samples were stored frozen before analysis (McKellar, 1972).

In another set of trials, plots of field corn in Illinois, Michigan, Mississippi and Missouri were treated with EC formulation at 1.12 or 1.68 kg ai/ha during the 2 to 6-leaf stage by ground or aerial spray. The Michigan and Missouri plots were also treated at planting with 1.2 or 1.5 kg ai/ha of a granular formulation. Samples of green forage were taken at the silage stage (47 to 102 days after the last application) and grain and fodder at normal harvest 104 to 127 days after the last application (Wetters *et al.*, 1977).

In two trials in 1979 chlorpyrifos was applied to plots of field corn at two locations, either as a pre-plant broadcast and incorporated application of a granular formulation or of the EC formulation, both at 3.36 kg ai/ha. In one trial in Michigan, the EC treatment was followed by post-emergence foliar applications at 1.68 kg ai/ha at the 5-leaf stage and then again 7 days later. Samples of green forage were collected at the silage stage 44 to 105 days after the last application and fodder and grain at normal harvest 143 to 176 days after the last application (Norton, 1980a).

Trials in four US States in 1979 were with multiple applications of chlorpyrifos 15 G and EC formulations were made. At three of the locations, 5 or 6 ground applications were made to separate plots. Five aerial treatments were applied at the fourth site. Field corn was treated at planting with the granular formulation followed by foliar treatments with the EC formulation plus either two applications of granular or three applications of EC. A total of 6.6 to 9.9 kg ai/ha was applied. Samples of green forage were taken 0 to 20 days after the last application and fodder and grain at normal harvest (Norton, 1981).

The results are shown in Table 86.

Table 86. Residues of chlorpyrifos in field corn from supervised trials in the USA.

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-USA | EC | 1.7 foliar<br>3.4 pre-plant incorp<br>1.12 soil pre-emergence | | 5 | 35 grain<br>14 silage<br>35 fodder | | |
| | 15 G | 1.1 broadcast (ground/aerial)<br>2.2 soil incorp pre-plant | | 1 | | | |

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | 50 WP | 62g ai/ 100 kg seeds | | 1 | NA | | |
| Nebraska, 1970 | | | | | | | |
| Green forage | 10 G | 1.12 | | 1 | 27 | <0.01, <0.01, <0.01 | GH-C 530 At planting |
| | | | | | 57 | <0.01, 0.01, <0.01 | |
| | | | | | 87 | <0.01, <0.01, <0.01 | |
| | | | | | 112 | <0.01, <0.01, <0.01 | |
| | 10 G | 3.36 | | 1 | 112 | <0.01, <0.01, <0.01 | |
| Grain | 10 G | 1.12 | | 1 | 149 | <0.01, <0.01, <0.01 | |
| | | 3.36 | | 1 | 149 | <0.01, <0.01, <0.01 | |
| Stover | 10 G | 1.12 | | 1 | 149 | <0.01, <0.01, <0.01 | |
| | | 3.36 | | 1 | 149 | <0.01, <0.01, <0.01 | |
| Nebraska, 1970 | | | | | | | |
| Green forage | EC | 1.12 | 23 | 1 | 27 | <0.01, <0.01, 0.01 | GH-C 530 At planting |
| | | | | | 57 | <0.01, <0.01, <0.01 | |
| | | | | | 87 | <0.01, <0.01, <0.01 | |
| | | | | | 112 | <0.01, <0.01, <0.01 | |
| Grain | EC | 1.12 | 23 | 1 | 149 | <0.01, <0.01, <0.01 | |
| Stover | EC | 1.12 | 23 | 1 | 149 | <0.01, <0.01, <0.01 | |
| Illinois, 1970 | | | | | | | |
| Green forage | G | 1.12 | | 1 | 27 | <0.01, <0.01, <0.01 | GH-C 530 At planting |
| | | | | | 59 | <0.01, <0.01, <0.01 | |
| | | | | | 89 | <0.01, <0.01, <0.01 | |
| | | | | | 100 | 0.01, <0.01, 0.01 | |
| | G | 3.36 | | 1 | 100 | 0.01, <0.01, <0.01 | |
| Grain | G | 1.12 | | 1 | 130 | <0.01, <0.01, <0.01 | |
| | | 3.36 | | 1 | 130 | <0.01, <0.01, <0.01 | |
| Stover | G | 1.12 | | 1 | 130 | <0.01, <0.01, 0.01 | |
| | | 3.36 | | 1 | 130 | <0.01, <0.01, <0.01 | |
| Illinois, 1970 | | | | | | | |
| Green forage | EC | 1.12 | 23 | 1 | 27 | 0.01, 0.01, <0.01 | GH-C 530 At planting |
| | | | | | 59 | <0.01, <0.01, <0.01 | |
| | | | | | 89 | <0.01, <0.01, <0.01 | |
| | | | | | 100 | <0.01, <0.01, <0.01 | |
| Grain | EC | 1.12 | 23 | 1 | 130 | <0.01, <0.01, <0.01 | |
| Stover | EC | 1.12 | 23 | 1 | 130 | 0.01, <0.01, 0.01 | |
| Ohio, 1970 | | | | | | | |
| Green forage | G | 1.12 | | 1 | 117 | <0.01, <0.01, <0.01, <0.01 | GH-C 530 At planting |
| | | | | | | <0.01, <0.01, <0.01, <0.01 | |
| Grain | G | 1.12 | | 1 | 159 | <0.01, <0.01, <0.01, <0.01 | |
| Stover | G | 1.12 | | 1 | 159 | <0.01, <0.01, <0.01, <0.01 | |

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| Kansas, 1970 | | | | | | | |
| Green forage | G | 1.12 | | 1 | 52 | <0.01 | GH-C 530 At planting |
| Grain | G | 1.12 | | 1 | 170 | <0.01 | |
| Stover | G | 1.12 | | 1 | 170 | <0.01 | |
| Illinois, 1977 | 4 E | | | | | | GH-C 1068 |
| Green forage | 4 E | 1.12 | | 1 | 102 | <0.01, <0.01, <0.01, <0.01 | Foliar, ground |
| Green forage | 4 E | 1.12 | | 1 | 102 | <0.01, <0.01, <0.01, <0.01 | GH-C1068 Foliar, aerial |
| Fodder | 4 E | 1.12 | | 1 | 127 | <0.01, <0.01, <0.01, <0.01 | Ground |
| Fodder | 4 E | 1.12 | | 1 | 127 | 0.01, <0.01, <0.01, <0.01 | Aerial |
| Grain | 4 E | 1.12 | | 1 | 127 | <0.01, <0.01, <0.01, <0.01 | Ground |
| Grain | 4 E | 1.12 | | 1 | 127 | <0.01, <0.01, <0.01, <0.01 | Aerial |
| Michigan, 1977 | | | | | | | |
| Green forage | G + EC | 1.5 + 1.12 | | 2 | 89 | <0.01, <0.01, <0.01, 0.01 | GH-C 1068 Foliar |
| Fodder | G + EC | 1.5 + 1.12 | | 2 | 114 | <0.01, <0.01, <0.01, 0.01 | |
| Fodder | G + EC | 1.5 + 1.68 | | 2 | 114 | 0.01, <0.01, <0.01, <0.01 | |
| Grain | G + EC | 1.5 + 1.12 | | 2 | 114 | <0.01, <0.01, <0.01, 0.01 | |
| | G + EC | 1.5 + 1.68 | | 2 | 114 | <0.01, <0.01, <0.01, 0.01 | |
| Mississippi, 1977 | | | | | | | |
| Green forage | EC | 1.12 | | 1 | 47 | 0.01, <0,01, <0,01, 0.01 | GH-C 1068 |
| Grain | EC | 1.12 | | 1 | 104 | <0.01, <0.01, <0.01 | Foliar |
| Mississippi, 1977 | | | | | | | |
| Green forage | EC | 1.12 | | 1 | 66 | 0.01, 0.01, 0.02, 0.02 | GH-C 1068 |
| Grain | EC | 1.12 | | 1 | 109 | <0.01, <0.01, <0.01, 0.01 | Foliar |
| Missouri, 1977 | | | | | | | |
| Green forage | G + EC | 1.23 + 1.12 | | 2 | 63 | <0.01, <0.01, <0.01, 0.01 | GH-C 1068 |
| Fodder | G + EC | 1.23 + 1.12 | | 2 | 126 | <0.01, <0.01, <0.01, 0.01 | Foliar |
| Grain | G + EC | 1.23 + 1.12 | | 2 | 126 | <0.01, <0.01, <0.01, 0.01 | |
| Illinois, 1978 | | | | | | | |
| Green forage | G | 3.36 | | 1 | 105 | 0.02, 0.01, 0.01 | GH-C 1284 |
| | EC | 3.36 | | 1 | 105 | 0.01, 0.01, 0.01 | Pre-plant |
| Fodder | G | 3.36 | | 1 | 151 | 0.01, 0.01, 0.01 | |
| | EC | 3.36 | | 1 | 151 | 0.01, 0.01, 0.01 | |
| Grain | G | 3.36 | | 1 | 151 | <0.01, <0.01, <0.01 | |
| | EC | 3.36 | | 1 | 151 | <0.01, <0.01, <0.01 | |
| Michigan, 1978 | | | | | | | |
| Green forage | G | 3.36 | | 1 | 77 | 0.01, 0.01, <0.01, <0.01 | GH-C 1284 |

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | EC | 3.36 + 1.68 (x 2) | | 3 | 44 | 0.01, <0.01, <0.01, <0.01 | Pre-plant |
| Fodder | G | 3.36 | | 1 | 176 | 0.01, 0.01, 0.01, 0.01 | |
| | EC | 3.36 + 1.68 (x 2) | | 3 | 143 | 0.01, 0.02, 0.01, 0.01 | |
| Grain | G | 3.36 | | 1 | 174 | <0.01, <0.01, <0.01, 0.01 | |
| | EC | 3.36 + 1.68 (x 2) | | 3 | 141 | <0.01, <0.01, <0.01, 0.01 | |
| Illinois, 1979 | | | | | | | |
| Green forage | G + EC | 1.22 (x 3) + 1.68 (x 2) | | 5 | 0 | 2.4, 1.9, 0.71, <0.01 | GH-C 1440 |
| | | | | | 6 | 2.3, 1.9, 0.27, <0.01 | At planting + foliar |
| | | | | | 13 | 2.8, 0.72, 0.31, 2.5 | |
| | | | | | 20 | 1.5, 2.0, 0.33, 0.32 | |
| Illinois, 1979 | | | | | | | |
| Green forage | G + EC | 1.22 + 1.68 (x 5) | | 6 | 0 | 2.05, 22.6, 17.7, 19 | GH-C 1440 |
| | | | | | 6 | 1.6, 3.4, 3.4, 3.1 | At planting + foliar |
| | | | | | 13 | 2.0, 1.3, 5.5, 2.6 | |
| | | | | | 20 | 2.5, 1.1, 2.1, 3.9 | |
| Fodder | G + EC | 1.22 (x 3) + 1.68 (x 2) | | 5 | 34 | 1.1, 0.21, 3.4, 5.9 | |
| Fodder | G + EC | 1.22 + 1.68 (x 5) | | 6 | 34 | 2.3, 0.67, 0.25, 0.92 | |
| Grain | G + EC | 1.22 (x 3) + 1.68 (x 2) | | 5 | 20 | <0.01, <0.01, 0.01, <0.01 | |
| | | | | | 34 | 0.04, 0.01, 0.01, 0.03 | |
| Grain | G + EC | 1.22 + 1.68 (x 5) | | 6 | 20 | <0.01, <0.01, 0.01, 0.01 | |
| | | | | | 34 | 0.02, 0.03, 0.02, 0.02 | |
| Michigan, 1979 | | | | | | | |
| Green Forage | G + EC | 1.46 (x 3) + 1.68 (x 2) | | 5 | 0 | 6.1, 1.5, 0.14, 3.1 | GH-C 1440 |
| | | | | | 4 | 3.4, 2.85, 6.93, 4.4 | At planting + foliar |
| | | | | | 11 | 1.7, 0.40, 2.92, 3.0 | |
| | | | | | 18 | 0.83, 0.66, 7.2, 1.5 | |
| Michigan, 1979 | | | | | | | |
| Green Forage | G + EC | 1.46 + 1.68 (x 5) | | 6 | 0 | 27, 26, 18, 23 | GH-C 1440 |
| | | | | | 4 | 7.8, 9.3, 5.5, 8.0 | At planting + foliar |
| | | | | | 11 | 2.2, 1.9, 3.0, 2.2 | |
| | | | | | 18 | 1.3, 1.5, 1.3, 0.65 | |
| Fodder | G + EC | 1.46 (x 3) + 1.68 (x 2) | | 5 | 32 | 1.4, 1.6, 1.9, 3.1 | |
| Fodder | G + EC | 1.46 + 1.68 (x 5) | | 6 | 32 | 1.0, 1.1, 1.5, 1.6 | |
| Grain | G + EC | 1.46 (x 3) + 1.68 (x 2) | | 5 | 32 | <0.01, <0.01, 0.01, 0.01 | |
| Grain | G + EC | 1.46 + 1.68 (x 5) | | 6 | 32 | <0.01, <0.01, <0.01, 0.01 | |
| Mississippi, 1979 | | | | | | | |
| Green forage | G + EC | 1.09 (x 3) + 1.68 (x 2) | | 5 | 0 | 2.2, 1.7, 2.2, 3.2 | GH-C 1440 |
| | | | | | 8 | 1.8, 1.2, 1.9, 1.4 | At planting + foliar |
| | | | | | 11 | 1.1, 1.4, 1.0, 1.4 | |
| | | | | | 17 | 1.5, 1.4., 2.1, 1.6 | |

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| Mississippi, 1979 | | | | | | | |
| Green forage | G + EC | 1.09 + 1.68 (x 5) | | 6 | 0 | 11, 7.8, 9.0, 7.5 | GH-C 1440 |
| | | | | | 8 | 2.9, 4.4, 2.9, 2.9 | At planting + foliar |
| | | | | | 11 | 2.7, 1.6, 2.0, 1.7 | |
| | | | | | 17 | 3.0, 3.4, 3.6, 2.9 | |
| Fodder | G + EC | 1.09 (x 3) + 1.68 (x 2) | | 5 | 31 | 1.7, 1.3, 1.7, 1.5 | |
| Fodder | G + EC | 1.09 + 1.68 (x 5) | | 6 | 31 | 0.87, 1.6, 1.4, 2.0 | |
| Grain | G + EC | 1.09 (x 3) + 1.68 (x 2) | | 5 | 17 | 0.06, 0.02, 0.01, 0.02 | |
| | | | | | 31 | 0.02, 0.02, 0.03, 0.01 | |
| Grain | G + EC | 1.09 + 1.68 (x 5) | | 6 | 17 | 0.03, 0.02, 0.02, 0.02 | |
| | | | | | 31 | 0.02, 0.01, 0.01, 0.01 | |
| Nebraska, 1979 | | | | | | | |
| Green forage | G + EC | G=1.46 + 1.09 (x 2)+ | | | | | GH-C 1440 |
| | | EC=1.68 (x 2) | | 5 | 0 | 2.2, 4.0, 6.3, 1.6 | At planting + foliar |
| Fodder | G + EC | G=1.46 + 1.09 (x 2)+ | | | | | |
| | | EC=1.68 (x 2) | | 5 | 35 | 6.0, 3.6, 7.2, 5.7 | |
| Grain | G + EC | G=1.46 + 1.09 (x 2)+ | | | | | |
| | | EC=1.68 (x 2) | | 5 | 35 | <0.01, <0.01, <0.01, 0.01 | |

[1] LOQ 0.01 mg/kg.
[2] Recoveries 81% to 92% from green forage, 82% to 84% from fodder and 83% to 93% from grain.

Sweet corn. Supervised trials were conducted in Canada and the USA. The results are shown in Tables 87 and 88.

Sweet corn in Ontario, Canada, in 1977 received a single foliar treatment of an EC formulation at 1.12 kg ai/ha at the seedling stage. Samples of kernels + cob with husk removed were taken 73 and 85 days after treatment (Fairbairn and Norton, 1980).

In US trials in 1983 12 to 23 foliar applications of 50W formulation were made to sweet corn at 1.12 kg ai/ha. One additional plot in California was treated with 21 foliar sprays of the EC formulation at the same rate. Samples of the ears (kernel + cob) and green forage were collected for analysis 7-8, 14-15 and 21-22 days after the last application (Wetters *et al.*, 1986).

Multiple ground applications of the EC formulation were made in trials in California, Florida, Georgia, Minnesota and Wisconsin, and aerial applications in Florida and Georgia only. Ground applications were 5 x 1.67 kg ai/ha and aerial 11 x 1.12 kg ai/ha. Samples of ears and forage from each plot were analysed (Nugent and Schotts, 1991).

Table 87. Residues of chlorpyrifos in sweet corn from supervised trials in Canada and the USA.

| Country, year | Application | | | | PHI, days | Residues, mg/kg[1] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Canada | 500 WP | 1.15 | (0.29) | 1 | 70 | | |
| Canada, 1977 | EC | 1.12 | 0.14 | 1 | 73 | <0.01, <0.01, ≤0.01 | GHS-C 13 |
| Grain + cob | | | | | 85 | <0.01, <0.01, <0.01 | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg[1] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-USA | EC | 3.4 broadcast soil incorp pre-plant<br>1.1 directed at cultivation<br>1.7 foliar<br>1.1 foliar (FL and GA)<br>0.5 foliar (DE) | | 1 pre-plant | 35 grain<br>35 fodder<br>14 silage | | Grain PHI is 21 days FL and GA and 7 days DE (for special rates) |
| | 15 G | 2.3 pre-plant, at planting<br>1.1 foliar and post plant | | 1 | | | |
| | 50 WP | 62 g ai/<br>100 kg seeds | | 1 | | | |
| CA, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 12 | 7 | 0.21 | GH-C 1797 |
| | | | | | 14 | 0.11 | |
| DE, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 17 | 7 | 1.3 | GH-C 1797 |
| | | | | | 14 | 0.38, 0.23 | |
| | | | | | 21 | 0.66 | |
| FL, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 17 | 7 | 3.5 | GH-C 1797 |
| | | | | | 14 | 1.2 | |
| | | | | | 21 | 2.0 | |
| FL, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 15 | 7 | 2.9 | GH-C 1797 |
| | | | | | 14 | 1.2 | |
| | | | | | 21 | 0.77 | |
| IL, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 19 | 7 | 1.3 | GH-C 1797 |
| | | | | | 14 | 1.1 | |
| | | | | | 21 | 0.31 | |
| MI, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 14 | 8 | 0.4 | GH-C 1797 |
| | | | | | 15 | 0.11 | |
| | | | | | 22 | 0.04 | |
| OR, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 17 | 7 | 6.27, 6.51, 8.56, 6.03 | GH-C 1797 |
| | | | | | 14 | 3.4 | |
| | | | | | 21 | 3.0 | |
| TX, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 23 | 7 | 1.3 | GH-C 1797 |
| | | | | | 14 | 0.81 | |
| | | | | | 21 | 0.78 | |
| CA, 1983 | | | | | | | |
| Forage | 50 WP | 1.12 | | 21 | 7 | 1.0 | GH-C 1797 |
| | | | | | 14 | 0.24 | |
| | | | | | 21 | 0.48 | |
| CA, 1983 | | | | | | | |
| Forage | EC | 1.12 | | 21 | 7 | 1.8 | GH-C 1797 |
| | | | | | 14 | 0.64 | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg[1] | Reference/ comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| | | | | | 21 | 0.15 | |
| CA, 1990 | | | | | | | |
| Ears | EC | 1.67 | | 5 | 35 | <0.01, <0.01, <0.01, <0.01 | GH-C 2569 |
| Fodder | EC | 1.67 | | 5 | 35 | 0.13, 0.09, 0.15, 0.23 | |
| FL, 1990 | | | | | | | |
| Ears | EC | 1.68 | | 5 | 35 | <0.01, <0.01, <0.01, <0.01 | GH-C 2569 |
| Fodder | EC | 1.68 | | 5 | 35 | 0.06, 0.03, 0.06, 0.04 | |
| Ears | EC | 1.12 | | 11 | 21 | <0.01, <0.01, <0.01, <0.01 | |
| Fodder | EC | 1.12 | | 11 | 21 | 0.05, 0.04, 0.06, 0.16 | |
| GA, 1990 | | | | | | | |
| Ears | EC | 1.68 | | 5 | 35 | <0.01, <0.01, <0.01, <0.01 | GH-C 2569 |
| Fodder | EC | 1.68 | | 5 | 35 | 0.28, 0.08, 1.3, 0.35 | |
| Ears | EC | 1.12 | | 11 | 21 | <0.01, <0.01, <0.01, <0.01 | |
| Fodder | EC | 1.12 | | 11 | 21 | 0.10, 0.14, 0.11, 0.14 | |
| MN, 1990 | | | | | | | |
| Ears | EC | 1.68 | | 5 | 35 | <0.01, <0.01, <0.01, <0.01 | GH-C 2569 |
| Fodder | EC | 1.68 | | 5 | 35 | 0.39, 0.52, 0.68, 0.77 | |
| WI, 1990 | | | | | | | |
| Ears | EC | 1.68 | | 5 | 35 | <0.01, <0.01, <0.01, <0.01 | GH-C 2569 |
| Fodder | EC | 1.68 | | 5 | 35 | 1.6, 1.3, 0.75, 0.26 | |

[1] LOQ 0.01 mg/kg

In trials in six US States in 1972, sweet corn seeds were treated before planting with WP slurry formulations containing 25% or 50% ai at 0.63 to 1.9 g ai/100 kg seeds. Green plant samples were collected at intervals, and kernels, kernels + cobs, cobs + husks and husks were collected at normal harvest (Wetters, 1973). The results are shown in Table 88.

Table 88. Residues of chlorpyrifos in sweet corn grown from treated seed in supervised trials in the USA.

| Location, Year | Application | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | g ai/100kg | No. | | | |
| GAP-USA | WP | 62 | 1 | NA | | |
| FL, 1972 | WP | 190 | 1 | | | GH-C 664 |
| Green plant | | | | 28 | <0.01, 0.01, 0.01, <0.01 | |
| | | | | 42 | 0.01, 0.01, <0.01, <0.01 | |
| | | | | 83 | <0.01, <0.01, <0.01, <0.01 | |
| Kernel + cob | WP | 190 | 1 | 83 | <0.01, <0.01, <0.01, <0.01 | |
| Husks | WP | 190 | 1 | 83 | <0.01, <0.01, <0.01, <0.01 | |
| MS, 1971 | WP | 190 | 1 | | | GH-C 664 |

| Location, Year | Application | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | g ai/100kg | No. | | | |
| Green plant | | | | 28 | <0.01, <0.01, 0.01, 0.01 | |
| | | | | 41 | <0.01, <0.01, <0.01, <0.01 | |
| | | | | 72 | <0.01, 0.01, <0.01, <0.01 | |
| Kernels | WP | 190 | 1 | 72 | <0.01, <0.01, <0.01, 0.01 | |
| Kernels + cobs | WP | 190 | 1 | 72 | <0.01, 0.01, <0.01, <0.01 | |
| Cobs + husk | WP | 190 | 1 | 72 | <0.01, 0.01, <0.01, <0.01 | |
| OR, 1971 | WP | 190 | 1 | | | GH-C 664 |
| Green plant | | | | 48 | <0.01, 0.01, <0.01, <0.01 | |
| | | | | 61 | <0.01, <0.01, <0.01, <0.01 | |
| | | | | 126 | <0.01 | |
| Kernels | WP | 190 | 1 | 126 | <0.01, <0.01, <0.01, <0.01 | |
| Kernels + cob | WP | 190 | 1 | 126 | <0.01, <0.01, <0.01, <0.01 | |
| Cobs + husks | | | | 126 | <0.01, <0.01, <0.01, <0.01 | |
| IL, 1969 | WP | 63 | 1 | | | GH-C 664 |
| Green plant | | | | 80 | <0.01 | |
| | | | | 81 | <0.01 | |
| | | | | 85 | <0.01 | |
| Kernels | WP | 63 | 1 | 80 | <0.01 | |
| | | | | 81 | <0.01 | |
| | | | | 85 | <0.01 | |
| NY, 1971 | | | | | | GH-C 664 |
| Green plant | WP | 190 | 1 | 46 | <0.01 | |
| | | | | 106 | <0.01 | |
| Kernels | WP | 190 | 1 | 106 | <0.01 | |
| Kernels + cobs | WP | 190 | 1 | 106 | <0.01 | |
| Cobs + husks | WP | 190 | 1 | 106 | <0.01 | |
| WI, 1971 | | | | | | GH-C 664 |
| Green plant | WP | 190 | 1 | 29 | <0.01 | |
| | | | | 45 | <0.01 | |
| | | | | 108 | <0.01 | |
| Kernels | WP | 190 | 1 | 108 | <0.01 | |
| IA, 1971 | | | | | | GH-C 664 |
| Green plant | WP | 120 | 1 | 31 | <0.01, <0.01, <0.01 | |
| | | | | 90 | <0.01, <0.01, <0.01 | |
| Green plant | WP | 190 | 1 | 31 | <0.01, <0.01, <0.01 | |
| | | | | 90 | <0.01, <0.01, <0.01 | |

[1] LOQ 0.01 mg/kg. Recoveries averaged 97% from green plants, 94% from kernels, 95% from kernels + cobs, 101% from cobs + husks and 82% from husks, over the range 0.01 to 1.0 mg/kg.

Rice. Supervised trials on rice were conducted in 1998 in Australia, Columbia, the Philippines, Thailand and Vietnam. The results are shown in Tables 89 and 90.

In the trials in Australia, the 500 EC formulation was applied once at 0.45 kg ai/ha 10 days before harvest, and samples of grain and straw taken randomly were analysed (Cowles *et al.*, 1999a).

In two trials in Columbia according to current GAP, chlorpyrifos was applied 3 times to paddy at 0.96 kg ai/ha, followed 15 days later with an application of 0.79 kg ai/ha and another of 0.38 kg ai/ha 20-21 days before harvest. Threshed samples were taken for analysis (Pinheiro and De Vito, 1999).

In the two trials in the Philippines the 3 E formulation at 0.30 kg ai/ha was applied 25 and 40 days after transplanting and 25 days before harvest (Cowles *et al.*, 1999b). In the trials in Vietnam one application of the 30 EC formulation was made 10 days before harvest at 0.42 kg ai/ha (Cowles *et al.*, 1999c). In the trials in Thailand only one application of 40 EC formulation at 0.4 g ai/ha was made 44 days before harvest in one trial and 62 days before harvest in the other (Cowles *et al.*, 1999d).

Table 89. Residues of chlorpyrifos in rice from supervised trials in Australia and Colombia.

| Country, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-Australia | 500 EC | 0.75 | | 10 | | |
| Australia, 1998 | | | | | | |
| Rice grain | 500 EC | 0.45 | 1 | 10 | 0.17 | GHF-P 1790 |
| | | | | | 0.13 | |
| Rice straw, dry weight | 500 EC | 0.45 | 1 | 10 | 1.26 | |
| | | | | | 1.25 | |
| GAP-Columbia | 480 EC | 0.8 | | 15 | | |
| Colombia, 1998 | | | | | | |
| Rice grain | 480EC | 0.96+0.72+ 0.38 | 3 | 20 | 0.09, 0.09, 0.19 | GHB-P 406 |
| | | | | | 0.08,0.08, 0.07 | |

[1] LOQ 0.01 mg/kg in both trials. Recoveries were 92% and 89%, from grain and straw, respectively, in Australia and 81% from grain in Colombia.

Table 90. Residues of chlorpyrifos in rice from supervised trials in the Philippines, Vietnam and Thailand.

| Country, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference/ Comments |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP- Philippines | 300 EC | 0.30 | 3 | 7 | | 160-192 l water/ha |
| Philippines, 1998 | | | | | | |
| Rice grain | 300 EC | 0.30 | 3 | 25 | 0.02 | GHF-P 1791 |
| | | | | | 0.06 | |
| Rice straw, dry weight | 300 EC | 0.30 | 3 | 25 | 0.19 | |
| | | | | | 0.45 | |
| GAP-Vietnam- NONE | | | | | | |
| Vietnam, 1998 | | | | | | |
| Rice grain | 300 EC | 0.42 | 1 | 10 | 0.15 | GHF-P 1792 |
| | | | | | 0.28 | |
| Rice straw, dry weight | 300 EC | 0.42 | 1 | 10 | 1.83 | |
| | | | | | 2.33 | |
| GAP-Thailand | 200 EC | 0.4 | | 7 | | |
| Thailand, 1998 | | | | | | |
| Rice grain | 400 EC | 0.4 | 1 | 44 | <0.01, <0.01 | GHF-P 1793 |
| | | | | 62 | <0.01, <0.01 | |

| Country, year | Application | | | PHI, days | Residues, mg/kg [1] | Reference/ Comments |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| Rice straw, dry weight | 400 EC | 0.4 | 1 | 44 | 0.08, 0.06 | |
| | | | | 62 | 0.17, 0.13 | |

[1] LOQ 0.01 g/kg in all trials. Recoveries were 91% and 96% from grain and straw respectively in all trials.

A trial in India in 1978 was reported. Proposed GAP in India requires 0.1-0.375 ka ai/ha with a 30-day PHI. Chlorpyrifos residues were determined in brown rice grain and straw harvested after treatment with the 20 EC formulation at 0.19 and 0.38 kg ai/ha. Samples were harvested 1, 7, 14 and 21 days after the last application (Leung, 1978). The results are shown in Table 91.

Table 91. Residues of chlorpyrifos in brown rice from supervised trials in India.

| Location, year | Application | | | | PHI, days | Residues, mg/kg [1,2] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-India | 200 EC | 0.38 | 0.08 | | 30 | | PHI proposed . |
| Delhi, 1978 | | | | | | | |
| Grain | 200 EC | 0.19 | | 3 | 1 | 0.62, 0.37, 0.45 | GHF-P 084 |
| | | | | | 7 | 0.28, 0.26, 0.34 | |
| | | | | | 14 | 0.33, 0.17, 0.18 | |
| | | | | | 21 | 0.11, 0.17, 0.08 | |
| Straw | 200 EC | 0.19 | | 3 | 1 | 0.47 0.52, 0.41 | GHF-P 084 |
| | | | | | 7 | 0.23, 0.27, 0.34 | |
| | | | | | 14 | 0.39, 0.31, 0.34 | |
| | | | | | 21 | 0.08, 0.15, 0.11 | |
| 1978 | | | | | | | |
| Grain | 200 EC | 0.38 | | 3 | 1 | 0.77, 0.62, 0.78 | GHF-P 084 |
| | | | | | 7 | 0.45, 0.54, 0.39 | |
| | | | | | 14 | 0.20, 0.34, 0.40 | |
| | | | | | 21 | 0.23, 0.11, 0.25 | |
| | | | | | | | |
| Straw | 200 EC | 0.38 | | 3 | 1 | 0.62, 0.76, 0.56 | GHF-P 084 |
| | | | | | 7 | 0.28, 0.63, 0.42 | |
| | | | | | 14 | 0.27, 0.20, 0.32 | |
| | | | | | 21 | 0.15, 0.25, 0.22 | |

[1] LOQ 0.01 mg/kg. Recoveries were 94% and 89% from grain and straw respectively
[2] Sample storage period from harvest to analysis not indicated.

Sorghum. Supervised trials on sorghum were conducted with EC formulations in Australia, Brazil and the USA. The results are shown in Table 92.

In trials in Australia, sorghum plots were hand-sprayed with EC formulation diluted to 0.05 to 0.1 kg ai/hl at rates of 0.14 and 0.28 kg ai/ha. Heads were randomly picked from each of the treated plots 1 and 7 days after treatment, sealed in paper bags and forwarded for analysis. On receipt, the grain was separated from the heads, ground and stored frozen before analysis (Tucker, 1974).

In two trials in Brazil chlorpyrifos was applied three times at 0.36 or 0.72 kg ai/ha to plants near the final crop stage. Sampling was made at 21 days after the last application (Balderrama and De Vito, 1994).

In a trial in Kansas, USA, a broadcast band application of the 15 G formulation at 2.2 kg ai/ha at planting was followed by three broadcast foliar sprays of the EC formulation at 0.28 kg ai/ha, made at 3-day intervals with the last application 63 days before harvest. Samples of green forage were collected at the silage stage, 14 days after the last application, and grain and fodder at normal harvest (Miller and McKellar, 1986b).

In trials in 1993 in Kansas and Texas, plants received a total of 1.68 kg ai/ha of chlorpyrifos with separate plots for two different treatment modes at each location the first being three foliar applications of 0.56 kg ai/ha at 44, 36-37 and 30 days before harvest and the second two foliar applications of 0.56 and 1.12 kg ai/ha at 67 and 60 days before harvest respectively. Samples of sorghum grain, green forage and fodder were analysed (Robb, 1994). The water content of the forage and fodder was not reported.

Sorghum plots were treated with three aerial or ground applications of an EC formulation applied at 0.56 kg ai/ha, in trials in the mid-United States in 1975-6. The applications were made at two-week intervals with the last application two weeks before harvest. Grain and stover samples were analysed (Miller and Ervick, 1977).

Trials at three locations in the USA were with three applications of chlorpyrifos at 0.28 kg ai/ha at 3-day intervals. Samples of green plant were collected at 0, 7, and 14 days after the last treatment and at the silage stage if the sorghum was of a silage variety. Samples of dry plant and grain were taken at normal harvest (Wetters and Dishburger, 1976).

Sorghum grown in Kansas and Texas was treated in two ways with an EC formulation at both locations: three foliar applications of 0.56 kg ai/ha with samples collected 30 days after the last application, and two foliar applications of 0.56 and 1.12 kg ai/ha with samples collected 60 days after the second application (Robb, 1991b).

Table 92. Residues of chlorpyrifos in sorghum from supervised trials in Australia, Brazil and the USA.

| Country, year, sample | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-Australia | 500 EC | 0.75 | | 2 | 2 | | |
| Australia, 1974 | EC | 0.05 | | 1 | 1 | 0.87 | GHF-P 019 |
| Grain | | | | | 7 | 0.10 | |
| Australia, 1974 | EC | 0.10 | | 1 | 1 | 1.0 | GHF-P 019 |
| Grain | | | | | 7 | 0.17 | |
| GAP-Brazil | 480 EC | 0.36 | 2 | | 21 | | |
| Mogi Mirim, Brazil, 1992 (BR301) | EC | 0.36 | | 3 | 21 | 0.06, 0.05, 0.07 | GHB-P 188 |
| | EC | 0.72 | | 3 | 21 | 0.07, 0.17, 0.06 | GHB-P 188 |
| GAP-USA | 480 EC | 1.1 | | | 30 @ 0.56<br>60 @ 1.1 | | 1.7 kg ai/ha/season max |
| | 15 G | 2.0 | | 1 | | | At planting |
| KS, 1985 | | | | | | | |
| Green forage | G + EC | 2.24 + 0.28(x3) | | 4 | 14 | 0.48, 0.63, 0.42, 0.55 | GH-C 1813 |
| Fodder | G + EC | 2.24 + 0.28(x3) | | | 63 | 0.15, 0.35, 0.2,0.25 | |
| Grain | G + EC | 2.24 + 0.28(x3) | | | 63 | 0.02, 0.02', 0.02, 0.02 | |

| Country, year, sample | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| KS, 1993 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 30 | 0.20 | GH-C 3226 |
| | EC | 0.56 + 1.12 | | 2 | 60 | <0.01 | |
| Green forage | EC | 0.56 | | 3 | 30 | 0.14 | |
| | EC | 0.56 + 1.12 | | 2 | 60 | 0.04 | |
| Fodder | EC | 0.56 | | 3 | 30 | 1.3 | |
| | EC | 0.56 + 1.12 | | 2 | 60 | 0.34 | |
| TX, 1993 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 30 | 0.02 | GH-C 3226 |
| | | 0.56 + 1.12 | | 2 | 60 | <0.01, <0.01 | |
| Green forage | EC | 0.56 | | 3 | 30 | 0.03 | |
| | EC | 0.56 + 1.12 | | 2 | 60 | 0.01 | |
| Fodder | EC | 0.56 | | 3 | 30 | 0.17 | |
| | EC | 0.56 + 1.12 | | 2 | 60 | 0.08 | |
| IL, 1976 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 14 | 0.03, 0.08, 0.06, 0.03 | GH-C 998 |
| Stover | EC | 0.56 | | 3 | 14 | 0.07, 0.05, 0.05, 0.05 | |
| KS, 1976 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 15 | 0.32, 0.58, 0.42, 0.28 | GH-C 998 |
| Stover | EC | 0.56 | | 3 | 15 | 0.70, 0.2, 0.7, 0.83 | |
| TX, 1976 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 14 | 0.25, 0.27, 0.31, 0.28 | GH-C 998 |
| Stover | EC | 0.56 | | 3 | 14 | 2.0, 2.3, 2.2, 1.15 | |
| NE, 1975 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 14 | 0.27, 0.27, 0.17, 0.13 | GH-C 998 |
| Stover | EC | 0.56 | | 3 | 14 | 1.1, 0.90, 0.91, 1.0 | |
| MS, 1975 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 14 | 0.31, 0.41, 0.57, 0.70 | GH-C 998 |
| Stover | EC | 0.56 | | 3 | 14 | 1.2, 1.7, 2.5, 2.4 | |
| MS, 1974 | | | | | | | |
| Green plant | EC | 0.28 | | 3 | 0 | 2.1, 2.4, 1.8, 4.1 | GH-C 900 |
| | | | | | 7 | 0.24, 0.46, 0.52, 0.38 | |
| | | | | | 14 | 0.21, 0.17, 0.17, 0.17 | |
| Fodder | EC | 0.28 | | 3 | 47 | 0.46, 1.2, 0.35, 0.76 | |
| Grain | EC | 0.28 | | 3 | 47 | 0.02, 0.01, 0.01, 0.01 | |
| TX, 1974 | | | | | | | |
| Green plant | EC | 0.28 | | 3 | 0 | 2.2, 2.0, 3.0, 3.4 | GH-C 900 |
| | | | | | 7 | 0.27, 0.16, 0.27, 0.36 | |
| | | | | | 14 | 0.06, 0.02, 0.03, 0.04 | |
| Fodder | EC | 0.28 | | 3 | 38 | 0.02, 0.02, 0.02, 0.10 | |
| | | | | | | | |
| Grain | EC | 0.28 | | 3 | 38 | <0.01, <0.01, <0.01, 0.01 | |
| MS, 1974 | | | | | | | |
| Green plant | EC | 0.28 | | 3 | 8 | 0.70, 0.76, 0.98, 0.91 | GH-C 900 |
| | | | | | 15 | 0.63, 0.66, 0.56, 0.69 | |
| Forage | EC | 0.28 | | 3 | 29 | 0.24, 0.15, 0.26, 0.34 | |

| Country, year, sample | Application | | | | PHI, days | Residues, mg/kg [1] | Reference/ Comments |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| Fodder | EC | 0.28 | | 3 | 72 | 0.17, 0.29, 0.25, 0.38 | |
| Grain | EC | 0.28 | | 3 | 72 | <0.01, <0.01, <0.01, 0.01 | |
| KS, 1990 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 29 | 0.27, 0.20, 0.26, 0.26 | GH-C 2555 |
| | EC | 1.12 + 0.56 | | 2 | 60 | 0.09, 0.09, 0.17, 0.21 | |
| Fodder | EC | 0.56 | | 3 | 29 | 0.28, 0.39, 0.38, 0.36 | |
| | EC | 1.12 + 0.56 | | 2 | 60 | 0.06, 0.06, 0.04, 0.15 | |
| TX, 1990 | | | | | | | |
| Grain | EC | 0.56 | | 3 | 30 | 0.04, 0.03, 0.03, 0.04 | GH-C 2555 |
| | EC | 1.12 + 0.56 | | 2 | 60 | 0.01, 0.01, 0.03, 0.01 | |
| Fodder | EC | 0.56 | | 3 | 30 | 0.01, 0.01, 0.01, 0.01 | |
| | EC | 1.12 + 0.56 | | 2 | 60 | 0.01, 0.02, 0.01, 0 01 | |

[1] LOQ 0.01 mg/kg. Recoveries 83% to 92% from grain, 84% to 87% from fodder and 82% to 93% from green forage at the 95% confidence limit of the mean

Wheat. In residue trials in Brazil in 1992, 1993 and 1997 an EC formulation was applied once or three times at PHIs of 17 or 72 days (Balderrama, 1994), once or twice at rates up to 1.4 kg ai/ha (Balderrama and Matos, 1994j), or in single applications from 0.24 to 1.4 kg ai/ha (Do Amaral and De Vito, 1999). The results are shown in Table 93.

Table 93. Residues of chlorpyrifos in wheat grain in Brazil. EC formulation.

| Location, Year | Application | | | PHI, days | Residue, mg/kg | Reference |
|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | | |
| Brazil GAP | 0.72 | | 2 | 21 | | |
| Guarapuava (BR-28) 1992 | 0.48 | | 1 | 72 | 0.02 | GHB-P 197 |
| | 0.72 | | 3 | 17 | 0.30 | |
| | 0.96 | | 1 | 72 | 0.02 | |
| | 1.4 | | 3 | 17 | 0.35 | |
| Cruz Alta (BR-34) 1993 | 0.72<br>0.19 | | 2 | 20 | <0.01 | GHB-P 200 |
| | 1.4<br>0.38 | | 2 | 20 | 0.02 | |
| | 0.48 | | 1 | 29 | <0.01 | |
| | 0.96 | | 1 | 29 | 0.01 | |
| Mogi Mirim (IAC-5) 1997 | 0.24 | | 1 | 21 | <0.01 | GHB-P 411 |
| | 0.48 | | 1 | 21 | 0.03 | |
| | 0.72 | | 1 | 21 | 0.04 | |
| | 0.96 | | 1 | 21 | 0.06 | |
| | 1.4 | | 1 | 21 | 0.17 | |

In a trial in the UK in 1992 three applications of the EC formulation containing 480 g ai/l were applied to winter wheat plants at various growth stages at rates of 0.72 kg ai/ha for the 1st and 2nd applications and 0.34 kg ai/ha for the 3rd. Grain and straw were sampled at harvest 18 days after application (Khoshab and Berryman, 1994f).

In two trials on winter wheat in the UK in 1995 three treatments with the 480 EC formulation were applied at growth stages Zd 12 or Zd 25 (autumn applications), 14-27 (winter applications) and 83-85 (summer applications) at 0.72 kg ai/ha for the 1st and 2nd applications and 0.34 kg ai/ha for the 3rd. Whole plants were sampled at intervals, and grain and straw at harvest 24 or 31 days after the last application (Khoshab, 1996).

In two trials on winter wheat in the UK in 1994-1995, chlorpyrifos WG formulation containing 750 g ai/kg was applied three times at 0.72 g ai/ha for the 1st and 2nd applications and 0.34 kg ai/ha for the 3rd, and the wheat sampled at intervals from 0 to 24 or 31 days after treatment (Portwood and Williams, 1996).

In a trial in Germany in 1995 plots of winter wheat were treated with three applications of the 480 EC formulation at 0.72 kg ai/ha for the 1st and 2nd and 0.34 kg ai/ha for the 3rd, and immature plants were sampled immediately before the 3rd application and as soon as the spray had dried. Further plant samples were taken 6 and 11 days later, and grain and straw were sampled at harvest 17 days after the last application (Teasdale, 1997).

Table 94. Residues of chlorpyrifos in wheat from supervised trials in the UK and Germany.

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1,2,3] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-UK | 480 EC | 0.72 spring<br>0.72 winter | 0.36 | 3<br>3 | 14<br>14 | | |
| UK, 1992 | EC | | | | | | |
| Straw | | 0.72 + 0.72 + 0.34 | | 3 | 18 | 0.07 | GHE-P 3720 |
| Grain | | | | | 18 | <0.01 | |
| UK, 1995 | | | | | | | |
| Whole plant | EC | 0.72 + 0.72 + 0.34 | | 3 | 0 | 2.6 | GHE-P 5204 |
| Whole plant | | | | | 8 | 0.64 | |
| Whole plant | | | | | 16 | 0.50 | |
| Straw | | | | | 24 | 0.48 | |
| Grain | | | | | 24 | 0.02 | |
| UK, 1995 | | | | | | | |
| Whole plant | EC | 0.72 + 0.72 + 0.34 | | 3 | 0 | 9.7 | GHE-P 5204 |
| Whole plant | | | | | 7 | 1.7 | |
| Whole plant | | | | | 12 | 1.81 | |
| Straw | | | | | 31 | 1.1 | |
| Grain | | | | | 31 | 0.11 | |
| UK, 1995 | WG | | | | | | |
| Whole plant | | 0.72 + 0.72 + 0.34 | | 3 | 0 | 3.1 | GHE-P 5471 |
| Whole plant | | | | | 8 | 1.0 | |
| Whole plant | | | | | 16 | 0.97 | |
| Grain | | | | | 24 | 0.03 | |
| Straw | | | | | 24 | 0.88 | |
| UK, 1995 | WG | | | | | | |
| Whole plant | | 0.72 + 0.72 + 0.34 | | 3 | 0 | 5.0 | GHE-P 5471 |
| Whole plant | | | | | 7 | 0.89 | |

| Country, year | Application | | | | PHI, days | Residues, mg/kg [1,2,3] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| Whole plant | | | | | 12 | 0.63 | |
| Grain | | | | | 31 | 0.03 | |
| Straw | | | | | 31 | 0.48 | |
| GAP-Germany NONE | | | | | | | |
| Germany, 1995 | | | | | | | |
| Whole plant | | 0.72 + 0.72 | | 2 | 0[4] | <0.01 | GHE-P 4834 |
| Whole plant | | 0.72 + 0.72 + 0.34 | | 3 | 0 | 3.7 | |
| Whole plant | | | | | 6 | 1.1 | |
| Whole plant | | | | | 11 | 1.0 | |
| Grain | | | | | 17 | 0.04 | |
| Straw | | | | | 17 | 0.76 | |

[1] All results are averages of duplicate analyses
[2] LOQ 0.01 mg/kg
[3] Recoveries were 86% to 92% from whole plant, 76% to 91% from grain and 82 to 102% from straw
[4] Immediately before 3rd application

In the USA multiple aerial or single ground applications of EC formulation were made at 0.56 or 1.12 kg ai/ha per application to winter or spring wheat plots at various locations (total 1.12, 1.68, or 2.8 kg ai/ha per season). Grain and straw samples were collected 25-31 days after the last of the multiple applications, and green forage at 7 and 14-15 days after the five single ground applications at 1.12 kg ai/ha (Norton and Wetters, 1983). The moisture content of the green forage was not reported.

Samples of grain and straw were collected from 10 trials in the main wheat growing areas of the USA. Chlorpyrifos was applied by air twice, at 1.12 kg ai/ha 41 to 57 days and at 0.56 kg ai/ha 15 to 35 days before harvest. Green forage was collected 14 or 15 days after treatment in eight other trials at some of the same locations, in which the EC formulation was applied once at 1.12 kg ai/ha as a broadcast ground application when the plants were 15 to 20 cm tall (Miller and McKellar, 1986c).

In trials in Canada EC was applied once at 0.375 kg ai/ha to each of four wheat plots 7, 16-19, 26-29 and 36-40 days before normal harvest. All the plots were harvested at the same time and only grain samples were collected (McKellar and Ordiway, 1986b).

The results of all the trials are shown in Table 95.

Table 95. Residues of chlorpyrifos in wheat from supervised trials in the USA and Canada.

| Country, year, sample | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-USA | 480 EC | 0.56 | (2.9) | 2 | 28 grain | | |
| | | | | | 14 forage/hay | | |
| IL, 1980 | EC | 1.12 + 1.12 + 0.56 | | 3 | | | |
| Grain | | | | | 25 | 0.01, 0.01, 0.02, 0.02 | GH-C 1639 |
| Straw | | | | | 25 | 0.08, 0.28, 0.28, 0.39 | |
| IL, 1980 | EC | 1.12 + 1.12 + 0.56 | | 3 | | | |
| Grain | | | | | 26 | 0.01, <0.01, <0.01, 0.01 | GH-C 1639 |
| Straw | | | | | 26 | 0.09, 0.05, 0.07, 0.04 | |

| Country, year, sample | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| ID, 1982 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 27 | 0.02, 0.05 | GH-C 1639 |
| Straw | | | | | 27 | 2.1, 1.4 | |
| ID, 1981 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | 0.19 | GH-C 1639 |
| Straw | | | | | 28 | 1.2 | |
| KS, 1980 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 31 | 0.01, 0.01, 0.01, 0.01 | GH-C 1639 |
| Straw | | | | | 31 | 0.32, 0.38, 0.37, 0.48 | |
| KS, 1980 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 27 | 0.01, 0.01, 0.01, 0.01 | GH-C 1639 |
| Straw | | | | | | 0.44, 0.39, 0.42, 0.47 | |
| ND, 1982 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | <0.01 | GH-C 1639 |
| Straw | | | | | 28 | 0.2 | |
| TX, 1981 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | 0.03 | GH-C 1639 |
| Straw | | | | | 28 | 1.20 | |
| OR, 1981 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | 0.23 | GH-C 1639 |
| Straw | | | | | 28 | 4.1, 3.2 | |
| TX, 1981 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | 0.02 | GH-C 1639 |
| Straw | | | | | 28 | 0.63 | |
| TX, 1981 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | 0.02 | GH-C 1639 |
| Straw | | | | | 28 | 0.64 | |
| WA, 1981 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | 0.03, 0.03 | GH-C 1639 |
| Straw | | | | | 28 | 0.23, 0.21 | |
| CA, 1982 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 7 | 18, 1.4, 1.6, 2.6 | GH-C 1639 |
| Green forage | | | | | 14 | 0.58, 0.51, 0.28, 0.31 | |
| IL, 1981 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 7 | 1.1, 0.71, 0.63, 1.1 | GH-C 1639 |
| Green forage | | | | | 14 | 0.17, 0.08, 0.04, 0.07 | |
| MI, 1981 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 7 | 0.96, 1.1, 0.54, 0.91 | GH-C 1639 |
| Green forage | | | | | 15 | 0.11, 0.06, 0.08, 0.08 | |
| OK, 1981 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 7 | 3, 15., 9.1, 2.8 | GH-C 1639 |
| Green forage | | | | | 14 | 1.6, 15, 9.0, 12 | |
| TX, 1981 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 7 | 4.9, 5.9, 5.0, 4.0 | GH-C 1639 |
| Green forage | | | | | 14 | 1.6, 1.7, 2.0, 2.1 | |

| Country, year, sample | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| AL, 1983 | EC | 1.12 +.0.56 | | 2 | | | |
| Grain | | | | | 35 | 0.01 | GH-C 1790 |
| Straw | | | | | 35 | 0.03 | |
| CA, 1984 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | 0.05, 0.07, 0.08, 0.06 | GH-C 1790 |
| Straw | | | | | 28 | 2.6, 1.9, 2.4, 2.3 | |
| CO, 1983 | EC | 1.12 + 0.05 | | 2 | | | |
| Grain | | | | | 30 | -- | GH-C 1790 |
| Straw | | | | | 30 | 0.16 | |
| MN, 1984 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 27 | <0.01 | GH-C 1790 |
| Straw | | | | | 27 | 2.2 | |
| MS, 1983 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 27 | <0.01, <0.01, <0.01, 0.01 | GH-C 1790 |
| Straw | | | | | 27 | 0.53, 0.51, 0.85, 0.96 | |
| MO, 1983 | EC | 1.12 + .0.56 | | 2 | | | |
| Grain | | | | | 29 | 0.01 | GH-C 1790 |
| Straw | | | | | 29 | 0.11 | |
| NE, 1983 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 15 | <0.01 | GH-C 1790 |
| Straw | | | | | 15 | 1.9 | |
| NY, 1983 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 28 | 0.01 | GH-C 1790 |
| Straw | | | | | 28 | 0.60 | |
| NC, 1983 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 23 | -- | GH-C 1790 |
| Straw | | | | | 23 | 0.01 | |
| OH, 1984 | EC | 1.12 + 0.56 | | 2 | | | |
| Grain | | | | | 29 | <0.01 | GH-C 1790 |
| Straw | | | | | 29 | 0.48 | |
| AL, 1983 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 14 | 0.71 | GH-C 1790 |
| CO, 1983 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 14 | 0.31 | GH-C 1790 |
| MN, 1984 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 14 | 0.05 | GH-C 1790 |
| MS, 1983 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 14 | 0.30, 0.33, 0.30, 0.28 | GH-C 1790 |
| MO, 1983 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 15 | 0.05 | GH-C 1790 |
| NE, 1983 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 14 | 0.29 | GH-C 1790 |
| NY, 1983 | EC | 1.12 | | 1 | | | |
| Green forage | | | | | 14 | 0.15 | GH-C 1790 |
| NC, 1983 | EC | 1.12 | | 1 | | | |

| Country, year, sample | Application | | | | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| Green forage | | | | | 14 | 0.87 | GH-C 1790 |
| GAP- Canada | EC 480 g/l | 0.58 | | 5 | 60 | | |
| Canada, 1985 | EC | 0.38 | | 1 | 7 | 0.12, 0.13, 0.11, 0.09 | GH-C 1804 |
| Grain | | | | | 16 | 0.10, 0.09, 0.13, 0.10 | |
| | | | | | 26 | 0.03, 0.03, 0.02, 0.03 | |
| | | | | | 36 | 0.01, 0.01, 0.01, 0.02 | |
| Canada, 1985 | EC | 0.36 | | 1 | 29 | 0.01, 0.02, 0.02, 0.03 | GH-C 1804 |
| Grain | | | | | 39 | 0.01, <0.01, <0.01, 0.01 | |
| | | | | | 49 | <0.01, <0.01, <0.01, <0.01 | |
| Canada, 1985 | EC | 0.36 | | 1 | 7 | 0.11, 0.12, 0.13, 0.17 | GH-C 1804 |
| Grain | | | | | 19 | 0.03, 0.02, 0.01, 0.01 | |
| | | | | | 29 | 0.01, 0.02, 0.01, 0.01 | |
| | | | | | 40 | <0.01, <0.01, 0.01, <0.01 | |

[1] LOQ 0.01 mg/kg. Recoveries 91% to 93% from forage, 90% to 91% from grain and 85% to 88% from straw

Grasses

Alfalfa. Several supervised trials were conducted in the USA from 1982 to 1995, with the results shown in Tables 96 and 97.

In four trials at different locations in 1982, EC formulation was applied as a single foliar spray with ground equipment at 0.28 kg ai/ha 7-8 days before cutting, and samples of green forage were collected 7-8 days and cured alfalfa hay 9-14 days after application (Miller, 1983b).

In trials in California, Michigan and Illinois in 1984 an EC formulation was applied to plots according to two treatment schedules. In the first applications were made before each of four separate cuttings during the season: 28 days before the first cutting, 21 days before the second, 14 days before the third and 7 days before the fourth at 1.12, 1.12, 0.56 and 0.28 kg ai/ha respectively. The second set of plots was treated with single applications of 0.56 kg ai/ha seven days before each of four cuttings during the season. Samples of green forage were collected seven days after the fourth application and cured hay 7-14 days later (Wetters, 1990b).

In a trial in California in 1985 EC formulation was applied at 1.12 kg ai/ha and seed samples were collected after 15 and 22 days (McKellar and Ordiway, 1986c).

In California, Illinois and Michigan alfalfa plots were treated with single applications of chlorpyrifos at 1.12 kg ai/ha, followed by post-plant applications at 1.12 kg ai/ha before each of 3 or 4 cuttings. Four replicate samples of green forage and hay cut 21 and 28 or 29 days after the last application respectively were analysed. The hay samples were field- or greenhouse-cured for 7-8 days (Deubelbeis, 1990).

Chlorpyrifos EC was applied to plots of alfalfa in Illinois, Michigan and Mississippi at 0.80 kg ai/ha 12 to 14 days before each of four cuttings. Samples of green forage and cured hay were collected from the fourth cutting only (Wetters and Ervick, 1990b).

Four applications of chlorpyrifos EC formulation corresponding to four cuttings of the crop were made in California, Illinois, New York and Wisconsin. Residues were determined in samples taken at the fourth cutting after 0.56 kg ai/ha had been applied 7 days, and 1.12 kg/ha 7 or 14 days before harvest (Robb and Schotts, 1993a).

In trials in Colorado, Ohio, North Dakota and Washington, the EC and WG formulations were compared at both 0.28 and 1.12 kg ai/ha. The WG was applied 21 days and the EC 7 days before each cutting. Three cuttings were obtained from Colorado and four from the other sites, and forage and hay samples were taken each time. Only samples from the last cutting were analysed except in North Dakota, where those from the third cutting were also analysed (McCormick and Bormett, 1996b).

Table 96. Residues of chlorpyrifos in alfalfa from supervised trials in the USA (1982-1986).

| Country, year, sample | Application | | | PHI, days | Chlorpyrifos, mg/kg[1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA | 480 EC | 0.28 | 4 | 7 | | One application per cutting cycle. |
| | | 0.56 | 4 | 14 | | |
| | | >0.56-1.12 | 4 | 21 | | |
| | 240 EC | 0.56 | 4 | 4 | | CA and AZ only. One application per cutting cycle. |
| California, 1982 | | | | | | |
| Green forage | EC | 0.28 | 1 | 7 | 0.22, 0.16, 0.05, 0.15 | GH-C 1610 |
| Hay | EC | 0.28 | 1 | 7+7 | 0.74, 0.45, 0.92, 0.40 | |
| Illinois, 1982 | | | | | | |
| Green forage | EC | 0.28 | 1 | 7 | 0.23, 0.20, 0.10, 0.25 | GH-C 1610 |
| Hay | EC | 0.28 | | 7+3 | 0.33, 0.29, 0.43, 0.32 | |
| Michigan, 1982 | | | | | | |
| Green forage | EC | 0.28 | 1 | 8 | 0.09, 0.20, 0.17, 0.10 | GH-C 1610 |
| Hay | EC | 0.28 | | 8+5 | 0.40, 0.43, 0.37, 0.36 | |
| Mississippi, 1982 | | | | | | |
| Green forage | EC | 0.28 | 1 | 7 | 0.65, 0.45, 0.57, 0.35 | GH-C 1610 |
| Hay | EC | 0.28 | | 7+2 | 1.8, 1.3, 1.4, 1.3 | |
| California, 1984 | | | | | | |
| Green forage | EC | 1.12(x2) + 0.56 + 0.28 | 4 | 7 | 0.65, 0.81, 0.72, 0.90 | GH-C 2334 |
| Hay | EC | 1.12(x2) + 0.56 + 0.28 | | 7 + 14 | 1.3, 1.3, 1.2, 1.2 | |
| Illinois, 1984 | | | | | | |
| Green forage | EC | 1.12(x2) + 0.56 + 0.28 | 4 | 7 | 0.40, 0.49, 0.62, 0.62 | GH-C 2334 |
| Hay | EC | 1.12(x2) + 0.56 + 0.28 | | 7 + 10 | 1.2, 1.0, 0.92, 0.95 | |
| Michigan, 1984 | | | | | | |
| Green forage | EC | 1.12(x2) + 0.56 + 0.28 | 4 | 7 | 0.85, 1.3, 1.1, 1.0 | GH-C 2334 |
| Hay | EC | 1.12(x2) + 0.56 + 0.28 | | 7 + 7 | 2.3, 2.3, 2.6, 3.1 | |
| California, 1984 | | | | | | |
| Green forage | EC | 0.56 | 4 | 7 | 2.1, 2.5, 2.0, 2.4 | GH-C 2334 |
| Hay | EC | 0.56 | | 7 + 14 | 4.8, 5.7, 6.4, 4.6 | |
| Illinois, 1984 | | | | | | |
| Green forage | EC | 0.56 | 4 | 7 | 0.79, 0.72, 0.69, 0.95 | GH-C 2334 |
| Hay | EC | 0.56 | | 7 + 10 | 1.7, 1.9, 2.3, 2.5 | |

| Country, year, sample | Application | | | PHI, days | Chlorpyrifos, mg/kg[1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| Michigan, 1984 | | | | | | |
| Green forage | EC | 0.56 | 4 | 7 | 2.2, 2.3, 1.8, 2.0 | GH-C 2334 |
| Hay | EC | 0.56 | | 7 + 7 | 7.0, 7.2, 6.8, 6.2 | |
| California, 1985 | EC | 1.12 | 1 | 15 | 0.06, 0.05, 0.07, 0.12 | GH-C 1803 |
| Seed | | | | 22 | 0.46, 0.23, 0.23, 0.37 | |
| California, 1985 | | | | | | |
| Green forage | EC | 1.12 + 1.12/cutting | 5 | 21 | 0.61, 0.77, 0.89, 0.85 | GH-C 2288 |
| Cured hay | EC | 1.12+ 1.12/cutting | | 21 + 8 | 1.3, 1.4, 1.2, 1.7 | |
| Illinois, 1985 | | | | | | |
| Green forage | EC | 1.12 + 1.12/cutting | 4 | 21 | 2.7, 1.7, 1.6, 1.5 | GH-C 2288 |
| Cured hay | EC | 1.12 + 1.12/cutting | | 21 + 7 | 1.1, 0.99, 0.32, 0.18 | |
| Michigan, 1985 | | | | | | |
| Green forage | EC | 1.12 + 1.12/cutting | 4 | 21 | 3.6, 4.4, (4.9), 4.0 | GH-C 2288 |
| Cured hay | EC | 1.12 + 1.12/cutting | | 21 + 8 | 19, (23), 21, 20 | |
| Illinois, 1986 | | | | | | |
| Green forage | EC | 0.80 | 5 | 14 | 0.32, 0.32, 0.29, 0.32 | GH-C 2294 |
| Cured hay | EC | 0.80 | | 14 + 10 | 0.52, 0.55, 0.57, 0.56 | |
| Michigan, 1986 | | | | | | |
| Green forage | EC | 0.80 | 4 | 12 | 9.3, 10, 10, 10 | GH-C 2294 |
| Hay | EC | 0.80 | | 12 + 2 | 21, 29, 30, 31 | |
| Mississippi, 1986 | | | | | | |
| Green forage | EC | 0.80 | 4 | 14 | 0.63, 0.83, 0.77, 0.31 | GH-C 2294 |
| Cured hay | EC | 0.80 | | 14 + 2 | 1.7, 1.9, 2.2, 2.0 | |

[1] LOQ 0.05 mg/kg in all trials. Recoveries were 94% to 97% from forage and 94% to 107% from hay

Table 97: Residues of chlorpyrifos in alfalfa from supervised trials in the USA, 1991-1995.

| Location, year | Application | | | PHI, days | Chlorpyrifos, mg/kg[1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA | 480 EC | 0.28 | 4 | 7 | | One application per cutting cycle |
| | | 0.56 | 4 | 14 | | |
| | | >0.56-1.12 | 4 | 21 | | |
| | 240 EC | 0.56 | 4 | 4 | | One application per cutting cycle. AZ and CA only |
| California, 1991 | | | | | | |
| Green forage | EC | 0.56 | 4 | 7 | 1.0, 0.48, 0.75, 0.51 | GH-C 2752R |
| | EC | 1.12 | 4 | 7 | 2.9, 3.0, 1.4, 4.3 | |
| | EC | 1.12 | 4 | 14 | 0.31, 0.21, 0.30, 0.27 | |
| Cured Hay | EC | 0.56 | 4 | 7 | 1.8, 2.6, 2.1, 2.2 | |
| | EC | 1.12 | 4 | 7 | 3.1, 5.1, 4.3, 4.6 | |
| | EC | 1.12 | 4 | 14 | 1.4, 1.2, 1.5, 2.3 | |
| Illinois, 1991 | | | | | | |
| Green forage | EC | 0.56 | 4 | 7 | 1.6, 1.5, 1.8, 2.2 | GH-C 2752R |
| | EC | 1.12 | 4 | 7 | 5.7, 4.4, 5.0, 5.5 | |

| Location, year | Application | | | PHI, days | Chlorpyrifos, mg/kg[1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| | EC | 1.12 | 4 | 14 | 1.8, 1.7, 1.7, 2.3 | |
| Illinois, 1991 | | | | | | GH-C 2752R |
| Cured hay | EC | 0.56 | 4 | 7 | 5.7, 8.4, 7.4, 8.2 | |
| | EC | 1.12 | 4 | 7 | 19, 21, 21, 26 | |
| | EC | 1.12 | 4 | 14 | 8.7, 7.8,10, 9.4 | |
| New York, 1991 | | | | | | |
| Green forage | EC | 0.56 | 4 | 7 | 4.0, 3.3, 3.9, 3.5 | GH-C 2752R |
| | EC | 1.12 | 4 | 7 | 11, 11, 13, 14 | |
| | EC | 1.12 | 4 | 14 | 8.3, 7.6, 8.0, 8.3 | |
| Cured hay | EC | 0.56 | 4 | 7 | 16, 17, 16, 15 | GH-C 2752R |
| | EC | 1.12 | 4 | 7 | 33, 25, 28, 25 | |
| | EC | 1.12 | 4 | 14 | 30, 37, 28, 21 | |
| Wisconsin, 1991 | | | | | | |
| Green forage | EC | 0.56 | 4 | 7 | 5.3, 5.3, 5.0, 5.2 | GH-C 2752R |
| | EC | 1.12 | 4 | 7 | 9.3, 14, 11, 6.7 | |
| | EC | 1.12 | 4 | 14 | 2.8, 5.9, 4.0, 2.6 | |
| Cured hay | EC | 0.56 | 4 | 7 | 9.2, 17, 18, 13 | GH-C 2752R |
| | EC | 1.12 | 4 | 7 | 37, 51, 41, 24 | |
| | EC | 1.12 | 4 | 14 | 5.1, 16, 9.2, 17 | |
| Colorado, 1995 | | | | | | |
| Green forage | EC | 0.28 | 3 | 8 | 0.32, 0.45 | GH-C 4198 |
| | WG | 0.28 | 3 | 8 | 0.19, 0.30 | |
| | EC | 1.12 | 3 | 23 | 0.06, 0.05 | |
| | WG | 1.12 | 3 | 23 | 0.04, 0.06 | |
| Hay | EC | 0.28 | 3 | 8 | 1.0, 0.84 | GH-C 4198 |
| | WG | 0.28 | 3 | 8 | 1.2, 1.2 | |
| | EC | 1.12 | 3 | 23 | 0.45, 0.31 | |
| | WG | 1.12 | 3 | 23 | 0.23, 0.28 | |
| North Dakota, 1995 | | | | | | |
| Green forage | EC | 0.28 | 3 | 7 | 0.43, 0.40 | GH-C 4198 |
| | WG | 0.28 | 3 | 7 | 0.42, 0.33 | |
| | EC | 1.12 | 3 | 21 | 0.27, 0.18 | |
| | WG | 1.12 | 3 | 21 | 0.08, 0.08 | |
| Hay | EC | 0.28 | 3 | 7 | 0.46, 0.22 | GH-C 4198 |
| | WG | 0.28 | 3 | 7 | 0.56, 0.59 | |
| | EC | 1.12 | 3 | 21 | 0.43, 0.78 | |
| | WG | 1.12 | 3 | 21 | 0.27, 0.35 | |
| North Dakota, 1995 | | | | | | |
| Green forage | EC | 0.28 | 4 | 9 | 1.4, 1.2 | GH-C 4198 |
| | WG | 0.28 | 4 | 9 | 1.5, 1.1 | |
| | EC | 1.12 | 4 | 23 | 4.3,(5.6) | |
| | WG | 1.12 | 4 | 23 | 2.2, 0.12 | |
| Hay | EC | 0.28 | 4 | 9 | 2.1, 2.3 | |
| | WG | 0.28 | 4 | 9 | 2.0, 1.5 | |
| | EC | 1.12 | 4 | 23 | (12,) 11 | |

| Location, year | Application | | | PHI, days | Chlorpyrifos, mg/kg[1] | Reference/ Comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| | WG | 1.12 | 4 | 23 | 1.8, 1.7 | |
| Ohio, 1995 | | | | | | |
| Green forage | EC | 0.28 | 4 | 7 | 0.31, 0.38 | GH-C 4198 |
| | WG | 0.28 | 4 | 7 | 0.57, 0.55 | |
| | EC | 1.12 | 4 | 21 | 0.17, 0.17 | |
| | WG | 1.12 | 4 | 21 | 0.43, 0.41 | |
| Hay | EC | 0.28 | 4 | 7 | 0.40, 0.66 | |
| | WG | 0.28 | 4 | 7 | 0.90, 0.93 | |
| | EC | 1.12 | 4 | 21 | 0.36, 0.31 | |
| | WG | 1.12 | 4 | 21 | 0.59, 0.63 | |
| Washington, 1995 | | | | | | |
| Green forage | EC | 0.28 | 4 | 7 | 0.18, 0.21 | GH-C 4198 |
| | WG | 0.28 | 4 | 7 | 0.09, 0.12 | |
| | EC | 1.12 | 4 | 21 | <0.01, <0.01 | |
| | WG | 1.12 | 4 | 21 | 0.01, 0.01 | |
| Hay | EC | 0.28 | 4 | 7 | 0.64, 0.42 | |
| | WG | 0.28 | 4 | 7 | 0.40, 0.37 | |
| | EC | 1.12 | 4 | 21 | 0.04, 0.02 | |
| | WG | 1.12 | 4 | 21 | 0.02, 0.02 | |

[1] LOQ 0.01 mg/kg. Recoveries 84% to 90% from forage and 84% to 89% from hay

In the trials in Michigan (GH-C 2288; GH-C 2294) residues were much higher from the Midland site than from other locations. According to the manufacturer, a possible explanation is that the Midland alfalfa was very sparse and small because of the colder climate and shorter growing season in mid-Michigan, where only the first two cuttings produce adequate forage for commercially acceptable hay. In an unusual year a third cutting may be taken, but very seldom are four commercially acceptable cuttings taken. Fourth cutting samples are therefore not typical of Michigan cultural practices and the residues are not representative.

Tree nuts

Almonds. Plots of trees in Fresno Country, California, USA, were foliar-sprayed three times with an EC formulation and once or twice with a soil application about the trees (Wetters and McKellar, 1989). Almonds were shaken from the trees 14 days after the last application and left to dry on the ground for 7 days. Kernels were separated from the hulls and both were analysed for chlorpyrifos and TCP. In another trial in California, dormant spray applications were made and almonds collected at normal harvest were analysed for chlorpyrifos plus TCP, determined as TCP (Wetters and Dishburger, 1975). The results are shown in Table 98.

Table 98. Residues of chlorpyrifos and TCP in almond kernels and hulls from the foliar and ground application of an EC formulation in the USA.

| Location, Year | Application | | | PHI, days | Residue, mg/kg | | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Chlorpyrifos | TCP | |
| USA GAP | 2.2 foliar<br>4.5 ground | | 3 | 14 | | | |

| Location, Year | Application | | | PHI, days | Residue, mg/kg | | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | Chlorpyrifos | TCP | |
| Fresno, California 1987 | 2.2 foliar<br>4.5 ground<br>2.2 foliar<br>2.2 foliar<br>4.5 ground | 0.06<br>1.2 | 5 | 14 | 0.010 kernel<br>2.3 hull | 0.056 kernel<br>2.2 hull | GH-C 2180. Foliar and ground applications were on the same day. Foliar 3700 l/ha. Ground 370 l/ha. Plot = 1 tree. |
| Fresno, California 1987 | 2.2 foliar<br>2.2 foliar<br>2.2 foliar<br>4.5 ground | 0.06<br><br><br>1.2 | 4 | 14 | ≤0.01 kernel<br>1.9 hull | 0.043 kernel<br>1.4 hull | GH-C 2180. Foliar and ground applications were on the same day. Foliar 3700 l/ha. Ground 370 l/ha. Plot = 1 tree |
| Fresno, California 1987 | 2.2 foliar<br>4.5 ground<br>2.2 foliar<br>2.2 foliar | 0.06<br>1.2 | 4 | 14 | 0.010 kernel<br>3.2 hull | 0.066 kernel<br>2.8 hull | GH-C 2180. Foliar and ground applications were on the same day. Foliar 3700 l/ha. Ground 370 l/ha. Plot = 1 tree |
| US GAP | Dormant<br>EC: 2.2<br>WP: 4.5 | <br>0.06<br>0.24 | <br>1<br>1 | <br>-<br>- | Total | | |
| Fresno, California 1972 | 2.2 | 0.06 | 1 | 204 | ≤0.05 nut<br>≤0.05 hull | | GH-C 783. EC formulation. Total residue determined. |
| | 2.2 | 0.06 | 1 | 191 | 0.05 nut<br>0.07 hull | | GH-C 783. EC formulation. Total residue determined. |
| | 2.2 | 0.06 | 1 | 185 | ≤0.05 nut<br>≤0.05 hull | | GH-C 783 EC formulation. Total residue determined |
| | 2.2 | 0.06 | 1 | 185 | ≤0.05 nut<br>≤0.05 hull | | GH-C 783 WP formulation. Total residue determined. |

Pecans. Five foliar sprays of an EC formulation were applied to pecan trees in Mississippi, USA (Wetters, 1989). Samples were harvested 28 days after the last application and stored frozen. Before analysis, the kernels were separated from the shells. The recovery of chlorpyrifos from fortified kernels at 0.01 mg/kg (method ACR 73.5.S1) was 96±7%, n = 9, and that of TCP at 0.05 mg/kg (method ACR 71.19R.S6) was 110 ± 15%, n = 4. The conditions and results are shown in Table 99.

In earlier trials in 1982 in the USA, 5-8 foliar applications of an EC formulation were made to pecan trees at 0.12 kg ai/hl. Kernels were analysed for total residue, chlorpyrifos plus TCP (Miller, 1983c).

The results are shown in Table 99.

Table 99. Residues of chlorpyrifos and TCP in pecans from the application of EC to trees in the USA.

| Location, year, (Variety) | Application | | | PHI, days | Residue, mg/kg[1] | | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | chlorpyrifos | TCP | |
| USA GAP | 2.2 foliar<br>1.1 foliar | (0.23)<br>0.12 | 5<br>5 | 28<br>28 | | | 480 g/l EC<br>120 g/l EC |
| Helm, Mississippi 1985 (Stewart) | 1.1 | | 5 | 28 | ≤0.01 | <0.05 | GH-C 2195. 50 or 100 l/ha. Plot = 1.6 ha. |
| Leland, Mississippi 1985 (Stewart) | 1.1 | | 5 | 28 | ≤0.01 | <0.05 | GH-C 2195. 50 or 100 l/ha. Plot = 1.6 ha |
| | | | | | Total | | |

| Location, year, (Variety) | Application | | | PHI, days | Residue, mg/kg[1] | | Reference/ Comment |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | No. | | chlorpyrifos | TCP | |
| South Carolina 1982 | 0.05 kg/tree | 0.12 | 5 | 30 | <0.05[3] | | GH-C 1652 Plot=2 trees |
| New Mexico 1982 | 0.11 kg/tree | 0.12 | 5 | 31 | <0.05 | | GH-C 1652 Plot= 1 tree Duplicate plots. |
| Texas 1982 | 4 | 0.12 | 5 | 31 | <0.05 | | GH-C 1652 Plot=1 tree |
| Mississippi 1982 | 0.014 kg/tree | 0.12 | 5 | 30 | <0.05 | | GH-C 1652 Plot=3 trees |
| Mississippi 1982 | 0.023 kg/tree | 0.12 | 5 | 30 | <0.05 | | GH-C 1652 Plot=3 trees |
| Mississippi 1982 | 0.023 kg/tree | 0.12 | 5 | 30 | <0.05 | | GH-C 1652 Plot=2 trees |
| Georgia 1981 | 0.057 kg/tree (0.7) | 0.15 | 6 | 14 | <0.05 | | GH-C 1652 Plot= 8 trees with 12 trees/A |
| Georgia 1982 | 0.028 kg/tree (0.34) | 0.08 | 8 | 21 | 0.17 | | GH-C 1652 Plot=8 trees with 12 trees/A |

[1] LOQ 0.01 mg/kg for chlorpyrifos, 0.05 mg/kg for TCP. Recoveries averaged 94% from GH-C 2195 and 82% from GH-C 1652.

Walnuts. In supervised trials in California, USA, three applications of chlorpyrifos were made either as a dilute or concentrated spray, at 2.2 to 2.8 kg ai/ha. Samples were collected at normal harvest, 12-17 days after the last application. Kernels were analysed for total residues (chlorpyrifos + TCP) measured as TCP (Miller, 1982). The results are shown in Table 100.

Table 100: Residues of chlorpyrifos in walnut kernels from supervised trials in California, USA.

| Country, year | Application | | | | PHI, days | Residues, mg/kg[1,2] | Reference |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | kg ai/hl | No. | | | |
| GAP-USA | 50 WP | 2.24 | 0.24 | 1 | 14 | | |
| | 4 EC | 2.24 | 0.24 | 2 | 14 | | |
| 1977 | WP+EC | 2.2 | 0.06 | 3 | 12 | <0.05, <0.05, <0.05, <0.05 | GH-C 1579 |
| | WP+EC | 2.2 | 0.60 | 3 | 12 | <0.05, <0.05, <0.05, <0.05 | |
| 1979 | EC | 2.2 | 0.06 | 3 | 14 | <0.05, <0.05, <0.05, <0.05 | GH-C 1579 |
| | EC | 2.2 | 0.06 | 3 | 14 | <0.05, <0.05, <0.05, <0.05. | |
| 1979 | EC | 2.8 | 0.06 | 3 | 17 | <0.05, <0.05, <0.05, <0.05 | GH-C 1579 |
| 1979 | EC | 2.2 | 0.06 | 3 | 14 | <0.05, <0.05 | GH-C 1579 |
| 1972 | EC | 2.2 | 0.06 | 1 | 216 | <0.05, <0.05, <0.05, <0.05 | GH-C 783 |

[1] Total residue (chlorpyrifos + TCP), measured as TCP
[2] LOQ 0.05 mg/kg for total residue. Recoveries averaged 83% to 88%

Oil seeds

Cotton seed. Supervised trials were conducted in Brazil and the USA. In a trial in Brazil in 1992 the EC formulation of chlorpyrifos was applied to plants three times at 0.96 or 1.92 kg ai/ha or once at 0.72 or 1.44 kg ai/ha. Samples were taken 21 days after the last application from 0.96 and 1.92 kg ai/ha plots and 106 days after application from 0.72 and 1.44 kg ai/ha plots (Balderrama and Matos, 1994h).

In a separate trial in Brazil, an EC formulation of chlorpyrifos was applied to plants twice at 0.96 or 1.92 kg ai/ha and once at 0.72 or 1.44 kg ai/ha. Samples were taken from the 0.96 and 1.92 kg treatments 21 days and from the 0.72 and 1.44 kg treatments 85 days after the last application, kept 2 days at 30°C in an oven and 1 day exposed to sunlight, then packed and stored at -20°C before analysis (Balderrama and Matos, 1994i).

In trials in the USA in Arizona, California and Mississippi, ten foliar applications of an EC formulation at 0.56 kg ai/ha were made with aerial or ground equipment, the last 30-33 or 40-41 days before harvest. Samples were harvested at random and ginned, and the seeds stored frozen until analysis. The recoveries of chlorpyrifos from cotton seed fortified at 0.01 mg/kg were 84 ± 5%, n = 5 (Doom, 1987).

In trials in Mississippi EC was applied nine times as a foliar spray at 1.12 or 2.24 kg ai/ha and samples collected at intervals from 0 to 14 days after the last application were ginned and the seed analysed (McKellar and Dishburger, 1974).

In trials in Texas, Mississippi and South Carolina, plants were treated with multiple foliar applications of an EC formulation, using ground and aerial equipment, at 1.12 or 0.28 and 1.12 kg ai/ha. Samples were taken at normal harvest (McKellar, 1975).

In a trial in 1986 in California chlorpyrifos was applied 5 times at 1.12 kg ai/ha. Samples collected 14 days after the last application were mechanically delinted and the seeds collected from the gin chute and stored frozen before analysis (Wetters, 1987b).

Table 101 summarizes the results of all the trials.

Table 101. Residues of chlorpyrifos in cotton seed from supervised trials in Brazil and the USA. Seed analysed.

| Location/Year | Application | | | PHI, days | Residues, mg/kg [1] | Reference/ comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-Brazil | 480 EC | 0.96 | 3 | 21 | | |
| Brazil, 1992 | EC | 0.72 | 1 | 106 | 0.01 | GHB-P 195 |
| | EC | 0.96 | 3 | 21 | 0.07 | |
| | EC | 1.44 | 1 | 106 | 0.02 | |
| | EC | 1.92 | 3 | 21 | 0.44 | |
| Brazil, 1992 | EC | 0.72 | 1 | 85 | <0.01 | GHB-P 196 |
| | EC | 0.96 | 2 | 21 | 0.02 | |
| | EC | 1.44 | 1 | 85 | 0.02 | |
| | EC | 1.92 | 2 | 21 | 0.04 | |
| GAP-USA | 480 EC | 1.1 | 6 | 14 | | |
| Mississippi, 1973 | EC | 1.12 | 9 | 0 | 1.4, 0.94, 0.97, 1.1 | GH-C 739 |
| | | | | 3 | 0.28, 0.37, 0.48, 0.47 | |
| | | | | 7 | 0.02, 0.02, 0.02 | |
| | | | | 14 | 0.16, 0.02, 0.03 | |
| Mississippi, 1973 | EC | 2.24 | 9 | 0 | 3.0, 3.7, 1.8, 2.7 | GH-C 739 |
| | | | | 3 | 0.63, 0.54, 0.66, 1.1 | |
| | | | | 7 | 0.07, 0.04, 0.07 | |
| | | | | 14 | 0.05, 0.14, 0.04 | |
| MS, 1980 | EC | 0.56 | 2 | 41 | 0.01, <0.01, 0.01, 0.01 | GH-C 1658 |
| CA, 1982 | EC | 0.56 | 4 | 14 | 0.08, 0.08, 0.17, 0.25 | GH-C 1658 |
| | | | | 21 | 0.08, 0.05, 0.07, 0.04 | |

| Location/Year | Application | | | PHI, days | Residues, mg/kg [1] | Reference/ comment |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| MS, 1982 | EC | 0.56 | 8 | 14 | 0.01, 0.01, 0.03, 0.03 | GH-C 1658 |
| | | | | 21 | 0.03, 0.03, 0.01, 0.02 | |
| MS, 1982 | EC | 0.56 | 8 | 14 | 0.03, 0.06, 0.06, 0.02 | GH-C 1658 |
| | | | | 21 | 0.12, 0.06, 0.14, 0.15 | |
| CA, 1986 | EC | 1.12 | 5 | 14 | 0.12, 0.17, 0.15, 0.10 | GH-C 1893 |
| Arizona, 1985 | EC | 0.56 | 10 | 30<br>40 | <0.01<br><0.01 | GH-C 1879<br>2.3 l/ha |
| Arizona, 1985 | EC | 0.56 | 10 | 30<br>40 | <0.01<br><0.01 | GH-C 1879.<br>Aerial 28 l/ha |
| Arizona, 1985 | EC | 0.56 | 10 | 40 | 0.010 | GH-C 1879.<br>Aerial 28 l/ha |
| Arizona, 1985 | EC | 0.56 | 10 | 30<br>40 | 0.019<br><0.01 | GH-C 1879.<br>2.3 l/ha |
| California, 1985 | EC | 0.56 | 10 | 30<br>40 | <0.01<br><0.01 | GH-C 1879.<br>Aerial 47 l/ha |
| California, 1985 | EC | 0.56 | 10 | 31<br>41 | 0.040<br>0.086 | GH-C 1879<br>Aerial 28 l/ha |
| Mississippi, 1985 | EC | 0.56 | 10 | 33<br>40 | 0.018<br>0.014 | GH-C 1879<br>2.3 l/ha |
| Texas, 1974 | EC | 0.28 x 4<br>1.12 x 13 | 17 | 6 | 0.062, 0.037, 0.015, 0.029 | GH-C 840 |
| Mississippi, 1974 | EC | 0.28 x 4<br>1.12 x 12 | 16 | 15 | 2.0, 0.92, 1.0, 1.3 | GH-C 840 |
| Mississippi, 1974 | EC | 1.12 | 11 | 31 | <0.01, <0.01, <0.01, <0.01 | GH-C 840 |
| South Carolina, 1974 | EC | 0.28 x 4<br>1.12 x 13 | 17 | 34 | 0.13, 0.11, 0.094 | GH-C 840 |
| South Carolina, 1974 | EC | 1.12 | 13 | 38 | 0.055, 0.053, 0.023, 0.038 | GH-C 840 |
| Texas. 1974 | EC | 0.28 x 4<br>1.12 x 12 | 16 | 18 | 0.12, 0.078, 0.11, 0.098 | GH-C 840 |

[1] LOQ 0.01 mg/kg and mean recoveries 81% for Brazilian trials. LOQ 0.01 mg/kg and recoveries 84% to 87% for US trials.

Peanuts. Residue trials were reported from Georgia and Mississippi, USA, in which a granular formulation was applied at planting and again 30 to 72 days before harvest, and in some trials one or two EC foliar applications between the granular treatments. Peanuts were harvested at normal maturity, separated into hulls and kernels, and analysed for chlorpyrifos and TCP by method ACR 84.4.

Table 102. Residues of chlorpyrifos and TCP in peanuts from soil and foliar applications in the USA.

| Location, (Variety) Year | Application | | | PHI, days | Residue, mg/kg [1] | | Reference/comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | chlorpyrifos | TCP | |
| USA GAP | G | 2.2 broadcast 15 oz/1000 ft row | 2 | 21 | | | All uses not to exceed 4.5 kg ai/ha/season |
| | EC | 2.2 pre-plant (soil); foliar | | 21 | | | |

| Location, (Variety) Year | Application | | | PHI, days | Residue, mg/kg [1] | | Reference/comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | chlorpyrifos | TCP | |
| Donalson-ville, Georgia (Florunner) 1986 | G<br>EC<br>G | 2.2<br>0<br>2.2 | 2 | 30 | 0.017, 0.011, 0.016, 0.014 | 0.084, 0.084, 0.093, 0.11 | GH-C 2665<br>The first G application was in an 8 inch band at 15 oz/1000 ft., 36 inch centre. The second G application was an 8 inch band at the rate o f 22.5 oz/1000ft row. |
| | G<br>EC<br>G | 2.2<br>0<br>3.4 | 2 | 30 | 0.032, 0.029, 0.034, 0.038 | 0.13, 0.11, 0.16, 0.18 | |
| | G<br>EC<br>G | 2.2<br>1.1<br>2.2 | 3 | 30 | 0.037, 0.053, 0.034, 0.028 | 0.23, 0.22, 0.11, 0.22 | EC was broadcast, 187 l/ha |
| | G<br>EC<br>G | 2.2<br>0<br>2.2 | 2 | 58 | 0.020, 0.019, 0.035, 0.30 | 0.16, 0.10, 0.10, 0.20 | |
| | G<br>EC<br>G | 2.2<br>0<br>3.4 | 2 | 58 | 0.040, 0.044, 0.050, 0.045 | 0.16, 0.20, 0.17, 0.23 | |
| Wayside, Mississippi (Florunner) 1986 | G<br>EC<br>G | 2.0<br>0<br>2.0 | 2 | 41 | 0.007, 0.010, 0.008, 0.005 | 0.12, 0.11, 0.094, 0.058 | GH-C 2665.<br>First G application at 15 oz/1000ft of row, 40 in centre. |
| | G<br>EC<br>G | 2.0<br>0<br>3.1 | 2 | 41 | 0.007, 0.011, 0.009, 0.005 | 0.10, 0.11, 0.13, 0.072 | Second G application at a rate of 22.5 oz/1000ft row, 40 inch centres. |
| | G<br>EC<br>G | 2.0<br>1.1<br>2.0 | 3 | 41 | 0.006, 0.004, 0.009, 0.004 | 0.086, 0.052, 0.078, 0.068 | EC was broadcast |
| | G<br>EC<br>G | 2.0<br>0<br>2.0 | 2 | 72 | 0.004, 0.002, 0.004, 0.003 | 0.12, 0.13, 0.12, 0.19 | |
| | G<br>EC<br>G | 2.0<br>0<br>3.1 | 2 | 72 | 0.004, 0.005, 0.005, 0.002 | 0.19, 0.13, 0.20, 0.11 | |
| North Carolina 1973 | EC<br>EC<br>G | 0.28<br>0.28<br>2.24 | 2<br>2<br>1 | | | | GH-C 1071 |
| Green forage | | | | 0<br>8<br>15 | 16, 21, 17, 19<br>8.6, 8.2, 8.4, 8.8<br>4.1, 5.4, 5.8, 5.6 | | |
| Kernels | | | | 15 | 0.01, 0.01, <0.01, 0.01 | | |
| Hay | | | | 15 | 5.1, 5.8, 6.3, 4.2 | | |
| Virginia 1974 | EC<br>EC<br>G | 0.28<br>1.12<br>2.24 | 2<br>2<br>1 | | | | GH-C 1071 |
| Green forage | | | | 0<br>6<br>14 | 74, 42, 56, 52<br>15, 17, 20, 13<br>6.0, 7.2, 5.6, 8.6 | | |

| Location, (Variety) Year | Application | | | PHI, days | Residue, mg/kg [1] | | Reference/comment |
|---|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | chlorpyrifos | TCP | |
| Kernels | | | | 14<br>44 | 0.01, 0.01,<br>0.02, 0.01<br>0.01, 0.01,<br>0.01, 0.01 | | |
| Hay | | | | 36 | 5.5, 5.0, 3.6,<br>5.6 | | |
| Georgia<br>1974 | EC<br>EC<br>G | 0.28<br>1.12<br>2.24 | 2<br>2<br>1 | | ` | | GH-C 1071 |
| Kernels | | | | 18 | <0.01, <0.01,<br><0.01, <0.01 | | |
| Hay | | | | 18 | <0.01, <0.01,<br><0.01, <0.01 | | |
| Georgia<br>1975 | EC<br>EC<br>G | 0.28<br>1.12<br>2.24 | 2<br>2<br>1 | | | | GH-C 1071 |
| Kernels | | | | 27 | <0.01, <0.01,<br><0.01, <0.01 | | |
| Hay | | | | 27 | 0.01, 0.01,<br>0.01, 0.01 | | |
| Mississippi<br>1974 | EC<br>EC<br>G | 0.28<br>1.12<br>2.24 | 2<br>2<br>1 | | | | GH-C 1071 |
| Kernels | | | | 27 | <0.01, <0.01,<br><0.01, <0.01 | | |
| Hay | | | | 27 | 1.0 | | |

[1] LOQ 0.01 mg/kg.

Sunflowers. In two supervised trials in the USA chlorpyrifos EC formulation was applied as a band-over-row treatment to plants at cracking to the 4th leaf stage of growth at the broadcast equivalent of 5 or 10 kg ai/ha. Samples of seeds were taken at normal harvest 77 days after treatment (Wetters, 1979).

In four US trials samples of seed and forage were collected after soil incorporation of the 15G formulation at planting at 1.5 kg ai/ha followed by three to five applications of EC at 0.56 to 1.68 kg ai/ha as a broadcast post-emergence spray using aerial or ground equipment. Samples of seed were collected at harvest, 44 to 75 days after the last application, and of forage at the early seed development stage (Miller, 1980c).

Sunflowers in North Dakota were treated with three applications of EC at 1.6 kg ai/ha, and samples collected 42 days after the last application (Robb, 1991c).

In a 1993 trial sunflowers were treated with 3 applications of chlorpyrifos at the maximum label rate of 1.68 kg ai/ha, and samples of whole seeds collected and processed. Residues of chlorpyrifos were determined in forage, seed and hulls (Turner, 1994).

In trials in Michigan, North Dakota and Minnesota in 1979 and 1980 four or six applications of chlorpyrifos 15 G and EC formulations were made to plots of sunflowers. The former was applied at planting, incorporated into the soil at 1.5 kg ai/ha, and the latter twice post-emergence to the leaves at 1.68 kg ai/ha 4 to 8 weeks after planting. The four-treatment schedule concluded with one further application and the six-treatment with three additional applications of EC at 0.56 kg ai/ha 7 to 22 days

apart. Seeds were sampled 69 to 71 days after the fourth application and 42 to 46 days after the sixth (Miller, 1981b).

Sunflowers in Canada were treated with a single foliar application of an EC formulation at the 4- to 6-leaf stage at 1.12 kg ai/ha 97 days before harvest, and residues were determined in the seed (Fairbairn *et al.*, 1980).

The results are shown in Table 103.

Table 103: Residues of chlorpyrifos in sunflowers from supervised trials in the USA and Canada.

| Location, year, sample | Application | | | PHI, days | Residues, mg/kg[1,2] | Reference/ Comments |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| GAP-USA | 480 EC | 2.2 pre-plant<br>1.7 foliar | 1<br>3 | <br>42 | | 5.0 kg ai/ha/season |
| | 15G | 2.4 for 18 in row spacing. | 1 | NA | | At planting |
| MN, 1978 | | | | | | |
| Seed | EC | 1.68 | 1 | 77 | <0.01, <0.01, <0.01, <0.01 | GH-C 1180 |
| | EC | 3.36 | 1 | 77 | <0.01, <0.01, <0.01, <0.01 | |
| ND, 1993 | | | | | | |
| Forage | EC | 1.68 | 3 | 42 | 0.09 | GH-C 3239 |
| Seed | EC | 1.68 | 3 | 42 | 0.05 | |
| Hulls | EC | 1.68 | 3 | 42 | 0.15 | |
| ND, 1990 | | | | | | |
| Seed | EC | 1.68 | 3 | 42 | 0.02[3],0.04, 0.04, 0.09[3] | GH-C 2683 |
| MN, 1979 | | | | | | |
| Seed | G + EC | 1.34 + 1.68 (x2) + 0.56 | 4 | 59 | 0.02, 0.03, 0.03, 0.02 | GH-C 1371 |
| | G + EC | 1.34 + 1.68 (x2) + 0.56 (x3) | 6 | 45 | 0.05, 0.05, 0.02, 0. 04 | |
| Forage | G + EC | 1.34+1.68 (x2)+0.56 | 4 | 27 | 2.1 | |
| | G + EC | 1.34 + 1.68 (x2) + 0.56 (x3) | 6 | 13 | 3.9 | |
| MS, 1979 | | | | | | |
| Seed | G + EC | 1.5 + 1.68 (x2) + 0.56 | 4 | 65 | 0.02[3], 0.01, 0.03, 0.02 | GH-C 1371 |
| | G + EC | 1.5 + 1.68 (x2) + 0.56 (x3) | 6 | 44 | 0.04, 0.05[3], 0.06, 0.03 | |
| NE, 1979 | | | | | | |
| Seed | G + EC | 1.5 + 1.68 (x2) + 0.56 | 4 | 67 | 0.05, 0.05, 0.03[3], 0.03 | GH-C 1371 |
| | G + EC | 1.5 + 1.68 (x2) + 0.56 (x3) | 6 | 46 | 0.12, 0.12[3], 0.16, 0.11[3] | |
| ND, 1979 | | | | | | |
| Seed | G + EC | 1.5 + 1.68 (x2) + 0.56 | 4 | 75 | 0.04 | GH-C 1371 |
| | G + EC | 1.5 + 1.68 (x2) + 0.56 (x3) | 6 | 44 | 0.10[3] | |
| MI, 1980 | | | | | | |
| Seed | G + EC | 1.5 + 1.68 (x2) + 0.56 | 4 | 69 | 0.04, 0.04, 0.05, 0.01 | GH-C 1468 |
| | G + EC | 1.5 + 1.68 (x2) +0.56 (x3) | 6 | 42 | 0.07, 0.07, 0.07, 0.13 | |
| ND, 1980 | | | | | | |
| Seed | G + EC | 1.5 + 1.68 (x2) + 0.56 | 4 | 71 | 0.06, 0.02, 0.04, 0.01 | GH-C 1468 |
| | G + EC | 1.5 + 1.68 (x2) + 0.56 (x3) | 6 | 46 | 0.03, 0.02, 0.01[3], 0.01[3] | |
| MN, 1979 | | | | | | |
| Seed | G + EC | 1.8 + 1.68 (x2) + 0.56 (x3) | 6 | 45 | 0.03 | GH-C 1468 |
| GAP-Canada | 480 EC | 0.576 | 1 | 42 | | |
| Canada, 1977 | | | | | | GHS-C 11 |

| Location, year, sample | Application | | | PHI, days | Residues, mg/kg[1,2] | Reference/ Comments |
|---|---|---|---|---|---|---|
| | Form. | kg ai/ha | No. | | | |
| Seeds | EC | 1.12 | 1 | 97 | | |

[1] LOQ 0.01 mg/kg
[2] Recoveries 78% to 92% from seeds
[3] Average of duplicate analyses

Seeds for beverages

Coffee. In a trial on the foliar application of an EC formulation to plants in Brazil three treatments were made at 0.72 or 1.4 kg ai/ha and beans sampled 21 days after the final application (Pinto and Matos, 1994). The peel was mechanically separated from the bean (grain).

In a field trial in Brazil in 1994-1995 the EC formulation was applied 3 times to plants at 0.72 and 1.44 kg ai/ha and samples were taken 21 days after the last application (Catta-Preta and Rampazzo, 1997). The beans were processed into roasted beans and instant coffee.

In further trials in Brazil, trees at three different locations were treated at 0.72 kg ai/ha three times during the growing season, with the last application 7 days before normal harvest. Samples of beans were collected 7, 14, 21 and 35-36 days after the last application. Following commercial practice, the beans were de-pulped and sun-dried before analysis (Miller, 1981c).

In field trials in Columbia an EC formulation was applied three times to berry-containing plants at 2.0 or 4.0 l/ha (0.96 kg ai/ha and 1.92 kg ai/ha respectively). Samples were collected at intervals from 0 to 28 days, shelled, soaked in water and dried in the sun. The green beans were stored frozen and sub-samples were processed (Catta-Preta and Rampazzo, 1999).

In a trial in Tanzania during 1986, an EC was applied at rates up to 1.92 kg ai/ha. Samples of beans were taken 1, 8, 15, 22 and 29 days after application and some were processed before analysis (Day, 1987b).

The results are shown in Table 104.

Table 104. Residues of chlorpyrifos in coffee beans from the foliar application of an EC formulation.

| Location, (Variety) Year | Application | | PHI, days | Residue, mg/kg[1] | Reference/ comment |
|---|---|---|---|---|---|
| | kg ai/ha | No. | | | |
| Brazil GAP | 0.72 | 2 | 21 | | |
| Campinas, Brazil (Catuai)<br>1992-1993 | 0.72 | 3 | 21 | 0.03 bean (green)<br>0.39 peel | GHB-P 201.<br>400 l/ha |
| | 1.4 | 3 | 21 | 0.11 bean (green)<br>1.0 peel | |
| Mogi Mirim, Brazil (Catuai)<br>1994-1995 | 0.70 | 3 | 21 | 0.03 bean (green) | GHB-P310<br>400 l/ha |
| | 1.4 | 3 | 21 | 0.12 bean (green) | |
| Brazil<br>1979 | 0.72 | 3 | 7<br>14<br>21<br>36 | 0.01 bean (green)<br>0.01<br>0.01<br>0.01 | GH-C 1462 |
| Brazil<br>1979 | 0.72 | 3 | 7<br>14<br>21<br>35 | 0.01 bean (green)<br>0.02<br>0.01<br>0.01 | GH-C 1462 |

| Location, (Variety) Year | Application | | PHI, days | Residue, mg/kg[1] | Reference/ comment |
|---|---|---|---|---|---|
| | kg ai/ha | No. | | | |
| Brazil<br>1979 | 0.72 | 3 | 7<br>14<br>21<br>35 | 0.01 bean (green)<br>0.02<br>0.01<br>0.01 | GH-C 1462 |
| GAP- Columbia | 1.4 | 3 | 21 | | 480 EC |
| Columbia<br>1999 | 0.96 | 3 | 0<br>2<br>7<br>14<br>21<br>28 | 1.1, 2.0, 0.84, 2.3<br>0.29, 0.77, 0.58, 0.58<br>0.1, 0.29, 0.26, 0.38<br>0.11, 0.17, 0.14, 0.23<br>0.05, 0.08, 0.15, 0.05<br>0.04, 0.04, 0.05, 0.07<br>bean (green) | GHB-P 413 |
| | 1.92 | 3 | 0<br>2<br>7<br>14<br>21<br>28 | 2.1, 2.9, 4.0, 3.4<br>0.85, 2.3, 0.60, 1.7<br>0.72, 1.4, 0.68, 0.79<br>0.31, 0.60, 0.44, 0.79<br>0.22, 0.44, 0.43, 0.38<br>0.08, 0.20, 0.28, 0.18<br>bean (green) | |
| GAP-Tanzania | 0.96 | | 7 | | |
| Tanzania<br>1986 | 0.96<br>1.44<br>1.92 | | 8<br>8<br>8 | 0.04<br>0.13<br>0.08<br>green bean | GHE-P 1737 |
| Tanzania<br>1986 | 0.96<br>1.44<br>1.92 | | 15<br>15<br>15 | 0.07<br>0.11<br>0.06<br>green bean | GHE-P 1737 |
| Tanzania<br>1986 | 0.96<br>1.44<br>1.92 | | 22<br>22<br>22 | 0.07<br>0.08<br>0.08<br>green bean | GHE-P 1737 |

[1] LOQ 0.01 mg/kg for GH-C 1462, 0.02 mg/kg for GHB-P 201 and GHE-P 1737. Recoveries 87-88% for GHB-P 201 and GHE-P 1737.

## Animal feeding studies

Feeding studies were carried out on cattle, pigs and chickens to determine the potential for residues of chlorpyrifos in meat, milk and eggs. The results are shown in Tables 105 to 110. No corrections were made for recoveries or residues in control samples.

Cattle (Dishburger *et al.*, 1972a). Eighteen Hereford cross-bred heifers were subdivided into 6 groups of 3 according to body weight (158-243 kg). Each group was penned together and allowed to share a conditioning ration containing 50% concentrate and 50% roughage for 36 days. The feed was then changed to 75% concentrate and 25% roughage and doses of chlorpyrifos were administered in capsules by balling gun each day at the equivalent of 0, 3, 10, 30 or 100 ppm in the diet, calculated from each group's average total dry matter intake of the concentrate and roughage. The cattle were slaughtered at the end of the 30-day period without withdrawal and samples of muscle, liver, kidneys, and omental and subcutaneous fat were collected for residue determination. All the cattle, including the control group, gained from 2 to 34 kg during the treatment period. Three other cattle were dosed with the equivalent of 100 ppm chlorpyrifos in the diet for 30 days and then samples of omental fat were collected by surgical biopsy at weekly intervals for 5 weeks.

The results are shown in Tables 105 and 106, and indicate that residues of chlorpyrifos were mainly in the fat, where they ranged from <0.01-0.04 mg/kg in the 3 ppm group to 2.0-4.2 mg/kg in the 100 ppm group with no withdrawals for 30 days. After withdrawal the residues of chlorpyrifos in the omental fat decreased steadily to <0.01-0.03 mg/kg.

Table 105. Residues in the tissues of cattle dosed with chlorpyrifos for 30 days.

| Dose, ppm in diet | Chlorpyrifos, mg/kg[1,2,3] | | | | | | Reference |
|---|---|---|---|---|---|---|---|
| | Muscle | Liver | Kidney | Omental fat | Renal fat | Subcutaneous fat | |
| 0 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | GH-C 566 |
| | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | |
| | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | |
| 3 | <0.01 | <0.01 | <0.01 | 0.01 | 0.01 | 0.01 | GH-C 566 |
| | <0.01 | <0.01 | <0.01 | 0.04 | 0.03 | 0.03 | |
| | <0.01 | <0.01 | <0.01 | 0.01 | 0.01 | <0.01 | |
| 10 | 0.02 | 0.02 | <0.01 | 0.10 | 0.14 | 0.15 | GH-C 566 |
| | <0.01 | <0.01 | <0.01 | 0.07 | 0.09 | 0.06 | |
| | <0.01 | <0.01 | <0.01 | 0.10 | 0.13 | 0.07 | |
| 30 | 0.01 | <0.01 | <0.01 | 0.38 | 0.41 | 0.18 | GH-C 566 |
| | 0.01 | 0.01 | 0.01 | 0.75 | 0.99 | 0.51 | |
| | 0.02 | <0.01 | 0.01 | 0.31 | 0.42 | 0.23 | |
| 100 | 0.11 | 0.01 | 0.02 | 2.6 | 3.1 | 3.1 | GH-C 566 |
| | 0.19 | 0.02 | 0.01 | 2.4 | 4.2 | 3.8 | |
| | 0.29 | <0.01 | <0.01 | 2.0 | 2.4 | 2.5 | |

[1] LOQ 0.01 mg/kg
[2] Recoveries averaged 86% from muscle, 78% from liver, 74% from kidney, 88-90% from fat
[3] Analyses were also conducted for TCP and the oxygen analogue of chlorpyrifos. The latter was found only in two omental fat samples from cattle fed at the 100 ppm level, at the LOQ, 0.01 mg/kg.

Table 106. Residues of chlorpyrifos in omental fat of cattle during the withdrawal phase after oral administration of 100 ppm for 30 days.

| Period, days | Chlorpyrifos residues, mg/kg[1,2,3] | | Reference |
|---|---|---|---|
| | Individual cow | Average | |
| 0 | 2.6, 2.4, 2.0 | 2.3 | GH-C 566 |
| 7 | 1.0, 0.86, 0.58 | 0.81 | GH-C 566 |
| 14 | 0.59, 0.13, 0.23 | 0.32 | |
| 21 | 0.51, 0.11, 0.08 | 0.23 | |
| 28 | 0.13, 0.06, 0.02 | 0.07 | |
| 35 | 0.03, <0.01 | 0.02 | |

[1] LOQ 0.01 mg/kg
[2] Recoveries averaged 88 to 90%
[3] Linear regression correlation coefficient is 0.8447

Dairy cattle. Three dairy cows were fed complete rations containing chlorpyrifos at 0.3, 1, 3, 10 or 30 ppm for 14 days consecutively at each level. At the end of this time, the fortified feed was withdrawn and the animals fed the basal ration for 14 days. A total of 16.3 kg of feed was given to each cow daily, half at each milking. Any feed not eaten between milkings was removed and weighed. All of the feed was

consumed most of the time. Milk samples from each cow were taken at intervals by combining equal volumes from the evening and the following morning milkings. All milking was done by machine.

Cream samples were collected only from the morning milk, by pooling 5.6 l of milk from each cow of its group. This was then separated on an electric farm separator adjusted to give medium heavy cream (about 45% butterfat), and samples of milk and cream were stored frozen for analysis (Dishburger *et al.*, 1972b). The results are shown in Table 107. Residues of chlorpyrifos in milk ranged from <0.01 mg/kg for cows fed at the 10 ppm level to 0.02 mg/kg at 30 ppm. In cream the residues ranged from <0.01 at 3 ppm to 0.15 mg/kg at 30 ppm. After withdrawal from the 30 ppm diet, milk and cream showed no detectable residues in the withdrawal period.

Table 107. Residues of chlorpyrifos in milk and cream of cows fed diets containing chlorpyrifos for 14 days.

| Chlorpyrifos, ppm in diet | Days fed | Chlorpyrifos, mg/kg [1] | | Reference |
|---|---|---|---|---|
| | | Milk | Cream | |
| 3 | 10 | -- | 0.01 | GH-C 533 |
| | 11 | -- | <0.01 | |
| | 12 | -- | <0.01 | |
| | 13 | -- | 0.01 | |
| 10 | 3 | <0.01, <0.01, <0.01 | | GH-C 533 |
| | 6 | <0.01,<0.01, <0.01 | | |
| | 10 | <0.01,<0.01, <0.01 | 0.03, 0.04 | |
| | 11 | <0.01,<0.01, <0.01 | 0.02, 0.03 | |
| | 12 | <0.01,<0.01, <0.01 | 0.03, 0.03 | |
| | 13 | <0.01,<0.01, <0.01 | 0.03, 0.03 | |
| 30 | 3 | 0.01, 0.02, 0.01 | | GH-C 533 |
| | 6 | 0.01, 0.02, 0.01 | | |
| | 10 | <0.01, 0.01, 0.01 | 0.15 | |
| | 11 | 0.01, 0.01, 0.01 | 0.12 | |
| | 12 | 0.01, 0.01, 0.01 | 0.11 | |
| | 13 | 0.01, 0.01, 0.01 | 0.10 | |
| Withdrawal from | Withdrawal (days) | | | GH-C 533 |
| 30 ppm diet | 1 | <0.01, <0.01, <0.01 | | |
| | 3 | <0.01, <0.01, <0.01 | <0.01 | |
| | 4 | <0.01, <0.01, <0.01 | <0.01 | |
| | 5 | <0.01, <0.01, <0.01 | <0.01 | |

[1] LOQ 0.01 mg/kg. Recoveries averaged 88% from milk and cream

Pigs. Eighteen weaned Landrace pigs weighing about 23 kg each were divided into 6 treatment groups, each consisting of 2 males and 1 female, placed in separate pens and fed *ad libitum* throughout the trial. Groups were fed the basal ration containing 0, 1, 3 or 10 ppm (3 groups) chlorpyrifos for 30 days. The groups fed 0, 1 and 3 ppm and one of the 10 ppm groups were slaughtered with no withdrawal period. The remaining two 10 ppm groups were put on untreated feed and one group was slaughtered at 7 days and the other after 21 days.

Samples of muscle, liver, kidney, omental fat, renal fat and subcutaneous fat were collected from each pig at slaughter (Dishburger *et al.*, 1972c). The results (Table 106) showed that during the treatment period residues in the fat ranged from <0.01-0.02 mg/kg at the 1 ppm level to 0.05-0.18 mg/kg at the 10

ppm level, and in the muscle 0.01-0.03 mg/kg at the 10 ppm level. After withdrawal the only detectable residues were 0.01-0.03 mg/kg in the fat at 7 days.

Table 108. Residues of chlorpyrifos in tissues of pigs fed diets containing chlorpyrifos for 30 days.

| Chlorpyrifos, ppm in diet | Chlorpyrifos, mg/kg [1] | | | | | | Reference |
|---|---|---|---|---|---|---|---|
| | Muscle | Liver | Kidney | Omental fat | Renal fat | Subcutaneous fat | |
| 0 | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 | 0.00 | GH-C 549 |
| | 0.00 | <0.01 | 0.00 | 0.00 | 0.01 | 0.00 | |
| | 0.00 | 0.00 | 0.00 | 0.00 | 0.01 | 0.00 | |
| 1 | --- | --- | --- | <0.01 | 0.02 | 0.01 | GH-C 549 |
| | --- | --- | --- | <0.01 | <0.01 | <0.01 | |
| | --- | --- | --- | 0.01 | 0.01 | 0.01 | |
| 3 | <0.01 | <0.01 | --- | 0.01 | 0.02 | 0.02 | GH-C 549 |
| | <0.01 | <0.01 | --- | <0.01 | 0.01 | 0.01 | |
| | <0.01 | <0.01 | --- | 0.01 | 0.04 | 0.03 | |
| 10 | 0.03 | <0.01 | <0.01 | 0.18 | 0.18 | 0.18 | GH-C 549 |
| | 0.02 | 0.01 | <0.01 | 0.05 | 0.12 | 0.12 | |
| | 0.01 | 0.01 | <0.01 | 0.06 | 0.11 | 0.12 | |
| Withdrawal from 10 ppm | | | | | | | |
| 7 days | <0.01 | <0.01 | --- | <0.01 | 0.01 | 0.01 | GH-C 549 |
| | <0.01 | <0.01 | --- | <0.01 | 0.02 | 0.03 | |
| | <0.01 | <0.01 | --- | <0.01 | <0.01 | 0.01 | |
| 21 days | --- | --- | --- | <0.01 | <0.01 | <0.01 | GH-C 549 |
| | --- | --- | --- | <0.01 | <0.01 | <0.01 | |
| | --- | --- | --- | <0.01 | <0.01 | <0.01 | |

[1] LOQ 0.01 mg/kg. Recoveries averaged 88% from muscle, 90% from liver, 88% from kidney and 83% from fat

Poultry. 288 hens were divided into 8 groups of 36 birds each, kept in certified facilities, and fed chlorpyrifos at 0 (2 groups) 0.3, 1, 3 and 10 (3 groups) ppm in their diet for 30 days. After the 30 days, all the hens in the two control groups and 24 hens from each of 0.3, 1, 3 and 10 (1 group only) ppm groups were killed with no withdrawal. Samples of muscle with fat and skin, liver, kidneys and peritoneal fat, as well as eggs from laying hens fed 10 ppm chlorpyrifos for 45 days, were analysed for residues (Dishburger *et al.*, 1972d). The residues in tissues are shown in Table 109 and in whole eggs in Table 110. Residues were found in peritoneal fat from <0.01 mg/kg at 1 ppm to 0.05 mg/kg at 10 ppm. No residues were detected at 7 days after withdrawal from the 10 ppm dose level. Feed consumption during the 30-day trial was 0.1 kg/hen/day for the control groups, and 0.12 kg/hen/day for the treated groups. Egg production was consistent in all groups (0.62-0.72 egg/hen/day).

Table 109. Residues of chlorpyrifos in tissues of chickens fed diets containing chlorpyrifos for 30 days.

| Chlorpyrifos, ppm in diet | Residues, mg/kg [1] | | | | Reference |
|---|---|---|---|---|---|
| | Muscle | Liver | Kidney | Peritoneal fat | |
| 0 | 0.00 | 0.004 | 0.00 | 0.00 | GH-C 555 |
| | 0.01 | 0.001 | 0.00 | 0.00 | |

| Chlorpyrifos, ppm in diet | Residues, mg/kg [1] | | | | Reference |
|---|---|---|---|---|---|
| | Muscle | Liver | Kidney | Peritoneal fat | |
| | 0.001 | 0.001 | 0.00 | 0.00 | |
| | 0.00 | 0.003 | 0.00 | 0.00 | |
| | 0.00 | 0.002 | 0.00 | 0.00 | |
| | 0.00 | 0.000 | 0.00 | 0.00 | |
| 0.3 | No residues detected | | | | GH-C 555 |
| | | | | | |
| 1.0 | | --- | --- | <0.01 | GH-C 555 |
| | | --- | --- | <0.01 | |
| | | --- | --- | <0.01 | |
| | | --- | --- | <0.01 | |
| | | --- | --- | <0.01 | |
| | | --- | --- | <0.01 | |
| 3 | | --- | --- | <0.01 | GH-C 555 |
| | | --- | --- | 0.01 | |
| | | --- | --- | <0.01 | |
| | | --- | --- | 0.01 | |
| | | --- | --- | 0.01 | |
| | | --- | --- | 0.01 | |
| 10 | 0.01 | <0.01 | <0.01 | 0.03 | GH-C 555 |
| | <0.01 | <0.01 | <0.01 | 0.05 | |
| | <0.01 | <0.01 | <0.01 | 0.03 | |
| | <0.01 | <0.01 | <0.01 | 0.02 | |
| | <0.01 | <0.01 | <0.01 | 0.02 | |
| | <0.01 | <0.01 | <0.01 | 0.02 | |
| Withdrawal from 10 ppm feed | | | | | |
| 7 days | --- | --- | --- | No residues detected | GH-C 555 |

[1] LOQ 0.01 mg/kg. Recoveries averaged 98% from muscle, 90% from liver, 91% from kidney, 83% from fat.

Table 110. Residues of chlorpyrifos in whole eggs of chickens fed diets containing chlorpyrifos for 7-45 days.

| Chlorpyrifos, ppm in diet | Days feed | Chlorpyrifos, mg/kg [1] | Reference |
|---|---|---|---|
| 0 | 0 | 0.00, 0.00, | GH-C 555 |
| | 10 | 0.00, 0.00 | |
| 10 | 7 | 0.01 | GH-C 555 |
| | 14 | 0.01 | |
| | 21 | <0.01 | |
| | 28 | 0.01 | |
| | 35 | <0.01 | |
| | 36 | 0.01 | |
| | 37 | <0.01 | |
| | 38 | 0.01 | |
| | 39 | <0.01 | |
| | 40 | <0.01 | |
| | 41 | <0.01 | |
| | 42 | <0.01 | |
| | 43 | <0.01 | |

| Chlorpyrifos, ppm in diet | Days feed | Chlorpyrifos, mg/kg [1] | Reference |
|---|---|---|---|
| | 44 | 0.01 | |
| | 45 | <0.01 | |

[1] LOQ 0.01 mg/kg. Recoveries averaged 83%

## FATE OF RESIDUES IN STORAGE AND PROCESSING

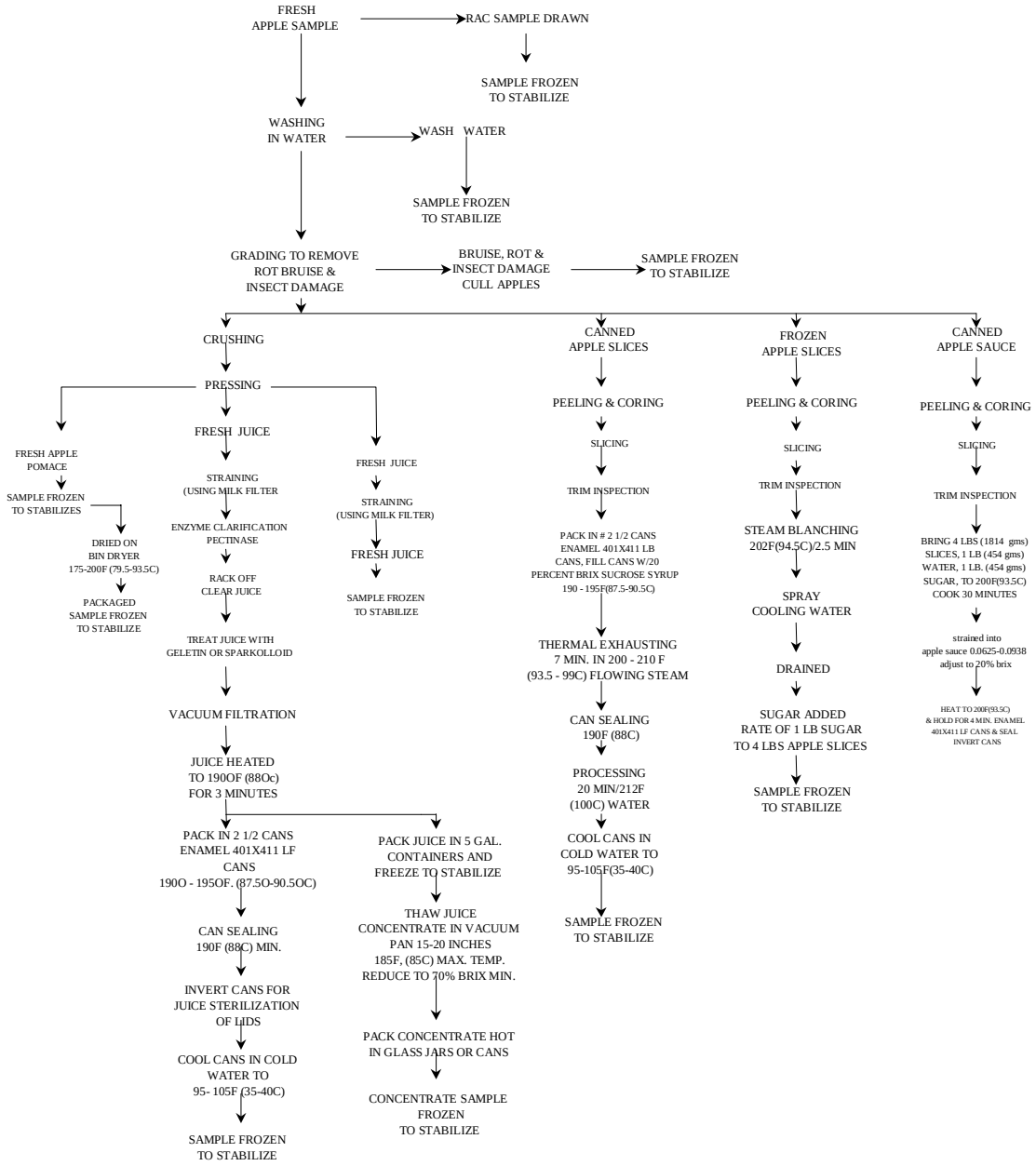
### In processing

Processing trials were conducted on apples, citrus, grapes, sugar beet, tomatoes**,** maize, wheat, cotton seed, peanuts, sunflower and coffee. The results are shown in Tables 111-129, where processing factors used in the estimation of STMR-Ps, HRs and/or maximum residue levels are underlined.

None of the results were corrected for recoveries or apparent residues in control samples.

Apples. A typical small-batch apple processing simulating commercial practice is shown in Figure 4. The process involves washing and processing into juice and wet and dry pomace.

Figure 4. Apple processing.



In processing trials in Midland, Michigan in 1976, apple trees received 7 applications of chlorpyrifos 50 WP at the label rate of 2.24 kg ai/ha or 0.06 kg ai/hl. Samples harvested on the day of the last application were processed and residues of chlorpyrifos determined in unwashed, washed, and peeled apples as well as in the peel, juice, and wet and dry pomace (Wetters and Ervick, 1978).

In further processing trials 12 applications of chlorpyrifos 50 WP were made at 2.24 kg ai/ha or 0.12 kg ai/hl, and samples harvested and processed on the last treatment date (0-day PHI). Residues were determined in whole apples, juice, and wet and dry pomace (Miller, 1981a). The results are shown in Table 111.

Table 111. Residues of chlorpyrifos in processed apple fractions, 0-day PHI.

| Sample | Application, kg ai/ha | Residues and (mean), mg/kg [1,2] | Processing factor | Reference |
|---|---|---|---|---|
| Unwashed apples | 2.24 x7 | 4.4, 3.2, 2.1, 2.9 [3] (3.2) | | GH-C 1107 |
| | 2.24 x12 | 0.53 | 1.0 | GH-C 1488 |
| Washed apples | 2.24 x7 | 4.0, 3.3, 2.9, 3.6 (3.4) | 1.0 | GH-C 1107 |
| Peeled apples | 2.24 x7 | 0.52, 0.27, 0.21 0.43 (0.36) | 0.11 | GH-C 1107 |
| Peels | 2.24 x7 | 17, 17, 11, 16 (15) | 4.4 | GH-C 1107 |
| Juice | 2.24 x7 | 0.27, 0.40, 0.23, 0.30 (0.30) | 0.09 | GH-C 1107 |
| | 2.24 x12 | 0.11 | 0.20 | GH-C 1488 |
| Wet pomace | 2.24 x7 | 8.2, 7.9, 7.2, 7.0 (7.6) | 2.2 | GH-C 1107 |
| | 2.24 x12 | 0.88 | 1.7 | GH-C 1488 |
| Dry pomace | 2.24 x12 | 3.5 | 6.6 | GH-C 1488 |

[1] LOQ 0.01 mg/kg. Results not corrected for recoveries
[2] Recoveries averaged 92 to 97% from whole apples, 94% from peeled apples, 87% from peel, 87% from pomace and 101% from juice.
[3] Four samples of 20 apples each randomly picked from 10 trees.

## Citrus fruit

Oranges. Three processing trials were conducted in California in 1975. In the first chlorpyrifos was applied to fruit at 12 kg ai/ha using low- and high-volume sprays (California, 1975). Samples of whole oranges, peel, peel + pulp and juice were taken after 14 days and residues determined. The juice was extracted with a stainless steel electric juicer. In the second trial, the fruit were treated at 15 kg ai/ha and processed after 14 days, and in the third at 17 kg ai/ha with samples taken 0, 3, 14 and 30 days later (Wetters, 1977a).

In further processing trials chlorpyrifos was applied to oranges at 8 kg ai/ha (Trial 1) and 11 kg ai/ha (Trial 2) and residues were determined 14 and 21 days after treatment respectively in whole oranges, peel, pulp and juice. The oranges were peeled by hand and juice extracted in the laboratory with a Hobart juice extractor (Wetters, 1978). The results are shown in Table 112.

Table 112. Residues in oranges, juice, peel and pulp after foliar applications of chlorpyrifos and subsequent simulated home processing in the USA.

| Sample | Application, kg ai/ha | PHI, days | Residues and (mean), mg/kg[1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| Whole oranges (unwashed) | 12 | 14 | 0.40, 0.55, 0.49, 0.36 (0.45) (13400 l/ha) | 1 | GH-C 1041 |
| | 12 | 14 | 1.2, 1.0, 1.3, 1.1, 1.1 (1.1) (935 l/ha) | 1 | |
| | 15 | 14 | 0.86, 0.84, 0.77, 0.80 (0.82) (16800 l/ha) | 1 | |

| Sample | Application, kg ai/ha | PHI, days | Residues and (mean), mg/kg[1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| | 15 | 14 | 0.56, 0.47, 0.24, 0.82 (0.52) (935 l/ha) | 1 | |
| Whole oranges (unwashed) | 17 | 0 | 1.5, 1.6, 1.5, 1.3 (18700 l/ha) | | GH-C 1041 |
| | 17 | 3 | 0.54, 0.60, 0.58, 2.6 | | |
| | 17 | 14 | 0.38, 0.70, 0.52, 0.35 (0.49) | | |
| | 17 | 30 | 0.21, 0.21, 0.13, 0.18 | | |
| Whole oranges (unwashed) | 17 | 0 | 6.8, 5.0, 7.1, 3.2 (935 l/ha) | | GH-C 1041 |
| | 17 | 3 | 3.8, 3.7, 3.6, 1.8 | | |
| | 17 | 14 | 3.3, 2.0, 2.3, 0.70 (2.1) | 1 | |
| | 17 | 30 | 0.80, 0.69, 0.89, 0.63 | | |
| Peel + pulp | 12 | 14 | 0.52, 0.56, 0.55, 0.51 (0.54) | 1.2 | GH-C 1041 |
| | 12 | 14 | 1.6, 1.5, 1.4, 1.3 (1.4) | 1.3 | |
| | 15 | 14 | 1.5, 1.6, 1.5, 1.7 (1.6) | 2.0 | |
| | 15 | 14 | 0.59, 0.85, 0.62, 0.60 (0.66) | 1.3 | |
| Juice | 12 | 14 | 0.01, 0.01, 0.01, 0.01 (0.01) | 0.02 | GH-C 1041 |
| | 12 | 14 | 0.07, 0.03, 0.02, 0.02 (0.04) | 0.04 | |
| . | 15 | 14 | 0.01, 0.01, <0.01, 0.01 (0.01) | 0.01 | GH-C 1041 |
| | 15 | 14 | <0.01, <0.01, <0.01, 0.01 (0.01) | 0.02 | |
| Peel + pulp | 17 | 0 | 3.2, 2.4 (18700 l/ha) | | |
| | 17 | 3 | 1.5, 1.02 | | |
| | 17 | 14 | 0.88, 0.68 (0.78) | 1.6 | |
| | 17 | 30 | 0.40, 0.39 | | |
| | 17 | 0 | 16, 5.4 (935 l/ha) | | |
| | 17 | 3 | 8.4, 4.6 | | |
| | 17 | 14 | 2.0, 4.9 (3.4) | 1.6 | |
| | 17 | 30 | 1.4, 0.68 | | |
| Juice | 17 | 0 | 0.038, 0.046 (18700 l/ha) | | |
| | 17 | 3 | 0.018, 0.016 | | |
| | 17 | 14 | 0.013, 0.013 (0.013) | 0.03 | |
| | 17 | 30 | 0.016, 0.011 | | |
| | 17 | 0 | 0.78, 0.071 (935 l/ha) | | |
| | 17 | 3 | 0.22, 0.22 | | |
| | 17 | 14 | 0.17, 0.062 (0.12) | 0.06 | |
| | 17 | 30 | 0.050, 0.027 | | |
| Whole oranges (unwashed) | 8 | 14 | 0.30, 0.28, 0.33, 0.33 (0.31) | 1 | GH-C 1141 |
| | | 21 | 0.34, 0.44, 0.35, 0.27 | | |
| Whole oranges (unwashed) | 11 | 14 | 0.59, 0.56, 0.58, 0.46 (0.55) | 1 | GH-C 1141 |
| | | 21 | 0.47, 0.44, 0.44, 0.52 | | |
| Peel | 8 | 14 | 1.0, 0.98, 1.0, 1.0 (1.0) | 3.2 | GH-C 1141 |
| | | 21 | 0.97, 0.84, 0.92, 0.89 | | |
| Peel | 11 | 14 | 1.9, 1.9, 2.0, 1.6 (1.8) | 3.3 | GH-C 1141 |
| | | 21 | 2.1, 1.9, 1.7, 1.7 | | |
| Pulp | 8 | 14 | 0.03, 0.02, 0.03, 0.01 (0.02) | 0.06 | GH-C 1141 |
| | | 21 | 0.01, 0.01, 0.01, 0.02 | | |
| Pulp | 11 | 14 | 0.06, 0.03, 0.04, 0.07 (0.05) | 0.09 | GH-C 1141 |
| | | 21 | 0.02, 0.07, 0.02, 0.02 | | |
| Juice | 8 | 14 | <0.01, 0.01, <0.01, <0.01 (0.01) | 0.03 | GH-C 1141 |
| | | 21 | <0.01, 0.01, <0.01, 0.01 | | |
| Juice | 11 | 14 | <0.01, <0.01, <0.01, 0.01 (0.01) | 0.02 | GH-C 1141 |

| Sample | Application, kg ai/ha | PHI, days | Residues and (mean), mg/kg[1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| | | 21 | 0.01, 0.01, 0.01, 0.01 | | |

[1] LOQ 0.01 mg/kg. Results not corrected for recoveries
[2] Recoveries averaged 87 to 88% from whole oranges, 90% from peel + pulp, 86% from peel, 87% from pulp and 87 to 92% from juice.

Batches of 10-15 field boxes of citrus fruit were processed according to the scheme in Figure 5, simulating the commercial process. Residues in the unwashed fruit and fractions through to juices and oils are shown in Table 113 (Wetters, 1981).

Figure 5. Citrus fruit processing.

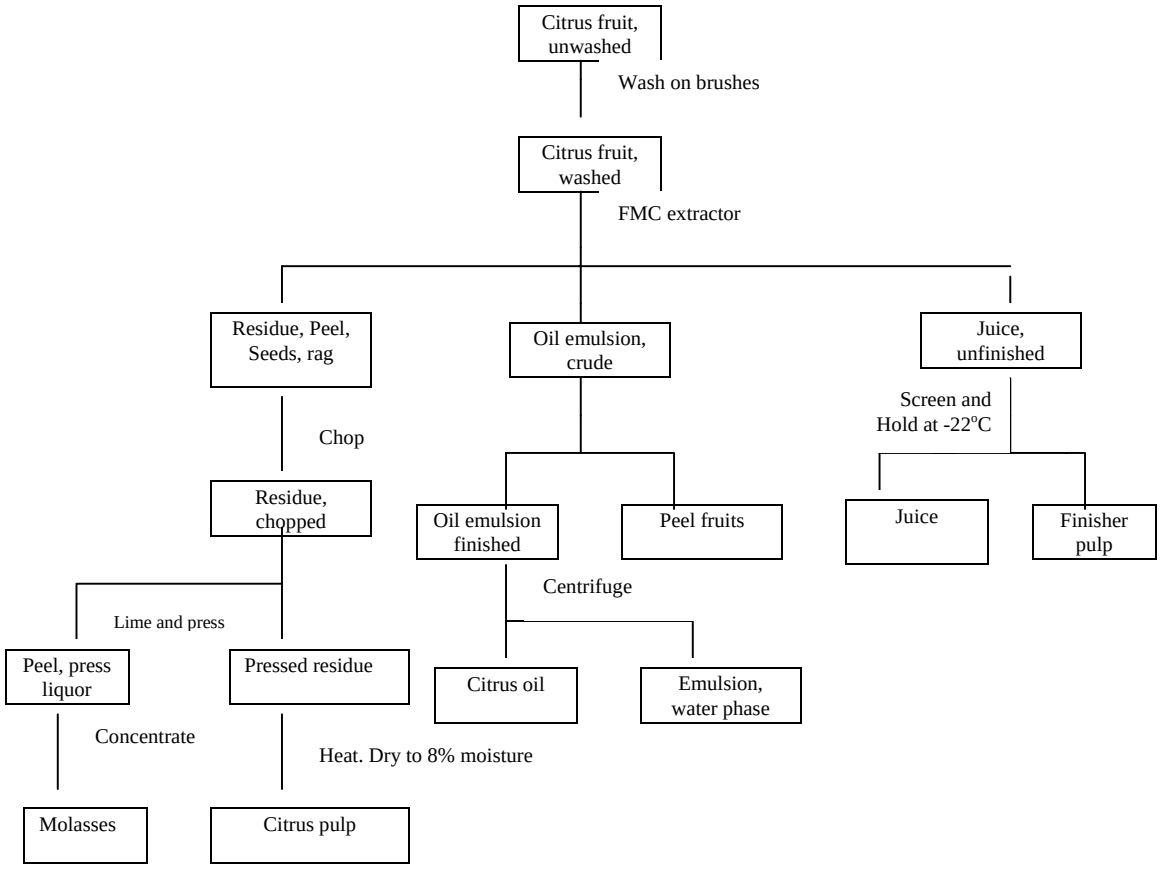


Table 113. Residues of chlorpyrifos in citrus fruit and commercial processed fractions.

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg[1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| Grapefruit (unwashed) | 5.9 x2 | 15 | 0.36 | | GH-C 1441 |
| Grapefruit (washed) | 5.9 x2 | 15 | 0.29 | 1 | |
| Dried pulp | 5.9 x2 | 15 | 1.1 | 3.8 | |
| Juice | 5.9 x2 | 15 | <0.01 | 0.03 | |
| Peel | 5.9 x2 | 15 | 1.3 | 4.5 | |
| Pulp | 5.9 x2 | 15 | 0.01 | 0.03 | |
| Oil | 5.9 x2 | 15 | 6.3 | 22. | |
| Lemons (unwashed) | 5.8 x2 | 14 | 0.38 | | GH-C 1441 |
| Lemons (washed) | 5.8 x2 | 14 | 0.31 | 1 | |
| Dried pulp | 5.8 x2 | 14 | 0.48 | 1.5 | |
| Juice | 5.8 x2 | 14 | <0.01 | 0.03 | |
| Peel | 5.8 x2 | 14 | 0.71 | 2.3 | |
| Pulp | 5.8 x2 | 14 | 0.01 | 0.03 | |
| Oil | 5.8 x2 | 14 | 1.0 | 3.2 | |
| Oranges (unwashed) | 8.6 x2 | 15 | 0.52 | | GH-C 1441 |
| Oranges (washed) | 8.6 x2 | 15 | 0.47 | 1 | |
| Dried pulp | 8.6 x2 | 15 | 1.2 | 2.6 | |
| Fruit juice | 8.6 x2 | 15 | <0.01 | 0.02 | |
| Peel | 8.6 x2 | 15 | 1.5 | 3.2 | |
| Pulp | 8.6 x2 | 15 | 0.02 | 0.04 | |
| Oil | 8.6 x2 | 15 | 3.0 | 6.4 | |
| Tangelos (unwashed) | 4.5 x2 | 15 | 0.59 | | GH-C 1441 |
| Tangelos (washed) | 4.5 x2 | 15 | 0.42 | 1 | |
| Dried pulp | 4.5 x2 | 15 | 1.7 | 4.0 | |
| Juice | 4.5 x2 | 15 | <0.01 | 0.02 | |
| Peel | 4.5 x2 | 15 | 3.6 | 8.6 | |
| Pulp | 4.5 x2 | 15 | 0.08 | 0.20 | |
| Oil | 4.5 x2 | 15 | 5.6 | 13. | |

[1] LOQ 0.01 mg/kg. Results not corrected for recoveries
[2] Recoveries averaged 86 to 89% from whole fruit and 78 to 79% from the processed fractions

Grapes. Processing trials were conducted in various countries from 1978 to 1988. Processing included drying to raisins and processing to juice, pomace, must and wine. In the USA chlorpyrifos was applied at 1.12 kg ai/ha, and grapes sampled 0 and 3 days after application. A portion of the sample was sun-dried for 14 days on paper-lined trays to produce raisins, and as it was late season the samples had to be put under cover each evening to protect them from dew (Wetters, 1984).

The results are shown in Table 114.

Table 114. Residues of chlorpyrifos in grapes, raisins and raisin waste (USA).

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg[1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| Grapes | 1.12 | 0 | 1.3 | 1 | GH-C 1698 |
| | | 3 | 0.38 | 1 | |
| Raisins | 1.12 | 0 + 14 | 0.28 | 0.22 | GH-C 1698 |
| | | 3 +14 | 0.07, 0.08 | 0.20 | |
| Raisin waste | 1.12 | 0 + 14 | 0.51 | 0.39 | GH-C 1698 |
| | | 3 +14 | 0.26 | 0.68 | |

[1] LOQ 0.01 mg/g. Results not corrected for recoveries.
[2] Recoveries averaged 85% from grapes, 91% from raisins and 94% from raisin waste

Chlorpyrifos was applied 3 times at 1.12 kg ai/ha in processing trials in the USA in 1982. Samples were harvested 44 days after the last treatment and processed. Residues of chlorpyrifos were determined in the grapes, juice and pomace (Wetters, 1983). The results are shown in Table 115.

Table 115. Residues of chlorpyrifos in grapes, juice, wet pomace and dry pomace.

| Sample | Application, kg ai/ha | PHI, days | Residues and (mean), mg/kg[1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| Grapes | 1.12 (x3) | 44 | 0.42, 0.40, 0.49, 0.63 (0.48) | 1 | GH-C 1611 |
| Juice | | | 0.03 | 0.06 | |
| Wet Pomace | | | 0.90 | 1.9 | |
| Dry pomace | | | 1.9 | 4.0 | |

[1] LOQ 0.01 mg/kg. Results not corrected for recoveries
[2] Recoveries averaged 89% from grapes, 85% from wet pomace, 92% from dry pomace and 91% from juice

Results of processing trials in which grapes were processed into must and wine are shown in Table 116. The water dispersible granule formulation of chlorpyrifos (WG 750 g ai/kg) was applied four times to wine grapes at 0.34 kg ai/ha in Northern France (Mesland). In a trial on white wine grapes (stages BBCH 65, 79 (2) and 85) and another on red wine grapes (stages BBCH 65, 77, 79 and 83) samples of grapes were taken just before, just after and 7, 14, 21 and 28 days after the last application. At harvest (28-day PHI), separate samples of grapes were taken for residue analysis and for processing to must, pomace, and wine at 2 months and wine at 6 months (Gale, 1999). The recovery of chlorpyrifos from fortified control samples was 81% from grapes (0.05 mg/kg), 106% from pomace (0.01 mg/kg), 102, 104% from must (0.02 mg/kg) and 96, 98% from wine (0.02 mg/kg).

In trials in Italy in 1986 single applications of chlorpyrifos were applied to vines at either 0.69 or 0.77 kg ai/ha. In one trial, a double application at 0.69 kg ai/ha was followed by another double application at 0.77 kg ai/ha. Samples of grapes were taken at intervals after application ranging between 7 and 69 days. At harvest, additional grapes were taken for wine production (Teasdale, 1988b).

In a trial in Israel in 1975 wine grapes were treated with chlorpyrifos at 0.48 kg ai/ha and sampled 0, 7, 14 and 21 days after application. A second trial involved treatment at 0.72 kg ai/ha with grapes sampled on 0 only. Wine was prepared from grapes taken at each sampling interval after which both wine and grapes were analysed for chlorpyrifos residues (Hollick and Iosson, 1978).

Table 116. Residues of chlorpyrifos in grapes, must and wine.

| Country, year | Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg [1,2] and (mean) | Processing factor | Reference |
|---|---|---|---|---|---|---|
| Israel, 1975 | Grape | 0.48 | 0 | 3.0, 3.4 (3.2) | 1 | GHE-P 603 |
| | | | 7 | 0.90, 0.94 (0.92) | 1 | |
| | | | 14 | 1.1, 1.1 (1.1) | 1 | |
| | | | 21 | 0.48, 0.49 (0.48) | 1 | |
| | | 0.72 | 0 | 0.01, 0.01 (0.01) | 1 | |
| Israel, 1975 | Wine | 0.48 | 0 | 0.02, 0.02 (0.02) | 0.006 | GHE-P 603 |
| | | 0.48 | 7 | 0.01, 0.01 (0.01) | 0.01 | |
| | | 0.48 | 14 | 0.01, 0.01 (0.01) | 0.009 | |
| | | 0.48 | 21 | <0.01, <0.01 (0.01) | 0.02 | |

| | | | | | | |
|---|---|---|---|---|---|---|
| | | 0.72 | 0 | 0.01, 0.01 (0.01) | 1 | |
| Italy, 1986 | Grapes | 0.69 | 7 | 0.50, 0.49 | | GHE-P 1818R |
| | | | 14 | 0.19, 0.19, 0.03, 0.03 | | |
| | | | 33 | 0.02, 0.02 | | |
| | | | 49 | 0.03, 0.03 (0.03) | 1 | |
| Italy, 1986 | Grapes | 0.77 | 12 | 0.71, 0.66 | | GHE-P 1818R |
| | | | 19 | 0.30, 0.26 | | |
| | | | 38 | 0.10, 0.10 | | |
| | | | 69 | 0.05, 0.05 (0.05) | 1 | |
| Italy, 1986 | Grapes | 0.69 x2 + 0.77 x2 | 13 | 0.50, 0.50 | | GHE-P 1818R |
| | | | 44 | 0.29, 0.24 (0.26) | 1 | |
| Italy, 1986 | Must | 0.69 | | <0.01, <0.01, <0.01 (0.01) | 0.3 | GHE-P 1818R |
| | | 0.77 | | <0.01 (0.01) | 0.2 | |
| | | 0.69 x2 + 0.77 x2 | | <0.01, <0.01 (0.01) | 0.04 | |
| Italy, 1986 | Wine | 0.69 | | <0.01, <0.01, <0.01 (0.01) | 0.3 | GHE-P 1818R |
| | | 0.77 | | <0.01, <0.01 (0.01) | 0.2 | |
| | | 0.69 x2 + 0.77 x2 | | <0.01, <0.01 (0.01) | 0.04 | |
| France, 1996 | Grapes (red) | 0.34 x4 | 28 | 0.33 | 1 | GHE-P 7467 |
| | Must | | | 0.17 | 0.5 | |
| | Pomace | | | 1.7 | 5.2 | |
| | Wine | | | <0.02 | 0.06 | |
| | Grapes (white) | 0.34 x4 | 28 | 0.50 | 1 | |
| | Must | | | 0.06 | 0.1 | |
| | Pomace | | | 1.3 | 2.6 | |
| | Wine | | | <0.02 | 0.04 | |

[1] Limit of quantification 0.01 mg/kg, except for GH-C 788, where the limit of determination = 0.001 mg/kg and GHE-P 7467, where the limit of determination (wine) = 0.02 mg/kg.
[2] Recoveries averaged 96% from must, 92 to 98% from wine

Tomatoes. Processing trials on tomatoes were conducted in the USA and Israel. In the US trial tomatoes were treated 5 times with 1.12 kg ai/ha and samples were taken 7 days after the last application for processing (Miller, 1980d). The results, not corrected for recoveries or control, are shown in Table 117.

Table 117. Residues of chlorpyrifos in tomatoes and processed fractions (USA).

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg[1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| Whole tomatoes | 1.12 x5 | 7 | 0.33 | 1 | GH-C 1282 |
| Tomato juice | | | <0.01, <0.01 (0.01) | 0.03 | |
| Whole tomato less peel | | | <0.01, 0.01 (0.01) | 0.03 | |
| Purée | | | 0.05, 0.03 (0.04) | 0.1 | |
| Seeds and peelings from purée | | | 0.80, 0.43 (0.62) | 1.9 | |
| Seeds and peelings from juice | | | 1.4, 0.47 (0.94) | 2.8 | |

[1] LOQ 0.01 mg/kg
[2] Recoveries averaged 87% from whole tomato, 100% from juice, 94% from tomato less peel, 92% from purée and 90% from seeds/peelings.

In processing trials in Israel, tomatoes were treated four times with chlorpyrifos at 0.96 kg ai/ha. Samples were collected at 0, 7, 14 and 21 days after the last treatment. A portion of the fruit collected at each sampling was processed to juice and concentrated to paste. Fresh tomatoes, juice and paste were analysed for residues of chlorpyrifos (Hollick and Iosson, 1977). The results are shown in Table 118.

Table 118. Residues of chlorpyrifos in fresh tomatoes, juice and paste (Israel).

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg [1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| 1975 Trials | | | | | |
| Whole tomatoes | 0.96 | 0 | 0.36, 0.16, 0.15, 0.11 (0.20) | 1 | GHE-P 489 |
| | | 7 | 0.12, 0.10, 0.10, 0.13 (0.11) | 1 | |
| | | 14 | 0.10, 0.06, 0.08, 0.06 (0.08) | 1 | |
| | | 21 | 0.06, 0.05, 0.04, 0.08 (0.06) | 1 | |
| Juice | 0.96 | 0 | 0.04, 0.02, 0.01, <0.01 (0.02) | 0.1 | GHE-P 489 |
| | | 7 | 0.02, 0.02, 0.01, 0.02 (0.02) | 0.2 | |
| | | 14 | 0.01, 0.01, 0.01, 0.01 (0.01) | 0.1 | |
| | | 21 | 0.01, <0.01, <0.01, 0.01 (0.01) | 0.2 | |
| Paste | 0.96 | 0 | 0.05, 0.06, 0.03, 0.02 (0.04) | 0.2 | GHE-P 489 |
| | | 7 | 0.04, 0.04, 0.02, 0.03 (0.03) | 0.3 | |
| | | 14 | 0.03, 0.02, 0.03, 0.02 (0.02) | 0.2 | |
| | | 21 | 0.03, 0.02, 0.02, 0.02 (0.02) | 0.3 | |
| 1976 Trials | | | | | |
| Whole tomatoes | 0.96 | 0 | 0.38, 0.65, 0.29, 0.26 (0.52) | 1 | GHE-P 489 |
| | | 7 | 0.49, 0.21, 0.22, 0.13 (0.26) | 1 | |
| | | 14 | 0.11, 0.08, 0.05, 0.09 (0.08) | 1 | |
| | | 21 | 0.14, 0.11, 0.19, 0.05 (0.12) | 1 | |
| Juice | 0.96 | 0 | 0.03, 0.01, 0.01, 0.03 (0.02) | 0.04 | GHE-P 489 |
| | | 7 | <0.01, 0.01, 0.01, <0.01 (0.01) | 0.04 | |
| | | 14 | <0.01 (0.01), <0.01, <0.01, <0.01 (0.01) | 0.1 | |
| | | 21 | <0.01, <0.01, <0.01, <0.01 (0.01) | 0.08 | |
| Paste | 0.96 | 0 | 0.03, 0.01, 0.01, 0.03 (0.02) | 0.04 | GHE-P 489 |
| | | 7 | <0.01, 0.02, <0.01, <0.01 (0.01) | 0.04 | |
| | | 14 | <0.01, <0.01, <0.01, <0.01 (0.01) | 0.1 | |
| | | 21 | <0.01, <0.01, <0.01, <0.01 (0.01) | 0.08 | |

[1] LOQ 0.01 mg/kg. Results not corrected for recoveries
[2] Recoveries from all fractions averaged 98%

Soya beans. Lorsban 4E was applied 5 times at exaggerated rates in the USA and seeds harvested 14 days after the last application for processing by a commercial solvent extraction procedure (Miller, 1979a). The recovery of chlorpyrifos from fortified control samples was 88±15%% from soya beans (0.01 mg/kg, n = 5), 80% from hulls (0.05 mg/kg), 82% from extracted meal (0.05 mg/kg), 90% from crude oil (0.01 mg/kg), 80% from refined oil (0.01 mg/kg), 90% from refined bleached oil (0.01 mg/kg) and 100% from soapstock (0.02 mg/kg). The results are shown in Table 119. No details of the processing were provided.

Table 119. Residues of chlorpyrifos in soya beans and processed fractions (USA).

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg[1] | Processing factor | Reference |
|---|---|---|---|---|---|
| Soya beans (RAC) | 4.4 + 1.1 x2 + 2.2 x2 | 14 | 0.04 | 1 | GH-C 1224 |
| Hulls | 4.4 + 1.1 x2 + 2.2 x2 | 14 | 0.02 | 0.5 | |

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg[1] | Processing factor | Reference |
|---|---|---|---|---|---|
| Extracted meal | 4.4 + 1.1 x2 + 2.2 x2 | 14 | <0.05 | 1.2 | |
| Crude oil | 4.4 + 1.1 x2 + 2.2 x2 | 14 | 0.02 | 0.5 | |
| Refined oil | 4.4 + 1.1 x2 + 2.2 x2 | 14 | 0.02 | 0.5 | |
| Refined bleached oil | 4.4 + 1.1 x2 + 2.2 x2 | 14 | 0.02 | 0.5 | |
| Soapstock | 4.4 + 1.1 x2 + 2.2 x2 | 14 | <0.02 | 0.5 | |

[1] LOQ 0.01-0.05 mg/kg. Results not corrected for recoveries

Sugar beet. Sugar beet were treated at planting and/or post-emergence with chlorpyrifos at 2.2 kg ai/ha in the USA. Samples were collected at normal harvest (133 days after the last treatment) and processed, simulating commercial practices. Untreated samples were also fortified with 1 mg/kg chlorpyrifos before processing. Residues of chlorpyrifos were determined in the roots, pulp, cake and juice (Wetters and Dishburger, 1974). The results are shown in Table 120.

Table 120. Residues of chlorpyrifos in sugar beet and processed fractions (USA).

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg [1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| Roots | 2.2 x2 | 133 | <0.01 | | GH-C 729 |
| Wet pulp | 2.2 x2 | 133 | <0.01 | | |
| Wet pulp, fortified | + 1 mg/kg | | 0.43 | 0.43 | |
| Dry pulp | 2.2 x2 | 133 | 0.01 | | |
| Dry pulp, fortified | +1 mg/kg | | 0.48 | 0.48 | |
| Lime cake | 2.2 x2 | 133 | <0.01 | | |
| Lime cake, fortified | +1 mg/kg | | 0.01 | 0.01 | |
| Diffusion juice | 2.2 x2 | 13. | <0.01 | | |
| Diffusion juice, fortified | +1 mg/kg | | 0.12 | 0.12 | |
| Thin juice | 2.2 x2 | 133 | <0.01 | | |
| Thin juice, fortified | +1 mg/kg | | <0.01 | 0.01 | GH-C 729 |
| Thick juice | 2.2 x2 | 133 | <0.01 | | |
| Thick juice, fortified | +1 mg/kg | | <0.01 | 0.01 | |
| Sugar | 2.2 x2 | 133 | ---- | | |
| Sugar, fortified | + 1 mg/kg | | ---- | | |
| Molasses | 2.2 x2 | 133 | ---- | | |
| Molasses, fortified | + 1 mg/kg | | ---- | | |

[1] LOQ 0.01 mg/kg. Results not corrected for recoveries
[2] Recoveries averaged 87% from roots, 99% from lime cake, 103% to 106% from juice, 90% from wet pulp and 100% from dry pulp.

Maize (corn). In two processing studies in 1990 in the USA, maize was treated with 5 applications of chlorpyrifos at 8.4 kg ai/ha total and samples harvested 35 days after the last treatment were processed in a processing plant. Residues of chlorpyrifos in the processed fractions from dry and wet milling were determined. The simulated commercial process is shown in Figures 6a and b (Robb and Schotts, 1993b). The results are shown in Table 121.

Figure 6a. Wet milling processing of maize (corn).

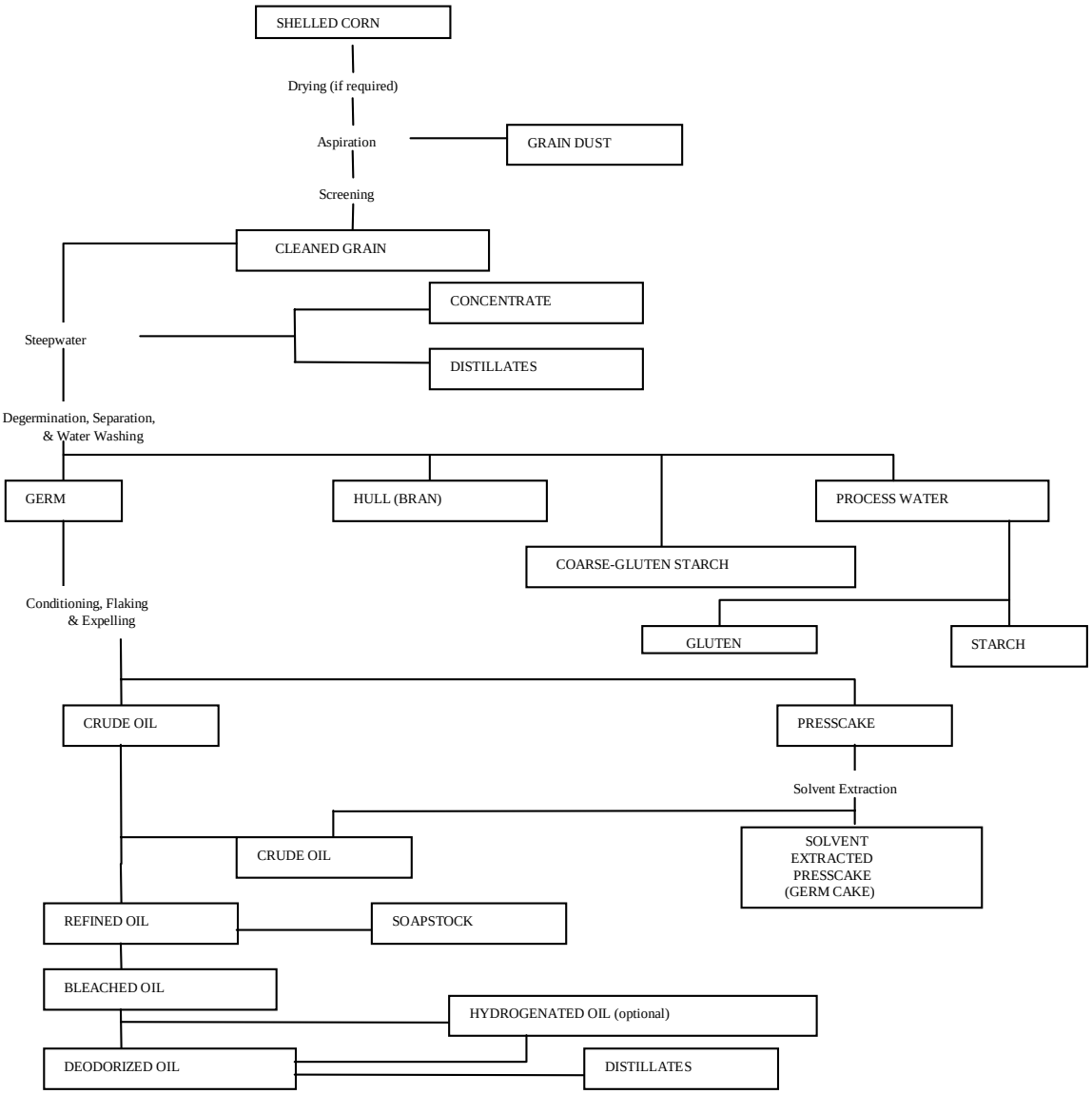


Figure 6b. Dry milling processing of maize.

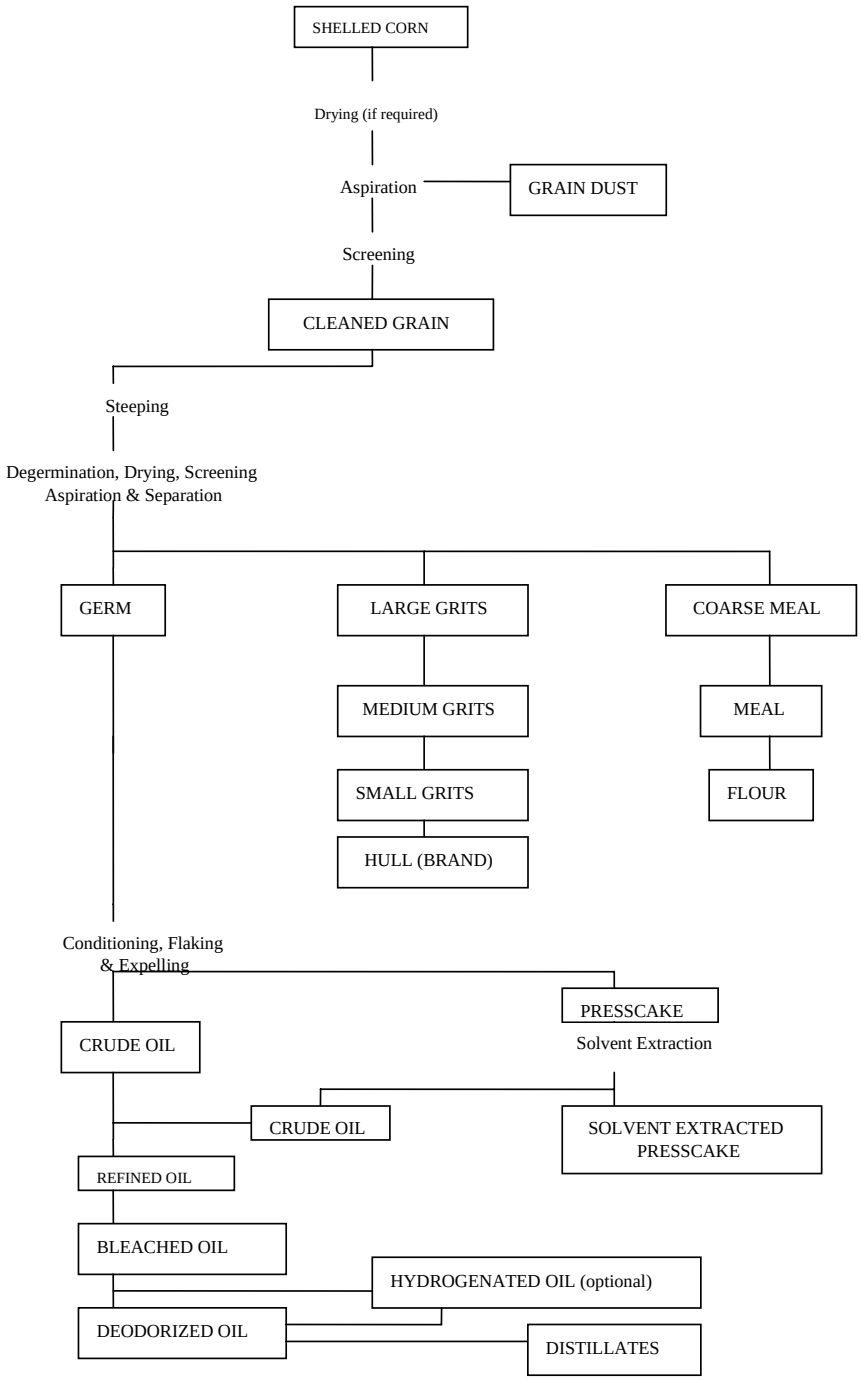


Table 121. Chlorpyrifos residues in maize and its processed fractions.

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg [1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| **Illinois** | | | | | |
| Bulk grain | 8.4 | 35 | 0.01 | 1 | GH-C 2878 |
| *Dry milling* | | | | | |
| Large grits | 8.4 | 35 | <0.01 | 1 | GH-C 2878 |
| Medium grits | 8.4 | 35 | <0.01 | 1 | |
| Small grits | 8.4 | 35 | <0.01 | 1 | |
| Coarse meal | 8.4 | 35 | <0.01 | 1 | |
| Meal | 8.4 | 35 | <0.01 | 1 | |
| Flour | 8.4 | 35 | <0.01 | 1 | |
| Crude oil | 8.4 | 35 | 0.01 | 1 | |
| Refined oil | 8.4 | 35 | 0.01 | | |
| *Wet milling* | | | | | |
| Coarse gluten-starch | 8.4 | 35 | 0.01 | 1 | GH-C 2878 |
| Starch | 8.4 | 35 | <0.01 | 1 | |
| Crude oil | 8.4 | 35 | 0.01 | 1 | |
| Refined oil | 8.4 | 35 | 0.01 | 1 | |
| **Michigan** | | | | | |
| Bulk grain | 8.4 | 35 | 0.04, 0.03 (0.04) | | GH-C 2878 |
| *Dry milling* | | | | | |
| Large grits | 8.4 | 35 | 0.01 | 0.2 | GH-C 2878 |
| Medium grits | 8.4 | 35 | 0.03 | 0.8 | |
| Small grits | 8.4 | 35 | 0.04 | 1 | |
| Coarse meal | 8.4 | 35 | 0.05 | 1.2 | |
| Meal | 8.4 | 35 | 0..5 | 1.2 | |
| Flour | 8.4 | 35 | 0.07 | 1.8 | |
| Crude oil | 8.4 | 35 | 0.06 | 1.5 | |
| Refined oil | 8.4 | 35 | 0.06 | 1.5 | |
| *Wet milling* | | | | | |
| Coarse gluten-starch | 8.4 | 35 | 0.07 | 1.8 | GH-C 2878 |
| Starch | 8.4 | 35 | <0.01 | 0.2 | |
| Crude oil | 8.4 | 35 | 0.12 | 3 | |
| Refined oil | 8.4 | 35 | 0.13 | 3.2 | |

[1] LOQ 0.01 mg/kg. Results not corrected for recoveries
[2] Recoveries averaged 88% from grain, 90% from dry and wet milling fractions, 71% from oil.

Rice. Rice plants in the Philippines were treated with single applications of EC formulation at 1.05 kg ai/ha, 10 days before harvest. Samples of the grain were taken at random, sundried and milled. Chlorpyrifos residues were determined in the processed fractions by GLC with flame photometric detection (Cowles *et al.*, 1999e). In a trial in Australia, a single broadcast application of 1 kg ai/ha chlorpyrifos 500 EC was applied to rice 10 days before harvest, and samples were taken, sun-dried and processed at a commercial facility. Chlorpyrifos was determined in each of the processed fractions (Cowles *et al.*, 1999f). The results are shown in Table 122.

Table 122. Residues of chlorpyrifos in rice grain and its processed fractions.

| Sample | Application, kg ai/kg | PHI, days | Residues, mg/kg [1] | Processing factor | Reference |
|---|---|---|---|---|---|
| **Australia, 1999** | 1 | 10 | | | GHF-P 1795 |
| Paddy rice | | | 0.33 | 1 | |
| Brown rice | | | 0.06 | 0.2 | |
| White rice | | | 0.01 | 0.03 | |
| Rice hulls | | | 1.41 | 4.3 | |
| Rice pollard | | | 0.36 | 1.1 | |
| Rice thrash | | | 1.85 | 5.6 | |
| **Philippines, 1999** | 1.05 | 10 | | | GHF-P 1794 |
| Unhulled rice | | | 1.55 | 1 | |
| Hulled rice | | | 0.10 | 0.06 | |
| Rice bran | | | 3.89 | 2.5 | |
| Rice hulls | | | 4.06 | 2.6 | |

[1]LOQ 0.01 mg/kg. Recoveries averaged 90% from all processed fractions

Sorghum. Plants were treated 3 times with chlorpyrifos at 0.28 kg ai/ha beginning when heads were about 30% in flower. Samples of grain were collected at normal harvest, 49 days after the last treatment in Mississippi (6 x 15 m row) and 62 days in Kansas (0.2 ha)) and submitted for milling. One grain sample was collected in Mississippi (45 kg) and six replicate samples in Kansas (60 kg total). The grain was tempered to 15.5% moisture before milling, and the grain and milling fractions analysed for chlorpyrifos (Wetters and Miller, 1978). The results are shown in Table 123.

Table 123. Residues of chlorpyrifos in grain and milling fractions of sorghum.

| Sample | Application, kg ai/kg | PHI, days | Residues, mg/kg [1,2] | Processing factor | Reference |
|---|---|---|---|---|---|
| **MS** | | | | | |
| Grain | 0.28 x3 | 42 | 0.04 | 1 | GH-C 1109 |
| Flour | 0.28 x3 | 42 | 0.01 | 0.2 | |
| Shorts[3] | 0.28 x3 | 42 | 0.04 | 1 | |
| Middlings[4] | 0.28 x3 | 42 | 0.01 | 0.2 | |
| Bran[5] | 0.28 x3 | 42 | 0.06 | 1.5 | |
| Screenings | 0.28 x3 | 42 | 0.11 | 2.8 | |
| Germ | 0.28 x3 | 42 | 0.06 | 1.5 | |
| **KS** | | | | | |
| Grain | 0.28 x3 | 69 | 0.01 | 1 | GH-C 1109 |
| Flour | 0.28 x3 | 69 | 0.01 | 1 | |
| Shorts | 0.28 x3 | 69 | 0.01 | 1 | |
| Middlings | 0.28 x3 | 69 | 0.01 | 1 | |
| Bran | 0.28 x3 | 69 | 0.01 | 1 | |
| Screenings | 0.28 x3 | 69 | <0.01 | 1 | |
| Germ | 0.28 x3 | 69 | <0.01 | 1 | |

[1] LOQ 0.01 mg/kg in grain, 0.05 mg/kg in milling and baking fractions
[2] Recoveries averaged 85% from grain, 79% from flour, 92% from shorts, 88% from middlings, 90% from bran, 82% from screenings and 77% from germ
[3] Shorts is finely ground particles of bran, endosperm, and some germ
[4] Middlings refers to a product between whole grain and flour
[5] Bran refers to pericap, seed coat and the aleurone layer

Wheat. Wheat plots in Illinois, USA, were treated with three aerial or ground applications of chlorpyrifos at 0.56 kg ai/ha, and samples of grain collected at normal harvest for determination of the residues before and after milling and baking (Norton, 1980b). The results are shown in Table 124.

Table 124. Residues of chlorpyrifos in wheat grain and milling and baking fractions.

| Sample | Application, kg ai/kg | PHI, days | Residues, mg/kg [1] | Processing factor | Reference |
|---|---|---|---|---|---|
| Grain | 0.56 x3 | 14 | 0.51 | 1 | GH-C 1346 |
| Bran | 0.56 x3 | 14 | 1.3 | 2.5 | |
| Straight grade flour | 0.56 x3 | 14 | 0.08 | 0.2 | |
| Break shorts | 0.56 x3 | 14 | 0.88 | 1.7 | |
| Reduction shorts | 0.56 x3 | 14 | 1.2 | 2.4 | |
| Red dog | 0.56 x3 | 14 | 0.41 | 0.8 | |
| Bread | 0.56 x3 | 14 | 0.05 | 0.1 | |

[1] LOQ 0.01 mg/kg in grain and 0.05 mg/kg in milling and baking fractions. Recoveries averaged 92% from grain, 84% from baking and milling fractions.

Cotton. Chlorpyrifos was applied twice or four times at 0.28 and 12 or 13 times at 1.12 kg ai/ha at various times during May/October 1974 to cotton in Mississippi (ground) and Texas (aerial). Cotton was picked by hand and samples collected 18 days after aerial and 7 days after ground applications. The cotton samples were ginned and seeds and gin trash collected. The seed samples were processed into linters, hulls, solvent-extracted meal, crude oil and refined bleached oil (McKellar, 1975). The process is shown in Figure 7 and the results in Table 125.

Figure 7. Processing of cotton seed.

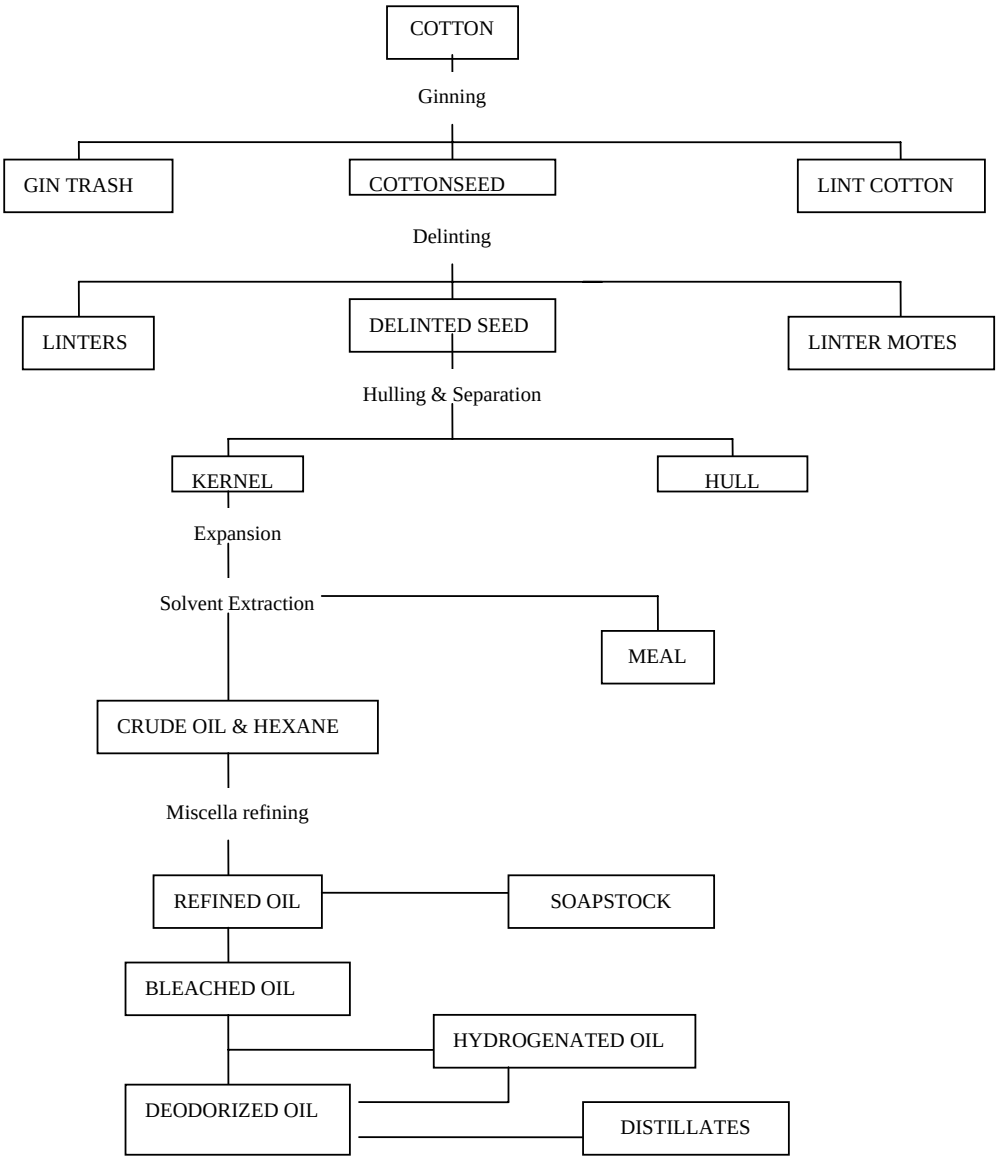


Table 125. Residues of chlorpyrifos in cotton seed and processed fractions.

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg [1] | Processing factor | Reference |
|---|---|---|---|---|---|
| Ground application (MS) | | | | | |
| Seed | 0.28 x2 + 1.12 x12 | 15 | 0.043±0.016 (0.031-0.066) (control 0.009) | 1 | GH-C 840 |
| Gin trash | | | 75 (control 1.3) | 1700 | |
| Hulls | | | <0.01 | 0.2 | |
| Linters | | | 0.04 | 1 | |
| Solvent extracted meal | | | <0.01 | 0.2 | |
| Crude oil | | | 0.01 | 0.2 | |
| Refined oil | | | 0.01 | 0.2 | |
| Aerial application (TX) | | | | | |
| Seed | 0.28 x4 + 1.12 x13 | 18 | 0.103 ±0.019 (0.078-0.123) (control 0.016) | 1 | GH-C 840 |
| Gin trash | | | 0.50 (control 0.36) | Invalid | |
| Hulls | | | 0.07 | 0.5 | |
| Linters | | | 0.52 | 5.0 | |
| Solvent extracted meal | | | 0.01 | 0.1 | |
| Crude oil | | | 0.14 | 1.4 | |
| Refined oil | | | <0.01 | 0.1 | |

[1] LOQ 0.01 mg/kg. Recoveries averaged 94% from seed, 90% from gin trash, 100% from crude oil.

Peanuts. In several processing trials on peanuts to determine the residues of chlorpyrifos in processed oil the 15G formulation of chlorpyrifos was applied twice at a rate equivalent to 1.12 kg ai/ha. Samples of peanuts were taken 49 days after the last treatment and processed to pressed cake and oil (Miller, 1979b). The results are shown in Table 126.

Table 126. Residues of chlorpyrifos in peanuts and processed fractions (USA).

| Sample | Application, kg ai/kg | PHI, days | Residues, mg/kg[1] | Processing factor | Reference |
|---|---|---|---|---|---|
| Kernels | 1.12 x2 | 49 | 0.03 | 1 | GH-C 1199 |
| Press cake | 1.12 x2 | 49 | 0.04 | 1.3 | |
| Pressed oil | 1.12 x2 | 49 | 0.07 | 2.3 | |

[1] LOQ 0.02 mg/kg for kernels and 0.05 mg/kg for processed fractions. Recoveries averaged 90% from kernels, 88% from pressed cake and 86% from oil.

In another trial, chlorpyrifos was applied to the plants at 1.12 kg ai/ha at bloom and again 49 days before harvest. Samples were collected at normal harvest for processing into crude oil, with a Carver press, and then to refined oil and soapstock. The concentration of chlorpyrifos in the peanuts was not determined, but fortified crude oil from untreated peanuts was also processed to refined oil and soapstock (Miller, 1980e). The results are shown in Table 127.

Table 127. Residues of chlorpyrifos in peanut oil fractions from peanuts treated with two applications of 15 G formulation (USA).

| Sample | Application, kg ai/kg | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|
| Crude oil | 1.12 x2 | 49 | 0.07 | GH-C 1278 |
| Refined oil | 1.12 x2 | 49 | 0.05 | |
| Soapstock | 1.12 x2 | 49 | 0.02 | |
| Crude oil fortified with 0.25 mg/kg chlorpyrifos | | | | |
| Refined oil | | | 0.18 | GH-C 1278 |

| Sample | Application, kg ai/kg | PHI, days | Residues, mg/kg [1] | Reference |
|---|---|---|---|---|
| Soapstock | | | 0.07 | |

[1] LOQ 0.01 mg/kg.
[2] Recoveries averaged 92% from crude oil, 90% from refined oil and 91% from soapstock.

Sunflowers. In a processing study in 1993 sunflowers were treated with 3 applications of EC at 1.7 kg ai/ha/application. Samples of whole seeds were collected and cracked and residues determined in the whole seed (kernel + hull), seed (RAC) and hulls (Turner, 1994).

In another trial, application of the 15 G chlorpyrifos formulation at planting was followed by 5 aerial applications of the EC formulation. Samples were collected 45 days after the last treatment (0.6 kg ai/ha) and processed, following commercial practices. Residues of chlorpyrifos were determined in the processed fractions (Miller, 1981b). The results are shown in Table 128.

Table 128. Residues of chlorpyrifos in sunflower seed and processed fractions.

| Sample | Application, kg ai/ha | PHI, days | Residues, mg/kg [1] | Processing factor | Reference |
|---|---|---|---|---|---|
| Hull + kernel | 1.7 x3 | 42 | 0.08 | - | GH-C 3239 |
| Hull | 1.7 x3 | 42 | 0.16 | - | |
| Seed | 1.7 x3 | 42 | 0.04 | - | |
| Seed | 0.6 x3 + 1.7 x2 + 0.012 kg ai/100 m row x1 | 45 | 0.03 | 1 | GH-C 1468 |
| Meal | 0.6 x3 + 1.7 x2 + 0.012 kg ai/100 m row x1 | 45 | 0.01 | 0.3 | GH-C 1468 |
| Hulls | 0.6 x3 + 1.7 x2 + 0.012 kg ai/100 m row x1 | 45 | 0.51 | 17 | GH-C 1468 |
| Crude oil | 0.6 x3 + 1.7 x2 + 0.012 kg ai/100 m row x1 | 45 | 0.02 | 0.7 | GH-C 1468 |
| Refined oil | 0.6 x3 + 1.7 x2 + 0.012 kg ai/100 m row x1 | 45 | 0.01 | 0.3 | GH-C 1468 |
| Soapstock | 0.6 x3 + 1.7 x2 + 0.012 kg ai/100 m row x1 | 45 | 0.01 | 0.3 | GH-C 1468 |

[1]LOQ 0.01 mg/kg. Recoveries averaged 91% from seeds, 77% from meal, 97% from hulls, 75% from oil and 81% from soapstock.

Coffee. In a trial in Brazil coffee plants were treated with 3 applications of 480 EC formulation at the maximum label rate of 0.72 kg ai/ha (1.5 l/ha) or at a double rate (Catta-Preta and Rampazzo, 1997). Twenty-one days after the last application, samples of unshelled beans were allowed to dry for 20 days at 17-48$^{o}$ C in a greenhouse, shelled in a small-scale machine and some were then roasted in a country-style coffee roaster. A freeze-drying process further processed a sub-set of the latter into instant coffee. After each process samples were frozen and shipped for analysis.

In another field trial in Columbia a 480 EC chlorpyrifos formulation was applied 3 times to plants at 0.96 and 1.92 kg ai/ha. Samples were collected 0, 2, 7, 14, 21 and 28 days after the last application. Ripe beans gathered by hand were shelled wet in a small-scale machine, fermented in water for 48 hours and dried in sunshine for 4-6 days. The unshelled fraction (green beans) and the shelled and dried fraction (divided into 3 subfractions) were packed in polyethylene bags, frozen and sent for processing. Two of the subfractions were roasted in a home-style roaster and one of these was converted to instant coffee by grinding and extracting with boiling water. All fractions were analysed for chlorpyrifos (Catta-Preta and Rampazzo, 1999). The results are shown in Table 129.

Table 129. Residues of chlorpyrifos in coffee and processed fractions.

| Location, year, sample | Application, kg ai/ha | PHI, days | Residues, mg/kg [1] | Processing factor | Reference |
|---|---|---|---|---|---|
| **Brazil, 1997** | | | | | |
| Shelled beans | 0.72 | 21 | 0.02, 0.02, 0.03 (0.02) | 1 | GHB-P 310 |
| | 1.44 | 21 | 0.06, 0.05, 0.12 (0.08) | 1 | |
| Roasted beans | 0.72 | 21 | <0.01, <0.01, <0.01 (0.01) | 0.5 | |
| | 1.44 | 21 | <0.01, <0.01, <0.01 (0.01) | 0.1 | |
| Instant coffee | 0.72 | 21 | <0.01, <0.01, <0.01 (0.1) | 0.5 | |
| | 1.44 | 21 | <0.01, <0.01, <0.01 (0.01) | 0.1 | |
| **Columbia, 1999** | | | | | |
| Green beans with shell | 0.96 | 0 | 1.14, 1.98, 0.84,, 2.28 | | GHB-P 413 |
| | | 2 | 0.29, 0.77, 0.58, 0.58 | | |
| | | 7 | 0.1, 0.29, 0.26, 0.38 | | |
| | | 14 | 0.11, 0.17, 0.14, 0.23 (0.14) | | |
| | | 21 | 0.05, 0.08, 0.15, 0.05 | | |
| | | 28 | 0.04, 0.04, 0.05, 0.07 | | |
| Green beans with shell | 1.92 | 0 | 2.12, 2.90, 3.98, 3.45 | | |
| | | 2 | 0.85, 2.27, 0.60, 1.67 | | |
| | | 7 | 0.72, 1.4, 0.68, 0.79 | | |
| | | 14 | 0.31, 0.60, 0.44, 0.79 (0.54) | | |
| | | 21 | 0.22, 0.44, 0.43, 0.38 | | |
| | | 28 | 0.08, 0.20, 0.28, 0.18 | | |
| Shelled dried beans | 0.96 | 0 | <0.01, 0.03, 0.05, 0.04 (0.03) | | |
| | | 2 | <0.01, 0.03, 0.03, 0.03 (0.02) | | |
| | | 7 | <0.01, 0.02, 0.02, 0.02 (0.02) | | |
| | | 14 | <0.01, 0.02, 0.02, 0.02 (0.02) | 1 | |
| | | 21 | <0.01, 0.01, 0.02, 0.02 (0.02) | | |
| | | 28 | <0.01, 0.01, 0.02, 0.02 (0.02) | | |
| | 1.92 | 0 | 0.04.0.14, 0.10, 0.09 (0.09) | | |
| | | 2 | 0.02, 0.07, 0.08, 0.09 (0.06) | | |
| | | 7 | 0.02, 0.06, 0.06, 0.04 (0.04) | | |
| | | 14 | 0.01, 0.05, 0.05, 0.03 (0.04) | 1 | |
| | | 21 | 0.03, 0.04, 0.05, 0.03 (0.04) | | |
| | | 28 | 0.02, 0.02, 0.05, 0.02 (0.03) | | |
| Roasted beans | 0.96 | 14 | <0.01, <0.01, <0.01, <0.01 (0.01) | 0.05 | |
| | 1.92 | 14 | <0.01, 0.01, 0.02, <0.01 (0.01) | 0.25 | |
| Instant coffee | 0.96 | 14 | <0.01, <0.01, <0.01, <0.01 (0.01) | 0.07 | |
| | 1.92 | 14 | <0.01, <0.01, <0.01, <0.01 (0.01) | 0.02 | |

[1] LOQ 0.01 mg/kg. Recoveries in Brazil averaged 83%, 80% and 84% from dried beans, roasted beans and instant coffee. Recoveries in Columbia averaged 82%, 80%, 95%, and 95% from green beans, shelled dried beans, roasted beans and instant coffee respectively.

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The manufacturer reported monitoring data from the USA, where chlorpyrifos is determined in several pesticide monitoring programmes. Among them are the California Environmental Protection Agency Food Safety Program, the Food and Drug Administration Enforcement

Monitoring Program, the US Department of Agriculture Pesticide Data Program (PDP), and the Pesticide Residues Information System (PRIS).

The residue monitoring of the California Environmental Protection Agency (Cal-EPA) Food Safety Programs is designed to enforce laws and regulations involving pesticide registrations in California. Samples of the commodities are collected randomly at points of entry, packing sites, wholesale distribution centres and retail outlets and analysed for a wide range of pesticides.

The FDA Enforcement Monitoring Programs are designed to enforce tolerances established by the US EPA. Raw agricultural commodities sampled at the "farm gate" are analysed. Samples are classified as either surveillance or compliance. Most of them are surveillance samples. Compliance samples are collected as follow-up to findings of illegal residues or when there is evidence of a pesticide residue problem.

The USDA Pesticide Data Program (PDP) was developed to collect residue data on fresh fruits and vegetables. Sampling has been designed to be statistically representative of the US food supply. Samples of peeled and/or washed fruits and vegetables are analysed. The Program collects residue data for the US EPA dietary risk assessments and to aid decisions on re-registration and special reviews.

The Pesticide Residue Information System (PRIS) has developed the FOODCONTAM national database to compile and summarize existing data on the analyses of domestically-produced foods for residues of pesticides and industrial chemicals. The system is administered by The Mississippi State Chemical Laboratory under contract with the US FDA. Most of the data are from routine state surveillance programmes.

Table 130 summarizes the residue data for chlorpyrifos compiled by the manufacturer from the above US sources (Tomerlin *et al.*, 1995).

Table 130. Summary of chlorpyrifos residue data from national food survey and monitoring programmes in the USA.

| Commodity | No. of samples | Mean, mg/kg | Min., mg/kg | Max., mg/kg | No. >LOD | % >LOD |
|---|---|---|---|---|---|---|
| Cal-EPA 1986-1991 | | | | | | |
| Alfalfa | 8 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Almonds | 188 | 0.067 | 0.005 | 2.6 | 10 | 5.32 |
| Apples | 1733 | 0.007 | 0.005 | 1.0 | 34 | 2.0 |
| Bananas | 404 | 0.006 | 0.005 | 1.0 | 10 | 2.5 |
| Beans | 1096 | 0.005 | 0.005 | 0.09 | 2 | 0.18 |
| Beets, roots | 207 | 0.005 | 0.005 | 0.01 | 1 | 0.48 |
| Beets, tops | 228 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Brussels | 378 | 0.010 | 0.005 | 0.79 | 8 | 2.1 |
| Broccoli | 825 | 0.005 | 0.005 | 0.03 | 3 | 0.36 |
| Cabbage | 1071 | 0.005 | 0.005 | 0.48 | 1 | 0.09 |
| Carrots | 661 | 0.006 | 0.005 | 0.73 | 2 | 0.30 |
| Cauliflower | 572 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Citrus | 8 | 0.051 | 0.005 | 0.28 | 2 | 25 |
| Corn | 806 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Egg plant | 494 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Grapefruit | 733 | 0.007 | 0.005 | 0.19 | 17 | 2.3 |
| Grapes | 1930 | 0.005 | 0.005 | 0.40 | 2 | 0.10 |
| Kale | 136 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Kiwifruit | 180 | 0.01 | 0.005 | 0.23 | 19 | 10 |
| Lemons | 607 | 0.01 | 0.005 | 0.39 | 65 | 11 |

| Commodity | No. of samples | Mean, mg/kg | Min., mg/kg | Max., mg/kg | No. >LOD | % >LOD |
|---|---|---|---|---|---|---|
| Lettuce-head | 943 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Lettuce-leaf | 1670 | 0.005 | 0.005 | 0.26 | 3 | 0.18 |
| Mushrooms | 304 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Onions | 1607 | 0.005 | 0.005 | 0.06 | 1 | 0.06 |
| Oranges | 1262 | 0.04 | 0.005 | 0.84 | 235 | 19 |
| Other nuts | 221 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Peaches | 741 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Peanuts | 11 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Pears | 955 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Peas | 596 | 0.006 | 0.005 | 0.34 | 2 | 0.33 |
| Peppers | 3049 | 0.01 | 0.005 | 0.71 | 166 | 5.4 |
| Plums | 684 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Potatoes | 1704 | 0.007 | 0.005 | 2.0 | 3 | 0.18 |
| Strawberries | 781 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Sugar beet | 286 | 0.007 | 0.005 | 0.27 | 2 | 0.70 |
| FDA Monitoring, 1992-1993 | | | | | | |
| Apples | 919 | 0.01 | 0.002 | 0.32 | 139 | 15 |
| Banana | 342 | 0.009 | 0.005 | 0.23 | 72 | 21 |
| Berries | 1593 | 0.005 | 0.002 | 0.26 | 21 | 1.3 |
| Br. sprouts | 40 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Broccoli | 378 | 0.005 | 0.005 | 0.04 | 4 | 1.1 |
| Cabbage | 306 | 0.006 | 0.005 | 0.12 | 3 | 0.98 |
| Carrots | 303 | 0.005 | 0.005 | 0.009 | 1 | 0.33 |
| Cauliflower | 210 | 0.005 | 0.005 | 0.00 | 0 | 0.00 |
| Citrus fruit | 737 | 0.02 | 0.002 | 0.450 | 165 | 22 |
| Corn grain | 48 | 0.005 | 0.005 | 0.00 | 0. | 0.00 |
| Egg plant | 119 | 0.005 | 0.005 | 0.04 | 1 | 0.84 |
| Eggs | 526 | 0.005 | 0.005 | 0.01 | 1 | 0.19 |
| Kale | 109 | 0.01 | 0.005 | 0.40 | 4 | 3.7 |
| Kiwifruit | 199 | 0.03 | 0.005 | 0.800 | 24 | 12 |
| Lettuce, head | 360 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Lettuce, leaf | 615 | 0.005 | 0.005 | 0.04 | 9 | 1.5 |
| Milk | 636 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Mushrooms | 383 | 0.007 | 0.005 | 0.13 | 24 | 0.06 |
| Nuts | 175 | 0.005 | 0.005 | 0.02 | 4 | 2.3 |
| Onion, bulb | 210 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Peach | 353 | 0.007 | 0.005 | 0.20 | 22 | 6.2 |
| Pears | 282 | 0.005 | 0.005 | 0.07 | 5 | 1.8 |
| Peppers | 1497 | 0.010 | 0.005 | 0.93 | 105 | 7.0 |
| Plum | 145 | 0.005 | 0.005 | 0.03 | 2 | 1.4 |
| Potato | 520 | 0.005 | 0.005 | 0.02 | 1 | 0.19 |
| Rice grain | 120 | 0.005 | 0.005 | 0.01 | 10 | 8.3 |
| Soya beans | 127 | 0.007 | 0.005 | 0.08 | 6 | 4.7 |
| Tomatoes | 627 | 0.007 | 0.005 | 0.33 | 28 | 4.5 |
| Wheat grain | 197 | 0.005 | 0.005 | 0.03 | 9 | 4.6 |
| PDP Monitoring, 1992 | | | | | | |
| Apples | 567 | 0.010 | 0.002 | 0.640 | 100 | 18 |
| Bananas | 564 | 0.003 | 0.002 | 0.006 | 0 | 0.00 |
| Broccoli | 153 | 0.005 | 0.002 | 0.140 | 8 | 5.2 |
| Carrots | 153 | 0.003 | 0.002 | 0.006 | 0 | 0.00 |
| Grapefruit | 567 | 0.003 | 0.002 | 0.01 | 1 | 0.18 |
| Grapes | 552 | 0.007 | 0.002 | 0.77 | 24 | 4.3 |
| Beans | 466 | 0.003 | 0.002 | 0.01 | 1 | 0.21 |
| Lettuce | 565 | 0.003 | 0.002 | 0.09 | 3 | 0.53 |
| Oranges | 569 | 0.004 | 0.002 | 0.01 | 18 | 3.2 |

| Commodity | No. of samples | Mean, mg/kg | Min., mg/kg | Max., mg/kg | No. >LOD | % >LOD |
|---|---|---|---|---|---|---|
| Peaches | 360 | 0.004 | 0.002 | 0.07 | 31 | 8.61 |
| Potatoes | 568 | 0.003 | 0.002 | 0.006 | 0 | 0.00 |
| PRIS (FOODCONTAM) Monitoring, 1988-1991 | | | | | | |
| Apples | 1638 | 0.03 | 0.003 | 24.0 | 137 | 8.4 |
| Banana | 283 | 0.02 | 0.005 | 0.30 | 81 | 29 |
| Blueberries | 105 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Broccoli | 814 | 0.005 | 0.005 | 0.03 | 1 | 0.12 |
| Br. sprouts | 227 | 0.009 | 0.005 | 0.50 | 4 | 1.76 |
| Cabbage | 1190 | 0.007 | 0.005 | 1.13 | 5 | 0.42 |
| Carrots | 713 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Cauliflower | 506 | 0.005 | 0.005 | 0.07 | 1 | 0.20 |
| Ch. cabbage | 310 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Egg plant | 280 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Grapefruit | 300 | 0.007 | 0.005 | 0.18 | 6 | 2.0 |
| Grapes | 1063 | 0.006 | 0.005 | 0.24 | 10 | 0.94 |
| Kale | 352 | 0.008 | 0.003 | 0.51 | 6 | 1.7 |
| Lemon | 394 | 0.02 | 0.005 | 0.39 | 56 | 14 |
| Lettuce | 3276 | 0.005 | 0.005 | 0.26 | 6 | 0.18 |
| Milk/cream | 7290 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Mushrooms | 261 | 0.005 | 0.005 | 0.005 | 0 | 0.00 |
| Oranges | 871 | 0.03 | 0.005 | 0.63 | 183 | 21 |
| Peaches | 597 | 0.005 | 0.005 | 0.03 | 6 | 1.0 |
| Pears | 616 | 0.005 | 0.005 | 0.02 | 4 | 0.65 |
| Pepper, sweet | 1031 | 0.007 | 0.005 | 0.20 | 22 | 2.1 |
| Plums | 458 | 0.005 | 0.005 | 0.04 | 1 | 0.22 |
| Potatoes | 871 | 0.006 | 0.005 | 0.26 | 7 | 0.80 |
| Strawberry | 691 | 0.005 | 0.002 | 0.06 | 5 | 0.72 |
| Tomatoes | 1171 | 0.006 | 0.005 | 0.45 | 17 | 1.4 |

The Government of The Netherlands reported monitoring data on food in commerce for 1994-1996, 1997 and 1998. The information is shown in Table 131.

Table 131. Residues of chlorpyrifos in food in commerce in The Netherlands, 1994-1998.

| Commodity | No. of samples | | | % ≥ 0.05 mg/kg | | |
|---|---|---|---|---|---|---|
| | 1994-1996 | 1997 | 1998 | 1994- 1996 | 1997 | 1998 |
| **Citrus fruit** | | | | | | |
| Grapefruit | 301 | 87 | 35 | 9.6 | 1.1 | 11 |
| Lemon | 243 | 335 | 24 | 8.6 | 15 | 8.3 |
| Orange | 902 | 112 | 124 | 11. | 14 | 18 |
| Pomelo | | | 4 | | | 25 |
| Tangerine | 560 | 21 | 70 | 12 | 4.8 | 16 |
| Other citrus fruit | | | 12 | | | 8.4 |
| **Pome fruit** | | | | | | |
| Apple | 1495 | 398 | | 1.9 | 1.2 | |
| **Stone fruit** | | | | | | |
| Apricot | 80 | | | 0 | | |
| Cherry | 252 | | | 0.4 | | |
| Peach | 252 | | | 1.2 | | |
| Nectarine | 221 | | | 0 | | |
| Plum | | 85 | | | 1.2 | |
| **Berries and small fruit** | | | | | | |
| Grape | 667 | 196 | 99 | 3.6 | 1.0 | 3.0 |
| Strawberry | 2378 | 779 | | 0.2 | 0.2 | |

| Commodity | No. of samples | | | % ≥ 0.05 mg/kg | | |
|---|---|---|---|---|---|---|
| | 1994-1996 | 1997 | 1998 | 1994- 1996 | 1997 | 1998 |
| Currant | 450 | | | 0 | | |
| **Miscellaneous fruit** | | | | | | |
| Avocado | 125 | | | 0.8 | | |
| Banana | 57 | | | 0 | | |
| Date | | 8 | | | 12 | |
| Kiwifruit | 223 | 60 | | 2.2 | 1.7 | |
| Litchi | 351 | | | 0 | | |
| Mango | 191 | | | 0.5 | | |
| Other fruits and fruit products | 385 | 152 | 51 | 1.3 | 1.3 | 2.0 |
| **Fruiting vegetables** | | | | | | |
| Tomato | 1108 | | | 0.2 | | |
| Pepper | 1525 | 607 | 213 | 0.3 | 0.5 | 0.5 |
| Aubergine (egg plant) | 148 | | | 0.7 | | |
| Melon | 390 | | | 0 | | |
| **Brassica vegetables** | | | | | | |
| Broccoli | 154 | 62 | 42 | 0.6 | 3.2 | 4.8 |
| Brussels sprouts | | | 47 | | | 2.1 |
| Chinese cabbage | 297 | | | 0.3 | | |
| **Leaf vegetables and fresh herbs** | | | | | | |
| Lamb's lettuce | 268 | | | 0.7 | | |
| Iceberg lettuce | 471 | | | 0 | | |
| Lettuce | 3306 | | | 0.09 | | |
| Endive | 1137 | | | 0 | | |
| Other herbs | 148 | | | 0 | | |
| **Stem vegetables** | | | | | | |
| Asparagus | 244 | | | 0.4 | | |
| Celery | 233 | | 105 | 1.7 | | 0.9 |
| Other stem vegetables | 341 | | | 0 | | |
| **Pulses** | | | | | | |
| Beans, fresh with pods | 617 | | | 0 | | |
| **Root and tuber vegetables** | | | | | | |
| Potatoes | 325 | | | 0.3 | | |
| Carrot | | 164 | | | 0.6 | |
| Beetroot | | | 20 | | | 5.0 |
| **Cereals** | | | | | | |
| Rice | 96 | | | 0 | | |
| Other arable products | 699 | | | 0.1 | | |

The Government of Poland submitted monitoring data on chlorpyrifos for the years 1997 and 1998, as shown in Table 132.

Table 132. Residues of chlorpyrifos in food in commerce in Poland, 1997-1998.

| Commodity | LOQ, mg/kg | No. of samples | | % ≥LOQ | |
|---|---|---|---|---|---|
| | | 1997 | 1998 | 1997 | 1998 |
| Apple | 0.05 | 78 | 77 | 0 | 0 |
| Carrot | 0.02 | 180 | 167 | 0 | 0 |
| Cabbage, head | 0.03 | 141 | 119 | 7.8 | 7.6 |
| Onion, bulb | 0.04 | 46 | 90 | 0 | 1.1 |
| Potato | 0.02 | 277 | 258 | 0 | 0 |

## NATIONAL MAXIMUM RESIDUE LIMITS

The National MRLs listed below were supplied by the manufacturer and the governments of The Netherlands, Poland and the USA. The USA supplied revised tolerances (MRLs) arising from the Reregistration Eligibility Decision for chlorpyrifos.

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| Argentina | Alfalfa | 4 |
| | Apple | 0.2 |
| | Apple | 0.2 |
| | Asparagus | 0.5 |
| | Barley | 0.05 |
| | Bell pepper | 0.5 |
| | Citrus fruits | 0.3 |
| | Corn | 0.05 |
| | Cotton seed | 0.05 |
| | Cotton seed oil | 0.05 |
| | Flax seed | 1 |
| | Garlic | 0.5 |
| | Oats | 0.05 |
| | Olive | 0.5 |
| | Onion bulb | 0.05 |
| | Pear | 0.2 |
| | Potato | 0.05 |
| | Rye | 0.05 |
| | Sorghum | 0.05 |
| | Soya bean | 0.01 |
| | Stone fruits | 0.5 |
| | Sunflower seed | 0.05 |
| | Sunflower seed oil | 0.1 |
| | Sweet corn (kernels) | 0.05 |
| | Tomato | 0.5 |
| | Wheat | 0.05 |
| Austria | Cereal | 0.05 |
| | Corn | 0.05 |
| | Carrot | 0.1 |
| | Hops (dry) | 0.1 |
| | Kiwifruit (pulp) | 0.1 |
| | Kiwifruit | 2 |
| | Fruit (others) | 0.2 |
| | Citrus fruits without peel | 0.2 |
| | Sugar beet | 0.05 |
| Australia | Asparagus | 0.5 |
| | Avocado | 0.5 |
| | Banana | 0.5T |
| | Brassica (cole or cabbage) vegetables | 0.5 |
| | Cabbage, head | 0.5 |
| | Flowerhead brassicas | 0.5 |
| | Cassava | 0.02* |
| | Cattle, edible offal | 2 |
| | Cattle meat (in the fat) | 2 |
| | Cereal grains (except sorghum) | 0.1 |
| | Citrus fruits | 0.5 |
| | Cotton seed | 0.05 |
| | Cotton seed oil, crude | 0.2 |
| | Dried fruits | 2 |
| | Grapes | 1 |
| | Eggs | 0.01T |
| | Ginger, root | 0.01* |
| | Kiwifruit | 2 |
| | Mango | 0.05* |
| | Milks (in the fat) | 0.2T |
| | Oilseed | 0.01 |
| | Passion fruit | 0.05* |
| | Pig, edible offal | 0.1 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Pig meat (in the fat) | 0.1 |
| | Pineapple | 0.5 |
| | Pome fruits | 0.2 |
| | Potato | 0.05 |
| | Poultry, edible offal | 0.1T |
| | Poultry meat (in the fat) | 0.1T |
| | Sheep, edible offal | 0.1 |
| | Sheep, meat (in the fat) | 0.1 |
| | Sorghum | 3 |
| | Stone fruits | 1 |
| | Strawberry | 0.05 |
| | Sugar cane | 0.1 |
| | Tomato | 0.5 |
| | Tree nuts | 0.2 |
| | Vegetables (except asparagus, brassica, cassava, celery, potato, tomato, sweet potato) | 0.01* |
| | Cotton fodder, dry | 30 |
| | Cotton meal and hulls | 0.05 |
| Belgium | Beef (meat) | 2 |
| | Beet | 0.2 |
| | Bilberry (red or red whortleberry) | 0.1 |
| | Blackberry | 0.1 |
| | Carrot | 0.1 |
| | Citrus fruit | 0.3 |
| | Corn (sweet) | 0.2 |
| | Corn salad | 0.2 |
| | Currant | 0.1 |
| | Endive | 0.2 |
| | Eggs, egg products | 0.01 |
| | Food (except as otherwise listed) | 0.05 |
| | Gooseberry | 0.1 |
| | Grapes | 0.5 |
| | Herbs | 0.2 |
| | Kiwifruit | 2 |
| | Lettuce | 0.2 |
| | Milk and milk products | 0.01 |
| | Onion | 0.2 |
| | Parsnip | 0.2 |
| | Plum | 0.2 |
| | Pome fruits | 0.5 |
| | Sheep (meat | 0.2 |
| | Strawberries | 0.2 |
| | Tea (green, black) | 0.1 |
| | Turnip (garden) | 0.2 |
| | Turnip (Swedish turnip or Swede fodder) | 0.2 |
| | Vegetables (legume) | 0.2 |
| | Vegetables (stalk)) | 0.2 |
| Brazil | Apple | 1 |
| | Bean (field) | 0.1 |
| | Bean (dry) | 0.1 |
| | Beef (fat) | 2 |
| | Cabbage | 1 |
| | Carrot | 0.5 |
| | Cereal grains | 0.75 |
| | Citrus fruit | 0.3 |
| | Coffee (bean) | 0.02 |
| | Corn | 0.1 |
| | Cotton (seed oil), cotton (seed, whole) | 0.05 |
| | Grasses | 2 |
| | Hay or fodder (dry) or grasses | 6 |
| | Kale | 1 |
| | Milk (fat basis) | 0.01 |
| | Peanut | 0.01 |
| | Miscellaneous fodder and forage | 1.5 |
| | Potato | 0.01 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Poultry fats | 0.2 |
| | Sheep fat | 0.2 |
| | Sorghum | 0.01 |
| | Soya bean | 0.01 |
| | Tomato | 0.5 |
| | Wheat | 0.01 |
| Canada | Apples | 1.5 |
| | Citrus fruits | 1 |
| | Kiwifruit | 2 |
| | Peppers | 1 |
| | Rutabagas | 0.5 |
| | Meat and meat by-products of cattle, fat content basis | 1 |
| | Fat, liver and kidney of cattle | 1 |
| | Apples | 1 |
| | Beans (dry) | 0.2 |
| | Beef (carcasses, fat) | 2 |
| | Carrot | 0.5 |
| | Citrus fruit | 0.3 |
| Chile | Eggs | 0.01* |
| | Grapes | 1 |
| | Lettuce | 0e1 |
| | Pear | 0.5 |
| | Potato | 0.05 |
| | Poultry (fat) | 0.1 |
| | Rice | 0.1 |
| | Sheep (carcasses, fat basis) | 0.2 |
| | Sugar beet | 0.05 |
| | Tomato | 0.5 |
| Denmark | Carrot | 0.5 |
| | Citrus fruit | 0.5 |
| | Fruit (other) | 0.5 |
| | Potato | 0.05 |
| | Vegetables (leafy) | 0.5 |
| | Artichoke | 1 |
| | Bananas | 3 |
| | Barley | 0.2 |
| | Blackberries | 0.5 |
| EU | Cherries | 0.3 |
| | Chinese cabbage | 0.5 |
| | Cranberries | 0.05* |
| | Currants | 1 |
| | Flowerhead Brassica | 0.05* |
| | Gooseberries | 1 |
| | Head cabbage | 1 |
| | Herbs | 0.05* |
| | Lamb | 0.05* |
| | Legume vegetables | 0.05* |
| | Lemon | 0.2 |
| | Lettuce | 0.05* |
| | Mandarin | 2 |
| | Onions | 0.2 |
| | Other cane fruits | 0.05* |
| | Other head brassicas | 0.05* |
| | Other leaf brassicas | 0.05* |
| | Other stem vegetables | 0.05* |
| | Peaches | 0.2 |
| | Radish | 0.2 |
| | Raspberries | 0.5 |
| | Scarole | 0.05* |
| Finland | Vegetables | 0.1 |
| | Kiwifruit | 2 |
| France | Beans, French | 0.2 |
| | Carrot | 0.1 |
| | Cereals | 0.05 |
| | Citrus fruit | 0.3 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Fruit (Other), vegetables (Other) | 0.05* |
| | Grapes | 0.5 |
| | Kiwifruit | 2 |
| | Maize | 0.05 |
| | Oilseed | 0.05 |
| | Onion | 0.2 |
| | Pome fruits | 0.5 |
| | Soya beans (dry) | 0.05 |
| | Stone fruit | 0.2 |
| | Strawberries | 0.2 |
| | Tea (green, black) | 0.1 |
| | Sunflower seed | 0.05 |
| Germany | Carrot | 0.1 |
| | Citrus fruit | 0.3 |
| | Coffee (bean, raw) | 0.2 |
| | Corn | 0.05 |
| | Fruit | 0.1 |
| | Grapes | 0.5 |
| | Hops (dry) | 0.1 |
| | Kiwifruit | 2 |
| | Legumes (Stored or for direct consumption) | 0.05 |
| | Peppers (bell) | 0.1 |
| | Plum | 0.2 |
| | Pome fruit | 0.5 |
| | Strawberry | 0.2 |
| | Sugar beet | 0.05 |
| | Tomato | 0.1 |
| | Vegetables (bulb) | 0.1 |
| | Vegetables (root, tuber, stalk, sprouts) | 0.05 |
| Hungary | Cereals | 0.1 |
| | Corn | 0.05 |
| | Pome fruits | 0.2 |
| | Sunflower seed | 0.1 |
| India | Cabbage | 0.01 |
| | Cauliflower | 0.01 |
| | Cotton (seed) | 0.05 |
| | Cotton (seed, oil, crude) | 0.025 |
| | Food grains | 0.05 |
| | Food grains (milled) | 0.01 |
| | Fruit | 0.5 |
| | Onion | 0.01 |
| | Potato | 0.01 |
| | Vegetables (other) | 0.2 |
| Israel | Alfalfa forage (green) | 0.1 |
| | Artichoke (globe) | 0.05 |
| | Citrus fruit | 0.3 |
| | Corn (Sweet, kernels) | 0.05 |
| | Cotton (seed) | 0.05 |
| | Grapes | 1 |
| Italy | Cabbage | 0.2 |
| | Citrus (fruit) | 0.2 |
| | Corn | 0.05 |
| | Fruit | 0.5 |
| | Peach | 0.2 |
| | Pear (Japanese) | 0.5 |
| | Pome fruits | 0.2 |
| | Potato | 0.2 |
| | Sugar beet | 0.2 |
| | Tomato | 0.5 |
| Japan | Apple | 1 |
| | Beet root | 0.05 |
| | Citrus fruit | 0.3 |
| | Grapes | 1 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Mandarin | 0.3 |
| | Oranges, sweet, sour | 0.5 |
| | Peach | 1 |
| | Pear, Japanese | 0.5 |
| | Plums (including prunes) | 1 |
| | Sweet potato | 0.1 |
| | Tea, green, black | 3 |
| Korea | Apple | 1 |
| | Cabbage | 0.5 |
| | Chinese cabbage | 1 |
| | Citron | 0.5 |
| | Citrus fruits | 0.5 |
| | Garlic | 0.5 |
| | Onion bulb | 0.5 |
| | Peach | 0.5 |
| | Pear | 0.5 |
| | Tea, green, black | 1 |
| Malaysia | Cocoa | 0.01 |
| Mexico | Alfalfa fodder | 4 |
| | Apple | 1.5 |
| | Bean (forage, green) | 1 |
| | Bean (string) | 0.05 |
| | Broccoli | 1 |
| | Cattle meat | 2 |
| | Citrus fruit | 1 |
| | Coffee beans | 0.2 |
| | Corn | 0.1 |
| | Cotton (seed) | 0.5 |
| | Cucumber | 0.1 |
| | Peach | 0.05 |
| | Pear | 0.05 |
| | Peppers | 1 |
| | Rice | 0.1 |
| | Sorghum (grain) | 0.75 |
| | Sorghum forage, green | 1.5 |
| | Soya beans | 0.5 |
| | Sugarcane | 0.1 |
| | Sweet potato | 0.1 |
| | Tomato | 0.5 |
| | Wheat | 0.5 |
| New Zealand | Banana | 2 |
| | Fruit (other) | 0.2 |
| | Fruit (stone) | 1 |
| | Grapes | 1 |
| | Kiwifruit | 2 |
| | Meat fat in any food | 1.5 |
| | Tomato | 0.2 |
| Netherlands | Beef (meat) | 2 |
| | Bananas | 3 |
| | Barley | 0.2 |
| | Blackberries | 0.5 |
| | Cabbage, head | 1 |
| | Carrots | 0.1 |
| | Cherries | 0.3 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Chicken (meat) | 0.05* |
| | Chinese cabbage | 0.5 |
| | Citrus fruit (except lemons and mandarins) | 0.3 |
| | Currants (red, black and white) | 1 |
| | Eggs | 0.01* |
| | Globe artichokes | 1 |
| | Gooseberries | 1 |
| | Grapes (table and wine) | 0.5 |
| | Hops | 0.1* |
| | Kiwifruit | 2 |
| | Lemons | 0.2 |
| | Mandarins | 2 |
| | Onions | 0.2 |
| | Peaches (including nectarines) | 0.2 |
| | Plums | 0.2 |
| | Radishes | 0.2 |
| | Raspberries | 0.5 |
| | Sheep (meat) | 0.2 |
| | Strawberries | 0.2 |
| | Milk | 0.01* |
| | Pome fruit | 0.5 |
| | Raspberries (red, black) | 0.2 |
| | Solanacea | 0.5 |
| | Tea | 0.1* |
| Poland | Cereal grains | 0.05 |
| | Citrus fruits | 0.3 |
| | Eggs | 0.01 |
| | Fruiting vegetables, cucurbits | 0.5 |
| | Fruiting vegetables, other than cucurbits | 0.5 |
| | Meat and meat products | 0.2 |
| | Milks and milk product | 0.01 |
| | Poultry meat | 0.05 |
| | Pome fruits | 0.5 |
| | Potato | 0.05 |
| | Stone fruits | 0.2 |
| | Sugar beet | 0.05 |
| | Tea | 0.1 |
| | Vegetables except as otherwise noted | 0.1 |
| Romania | Eggs (less shell) | 0.01 |
| | Poultry (meat) | 0.2 |
| | Meat (cattle, pigs and sheep) | 2 |
| South Africa | Apple | 0.05 |
| | Apricot | 0.05 |
| | Banana | 0.5 |
| | Carrot | 0.05 |
| | Citrus | 0.3 |
| | Grapes | 0.5 |
| | Lettuce | 0.05 |
| | Peach | 0.3 |
| | Pear | 0.3 |
| | Plum | 0.3 |
| | Potato | 0.3 |
| | Tomato | 0.5 |
| | Wheat | 0.3 |
| Spain | Alfalfa | 0.05 |
| | Aubergine | 0.5 |
| | Banana | 0.2 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Brassica | 0.05 |
| | Cabbage | 1 |
| | Carrot | 0.1 |
| | Cereal grains | 0.05 |
| | Citrus fruit | 0.3 |
| | Cucurbits | 0.05 |
| | Grapes | 0.5 |
| | Hazelnuts | 0.05 |
| | Leafy vegetables | 0.05 |
| | Lettuce head | 0.5 |
| | Lettuce leaves | 0.5 |
| | Maize | 0.05 |
| | Olive | 0.2 |
| | Pepper | 0.5 |
| | Pome fruit | 0.5 |
| | Potato | 0.05 |
| | Sorghum | 0.05 |
| | Strawberry | 0.2 |
| | Sugar beet | 0.05 |
| | Tomato | 0.5 |
| | Tree nuts | 0.05 |
| Sweden | Milk | 0.003 |
| | Eggs | 0.01 |
| | Meat (raw material) | 0.02 |
| | Potato | 0.05 |
| | Butter | 0.1 |
| | Cheese | 0.1 |
| Switzerland | Milk | 0.005 |
| | Vegetables | 0.05 |
| | Citrus (fruit, in pulp) | 0.1 |
| | Kiwifruit )pulp) | 0.1 |
| | Citrus (whole fruit) | 0.3 |
| | Oil (wheat germ) | 0.5 |
| | Kiwifruit (whole) | 2 |
| Taiwan | Banana | 1 |
| | Bulb vegetable | 0.5 |
| | Citrus | 2 |
| | Coconut | 1 |
| | Large berry | 1 |
| | Leafy vegetable | 1 |
| | Litchi | 1 |
| | Maize | 0.5 |
| | Pineapple | 1 |
| | Pome fruit | 1 |
| | Rice | 0.1 |
| Ukraine | Apples | 0.05 |
| | Hops, dry | 1 |
| | Peach | 0.05 |
| | Potato | 0.05 |
| | Sugar beet | 0.05 |
| USA | Alfalfa forage | 3 |
| | Alfalfa hay | 13 |
| | Almonds | 0.2 |
| | Almond hulls | 12 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Apple | 0.01 |
| | Apple pomace, wet | 0.02 |
| | Asparagus | 5 |
| | Banana, whole | 0.1 |
| | Banana, pulp | 0.01 |
| | Cattle fat | 0.3 |
| | Cattle meat | 0.05 |
| | Cattle, meat by-products | 0.05 |
| | Chinese Cabbage | 1 |
| | Citrus | 1 |
| | Citrus oil | 20 |
| | Citrus pulp, dried | 5 |
| | Corn, field, grain | 0.05 |
| | Corn, field, fodder | 8 |
| | Corn, field, forage | 8 |
| | Corn oil | 0.25 |
| | Cotton seed | 0.2 |
| | Cranberries | 1 |
| | Cucumber | 0.05 |
| | Eggs | 0.01 |
| | Figs | 0.01 |
| | Filbert | 0.2 |
| | Goats, fat | 0.2 |
| | Goat, meat | 0.05 |
| | Goat, meat by-products | 0.05 |
| | Grapes | 0.01 |
| | Hogs, fat | 0.2 |
| | Hogs, meat | 0.05 |
| | Hogs, meat by-products | 0,05 |
| | Horses, fat | 0.25 |
| | Horses, meat | 0.25 |
| | Horses, meat by-products | 0.25 |
| | Kiwifruit | 2 |
| | Legumes (except soya beans) | 0.05 |
| | Lettuce | 1 |
| | Macadamia nuts | 0.2 |
| | Milk fat | 0.25 |
| | Nectarines | 0.05 |
| | Onions, dry bulb | 0.5 |
| | Peach | 0.05 |
| | Peanuts | 0.2 |
| | Peanut oil | 0.2 |
| | Pears | 0.01 |
| | Pecans | 0.2 |
| | Peppermint, tops | 0.8 |
| | Peppermint oil | 8 |
| | Peppers | 1 |
| | Plums | 0.05 |
| | Poultry, fat (incl turkeys) | 0.1 |
| | Poultry, meat (incl turkeys) | 0.1 |
| | Pumpkin | 0.05 |
| | Radishes | 2 |
| | Rutabagas | 0.5 |
| | Sheep, fat | 0.2 |

| Country | Commodity | MRL, mg/kg |
|---|---|---|
| | Sheep, meat | 0.05 |
| | Sheep, meat by-products | 0.05 |
| | Soya bean, grain | 0.3 |
| | Sorghum, fodder | 6 |
| | Sorghum, forage | 1.5 |
| | Sorghum, Grain | 0.75 |
| | Spearmint, tops | 0.8 |
| | Spearmint oil | 8 |
| | Strawberries | 0.2 |
| | Sugar beet molasses | 15 |
| | Sugar beet, pulp, dried | 5 |
| | Sugar beet, roots | 1 |
| | Sugar beet, tops | 8 |
| | Sunflower seeds | 0.1 |
| | Sweet potato | 0.05 |
| | Turnip greens | 0.3 |
| | Turnips | 1 |
| | Vegetables, leafy brassica cole | 1 |
| | Walnuts | 0.2 |
| | Wheat forage | 3.0 |
| | Wheat grain | 0.5 |
| | Wheat straw | 6.0 |
| Venezuela | Banana | 0.25 |
| | Cabbage | 0.05 |
| | Coffee beans | 0.01 |
| | Corn | 0.1 |
| | Cotton seed | 0.05 |
| | Onion, bulb | 0.01 |
| | Peanut | 0.01 |
| | Plantain | 0.1 |
| | Potato | 0.05 |
| | Rice | 0.1 |
| | Sesame | 0.01 |
| | Sugarcane | 0.1 |
| | Sorghum | 10 |
| | Tomato | 0.5 |
| Zimbabwe | Carrots | 0.05 |
| | Citrus | 0.3 |
| | Cucurbits | 0.05 |
| | Fruiting vegetables, except cucurbits | 0.05 |
| | Leafy vegetables | 0.05 |
| | Maize | 0.05 |
| | Potato | 0.05 |

* Residue at the LOQ.
T: temporary

## APPRAISAL

### *Metabolism*

*Animals*

Two female *goats* were fed [$^{14}$C]chlorpyrifos in gelatin capsules twice daily for 10 days for a total dose of 0.26 mCi/goat per day and dietary intakes of 15 and 19 ppm. Urine and faeces contained 79-89% of the administered dose, and about 2% was found in milk and tissues combined. The concentration of residue in milk attained a maximum on day 8 (0.047 mg/kg) and then fell slightly. The concentrations in the tissues of the two goats, respectively, expressed as equivalents of chlorpyrifos, were fat, 0.10 and 0.22; liver, 0.18 and 0.27; kidney, 0.26 and 0.35; muscle, 0.03 and 0.03; and skin, 0.11 and 0.18 mg/kg. When tissues were hydrolysed with 0.6 N potassium hydroxide, >94% of the radiolabelled residue in all tissues and 92-94% of that in milk was 3,5,6-trichloropyridinol. Chlorpyrifos and 3,5,6-trichloropyridinol represented 70 and 14% of the recovered activity in solvent extracts of milk, 76 and 21% in fat, 1.9 and 84% in liver, and 0.9 and 92% in kidney. The oxygen analogue of chlorpyrifos was not detected.

In a study in *poultry*, acclimatized white Leghorn laying hens received a daily oral dose of 2.26 mg of [$^{14}$C-2 and $^{14}$C-6]chlorpyrifos for 10 days. Ring C-2 is adjacent to the thiophosphate. The concentrations of chlorpyrifos equivalents in treated tissues were kidney, 0.15 mg/kg; liver, 0.054 mg/kg; muscle, 0.10 mg/kg; fat, 0.20 mg/kg; skin, 0.13 mg/kg; gizzard, 0.024 mg/kg; and heart, 0.068 mg/kg. Eggs were separated into yolk and whites and combined by group and day. The concentration of radiolabel in the whites reached a plateau of 0.026 mg/kg on day 7, and that in the yolks appeared to reach a plateau of 0.15 mg/kg on day 9 or 10. Chlorpyrifos and 3,5,6-trichlorpyridinol accounted for 72% of the total radiolabel in kidney, 81% in egg yolk, <2% in liver, 65% in hydrolysed liver, 83% in skin, and 89% in fat.

The Meeting concluded that chlorpyrifos is metabolized in livestock to 3,5,6-trichloropyridinol and derivatives thereof, which are released by base hydrolysis. The Meeting also concluded that the residues are concentrated to a greater degree in fat than in muscle.

*Plants*

The metabolism of [$^{14}$C-2 and $^{14}$C-6]chlorpyrifos was studied in leaves of *maize (corn), soya bean, and sugar beet.* A total of 24 maize plants were maintained in a chamber which permitted collection of volatile products, and radiolabelled chlorpyrifos was applied to the upper surfaces of the leaves as 1-µl drops up to a typical total dose of 200 µg of chlorpyrifos per plant. At intervals, the treated leaf areas were excised, rinsed with methanol, and analysed or homogenized in 75% acetone to extract metabolites. The untreated plant parts were also analysed to determine the extent of translocation as a function of time. The radiolabel that could be removed by rinsing with a solvent decreased from 99% on the day of application to 1% on day 4, while the volatile radiolabel increased from 0 to 84% of the applied dose. The amount of translocated radiolabel did not represent more than 0.8% of the applied dose. The combined surface rinses and leaf extracts did not contain more than 10% of the applied dose 8-16 days after application, and the amount of radiolabel that could not be extracted did not exceed 3% of the applied dose. The extracts contained chlorpyrifos (0.1-0.4% of the applied dose) and polar metabolites. Acid hydrolysis, base hydrolysis, or enzyme hydrolysis of the extracts released 25-58% of the radiolabel in the extracts as 3,5,6-trichlorpyridinol.

*Maize (field corn)* was treated twice with radiolabelled chlorpyrifos, once by ground application at planting (223 mg ai/m of row) and again by foliar application (1.7 kg ai/ha). Green forage was harvested 49 days after the foliar application, and grain and fodder were harvested after 153 days. The concentrations of chlorpyrifos equivalents were 1.6 mg/kg in green forage, 4.2 mg/kg in dry fodder, and 0.13 mg/kg in grain. About 3% of the total residue in forage was chlorpyrifos, and 1% was 3,5,6-trichloropyridinol. Base hydrolysis of the green forage solubilized 90% of the total radiolabelled residue, and 30% was identified as 3,5,6-trichloropyridinol. A similar result was obtained with dry fodder, except that trichloromethoxypyridine was tentatively identified as representing 3% of the residue. Corn forage was further characterized by sequential extraction as containing 17% polysaccharide, 10% hemicellulose, and 26% lignin. The residue in grain could not be released by mild base hydrolysis, but sequential extraction revealed 4% in protein, 14% in cellulose, 8% in gluten, and 34% in starch.

*Soya beans* were sprayed in mid-season with [$^{14}$C]chlorpyrifos at a rate of 1.12 kg ai/ha. Forage was sampled 14 days after treatment, and beans and field trash were sampled at the normal harvesting time 52 days after treatment. The forage was found to contain 46% of the total radiolabelled residue as chlorpyrifos and 24% as 3,5,6-trichloropyridinol, free and conjugated. The beans contained 2.6% of the residue as chlorpyrifos, 8.8% as free 3,5,6-trichloropyridinol, and 66% as incorporated (protein).

*Sugar beets* were given two applications of [$^{14}$C]chlorpyrifos in a manner analogous to the field corn. Green foliage was taken 38 days after the soil application and before the foliar application, and tops and mature beets were harvested 108 days after the foliar application. The green foliage contained primarily polar radiolabelled compounds, 90% of which were extractable. Alkaline hydrolysis of the extract released 3,5,6-trichloropyridinol, representing 57% of the total residue. When mature beet tops were hydrolysed with base, 65% of the total radiolabel was released. About 29% of the radiolabel was associated with 3,5,6-trichloropyridinol. Solvent extraction of the tops released a mixture of polar compounds, accounting for 45% of the total residue. Methanol extraction of the beet roots released 85% of the residue. About 40% of the total residue was shown to be sucrose. Also present were the methoxypyridine (7% of the total residue), 3,5,6-trichloropyridinol (36%), and chlorpyrifos (<0.5%).

An *apple* tree was sprayed nine times with a wettable powder formulation of chlorpyrifos, and in the last two applications [$^{14}$C]chlorpyrifos was admixed with unlabelled compound. The apples were harvested 14 days after the final treatment. Most of the radiolabel was found in the peel, with 0.8 mg/kg in peel and 0.005 mg/kg in flesh. In the peel, 36% of the residue was chlorpyrifos, 5.3% was free 3,5,6-trichloropyridinol, 1.2% was conjugated 3,5,6-trichloropyridinol, 5% was unknown compounds converted by refluxing base hydrolysis to 3,5,6-trichloropyridinol, and 15% was postulated to be natural products.

A study of confined rotational crops was conducted in which *carrots*, *lettuce*, and *wheat* were planted in soil treated with [$^{14}$C]chlorpyrifos 30 and 132 days after treatment. The concentrations of residue ranged from 0.19 mg/kg in carrot roots planted 30 days after treatment to 1.3 mg/kg in wheat straw planted after 132 days. In the carrot roots, chlorpyrifos represented 2.0% of the residue; trichloropyridinol, 10%; trichloromethoxy-pyridine, 26%; and glucose, 21%. In wheat straw planted 30 days after treatment, trichloropyridinol represented 4.3% of the residue; cellulose, 13%; lignin, 17%; and glucose, 21%, In wheat grain planted 30 days after treatment, the values were trichloropyridinol, 0.3%; cellulose, 8.5%; starch, 46%; and glucose, 49%. The identification of glucose was tentative.

The Meeting concluded that chlorpyrifos is metabolized to 3,5,6-trichloropyridinol, which is then conjugated or further degraded. Much of the chlorpyrifos is ultimately

incorporated into natural components (such as protein, cellulose, and lignin) of the plants. The Meeting also concluded that chlorpyrifos has a low to moderate tendency to translocate from the site of application.

***Environmental fate***

Under aerobic conditions in loam soil, chlorpyrifos degraded to $CO_2$ over 360 days. The maximal concentration of 3,5,6-trichloropyridinol represented 4.3% of the applied dose at about day 60, and that of 3,5,6-trichloromethoxypyridine represented 1.6% at about day 30. The conversion was slower in clay soil. The degradation of 3,5,6-trichloropyridinol in soil involved extensive mineralization, with an average half-life of 73 days. Under anaerobic conditions in loam soil, 92% was converted to 3,5,6-trichloropyridinol over 360 days and none to 3,5,6-trichloromethoxypyridine.

Owing to its nonpolar nature, chlorpyrifos is sparsely soluble in water and tends to partition from aqueous into organic phases in the environment. It has a strong affinity for soil, as evidenced by an average soil and sediment sorption coefficient ($K_{oc}$) of 8500 ml/g (range, 970-31 000) in 28 laboratory studies in which the batch equilibrium method was used. 3,5,6-Trichloropyridinol shows only moderate sorption, with $K_{oc}$ values of 18-390 ml/g (average, 160 ml/g).

Several studies have been conducted of leaching, in both the laboratory and the field. A typical study involved 30-cm glass columns packed with Commerce loam (0.68% organic carbon), Tracy sandy loam (1.1% organic carbon), or Catlin silty clay loam (2.0% organic carbon) which were treated with [$^{14}$C]chlorpyrifos at 0.5 kg/ha and eluted with 51 cm of water. Most of the chlorpyrifos (95-99%) remained in the top 2 cm of the column, and none moved beyond the upper 5 cm of soil. A maximum of 1.3% of the applied radiolabel appeared in the leachates. Field studies were conducted under natural conditions of rainfall and irrigation. Chlorpyrifos applied at 1.1-2.2 kg ai/ha remained in the top 20 cm of soil throughout the growing season. One of the studies indicated that 3,5,6-trichloropyridinol has at least a moderate tendency to leach. When chlorpyrifos was applied three times at 1.12 kg ai/ha during the growing season in a citrus grove, with a rainfall of 110 cm and irrigation with 48 cm, it was confined to the upper 15 cm of soil, but 3,5,6-trichloropyridinol was found at a depth of 46 cm.

The Meeting concluded that chlorpyrifos is converted in soil to 3,5,6-trichloropyridinol and ultimately to $CO_2$. The Meeting also concluded that chlorpyrifos has no tendency to leach from the soil, but that the metabolite 3,5,6-trichloropyridinol has a moderate tendency to do so.

***Methods of analysis***

Methods for both enforcement and data collection and monitoring have been developed for the determination of chlorpyrifos in plant and animal matrices, soil, and water. Various extraction and clean-up methods are followed by analysis by gas chromatography with a flame photometric detector or, infrequently, an electron capture detector. Gas chromatography with mass spectrometry may be used for confirmation. A variation involves base hydrolysis of the matrix, which converts chlorpyrifos and conjugated 3,5,6-trichloropyridinol to 3,5,6-trichloropyridinol. The limit of determination is 0.01 mg/kg for methods for the determination of chlorpyrifos and 0.05 mg/kg for those for 3,5,6-trichloropyridinol.

The Meeting concluded that adequate analytical methods are available for the enforcement of MRLs and for monitoring.

***Stability of residues in stored analytical samples***

Substantial data were made available on the stability of chlorpyrifos in frozen crop matrices. Generally, no loss occurred over 360 days of frozen storage, except from walnuts and almonds (20-23% loss within 258 days), oranges and orange juice (20% loss within 170 days), sorghum silage (23% loss within 65 days), and sugar beet roots (37% loss within 150 days).

Only summary information was provided on the stability of chlorpyrifos in animal commodities. The data for muscle, liver, and kidney were variable. In subcutaneous fat, 60-86% of the incurred residue remained after 41 months of frozen storage. About 74% of chlorpyrifos added at 0.1 or 1.0 mg/kg to whole milk remained after 49 months of frozen storage.

The Meeting concluded that chlorpyrifos is stable in crop matrices stored frozen for up to 1 year. Insufficient detail was provided on animal commodities.

***Definition of the residue***

The studies on animal and plant metabolism and on environmental fate indicate that use of chlorpyrifos could result in the presence of the parent compound and the major metabolite 3,5,6-trichloropyridinol (free and conjugated) in agricultural commodities. The 1999 Meeting considered the trichloropyridinol metabolite during its deliberations, but established an ADI and an acute RfD only for the parent compound. Analytical methods for enforcement purposes are available for the determination of chlorpyrifos residues in plant and livestock commodities, soil, and water.

The octanol/water partition coefficient for chlorpyrifos, log $P_{OW}$ = 4.7, indicates that chlorpyrifos is fat-soluble. This conclusion is confirmed by the results of studies in goats and poultry, in which the concentration of radiolabelled material in fat was up to 10 times that in muscle.

The Meeting concluded that the residue definition both for compliance with MRLs and estimation of dietary intake should be chlorpyrifos and that chlorpyrifos should be designated as fat-soluble.

***Results of supervised trials***

The results of supervised trials were provided for citrus (mandarin, orange, grapefruit, lemon), apple, pear, peach, plum, blueberry, caneberry, strawberry, grape, banana, kiwifruit, broccoli, Brussels sprout, cabbage, Chinese cabbage, cauliflower, pepper, tomato, soya, pea, carrot, potato, onion, lettuce, common bean, sugar beet, maize (corn), sweet corn, grain sorghum, rice, wheat, alfalfa, almond, pecan, peanut, sunflower, and coffee.

Data on the relevant GAP were not available to evaluate the data on blueberry, egg plant, and leaf lettuce. The percentage moisture was not available for any of the animal feed commodities, such as alfalfa, and the default values for dry matter from the *FAO Manual* (FAO, 1997) were used to estimate MRLs on a dry-weight basis, where appropriate.

The results of five field trials on *mandarin orange* conducted according to GAP were presented from Spain (0.10 kg ai/hl, 3 kg ai/ha, 21-day PHI), in which the residue concentrations were 0.15, 0.33, 0.55, 0.99, and 1.2 mg/kg. Five trials on *oranges* were reported from South Africa at GAP (0.048 kg ai/hl, 60-day PHI), showing concentrations of 0.05, 0.12, 0.14, 0.19, and 0.21 mg/kg. In three trials from the USA (GAP, 0.7 kg ai/hl, 6.7 kg ai/ha foliar treatment, 35-day PHI; 0.5 kg ai/hl, 1.1 kg ai/ha, ground treatment, 28-day PHI),

the concentrations were 0.26 (foliar), 0.41 (foliar), and 0.66 mg/kg (foliar and ground). One trial on *grapefruit* from Spain showed a concentration of 0.10 mg/kg. Trials on citrus fruit from Italy and the USA were not conducted according to GAP and were not evaluated further.

Thus, six trials at GAP rates were available for small citrus (mandarin, lemon) and eight for large citrus (orange, grapefruit). The ranked order of concentrations of chlorpyrifos residues (median in italics) was: 0.05, 0.10, 0.12, 0.14, 0.15, 0.19, ***0.21***, ***0.26***, 0.33, 0.41, 0.55, 0.66, 0.99, and 1.2 mg/kg. The concentrations in small citrus and on large citrus were similar. No data were presented from analyses of pulp, but a study of orange processing showed a threefold reduction in the concentration between a whole orange and its pulp. Using this factor, the Meeting estimated an STMR value of 0.08 mg/kg for citrus pulp from the STMR value for whole citrus fruit (0.24/3). The Meeting estimated a HR value of 0.4 mg/kg for citrus pulp from the HR value for whole citrus fruit (1.2/3), and a maximum residue limit of 2 mg/kg for whole citrus.

One field trial on *apple* from Chile (GAP, 0.06 kg hl/hl, 28-day PHI) showed a concentration of 0.09 mg/kg, two from Italy (GAP, 0.053 kg ai/hl, 30-day PHI) gave values of 0.17 and 0.19 mg/kg, two from New Zealand (GAP, 0.025-0.038 kg ai/hl, 1 kg ai/ha minimum, 14-day PHI) gave values of 0.16 and 0.19 mg/kg, six trials from Germany (at the GAP rate of the UK of 0.96 kg ai/ha, 14-day PHI) showed concentrations of 0.08, 0.13, 0.17, 0.43, 0.53, and 0.94 mg/kg, and two from the UK resulted in values of 0.17 and 0.18 mg/kg. Trials were reported from Brazil, Canada, and the USA but were not conducted according to GAP and were not evaluated further.

For *pear*, field trials were reported from Canada, the UK, and the USA, but no information on GAP was available or the trials were not conducted according to GAP. As GAP rates for apple and pear in the UK are similar, the Meeting agreed to extrapolate the results for apples to pears and to estimate an STMR value and MRL for pome fruit. The ranked order of concentrations in the 13 trials for apples conducted according to GAP was: 0.08, 0.09, 0.13, 0.16. ***0.17*** (3 trials), 0.18, 0.19 (2 trials), 0.43, 0.53, and 0.94 mg/kg. The Meeting estimated an STMR value of 0.17 mg/kg, a HR value of 0.94 mg/kg, and a maximum residue level of 1 mg/kg. The latter replaces the existing MRLs for apples and pears.

Supervised field trials on *peach* were conducted in Chile (14 trials at the GAP rate of 0.06 kg ai/hl, 45-day PHI), with concentrations of 0.017, 0.023, 0.03, 0.04 (4 trials), 0.045, 0.05, 0.07, 0.08, 0.09, 0.13, and 0.25 mg/kg), Greece (one trial at the GAP rate of 0.08 kg ai/hl, 20-day PHI) with a value of 0.33 mg/kg, Spain (one trial at the Greek GAP) showing a concentration of 0.04 mg/kg), Italy (two trials at the GAP rate of 0.054 kg ai/hl, 0.80 kg ai/ha, 30-day PHI) with values of 0.04 and 0.05 mg/kg, and the USA (four trials at the GAP rate of 0.36 kg ai/ha directed to trunk, 14-day PHI), which showed <0.01 mg/kg, reflecting the non-foliar use pattern.

The ranked order of the concentrations of residues after foliar application in 18 trials was 0.017, 0.023, 0.03, ***0.04*** (6 trials), ***0.045***, 0.05 (2 trials), 0.07, 0.08, 0.09, 0.13, 0.25, and 0.33 mg/kg. These values represent the whole fruit, including the pit. For the whole fruit, the Meeting estimated an STMR value of 0.042 mg/kg, a HR value of 0.33 mg/kg, and a maximum residue level of 0.5 mg/kg.

Supervised field trials on *plum* were submitted from Chile (three trials at the GAP rate of 0.06 kg ai/hl, 45-day PHI) with values of 0.002 (2 trials) and 0.005 mg/kg, Japan (two trials at the GAP rate of 0.025 kg ai/hl, 14-day PHI) with values of 0.03 and 0.05 mg/kg, and Germany (four trials at UK GAP rate of 0.38 kg ai/hl, 0.96 kg ai/ha, 14-day PHI) with concentrations of 0.04, 0.08, 0.14, and 0.20 mg/kg. The ranked order of concentrations in the nine trials was 0.002 (2 trials), 0.005, 0.03, ***0.04***, 0.05, 0.08, 0.14, and 0.20 mg/kg. These values represent the whole fruit, including the pit. For the whole fruit, the Meeting estimated

an STMR value of 0.04 mg/kg, a HR value of 0.2 mg/kg, and a maximum residue level of 0.5 mg/kg.

Seven trials on *blueberry* were reported from the USA, but no GAP was reported. The Meeting could not estimate an STMR value or maximum residue level.

Eleven trials on *blackberry*, *boysenberry*, and *raspberry* were reported from the USA, but no GAP was reported. The results of two trials on raspberries were reported from the UK at the GAP rate of 0.14 kg ai/hl, 0.72 kg ai/ha,7-day PHI. The Meeting decided that the results of two trials (0.25 and 0.52 mg/kg) were insufficient for estimating a maximum residue level or an STMR value and recommended withdrawal of the existing MRL for red and black raspberries of 0.2 mg/kg.

Supervised field trials on *strawberry* were reported from the UK (eight trials at the GAP rate of 0.072 kg ai/hl, 0.72 kg ai/ha, 7-day PHI) showing concentrations of 0.04, 0.09 (2 trials), 0.10 (2 trials), 0.12, 0.14, and 0.15 mg/kg) and from the USA (three trials at the GAP rate of 0.30 kg ai/hl, 1.1 kg ai/ha, 21-day PHI) with values of 0.02, 0.04, and 0.07 mg/kg. The ranked order of the concentrations of residues in the 11 trials was 0.02, 0.04 (2 trials), 0.07, ***0.09*** (2 trials), 0.10 (2 trials), 0.12, 0.14, and 0.15 mg/kg. The Meeting estimated an STMR value of 0.09 mg/kg, a HR value of 0.15 mg/kg, and a maximum residue level of 0.3 mg/kg.

Supervised field trials on *grape* were available from France (10 trials at the GAP rate of 0.34 kg ai/ha, 21-day PHI) showing concentrations of 0.02, 0.04, 0.06, 0.07, 0.08 (2 trials), 0.10, 0.14, and 0.15 (2 trials) mg/kg, Italy (two trials at the GAP rate of 0.05 kg ai/hl, 30-day PHI) with concentrations of 0.02 and 0.04 mg/kg, Greece (two trials at the GAP rate of 0.065 kg ai/hl, 0.54 kg ai/ha, 28-day PHI) showing values of 0.09 and 0.32 mg/kg, and South Africa (two trials at the GAP rate of 0.036 kg ai/hl, 28-day PHI) with values of 0.13 and 0.17 mg/kg. The ranked order of concentrations in the 16 trials conducted at GAP was 0.02 (2 trials), 0.04 (2 trials), 0.06, 0.07, ***0.08*** (2 trials), 0.09, 0.1, 0.13, 0.14, 0.15 (2 trials), 0.17, and 0.32 mg/kg. The Meeting estimated an STMR value of 0.085 mg/kg, a HR value of 0.32 mg/kg, and a maximum residue level of 0.5 mg/kg. Although trials were reported from the USA, none was at GAP.

The results of supervised trials on *banana* treated by foliar application were reported from Australia (one trial at the GAP rate of 0.1 kg ai/hl, 1.0 kg ai/ha, 14-day PHI) with a value of 0.03 mg/kg whole fruit and <0.02 mg/kg pulp; South Africa (two trials at the GAP rate of 0.036 kg ai/hl, 28-day PHI) showing 0.07 mg/kg assuming 20% of banana is peel, 0.33 mg/kg of peel, and 0.01 mg/kg of pulp, <0.01 mg/kg of pulp, no data on peel; and Spain (seven trials including five in glasshouses, at the GAP rate of 0.1 kg ai/hl, 21-day PHI) giving values of 0.37 (<0.01 pulp), 0.48 (0.01 pulp), 0.75, 1.1 (2 trials), 1.6 (2 trials) mg/kg of whole fruit. Additional trials were reported on the use of plastic bags impregnated with chlorpyrifos, from Ecuador (one trial at the GAP rate of Colombia, 1%, 1 bag per season, PHI, about 12 weeks: 0.06 whole fruit, <0.01 mg/kg of pulp), Costa Rica (five trials at the GAP rate of Colombia: 0.01, 0.02, 0.04, 0.05, and 0.13 mg/kg of whole fruit; <0.01 (4 trials), 0.01 mg/kg of pulp), Honduras (two trials at the GAP rate of Colombia: 0.01 (2 trials) mg/kg of whole fruit; <0.01 (2 trials) mg/kg of pulp), and the Philippines (two trials at the GAP rate of 1%, 1 bag/season, PHI, about 12 weeks: 0.13 and 0.21 mg/kg of whole fruit; 0.04 and 0.05 mg/kg of pulp).

The ranked order of concentrations of residues on whole bananas after bag treatment was 0.01 (3 trials), 0.02, 0.04, 0.05, 0.06, 0.13 (2 trials), and 0.21 mg/kg. The ranked order of concentrations on whole bananas after foliar treatment was 0.03, 0.07, 0.37, 0.48, 0.75, 1.1 (2 trials), and 1.6 (2 trials) mg/kg. The Meeting estimated a maximum residue level of 2 mg/kg. The ranked order of the concentrations in pulp samples after bag treatment was: <0.01 (7 trials), 0.01, 0.04, and 0.05 mg/kg, whereas those in pulp samples after foliar treatment were

0.01 (4 trials) and 0.05 mg/kg. The Meeting estimated an STMR value of 0.01 mg/kg and a HR value of 0.05 mg/kg for banana pulp.

Four trials on *kiwifruit* were reported from New Zealand, conducted at the GAP rate of 0.025 kg ai/hl, 0.50 kg ai/ha, 14-day PHI, with concentrations of 0.26, 0.75, 1.0, and 1.9 mg/kg. The Meeting concluded that four trials were insufficient to estimate an STMR value, and recommended withdrawal of the existing MRL of 2 mg/kg.

Supervised trials on *onion* were reported from Greece (seven trials at the GAP rate of 0.3 kg ai/hl, 0.96 kg ai/hl when banded, 7- or 20-day PHI) with concentrations of <0.01 (2 trials), 0.02 (2 trials), 0.03, and 0.05 (2 trials) mg/kg; and the UK (four trials at the GAP rate of 0.16 kg ai/hl, 0.96 kg ai/ha, 21-day PHI) with values of 0.04, 0.06, 0.07, and 0.08 mg/kg. One trial of application to seeds at the time of planting was reported from Canada, resulting in a concentration of 0.14 mg/kg. This trial represented a substantially different use, and the results were not used, even though they represent the maximum residue; however, one trial was considered insufficient to estimate a maximum residue level. The ranked order of concentrations in the 11 trials of foliar application at GAP was: <0.01 (2 trials), 0.02 (2 trials), 0.03, ***0.04***, 0.05 (2 trials), 0.06, 0.07, and 0.08 mg/kg. The Meeting estimated an STMR value of 0.04 mg/kg, a HR value of 0.08 mg/kg, and a maximum residue level of 0.2 mg/kg. The latter replaces the existing MRL of 0.05* mg/kg.

Reports were submitted of eight trials on *broccoli* in the USA at the GAP rate of 1.1 kg ai/ha. The PHI is 21 days in California and Arizona and 30 days elsewhere; the Meeting agreed to consider the data from all states at the 21-day PHI. The ranked order of residue concentrations was: <0.01 (3 trials), 0.01, 0.03, 0.05, 0.07, and 1.4 mg/kg. The latter value, from a trial in New Jersey, seemed excessive, but there was no indication of error in the trial conduct. The Meeting estimated an STMR value of 0.02 mg/kg, a maximum residue level of 2 mg/kg, and a HR value of 1.4 mg/kg.

One trial on *Brussels sprouts* was submitted from the USA, but the application rate did not comply with GAP. The Meeting decided that there were insufficient data to estimate a maximum residue level or an STMR value.

Reports of supervised field trials on *cabbage* were available from South Africa (three trials at the GAP rate of 0.024 kg ai/hl, 7-day PHI) showing concentrations of 0.01, 0.21, and 0.22 mg/kg, the UK (five trials at the GAP rate of 0.72 kg ai/ha, 21-day PHI) with values of 0.01, 0.02, 0.10, 0.15, and 0.26 mg/kg, and the USA (15 trials at the GAP rate of 2.5 kg ai/ha at the time of planting, 1.12 kg ai/ha foliar treatment, 21-day PHI. The ranked order of concentrations of residues was: <0.01 (3 trials), 0.01 (3 trials), 0.02, 0.03 (3 trials), 0.10, ***0.15*** (2 trials), 0.21, 0.22 (3 trials), 0.26 (2 trials), 0.4, 0.5, 0.71, and 0.94 mg/kg. The Meeting estimated an STMR value of 0.15 mg/kg, a HR value of 0.94 mg/kg, and a maximum residue level of 1.0 mg/kg. The latter is recommended to replace the existing MRL of 0.05* mg/kg. Trials reported from Brazil did not correspond to GAP.

Six trials on *Chinese cabbage* were reported from the UK at the GAP rate of 0.16 kg ai/hl, 0.96 kg ai/ha, 21-day PHI. The ranked order of the concentrations of residues was 0.04 (2 trials), 0.17, 0.19, 0.34, and 0.60 mg/kg. The Meeting estimated an STMR value of 0.18 mg/kg, a HR value of 0.60 mg/kg, and a maximum residue level of 1.0 mg/kg. The latter confirms the existing MRL of 1 mg/kg.

Five trials on *cauliflower* were reported from the UK at the GAP rate of 0.96 kg ai/ha, 21-day PHI. The ranked order of concentrations was <0.01 (3 trials), 0.01, and 0.02 mg/kg. The Meeting considered that the results of five trials were sufficient, as the residue values were low and showed little variation. The Meeting estimated an STMR value of 0.01 mg/kg, a

HR value of 0.02 mg/kg, and a maximum residue level of 0.05 mg/kg. This replaces the existing MRL of 0.05* mg/kg.

Results for *peppers, sweet* were reported from Spain (three trials at the GAP rate of 0.1 kg ai/hl, 7-day PHI) with values of 0.37, 0.45, and 0.47 mg/kg and the USA (17 trials at the GAP rate of 1.12 kg ai/hl, 7-day PHI). The ranked order of concentrations was 0.01, 0.06, 0.10 (2 trials), 0.13, 0.14, 0.27 (2 trials), 0.30, ***0.37***, ***0.39***, 0.40, 0.45, 0.47, 0.48, 0.52, 0.60 (2 trials), 0.81, and 1.4 mg/kg. The Meeting estimated an STMR value of 0.38 mg/kg, a HR value of 1.4 mg/kg, and a maximum residue level of 2.0 mg/kg. The latter replaces the existing MRL of 0.5 mg/kg.

Reports of supervised field trials on *tomato* were provided from Australia (one trial at the GAP rate of 0.10 kg ai/hl, 3-day PHI) giving a value of 0.13 mg/kg, Brazil (one trial at the GAP rate of 0.72 kg ai/ha, 21-day PHI) showing a concentration of 0.03 mg/kg, Mexico (three trials at the GAP rate of 1 kg ai/ha, 1-day PHI) with concentrations of 0.06, 0.19, and 0.33 mg/kg, South Africa (two trials at the GAP rate of 0.1 kg ai/hl, 4-day PHI) with a value of 0.23 (2 trials) mg/kg, and Spain (two trials at the GAP rate of 0.1 kg ai/hl, 7-day PHI) with values of 0.06 and 0.08 mg/kg. The ranked order of concentrations was 0.03, 0.06 (2 trials), 0.08, ***0.13***, 0.19, 0.23 (2 trials), and 0.33 mg/kg. The Meeting estimated an STMR value of 0.13, mg/kg, a HR value of 0.33 mg/kg, and a maximum residue level of 0.5 mg/kg. This confirms the existing MRL. Although trials were conducted in the USA, none conformed with GAP.

A report on one supervised trial on *egg plant* was received from Turkey, but no GAP was reported. The Meeting regarded the database as inadequate.

Reports on field trials on *head lettuce* were provided from Spain and on *leaf lettuce* from the USA, but no information was provided on GAP. The Meeting could not estimate STMR values or maximum residue levels, given the lack of data.

The results of supervised field trials on *common bean (snap and kidney)* were reported from Italy (three trials at the GAP rate of 0.53 kg ai/ha, foliar treatment, 15-day PHI) and the USA (four trials at the GAP rate of 0.62 g ai/kg, seed treatment). The ranked order of concentrations of residues after foliar treatment was <0.01 (2 trials) and 0.05 mg/kg, and that after seed treatment was <0.01 (2 trials) and 0.01 (2 trials) mg/kg. The Meeting concluded that three or four trials were insufficient for estimating a maximum residue limit or STMR value. The results of seed treatment of peas (see below) were considered suitable for evaluating bean seed treatment. The ranked order of concentrations of residues of chlorpyrifos in common beans and peas with pods after seed treatment at 0.62 kg ai/kg of seed, was <0.01 (3 trials) and 0.01 (5 trials) mg/kg. The Meeting estimated a HR value of 0.01 mg/kg, a maximum residue level of 0.01 mg/kg, and an STMR value of 0.01 mg/kg for common beans. The MRL would replace the existing MRL of 0.2 mg/kg.

The results of four supervised trials on *pea* that conformed to GAP were reported from the USA (GAP, 0.62 kg ai/kg of seed, seed treatment), resulting in a concentration of 0.01 mg/kg in all four trials. The results for seed treatment of common beans (see above) may be used to support the results for pea seed treatment. The ranked order of concentrations of residues of chlorpyrifos in common beans and peas with pods after seed treatment at 0.62 kg ai/kg seed was <0.01 (3 trials) and 0.01 (4 trials) mg/kg. The Meeting estimated a HR value of 0.01 mg/kg, a maximum residue level of 0.01 mg/kg, and an STMR value of 0.01 mg/kg for peas with pods. Trials reported from the UK did not conform to GAP and were not considered.

Reports were received on supervised trials conducted on *soya* in Thailand (two trials at the GAP rate of 0.72 kg ai/ha, 7-day PHI) giving concentrations of 0.23 and 1.6 mg/kg and

the USA (five trials at the GAP rate of 1.1 kg ai/ha, 28-day PHI) showing values of <0.01 (2 trials), 0.01 (2 trials), and 0.05 mg/kg). The Thai and USA data represent different populations of residues and cannot be grouped. The Meeting concluded that five data values were insufficient to permit estimation of a maximum residue level or an STMR value.

Supervised trials were conducted on *carrot* in The Netherlands (two trials at the GAP rate of the UK of 0.96 kg ai/ha, 14-day PHI) giving values of 0.01 and 0.03 mg/kg, South Africa (one trial at the GAP rate of 0.48 kg ai/ha, 21-day PHI) showing a value of 0.05 mg/kg, and the UK (three trials) resulting in concentrations of <0.01, 0.02, and 0.03 mg/kg. The ranked order of concentrations of residues found in the six trials was <0.01, 0.01, ***0.02***, ***0.03*** (2 trials), and 0.05 mg/kg. The Meeting estimated an STMR value of 0.025 mg/kg, a HR value of 0.05 mg/kg, and a maximum residue level of 0.1 mg/kg. The latter replaces the existing MRL of 0.5 mg/kg.

Reports were available for supervised trials of ground application to *potato* at the time of planting in Brazil (four trials at the GAP rate of Argentina of 3 kg ai/ha) with residue concentrations of 0.02, 0.13, 0.29, and 0.51 mg/kg. Data were also provided from trials of foliar and planting plus foliar treatment from Australia (two trials at the GAP rate of 3 kg ai/ha before planting, 0.5 kg ai/ha at hilling up) showing a value of <0.01 mg/kg in both trials, Brazil (one trial at the GAP rate of 0.72 kg ai/ha, 21-day PHI) with a value of 0.01 mg/kg, Canada (one trial at the GAP rate of 0.48 kg ai/ha for emulsifiable concentrate, 7-day PHI) with a value of 0.01 mg/kg, and Poland (one trial at the GAP rate of 0.42 kg ai/ha, 30-day PHI) showing <0.02 mg/kg. The ranked order of concentrations in the five trials of foliar residues was: <0.01 (2 trials), 0.01 (2 trials), and <0.02 mg/kg. The ranked order in the four trials of ground application at the time of planting was 0.02, 0.13, 0.29, and 0.51 mg/kg. The Meeting concluded that neither data set contained an adequate number of values for estimating a maximum residue level or an STMR value. The Meeting also recommended withdrawal of the existing MRL of 0.05* mg/kg. Trials reported from Colombia, South Africa, and the UK were not conducted according to GAP and not evaluated.

Supervised trials on *sugar beet* were conducted in Canada (one trial at the GAP rate of 1.2 kg ai/ha for foliar application, 90-day PHI) showing a residue concentration of <0.01 mg/kg, France (one trial at the GAP rate of 1.5 kg ai/ha before planting) with a value of <0.01 mg/kg, and the USA (eight trials at the GAP rate of 1.1 kg ai/ha for foliar application, 30-day PHI) with values of 0.01 (4 trials), 0.02 (3 trials), and 0.03 mg/kg). The ranked order of concentrations of residues in the nine trials in roots after foliar treatment was: <0.01, ***0.01*** (4 trials), ***0.02*** (3 trials), and 0.03 mg/kg. The Meeting estimated an STMR value of 0.015 mg/kg, a HR value of 0.03 mg/kg, and a maximum residue level of 0.05 mg/kg. The latter replaces the existing MRL of 0.05* mg/kg. Trials in Germany and the UK did not comply with GAP, and although trials were reported from Japan, no GAP was reported.

Supervised field trials on *maize* were reported for application at the time of planting in Brazil (two trials at the GAP rate of Argentina of 1.9 kg ai/ha, incorporated into soil) both showing <0.01 mg/kg). Trials from the USA were not according to GAP. Trials were also reported for foliar application or preplanting plus foliar application in Brazil (one trial at the GAP rate of 0.48 kg ai/ha, 2-day PHI) with a value of <0.01 mg/kg and the USA (seven trials at the GAP rate of 3.4 kg ai/ha before planting, 1.7 kg ai/ha for foliar treatment, 35-day PHI for grain and fodder, 14-day PHI for silage). The ranked order of the concentrations of residues in grain after foliar application was: <0.01, 0.01 (3 trials), 0.02, 0.03 (2 trials), and 0.04 mg/kg. The Meeting estimated an STMR value of 0.015 mg/kg and a maximum residue level of 0.05 mg/kg.

Supervised field trials were conducted on *sweet corn* in Canada (one trial at the GAP rate of 1.15 kg ai/ha, 70-day PHI) and the USA (six trials at the GAP for grain and 10 at the GAP for forage of 3.4 kg ai for emulsifiable concentrate before planting and 1.7 kg/ai for

foliar emulsifiable concentrate, 2.3 kg ai for granular formulation before planting and 1.1 kg/ai for foliar treatment, 35-day PHI for grain and fodder, 14-day PHI for silage). The concentration of residues in grain was <0.01 mg/kg in all seven trials. Information was also supplied on seed treatment in the USA (seven trials at the GAP rate of 62 g ai/100 kg of seed, wettable powder). The concentration was <0.01 mg/kg in all five trials. In two trials, results were not reported for kernel with cob. On the basis of the values after foliar application, the Meeting estimated an STMR value of 0.01 mg/kg, a HR value of 0.01 mg/kg, and a maximum residue level of 0.01* mg/kg.

Trials of use of chlorpyrifos in *rice* were reported from Australia, Colombia, India, the Philippines, Thailand, and Vietnam, but none was at the relevant GAP. As no data were available on treatment of rice under GAP conditions, the Meeting decided that the database was inadequate for estimating either an STMR value or a maximum residue level. The Meeting further recommended withdrawal of the existing MRL of 0.1 mg/kg.

Supervised field trials on *sorghum* were reported from Brazil (one trial at the GAP rate of 0.36 kg ai/ha, 21-day PHI) showing a residue concentration of 0.07 mg/kg, and the USA (six trials at the GAP rate of 1.1 kg ai/ha, emulsifiable concentrate, 60-day PHI; 2 kg ai/ha of granular formulation at the time of planting). The ranked order of concentrations was: <0.01 (2 trials), 0.02, ***0.04***, 0.07, 0.20, and 0.27 mg/kg. The Meeting estimated an STMR value of 0.04 mg/kg and a maximum residue level of 0.5 mg/kg. Two trials from Australia did not comply with GAP and were discarded.

Supervised field trials on *wheat* were reported from Brazil (three trials at the GAP rate of 0.72 kg ai/ha, 21-day PHI) with values of 0.04, 0.06, and 0.30 mg/kg and the USA (17 trials at the GAP rate of 0.56 kg ai/ha, 28-day PHI for grain, 14-day PHI for forage or hay). The ranked order of the concentrations of residues after use on grain were <0.01 (3 trials), ***0.01*** (7 trials), ***0.02*** (3 trials), 0.03, 0.04, 0.05, 0.06, 0.19, 0.23, and 0.30 mg/kg. The Meeting estimated an STMR value of 0.015 mg/kg and an MRL of 0.5 mg/kg. Trials from Canada and the UK were not in accordance with GAP in those countries, and although trials were reported from Germany no GAP was provided.

Supervised trials on *almond* were conducted in the USA (three trials at the GAP rate of 2.2 kg ai/ha for foliar application, 4.5 kg ai/ha for ground application, 14-day PHI; four trials at the GAP rate of 2.2 kg ai/ha for dormant crop). The ranked order of the concentrations in almond nutmeat was: <0.01, 0.01 (2 trials), ***<0.05*** (3 trials), and 0.05 mg/kg. The highest concentration resulted from the use on dormant crop. The two uses are distinguished by the PHI, 14 days versus about 180 days for use on dormant crop (with no nuts). As metabolic studies showed that chlorpyrifos is not readily translocated, any residues on almond nutmeat probably result from contamination during removal of the shells. The Meeting estimated an STMR value of 0.05 mg/kg, a HR value of 0.05 mg/kg, and a maximum residue level of 0.05 mg/kg.

Supervised trials on *pecan* were conducted in the USA (eight trials at the GAP rate of 2.2 kg ai/ha for foliar application, 28-day PHI). The ranked order of the concentrations of residues on the nutmeat was: <0.01 (2 trials) and <0.05 (6 trials). The latter value resulted from use of a method to determine combined residues of chlorpyrifos and 3,5,6-trichloropyridinol. The Meeting estimated an STMR value of 0.05 mg/kg, a HR value of 0.05 mg/kg, and a maximum residue level of 0.05* mg/kg.

Six supervised trials were conducted on *walnut* in the USA (at the GAP rate of 2.24 kg ai/ha, 14-day PHI). The concentration of residues on the nutmeat was <0.05 mg/kg in all six trials. The Meeting estimated an STMR value of 0.05 mg/kg, a HR value of 0.05 mg/kg, and a maximum residue level of 0.05* mg/kg.

Supervised field trials were conducted on *cotton seed* in Brazil (two trials at the GAP rate of 0.96 kg ai/ha, 21-day PHI: 0.02 and 0.07 mg/kg) and the USA (three trials at the GAP rate of 1.1 kg ai/ha, 14-day PHI). The ranked order of concentrations of residues in cotton seed was 0.02, 0.07, 0.16, 0.17, and 2.0 mg/kg. The Meeting concluded that five values were insufficient for estimating an STMR value or a maximum residue level. The Meeting further recommended the withdrawal of the existing MRL of 0.05* mg/kg

The results of a supervised field trial on *peanut* conducted in the USA at the GAP rate of 2.2 kg ai/ha, 21-day PHI were available. The Meeting concluded that the data were insufficient to estimate an STMR value or a maximum residue level.

A supervised field trial was conducted on *sunflower* in the USA at the GAP rate of 2.2 kg ai/ha before planting, 1.7 kg ai/ha foliar, 42-day PHI. A trial in Canada did not comply with GAP. The Meeting concluded that the data were insufficient to estimate an STMR value or a maximum residue level.

The results of supervised field trials on *coffee* were reported from Brazil (five trials at the GAP rate of 0.72 kg ai/ha, 21-day PHI) and the United Republic of Tanzania (one trial at the GAP rate of 0.96 kg ai/ha, 7-day PHI) with a residue concentration of 0.04 mg/kg. Two trials conducted in Colombia did not comply with GAP. The ranked order of concentrations of residues was 0.01 (3 trials), ***0.03*** (2 trials), and 0.04 mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR value of 0.03 mg/kg, and a HR value of 0.04 mg/kg.

Supervised trials were conducted on *alfalfa* in the USA, where the GAP specifies tiered application rates and PHIs: 0.28 kg ai/ha, 7-day PHI; 0.56 kg ai/ha, 14-day PHI; and >0.56-1.12 kg ai/ha, 21-day PHI. Additionally, a specific GAP applies to California and Arizona: 0.56 kg ai/ha, 4-day PHI. In all cases, only one foliar application may be made per cutting cycle, and the maximum number of applications is four per season. Of the trials reported, 29 conformed to GAP. The ranked order of concentrations of residues in green alfalfa forage was: <0.01, 0.01, 0.06 (2 trials), 0.08, 0.12, 0.17, 0.20, 0.21, 0.22, 0.25, 0.27, 0.30, 0.38, ***0.42***, 0.43 (2 trials), 0.45, 0.57, 0.62, 0.65, 0.89, 0.90, 1.3, 1.4, 1.5, 2.2, 2.7, and 5.6 mg/kg (fresh weight). As the moisture contents were not determined, the Meeting used the value given in the *FAO Manual* (FAO, 1997) of 35% dry matter. The Meeting estimated an STMR value of 1.2 mg/kg (0.42/0.35) and a maximum residue level of 20 mg/kg (5.6/0.35 = 16).

The ranked order of the concentrations of residues in the 28 trials on alfalfa hay was: 0.02, 0.04 (2 trials), 0,28, 0.35, 0.36, 0.43 (2 trials), 0.45, 0.46, 0.59, 0.63, 0.64, ***0.66***, ***0.78***, 0.92, 0.93, 1.0, 1.1, 1.2 (2 trials), 1.3, 1.7, 1.8 (2 trials), 2.0, 2.3, and 2.6 mg/kg (fresh weight). One value of 12 mg/kg for hay was discarded. In numerous comparative trials of the emulsifiable concentrate and water-dispersible granule formulations, the concentrations of residue were comparable within a factor of 2. However, in the case in which the emulsifiable concentrate yielded 12 mg/kg, the water-dispersible granule formulation yielded 1.8 mg/kg. Using the value for moisture in the *FAO Manual* of 89% dry matter, the Meeting estimated an STMR value of 0.81 mg/kg (0.72/0.89) and a maximum residue level of 5 mg/kg (2.6/0.89 = 2.9).

Three supervised trials on *almond hull* were conducted in the USA at the GAP rate of 2.2 kg ai/ha for foliar application, 4.5 kg ai/ha for ground application, 14-day PHI. The ranked order of concentrations in almond hulls was 1.9, 2.3, and 3.2 mg/kg. The Meeting estimated an STMR value of 2.3 mg/kg and a HR value of 3.2 mg/kg.

Residues in *green pea vine* after seed treatment were reported from the USA (four trials at the GAP rate of 0.62 kg ai/kg of seed). The ranked order of concentrations of residues

on pea vines was 0.01, 0.02, 0.05, and 0.17 mg/kg**.** These data are comparable to those for common bean vines: 13 trials, six at the GAP rate of 0.62 g ai/kg, water-dispersible granule; ranked order of concentrations of residues: <0.01 (2 trials), 0.01, 0.03, 0.05, and 0.06 mg/kg. The ranked order in the combined database was <0.01 (2 trials), 0.01 (2 trials), ***0.02***, ***0.03***, 0.05 (2 trials), 0.06, and 0.17 mg/kg. As no data were provided on the moisture content of the vines, the value in the *FAO Manual*, 25% dry matter, was used. The Meeting estimated an STMR value of 0.10 mg/kg (0.025/0.25) and a maximum residue limit of 1 mg/kg (0.17/0.25 = 0.68), both for dry weight.

Reports were available from supervised trials on *soya forage and hay* in Thailand (two trials at the GAP rate of 0.72 kg ai/ha, 7-day PHI) and the USA (six trials at the GAP rate of 1.1 kg ai/ha, 28-day PHI). One value was reported from the USA for green forage, 0.38 mg/kg. Additional data were supplied for straw, which is not a commodity listed by Codex. The Meeting could not estimate STMR values or maximum residue levels for forage and hay.

Supervised trials of residues in *sugar-beet tops and leaves* after foliar application or preplanting plus foliar application to sugar beets were conducted in Canada (one trial at the GAP rate of 1.2 kg ai/ha, 90-day PHI) with a residue concentration of <0.01 mg/kg and the USA (eight trials at the GAP rate of 2.3 kg/ai of granular formulation at the time of planting, 1.1 kg ai/ha for foliar application, 30-day PHI). Although trials were reported from Japan, no GAP was reported, and of trials carried out in the UK, none was according to GAP. The results of trials of application of chlorpyrifos to soil before or at the time of planting were reported from France (one trial at the GAP rate of 1.5 kg ai/ha before planting, with a concentration of <0.01 mg/kg. None of the trials from Germany was according to GAP. The ranked order of concentrations of residues in samples of tops after foliar application was: <0.01, 0.15, 0.42, 0.44, ***0.68***, 1.3, 1.4, 3.1, and 6.6 mg/kg. As no information was provided on the moisture content, the value in the *FAO Manual*, 23% of dry matter, was used. The Meeting estimated an STMR value of 3.0 mg/kg (0.68/0.23) and a maximum residue level of 40 mg/kg (6.6/.23 = 28.6), both on a dry weight basis.

Supervised field trials were reported of residues on *maize (field corn) fodder and forage* after application at the time of planting of maize in Brazil (two trials at the GAP rate of Argentina of 1.9 kg ai/ha, incorporated into soil), but with no data on fodder or forage. Of six trials in the USA, none was at the GAP). Additional trials were reported of early-to-late seasonal foliar application of chlorpyrifos to maize in Brazil (one trial at the GAP rate of 0.48 kg ai/ha, 21-day PHI; no data on forage or fodder) and the USA (seven trials at the GAP rate of 3.4 kg ai/ha before planting, 1.7 kg ai/ha for foliar application, 35-day PHI for grain and fodder, 14-day PHI for silage).

The ranked concentrations of residues in fodder were 1.6, 1.7, 2.0, *2.3*, 3.1, 5.9, and 7.2 mg/kg. As no data were provided on moisture content, the value in the *FAO Manual* (Appendix IX) for stover of 83% of dry matter was used. The ranked order on a dry weight basis was 1.9, 2.0, 2.4, ***2.8***, 3.7, 7.1, and 8.7 mg/kg. The Meeting estimated a maximum residue level of 10 mg/kg and an STMR value of 2.8 mg/kg for maize fodder, both on a dry weight basis**.**

The ranked concentrations of residues in maize forage were 2.1, 2.8, *3.0*, *3.6*, 5.5, and 7.2 mg/kg. As data on moisture content were not available, the value in the *FAO Manual* (Appendix IX) of 40% of dry matter was used. The ranked order of concentrations on a dry-weight basis was 5.2, 7.0, ***7.5***, ***9.0***, 14, and 18 mg/kg. The Meeting estimated an STMR value of 8.2 mg/kg and a maximum residue level of 20 mg/kg, both for dry-weight.

Supervised field trials on residues in *sweet corn fodder (stover) and forage* after treatment of sweet corn were conducted in Canada (one trial at the GAP rate of 1.15 kg ai/ha,

70-day PHI, no data on forage or fodder) and the USA (six trials at the GAP for grain, 10 at the GAP for forage, and seven at the GAP for fodder of 3.4 kg ai of emulsifiable concentrate before planting, 1.7 kg/ai for foliar application of emulsifiable concentrate, 2.3 kg ai of granular formulation before planting, and 1.1 kg/ai for foliar application, respectively; 35-day PHI for grain and fodder, 14-day PHI for silage). The ranked order of concentrations in forage was 0.11 (2 trials), 0.24, 0.38, 0.64, 0.81, 1.1, 1.2 (2 trials), and 3.4 mg/kg. As data on moisture content were not available, the value in the *FAO Manual* (Appendix IX), 48% of dry matter, was used to arrive at the following ranked order (dry-matter basis): 0.23 (2 trials), 0.50, 0.79, ***1.3***, 1.7, 2.3, 2.5 (2 trials), and 7.1 mg/kg. The Meeting agreed that sweet corn forage represented a different population from maize forage and considered that the STMR value and maximum residue limit for maize forage would suffice for sweet corn forage.

Seven values were available for concentrations of residues in sweet corn fodder (stover), ranked as follows: 0.06, 0.14, 0.16, 0.23, 0.77, 1.3, and 1.6 mg/kg. As data on moisture content were not available, the value in the *FAO Manual* (Appendix IX) for the moisture content of stover, 83% of dry matter, was used to arrive at the following ranked order of values (dry-matter basis): 0.07, 0.17, 0.19, ***0.28***, 0.93, 1.6, and 1.9 mg/kg. The Meeting agreed that sweet corn stover represents a different population from maize fodder and considered that the MRL and STMR value for maize fodder would suffice for sweet corn fodder (stover).

Supervised field trials of the residues in *sorghum forage and fodder* after treatment of sorghum were reported from Brazil (one trial at the GAP rate of 0.36 kg ai/ha, 21-day PHI; no data on fodder) and the USA (six trials at the GAP rate of 1.1 kg ai/ha, emulsifiable concentrate, 60-day PHI; 2 kg ai/ha of granular formulation at the time of planting). Two trials from Australia did not comply with the GAP. The ranked order of concentrations was 0.01, 0.08, ***0.17***, ***0.34***, 0.39, and 1.3 mg/kg. Using the value in the *FAO Manual* (Appendix IX) for water content, 88% of dry matter, the Meeting estimated an STMR value of 0.29 mg/kg (0.255/0.88), and a maximum residue limit of 2 mg/kg (1.3/0.88 = 1.5).

Only four values were available for residues in green forage, ranging from 0.01 to 0.14 mg/kg, and the Meeting concluded that this was an insufficient database for estimating an STMR or HR value.

The residues in *wheat fodder and straw* after treatment of wheat were reported from Brazil (three trials at the GAP rate of 0.72 kg ai/ha, 21-day PHI; no data on forage) and the USA (19 trials at the GAP rate of 0.56 kg ai/ha, 28-day PHI for grain, 14-day PHI for forage and hay). Trials from Canada and the UK were not according to GAP, and for one trial from Germany no GAP was reported. The ranked order of concentrations was 0.01, 0.03, 0.09, 0.11, 0.2, 0.39, 0.47, ***0.48*** (2 trials), ***0.60***, 0.63, 0.64, 0.96, 1.2 (2 trials), 2.1, 2.2, and 4.1 mg/kg. Using the value of 88% of dry matter from the *FAO Manual* (Appendix IX), the Meeting estimated an STMR value of 0.54 mg/kg (0.48/0.88) and a maximum residue limit of 5 mg/kg (4.1/0.88 = 4.6), both on a dry-weight basis.

No studies that were conducted in accordance with GAP were provided for green forage.

***Fate of residues during processing***

The Meeting received data on the fate of incurred residues of chlorpyrifos during the processing of apples, citrus, grapes, tomatoes, soya beans, maize (corn), rice, sorghum, wheat, cotton, peanuts, sunflower, and coffee. MRLs were not estimated for cotton, peanuts, soya beans, sunflower, or coffee, and these studies are not considered further. Moreover, a study in which fortified sugar beets as opposed to incurred residues were used was considered inappropriate.

*Apples* with an average residue concentration of 3.2 or 0.53 mg/kg were processed into juice, wet pomace, and dry pomace, with average concentration factors of 0.15, 2.0, and 6.6, respectively. The factors for juice and dry pomace applied by the Meeting to the STMR value for apple (0.18) yield STMR-P values of 0.027 mg/kg for juice and 1.2 mg/kg for dry pomace. The HR value for apple, 0.94 mg/kg, yields HR-P values of 6.2 mg/kg for dry apple pomace and 1.9 mg/kg for wet apple pomace.

*Oranges* bearing residues of chlorpyrifos were processed into orange juice in eight studies in which home processing was simulated. The processing factors ranged from 0.02 to 0.06. Single studies of commercial processing were conducted with oranges, *grapefruit*, *lemons*, and *tangelos*, in which the processing factors were 0.02-0.03. The average processing factor for the 12 studies was 0.03. By applying the factor to the median concentration for whole citrus (0.24) the Meeting estimated the STMR-P value to be 0.007 mg/kg for juice.

Oranges, grapefruit, lemons, and tangelos with incurred residues of chlorpyrifos were processed commercially into juice, dried pulp, and oil. The processing factors for pulp were 3.8 for grapefruit, 1.5 for lemons, 2.6 for oranges, and 4.0 for tangelos, with an average of 3.0. The respective processing factors for oil were 22, 3.2, 6.4, and 13, with an average of 11. With the average processing factor for citrus oil, the median residue for whole citrus (0.24 mg/kg) and the HR value for whole citrus (1.2 mg/kg), the STMR-P value for citrus oil is 2.6 mg/kg and the HR value is 13 mg/kg. With the average processing factor for citrus pulp, the HR value for whole citrus (1.2 mg/kg), and the median residue for whole citrus (0.24 mg/kg), the HR value for dried citrus pulp was estimated by the Meeting to be 3.6 mg/kg and the STMR-P value to be 0.72 mg/kg.

When *grapes* with concentrations of incurred residues of chlorpyrifos of 1.3 or 0.38 mg/kg were sun-dried, the processing factors for raisins were 0.22 and 0.20 (average, 0.21). The Meeting applied this average factor to the HR and STMR values for grapes (0.32 and 0.08 mg/kg) and estimated a HR value of 0.07 mg/kg and an STMR-P value of 0.017 mg/kg for raisins. The Meeting also estimated a maximum residue level of 0.1 mg/kg for raisins.

Grapes containing chlorpyrifos at 0.48 mg/kg were processed into juice, with a processing factor of 0.06. Using the STMR value for grapes (0.08 mg/kg), the Meeting estimated an STMR-P value of 0.005 mg/kg for juice.

In studies in France, Israel, and Italy in which grapes were processed into wine, the processing factor ranged from 0.006 to 0.3, with an average of 0.08. The wide range may be due to the absence of quantifiable residue in the wine (<0.01 mg/kg). The Meeting applied the average factor to the STMR value for grapes (0.08 mg/kg) to estimate an STMR-P value for wine of 0.007 mg/kg.

*Tomatoes* were processed into juice and tomato paste in a study in Israel and into juice and purée in a study in the USA. The processing factors for juice ranged from 0.03 to 0.4 ($\nu$ = 9; average, 0.18 or 0.2). The processing factor for purée was 0.1, and those for paste ranged from 0.08 to 0.3 ($\nu$ = 8; average, 0.16 or 0.2). Using the average processing factors and the STMR value for tomatoes (0.13 mg/kg), the Meeting estimated STMR-P values of 0.026 mg/kg for tomato paste and juice.

*Corn (maize)* with an incurred residue of 0.04 mg/kg was processed by both wet and dry milling in the USA. The processing factors for dry milling were 1.2 for meal, 1.8 for flour, and 1.5 for crude and refined oil. Those for wet milling were 3 for crude oil and 3.2 for refined oil. The Meeting decided to use the processing factor for wet milling for oil. Using the STMR value for corn grain (0.01 mg/kg), the Meeting estimated the following STMR-P values: meal, 0.01 mg/kg; crude oil, 0.03 mg/kg; refined oil, 0.03 mg/kg; and milled by-

products, 0.02 mg/kg based on flour. The Meeting also estimated a maximum residue level of 0.2 mg/kg for refined oil and a HR-P value of 0.09 mg/kg for milled by-products, on the basis of the factor of 1.8 for flour.

*Sorghum* grain bearing chlorpyrifos residue at 0.04 mg/kg was milled into flour in the USA, with a processing factor of 0.2. Using the sorghum grain STMR value of 0.04 mg/kg, the Meeting estimated an STMR-P value of 0.008 mg/kg for sorghum flour.

*Wheat* grain with an incurred concentration of chlorpyrifos residue of 0.51 mg/kg was milled in the USA into bran, flour, shorts, and milled by-products, with processing factors of 2.5, 0.2, 2.4, and 2.5. Using the STMR value for wheat grain (0.01 mg/kg), the Meeting estimated the following STMR-P values: bran, 0.03 mg/kg; flour, 0.002 mg/kg; shorts, 0.03 mg/kg; and milled by-products, 0.03 mg/kg. The Meeting also estimated a maximum residue level of 0.1 mg/kg for wheat flour. Using the HR value of wheat, 0.30 mg/kg, the Meeting estimated a HR-P value for wheat milled by-products of 0.75 mg/kg.

*Coffee* beans (shelled and dried) with incurred residues of chlorpyrifos were roasted in trials in Brazil and Colombia. The processing factors were 0.5 and 0.1 in Brazil and 0.5 and 0.25 in Columbia (average factor, 0.34). Application of this factor to the STMR value for coffee (0.03 mg/kg) yields an STMR-P value of 0.01 mg/kg for roasted coffee beans.

***Residues in animal and poultry commodities***

The Meeting estimated the dietary burden of chlorpyrifos in farm animals and poultry on the basis of the diets listed in Appendix IX of the *FAO Manual*. Calculation from the MRLs yields maximum theoretical dietary intakes, or the concentrations of residues in feed suitable for estimating MRLs for animal commodities. Calculation from STMR values for feed allows estimation of STMR values for animal commodities. The diets are designed to maximize dietary intake of chlorpyrifos, and nutritional requirements are not taken into consideration.

***Maximum theoretical dietary burden***

| Commodity | Maximum residue level | Group | % dry matter | % o diet | | | Concentration of residue, mg/kg | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Beef cattle | Dairy cows | Poultry | Pigs | Beef cattle | Dairy cows | Poultry | Pigs |
| Alfalfa forage (green) | 20 | AL | 100 | 70 | 60 | | | 14 | 12 | | |
| Alfalfa hay | 5 | AL | | | | | | | | | |
| Almond hulls | 3.2 | - | 90 | | | | | | | | |
| Apple pomace, wet | 6.2 | AB | 40 | | | | | | | | |
| Citrus pulp, dried | 3.6 | AB | 91 | | | | | | | | |
| Maize | 0.05 | GC | 88 | | | | | | | | |
| Maize forage | 20 | AF | 100 | 10 | 30 | | | 2 | 6 | | |
| Maize fodder | 10 | AS | 100 | | | | | | | | |
| Maize, milled | 0.09 | - | 85 | | | | | | | | |
| by-products | | | | | | | | | | | |
| Pea vines (green) | 1 | AL | 100 | | | | | | | | |
| Sorghum | 0.5 | GC | 86 | | | 50 | 50 | | | 0.29 | 0.29 |
| Sorghum stover | 2 | AS | 100 | | | | | | | | |
| (fodder) | | | | | | | | | | | |
| Sugar beet, tops | 40 | AV | 100 | 20 | 10 | | | 8 | 4 | | |
| Wheat | 0.5 | GC | 89 | | | | | | | | |
| Wheat, milled | 0.75 | - | 88 | | | 50 | 50 | | | 0.43 | 0.43 |

| Commodity | Maximum residue level | Group | % dry matter | % o diet | | | | Concentration of residue, mg/kg | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Beef cattle | Dairy cows | Poultry | Pigs | Beef cattle | Dairy cows | Poultry | Pigs |
| by-products | | | | | | | | | | | |
| Wheat, straw | 5 | AS | 100 | | | | | | | | |
| | | | | | | | | | | | |
| Total | | | | 100 | 100 | 100 | 100 | 24 | 22 | 0.77 | 0.77 |
| | | | | | | | | | | | |

***Average dietary burden***

| Commodity | STMR/ STMR-P | Group | % dry matter | % of diet | | | | Concentration of residue, mg/kg | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Beef cattle | Dairy cows | Poultry | Pigs | Beef cattle | Dairy cows | Poultry | Pigs |
| Alfalfa forage (green) | 1.2 | AL | 100 | | | | | | | | |
| Alfalfa hay | 0.81 | AL | | | | | | | | | |
| Almond hulls | 2.3 | AL | 90 | 10 | 10 | | | 0.26 | 0.26 | | |
| Apple pomace, wet | 0.34 | AB | 40 | 25 | 25 | | | 0.21 | 0.21 | | |
| Citrus pulp, dried | 0.72 | AB | 91 | | | | | | | | |
| Maize | 0.015 | GC | 88 | | | | | | | | |
| Maize forage | 8.2 | AF | 100 | 40 | 50 | | | 3.3 | 4.1 | | |
| Maize fodder | 2.8 | AS | 100 | 25 | 15 | | | 0.7 | 0.42 | | |
| Maize, milled | 0.02 | - | 85 | | | | | | | | |
| by-products | | | | | | | | | | | |
| Pea vines (green) | 0.10 | AL | 100 | | | | | | | | |
| Sorghum | 0.04 | GC | 86 | | | 80 | 90 | | | 0.037 | 0.042 |
| Sorghum stover | 0.29 | AS | 100 | | | | | | | | |
| (fodder) | | | | | | | | | | | |
| Sugar beet, tops | 3.0 | AV | 100 | | | | | | | | |
| Wheat | 0.015 | GC | 89 | | | | | | | | |
| Wheat, milled | 0.03 | - | 88 | | | 20 | 10 | | | 0.007 | 0.003 |
| by-products | | | | | | | | | | | |
| Wheat, straw | 0.54 | AS | 100 | | | | | | | | |
| Total | | | | 100 | 100 | 100 | 100 | 4.5 | 5.0 | 0.044 | 0.045 |

Acceptable feeding studies were provided for chickens, cows, and swine. Hens were fed chlorpyrifos in their daily rations at a rate of 0, 0.3, 3, or 10 ppm for 30 days. No residues (<0.01 mg/kg) of chlorpyrifos were found in muscle, liver, or kidney at any concentration. Chlorpyrifos was found in peritoneal fat at concentrations of <0.01-0.01 mg/kg in hens at 3 ppm and at 0.02-0.05 mg/kg at 10 ppm. Over a 45-day feeding period of chlorpyrifos at 10 ppm in the feed, the concentration in eggs was <0.01-0.01 mg/kg, reaching a plateau within 10 days. The calculated dietary burdens are 0.77 ppm on the basis of the MRL and 0.044 ppm on the basis of the STMR value. In hens at 2 ppm, residues were found at a concentration near the LOQ in fat only. The Meeting estimated the following maximum residue levels: poultry meat (fat), 0.01 mg/kg; eggs, 0.01* mg/kg; and offal, 0.01* mg/kg. The STMR values were estimated to be 0.001 mg/kg for meat (fat), 0.001 mg/kg for eggs, and 0.00 mg/kg for offal. The HR values were estimated to be 0.01 mg/kg for each of eggs, meat (fat), and offal.

Heifers were given capsules containing chlorpyrifos at a concentration of 0, 3, 10, 30, or 100 ppm for 30 days. In animals at 10 ppm, residues were found in muscle (0.02 mg/kg) and liver (0.02 mg/kg). At 100 ppm, the concentration in muscle increased to 0.29 mg/kg, but that in liver remained constant. Kidney was found to contain chlorpyrifos (0.02 mg/kg) only in animals at the highest dose (100 ppm). Fat showed concentrations of 0.01-0.03 mg/kg in animals at 3 ppm, which increased to 2.0-4.2 mg/kg at 100 ppm. At 30 ppm, which is comparable to the calculated dietary burden of 24 ppm based on MRLs, the concentrations were 0.02 mg/kg (<0.01-0.02 mg/kg) in muscle, 0.99 mg/kg (0.18-0.99 mg/kg) in fat, and

0.01 mg/kg in each of liver and kidney. In animals at 10 ppm, which is comparable to the 4.5 ppm dietary burden based on STMR values, the concentrations were <0.01-0.02 mg/kg in meat and liver, <0.01 mg/kg in liver, and 0.15 mg/kg (0.07-0.15 mg/kg) in fat. The Meeting estimated the maximum residue levels for cattle commodities to be: meat (fat), 1.0 mg/kg, liver, 0.01 mg/kg, and kidney, 0.01 mg/kg, and those for sheep commodities to be: meat (fat), 1.0 mg/kg; edible offal, 0.01 mg/kg. It estimated the STMR values for cattle commodities to be: meat, 0.02 mg/kg; liver, 0.01 mg/kg; and kidney, 0.01 mg/kg, and those for sheep commodities to be: meat, 0.02 mg/kg; edible offal, 0.01 mg/kg. The Meeting estimated the HR values for cattle commodities to be: meat, 0.02 mg/kg; kidney, 0.01 mg/kg; and liver, 0.01 mg/kg, and those for sheep commodities to be: meat, 1 mg/kg; edible offal, 0.01 mg/kg.

Cows were fed rations containing 0.3, 1, 3, 10, or 30 ppm of chlorpyrifos for 14 consecutive days. Residues were found in whole milk at a maximum of 0.02 mg/kg only in cows fed 30 ppm. Residues were found in cream at maximum concentrations of 0.01, 0.04, and 0.15 mg/kg at 3, 10, and 30 ppm, respectively. The concentration of chlorpyrifos residue reached a plateau within 6 days. No detectable residues were found in cows fed 30 ppm after a 1-day withdrawal period. The dietary burden, based on MRLs, was estimated to be 22 ppm for dairy cattle. The Meeting estimated the maximum residue level in whole milk to be 0.02 mg/kg on the basis of the maximum residue level of 0.02 mg/kg at 30 ppm. The dietary burden based on STMR values was estimated to be 5.0 ppm. The Meeting estimated the STMR value for whole milk to be 0.005 mg/kg, on the basis of the concentration of <0.01 mg/kg in milk of cows at 10 ppm.

Pigs were fed chlorpyrifos in their diets at a concentration of 0, 1, 3, or 10 ppm for 30 days. The concentrations of residues found in pigs at 30 ppm were 0.03 mg/kg in muscle, 0.01 mg/kg in liver, and 0.18 mg/kg in omental, renal, and subcutaneous fat. In pigs at 3 and 1 ppm, residues were found only in fat (0.02 mg/kg), muscle, liver, and kidney, each containing <0.01 mg/kg. The calculated dietary burdens are 0.77 and 0.045 ppm on the basis of MRLs and STMR values, respectively. At these levels, the estimated concentrations of chlorpyrifos in tissues are estimated to be <0.01 mg/kg, except for 0.02 mg/kg in fat, on the basis of MRLs, and 0.002 mg/kg in fat and 0.00 mg/kg in other tissues on the basis of STMR values. The MLR for pig meat (fat) was estimated to be 0.02 mg/kg, the STMR value was estimated to be 0.001 mg/kg, and the HR value was estimated to be 0.01 mg/kg. The STMR value and MRL for offal were estimated to be 0.00 mg/kg and 0.01* mg/kg, respectively. The HR value for pig offal was estimated to be 0.01 mg/kg.

Dermal application of chlorpyrifos is no longer a veterinary use.

## RECOMMENDATIONS

The Meeting estimated the maximum residue and STMR levels shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and estimation of dietary intake: chlorpyrifos.

The residue is fat-soluble.

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| AL 1020 | Alfalfa fodder | 5 | - | 0.81 | |
| AL 1021 | Alfalfa forage (green) | 20 | - | 1.2 | |
| TN 0660 | Almonds | 0.05 | - | 0.05 | 0.05 |
| | Almond hulls | | | 2.3 | 3.2 |
| FP 0226 | Apple | W[1] | 1 | | |

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| JF 0226 | Apple juice | | | 0.027 | |
| AB 0226 | Apple pomace, dry | | - | 1.2 | 6.2 |
| | Apple pomace, wet | | | 0.34 | 1.9 |
| FI 0327 | Banana | 2 | - | 0.01 | 0.05 |
| VB 0400 | Broccoli | 2 | - | 0.02 | 1.4 |
| VB 0041 | Cabbages, Head | 1 | 0.05* | 0.15 | 0.94 |
| VR 0577 | Carrot | 0.1 | 0.5 | 0.025 | 0.05 |
| MO 1280 | Cattle, kidney | 0.01 | - | 0.01 | 0.01 |
| MO 1281 | Cattle, liver | 0.01 | - | 0.01 | 0.01 |
| MM 0812 | Cattle meat | 1 (fat) | 2 (fat)V | 0.02 | 0.02 |
| VB 0404 | Cauliflower | 0.05 | 0.05* | 0.01 | 0.02 |
| VS 0624 | Celery | W | 0.05* | | |
| VL 0467 | Chinese cabbage (type Pe-tsai) | 1 | 1 | 0.18 | 0.60 |
| FC 0001 | Citrus fruits | 2 | 2 | 0.08 | 0.4 |
| JF 0001 | Citrus juice | | | 0.007 | |
| | Citrus oil | | | 2.2 | 11 |
| AB 0001 | Citrus pulp, dry | | | 0.72 | 3.6 |
| SB 0716 | Coffee | 0.05 | | 0.010 | 0.014 |
| VP 0526 | Common bean (pods and/or immature seeds) | 0.01 | 0.2 | 0.01 | 0.01 |
| SO 0691 | Cotton seed | W | 0.05* | | |
| OC 0691 | Cotton seed oil, crude | W | 0.05* | | |
| DF 0269 | Dried grapes (= Currants, Raisins and Sultanas) | 0.1 | 2 | 0.017 | 0.07 |
| VO 0440 | Egg plant | W | 0.2 | | |
| PE 0112 | Eggs | 0.01* | 0.05* | 0.001 | 0.01 |
| FB 0269 | Grapes | 0.5 | 1 | 0.085 | 0.32 |
| JF 0269 | Grape juice | | | 0.005 | |
| | Grapes, wine | | | 0.007 | |
| VL 0480 | Kale | W | 1 | | |
| FI 0341 | Kiwifruit | W | 2 | | |
| VL 0482 | Lettuce, Head | W | 0.1 | | |
| GC 0645 | Maize | 0.05 | - | 0.015 | |
| AS 0645 | Maize fodder | 10 | - | 2.8 | |
| AF 0645 | Maize forage | 20 | - | 8.2 | |
| | Maize, milled by-products | | | 0.02 | 0.09 |
| OR 0645 | Maize oil, edible | 0.2 | - | 0.03 | |
| CF 0645 | Maize meal | | | 0.01 | |
| ML 0106 | Milks | W | 0.01* | | |
| ML 0107 | Milk of cattle, goats and sheep | 0.02 | - | 0.005 | |
| VO 0450 | Mushrooms | W | 0.05* | | |
| VA 0385 | Onion, Bulb | 0.2 | 0.05* | 0.04 | 0.08 |
| VP 0063 | Peas (pods and succulent = immature seeds) | 0.01 | - | 0.01 | 0.01 |
| AL 0528 | Pea vines (green) | 1 | - | 0.10 | |
| FS 0247 | Peach | 0.5 | - | 0.042 | 0.33 |
| FP 0230 | Pear | W[1] | 0.5 | | |
| TN 0672 | Pecan | 0.05* | - | 0.05 | 0.05 |
| VO 0051 | Peppers | W | 0.5 | | |
| VO 0445 | Peppers, Sweet | 2 | - | 0.38 | 1.4 |
| FS 0014 | Plums (including Prunes) | 0.5 | - | 0.04 | 0.20 |
| MO 0818 | Pig, Edible offal of | 0.01* | - | 0.00 | 0.01 |
| MM 0818 | Pig meat | 0.02 (fat) | - | 0.001 | 0.01 |
| FP 0009 | Pome fruits | 1 | - | 0.17 | 0.94 |
| VR 0589 | Potato | W | 0.05* | | |
| PM 0110 | Poultry meat | 0.01 (fat) | 0.1 (fat) | 0.001 | 0.01 |
| PO 0111 | Poultry, Edible offal of | 0.01* | - | 0.00 | 0.01 |
| FB 0272 | Raspberries, Red, Black | W | 0.2 | | |
| GC 0649 | Rice | W | 0.1 | | |
| MO 0822 | Sheep, Edible offal of | 0.01 | - | 0.01 | 0.01 |
| MM 0822 | Sheep meat | 1 (fat) | 0.2 (fat)V | 0.02 | 0.02 |
| GC 0651 | Sorghum | 0.5 | - | 0.04 | |
| AS 0651 | Sorghum straw and fodder, dry | 2 | - | 0.29 | |

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| | Sorghum flour | | | 0.008 | |
| FB 0275 | Strawberry | 0.3 | | 0.09 | 0.15 |
| VR 0596 | Sugar beet | 0.05 | 0.05* | 0.015 | 0.03 |
| AV 0596 | Sugar beet leaves or tops | 40 | - | 3.0 | |
| VO 0447 | Sweet corn | 0.01* | - | 0.01 | 0.01 |
| VO 0448 | Tomato | 0.5 | 0.5 | 0.13 | 0.33 |
| JF 0448 | Tomato juice | | | 0.026 | |
| | Tomato paste | | | 0.026 | |
| PM 0848 | Turkey meat | W | 0.2 fat (V) | | |
| TN 0678 | Walnuts | 0.05* | - | 0.05 | 0.05 |
| GC 0654 | Wheat | 0.5 | - | 0.015 | |
| AS 0654 | Wheat straw and fodder, dry | 5 | - | 0.54 | |
| | Wheat, milled by-products | | | 0.03 | 0.75 |
| CF 1211 | Wheat flour | 0.1 | - | 0.002 | |
| CM 0654 | Wheat bran, unprocessed | | | 0.03 | |
| | Wheat shorts | | | 0.03 | |

[1] Now included in recommendation for Pome fruit

*Further work or information*

*Desirable*

Study of the stability of analytical samples of farm animal commodities in frozen storage

## Dietary risk assessment

### *Chronic intake*

STMR or STMR-P levels were estimated by the present Meeting for 61 commodities. When data on consumption were available, these values were used in the estimates of dietary intake.

The dietary intakes in the five GEMS/Food regional diets, on the basis of the new STMR values, represented 1-6% of the ADI (Annex 3). The Meeting concluded that the intake of residues of chlorpyrifos resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

### *Short-term intake*

The IESTI for chlorpyrifos was calculated for the commodities for which MRLs, STMR values, and HR values were established and for which data on consumption (of large portions and unit weight) were available. The results are shown in Annex 4.

The acute RfD for chlorpyrifos is 0.1 mg/kg bw. The calculated short-term intakes of those commodities for which calculations were possible were less than 100% of the acute RfDs for children and for the general population. The Meeting concluded that the intake of residues of chlorpyrifos resulting from uses that have been considered by the JMPR is unlikely to present a public health concern for consumers.

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# DDT (021)

## EXPLANATION

DDT was first evaluated for residues in 1966 and has been reviewed several times since, most recently in 1996. ERLs were proposed for carrot, cereal grains, eggs, meat (from mammals other than marine mammals; temporary) and milks. At the 30th Session of the CCPR (ALINORM 99/24 para. 102), DDT was tentatively scheduled for residue evaluation in 2000 for consideration of an ERL in chicken meat, and the Committee agreed to request national monitoring data by a Circular Letter. Monitoring data on chicken meat were provided by Israel, the USA, Poland, Germany, the UK, and Thailand.

The Meeting also received information on national MRLs from Poland and The Netherlands, and on methods of residue analysis and monitoring surveys on fruits and vegetables from The Netherlands. The Meeting was informed by the governments of Germany and The Netherlands that no authorized uses of DDT exist in those countries.

At the 31st Session of the CCPR (ALINORM 99/24A paras. 115-121), there was discussion of the temporary ERL in meat of 5 mg/kg proposed by the 1996 JMPR. At a 0.5% violation rate, 3 mg/kg seemed to be an appropriate level on the basis of the 1996 evaluation, but this value did not conform to the geometric progression approach used by the JMPR for estimating maximum residue levels or ERLs. The CCPR requested the JMPR to reconsider the statistical validity of its proposal and its non-conformity to the geometric progression, on the basis of the 1996 evaluation.

## METHODS OF RESIDUE ANALYSIS

### Analytical methods

In the official methods of analysis of The Netherlands (Ministry of Health, Welfare and Sport, 1996) *p,p´*-DDT, its isomer *o,p´*-DDT and its metabolites are determined by a multi-residue method for pesticides amenable to gas chromatography.

Animal products. Fat is rendered and dissolved in light petroleum. Depending on the sample the solution is cleaned up by gel permeation chromatography or adsorption chromatography. The final extract is analysed by GLC with an ECD or by GC-MS.

Plant products. The sample is extracted with acetone/dichloromethane/light petroleum and cleaned up and analysed as above.

The limit of quantification was 0.05 mg/kg. Recoveries and repeatabilities (relative SD, n=10) at a fortification level of 0.29 mg/kg in various samples (not specified) were 96% ± 4.2% for *p,p´*-DDE and 99% ± 6.0% for *p,p´*-DDT.

## USE PATTERN

The Meeting was informed that there were no authorized uses of products based on DDT in Germany and The Netherlands.

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The presentation of the data received differed from country to country, and the layouts in the Tables are consequently different. All residues are expressed as the sum of *p,p´*-DDT, *o,p´*-DDT, *p,p´*-DDE and *p,p´*-TDE (*p,p´*-DDD), in conformity with the Codex definition.

Monitoring of poultry fat in Israel (Table 1). Residues in poultry fat were reported for the period 1990-1998 by the Plant Protection and Inspection Services of Israel. The reporting limit was 2.5 mg/kg.

Table 1. Residues of DDT in poultry fat in Israel, 1990-1998 (Anon., 1999a).

| Year | No. of samples | No. of samples containing residues below 2.5 mg/kg |
|---|---|---|
| 1990 | 27 | 27 |
| 1991 | 58 | 58 |
| 1992 | 48 | 48 |
| 1993 | 71 | 71 |
| 1994 | 86 | 86 |
| 1998 | 187 | 187 |

Monitoring of chicken and turkey meat in the USA (Tables 2-5). The Meeting received information on the random monitoring of domestic samples in the period 1992-1998. The data from 1992, 1993 and 1994 were included in the 1996 JMPR evaluation. The data for the years 1995-1998 are shown in Tables 2-5. All residues are expressed on a fat basis. The reporting limit was not given, but presumably that of the 1996 JMPR evaluation of 0.01 mg/kg still applies.

Table 2. Residues of DDT in poultry meat in the USA, 1995 (Anon., 1999b)

| Class | Residues, mg/kg | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | None | 0.01-0.1 | 0.11-0.2 | 0.21-0.3 | 0.31-0.5 | 0.51-1.0 | 1.01-2.5 | 2.51-5.0 | Over 5.0 |
| Young chickens | 482 | 2 | 0 | 0 | 0 | 0 | 1 | 0 | 0 |
| Mature chickens | 524 | 6 | 1 | 1 | 0 | 0 | 0 | 0 | 0 |
| Young turkeys | 505 | 17 | 0 | 0 | 1 | 0 | 0 | 0 | 0 |
| Mature turkeys | 244 | 15 | 0 | 0 | 1 | 0 | 1 | 0 | 0 |

Table 3. Residues of DDT in poultry meat in the USA, 1996 (Anon., 1999b)

| Class | Residues, mg/kg | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | None | 0.01-0.1 | 0.11-0.2 | 0.21-0.3 | 0.31-0.5 | 0.51-1.0 | 1.01-2.5 | 2.51-5.0 | Over 5.0 |
| Young chickens | 176 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Mature chickens | 183 | 3 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Young turkeys | 381 | 9 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Mature turkeys | 64 | 4 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

Table 4. Residues of DDT in poultry meat in the USA, 1997 (Anon., 1999b)

| Class | Residues, mg/kg | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | None | 0.01-0.1 | 0.11-0.2 | 0.21-0.3 | 0.31-0.5 | 0.51-1.0 | 1.01-2.5 | 2.51-5.0 | Over 5.0 |
| Young chickens | 273 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Mature chickens | 101 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

| Class | Residues, mg/kg | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | None | 0.01-0.1 | 0.11-0.2 | 0.21-0.3 | 0.31-0.5 | 0.51-1.0 | 1.01-2.5 | 2.51-5.0 | Over 5.0 |
| Young turkeys | 148 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Mature turkeys | 107 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

Table 5. Residues of DDT in poultry meat in the USA, 1998 (Anon., 1999b)

| Class | Residues, mg/kg | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | None | 0.01-0.1 | 0.11-0.2 | 0.21-0.3 | 0.31-0.5 | 0.51-1.0 | 1.01-2.5 | 2.51-5.0 | Over 5.0 |
| Young chickens | 268 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Mature chickens | 94 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 |
| Young turkeys | 245 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 |
| Mature turkeys | 145 | 5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |

Monitoring of chicken fat in Poland (Table 6). Residues in chicken fat were reported for the period 1996-1999 from the National Veterinary Monitoring Program of Poland. Samples were from random monitoring of slaughtered animals. The reporting limit was 0.01 mg/kg.

Table 6. Residues of DDT in chicken fat in Poland, 1996-1999 (Anon., 1999c)

| Year | No. of samples | <LOQ | No. of samples in range, mg/kg | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | <0.10 | >0.10-0.20 | >0.20-0.50 | >0.50-1.00 | >1.00-2.00 | >2.00 |
| 1996 | 408 | 54 | 334 | 15 | 2 | 3 | 0 | 0 |
| 1997 | 406 | 124 | 261 | 8 | 4 | 5 | 4 | 0 |
| 1998 | 218 | 57 | 153 | 5 | 3 | 0 | 0 | 0 |
| 1999 | 182 | 59 | 121 | 1 | 1 | 0 | 0 | 0 |

Monitoring of chicken meat in Germany (Table 7). Monitoring data on domestic (81%) and import samples of chicken meat were supplied by the Bundesinstitut für gesundheitlichen Verbraucherschutz und Veterinärmedizin. Results apply to the fat of the meat. The limit of reporting (LOQ, mg/kg) was 0.0001-0.1 (mainly 0.001-0.01).

Table 7. Residues of DDT in fat of chicken meat in Germany, 1994-1997 (Anon., 1999d)

| No. of samples | No. of samples in range, mg/kg | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | <LOQ | <0.005 | 0.005-0.01 | >0.01-0.02 | >0.02-0.05 | >0.05-0.1 | >0.1-0.2 | >0.2-0.5 | >0.5-1.0 | >1.0-2.0 | >2.0-5.0 | >5.0 |
| 215 | 147 | 9 | 24 | 16 | 7 | 4 | 3 | 3 | 1 | 1 | - | - |

Monitoring of chicken meat in the UK (Table 8). Residues in poultry commodities were reported for the period 1990-1996. The various limits of reporting are indicated in the Table.

Table 8. Residues in poultry commodities in the UK, 1990-1996 (Anon.)

| Year | No. of samples | No. of samples in range, mg/kg | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | <LOQ | ≤0.005 | >0.005-0.01 | >0.01-0.02 | >0.02-0.05 | >0.05-0.1 | >0.1-0.2 | >0.2-0.5 | >0.5-1.0 | >1.0-2.0 | >2.0-5.0 | >5.0-10 |
| Chicken meat (LOQ, mg/kg: 0.002 (1995), 0.01 (1992 & 1990)) | | | | | | | | | | | | | |
| 1995 | 37[1] | 37 | 37 | | | | | | | | | | |
| | 35[2] | 35 | 35 | | | | | | | | | | |

| Year | No. of samples | No. of samples in range, mg/kg | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | <LOQ | ≤0.005 | >0.005-0.01 | >0.01-0.02 | >0.02-0.05 | >0.05-0.1 | >0.1-0.2 | >0.2-0.5 | >0.5-1.0 | >1.0-2.0 | >2.0-5.0 | >5.0-10 |
| 1992 | 72 | 64 | | 64 | | 7 | 1 | | | | | | |
| 1990 | 118 | 115 | | 115 | 2 | 1 | | | | | | | |
| Chicken liver (LOQ, mg/kg: 0.008 (1994), 0.01 (1991)) | | | | | | | | | | | | | |
| 1994 | 31 | 30 | 30 | | 1 | | | | | | | | |
| 91 | 18 | 18 | | | | | | | | | | | |
| Poultry (**may or may not be chicken**) (LOQ 0.002 mg/kg) | | | | | | | | | | | | | |
| 1996 | 24 | 21 | 24 | | | | | | | | | | |
| Total diet study<br>(1) carcass meat (may or may not be chicken), LOQ 0.01 mg/kg<br>(2) composite offal, LOQ 0.002 mg/kg<br>(3) composite meat products, LOQ 0.01 mg/kg | | | | | | | | | | | | | |
| 1996 | 24 (1) | 16 | | 16 | | | 8[3] | | | | | | |
| 1996 | 24 (2) | 18 | 18 | | 6[4] | | | | | | | | |
| 1996 | 24 (3) | 22 | | 22 | 2 | | | | | | | | |
| Canned meat (may or may not be chicken; LOQ 0.01 mg/kg) | | | | | | | | | | | | | |
| 1993 | 36 | 22 | | 22 | | 14[5] | | | | | | | |

[1] Samples were lean breast
[2] Samples were chicken, >10% fat
1992 & 1990 data reported and residues quoted on a fat basis
[3] these cover the range 0.01-0.07. No further details available
[4] these cover the range 0.002-0.02. No further details available
[5] these cover the range 0.01-0.05. No further details available

Monitoring of poultry meat in Thailand (Table 9). Data on random monitoring of poultry meat from various slaughter plants in the period 1997-1999 were supplied by the Thai Veterinary Public Health Laboratory. The portion analysed was the abdominal fat. The limit of quantification (LOQ) was 0.01 mg/kg, the limit of reporting was 0.003 mg/kg. Recoveries were within the limits 70-110%. Samples were stored before analysis below 0°C for 14 days or less.

Table 9. Residues of DDT in chicken and duck meat in Thailand, 1997-1999 (Anon.)

| Sample | Year | No. of samples | No. of residues detected | No. of residues <LOQ (<0.01 mg/kg) | No. of samples in range, mg/kg | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | 0.01-0.04 | 0.05-0.08 | 0.09-0.12 | 0.13-0.16 | 0.17-0.20 | 0.21-0.50 | 0.51-1.00 | 1.01-2.00 | >2.00 |
| Chicken meat | 1997 | 3882 | 3834 | 132 | 3113 | 477 | 71 | 13 | 11 | 13 | - | 2 (1.80, 1.10) | 2 (2.50, 2.40) |
| | 1998 | 2910 | 2734 | 747 | 1801 | 156 | 16 | 4 | 5 | 4 | - | 1 (1.20) | - |
| | 1999 | 2537 | 2033 | 690 | 1208 | 93 | 24 | 6 | 3 | 6 | 2 | 1 (1.54) | - |
| Duck meat | 1997 | 399 | 369 | 73 | 288 | 5 | - | - | 2 | 1 (0.24) | - | - | - |
| | 1998 | 464 | 381 | 264 | 115 | 2 | - | - | - | - | - | - | - |
| | 1999 | 96 | 46 | 36 | 10 | - | - | - | - | - | - | - | - |

Residues of DDT in foods of plant origin in The Netherlands (Table 10). The 1993 JMPR reported monitoring data on DDT residues for the period 1987-1991. The 1996 JMPR reported additional data for 1991-1993 and 1994. The present Meeting received information for 1994-1996.

Table 10. Residues of DDT in foods of plant origin in The Netherlands, 1994-1996 (Anon. 2000).

| Crop | Samples | | | | | | MRL, mg/kg |
|---|---|---|---|---|---|---|---|
| | No. | Without residues (LOQ* = 0.05) | Residues <MRL | Residues >MRL | Range, >MRL | Mean**, mg/kg | |
| Citrus fruit | | | | | | | |
| Grapefruit | 327 | 45 | - | - | - | <0.05 | 0.05* |
| Other fruit and fruit products | 469 | 467 | - | 2 | 0.05-0.12 | <0.05 | 0.05* |
| Root and tuber vegetables | | | | | | | |
| Carrots | 500 | 497 | - | 3 | 0.07-1.6 | <0.05 | 0.05* |
| Fruiting vegetables | | | | | | | |
| Tomatoes | 1242 | 1242 | - | - | - | < 0.05 | 0.05* |
| Melons | 455 | 455 | - | - | - | < 0.05 | 0.05* |
| Leaf vegetables and fresh herbs | | | | | | | |
| Iceberg lettuce | 535 | 534 | - | 1 | 0.05 | < 0.05 | 0.05* |
| Parsley | 390 | 390 | - | - | - | < 0.05 | 0.05* |
| Other herbs | 224 | 224 | - | - | - | < 0.05 | 0.05* |
| Legume vegetables | | | | | | | |
| Beans (without pods) | 45 | 45 | - | - | - | <0.05 | 0.05* |
| Other stem vegetables | 375 | 375 | - | - | - | <0.05 | 0.05* |

* Lower limit of quantification
** For samples without residues (<LOQ), half the LOQ is taken for the calculation of the mean.

## Statistical analysis

The monitoring data were reported as summaries in tables in which only the number of results within certain classes were given. This has advantages since it concentrates the data into a small table and allows rapid visual interpretation. The disadvantages are that comparison of the tables might be difficult (the definition of the classes can be different) and that parameters like "the critical level where only 0.2 % of the results is above the critical level" are hard to determine.

A statistical approach was used to solve this problem, based on the assumption that each set of data can be described by a log-normal distribution. The two parameters of the distribution were estimated by maximising the probability of observing the numbers reported in the classes. Since in the available data sets the amount of information was rather limited, combinations of sets were made (for example the same standard deviation was used for all data sets except those of Thailand and, for mammalian meat, of New Zealand).

Data on mammalian meat were collected from the 1996 JMPR evaluation of DDT. Some data on chicken meat also derive from the 1996 evaluation. Additional data on chicken meat were provided to the Meeting by Israel, the USA, Poland, Germany, the UK, and Thailand (monitoring data). The origin of the data is tabulated in Annex III. The poultry data set has 68 items and the mammalian data set 103.

The data were pretreated in the following way. Data which were in internally inconsistent (for example the sum of the numbers in the classes was not equal to the reported sums) were rejected. Mixed sets of data (for example poultry and pork) were rejected. The Australian data report the residues of DDT, DDE and TDE separately instead of expressing them as total DDT. To combine the data the standard deviations of the different components within one set are assumed equal and the resulting concentrations are then summed. Classes sometimes did not cover the whole range, presumably owing to rounding. For example in some cases the first class was 0-0.1 and the second was 0.11-0.2. Then the region between 0.1 and 0.11 would “not exist” in the distribution. Such situations are therefore interpreted as class 1 being 0.00000001-0.105 and class 2 as 0.105-0.2. Note also that zero is replaced by a very small number since the log of zero is not a number.

The estimated probability of a result falling in a certain class, $p_i$, is calculated by using the cumulative function of the log-normal distribution with the boundaries as the boundaries of the integration interval. Then for each class the value $f_i \ln(f_i/np_i)$ is calculated (n = total number of results

and $f_i$ is the reported number of results in this class)[1]. The sum of these values for all classes represents the log-probability of the particular values for the two parameters of the log-normal distribution. The difference in log-probability is also used to test whether one set of parameters fits significantly better than another set of parameters. Note that in the (unlikely) event of a perfect fit, all $np_i=f_i$, yields a zero value for the log-probability. For a fit that is less than perfect the log-probability value will be lower. In general, the better the fit the higher the log-probability.

Results, poultry. When a separate value for the median and standard deviation is allowed for each set the sum of all log-probabilities is -277. Using a log-normal distribution the log-probability for this data set cannot be larger. If a single value for all relative standard deviations (RSDs) is applied for all items in the poultry data set the log-probability decreases to -1109. This difference is quite significant compared to the difference in the number of parameters (67). The data sets from Thailand show RSDs that are generally lower than these combined value therefore this set was treated as a separate class. The log-probability is then –672. The latter calculations were used for further evaluation. The detailed results and calculations are shown in Annex III.

Annex III shows the treatment of the information for both poultry and mammals using the 2 RSDs calculation for poultry and the 3 RSDs calculation for mammals. It shows the source of the data and commodity/animal/sex/age and year(s) of sampling, then the total number of samples and the numbers in selected concentration intervals (mg/kg), and then a check on the consistency of these numbers and calculated distribution parameters (median and RSD). The average and the calculated number of samples above a certain level are also given. The last columns show for each item the estimated levels that correspond to the stated percentiles.

Results, mammals. Using the same calculation strategy for mammals as for poultry the log-probability for a separate value for the median and standard deviation for each set is -685. Using one value for all standard deviations the value decreases to -1510 (102 parameters). Again the data sets from Thailand show RSDs that are generally lower than the combined values so these sets were treated as a separate class, giving a log-probability of -1293. Furthermore, since the data sets from New Zealand contain higher residues than the other data sets these sets were also treated as a separate class. The log-probability is then –1155. The latter calculations are used for further evaluation. The details are shown in Annex III.

The advantage of combining the RSDs of various sets is a gain in accuracy, the disadvantage might be that some sets are combined with values that are significantly different. In principle, more sophisticated combinations of RSDs and even of medians are possible to refine the calculations. However the available information is not detailed enough to make additional valid combinations.

## NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was informed of the following national MRLs for DDT.

For plant and animal products the residue is defined as the sum of *o,p´*-DDT, *p,p´*-DDT, *p,p´*-DDE and *p,p´*-TDE (*p,p´*-DDD), expressed as DDT.

| Country | Commodity | MRL, mg/kg, expressed as DDT | Remarks |
|---|---|---|---|
| Israel | Poultry fat (PF 111) | 5 | |
| USA | poultry | No tolerance; 5 ppm action level | |

[1] R.R, Sokal and F.J. Rohlf, “Biometry” 3rd edition (1995) Freeman and Co New York

| Country | Commodity | MRL, mg/kg, expressed as DDT | Remarks |
|---|---|---|---|
| Germany | Chicken meat | 1 (fat) | |
| UK | Chicken meat | 1 | |
| | Chicken liver | 0.1 | |
| Thailand | Poultry meat | 1 (fat) | |
| | Poultry edible offal | 1 (fat) | |
| Netherlands | Tea | 0.2 | |
| | Cocoa products | 0.5 | 1) |
| | Meat | 1 | 2) |
| | Milk | 0.04 | 3) |
| | Eggs | 0.1 | 4) |
| | Eel | 1 | |
| | Liver of fish | 2 | |
| | Other fishery products | 0.5 | |
| | Wild and poultry | 1 | 2) |
| | Other animal oils and fats | 1 | |
| | Other food commodities | 0.05* | |
| Poland | Fruits except for citrus fruits | 0.05 | |
| | Cereal grains | 0.05 | |
| | Citrus fruits | 0.05 | |
| | Eggs | 0.1 | |
| | Hops, dry | 0.05 | |
| | Meat and meat products | 1.0 | |
| | Milks and milk products | 0.04 | |
| | Potato | 0.05 | |
| | Tea | 0.1 | |
| | Vegetables | 0.05 | |

1) Expressed as mg/kg fat
2) In the case of foodstuffs with a fat content of 10% or less by weight, the residue is related to the total weight of the boned foodstuff. In such cases, the maximum level is one-tenth of the value related to fat content, but must be no less than 0.01 mg/kg.
3) In determining the residues in raw cow's milk and whole cream cow's milk, a fat content of 4% by weight should be taken as a basis. For raw milk and whole cream milk of another animal the residues are expressed on the basis of the fat: with a fat content of less than 2% by weight, the maximum level is taken as half that set for raw milk and whole cream milk; with a fat content of 2% or more by weight, the maximum level is expressed in mg/kg of fat. In such cases, the maximum level is 25 times that set for raw milk and whole cream milk.
4) For eggs and egg products with a fat content higher than 10% the maximum level is expressed in mg/kg fat. In this case the maximum level is 10 times higher than the maximum level for fresh eggs.

* Indicates lower limit of quantification (LOQ)

## APPRAISAL

DDT was first evaluated in 1966 and has been reviewed several times since. The existing Codex MRL for meat, 5 mg/kg (fat), was converted to a temporary limit in 1993. The Joint Meeting in 1993 and 1994 proposed extraneous residue limits (ERLs) for carrot, eggs, meat, and milk and confirmed the previous temporary ERL proposed for cereal grains. For meat, the 1993 JMPR proposed an ERL of 1 mg/kg. On the basis of new data on residues received from the Government of New Zealand, the 1996 JMPR concluded that the ERL of 1 mg/kg for DDT in meat (fat) recommended by the 1993 JMPR should be increased to 5 mg/kg.

At its 31st session, the CCPR (ALINORM 99/24A paras. 115-121) discussed the temporary ERL in meat of 5 mg/kg. On the basis of a 0.5% rate of violation of this value, 3 mg/kg appeared to be an appropriate value from the 1996 evaluation. This value does not, however, conform to the geometric progression approach used by the Meeting for estimating MRLs and ERLs. The CCPR requested the JMPR to reconsider its proposal on statistical validity and non-conformity to the geometric progression, on the basis of the 1996 JMPR evaluation.

***Residues in animal commodities***

The CCPR at its 30th session (ALINORM 99/24 para. 102) requested the Meeting to evaluate data derived from monitoring of chicken meat in its consideration of an ERL for that commodity. These data were provided to the Meeting from Germany, Israel, Poland, Thailand, the UK, and the USA. The Meeting also received national residue limits from The Netherlands and Poland and methods for residue analysis and monitoring of fruits and vegetables from The Netherlands. The Meeting was informed by the Governments of Germany and The Netherlands that no uses for DDT are authorized in those countries.

The results of monitoring of DDT residues were summarized in tables in which only the number of results within certain classes was given. This method of reporting has the advantages of concentrating data and allowing rapid visual interpretation. The disadvantages are that such tables are difficult to compare (the definition of classes might differ) and parameters such as "the critical level at which only 0.2% of the results is above the critical level" are difficult to determine. A statistical solution to this problem was used which is based on the assumption that each set of data can be described by a log-normal distribution. The two parameters of this distribution were estimated by maximizing the likelihood of observing the numbers reported in the classes. Since the amount of information in the data sets was rather limited, combinations of sets were made. The same standard deviation was used for all data sets except those of Thailand, and, in the case of mammalian meat, New Zealand.

A total of 103 data sets on *mammalian meat* was abstracted from the 1996 JMPR evaluation of DDT. The data sets were derived from Australia, Germany, New Zealand, Norway, Thailand, the UK, and the USA. As the data from New Zealand showed higher concentrations of residues than those from other countries, the calculations were also performed exclusively for the New Zealand data. Nevertheless, one set of data on lamb meat from a region of New Zealand with a known history of exposure to DDT was not incorporated in either calculation (see 1996 JMPR DDT evaluation, Table 4).

Since the number of samples analysed in each data set varied widely, the calculations were repeated after introduction of a weighting factor to correct for the size of the data set, giving more weight to the large ones. This procedure does justice to each sample analysed, but it has a greater effect on the outcome of the calculations for those countries that provided the larger data sets.

The estimated percentage of samples in which the concentration of residues exceeds a certain concentration is called the "violation rate". Shown below for violation rates of 0.1. 0.2, and 0.5% are the average corresponding concentrations based on all the data sets, both giving each data set the same weight and weighting each data set according to the number of samples analysed. The second table gives the same information only from the data sets provided by New Zealand. In the parameter estimations, data sets are not included in the ranges where they have no discriminating power. For example, as the New Zealand data sets contain ≤310 samples they cannot discriminate below a violation rate of 0.3%. Once the parameters are established, they can be used to extrapolate to concentrations below 0.3%.

**Weighted average of the estimated concentration of DDT (sum of *ortho,para'*-DDT, *para,para'*-DDT, *para,para'*-DDE, and *para,para'*-TDE (*para,para'*-DDD), expressed as DDT) in mammalian meat (fat) samples at various violation rates. Calculations based on data sets from Australia, Germany, New Zealand, Norway, Thailand, the UK, and the USA.**

| Concentration, mg/kg | Violation rate (%) | | |
|---|---|---|---|
| | 0.1 | 0.2 | 0.5 |
| Average | 2.1 | 1.4 | 0.8 |
| Weighted average | 1.9 | 1.2 | 0.6 |

**Weighted average of the estimated concentration of DDT (sum of *ortho,para'*-DDT, *para,para'*-DDT, *para,para'*-DDE, and *para,para'*-TDE (*para,para'*-DDD), expressed as DDT) in mammalian meat (fat) samples at various violation rates. Calculations based on data sets from New Zealand only**

| Concentration, mg/kg | Violation rate (%) | | |
|---|---|---|---|
| | 0.1 | 0.2 | 0.5 |
| Average | 3.9 | 2.7 | 1.7 |
| Weighted average | 4.8 | 3.4 | 2.1 |

Data sets on *poultry meat* were provided to the Meeting by Germany, Israel, Poland, Thailand (monitoring data), the UK, and the USA, and additional data sets from Australia, Germany, Norway, Thailand, the UK, and the USA were collected from the 1996 JMPR evaluation on DDT, yielding a total of 68 data sets. The same calculations were performed as for mammalian meat, and the results are given below, where for violation rates of 0.1. 0.2, and 0.5% the average corresponding concentration is shown when each item has the same weight and when each item is weighted by the number of samples analysed in the set.

**Weighted average of the estimated concentration of DDT (sum of *ortho,para'*-DDT, *para,para'*-DDT, *para,para'*-DDE, and *para,para'*-TDE (*para,para'*-DDD), expressed as DDT) in poultry meat (fat) samples at various violation rates. Calculations based on data sets from Australia, Germany, Israel, Norway, Poland, Thailand, the UK, and the USA**

| Concentration, mg/kg | Violation rate (%) | | |
|---|---|---|---|
| | 0.1 | 0.2 | 0.5 |
| Average | 0.19 | 0.15 | 0.10 |
| Weighed average | 0.29 | 0.24 | 0.19 |

## RECOMMENDATIONS

The Meeting estimated the range of ERLs shown below from the available monitoring data as described above. It concluded that selection of an acceptable violation rate and the weight to be given to information provided by individual countries are risk management issues, not scientific ones. The CCPR should decide which violation rate is acceptable and whether each contributing country or each analysed sample should be given the same weight. When this is decided, suitable ERLs for mammalian and chicken meat can be derived from the Tables, in which the estimated concentrations of total DDT are given for violation rates of 0.1, 0.2, and 0.5%.

For dietary intake calculations, as a worst case assumption, the highest ERLs of 5 mg/kg for mammalian meat and 0.3 mg/kg for poultry meat were used. This resulted in exposures well below the PTDI.

| Commodity | | ERL, mg/kg | |
|---|---|---|---|
| CCN | Name | New[1] | Previous |
| MM 0095 | Meat (from mammals other than marine mammals) | 1-5 (fat) | 5 (fat)[2] |
| PM 0110 | Poultry meat | 0.1-0.3 (fat) | - |

[1] The Meeting estimated the total concentrations of DDT corresponding to violation rates of 0.1%, 0.2% and 0.5% for mammalian and poultry meat according to the procedure described in Sec. 4.7 of this Report.
[2] Recommendation by the 1996 JMPR

## Dietary risk assessment

### *Chronic intake*

ERLs for DDT exist for carrot, cereal grains, eggs, and milk. The present Meeting estimated the concentrations of DDT for violation rates of 0.1, 0.2, and 0.5% in meat from mammals other than marine mammals and from poultry. For dietary intake calculations, the 'worst case' was assumed to be the highest values in the tables. Thus, 5 mg/kg for mammalian meat and 0.3 mg/kg for poultry meat would be used.

The IEDI values from the five GEMS/Food regional diets, based on ERLs, were 10-30% of the PTDI of 0.01 mg/kg bw. The Meeting concluded that the long-term intake of residues of DDT resulting from its presence in carrots, cereal grains, eggs, milk, and meat (both mammalian and poultry) has been considered by the JMPR and is unlikely to present a public health concern.

### *Short-term intake*

The Meeting concluded that an acute RfD for DDT is unnecessary. This conclusion was based on a determination that the residues of this contaminant are unlikely to present an acute risk to consumers.

## REFERENCES

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# FENTHION (039)

## EXPLANATION

Fenthion was first evaluated in 1971 and has been reviewed several times since, most recently in 1995 as part of the CCPR Periodic Review Programme. On the basis of the results from supervised trials, the 1995 JMPR withdrew the previous recommendations for MRLs except those for cherries, olives, and husked rice. It recommended new MRLs for mandarins and sweet and sour oranges to replace the MRL for citrus fruit, and proposed an amended MRL for virgin olive oil.

The 29th CCPR decided to retain the CXLs for meat and milk for 4 years according to the Periodic Review procedure until results of animal feeding studies were available.

At the 30th Session of the CCPR, it was noted that ingestion of up to 200 ml virgin olive oil containing fenthion residues at the level of the MRL would not lead to exposure exceeding the acute RfD of 0.01 mg/kg bw. The CCPR noted that new GAP for olives was being developed in the European Union and consequently new data were to be expected. The 31st Session of the CCPR was informed that animal feeding studies and new data on olives, oranges and mandarins would be available for the 2000 JMPR. It retained the draft MRLs for mandarins and sweet and sour oranges at Step 7 (7B), awaiting the residue evaluation by the 2000 JMPR.

The 32nd Session of the CCPR was informed that animal feeding studies had been provided for evaluation by the 2000 JMPR and agreed to extend the 4-year period under the Periodic Review procedure for the MRLs for meat and milks pending the evaluation.

The 1995 JMPR had listed as desirable full details of olive trials in Greece begun in May 1995, additional information on residues in treated animal feeds and on residues in meat and offal arising either from dermal treatment or consumption of fenthion-treated animal feeds in transfer studies. Information on the measured octanol/water partition coefficients of the oxidative metabolites of fenthion was also requested.

The manufacturer reported new residue trials on peaches and olives in support of pending GAP within the European Union and also resubmitted the 1968 results of trials on cherries in Germany, but these results could not be evaluated as the relevant GAP is still pending.

Information on current GAP, the fate of residues in processing peaches, apples and olives, the octanol/water partition coefficients of the oxidative metabolites, methods of residue analysis, an animal feeding study and residues in food in commerce or at consumption was also provided.

Information on national MRLs and GAP was provided by the governments of Australia, Germany and The Netherlands as follows.

Information was submitted on the octanol/water partition coefficients of fenthion and its oxidative metabolites. Measurements were made at octanol:water volume ratios of 1:1 and 1:10 as listed below.

| | log $P_{ow}$ | |
|---|---|---|
| | O/W* = 1 | O/W = 1/10 |
| Fenthion | 4.04 ± 0.05 | 4.06 ± 0.04 |
| Fenthion sulfoxide | 1.98 ± 0.04 | 1.93 ± 0.03 |
| Fenthion sulfone | 2.02 ± 0.02 | 2.17 ± 0.02 |

*octanol/water (v/v )

## USE PATTERN

The manufacturer reported registered world-wide uses of fenthion on cherries, peaches, citrus fruit and olives.

The Meeting was informed that fenthion was under review in the European Union and revised GAP was pending. Information on current GAP was provided by the manufacturer and the governments of Germany and Australia. Product labels were supplied by the manufacturer and the government of Australia.

The external application of fenthion to animals is no longer registered in Europe, and it is no longer used on lactating dairy cattle in Australia. The current MRL for residues of fenthion in milk (0.2 mg/kg) was set as a temporary standard until August 2001.

Table 1. Registered use of fenthion in Australia for direct animal treatment.

| | Form | Liveweight, kg | Application | | Withholding period |
|---|---|---|---|---|---|
| | | | Dose, ml/animal | Dose, mg/kg bw | |
| Beef cattle | Spot-on | up to 110 | 2.5 | 4.5 | 10 days |
| | | 110 to 220 | 5 | 9-4.5 | |
| Non-lactating dairy cattle | | 220 to 550 | 10 | 9-3.6 | |
| | | Over 550 | 20 | 7 | |

### Farm animal feeding studies

Fenthion is not directly applied to fodder plants, but fenthion residues may occur in plant parts or processed plant products, e.g. from rice or citrus, that are used as animal feeding stuffs.

The 1995 JMPR concluded that additional information on residues in meat and offal arising either from dermal treatment or consumption of fenthion-treated animal feeds in transfer studies was desirable. In response, a cattle feeding trial was reported to the Meeting (Phillips *et al*., 1996).

Nine Holstein dairy cows each weighing 539-632 kg were dosed orally with gelatin capsules of fenthion for 28 consecutive days. Three cows were dosed with fenthion at each level: 0.075 mg/kg bw, 0.23 mg/kg bw and 0.75 mg/kg bw, equivalent to 1.9 ppm, 7.6 ppm, and 20 ppm fenthion in the feed. An additional cow served as the control.

Organs, tissues and milk were analysed for fenthion, fenoxon and their sulfoxides and sulfones. No significant effect on body weight, feed intake, or milk production was observed at any dosage. The cows were milked twice daily on days (-1), 1, 7, 14, 21, 26, 27, and 28, and tissue samples were collected from each cow not more than 24 hours after the last dose.

At the highest dose the total residues of fenthion in the liver ranged from <0.05 mg/kg to 0.07 mg/kg and in composite perirenal, omental and subcutaneous fat from <0.05 to 0.12 mg/kg. The residues in the kidneys and in composite round, flank and loin muscle were below the LOD of 0.05 mg/kg. Residues in liver and fat were below the LOD of 0.05 mg/kg in the 0.23 mg/kg bw group. Kidneys and muscle of this group and all tissues of the 0.075 dose group were not analysed because no residues were detected at the higher dose level.

Table 2. Total fenthion residues in the edible tissues of dairy cattle after daily dosing with fenthion for 28 consecutive days.

| Feeding level, ppm | Dose, mg/kg bw | Total residues, mg/kg | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | Liver | | Kidneys | | Composite muscle | | Composite fat | |
| | | Range | Av. | Range | Av. | Range | Av. | Range | Av. |
| 1.9 | 0.075 | NA | | NA | | NA | | NA | |
| 7.6 | 0.23 | <0.05 | <0.05 | NA | | NA | | <0.05 | <0.05 |
| 20 | 0.75 | <0.05-0.07 | 0.06 | <0.05 | <0.05 | <0.05 | <0.05 | <0.05-0.12 | 0.09 |

NA not analysed LOD 0.05 mg/kg

Residues in the milk at the 0.75 mg/kg bw dose level were 0.02, 0.04, 0.03, 0.04, and 0.04 mg/kg (average) at days 1, 7, 14, 21, and 28 respectively. A plateau of about 0.04 mg/kg was reached for total residues of fenthion in the cows dosed at 0.75 mg/kg bw within 7 days. At the 0.23 and 0.075 mg/kg bw dose levels the residues in the milk were below the LOD of 0.01 mg/kg (Table 3).

Table 3. Fenthion residues in the milk from dairy cattle after daily dosing with fenthion for 28 days.

| Feeding level, ppm | Dose, mg/kg bw | Days | Total fenthion residues, mg/kg | |
|---|---|---|---|---|
| | | | Range | Average |
| 20 | 0.75 | 1 | 0.02 | 0.02 |
| | | 7 | 0.03-0.047 | 0.039 |
| | | 14 | 0.025-0.034 | 0.03 |
| | | 21 | 0.037-0.043 | 0.04 |
| | | 28 | 0.036-0.042 | 0.041 |
| 7.6 | 0.23 | 28 | <0.01 | <0.01 |
| 1.9 | 0.075 | 28 | <0.01 | <0.01 |

LOD = 0.01 mg/kg

## FATE OF RESIDUES IN STORAGE AND PROCESSING

The Meeting received information on the fate of incurred residues of fenthion during the processing of apples, peaches, oranges and olives. Processing studies on apples, oranges and olives were evaluated by the 1995 JMPR. Although maximum residue levels were not estimated for apples, peaches and olives because GAP was only pending, processing factors for apples and peaches were calculated. Processing factors for the production of crude olive oil were reported in 1995.

Peaches. Three trials were conducted in Spain in 1994 and 1995. Fenthion was applied twice to peach trees at 1.1 kg ai/ha. The water volume was 1500 l/ha, corresponding to a spray concentration of 0.075% ai. Fruits were sampled 28 days after the last application from the treated and untreated plots.

The peaches were washed in running water with slow movement in all the processes and stoned. Jam was made from peeled and stoned peaches by household preparation. The processing into preserve and juice in the laboratory simulated industrial practice (Figure 1).

Table 4. Total fenthion residues in peaches and their processed commodities from supervised trials in Spain (Heinemann and Ohs, 1997).

| Country, year (variety) | Application | | | | | PHI, days | Commodity | Residues, mg/kg | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | Water, l/ha | No. | | | | |
| Spain, 1995 (Miraflores) | 500 EC | 1.125 | 0.075 | 1500 | 2 | 28 | whole fruit<br>washed fruit<br>juice<br>jam<br>preserve | 0.03<br>0.01<br>0.02<br><0.01<br><0.01 | *RA-3013/95*<br>0385-94<br>403857<br>(Heinemann and Ohs, 1997) |
| Spain, 1995 (Gladis) | 500 EC | 1.125 | 0.075 | 1500 | 2 | 28 | whole fruit<br>washed fruit<br>juice<br>jam<br>preserve | 0.13<br>0.06<br><0.01<br><0.01<br>0.01 | *RA-3013/95*<br>0242-95<br>502421<br>(Heinemann and Ohs, 1997) |

Figure 1. Processing of peaches to jam.

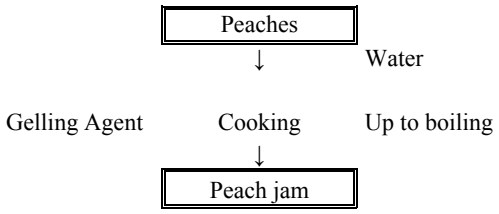



Figure 2. Processing of peaches to preserve.

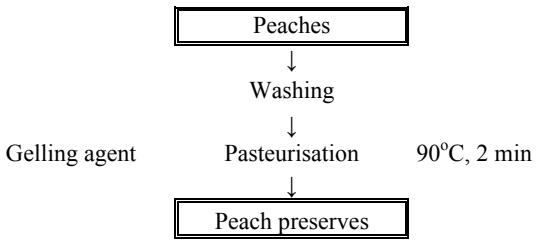
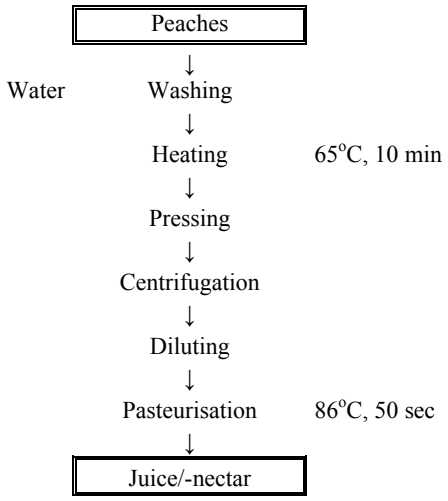



Figure 3. Processing of peaches to juice/-nectar.



**Processing factors**

Table 5. Processing factors calculated from unrounded residues of fenthion and of combined fenthion and fenthion sulfone expressed as fenthion.

| Commodity | Residues, mg/kg | | | Processing factors | | | |
|---|---|---|---|---|---|---|---|
| | Trial A | Trial B | Trial C | Trial A | Trial B | Trial C | Mean |
| Apples, 18 days PHI | 0.22 | | | - | | | |
| Apples, washed | 0.12 | | | 0.5 | | | 0.5 |
| Sauce, raw | 0.15 | | | 0.7 | | | 0.7 |
| Sauce | 0.11 | | | 0.5 | | | 0.5 |
| Juice, raw | 0.20 | | | 0.9 | | | 0.9 |
| Juice | 0.18 | | | 0.8 | | | 0.8 |
| Pomace, dry | 0.96 | | | 4.4 | | | 4.4 |
| | | | | | | | |
| Peaches, 28 days PHI | 0.17 | 0.03 | 0.13 | | | | |
| Peaches, washed | 0.40 | 0.01 | 0.06 | 2.4 | 0.33 | 0.46 | 1.06 |
| Jam | 0.06 | < 0.01 | < 0.01 | 0.35 | 0.33 | 0.08 | 0.25 |
| Preserve | < 0.01 | < 0.01 | 0.01 | 0.06 | 0.33 | 0.08 | 0.16 |
| Juice | 0.06 | 0.02 | < 0.01 | 0.35 | 0.67 | 0.08 | 0.37 |

## APPRAISAL

Fenthion was first evaluated by the Joint Meeting in 1971 and has been reviewed several times since, most recently in 1995 within the Periodic Review Programme of the CCPR. On the basis of data on residues found in supervised trials, the 1995 JMPR agreed to withdraw the previous recommendations for all MRLs except those for cherries, olives, and husked rice, which were confirmed. The 1995 JMPR recommended new MRLs for mandarins and sweet and sour oranges, replacing the existing MRL for citrus fruit, and amended the MRL for virgin olive oil.

At its twenty-ninth Session, the CCPR decided to retain the Codex MRLs for meat and milk for 4 years until data from animal feeding studies became available. At its thirtieth session, the CCPR noted that ingestion of up to 200 ml of virgin olive oil containing fenthion residues at the MRL would not exceed the acute RfD of 0.01 mg/kg bw. The Committee recognized that new GAP for olives was being developed within the European Union and, consequently, new data were to be expected. At its thirty-first Session, the CCPR was informed that the results of animal feeding studies and new data on olives, oranges, and mandarins would be available for the 2000 JMPR. The CCPR decided to retain the draft MRLs for mandarins and sweet and sour oranges to Step 7 (7B) until the residue evaluation of the 2000 JMPR became available. At its thirty-second Session, the CCPR was informed that the results of animal feeding studies had been provided to the 2000 JMPR and agreed to extend the 4-year extension for the MRLs for meat and milks, pending the review of the 2000 JMPR.

The 1995 JMPR considered that it would be desirable to have full details of the trials on olives conducted in Greece and additional information on residues in treated animal feeds and in meat and offal from animals treated externally or which had consumed fenthion-treated feeds in transfer studies. Information on the measured octanol-water partition coefficients of the oxidative metabolites of fenthion was also considered desirable.

The manufacturer provided new studies of residues in peaches and olives, to support pending GAP within the European Union, and data on cherries from a trial conducted in Germany in 1968 which had been submitted to the Meeting previously. These data were not considered, as the related GAP in the European Union is pending. In addition, information on current GAP, the fate of residues during the processing of peaches, apples, and olives, octanol-water partition coefficients for the

oxidative metabolites, methods of analysis for residues, an animal transfer study, and residues in food in commerce or at consumption were provided. Information on national MRLs and GAP was provided by Australia, Germany and The Netherlands.

***Octanol-water partition coefficients***

The manufacture submitted data on the octanol-water partition coefficients of fenthion, fenthion sulfoxide, and fenthion sulfone. The log $P_{OW}$ for fenthion is 4.04 and 4.06 at *n*-octanol:water ratios of 1 and 0.1, respectively. The two metabolites are polar, with log $P_{OW}$ values for fenthion sulfoxide of 1.98 and 1.93 and for fenthion sulfone of 2.02 and 2.17 at *n*-octanol:water ratios of 1 and 0.1, respectively.

***Residues in animal commodities***

Exposure to fenthion residues may occur during consumption of rice. In the evaluation of fenthion use on rice made by the Meeting in 1995, the concentrations of total fenthion residues in rice in the trials conducted according to GAP (median values in italics) were <0.001 (17 trials), <0.002 (2 trials), 0.008, 0.009, 0.01 (2 trials), 0.012, *<0.014* (8 trials), *<0.015* (17 trials), <0.016 (2 trials), <0.017, 0.018, <0.019 (2 trials), <0.02, <0.023, <0.024 (5 trials), and <0.028 (2 trials) mg/kg. The current Meeting estimated an STMR value of 0.0145 mg/kg.

The Meeting received data on the processing of oranges, which could have been used to assess the dietary burden from consumption of dried orange pulp; however, residues in this animal feed commodity were not determined, and the report was submitted as a summary. The Meeting decided that it was inadequate.

The Meeting considered that husked rice is a minor animal feed commodity and is therefore not suitable for calculating the dietary burden of fenthion residues in farm animals.

A study of transfer of fenthion residues in lactating dairy cows was made available to the Meeting. Encapsulated fenthion was given for 28 days to groups of three cows at a dose of 0.075, 0.23, or 0.75 mg/kg bw. The only edible tissues in which residues of fenthion were detected were liver and composite fat, and only in cows at the highest dose. In liver, the concentration of residues ranged from <0.05 to 0.07 mg/kg. In composite fat (perirenal, omental, subcutaneous), the concentration ranged from <0.05 to 0.12 mg/kg. The concentrations in kidney and composite muscle (round, flank, loin) were below the LOQ of 0.05 mg/kg. At 0.23 mg/kg bw, the concentration of total residues in liver and composite fat was below the LOQ. Kidney and muscle of cows at this dose and all tissues of cows at 0.075 mg/kg were not analysed because no residues were expected on the basis that none were found at the higher dose. The concentration of total residues of fenthion in milk reached a plateau in the cows given 0.75 mg/kg bw within 7 days, well before the 28-day slaughter. At the two lower doses, the concentration in milk was below the LOQ of 0.01 mg/kg.

The Meeting noted that, although the results of an animal feeding study had been submitted, the calculated dietary burden of fenthion residues in animal commodities was an underestimate. GAP exists for direct use of fenthion on animals, but data on dermal application to animals were not available. The Meeting was therefore unable to estimate maximum residue levels for fenthion in animal commodities. It confirmed the 1995 JMPR recommendation to withdraw the maximum residue levels for meat (of mammals other than marine mammals) and milk.

The Meeting received information on the fate of incurred residues of fenthion during the processing of apples, peaches, oranges, and olives, and processing factors were calculated for apples and peaches. Processing factors for olives were calculated by the 1995 JMPR. The processing data for oranges were not evaluated as the report submitted was inadequate.

## RECOMMENDATIONS

The 1995 recommendation to withdraw the MRLs for meat (from mammals other than marine mammals) and milks is confirmed.

Definition of the residue for compliance with MRLs and estimation of dietary intake: sum of fenthion, its oxygen analogue and their sulfoxides and sulfones, expressed as fenthion.

The residue is fat-soluble.

### Dietary risk assessment

#### *Chronic intake*

STMR values were estimated by the present Meeting for husked rice. When data on consumption were available, the STMR value was used with the existing MRLs and draft MRLs for six other food commodities in estimating dietary intake.

The dietary intakes from the five GEMS/Food regional diets, based on new and existing STMR values and MRLs, represented 1-12% of the ADI. The Meeting concluded that the dietary intake of fenthion residues would not exceed the ADI in any GEMS/Food regional diet (Annex 3). The estimates of dietary intake will be further refined during the next periodic review of residues.

#### *Short-term intake*

The acute RfD for fenthion established by the present Meeting is 0.01 mg/kg bw. The IESTIs for husked rice (Annex 3) was 0.0009 mg/kg bw (1% of the acute RfD) for aduts and 0.00018 mg/kg bw (2% of the acute RfD) for children. The Meeting concluded that it is highly unlikely that the short-term intake of fenthion would exceed the acute RfD. The lack of STMR values, except for husked rice, for fenthion in food commodities precluded a risk assessment for short-term intake, which will be assessed during the next periodic review of residues.

## REFERENCES

Heinemann, O and P. Ohs. 1997. Determination of residues of Lebaycid 500 EC on peach following spray application in peaches. Bayer Report RA-3013/95. Unpublished

Philips, J.D., L.L. Roesel, C.D. Chickering and R.R. Gronberg. 1966. A 28-day feeding study of fenthion in dairy cattle. Bayer Report No. 107313.

# MALATHION (049)

## EXPLANATION

Malathion was evaluated by the JMPR in 1999 within the Periodic Review Programme of the CCPR but no recommendations were made in relation to the existing CXLs for wheat bran, wheat flour and wheat wholemeal.

## APPRAISAL

At the 1999 JMPR, an MRL of 0.5 mg/kg was recommended and an STMR of 0.04 mg/kg estimated for malathion in wheat grain. The highest residue found in the trials was 0.28 mg/kg. A processing study on wheat was submitted to the 1999 Meeting, which gave processing factors of 0.23 for wheat flour and 0.41 for wheat bran. No processing factor was derived for wheat wholemeal. The 1999 JMPR concluded that it was unlikely that residues in grain would decrease after processing to bran, and agreed that the PF of 0.41 was unrealistic.

The present Meeting recommended an MRL of 0.2 mg/kg for wheat flour, derived by multiplying the maximum residue level estimated for wheat grain by the processing factor (0.5 mg/kg x 0.23 = 0.115 mg/kg), and an STMR-P of 0.0092 mg/kg, which corresponds to the processing factor multiplied by the STMR for wheat.

The Meeting recommended the withdrawal of the MRLs for wheat bran and wheat wholemeal.

## RECOMMENDATIONS

The Meeting concluded that the residue levels shown below are suitable for establishing maximum residue limits and for the assessment of dietary intake.

Definition of the residue for compliance with MRLs and for the estimation of dietary intake: malathion

| CCN | Commodity | MRL, mg/kg | | STMR or STMR-P, mg/kg |
|---|---|---|---|---|
| | | New | Previous | |
| CF 1211 | Wheat flour | 0.2 | 2 PoP | 0.0092 |
| CF 1212 | Wheat wholemeal | W | 2 PoP | |
| CM 0654 | Wheat bran, unprocessed | W | 20 PoP | |

## DIETARY RISK ASSESSMENT

Chronic. Currently, the ADI for malathion is 0.3 mg/kg body weight/day. International estimated daily intake (IEDI) was calculated for commodities of human consumption which STMRs were estimated in this evaluation and by the 1999 JMPR. The results are shown in Annex III.

International Estimated Daily Intakes for the five GEMS/Food regional diets, based on estimated STMRs were 0% of the ADI. The Meeting concluded that the intake of residues of pyrethrins resulting from its uses that have been considered by the JMPR is unlikely to present a public health concern.

Acute. The International Estimated Short-term Intake (IESTI) for malathion was calculated as described in Section 3 for wheat flour. The result is shown in Annex IV. The IESTI was for children and for adults. As no acute reference dose has been established the acute risk assessment for malathion was not finalised.

# MEVINPHOS (053)

## EXPLANATION

Mevinphos was evaluated in the Periodic Review Programme of the CCPR by the 1997 JMPR, which concluded that the existing MRLs for some crops (broccoli, Brussels sprouts, cauliflower, citrus fruits, cucumber, grapes, melons, peas, spinach, strawberry and tomatoes) should be withdrawn, owing to inadequacies in the available information. The 31st CCPR decided to maintain the CXLs for those commodities for 4 years, as the manufacturer had indicated its intention to submit new residue data to the 2000 JMPR, except on Brussels sprouts and cauliflower. The compound was reviewed for toxicology at the 1996 JMPR, which allocated an ADI of 0 - 0.0008 mg/kg bw and an acute RfD of 0.003 mg/kg bw.

The Meeting received reports of studies on storage stability and processing, analytical methods, additional information on GAP, and updated supervised residue trials on vegetables and fruits.

Information on analytical methods, national MRLs, and residues in food in commerce were supplied by The Netherlands.

## METHODS OF RESIDUE ANALYSIS

### Analytical methods for crops

The sample is extracted with acetonitrile and the homogenate filtered into a separatory funnel. Solid sodium chloride is added and the acetonitrile layer drained through anhydrous sodium sulfate. The solvent is evaporated to near dryness and the residue dissolved in acetone. The final extract is analysed by GLC with an FPD in the phosphorus mode. The results of validation experiments are shown in Table 1.

Table 1. Recovery of mevinphos from fortified crops.

| Crop | Fortification level of mevinphos (E + Z), mg/kg | Recovery, %[1] |
|---|---|---|
| Broccoli | 0.05 | 84, 104 |
| | 0.5 | 103, 95 |
| Cantaloupes | 0.05 | 102 |
| | 1.0 | 101 |
| Cucumbers | 0.05 | 86 |
| | 0.1 | 99, 90, 102 |
| | 0.2 | 92 |
| | 1.0 | 91, 96 |
| Grapes | 0.05 | 92, 69, 69 |
| | 0.1 | 89, 90 |
| | 0.2 | 94 |
| | 1.0 | 72, 79, 81 |
| Lemons | 0.05 | 91, 104 |
| | 0.50 | 102, 76 |
| Melons | 0.1 | 95 |
| | 1.0 | 91 |
| Peas | 0.05 | 102, 106 |
| | 0.1 | 103, 85, 104 |
| | 1.0 | 95, 104 |

| Crop | Fortification level of mevinphos (E + Z), mg/kg | Recovery, %[1] |
|---|---|---|
| Spinach | 0.1 | 97, 108 |
| | 1.0 | 90 |
| Strawberries | 0.05 | 98, 69, 104 |
| | 0.1 | 93, 105 |
| | 0.2 | 97 |
| | 1.0 | 101 |
| | 1.5 | 91, 97 |
| Tomatoes | 0.05 | 78 |
| | 0.1 | 94, 92 |
| | 0.2 | 90 |
| | 1.0 | 95, 98 |

[1] Each value corresponds to one sample.

## Stability of residues in stored analytical samples

Tomato, strawberry, broccoli, lettuce, and cucumber samples were fortified with mevinphos at 0.05 and 0.5 mg/kg and stored frozen at -20°C. Single samples taken after 0, 1, 3, and 6 months were analysed by GLC with an FPD. The percentages of residues remaining after storage are shown in Table 2. The complete study reports were not provided to the Meeting.

Table 2. Stability of mevinphos in fortified samples stored at -20°C.

| Commodity | Storage period, months | Fortification, mg/kg | % remaining | | |
|---|---|---|---|---|---|
| | | | *E*-isomer | *Z*-isomer | Total |
| Broccoli | 0 | 0.05 | 96 | 98 | 97 |
| | 1 | 0.05 | 65 | 83 | 70 |
| | 3 | 0.05 | 55 | 89 | 65 |
| | 6 | 0.05 | 58 | 107 | 72 |
| | 0 | 0.5 | 98 | 98 | 98 |
| | 1 | 0.5 | 85 | 96 | 88 |
| | 3 | 0.5 | 74 | 86 | 78 |
| | 6 | 0.5 | 88 | 105 | 93 |
| Cucumber | 0 | 0.05 | 91 | 91 | 91 |
| | 1 | 0.05 | 72 | 94 | 78 |
| | 3 | 0.05 | 63 | 89 | 70 |
| | 6 | 0.05 | 64 | 100 | 74 |
| | 0 | 0.5 | 89 | 90 | 90 |
| | 1 | 0.5 | 88 | 93 | 89 |
| | 3 | 0.5 | 84 | 90 | 86 |
| | 6 | 0.5 | 88 | 95 | 90 |
| Lettuce | 0 | 0.05 | 88 | 89 | 88 |
| | 1 | 0.05 | 62 | 94 | 71 |
| | 3 | 0.05 | 49 | 86 | 59 |
| | 6 | 0.05 | 51 | 85 | 61 |
| | 0 | 0.5 | 91 | 91 | 91 |
| | 1 | 0.5 | 88 | 95 | 90 |
| | 3 | 0.5 | 78 | 87 | 80 |
| | 6 | 0.5 | 84 | 97 | 88 |
| Strawberry | 0 | 0.05 | 97 | 97 | 97 |
| | 1 | 0.05 | 94 | 93 | 94 |
| | 3 | 0.05 | 80 | 81 | 80 |
| | 6 | 0.05 | 89 | 91 | 90 |
| | 0 | 0.5 | 96 | 97 | 96 |
| | 1 | 0.5 | 86 | 87 | 86 |
| | 3 | 0.5 | 87 | 88 | 88 |
| | 6 | 0.5 | 93 | 99 | 95 |

| Commodity | Storage period, months | Fortification, mg/kg | % remaining | | |
|---|---|---|---|---|---|
| | | | *E*-isomer | *Z*-isomer | Total |
| Tomato | 0 | 0.05 | 96 | 96 | 96 |
| | 1 | 0.05 | 94 | 99 | 95 |
| | 3 | 0.05 | 81 | 93 | 84 |
| | 6 | 0.05 | 80 | 97 | 85 |
| | 0 | 0.5 | 96 | 96 | 96 |
| | 1 | 0.5 | 98 | 101 | 99 |
| | 3 | 0.5 | 85 | 89 | 86 |
| | 6 | 0.5 | 89 | 102 | 93 |

## USE PATTERN

The Meeting received additional information on the registered uses of mevinphos on selected crops in Mexico (Table 3).

Table 3. Registered uses of mevinphos in Mexico.

| Crop | Application | | | | PHI, days |
|---|---|---|---|---|---|
| | Method | kg ai/ha | kg ai/hl | No. | |
| Cucumber | Foliar | 0.24-0.48 | not specified | not specified | 1 |
| Egg plant | Foliar | 0.24-0.48 | not specified | not specified | 2 |
| Grape vine | Foliar | 0.24-0.48 | not specified | not specified | 2 |
| Lettuce | Foliar | 0.24-0.48 | not specified | not specified | 2 |
| Muskmelon | Foliar | 0.24-0.48 | not specified | not specified | 1 |
| Pepper | Foliar | 0.24-0.48 | not specified | not specified | 2 |
| Potato | Foliar | 0.24-0.48 | not specified | not specified | 1 |
| Tomato | Foliar | 0.24-0.48 | not specified | not specified | 1 |
| Watermelon | Foliar | 0.24-0.48 | not specified | not specified | 1 |

## RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised residue field trials on broccoli, cantaloupes, cucumbers, grapes, lemons, melons, peas, spinach, strawberries and tomatoes.

| | |
|---|---|
| Table 4 | Lemons. USA |
| Table 5 | Grapes. Mexico, USA |
| Table 6 | Strawberries. Mexico, USA |
| Table 7 | Broccoli. Mexico, USA |
| Table 8 | Melons. Mexico, Cantaloupe. USA |
| Table 9 | Cucumbers. Mexico |
| Table 10 | Tomatoes. Mexico |
| Table 11 | Spinach. Mexico, USA |
| Table 12 | Peas. Mexico, USA |

When residues were not detected, data in the Tables are shown as below the limit of quantification (LOQ) e.g. <0.01 mg/kg.

Lemons. Mevinphos was applied by tractor-mounted air blast sprayer or backpack single air blast sprayer in the USA. The trials were conducted with plot sizes of 140 to 220 $m^2$. Established populations were normal density (about 6.7 m tree space, 3.6 to 5.7 m tree spread). Field samples (>16 fruits from 3 individual trees) were stored in a freezer for 12 to 24 days before analysis.

Table 4. Mevinphos residues in lemons (whole fruit) resulting from supervised trial in the USA in 1993 (Obrist, 1997g).

| Location | Form. | Application | | | | Variety | PHI, days | Residues mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| CA. Tulare Co. | SL | 1 | 0.98 | 1900 | 0.05 | Pryor | 0<br>1<br>2<br>4<br>7<br>10 | 0.14<br>0.14<br>0.05<br>0.04<br><0.02<br><0.02 | 0.07<br>0.08<br>0.05<br>0.05<br>0.03<br>0.03 | 0.21<br>0.22<br>0.10<br>0.09<br>0.03<br>0.03 | SARS -93-14 |
| CA. Tulare Co. | SL | 1 | 0.98 | 1900 | 0.05 | Lisbon | 4 | 0.08 | 0.11 | 0.19 | |
| AZ. Yuma Co. | SL | 1 | 0.98 | 850 | 0.12 | Lisbon Rough Lemon Stock | 4 | 0.15 | 0.16 | 0.31 | |

Grapes. Mevinphos was applied by $CO_2$ backpack sprayer with U-shaped boom applicator in Mexico, and by air blast or inverted U boom in the USA. In Mexico 6 trials were conducted with plot sizes of 260 or 280 $m^2$. Established populations were 1300 to 2600 plants/ha. Field samples (6 samples, each containing 2.0 kg) were stored in a freezer for 1 to 2 months before analysis.

US trials were conducted with plot sizes of 90 to 180 $m^2$. Established populations were normal density (about 1 plant per 2.1 to 2.4 m, 3.4 to 3.7 m between rows). Field samples (12 bunches) were stored in a freezer for 1 month before analysis.

Table 5. Mevinphos residues in grapes resulting from supervised trials in Mexico (Obrist and Kent, 1998a) and the USA (Obrist, 1997e). All SL formulations.

| Country, Year, Location | Application | | | | Variety | PHI, days | Residues, mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|
| | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| Mexico, 1997 Sonora St. Pesqueria Co. | 1 | 0.44 | 1000 | 0.04 | Flames | 5 | 0.01 | 0.01 | 0.02 | AMV9601 2 |
| Mexico, 1997 Sonora St. Pesqueria Co. | 1 | 0.44 | 1000 | 0.04 | Flames | 5 | <0.01 | <0.01 | <0.02 | |
| Mexico, 1997 Sonora St. Pesqueria Co. | 1 | 0.44 | 1000 | 0.04 | Flames | 5 | 0.01 | <0.01 | 0.01 | |
| Mexico, 1997 Sonora St. Heroica Caborca Co. | 1 | 0.44 | 1090 | 0.04 | Superior | 5 | <0.01 | <0.01 | <0.02 | |
| Mexico, 1997 Sonora St. Heroica Caborca Co. | 1 | 0.44 | 810 | 0.05 | Thompson Seedless | 5 | <0.01 | <0.01 | <0.02 | |
| Mexico, 1997 Sonora St. Ejido Tlaxcala Co. | 1 | 0.44 | 1090 | 0.04 | Cardinal | 5 | <0.01 | <0.01 | <0.02 | |
| USA 1993 CA. Riverside Co. | 1 | 1.2 | 880 | 0.14 | Thompson | 5 | <0.02 | 0.03 | 0.03 | SARS-93-11 |
| USA 1993 CA. Tulare Co. | 1 | 0.98 | 1800 | 0.05 | Emperor | 5 | 0.06 | 0.04 | 0.10 | |
| USA 1993 CA. Stanislaus Co. | 1 | 0.98 | 780 | 0.13 | Carrignan | 5 | 0.035 | 0.048 | 0.083 | |

| Country, Year, Location | Application | | | | Variety | PHI, days | Residues, mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|
| | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| USA<br>1993<br>WA.<br>Cowlitz Co. | 1 | 0.98 | 470 | 0.21 | Riesling | 0<br>1<br>3<br>5<br>7<br>10 | 0.63<br>0.43<br>0.25<br>0.10<br>0.09<br>0.06 | 0.30<br>0.24<br>0.16<br>0.10<br>0.09<br>0.07 | 0.93<br>0.67<br>0.41<br>0.20<br>0.18<br>0.13 | |

Strawberries. Mevinphos was applied by $CO_2$ backpack applicator in Mexico, and by $CO_2$ backpack sprayer or tractor-mounted sprayer in the USA. In Mexico, one trial was conducted on 3 plots of 60 $m^2$. Established populations were 80000 plants/ha. Field samples were stored in a freezer for 2.5 to 5 months before analysis.

US trials were conducted at 5 sites with 4 formulation types, and plot sizes of 46 to 140 m. Field samples from 12 areas of each plot were stored in a freezer for 11 days to 1 month before analysis.

Table 6. Mevinphos residues in strawberries resulting from supervised trials in Mexico (Obrist and Taylor, 1998e) and the USA (Obrist, 1997f). Analyses of replicate field samples from the treated plots in the Mexico trial are shown separately.

| Country, Year, Location | Form. | Application | | | | Variety | PHI, days | Residues mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| USA, 1997<br>CA.<br>Tulare Co | SL | 1 | 0.98 | 240 | 0.41 | Seascape | 3 | 0.21 | 0.15 | 0.36 | SARS-93-12 |
| | EC | 1 | 1.00 | 240 | 0.42 | Seascape | 3 | 0.19 | 0.14 | 0.33 | |
| | SL | 1 | 0.84 | 240 | 0.36 | Seascape | 3 | 0.17 | 0.05 | 0.22 | |
| | EC | 1 | 0.84 | 240 | 0.36 | Seascape | 3 | 0.18 | 0.06 | 0.24 | |
| USA 1997<br>CA.<br>Monterrey Co. | SL | 1 | 0.98 | 1400 | 0.07 | Selva | 3 | 0.51 | 0.35 | 0.86 | |
| | EC | 1 | 1.00 | 1400 | 0.07 | Selva | 3 | 0.68 | 0.41 | 1.09 | |
| | SL | 1 | 0.84 | 1400 | 0.06 | Selva | 3 | 0.61 | 0.15 | 0.76 | |
| | EC | 1 | 0.84 | 1400 | 0.06 | Selva | 3 | 0.77 | 0.18 | 0.95 | |
| USA 1997<br>CA. Frenso Co. | SL | 1 | 0.98 | 230 | 0.42 | Chandler | 3 | 0.48 | 0.28 | 0.76 | |
| | EC | 1 | 1.00 | 230 | 0.43 | Chandler | 3 | 0.48 | 0.27 | 0.75 | |
| | SL | 1 | 0.84 | 230 | 0.36 | Chandler | 3 | 0.30 | 0.07 | 0.37 | |
| | EC | 1 | 0.84 | 230 | 0.36 | Chandler | 3 | 0.39 | 0.09 | 0.48 | |
| USA 1997<br>FL<br>Alachura Co. | SL | 1 | 0.98 | 490 | 0.20 | Douglas | 3 | 0.35 | 0.36 | 0.71 | |
| | EC | 1 | 1.00 | 490 | 0.20 | Douglas | 3 | 0.72 | 0.46 | 1.18 | |
| | SL | 1 | 0.84 | 490 | 0.17 | Douglas | 3 | 0.55 | 0.15 | 0.70 | |
| | EC | 1 | 0.84 | 490 | 0.17 | Douglas | 3 | 0.76 | 0.19 | 0.95 | |
| USA 1997<br><br>OR<br>Washing-ton Co. | SL | 1 | 0.98 | 470 | 0.21 | Red Gem | 0<br>1<br>3<br>5<br>7<br>10 | 0.46<br>0.39<br>0.31<br>0.08<br>0.06<br>0.03 | 0.21<br>0.20<br>0.21<br>0.07<br>0.07<br>0.05 | 0.67<br>0.59<br>0.52<br>0.15<br>0.13<br>0.08 | |
| | EC | 1 | 1.00 | 470 | 0.21 | Red Gem | 0<br>1<br>3<br>5<br>7<br>10 | 0.83<br>0.49<br>0.36<br>0.23<br>0.15<br>0.07 | 0.29<br>0.21<br>0.21<br>0.17<br>0.14<br>0.11 | 1.12<br>0.70<br>0.57<br>0.40<br>0.29<br>0.18 | |
| | SL | 1 | 0.84 | 470 | 0.18 | Red Gem | 0<br>1<br>3<br>5 | 0.70<br>0.57<br>0.48<br>0.18 | 0.12<br>0.10<br>0.11<br>0.05 | 0.82<br>0.67<br>0.59<br>0.23 | |

| Country, Year, Location | Form. | Application | | | | Variety | PHI, days | Residues mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| | | | | | | | 7<br>10 | 0.15<br>0.08 | 0.05<br>0.04 | 0.20<br>0.12 | |
| | EC | 1 | 0.84 | 470 | 0.18 | Red Gem | 0<br>1<br>3<br>5<br>7<br>10 | 0.71<br>0.52<br>0.37<br>0.15<br>0.14<br>0.07 | 0.15<br>0.09<br>0.08<br>0.05<br>0.05<br>0.03 | 0.86<br>0.61<br>0.45<br>0.20<br>0.19<br>0.10 | |
| Mexico 1997 Guanajuato St. | SL | 1 | 0.44 | 230 | 0.19 | Solana | 3 | 0.16, 0.15, 0.15 | 0.07, 0.06, 0.07 | 0.23, 0.21, 0.22 | AMV96 008 |

Broccoli. Mevinphos was applied by $CO_2$ backpack applicator in Mexico and tractor-mounted boom in the USA. In Mexico, the trial was conducted on 3 plots of 6 m × 8 m each. Field samples (12 plants and >2 kg from each plot) were stored in a freezer for 3 to 5 months before analysis.

In the USA there were 5 trials with plots of 63 to 370 $m^2$. Field samples (12 heads from each plot) were stored in a freezer for 5 days to 2 months before analysis.

Table 7. Mevinphos residues in broccoli resulting from supervised trials in Mexico (Obrist and Taylor 1998a) and the USA (Obrist, 1997a). Analyses of replicate field samples from the treated plots in the Mexico trials are shown separately.

| Country, Year, Location | Form. | Application | | | | Variety | PHI, days | Residues mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| Mexico 1997 Guanajuato St. Villagran | SL | 1 | 0.44 | 200 | 0.22 | Pirata | 3 | 0.05, 0.05, 0.04 | 0.16, 0.18, 0.16 | 0.21, 0.24, 0.20 | AMV960 01 Control sample had 0.15 mg/kg as total mevinphos |
| USA 1994 CA Tulare Co. | SL | 1 | 1.0 | 240 | 0.43 | Early Green Sprouting | 0<br>1<br>3<br>5<br>7<br>10 | 0.53<br>0.30<br><0.015<br><0.007<br><0.015<br><0.015 | 0.49<br>0.38<br>0.10<br>0.03<br><0.006<br><0.003 | 1.02<br>0.68<br>0.10<br>0.03<br><0.02<br><0.02 | SARS-93-01 |
| USA 1994 CA Santa Cruz Co. | SL | 1 | 1.0 | 370 | 0.27 | Arcadia | 3 | <0.015 | 0.07 | 0.07 | |
| USA 1993 CA Fresno Co. | SL | 1 | 1.0 | 240 | 0.43 | EN C85-66 Early Sprouting | 3 | 0.22 | 0.36 | 0.58 | |
| USA 1994 CA Yolo Co. | SL | 1 | 1.0 | 210 | 0.49 | Pinnacle | 3 | 0.39 | 0.55 | 0.94 | |
| USA 1994 TX Uvalde Co. | SL | 1 | 1.0 | 190 | 0.54 | Southern Comet | 3 | <0.007 | 0.017 | 0.017 | |

Melons. Mevinphos was applied by $CO_2$ backpack applicator in Mexico. One trial was conducted on 3 plots of 6 m × 10 m each. Field samples (12 fruits, a quarter of each fruit was sent for analysis) from each plot were stored in a freezer for 3 months before analysis.

Cantaloupes. Mevinphos was applied by experimental backpack sprayer in the USA. Four trials were conducted with 4 different formulations, and a plot size of 62 $m^2$. Field samples (6 whole fruit from each plot) were stored in a freezer for 1 week before analysis.

Table 8. Mevinphos residues in melons and canteloupes resulting from supervised trials in Mexico (Obrist and Taylor, 1998b) and the USA (Obrist, 1997c).

| Crop Country, Year, Location | Form. | Application | | | | Sample (Variety) | PHI, days | Residues mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| Melon Mexico 1997 Guanajuato St. | SL | 1 | 0.22 | 240 | 0.09 | Pulp (Laguna) | 3 | 0.02, 0.03, 0.03 | 0.02, 0.02, 0.02 | 0.04, 0.05, 0.05 | AMV9 6005 |
| Cantaloupe USA AZ Yuma Co. | SL | 1 | 0.49 | 230 | 0.21 | Whole fruit | 3 | <0.015 | 0.01 | 0.01 | SARS-93-08 |
| | | | | | | Pulp (Top Score) | | <0.007 | <0.003 | <0.01 | |
| | EC | 1 | 0.50 | 230 | 0.22 | Whole fruit | 3 | <0.007 | 0.01 | 0.01 | |
| | | | | | | Pulp (Top Score) | | <0.007 | <0.003 | <0.01 | |
| | SL | 1 | 0.41 | 230 | 0.18 | Whole fruit | 3 | <0.007 | 0.006 | 0.01 | |
| | | | | | | Pulp (Top Score) | | <0.007 | <0.003 | <0.01 | |
| | EC | 1 | 0.41 | 230 | 0.18 | Whole fruit | 3 | 0.02 | 0.01 | 0.03 | |
| | | | | | | Pulp (Top Score) | | <0.007 | <0.003 | <0.01 | |

Cucumber. Mevinphos was applied by $CO_2$ backpack applicator in 7 trials in Mexico with plot sizes of 192 or 216 $m^2$. Field samples (6 samples, each of 12 fruits) from each trial site were stored in a freezer for 2 to 3 months before analysis.

Table 9. Mevinphos residues in cucumbers resulting from supervised trials in Mexico in 1997. All SL formulation. Report no. AMV9601 (Obrist and Kent, 1998b).

| Location | Application | | | | Variety | PHI, days | Residues mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|
| | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total |
| Sinaloa St. Culiacan | 1 | 0.22 | 330 | 0.07 | Cortez | 3 | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Culiacan | 1 | 0.22 | 330 | 0.07 | Conquistador | 3 | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Culiacan | 1 | 0.22 | 210 | 0.10 | Cortez | 3 | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Culiacan | 1 | 0.22 | 250 | 0.09 | Conquistador | 3 | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Culiacan | 1 | 0.22 | 340 | 0.06 | Cortez | 3 | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Culiacan | 1 | 0.22 | 330 | 0.07 | Conquistador | 3 | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Los Mochois | 1 | 0.22 | 280 | 0.08 | Napoleon | 3 | <0.01 | <0.01 | <0.02 |

Tomatoes. Mevinphos was applied by $CO_2$ backpack applicator in six trials in Mexico with plot sizes of 128 to 216 $m^2$. Established populations were 12500 to 20800 plants/ha. Field samples (12 fruits, >2 kg) were stored in a freezer for 1.5 to 2 months before analysis.

Table 10. Mevinphos residues in tomatoes resulting from supervised trials, all with 3-day PHIs and SL formulation, in Mexico in 1997. Report no. AMV96011 (Obrist and Taylor, 1998f).

| Location | Application | | | | Sample | Residues, mg/kg | | |
|---|---|---|---|---|---|---|---|---|
| | No. | kg ai/ha | water l/ha | kg ai/hl | (Variety) | *E*-isomer | *Z*-isomer | Total |

| Location | Application | | | | Sample | Residues, mg/kg | | |
|---|---|---|---|---|---|---|---|---|
| | No. | kg ai/ha | water l/ha | kg ai/hl | (Variety) | *E*-isomer | *Z*-isomer | Total |
| Sinaloa St. Culiacan | 1 | 0.22 | 240 | 0.09 | Cherry Tomato (Large) | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Culiacan | 1 | 0.22 | 240 | 0.09 | Cherry Tomato (PSO4420) | <0.01 | <0.01 | <0.02 |
| Sinaloa St. San Luis de Ahome | 1 | 0.22 | 200 | 0.11 | Saladet Tomato (Alta Hybrid) | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Ejido La Louisiana | 1 | 0.22 | 200 | 0.11 | Fresh Saladet Tomato (Alta Hybrid) | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Culiacan | 1 | 0.22 | 240 | 0.09 | Ground Tomato (Divine Hybrid) | <0.01 | <0.01 | <0.02 |
| Sinaloa St. Culiacan | 1 | 0.22 | 290 | 0.08 | Ground Tomato (Homestead) | <0.01 | <0.01 | <0.02 |

Spinach. Mevinphos was applied by $CO_2$ backpack applicator in Mexico, and by $CO_2$ backpack sprayer or tractor-mounted boom in the USA. In Mexico, the trial was on 3 plots of 40 $m^2$. Established populations were 166600 plants/ha. Field samples (2 kg of leaves) were stored in a freezer for 3 to 5 months before analysis. In the USA, 4 trials were conducted with plots of 93 to 120 $m^2$. Field samples (minimum 1 kg from 12 areas within the plot) were stored in a freezer for 8 days to 3 months before analysis.

Table 11. Mevinphos residues in spinach resulting from supervised trials in Mexico (Obrist and Taylor, 1998d) and the USA (Obrist, 1997b). Analyses of replicate field samples from the treated plots in the Mexico trial are shown separately.

| Country, Year, Location | Form. | Application | | | | Variety | PHI, days | Residues mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| USA 1994 CA Santa Cruz Co. | SL | 1 | 0.50 | 370 | 0.13 | Gladiator | 0<br>1<br>2<br>4<br>7 | 4.6<br>1.4<br>0.63<br>0.11<br>0.02 | 2.9<br>1.4<br>0.79<br>0.20<br>0.09 | 7.5<br>2.8<br>1.4<br>0.31<br>0.11 | SARS-93-06 |
| | | 1 | 1.0 | 370 | 0.27 | Gladiator | 0<br>1<br>3<br>5<br>7<br>10 | 10<br>4.0<br>0.51<br>0.16<br>0.05<br><0.015 | 6.1<br>3.0<br>0.74<br>0.36<br>0.17<br>0.04 | 16<br>7.0<br>1.2<br>0.52<br>0.22<br>0.04 | |
| USA 1993 CA San Diego Co. | SL | 1 | 0.51 | 470 | 0.11 | St. Helens | 4 | 0.15 | 0.20 | 0.35 | |
| USA 1994 | SL | 1<br>1 | 1.0<br>0.50 | 470<br>190 | 0.21<br>0.27 | St. Helens<br>Fall Green | 7<br>4 | 0.04<br><0.007 | 0.09<br>0.03 | 0.13<br>0.03 | |
| TX Uvalde Co. | | 1 | 1.0 | 190 | 0.54 | Fall Green | 7 | <0.007 | 0.01 | 0.01 | |
| USA 1994 TX Willacy Co. | SL | 1 | 0.50 | 190 | 0.26 | No.612 | 4 | 0.06 | 0.10 | 0.16 | |
| | | 1 | 1.0 | 190 | 0.53 | No.612 | 7 | 0.03 | 0.06 | 0.09 | |
| Mexico 1997 Guanajuato St. Cartazar | SL | 1 | 0.44 | 220 | 0.20 | Gladiator | 7 | <0.01, <0.01, <0.01 | <0.01, <0.01, <0.01 | <0.02, <0.02, <0.02 | AMV96 007 |

Peas. Mevinphos was applied by $CO_2$ backpack applicator in Mexico, and by $CO_2$ self-propelled bicycle, $CO_2$ backpack, and tractor-mounted sprayer in the USA. In Mexico the trial was on 3 plots of 40 $m^2$. Established populations were 150 kg/ha. Field samples (2.0 kg whole peas) were stored in a freezer for 2 to 4 months before analysis.

In the USA five trials were with plot sizes of 93 to 280 $m^2$. Established populations were normal density (about 525000 seeds per 4000 $m^2$). Field samples (12 areas within the plot, > 1 kg) were stored in a freezer for 1 to 2 months before analysis.

Table 12. Mevinphos residues in succulent peas resulting from supervised trials in Mexico (Obrist and Taylor, 1998c) and the USA (Obrist, 1997d). Analyses of replicate field samples from the treated plots in the Mexico trial are shown separately.

| Country, Year, Location | Form. | Application | | | | Sample (Variety) | PHI, days | Residues mg/kg | | | Report no. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | No. | kg ai/ha | water l/ha | kg ai/hl | | | ***E***-isomer | ***Z***-isomer | Total | |
| USA 1994 WI Columbia Co. | SL | 1 | 0.50 | 200 | 0.25 | Peas with pods | 3 | <0.007 | 0.006 | 0.006 | SARS-93-10 |
| | | | | | | vines | | 0.05 | 0.04 | 0.09 | |
| | | | | | | Hay (EGO) | | <0.015 | 0.01 | 0.01 | |
| USA 1994 WI Walworth Co. | SL | 1 | 0.50 | 240 | 0.21 | Peas with pods (Quad) | 0 | 0.47 | 0.27 | 0.74 | |
| | | | | | | | 1 | 0.13 | 0.08 | 0.21 | |
| | | | | | | | 2 | 0.04 | 0.02 | 0.06 | |
| | | | | | | | 3 | 0.02 | 0.01 | 0.03 | |
| | | | | | | | 5 | <0.007 | <0.006 | <0.021 | |
| | | | | | | | 7 | <0.007 | <0.003 | <0.01 | |
| | | | | | | vines | 0 | 4.2 | 2.4 | 6.6 | |
| | | | | | | | 1 | 0.80 | 0.81 | 1.6 | |
| | | | | | | | 2 | 0.16 | 0.26 | 0.42 | |
| | | | | | | | 3 | 0.03 | 0.07 | 0.10 | |
| | | | | | | | 5 | <0.007 | 0.01 | 0.01 | |
| | | | | | | | 7 | <0.007 | <0.003 | <0.01 | |
| | | | | | | hay | 0 | 0.10 | 0.31 | 0.41 | |
| | | | | | | | 1 | 0.03 | 0.12 | 0.15 | |
| | | | | | | | 2 | <0.015 | 0.03 | 0.03 | |
| | | | | | | | 3 | <0.007 | 0.02 | 0.02 | |
| | | | | | | | 5 | <0.007 | <0.003 | <0.01 | |
| | | | | | | | 7 | <0.007 | <0.003 | <0.01 | |
| USA 1994 MN Redwood Co. | SL | 1 | 0.50 | 187 | 0.27 | Peas with pods | 3 | <LOQ | 0.007 | 0.007 | |
| | | | | | | vines | | 0.02 | 0.03 | 0.05 | |
| | | | | | | hay (Del Monte 7071) | | <0.007 | <0.006 | <0.013 | |
| USA 1994 MN Polk Co. | SL | 1 | 0.50 | 190 | 0.27 | Peas with pods | 3 | <0.015 | 0.01 | 0.01 | |
| | | | | | | vines | | 0.06 | 0.16 | 0.22 | |
| | | | | | | hay (Wando) | | 0.03 | 0.10 | 0.13 | |
| USA 1994 WA Grant Co. | SL | 1 | 0.50 | 220 | 0.23 | Peas with pods | 3 | 0.03 | 0.04 | 0.07 | |
| | | | | | | vines (Scout) | | 0.20 | 0.23 | 0.43 | |
| Mexico 1997 Guana-juato St. Silao | SL | 1 | 0.22 | 240 | 0.09 | Peas with pods (Del Monte 5063) | 3 | <0.01, <0.01, <0.01 | <0.01, <0.01, <0.01 | <0.02, <0.02, <0.02 | AMV96006 |

## RESIDUES IN PROCESSING

The Meeting received a report of one commercial processing study on lemons (Obrist, 1997h). Lemons were treated with a single application at 5 times the maximum label rate of 5.0 kg ai/ha with

1900 l/ha SL formulation. Mature lemons were harvested 4 days after application. The samples were shipped to the processing facility and processed into fresh juice, wet pulp, dry pulp, oil, molasses, and pasteurized juice. The processing procedure is shown in Figure 1.

Figure 1. Processing of lemons.

Fresh sample
⇓
Washing in water
⇓
Grinding to remove rot bruise
⇓
Abrade or rasp off Flavedo ⇒ centrifuge ⇒ ***PEEL OIL***
⇓
Juice extraction ⇒ press peel ⇒ Liming w/CaO
⇓ ⇓
Pulper finisher ⇒ ***WET PULP*** (70-73% $H_2O$) Press shredded material
⇓ ⇓
***FRESH JUICE*** Dryer (79-93°C) ⇓
⇓ ⇓ Concentration
Pasteurize to 88 °C for 15-20 sec. ***DRIED PULP*** (<10% $H_2O$) ⇓
⇓ ***MOLASSES***
Pack in bottles
***(PASTEURIZED JUICE)***

The results are shown in Table 13.

Table 13**.** Residues of mevinphos in lemons and processed fractions in the USA in 1993.

| Application, Ref. | Sample | Residues (mg/kg) | | | Processing Factor |
|---|---|---|---|---|---|
| | | ***E***-isomer | ***Z***-isomer | Total | |
| 5.0 kg ai/ha × 1 | Lemons (RAC) | 0.10 | 0.24 | 0.34 | - |
| 4 PHI days | Fresh Juice | <0.02 | 0.02 | 0.04 | 0.12 |
| | Wet pulp | 0.07 | 0.19 | 0.25 | 0.74 |
| | Dry pulp | <0.02 | 0.07 | 0.09 | 0.26 |
| SARS-93-17 | Oil | <0.02 | <0.01 | <0.03 | <0.09 |
| | Molasses | <0.02 | <0.01 | <0.03 | <0.09 |
| | Pasteurized Juice | <0.02 | <0.01 | <0.03 | <0.09 |

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The government of The Netherlands reported monitoring data for mevinphos on several crops (Table 14).

Table 14. Monitoring data for mevinphos on crops in The Netherlands, 1994-1997.

| Commodity | Samples analysed [1] | Samples with residues | Mean residues, mg/kg [2] |
|---|---|---|---|
| Blackberries | 299 | 5 | <0.05 |
| Chinese cabbage | 413 | 5 | <0.05 |
| Celery | 412 | 5 | <0.05 |
| Currants | 576 | 2 | <0.05 |
| Endive | 1503 | 29 | <0.05 |
| Lettuce | 4134 | 116 | <0.05 |

| Commodity | Samples analysed [1] | Samples with residues | Mean residues, mg/kg [2] |
|---|---|---|---|
| Peppers | 607 | 1 | <0.05 |
| Spinach | 610 | 6 | <0.05 |
| Strawberries | 3157 | 35 | <0.05 |

[1] LOQ 0.05 mg/kg
[2] For samples without residues, half the LOQ is taken for the calculation of the mean value.

## NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported.

| Country | MRL, mg/kg | Commodity |
|---|---|---|
| Australia | 0.25 | Cabbages, head, flowerhead brassicas |
| | 0.05* | Edible offal (mammalian), meat (mammalian) |
| Austria | 0.05 | Sugar beets |
| | 0.1 | Cabbages, grapes |
| | 0.2 | Apples, apricots, pears |
| | 0.5 | Cherries, peaches, plums |
| Belgium | 0.1 | Cucumbers, peas, peppers, potato, tomato |
| | 0.2 | Apples, pears |
| France | 0.1 | Beans, courgettes, cucumbers, egg plants, gherkins, grapes, hazelnuts, melons, peas, peppers, strawberries, tomato |
| | 0.2 | Apples, apricots |
| | 0.5 | Almonds, beets, cabbages, corn salad, fodder beets, garden cress, lettuce, peaches, plums, spinach |
| Luxembourg | 0.1 | Peas, peppers, potato, tomato |
| | 0.2 | Apples, pears |
| Netherlands | 0.05* | Other food commodities |
| | 0.1 | Other vegetables, potatoes, other fruit |
| | 0.2 | Citrus fruit, pome fruit, apricots |
| | 0.5 | Other stone fruit, leaf vegetables (except herbs) |
| Portugal | 0.1 | Cucumbers, garlic, melons, tomato |
| Switzerland | 0.3 | Apples, grapes, pears, peas |

* Limit of quantification

## APPRAISAL

Mevinphos was evaluated by the Meeting in 1997 within the Periodic Review Programme of the CCPR. The Meeting concluded that the existing MRLs for mevinphos in some crops (broccoli, Brussels sprouts, cauliflowers, citrus fruits, cucumbers, grapes, melons, peas, spinach, strawberries, and tomatoes) should be withdrawn, owing to inadequacies in the available information. At its thirty-first session, the CCPR decided to maintain the Codex MRLs for those commodities for 4 years, as the manufacturer had indicated its intention to submit new data on residues to the 2000 JMPR, except for Brussels sprouts and cauliflower. The compound was reviewed for toxicity by the Meeting in 1996, which allocated an ADI of 0-0.0008 mg/kg bw and an acute RfD of 0.003 mg/kg bw.

The present Meeting received information on analytical methods, stability under storage on tomatoes, strawberries, broccoli, lettuce, and cucumbers, the results of supervised trials on broccoli, cantaloupes, cucumbers, grapes, lemons, melons, peas, strawberries, spinach, and tomatoes, and one study of processing of lemons. Information on analytical methods, national MRLs, and residues in food in commerce were supplied by The Netherlands.

### *Methods of analysis*

Mevinphos consists of two isomers, *E* and *Z*. In the analytical method, residues are extracted by maceration with acetonitrile, and solid sodium chloride is added for separation. The acetonitrile layer is dried with anhydrous sodium sulfate, the solvent is evaporated, and mevinphos is determined by gas-liquid chromatography with flame photometry. The LOQs are 0.01 mg/kg for *E* plus *Z*, 0.02 mg/kg for *E*, and 0.01 mg/kg for *Z*; the recovery ranges from 69 to 108%.

***Stability of residues in stored analytical samples***

Studies on the stability of residues in stored tomatoes, strawberries, broccoli, lettuce, and cucumbers were reported. Samples were fortified with mevinphos at 0.05 and 0.5 mg/kg and stored frozen (-20 °C) for 0, 1, 3, and 6 months. As complete descriptions of the studies were not provided, the data could not be evaluated.

Studies of the stability of residues in lettuce, strawberries, and turnip tops were evaluated by the Meeting in 1997. Residues were stable at about 0.68 mg/kg (*E* plus *Z*) on strawberry fruit for 4-10 months, at about 1.0 mg/kg (*E* plus *Z*) on lettuce stored frozen for 3-10 months, and at about 0.47 mg/kg on turnip tops stored for 3-10 months.

***Results of supervised trials***

Supervised trials on lemons, strawberries, broccoli, spinach, and peas were conducted in Mexico and in the USA, where there is no registered GAP. No relevant GAP was available to evaluate the data on these crops, and the Meeting confirmed its previous recommendation to withdraw the MRLs for citrus fruits, strawberry, broccoli, spinach, and peas.

Although supervised trials of the use of mevinphos on *grapes and melons* were reported from Mexico and the USA, no data were provided on trials conducted according to GAP. The Meeting confirmed its previous recommendations to withdraw the MRL for grapes and melons except watermelon.

Although supervised trials of use of mevinphos on *cucumbers and tomatoes* were reported from Mexico, no data were provided from trials conducted according to GAP, and the Meeting confirmed its previous recommendations to withdraw the MRLs for cucumber and tomato.

***Fate of residues during processing***

One study on processing lemons was reported. The samples were processed into fresh juice, wet pulp, dry pulp, oil, molasses, and pasteurized juice. The processing factors were 0.12 for fresh juice, 0.74 for wet pulp, 0.26 for dry pulp, and <0.09 for oil, molasses, and pasteurized juice.

## RECOMMENDATIONS

New data from trials on a number of commodities were available, but none were according to relevant GAP. The Meeting therefore confirmed the recommendations of the 1997 JMPR to withdraw the MRLs concerned.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: sum of (*E*)- and (*Z*)-mevinphos.

| Commodity | | MRL, mg/kg | |
|---|---|---|---|
| CCN | Name | New | Previous[1] |
| VB 0400 | Broccoli | W | W |

| Commodity | | MRL, mg/kg | |
|---|---|---|---|
| CCN | Name | New | Previous[1] |
| FC 0001 | Citrus fruits | W | W |
| VC 0424 | Cucumber | W | W |
| FB 0269 | Grapes | W | W |
| VC 0046 | Melons, except watermelon | W | W |
| VP 0063 | Peas (pods and succulent = immature seeds) | W | W |
| VL 0502 | Spinach | W | W |
| FB 0275 | Strawberry | W | W |
| VO 0506 | Tomato | W | W |

[1] Recommendations of the 1997 JMPR

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# PARATHION (58)

## EXPLANATION

Parathion was first evaluated in 1965 and has been reviewed several times since, most recently in 1995 and 1997. It was listed by the 1998 CCPR (30th Session, ALINORM 99/24, Appendix VII) under the Periodic Review programme for residues by the 2000 JMPR. A comprehensive data package was provided by the basic manufacturer. Information was also provided by Australia, Germany and The Netherlands.

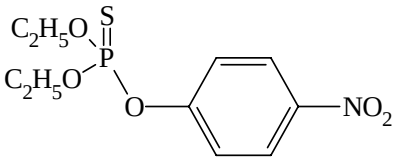
## IDENTITY

ISO common name: parathion

Chemical name
IUPAC: *O*,*O*-diethyl *O*-4-nitrophenyl phosphorothioate
CA: *O*,*O*-diethyl *O*-(4-nitrophenyl) phosphorothioate

CAS No.: 56-38-2

Structural formula:




Molecular formula: $C_{10}H_{14}NO_5PS$

Molecular weight: 291.3

### Physical and chemical properties

Pure active ingredient

Appearance: amber, with pungent garlic-like odour.

Vapour pressure: $0.965\ (\pm 0.055) \times 10^{-5}$ mm Hg at 25°C
$12.6\ (\pm 0.62) \times 10^{-5}$ mm Hg at 45°C

Melting point: 0°C

Octanol/water partition coefficient: $P_{ow} = 1598$

Solubility:

water: 12.4±0.7 mg/l at 25°C.
solvents: freely soluble in alcohols, esters, ketones and aromatic hydrocarbons; practically insoluble in petroleum oils (petroleum ether, kerosene) and paraffin oil.

Specific gravity: 1.26 g/ml

Hydrolysis:

half-life at 25°C in the dark under sterile conditions at initial conc. of 10 mg/l
pH 5: 133 days
pH 7: 247 days
pH 9: 102 days.

Photolysis:

half-life 88 hours when exposed as a thin film to UV light with peak energy output at 300 nm.

Thermal stability: heating at 150°C for 24 hours resulted in 80-90% decomposition. Parathion should not be heated above 55°C.

Technical material

Purity: minimum 96%

Main impurity: 4-nitrophenol

Stability: A sample of parathion, in its commercial package, was stored at 20-23°C in an air-conditioned room at the production plant for one year. The initial parathion content was 98.6/97.9% (duplicate analyses) and the final values were 98.1/97.7%.

Formulations

Novafos E500 Insecticide. Nova Parathion 25 EC. Parathion EC 50. Novafos E 20. E 605 forte. Microcap E 560. Parathion 8E.

## METABOLISM AND ENVIRONMENTAL FATE

### Animal metabolism

The Meeting received information on the metabolism of parathion in lactating goats and laying hens.

Residues were measured in the tissues (muscle, omental and perirenal fat, liver, kidney), milk and excreta of lactating dairy goats (2 goats weighing 57 and 42 kg initially and 47 and 34 kg finally, control goat 48 kg decreasing to 42 kg) dosed orally by capsule with 188 mg [*phenyl*-$^{14}$C]parathion equivalent to 96.9 ppm parathion in the diet for 5 consecutive days (Cheng, 1987a). The feed intake was 1.7 and 2.2 kg/animal/day. The two goats produced averages of 2.97 and 2.17 kg milk per day and were slaughtered 6 hours after the last dose. The total recovery of $^{14}$C was 42.4% and 36.65% for the two goats.

Table 1. Distribution of radioactivity in lactating goats dosed orally for 5 consecutive days by capsule with 188 mg [*phenyl*-$^{14}$C]parathion equivalent to 96.9 ppm parathion in the diet (Cheng, 1987a).

| Sample | Recovered $^{14}$C | | | |
|---|---|---|---|---|
| | Goat 092 | | Goat 088 | |
| | % of dose | mg/kg as parathion | % of dose | mg/kg as parathion |
| Kidney | 0.08 | 5.5 | 0.07 | 4.4 |
| Liver | 0.76 | 6.3 | 0.57 | 5.3 |
| Muscle | 0.08 | 0.75 | 0.03 | 0.32 |
| Omental fat | 0.03 | 0.86 | 0.03 | 0.34 |
| Renal fat | <0.01 | 0.90 | 0.03 | 0.34 |
| Bile | 0.16 | 35 | 0.08 | 31 |
| Milk | 0.20 | 1.0 | 0.18 | 0.45 |
| Urine + pan rinse | 25.6 | | 23.5 | |
| Faeces | 15.5 | | 12.1 | |
| Total | 42.4 | | 36.6 | |

83-97% of the radioactivity in the tissues and milk was extractable. The main component of the residue was *p*-acetamidoparaoxon and parathion itself was also present in all the samples (Cheng, 1988a).

Table 2. Compounds identified in tissues and milk from lactating goats dosed orally for 5 consecutive days by capsule with 188 mg [*phenyl*-$^{14}$C]parathion equivalent to 96.9 ppm parathion in the diet (Cheng, 1988a).

| Residue | Milk | | Liver | | Kidney | | Renal fat | | Muscle | |
|---|---|---|---|---|---|---|---|---|---|---|
| | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg |
| Unidentified | | | | | 8.3 | 0.45 | 3.3 | 0.030 | 1.7 | 0.013 |
| *p*- acetamido-phenol | | | 9.9 | 0.63 | 17 | 0.94 | 2.9 | 0.027 | 0.73 | 0.005 |
| *p*-nitrophenol | | | | | 1.7 | 0.094 | | | 9.2 | 0.066 |
| *p*-acetamido-paraoxon | 71 | 0.72 | 32 | 2.0 | 19 | 1.1 | 23 | 0.21 | 40 | 0.29 |
| *p*-amino-parathion | 2.5 | 0.026 | 3.8 | 0.24 | 3.5 | 0.19 | 2.2 | 0.020 | | |
| parathion | 1.8 | 0.019 | 8.9 | 0.56 | 8.8 | 0.48 | 16 | 0.15 | 2.7 | 0.019 |

In a trial on 15 White Leghorn laying hens (each weighing 1.34-2.1 kg), the hens were dosed orally by capsule with 1.5 mg [*phenyl*-$^{14}$C]parathion, equivalent to 16.5 ppm parathion in the diet, for 6 days (Cheng, 1987b). The average feed intake was 91g/bird/day. Eggs and excreta were collected throughout, the hens were killed 6 hours after the last dose for analysis of the liver, kidney, abdominal fat tissue, thigh muscle, breast muscle, skin with fat, and gizzard. The mean total recovery of $^{14}$C was 83%. The distribution of the $^{14}$C is shown in Table 3. The residue in the eggs has apparently not reached a plateau by day 6.

Table 3. Distribution of radioactivity in laying hens dosed orally by capsule with 1.5 mg [*phenyl*-$^{14}$C]parathion equivalent to 16.5 ppm parathion in the diet for 6 days (Cheng, 1987b).

| Sample | Mean recovered $^{14}$C | |
|---|---|---|
| | % of dose | mg/kg as parathion |
| Kidney | 0.04% | 0.25 |
| Liver | 0.05% | 0.093 |
| Thigh muscle | <0.01% | 0.008 |
| Breast muscle | <0.01% | 0.007 |
| Abdominal fat | 0.01% | 0.028 |
| Skin with fat | 0.04% | 0.081 |
| Gizzard | 0.03% | 0.10 |
| Eggs, total | 0.02% | |
| eggs day 3 | | 0.005 |
| eggs day 4 | | 0.009 |
| eggs day 5 | | 0.011 |
| eggs day 6 | | 0.014 |
| GI tract | 5.4% | |
| Excreta | 77% | |
| Total | 83% | |

Cheng (1988b) identified the $^{14}$C residues in the hen tissues and eggs. 53-86% of the radioactivity was extractable. The major components of the residue were *p*-nitrophenyl phosphate and *p*-acetamidophenol. Parathion itself was a minor component of the residue in all the samples.

Table 4. Compounds identified in tissues and eggs from laying hens dosed orally for 6 consecutive days by capsule with 1.5 mg [*phenyl*-$^{14}$C]parathion equivalent to 16.5 ppm parathion in the diet (Cheng, 1988b).

| Compound | Eggs | | Liver | | Kidney | | Skin + fat | |
|---|---|---|---|---|---|---|---|---|
| | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg | % of $^{14}$C in sample | $^{14}$C as parathion mg/kg |
| *p*-nitrophenyl phosphate | 19 | 0.003 | 8.1 | 0.009 | 35 | 0.087 | 7.5 | 0.013 |
| *p*-aminophenol | 3.5 | 0.001 | | | | | 4.7 | 0.008 |
| *p*- acetamido-phenol | 24 | 0.004 | 25 | 0.028 | 11 | 0.028 | 11 | 0.018 |
| *O*-ethyl *p*-nitrophenyl phosphorothioate | 7.1 | 0.001 | 13 | 0.014 | 3.5 | 0.009 | 8.6 | 0.014 |
| *p*-nitrophenol | 5.3 | 0.001 | 11 | 0.012 | 1.5 | 0.004 | 14 | 0.024 |
| *p*-acetamido-paraoxon | | | 4.1 | 0.004 | 0.98 | 0.002 | 5.3 | 0.009 |
| paraoxon | | | 0.96 | 0.001 | 0.60 | 0.001 | | |
| parathion | 4.4 | 0.001 | 0.73 | 0.001 | 1.8 | 0.004 | 1.3 | 0.002 |

The proposed metabolic pathways are shown in Figure 1.

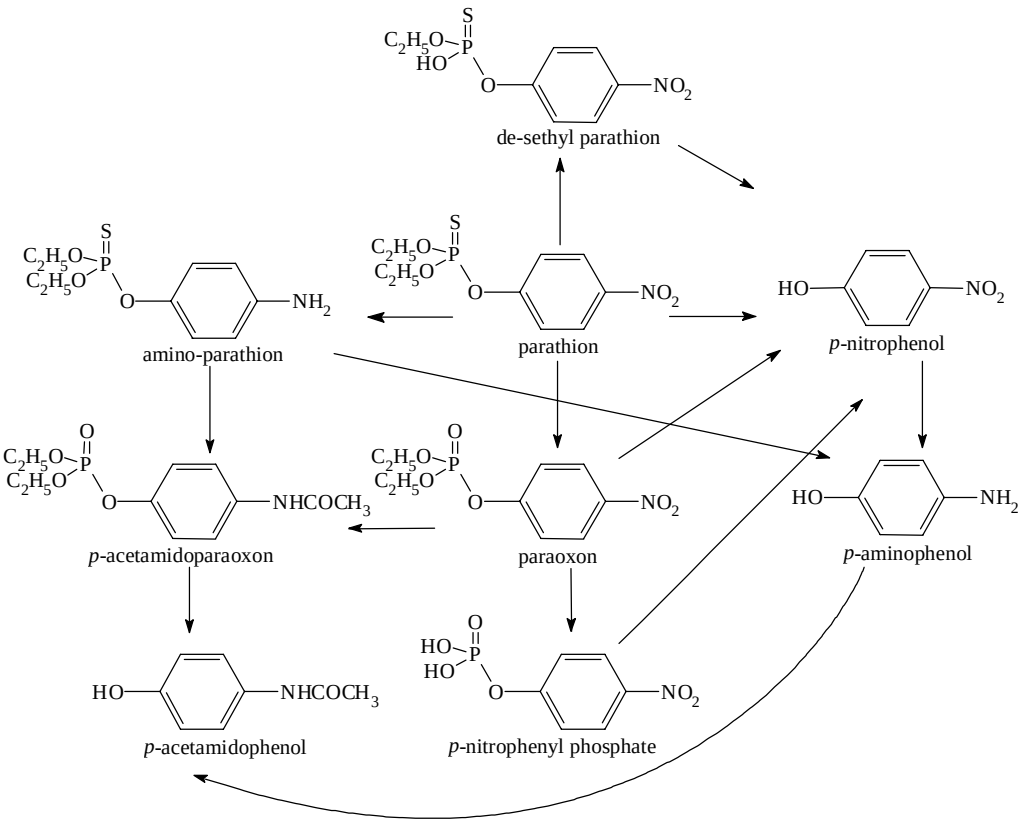



Figure 1. Proposed metabolic pathways of parathion in goats and hens.

## Plant Metabolism

The Meeting received information on the metabolism of parathion in wheat, cotton and potatoes.

Wheat plants, Marshall variety, were sprayed twice with [*phenyl*-$^{14}$C]parathion at 1.3 kg ai/ha with a 7-day interval between treatments, beginning approximately 15 days before the expected harvest (Hubert, 1988a). Samples of chaff, foliage and grain were taken 1, 3 and 7 days after each treatment and examined for $^{14}$C content (Table 5). Residues were present in the grain, but at much lower levels than in the chaff or foliage. The second spraying increased the residues from the first spraying.

Table 5. Levels of $^{14}$C in chaff, grain and foliage from wheat plants treated twice with [*phenyl*-$^{14}$C]parathion at 1.3 kg ai/ha (Hubert, 1988a).

| Day | $^{14}$C expressed as parathion, mg/kg | | | | | |
|---|---|---|---|---|---|---|
| | Chaff | | Grain | | Foliage | |
| -1 | 0.04 | ndr | ndr | 0.007 | 0.017 | ndr |
| 1st spraying | | | | | | |
| 1 | 66 | 169 | 1.4 | 5.2 | 20 | 47 |
| 3 | 86 | 107 | 1.6 | 3.7 | 24 | 36 |
| 7 | 62 | 75 | 3.4 | 5.1 | 21 | 22 |
| 2nd spraying | | | | | | |
| 8 | 164 | 178 | 3.3 | 6.0 | 70 | 79 |

| Day | $^{14}C$ expressed as parathion, mg/kg | | | | | |
|---|---|---|---|---|---|---|
| | Chaff | | Grain | | Foliage | |
| 10 | 111 | 188 | 3.0 | 9.8 | 47 | 78 |
| 14 | 234 | 318 | 6.7 | 10 | 86 | 111 |

ndr: no detectable residue

Hubert (1989) identified the components of the $^{14}C$ residue in the treated wheat plants (day 14, Table 5). Methanol/water extracts of the plant tissues were subjected to mild acid hydrolysis. The total accountable $^{14}C$ in the straw (previously termed foliage) chaff and seed was in the range of 94-99.5%, but 5.5-17% was not extracted. The composition of the residue was determined by TLC and HPLC. The compounds identified by HPLC are shown in Table 6. TLC also identified small amounts of *p*-nitrophenyl phosphate. Parathion itself constituted the main part of the residue in all the samples.

Table 6. Composition of the residue in extracts of straw, chaff and seed from wheat plants treated twice with [*phenyl*-$^{14}C$]parathion at 1.3 kg ai/ha (Hubert, 1989).

| Compound | straw | | chaff | | seed | |
|---|---|---|---|---|---|---|
| | % of $^{14}C$ | mg/kg as parathion | % of $^{14}C$ | mg/kg as parathion | % of $^{14}C$ | mg/kg as parathion |
| parathion | 51 | 66 | 62 | 197 | 65 | 6.7 |
| *p*-nitrophenol | 5.4 | 6.9 | 3.9 | 12 | 6.1 | 0.63 |
| paraoxon | 3.3 | 4.2 | 3.7 | 12 | 1.2 | 0.13 |
| *S*-phenyl parathion | 2.5 | 3.2 | 2.6 | 8.2 | 0.7 | 0.073 |
| *S*-ethyl parathion or *p*-amino-parathion | 2.2 | 2.8 | 1.4 | 4.4 | 0.7 | 0.073 |
| O-ethyl *p*-nitrophenyl phosphorothioate | 3.7 | 4.7 | 4.0 | 13 | 4.1 | 0.43 |
| *p*-nitrophenyl ß-D-glucopyranoside | 1.1 | 1.4 | 3.2 | 10 | 4.6 | 0.48 |

Sanger (1993) further examined the residue, especially the unextractable fractions in straw and grain using more vigorous solubilizing techniques. $^{14}C$ was found in protein, pectin, lignin, hemicellulose and cellulose. Small amounts of parathion and paraoxon were released from the lignin fraction of the straw. Parathion was confirmed as the major part of the residue, but some of the compounds identified as metabolites in the earlier study were not found.

Table 7. Compounds identified in grain and straw from wheat plants treated twice with [*phenyl*-$^{14}C$]parathion at 1.3 kg ai/ha (Sanger, 1993).

| Compound | $^{14}C$ expressed as parathion, mg/kg | |
|---|---|---|
| | grain | straw |
| parathion | 6.6 | 46 |
| *p*-nitrophenol | 0.79 | 13 |
| paraoxon | none | 1.2 |
| 4-acetylaminophenyl diethyl phosphate | none | 1.0 |
| *O*-ethyl p-nitrophenyl phosphorothioate | 0.027 | none |
| *p*-aminophenol | 0.086 | none |
| sugar conjugate of *p*-nitrophenol | 0.041 | 0.73 |
| complex polar metabolites (5+ TLC origin) | 0.47 | 14 |
| Total $^{14}C$ (measured) | 9.7 | 115 |

Hubert (1988b) sprayed cotton plants twice at a 7-day interval with [*phenyl*-$^{14}C$]parathion at 1.7 kg ai/ha and sampled the plants 0, 7, 14, 28 and 56 days after the first application for measurement of $^{14}C$. Residues in the seed were very low compared with levels on the leaves (Table 8).

Table 8. Levels of $^{14}C$ in cotton calyx, seed and leaves of plants sprayed twice at a 7-day interval with [*phenyl*-$^{14}C$]parathion at 1.7 kg ai/ha (Hubert, 1988b).

| Day | $^{14}C$ expressed as parathion, mg/kg | | |
|---|---|---|---|
| | calyx | seed | leaves |
| 1st spraying | | | |
| 0 | 0.84 | ndr | 24 |
| 7 | 0.59 | 0.005 | 13 |
| 2nd spraying | | | |
| 7 | 0.74 | ndr | 27 |
| 14 | 0.98 | 0.019 | 23 |
| 28 | 0.70 | 0.019 | 52 |
| 56 | 1.96 | 0.039 | 52 |

ndr: no detectable residue

Hubert (1990) extracted the cotton calyx, seed and leaves (day 14 samples, Table 8) with methanol + water and treated the remaining material with ethanolic hydrochloric acid. Compounds in the leaf and calyx extracts were identified by TLC and HPLC. Residues in the seed were too low for identification. The $^{14}C$ was accounted for by 62% extracted and 35% unextractable in the leaves, 59% extracted and 37% unextractable in the calyx and 46% extracted and 60% unextractable in the seed. Parathion was the main residue (Table 9).

Table 9. Compounds identified in the calyx and leaves of cotton plants treated twice with [*phenyl*-$^{14}C$]parathion at 1.7 kg ai/ha (Hubert, 1990). .

| Compound | $^{14}C$ expressed as parathion, mg/kg | |
|---|---|---|
| | calyx | leaves |
| parathion | 0.55 | 14 |
| *p*-nitrophenol | 0.16 | 1.4 |
| paraoxon | 0.080 | 1.2 |
| *S*-phenyl parathion | 0.077 | 1.1 |
| *S*-ethyl parathion or *p*-amino-parathion | 0.13 | 0.76 |
| *O*-ethyl *p*-nitrophenyl phosphorothioate | 0.094 | 2.5 |
| *p*-nitrophenyl ß-D-glucopyranoside | 0.043 | 0.71 |

Potato plants were sprayed twice with [*phenyl*-$^{14}C$]parathion at 3.0 kg ai/ha 15 and 30 days before harvest (Larson, 1990). At harvest most of the $^{14}C$ remained in the stems and foliage (20-31 mg/kg, n=12) with small amounts in the tubers (0.093-0.14 mg/kg, n=12).

Approximately 50% of the $^{14}C$ in the tubers was extracted with methanol + water and another 28% was released by hydrochloric acid hydrolysis. About 1% was identified by TLC as parathion and 10% as *p*-nitrophenol. Attempts at further identification were unsuccessful; the $^{14}C$ was generally in polar material and at a low level and the extracts contained substantial concentrations of endogenous compounds. Major components of the $^{14}C$ residue in the foliage and stems were parathion (8 mg/kg, 27% of the $^{14}C$), *O*-ethyl *p*-nitrophenyl phosphorothioate (5.3 mg/kg), *p*-nitrophenol (3.2 mg/kg) and *p*-aminoparathion (2.9 mg/kg). Metabolic pathways are shown in Figure 2.

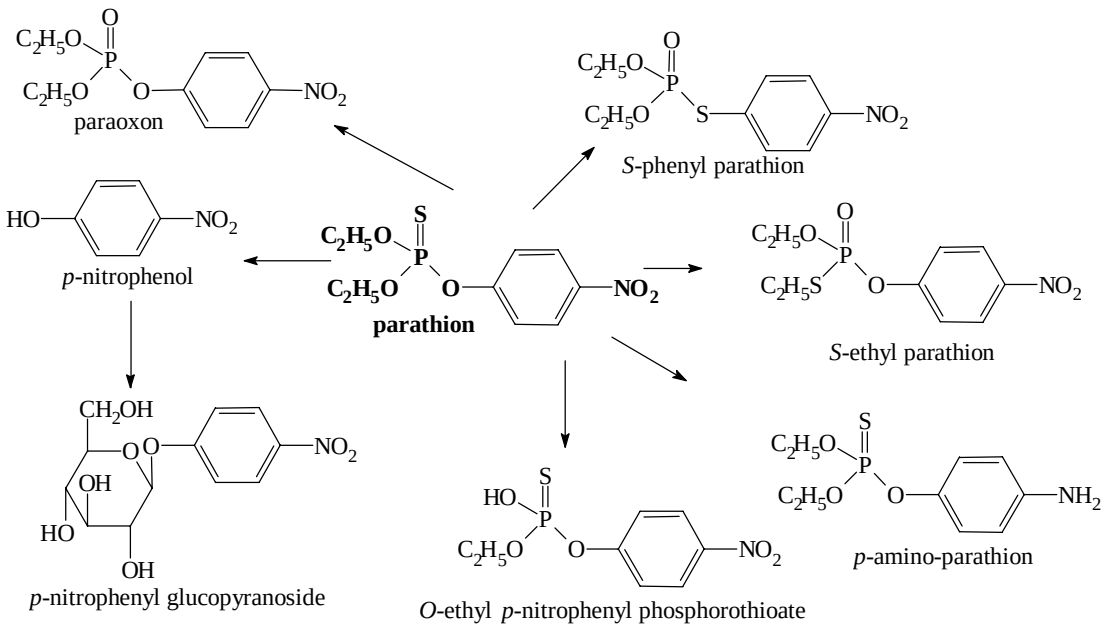



Figure 2. Metabolism of parathion by plants.

**Environmental fate in soil**

The Meeting received information on the aerobic and anaerobic degradation of parathion in soil.

Cranor (1989a) incubated [*phenyl*-$^{14}$C]parathion in the dark at 25°C in a sandy loam soil at a dose of 10 mg/kg for 12 months under aerobic conditions. The moisture in the soil was maintained at 70% field capacity. Volatiles were collected and soil samples were taken periodically for analysis. The sandy loam soil was microbiologically active with the following characteristics: 1.6% organic matter, 64% sand, 20% silt, 16% clay and pH 6.2. The total $^{14}$C recovered was in the range 97-105% in the first 6 months, decreasing to 88% at 12 months.

The half-life for the disappearance of parent parathion was 58 days. Levels of the primary metabolites paraoxon and nitrophenol always remained much less than the level of parent parathion, suggesting that parathion would normally be the main residue. After 1 month and 1 year 9.8% and 44% respectively of the residue had been mineralized. After 12 months 37% of the dose remained in the soil as unextractable material.

Table 10. Residues from aerobic incubation of [*phenyl*-$^{14}$C]parathion in the dark at 25°C in a sandy loam soil at a dose of 10 mg/kg for 12 months (Cranor, 1989a).

| Day | $^{14}$C, mg/kg expressed as parathion | | | | $CO_2$, cumulative % of dose |
|---|---|---|---|---|---|
| | parathion | *p*-nitrophenol | paraoxon | bis(4-nitrophenyl) ethyl phosphate | |
| 0 | 9.4 | 0.058 | 0.097 | 0 | 0.2 |
| 7 | 8.8 | 0.073 | 0.073 | 0 | 2.2 |
| 14 | 7.1 | 0.28 | 0.14 | 0 | 5.0 |
| 31 | | | | | 9.8 |
| 61 | 3.0 | 0.050 | 0.15 | 0.10 | 20 |
| 92 | | | | | 28 |
| 122 | 1.5 | 0.021 | 0.080 | 0.20 | 32 |
| 183 | 0.87 | 0 | 0.097 | 0.20 | 37 |
| 275 | | | | | 41 |
| 366 | 0.15 | | | | 44 |

Cranor (1990) further investigated the unextractable residues from the previous study. Soil samples were subjected to more vigorous treatments and extractions. Extracts from refluxing the bound residues with acidified aqueous acetonitrile produced traces of parent parathion. The day-10 soil sample released 0.82 mg/kg $^{14}C$ (expressed as parathion) of which 64% was parathion, while the day-275 soil released 1.1 mg/kg $^{14}C$ of which 17% was parathion. Incorporation of $^{14}C$ into humic acid (0.30 mg/kg), fulvic acid (1.8 mg/kg) and humin (0.64 mg/kg) was also demonstrated for the day 275 soil.

Cranor (1989b) incubated [*phenyl*-$^{14}C$]parathion in the dark at 25°C in the same sandy loam soil at a dose of 10 mg/kg for 12 months under anaerobic conditions. The soil had been flooded with well water and aged anaerobically for more than 30 days in preparation before dosing. Soil samples were taken periodically during the study for analysis. $^{14}C$ volatiles evolved during the incubation were negligible. The total recovered $^{14}C$ was in the range 92-110% of the dose.

The half-life for the loss of parathion calculated for the first 0.5-6 hours was 13 hours (Table 11), but became much longer after 24 hours, suggesting that part of the parathion became bound or less available for microbial attack. A small part of the dose was converted to *p*-nitrophenol and a trace of paraoxon, but a large part was converted to unextractable residues (Table 12). Bis(4-nitrophenyl) ethyl phosphate was also detected as a minor product. The $^{14}C$ in the aqueous phase was consistently less than in the soil.

Table 11. Disappearance of parathion during anaerobic incubation at 25°C in the dark of soil with an initial dose of 10 mg/kg [*phenyl*-$^{14}C$]parathion (Cranor 1989b).

| Hours after dosing | Parathion as % of dose | | |
|---|---|---|---|
| | soil extract | aqueous phase | Total |
| 0.5 | 51 | 28 | 79 |
| 1.0 | 49 | 26 | 75 |
| 2.0 | 49 | 22 | 71 |
| 3.0 | 30 | 35 | 64 |
| 4.0 | 52 | 22 | 74 |
| 6.0 | 27 | 28 | 55 |
| 24. | 21 | 12 | 33 |
| 72 | 20 | 8.4 | 28 |

Table 12. Formation of degradation products and bound residues during anaerobic incubation at 25°C in the dark of soil with an initial dose of 10 mg/kg [*phenyl*-$^{14}C$]parathion (Cranor 1989b).

| Day | Concentration expressed as % of dose | | |
|---|---|---|---|
| | *p*-nitrophenol | paraoxon | bound residues |
| 0 | 2.4 | 0 | 1.3 |
| 1 | 9.2 | 0 | 16 |
| 3 | 8.6 | 0 | 36 |
| 5 | 6.4 | 0 | 42 |
| 7 | 5.4 | 0 | 49 |
| 10 | 7.2 | 0 | 52 |
| 14 | 7.8 | 0 | 53 |
| 31 | 6.2 | 0 | 62 |
| 61 | 3.8 | 0 | 77 |
| 92 | 2.1 | 1.7 | 89 |
| 123 | 0.7 | 0.9 | 86 |
| 183 | | | 84 |
| 274 | | | 82 |

| Day | Concentration expressed as % of dose | | |
|---|---|---|---|
| | *p*-nitrophenol | paraoxon | bound residues |
| 366 | | | 76 |

Cranor (1992a) further investigated the nature of the unextractable residues from the previous study. Soil fractions were extracted under more vigorous conditions, including refluxing with acidified aqueous acetonitrile, following which the remaining soil containing the unextracted $^{14}C$ was separated into humic acid, fulvic acid and humin fractions. Small amounts of parathion were released by the vigorous extraction, but most of the released $^{14}C$ was in polar material. The $^{14}C$ in the humic acid + fulvic acid + humin fractions amounted to 28% of the dose in both day 7 and day 31 samples. The results show that $^{14}C$ was rapidly incorporated into the biomass of the soil by day 7.

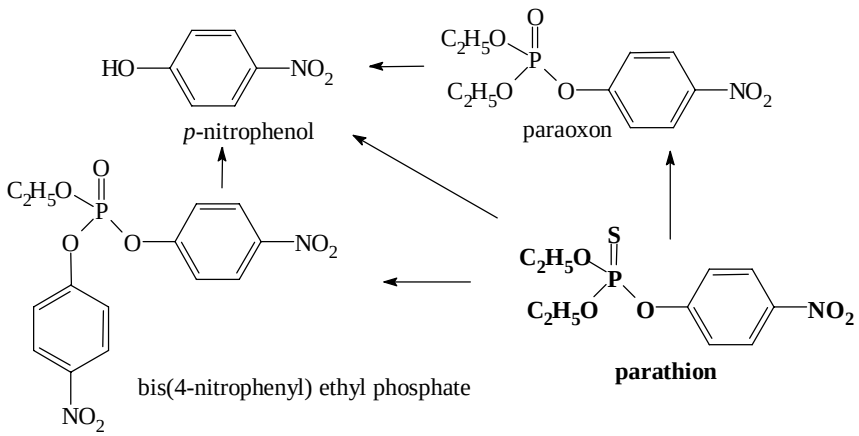



Figure 3. Degradation of parathion in soil.

**Environmental fate in water/sediment systems**

The Meeting received information on the aerobic degradation of parathion in a flooded soil.

Cranor (1989c) incubated [*phenyl*-$^{14}C$]parathion at a nominal dose of 10 mg/kg soil on a microbiologically active sandy loam soil flooded with well water under aerobic conditions in the dark at 25°C for 1 month. After 31 days parathion accounted for 2.5% of the dose, and evolved $CO_2$ for 3.0%. The sandy loam soil was characterized, as before, as 1.6% organic matter, 64% sand, 20% silt, 16% clay and pH 6.2. Total $^{14}C$ recovery was in the range 94-102%. The disposition of the residue with time is shown in Table 13.

Parathion disappeared quickly, with an initial half-life of 2.4 days (calculated on 0-14 days data). Half of the initial dose had become unextractable within 14 days. Analyses by TLC and by HPLC for *p*-nitrophenol and paraoxon differed substantially and identifications were therefore labelled as tentative.

When Cranor (1992b) re-examined the samples, HPLC analysis of samples at day 10 confirmed the identifications of parathion and *p*-nitrophenol by matching retention times with standards. A large HPLC peak in day 10 samples corresponding to 24% of the administered dose had been previously tentatively identified as paraoxon, but was shown not to be so. Identification by GC-MS was not possible, but the mass spectrum suggested a *p*-nitrophenol component. More vigorous extraction of day 10 and day 31 samples released more $^{14}C$ compounds from the unextractable

residues including traces of parathion. $^{14}C$ was shown to be incorporated into fulvic acid and humic acid.

Table 13. Parathion and degradation products from incubation of [*phenyl*-$^{14}C$]parathion at a nominal dose of 10 mg/kg of soil flooded with well water under aerobic conditions in the dark at 25°C (Cranor, 1989c).

| Day | Residue expressed as % of dose | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | extractable + aqueous residues | unextractable residues | cumulative volatiles | parathion | | *p*-nitrophenol | | paraoxon | | *O,O*-bis(4-nitrophenyl) ethyl phosphate |
| | | | | HPLC | TLC | HPLC | TLC | HPLC | TLC | HPLC |
| 0 | 94 | 0.15 | 0 | 89 | 87 | 0 | 0 | -[3] | 0 | 0 |
| 5 | 77 | 20 | 0.95 | 39 [1] | 37 | 3.2 [1] | 19 | | 0 | 0 |
| 7 | 70 | 27 | 1.4 | 2.6 [2] | 16 | 5.6 [2] | 27 | | 0 | |
| 14 | 50 | 50 | 2.3 | 0 [2] | 1.6 | 0 [2] | 23 | | 0.41 | |
| 21 | 43 | 54 | 2.6 | 0 [1] | 3.2 | 0 [1] | 15 | | 0 | 1.4 |
| 31 | 39 | 60 | 3.0 | 0.8 | 2.5 | 0.6 | 12 | | 0 | 1.6 |

[1] soil extract only
[2] water phase only
[3] original tentative HPLC identification of paraoxon could not be substantiated on re-examination

## METHODS OF RESIDUE ANALYSIS

### Analytical methods

The Meeting received information on analytical methods for determining residues of parathion, paraoxon and *p*-nitrophenol in supervised trials and suitable for enforcement.

Cassidy (1991) described such a method for a wide variety of raw agricultural and processed commodities. A solution of 100 ml of methanol + water + HCl is added to a finely divided sample of 25 g and thoroughly blended, then transferred with washings and rinsings to a round-bottom flask, refluxed gently for 30 minutes, cooled and filtered. The filtrate is transferred to a rotary evaporator and the methanol evaporated. The residual aqueous solution is treated with saturated sodium chloride and the residues extracted into ethyl acetate, which is dried with sodium sulfate and evaporated to 5-20 ml. This solution is analysed by GLC with an FPD in the P mode for parathion and paraoxon. An additional Florisil and C-18 Sep-Pak clean-up is required for *p*-nitrophenol residues before determination by HPLC with UV detection.

The LOQ was generally 0.05 mg/kg. Analytical recoveries were generally in the 80-90% range from numerous samples, many fortified at or about 0.05 mg/kg, in 3 laboratories on a wide range of substrates.

The method has been used on 39 different substrates including vegetables, nuts, forage, hay, olives, processed commodities and wheat, and tested for interferences from 230 pesticides. Eight compounds showed possible interference: dioxathion, phosphamidon, malathion and chlorpyrifos methyl had retention times close to paraoxon, and fenthion, chlorpyrifos and fensulfothion to parathion. The interference study was reported in detail by Szorik (1991).

The acid reflux extraction was introduced because the metabolism studies on wheat straw and grain demonstrated that acid released an additional 10-25% of parathion and paraoxon. Comparison of the reflux extraction with and aqueous methanol extraction of sweet peppers and celery at room

temperature did not result in significant differences, suggesting that cold neutral extraction is also generally suitable.

Sub-samples from the metabolism studies were analysed for parathion, paraoxon and *p*-nitrophenol and the results compared with levels derived from the HPLC measurements of $^{14}C$ (Table 14). The analysed sub-samples were not identical to those in the metabolism studies although from the same sampling period. The agreement for parathion is good in wheat commodities, but not in cotton leaves. The agreement for paraoxon in wheat grain is poor, but this was likely to be the result of oxidation of a small amount of parathion during storage (the wheat samples had been stored after blending) (Szorik, 1989).

Table 14. Comparison of residues found in plant metabolism studies determined by HPLC measurement of $^{14}C$ and by the analytical method of Cassidy, 1991 (Szorik, 1989).

| Sample | Residues, mg/kg | | | | | |
|---|---|---|---|---|---|---|
| | parathion | | paraoxon | | *p*-nitrophenol | |
| | $^{14}C$ HPLC | Cassidy method | $^{14}C$ HPLC | Cassidy method | $^{14}C$ HPLC | Cassidy method |
| wheat straw (forage) | 41.0 | 30.3 | 2.51 | 2.51 | 2.07 | 1.20 |
| wheat chaff | 147 | 102 | 8.25 | 16.8 | 4.39 | 2.65 |
| wheat grain | 3.97 | 4.30 | 0.07 | 0.35 | 0.18 | 0.23 |
| cotton leaves | 19.1 | 5.70 | 1.57 | | 0.92 | |
| cotton calyx | 0.27 | 0.18 | 0.031 | <0.05 | 0.032 | <0.05 |

Validation data were reported for wheat straw, grain and flour and sunflower seed oil (Sparacino, 1992). Recoveries were generally satisfactory at the levels tested: 0.05-2 mg/kg for parathion and 0.05-0.5 mg/kg for paraoxon. A confirmatory method using capillary GC-MS was also tested on wheat grain and proved satisfactory for parathion over the range tested (0.05-2 mg/kg). For paraoxon there was too much interference at 0.05 mg/kg and recoveries were 150-170% at 0.2-0.5 mg/kg.

Norby (1993a) provided validation data for a modification of the Cassidy method with an LOQ of 0.02 mg/kg for parathion and paraoxon in wheat grain, straw, forage, bran, flour, and milled samples. Lower sample weights were used for processed commodities and a capillary GC column for improved chromatography. Recoveries were generally 80-110%, with those of paraoxon at 0.02 mg/kg towards the higher end of the range.

Norby (1993b) validated the method for sorghum forage, fodder, grain, stover and flour. The LOQ for parathion and paraoxon was 0.05 mg/kg in sorghum forage and fodder and 0.02 mg/kg in the other commodities. Recoveries at spiking levels from the LOQs to 5 mg/kg were generally 80-110%.

Norby (1993c) tested the same method on canola seed, crude oil, refined oil, processing waste and meal. Recoveries at 0.02 mg/kg were often unacceptably high, so the LOQ for the 5 substrates was 0.05 mg/kg. Recoveries at spiking concentrations of 0.05-5 mg/kg were satisfactory at 74-110% for parathion and 81-116% for paraoxon.

Bower and Gillis (1996) validated a method with an LOQ of 0.01 mg/kg for residues of parathion and paraoxon in apples and grapes. Samples were extracted with acetone + water and the mixture filtered, evaporated to leave an aqueous solution, then treated with saturated sodium chloride and extracted with dichloromethane. The dichloromethane was evaporated and the residue taken up in methanol for clean-up on a C-18 column, then transferred to ethyl acetate for analysis by GLC with an FPD in the P-mode. Recoveries of parathion and paraoxon were generally 80-110% at levels of 0.01-2.0 mg/kg.

Nishioka (1996) described the Leoni method for determining residues of parathion, paraoxon and 4-acetylaminophenyl diethyl phosphate in animals and animal products. Goat liver is extracted with acetone and the filtered extract diluted, cleaned up by dichloromethane/water partition, and further purified on carbon/Celite and C-18 columns. The residues are determined by capillary GLC with an FPD in the P mode. The procedure is modified slightly for milk and fat. The LOQ for parathion and paraoxon in liver and fat was 0.05 mg/kg, and for 4-acetylaminophenyl diethyl phosphate 0.1 mg/kg. Residues in the tissues and milk from the goat and hen metabolism studies determined by $^{14}C$ measurement and the Leoni analytical method were in good agreement (Table 15).

Table 15. Residues from metabolism studies on goats and hens determined by radiotracer and Leoni methods (Nishioka, 1996).

| Compound | Residue, mg/kg | | | | | |
|---|---|---|---|---|---|---|
| | goat liver | | goat milk | | hen fat | |
| | $^{14}C$ HPLC | Leoni | $^{14}C$ HPLC | Leoni | $^{14}C$ HPLC | Leoni |
| parathion | <0.05 | <0.05 | <0.05 | <0.05 | 0.138 | 0.122 |
| paraoxon | <0.05 | <0.05 | | | | |
| 4-acetylaminophenyl diethyl phosphate | 0.848 | 0.730 | 0.360 | 0.390 | | |

Williams (1998) used a similar method for determining residues in milk and kidneys. The LOQs for parathion and paraoxon were 0.001 mg/kg in milk, and recoveries from milk spiked at 0.001 and 0.002 mg/kg were 83-96% (n=12) for parathion, 80-102% (n=12) for paraoxon and 17-90% (n=12) for 4-acetylaminophenyl diethyl phosphate. Two milk samples contained small amounts of 4-acetylaminophenyl diethyl phosphate, which affected the recoveries at 0.001 mg/kg (subtraction of control values from measured recoveries) and an LOQ of 0.001 or 0.002 was not demonstrated. The LOQ was 0.01 mg/kg for kidney, with recoveries from samples spiked at 0.01 and 0.02 mg/kg of 87-117% for parathion (n=4), 73-98% (n=4) for paraoxon and 71-91% (n=4) for 4-acetylaminophenyl diethyl phosphate.

Parathion is included in the multiresidue GLC analytical method for non-fatty and fatty foods published in the Official Methods of Analysis in The Netherlands (Netherlands, 1996). Detection is with an ion-trap or nitrogen-phosphorus detector. The LOQ is 0.05 mg/kg.

**Stability of pesticide residues in stored analytical samples**

The Meeting received data on the stability of residues in snap beans, kidney beans, cotton seed, strawberries, plums, apples, sunflower seed, almond kernels, spinach, green peppers, oranges, clover, canola seed, crude canola oil, canola meal, canola processing waste, sorghum flour, maize and processed maize commodities stored frozen.

Keller (1992) stored ground 25 g samples of snap beans, kidney beans and cotton seed, fortified at 1 mg/kg with parathion, paraoxon and *p*-nitrophenol, at approximately -20°C for up to 2 years. (The *p*-nitrophenol stability was tested but is not reported). Procedural recoveries validated the analytical results at each sampling interval. The results are shown in Table 16. The residues were stable except those of paraoxon in snap beans which had decreased substantially in samples stored for 12 months.

Table 16. Stability of parathion and paraoxon in snap beans, kidney beans and cotton seed fortified at 1 mg/kg and stored at approximately -20°C (Keller, 1992). The % remaining is not adjusted for procedural recoveries.

| Months storage | Snap beans | | | | Kidney beans | | | | Cotton seed | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | parathion | | paraoxon | | parathion | | paraoxon | | parathion | | paraoxon | |
| | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov |
| 0 | 80 | 75 | 74 | 74 | 89 | 70 | 89 | 82 | 80 | 80 | 92 | 97 |
| 1 | 90 | 100 | 97 | 113 | 88 | 89 | 95 | 93 | 86 | 84 | 92 | 91 |
| 2 | 80 | 90 | 84 | 94 | 80 | 76 | 87 | 87 | 84 | 87 | 98 | 98 |
| 3 | 102 | 100 | 109 | 112 | 77 | 75 | 80 | 79 | 85 | 90 | 104 | 109 |
| 4 | 90 | 102 | 74 | 106 | 82 | 77 | 88 | 88 | 78 | 80 | 93 | 98 |
| 6 | 89 | 91 | 121 | 115 | 80 | 95 | 83 | 112 | 98 | 91 | 67 | 97 |
| 12 | 96 | 93 | 31 | 98 | 96 | 96 | 95 | 103 | 96 | 95 | 102 | 105 |
| 18 | 87 | 94 | 26 | 102 | 96 | 96 | 99 | 106 | 74 | 80 | 104 | 104 |
| 24 | 84 | 92 | 29 | 99 | 79 | 84 | 78 | 94 | 73 | 72 | 88 | 90 |

Price (1991) tested the stability of parathion, paraoxon and *p*-nitrophenol added together at 1 mg/kg to ground samples of a number of commodities in 50 g lots and stored at approximately -20°C for up to 24 months. A decrease of less than 30% would not be distinguished from the variability of the analytical method. Parathion in almond kernels and oranges and paraoxon in spinach decreased to slightly below 70% of the initial level. Decreases of parathion may obscure losses of paraoxon if parathion has been converted to paraoxon. The results of the analyses for parathion and paraoxon are shown in Table 17.

Table 17. Stability of parathion and paraoxon in strawberry, plum, apple, sunflower seeds, almond kernels, spinach, green peppers, orange and clover fortified at 1 mg/kg and stored at approximately –20°C (Price, 1991). The % remaining is not adjusted for procedural recoveries.

| Months | parathion | | paraoxon | | parathion | | paraoxon | | parathion | | paraoxon | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov |
| | Strawberry | | | | Plum | | | | Apple | | | |
| 0 | 110 | 96 | 101 | 90 | 104 | 98 | 94 | 87 | 105 | 103 | 80 | 79 |
| 1 | 104 | 106 | 93 | 95 | 105 | 108 | 93 | 95 | 102 | 104 | 83 | 84 |
| 2 | 103 | 110 | 96 | 106 | 108 | 111 | 107 | 105 | 98 | 103 | 84 | 92 |
| 3 | 100 | 104 | 100 | 103 | 101 | 104 | 98 | 100 | 91 | 95 | 83 | 89 |
| 4 | 98 | 97 | 97 | 91 | 94 | 90 | 88 | 84 | 98 | 107 | 88 | 97 |
| 6 | 105 | 96 | 98 | 104 | 105 | 100 | 110 | 110 | 94 | 96 | 85 | 97 |
| 12 | 92 | 98 | 76 | 89 | 110 | 96 | 95 | 90 | 89 | 99 | 64 | 81 |
| 18 | 85 | 87 | 90 | 98 | 84 | 92 | 99 | 110 | 90 | 92 | 96 | 95 |
| 24 | 79 | 80 | 96 | 102 | 87 | 86 | 103 | 116 | 85 | 72 | 105 | 100 |
| | Sunflower seed | | | | Almond kernel | | | | Spinach | | | |
| 0 | 92 | 95 | 94 | 105 | 82 | 87 | 91 | 92 | 98 | 83 | 107 | 89 |
| 1 | 86 | 88 | 89 | 103 | 74 | 87 | 89 | 92 | 84 | 96 | 98 | 106 |
| 2 | 84 | 93 | 107 | 119 | 74 | 72 | 87 | 101 | 77 | 84 | 91 | 106 |
| 3 | 74 | 80 | 86 | 100 | 74 | 84 | 98 | 94 | 87 | 94 | 89 | 100 |
| 4 | 73 | 74 | 80 | 81 | 74 | 77 | 86 | 83 | 97 | 85 | 82 | 101 |
| 6 | 84 | 82 | 84 | 97 | 66 | 79 | 79 | 90 | 91 | 95 | 78 | 103 |
| 12 | 75 | 85 | 73 | 89 | 75 | 84 | 87 | 88 | 100 | 93 | 67 | 89 |
| 18 | 82 | 79 | 94 | 91 | 68 | 84 | 110 | 115 | 60 | 81 | 84 | 113 |
| 24 | 74 | 96 | 92 | 117 | 65 | 87 | 100 | 111 | 95 | 100 | 72 | 118 |
| | Green peppers | | | | Orange | | | | Clover | | | |
| 0 | 86 | 77 | 84 | 80 | 103 | 99 | 99 | 95 | 81 | 85 | 110 | 101 |
| 1 | 75 | 84 | 78 | 98 | 90 | 96 | 94 | 96 | 88 | 87 | 103 | 104 |

| Months | parathion | | paraoxon | | parathion | | paraoxon | | parathion | | paraoxon | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov |
| 2 | 81 | 95 | 89 | 101 | 83 | 89 | 92 | 98 | 88 | 74 | 114 | 106 |
| 3 | 88 | 84 | 90 | 96 | 82 | 82 | 98 | 95 | 84 | 82 | 81 | 106 |
| 4 | 87 | 89 | 73 | 94 | 80 | 81 | 78 | 83 | 95 | 85 | 95 | 113 |
| 6 | 82 | 98 | 66 | 92 | 85 | 74 | 93 | 91 | 88 | 87 | 85 | 102 |
| 12 | 84 | 94 | 45 | 80 | 92 | 93 | 80 | 84 | 99 | 100 | 82 | 105 |
| 18 | 93 | 82 | 69 | 89 | 71 | 72 | 100 | 104 | 105 | 104 | 73 | 98 |
| 24 | 74 | 80 | 83 | 85 | 68 | 87 | 80 | 114 | 89 | 92 | 77 | 102 |

Owen (1995) fortified homogenized canola seed and its processed commodities with parathion, paraoxon, parathion-methyl and paraoxon-methyl, and sorghum flour with parathion and paraoxon, all samples with 1 mg/kg of each compound. The analyses for parathion-methyl and paraoxon-methyl are not reported here. Sub-samples of 10 g each were stored in individual 250 ml bottles at -5°C, and duplicate bottles were withdrawn at intervals for analysis (Table 18). Parathion residues were generally stable for the duration of the study except in canola meal, where the stability was marginal. Paraoxon had decreased after 6 and 14 months in canola crude oil where recoveries were poor at 6 months and divergent duplicates of 38% and 108% occurred in the 14-month samples.

Table 18. Stability testing of parathion and paraoxon in canola commodities and sorghum flour fortified at 1 mg/kg and stored at -5°C (Owen, 1995). The reported % remaining is not adjusted for procedural recoveries.

| Storage period | parathion | | paraoxon | | parathion | | paraoxon | | parathion | | paraoxon | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov |
| | Canola seed | | | | Canola crude oil | | | | Canola meal | | | |
| 0 | 77 | 80 | 82 | 85 | 88 | 89 | 95 | 96 | 80 | 78 | 90 | 86 |
| 30 days | 96 | 93 | 99 | 105 | 87 | 89 | 101 | 104 | 87 | 85 | 100 | 105 |
| 3 months | 89 | 91 | 82 | 96 | 75 | 79 | 75 | 79 | 79 | 81 | 84 | 86 |
| 5 months | 87 | 92 | 87 | 100 | 85 | 95 | 81 | 87 | 99 | 97 | 111 | 103 |
| 6 months | 82 | 86 | 72 | 87 | 76 | 84 | 69 | 72 | 76 | 84 | 77 | 86 |
| 14 months | 77 | 88 | 51 | 76 | 82 | 85 | 38 108 | 58 | 67 | 89 | 67 | 92 |
| | Canola processing waste | | | | Sorghum flour | | | | | | | |
| 0 | 84 | 80 | 77 | 75 | 94 | 99 | 95 | 99 | | | | |
| 30 days | 87 | 89 | 80 | 89 | 93 | 96 | 91 | 96 | | | | |
| 3 months | 81 | 84 | 81 | 86 | 87 | 91 | 86 | 96 | | | | |
| 5 months | 83 | 89 | 69 | 74 | | | | | | | | |
| 6 months | 83 | 84 | 76 | 82 | 83 | 91 | 80 | 88 | | | | |
| 12 months | | | | | 104 | 110 | 102 | 110 | | | | |
| 14 months | 86 | 94 | 84 | 104 | | | | | | | | |
| 19 months | | | | | 94 | 90 | 92 | 101 | | | | |

McKinney and Crotts (1998) tested the stability of residues of parathion and paraoxon in untreated control samples from a maize processing study. Well-mixed samples (10 g for grain, grits and meal, 2 g for oil, 25 g for starch and flour) were weighed into glass jars, individually and separately fortified with parathion and paraoxon at 0.10 mg/kg, and stored at or below -10°C. The initial day zero and procedural recovery values are the means of the same 3 analyses. At other times the % remaining value is the mean result from duplicate samples and the procedural recovery is determined at 0.10 mg/kg.

Residues of parathion and paraoxon in maize commodities were stable during 4 months of freezer storage, but a decrease of less than 30% would not have been evident at 0.1 mg/kg.

Table 19. Stability of parathion and paraoxon in maize commodities fortified at 0.1 mg/kg and stored at -10°C (McKinney and Crotts, 1998). The reported % remaining is not adjusted for procedural recoveries.

| Storage period | parathion | | paraoxon | | parathion | | paraoxon | | parathion | | paraoxon | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov | % remain | % proced recov |
| | Maize grain | | | | Maize flour | | | | Maize starch | | | |
| 0 day | 73 | 73 | 81 | 81 | 98 | 98 | 97 | 97 | 95 | 95 | 109 | 109 |
| 1 month | 68 | 68 | 77 | 75 | 108 | 109 | 112 | 118 | 97 | 100 | 100 | 94 |
| 2 months | 76 | 70 | 75 | 84 | 90 | 92 | 99 | 92 | 96 | 95 | 100 | 100 |
| 4 months | 90 | 90 | 73 | 97 | 97 | 106 | 78 | 105 | 78 | 77 | 87 | 99 |
| | Maize oil | | | | Corn grits | | | | Maize meal | | | |
| 0 day | 102 | 102 | 97 | 97 | 79 | 79 | 80 | 80 | 72 | 72 | 85 | 85 |
| 1 month | 102 | 107 | 103 | 117 | 68 | 77 | 76 | 93 | 73 | 79 | 73 | 84 |
| 2 months | 85 | 89 | 93 | 96 | 72 | 82 | 69 | 88 | 68 | 73 | 68 | 81 |
| 4 months | 103 | 106 | 101 | 111 | 77 | 79 | 68 | 89 | 84 | 72 | 62 | 74 |

**Definition of the residue**

Parathion and paraoxon are the main components. Parathion is the major part of the residue at shorter intervals and at the higher levels. At low levels in some circumstances paraoxon may constitute a significant proportion of the residue. There were 227 cases where both parathion and paraoxon levels exceeded the LOQ in trials according to GAP on food and feed commodities. There was generally a close relation between the combined residue level and the parathion level.

The Meeting recommended that the definition of the residue for compliance with MRLs should continue to be parathion and for the estimation of dietary intake the sum of parathion and paraoxon expressed as parathion.

The log $P_{ow}$ of 3.2 and the results of the animal metabolism studies suggest that parathion is at the borderline of fat-solubility. In the goat metabolism study parathion residues in the renal fat, 0.15 mg/kg, were substantially higher than in the muscles, 0.019 mg/kg, but were 0.56 mg/kg in the liver and 0.48 mg/kg in kidney.

The Meeting recommended that parathion should be described as fat-soluble.

**USE PATTERN**

Parathion is an anti-cholinesterase compound used as a non-systemic insecticide and acaricide to control sucking and chewing insects in a wide range of agricultural and horticultural crops.

Information on registered uses was reported to the Meeting and is shown in Table 20.

Table 20. Registered uses of parathion.

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method[1] | Rate, kg ai/ha | Spray conc. kg ai/hl | Number | |
| Agricultural and horticultural crops | Netherlands | EC, GR | soil treatment in glasshouse | 0.8-5.0 | | 1 | note[1] |
| Alfalfa | Germany | EC | foliar | 0.11 | 0.018 | 2 | 21 |
| Alfalfa | USA | EC 960 g/l | foliar ( | 0.28-0.56 | | | 15 |
| Almond | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Apple[2] | France | | foliar | | 0.019-0.025 | | 14 |
| Apricot | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Artichoke | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Artichoke | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Asparagus | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Asparagus | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Barley | Germany | EC | bait | 0.051-0.15 | | 2 | |
| Barley | Germany | EC | cutworm bait | 0.10 | | 1 | 28 |
| Barley | Germany | EC | foliar | 0.11 | 0.018-0.028 | 1 | 21 |
| Barley | Germany | EC | spray | 0.15-0.23 | 0.025-0.038 | 2 | note[3] |
| Barley | USA | EC 960 g/l | foliar ( | 0.28-0.84 | | | 15[4] |
| Broad beans | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Broad beans | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Broccoli | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Broccoli | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Brussels sprouts | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Brussels sprouts | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Bulb vegetables | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Bulb vegetables | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Cabbage | France | EC 100 g/l | foliar | | 0.02 | | 15 |
| Cabbage | France | EC 30 g/l | foliar | | 0.02 | | 15 |
| Cabbage | France | EC 30 g/l + oil | foliar | 0.23 | | | 15 |
| Cabbage | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Cabbage | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Cauliflower | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Cauliflower | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Cereals | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Cereals | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Cherries | France | EC 93 g/l | dormant spray | | 0.047 | | |
| Cherries | France | EC 30 g/l + oil | foliar | | 0.023 | | 15 |
| Cherries | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Chinese cabbage | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Chinese cabbage | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Citrus fruit | France | EC 93 g/l | foliar | | 0.023 | | 15 |
| Citrus fruits | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Citrus fruits | Italy | EC 185 g/l | foliar | | 0.02-0.04 | | 20 |
| Citrus fruits | Italy | EC 185 g/l | foliar + mineral oil | | 0.02-0.04 | | 30 |
| Citrus fruits | Spain | EC 30 g/l | winter spray | | 0.022-0.06 | | |
| Citrus fruits | Spain | EC 50 g/l | winter spray | | 0.05 | | |
| Citrus fruits | Uruguay | CS 560 g/l | foliar + 1% oil | | 0.067-0.078 | | 15 |

[1] Before planting or sowing or at an early stage of crop development.

[2] Information from Index Phytosanitaire ACTA, 36th edition.

[3] Barley, oats, rye, triticale, wheat. Treatment at beginning of infestation, autumn and spring.

[4] Application rate 0.11 kg ai/ha for plants <50 cm. Application rate 0.16 kg ai/ha for plants >50 cm.

[5] Wheat, barley. Do not apply within 15 days of harvest, cutting or forage use.

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method[1] | Rate, kg ai/ha | Spray conc. kg ai/hl | Number | |
| Climbing French beans | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Climbing French beans | Germany | EC | foliar[1] | 0.11-0.21 | 0.018 | 2 | 14 |
| Clover | Germany | EC | foliar | 0.11 | 0.018 | 2 | 21 |
| Cotton | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Cotton | USA | EC 960 g/l | foliar ( | 0.28-1.1 | | | 7[2] |
| Crucifers | France | EC 100 g/l | foliar | 0.30 | | | 15 |
| Cucumbers | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Cucumbers | Germany | EC | foliar | 0.11 | 0.018 | 2 | 14 |
| Currants | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Currants | Germany | EC | foliar | 0.26 | 0.017 | 2 | 14 |
| Dwarf French beans | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Dwarf French beans | Germany | EC | foliar | 0.11 | 0.012-0.018 | 2 | 14 |
| Figs | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Fodder beet | Germany | EC | bait | 0.051-0.15 | | 2 | |
| Fodder beet | Germany | EC | foliar | 0.11-0.18 | 0.027-0.044 | 2 | 28 |
| Fruit trees | France | EC 100 g/l | dormant spray | | 0.15 | | |
| Fruiting vegetables | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Fruiting vegetables | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Garden peas | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Garden peas | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Gooseberries | Germany | EC | foliar | 0.26 | 0.017 | 2 | 14 |
| Grapes | France | EC 30 g/l + oil | dormant spray | | 0.045 | | |
| Grapes | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Grapes | Italy | EC 185 g/l | foliar | | 0.02-0.04 | | 20 |
| Grapes | Italy | EC 185 g/l | foliar + mineral oil | | 0.02-0.04 | | 30 |
| Grapes | Spain | EC 30 g/l | winter spray | | 0.022-0.06 | | |
| Grapes | Spain | EC 50 g/l | winter spray | | 0.05 | | |
| Grapes [2] | France | | foliar | | 0.020 | | 35 |
| Kale | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Kale | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Kohlrabi | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Kohlrabi | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Leafy vegetables | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Leafy vegetables | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Leek | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Leek | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Legume animal feeds | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Lettuce | France | EC 100 g/l | foliar | | 0.02 | | 15 |
| Lettuce | France | EC 30 g/l | foliar | | 0.02 | | 15 |
| Lettuce | France | EC 30 g/l + oil | foliar | 0.23 | | | 15 |
| Lettuce | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Lettuce | Germany | EC | foliar | 0.11 | 0.018 | 2 | 14 |
| Lupin | Germany | EC | foliar | 0.11 | 0.018 | 1 | 21 |
| Maize | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Maize | USA | EC 960 g/l | foliar ( | 0.28-0.84 | | | 12[3] |
| Oats | Germany | EC | bait | 0.051-0.15 | | 2 | |

[1] Application rate 0.11 kg ai/ha for plants <50 cm, 0.16 kg ai/ha for 50-125 cm, 0.21 kg ai/ha for >125 cm.

[2] 15 days PHI if cotton trash is to be used for animal feed.

[3] Maize. Do not apply within 12 days of harvest, cutting or forage use.

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method[1] | Rate, kg ai/ha | Spray conc. kg ai/hl | Number | |
| Oats | Germany | EC | cutworm bait | 0.10 | | 1 | 28 |
| Oats | Germany | EC | foliar | 0.11 | 0.018-0.028 | 1 | 21 |
| Oats | Germany | EC | spray | 0.15-0.23 | 0.025-0.038 | 2 | note[31] |
| Oats | Uruguay | CS 560 g/l | foliar | 0.19-0.24 | | | 42 |
| Oilseed | France | EC 100 g/l | foliar | 0.20 | | | 15 |
| Oilseed rape | France | EC 30 g/l | foliar | 0.21-0.30 | | | 15 |
| Oilseed rape | France | EC 30 g/l + oil | foliar | 0.21-0.30 | | | 15 |
| Oilseed rape | France | EC 93 g/l | foliar | 0.19-0.28 | | | 15 |
| Oilseed rape | USA | EC 960 g/l | foliar ( | 0.56[2] | | | 28[3] |
| Olive | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 60 |
| Olive | Italy | EC 185 g/l | foliar | | 0.02-0.04 | | 20 |
| Olive | Italy | EC 185 g/l | foliar + mineral oil | | 0.02-0.04 | | 30 |
| Pasture | Germany | EC | bait | 0.075-0.15 | | 2 | 28 |
| Pasture | Germany | EC | spraying | 0.15-0.23 | 0.025-0.038 | 2 | 28 |
| Peach | France | EC 93 g/l | dormant spray | | 0.047 | | |
| Peach | France | EC 100 g/l | foliar | | 0.020-0.025 | | 15 |
| Peach | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Peach | France | EC 30 g/l | foliar | | 0.025 | | 15 |
| Peach | France | EC 30 g/l + oil | foliar | | 0.023 | | 15 |
| Peas | France | EC 100 g/l | foliar | | 0.02 | | 15 |
| Peas | France | EC 30 g/l | foliar | | 0.02 | | 15 |
| Peas | France | EC 30 g/l + oil | foliar | 0.23 | | | 15 |
| Plums | France | EC 93 g/l | dormant spray | | 0.047 | | |
| Plums | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Pome fruits | France | EC 30 g/l | dormant spray | | 0.05 | | |
| Pome fruits | France | EC 30 g/l + oil | dormant spray | | 0.045 | | |
| Pome fruits | France | EC 93 g/l | dormant spray | | 0.047 | | |
| Pome fruits | France | EC 100 g/l | foliar | | 0.020-0.025 | | 15 |
| Pome fruits | France | EC 30 g/l | foliar | | 0.025 | | 15 |
| Pome fruits | France | EC 30 g/l + oil | foliar | | 0.023 | | 15 |
| Pome fruits | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Pome fruits | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Pome fruits | Italy | EC 185 g/l | foliar | | 0.02-0.04 | | 20 |
| Pome fruits | Italy | EC 185 g/l | foliar + mineral oil | | 0.02-0.04 | | 30 |
| Pome fruits | Spain | EC 30 g/l | winter spray | | 0.022-0.06 | | |
| Pome fruits | Spain | EC 50 g/l | winter spray | | 0.05 | | |
| Pome fruits | Uruguay | CS 560 g/l | foliar | | 0.034-0.078 | | 15 |
| Potato | Germany | EC | foliar | 0.18-0.27 | 0.045-0.068 | 3 | 21 |
| Potato | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Potato | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Potato | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Potato | Uruguay | CS 560 g/l | foliar | 0.28-0.49 | | | 15 |
| Rape | Germany | EC | bait | 0.051-0.15 | | 2 | |
| Rape | Germany | EC | foliar | 0.11 | 0.018 | 2 | |
| Red cabbage | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Red cabbage | Germany | EC | foliar[54] | 0.11-0.16 | 0.018 | 2 | 14 |

[1] Barley, oats, rye, triticale, wheat. Treatment at beginning of infestation, autumn and spring.

[2] Spray volume 30-100 l/ha

[3] Rapeseed. Do not graze treated fields or feed treated forage or threshing waste to livestock.

[4] Application rate 0.11 kg ai/ha for plants <50 cm, 0.16 kg ai/ha for 50-125 cm, 0.21 kg ai/ha for >125 cm.

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method[1] | Rate, kg ai/ha | Spray conc. kg ai/hl | Number | |
| Root vegetables | Italy | EC 185 g/l | foliar | | 0.03-0.04 | | 20 |
| Root vegetables | Italy | EC 185 g/l | foliar + mineral oil | | 0.03-0.04 | | 30 |
| Rye | Germany | EC | bait | 0.051-0.15 | | 2 | |
| Rye | Germany | EC | cutworm bait | 0.10 | | 1 | 28 |
| Rye | Germany | EC | foliar | 0.11 | 0.018-0.028 | 1 | 21 |
| Rye | Germany | EC | spray | 0.15-0.23 | 0.025-0.038 | 2 | note[1] |
| Savoy cabbage | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Savoy cabbage | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Sorghum | USA | EC 960 g/l | foliar ( | 0.28-1.1 | | | 12[2] |
| Soybean | USA | EC 960 g/l | foliar ( | 0.28-0.84 | | | 20[3] |
| Spinach | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Spinach | Germany | EC | foliar | 0.11 | 0.018 | 2 | 14 |
| Stone fruits | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Stone fruits | Italy | EC 185 g/l | foliar | | 0.02-0.04 | | 20 |
| Stone fruits | Italy | EC 185 g/l | foliar + mineral oil | | 0.02-0.04 | | 30 |
| Stone fruits | Spain | EC 30 g/l | winter spray | | 0.022-0.06 | | |
| Stone fruits | Spain | EC 50 g/l | winter spray | | 0.05 | | |
| Stone fruits | Uruguay | CS 560 g/l | foliar | | 0.034-0.078 | | 15 |
| Strawberry | Germany | EC | foliar | 0.36 | 0.018 | 1 | note[4] |
| Strawberry | Italy | EC 185 g/l | foliar | | 0.02-0.04 | | 20 |
| Strawberry | Italy | EC 185 g/l | foliar + mineral oil | | 0.02-0.04 | | 30 |
| Sugar beet | France | EC 100 g/l | foliar | 0.20 | | | 15 |
| Sugar beet | France | EC 30 g/l | foliar | 0.225 | | | 15 |
| Sugar beet | France | EC 30 g/l + oil | foliar | 0.23 | | | 15 |
| Sugar beet | France | EC 93 g/l | foliar | 0.14 | | | 15 |
| Sugar beet | Germany | EC | bait | 0.051-0.15 | | 2 | |
| Sugar beet | Germany | EC | foliar | 0.11-0.18 | 0.027-0.044 | 2 | 28 |
| Sugar beet | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Sugar beet | Italy | EC 185 g/l | foliar | | 0.02 | | |
| Sunflower | USA | EC 960 g/l | foliar ( | 0.56-1.1 | | | 30 |
| Sweet corn | USA | EC 960 g/l | foliar ( | 0.28-0.84 | | | 12 |
| Tomato | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| Tomato | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Tomato | Uruguay | CS 560 g/l | foliar | | 0.039 | | 15 |
| Tree nuts | France | EC 93 g/l | foliar | | 0.019 | | 15 |
| Triticale | Germany | EC | bait | 0.051-0.15 | | 2 | |
| Triticale | Germany | EC | cutworm bait | 0.10 | | 1 | 28 |
| Triticale | Germany | EC | foliar | 0.11 | 0.018-0.028 | 1 | 21 |
| Triticale | Germany | EC | spray | 0.15-0.23 | 0.025-0.038 | 2 | note[5] |
| Turnips | Germany | EC | foliar | 0.11 | 0.018 | 1 | 21 |
| Vegetables | Greece | EC 200 g/l | foliar | | 0.016-0.024 | | 14 |
| Vines | France | EC 100 g/l | dormant spray | | 0.15 | | |
| Vines | France | EC 30 g/l | dormant spray | | 0.05 | | |
| Vines | France | EC 93 g/l | dormant spray | | 0.047 | | |
| Vines | France | EC 100 g/l | foliar | | 0.020-0.025 | | 15 |
| Wheat | Germany | EC | bait | 0.051-0.15 | | 2 | |
| Wheat | Germany | EC | cutworm bait | 0.10 | | 1 | 28 |
| Wheat | Germany | EC | foliar | 0.11 | 0.018-0.028 | 1 | 21 |

[1] Barley, oats, rye, triticale, wheat. Treatment at beginning of infestation, autumn and spring.

[2] Sorghum. Do not apply within 12 days of harvest, cutting or forage use.

[3] Soya bean. Do not apply within 20 days of harvest, cutting or forage use.

[4] Strawberries, use at beginning of infestation (biting insects) before flowering or after harvest.

[5] Barley, oats, rye, triticale, wheat. Treatment at beginning of infestation, autumn and spring.

| Crop | Country | Form. | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method[1] | Rate, kg ai/ha | Spray conc. kg ai/hl | Number | |
| Wheat | Germany | EC | spray | 0.15-0.23 | 0.025-0.038 | 2 | note[1] |
| Wheat | Uruguay | CS 560 g/l | foliar | 0.19-0.24 | | | 42 |
| Wheat | USA | EC 960 g/l | foliar ( | 0.28-0.84 | | | 15[2] |
| White cabbage | Germany | EC | cutworm bait | 0.10 | | 1 | 14 |
| White cabbage | Germany | EC | foliar[5] | 0.11-0.16 | 0.018 | 2 | 14 |
| Wine grapes | Germany | EC | foliar | 0.03-0.06 | 0.0075 | 1 | note[3] |

( aerial application. The US label states that parathion may only be applied aerially and only by a certified commercial applicator.

## RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on the following supervised field trials.

| | | |
|---|---|---|
| Fruits | Table 22. | Grapefruit, lemon, orange |
| | Table 23. | Apples |
| | Table 24. | Pears |
| | Table 25. | Apricots |
| | Table 26. | Cherries |
| | Table 27. | Plums and prunes |
| | Table 28. | Blackberries |
| | Table 29. | Grapes |
| | Table 30. | Strawberries |
| | Table 31. | Olives |
| Vegetables | Table 32. | Garlic |
| | Table 33. | Onions |
| | Table 34. | Broccoli |
| | Table 35. | Cabbage |
| | Table 36. | Peppers |
| | Table 37. | Sweet corn |
| | Table 38. | Tomatoes |
| | Table 39. | Field peas |
| | Table 40. | Kale |
| | Table 41. | Lettuce |
| | Table 42. | Spinach |
| | Table 43. | Snap beans |
| | Table 44. | Dry beans |
| | Table 45. | Soya beans |
| | Table 46. | Carrots |
| | Table 47. | Potatoes |
| | Table 48. | Radish |
| | Table 49. | Sugar beet |
| | Table 50. | Turnips |
| | Table 51. | Celery |
| Tree nuts | Table 52. | Almonds |
| | Table 53. | Pecans, walnuts |

[1] Barley, oats, rye, triticale, wheat. Treatment at beginning of infestation, autumn and spring.

[2] Wheat, barley. Do not apply within 15 days of harvest, cutting or forage use.

[3] Wine grapes, use at beginning of infestation (grape berry moth) at growth stages 12-55.

| | | |
|---|---|---|
| Cereals | Table 54. | Barley |
| | Table 55. | Maize |
| | Table 56. | Rice |
| | Table 57. | Sorghum |
| | Table 58. | Wheat |
| Oilseeds | Table 59. | Canola |
| | Table 60. | Cotton seed |
| | Table 61. | Sunflower seed |
| Feeds | Table 62. | Barley hay and straw |
| | Table 63. | Maize forage and fodder |
| | Table 64. | Rice straw |
| | Table 65. | Sorghum hay, forage and stover |
| | Table 66. | Wheat hay, straw and forage |
| | Table 67. | Alfalfa forage, hay |
| | Table 68. | Clover |
| | Table 69. | Bean forage and vines |
| | Table 70. | Field pea forage and vines |
| | Table 71. | Soya bean hay |
| | Table 72. | Almond hulls |
| | Table 73. | Cotton gin trash |
| | Table 74. | Sugar beet fodder |
| | Table 75. | Sunflower forage |

Trials were generally well documented with full laboratory and field reports. Laboratory reports included method validation with batch recoveries at spiking levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of sample storage, data on sprayers and their calibration, plot sizes, sample sizes and sampling dates were reported.

Where residues were not detected, results are recorded in the Tables as below the limit of quantification (LOQ) e.g. <0.01 mg/kg. Residues, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOQ, to 1 significant figure. Although trials included control plots, no control data are included in the Tables except where residues in control samples exceeded the LOQ. The results are not adjusted for analytical recoveries.

Some results of trials on alfalfa, broccoli, cabbages, carrots, garlic, kale, olives, onion, pecans, potatoes, radish, sugar beet, tomatoes, turnips, walnuts and wheat were based on unvalidated analytical data from Craven Laboratories and could not be used.

The conditions of the supervised trials are shown in Table 21. Most trials were not replicated or were duplicate applications to a split plot. Replicate residues shown in the Table represent samples from the split plots or replicate field samples from the single treated plot.

Periods of freezer storage between sampling and analysis were recorded for all trials and were within the acceptable proven stability period of 2 years except in a few cases. Snap bean samples from US trials in 1987 were stored for 680-700 days before analysis, which is acceptable for parathion residues but not for paraoxon. The stability of paraoxon in snap bean samples was poor after 6 months. Walnut samples from trials in 1987 for 970 days and prune samples from trials in 1987 were stored for 800-820 days before analysis. The validity of data from samples stored for more than 2 years must be questioned.

Table 21. Sprayers, plot sizes and field sample sizes in the parathion supervised trials.

| Crop | Country | Year | Sprayer | Plot size | Sample size |
|---|---|---|---|---|---|
| lemon | USA | 1988 | Helicopter | 5-6 trees | 16 fruit |
| lemon | USA | 1989 | commercial sprayer and power hand-gun sprayer | 2-3 trees | 16 fruit |
| orange | USA | 1987-89 | aerial - fixed wing and helicopter, and commercial sprayer | 3 trees - 1 ha | 16 fruit |
| apple | USA | 1988 | aerial - fixed wing and helicopter | 0.5 ha - 210 trees | 16 fruit |
| apple | France | 1994 | calibrated knapsack | | 1 kg and 4 kg |
| apple | USA | 1988 | calibrated knapsack | 12-14 trees | 16 fruit |
| pear | USA | 1988 | aerial - helicopter, power hand-gun sprayer | 2-18 trees | 16 fruit |
| apricot | USA | 1989 | aerial – helicopter | 56 trees | 1.1 kg |
| apricot | USA | 1989 | commercial sprayer | 4 trees | 1.1 kg |
| cherries | USA | 1988 | $CO_2$ powered backpack, commercial sprayer and handheld with diaphragm pump | 2-12 trees | 1.1-1.6 kg |
| prunes | USA | 1987-88 | aerial - helicopter | 6-25 trees | 1.1 kg |
| prunes | USA | 1987-88 | commercial and hand-gun sprayer | 2-4 trees | 1.1 - 4 kg |
| blackberries | USA | 1988 | aerial - fixed wing and helicopter | 0.2-1.6 ha | 1.1-3 kg |
| blackberries | USA | 1988-89 | $CO_2$ powered backpack, commercial sprayer | 6-93 $m^2$ | 1.1-1.4 kg |
| grapes | France | 1994 | knapsack airblast sprayer | 140-300 $m^2$ | 1 kg |
| grapes | USA | 1987 | helicopter | 35 $m^2$ - 0.35 ha | 1.1 kg |
| grapes | USA | 1987 | power hand-gun sprayer | 33-100 $m^2$ | 1.1 kg |
| strawberry | USA | 1988 | aerial - fixed wing | 200-1800 $m^2$ | 1.1-2 kg |
| strawberry | USA | 1988 | $CO_2$ powered backpack | 45 $m^2$ | 1.1-1.5 kg |
| olives | USA | 1988 | aerial - fixed wing and helicopter, and commercial sprayer | 3 trees - 1.2 ha | 1.1 kg |
| garlic | USA | 1988-89 | aerial - fixed wing | 60 $m^2$ - 1.2 ha | 0.4 kg |
| garlic | USA | 1988-89 | $CO_2$ powered backpack | 60 $m^2$ | 0.4 kg |
| onion | USA | 1987-89 | aerial - fixed wing and helicopter | 200 $m^2$ - 1.2 ha | 1.1-2 kg |
| onion | USA | 1987-89 | $CO_2$ powered backpack and self propelled research sprayer | 59-70 $m^2$ | 1.1-2 kg |
| broccoli | USA | 1989 | aerial - fixed wing and helicopter | 0.017-0.4 ha | 12-20 heads |
| broccoli | USA | 1989 | $CO_2$ powered backpack | 3-4 rows of 10-14 m | 12-20 heads |
| cabbage | USA | 1988 | aerial - fixed wing and helicopter | 0.017-0.1 ha | 12 heads |
| cabbage | USA | 1987-89 | $CO_2$ powered backpack | 4 rows of 14 m | 12-14 heads |
| kale | USA | 1987-88 | aerial - fixed wing | 170-740 $m^2$ | 1.1 kg |
| kale | USA | 1987-88 | $CO_2$ powered backpack | 33-930 $m^2$ | 1.1 kg |
| lettuce | USA | 1987-89 | aerial - fixed wing and helicopter | 84-2000 $m^2$ | 12 heads |
| lettuce | USA | 1987-89 | $CO_2$ powered backpack | 28-88 $m^2$ | 12 heads |
| spinach | USA | 1987-88 | aerial - helicopter, and $CO_2$ powered backpack | 28-50 $m^2$ | 0.8-1.6 kg |
| snap beans | USA | 1987 | aerial - fixed wing, and $CO_2$ powered backpack | 46 $m^2$ - 1.2 ha | 1 kg forage, pods, vines |
| beans, dry | USA | 1987-88 | aerial - fixed wing | 0.14-0.4 ha | 1.1 kg |
| beans, dry | USA | 1987-88 | $CO_2$ powered backpack, self-propelled research and tractor mounted | 51-150 $m^2$ | 1.1 kg |
| peas, dried | USA | 1988 | aerial - fixed wing and helicopter, and $CO_2$ powered backpack | 31 - 2900 $m^2$ | 1.1 kg |
| peppers | USA | 1987-88 | aerial - fixed wing, commercial sprayer and $CO_2$ powered backpack | 28-3500 $m^2$ | 1.1 kg |
| sweet corn | USA | 1987-88 | aerial - fixed wing and helicopter | 170-510 $m^2$ | 12 ears, 2 kg forage |
| sweet corn | USA | 1987-88 | $CO_2$ powered backpack | 16-110 $m^2$ | 12 ears, 2 kg forage |

| Crop | Country | Year | Sprayer | Plot size | Sample size |
|---|---|---|---|---|---|
| tomato | USA | 1987-88 | aerial | 0.012-0.4 ha | 16 fruit |
| tomato | USA | 1987-88 | $CO_2$ powered backpack | 45-100 $m^2$ | 16 fruit |
| carrot | USA | 1987-88 | aerial - fixed wing | 1.2 ha | 1.1-2 kg |
| carrot | USA | 1987-88 | $CO_2$ powered backpack | 23-50 $m^2$ | 1.1- 4 kg |
| potatoes | USA | 1989 | aircraft - fixed wing | 1400 $m^2$ | 16 tubers |
| potatoes | USA | 1989 | $CO_2$ powered backpack, self-propelled research sprayer and tractor mounted | 53-850 $m^2$ | 16 tubers |
| radish | USA | 1988 | aerial - fixed wing and helicopter | 100 $m^2$ | 1.1 kg |
| radish | USA | 1987-88 | $CO_2$ powered backpack | 23-74 $m^2$ | 1.1 kg |
| sugar beet | USA | 1988 | aerial - fixed wing and helicopter | 0.13-1.6 ha | 2.3 kg |
| sugar beet | USA | 1987-88 | $CO_2$ powered backpack, self-propelled research sprayer and tractor mounted | 74-150 $m^2$ | 2.3 kg |
| turnip | USA | 1987-88 | aerial - fixed wing and helicopter | 70-200 m row | 1.1 kg |
| turnip | USA | 1987-88 | $CO_2$ powered backpack | 29-46 $m^2$ | 1.1 kg |
| celery | USA | 1987-89 | aerial - fixed wing and helicopter | 84 $m^2$ - 0.4 ha | 12 stalks |
| celery | USA | 1988-89 | aerial - fixed wing and helicopter | 84 $m^2$ - 0.4 ha | 12 stalks |
| almond | USA | 1989 | aerial - fixed wing and helicopter | 0.34-4 ha | 1.1 kg |
| almond | USA | 1988-89 | commercial sprayer | 2-3 trees | 1.1 kg |
| barley | USA | 1997-98 | aerial - fixed wing and helicopter | 0.37-0.63 ha | 1.1-2.3 kg |
| maize | USA | 1987-89 | aerial - fixed wing and helicopter | 93-1300 $m^2$ | 1.1 kg grain, 0.8 kg forage |
| maize | USA | 1987-89 | $CO_2$ powered backpack and tractor mounted | 93-110 $m^2$ | 1.1 kg grain, 0.8 kg forage |
| rice | USA | 1987-88 | aerial - fixed wing, and CO2 powered backpack | 56-2800 $m^2$ | 1.1 kg grain 0.4 kg straw |
| sorghum | USA | 1987 | aerial - fixed wing | 0.9 ha | 1.1 kg |
| sorghum | USA | 1087 | $CO_2$ powered backpack | 186 $m^2$ | 1.1 kg |
| sorghum | USA | 1992 | aerial - fixed wing | 0.21-0.61 ha | 1.1-2.7 kg |
| wheat | USA | 1994 | aerial - fixed wing | 560 $m^2$ | 1 kg |
| wheat | USA | 1989 | aerial - fixed wing and helicopter | 560-730 $m^2$ | 1 kg grain, forage, straw |
| wheat | USA | 1989 | $CO_2$ powered backpack, bicycle sprayer and tractor mounted | 81-150 $m^2$ | 0.5 kg straw, 1 kg forage, grain |
| wheat | USA | 1993 | aerial - fixed wing and helicopter | 0.16-0.70 ha | 0.5-5 kg |
| canola | USA | 1992 | aerial - fixed wing | 0.4-0.5 ha | 0.8-1.2 kg |
| cotton | USA | 1997-98 | aerial - fixed wing and helicopter | 0.45-2.4 ha | 1.1-2.8 kg seed, 0.4 kg gin trash |
| cotton | USA | 1989 | $CO_2$ powered backpack and tractor mounted | 59-70 $m^2$ | 2.5 kg |
| soya bean | USA | 1988 | aerial - fixed wing | 0.12-0.49 ha | 2.2 kg |
| soya bean | USA | 1988 | $CO_2$ powered backpack and tractor mounted | 46-400 $m^2$ | 2.2 kg |
| sunflower | USA | 1989 | aerial -fixed wing | 0.14-0.67 ha | 1.1 kg |
| sunflower | USA | 1988-89 | $CO_2$ powered backpack, bicycle sprayer and tractor mounted | 56-260 $m^2$ | 1.1 kg |
| alfalfa | USA | 1995 | aerial - fixed wing and helicopter | 0.15-0.75 ha | 1.1 kg |
| alfalfa | USA | 1989 | $CO_2$ powered backpack, bicycle sprayer and tractor mounted | 46-139 $m^2$ | 1.1 kg forage, 0.4 kg hay |
| alfalfa | USA | 1989 | aerial - fixed wing and helicopter | 0.35-1.8 $m^2$ | 1.1 kg forage, 0.4 kg hay |
| clover | USA | 1988 | aerial - fixed wing | 450 $m^2$ | 0.8 kg forage, 0.4 kg hay |

| Crop | Country | Year | Sprayer | Plot size | Sample size |
|---|---|---|---|---|---|
| clover | USA | 1988 | $CO_2$ powered backpack and tractor mounted | 46-116 $m^2$ | 0.8 kg forage, 0.4 kg hay |

Table 22. Parathion residues in citrus fruits from supervised trials in the USA.

| FRUIT, State, year (variety) | Application | | | | | PHI | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | days | parathion | paraoxon | |
| GRAPEFRUIT | | | | | | | | | |
| FL, 1987 (Pink seedless) | WP | 19.5 | 0.24 | 8100 | 3 | 14<br>30 | 6.0 11<br>4.4 5.2 | 0.26 0.48<br>0.29 0.41 | EP-GF-5118 |
| FL, 1987 (Pink seedless) | WP | 19.5 +oil | 0.24 | 8100 | 3 | 14<br>30 | 7.2 6.2<br>3.4 2.9 | 0.3 0.25<br>0.34 0.33 | EP-GF-5118 |
| CA, 1989 (Marsh White) | WP | 11.2 | 0.24 | 4700 | 3 | 14<br>30 | 1.8 1.0<br>0.92 0.92 | 0.13 0.06<br>0.10 0.07 | EP-GF-1036 |
| CA, 1989 (Marsh White) | WP | 11.2 +oil | 0.24 | 4700 | 3 | 14<br>30 | 2.3 3.8<br>1.0 0.58 | 0.09 0.18<br>0.10 <0.05 | EP-GF-1036 |
| FL, 1989 (unknown, white) | EC | 11.2 | | 77 | ( 3 | 14<br>30 | 0.32 0.18<br>0.11 0.16 | <0.05 (2)<br><0.05 (2) | EP-GF-5119 |
| CA, 1989 (Marsh White) | EC | 11.2 | 0.24 | 4700 | 3 | 14<br>30 | 2.9 1.4<br>1.2 0.76 | 0.12 0.06<br>0.09 0.07 | EP-GF-1037 |
| LEMON, SWEET | | | | | | | | | |
| CA, 1988 (Lisbon) | WP +oil | | 0.24 | 4700 | 3 | 14<br>30 | 9.3 10.2<br>4.7 5.8 | 0.27 0.2<br>0.24 0.27 | EP-LM-1045 |
| CA, 1988 (Lisbon) | WP | | 0.24 | 4700 | 3 | 14<br>30 | 6.0 6.4<br>4.8 3.9 | 0.26 0.32<br>0.32 0.26 | EP-LM-1045 |
| CA, 1988 (Lisbon) | WP | | 0.24 | 3700 | 3 | 14<br>30<br>14 | 5.5 3.7<br>3.9 3.2<br>c 0.11 | 0.28 0.11<br>0.30 0.22 | EP-LM-1043 |
| CA, 1988 (Lisbon) | EC | 11.2 | | 94 | ( 3 | 14<br>30 | 1.6 0.39<br>0.46 0.34 | 0.08 <0.05<br><0.05 (2) | EP-LM-1047 |
| CA, 1988 (Lisbon) | EC | 11.2 | | 94 | ( 3 | 14<br>28<br>28 | 0.22 0.14<br>0.14 0.15<br>c 0.07 | <0.05 (2)<br><0.05 (2) | EP-LM-1048 |
| CA, 1988 (Lisbon) | EC | | 0.24 | 4700 | 3 | 14<br>30<br>14 | 4.8 4.6<br>2.8 3.7<br>c 0.05 | 0.16 0.13<br>0.12 0.15 | EP-LM-1046 |
| CA, 1988 (Lisbon) | EC | | 0.24 | 3700 | 3 | 14<br>30<br>14 | 2.7 2.1<br>1.5 1.7<br>c 0.11 | 0.09 0.10<br>0.06 0.12 | EP-LM-1044 |
| ORANGE, SWEET | | | | | | | | | |

| FRUIT, State, year (variety) | Application | | | | | PHI | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | days | parathion | paraoxon | |
| CA, 1988 (Nucellar) | EC | 9.0 | 0.24 | 3700 | 3 | 14<br>30 | 2.3 1.8<br>0.56 1.6 | 0.06 0.05<br><0.05 0.05 | EP-OR-1061 |
| CA, 1988 (Nucellar) | WP | 9.0 | 0.24 | 3700 | 3 | 14<br>30 | 2.0 1.0<br>2.0 1.0 | <0.05 0.06<br>0.08 <0.05 | EP-OR-1062 |
| CA, 1988 (Washington Navel) | EC | 11.2 | | 110 | ( 3 | 14<br>28<br>14<br>28 | 0.13 0.80<br>1.3 0.09<br>c 0.06<br>c 0.18 | <0.05 0.05<br>0.06 <0.05 | EP-OR-1063 |
| FL, 1987 (Hamlin) | WP +oil | 19.5 | 0.24 | 8100 | 3 | 14<br>30 | 6.8 12.2<br>4.4 7.2 | 0.11 0.20<br>0.18 0.35 | EP-OR-5116 |
| FL, 1987 (Hamlin) | WP | 19.5 | 0.24 | 8100 | 3 | 14<br>30 | 9.0 7.2<br>4.6 9.2 | 0.27 0.16<br>0.19 0.42 | EP-OR-5116 |
| FL, 1989 (Parson Brown) | EC | 11.2 | | 94 | ( 3 | 14<br>30 | 0.18 0.18<br><0.05 0.18 | <0.05<br><0.05 (2) | EP-OR-5117 |

( aerial application c: sample from control plot

Table 23. Parathion residues in apples from supervised trials. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| Country, year (variety) | Application | | | | | PHI, | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | days | parathion | paraoxon | |
| USA (NY), 1988 (Twenty Ounce) | EC<br>EC | 9.0<br>6.7 | 0.24 | 3700<br>370 | 6<br>6 | 14<br>14 | 2.4 2.7<br>3.7 3.5 | 0.05 0.06<br>0.06 0.10 | EP-AP-5140 |
| USA (NY), 1988 (McIntosh) | EC | 6.7 | | 46 | ( 6 | 14 | 0.2 6.0 | <0.05 0.14 | EP-AP-5141 |
| USA (WA), 1988 (Rome) | EC | 6.7 | | 190 | ( 6 | 14 | 0.10 0.095 | <0.05 (2) | EP-AP-1154 |
| USA (WA), 1988 (Rome) | EC<br>EC | 6.7<br>6.7 | 0.24 | 2800<br>470 | 6 | 14<br>14 | 1.4 1.6<br>0.2 2.7 | 0.05 <0.05<br><0.05 0.10 | EP-AP-1155 |
| France, 1994 (Red Chief) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.21<br>0.14<br>0.24<br>0.11<br>0.08 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51C/952132 Site II |
| France, 1994 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.51<br>0.28<br>0.16<br>0.13<br>0.08 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51C/952132 F4 |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| France, 1994 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.40<br>0.10<br>0.06<br>0.02<br>0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51C/952132 Site I |
| France, 1994 (Melrose) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.13<br>0.09<br>0.06<br>0.02<br>0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51C/952132 F1 |
| France, 1994 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.16<br>0.07<br>0.02<br>0.02<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51C/952132 F2 |
| France, 1994 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.26<br>0.10<br>0.06<br>0.04<br>0.02 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51C/952132 F3 |
| France, 1994 (Red Chief) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.44<br>0.27<br>0.18<br>0.19<br>0.14 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51A/952131 Site II |
| France, 1994 (Golden Delicious) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.39<br>0.37<br>0.21<br>0.18<br>0.16 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51A/952131 F4 |
| France, 1994 (Golden Delicious) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.36<br>0.15<br>0.09<br>0.09<br>0.02 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51A/952131 Site I |
| France, 1994 (Melrose) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.14<br>0.11<br>0.09<br>0.05<br>0.03 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51A/952131 F1 |
| France, 1994 (Golden Delicious) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.18<br>0.19<br>0.09<br>0.03<br>0.02 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51A/952131 F2 |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| France, 1994 (Golden Delicious) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.23<br>0.15<br>0.11<br>0.07<br>0.05 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 51A/952131 F3 |

( aerial application

Table 24. Parathion residues in pears from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| WA, 1987 (D'Anjou) | WP | 2.2 | 0.060 | 3700 | 4 | 14 | 0.88 0.52 | <0.05 (2) | EP-PR-1067 |
| CA, 1988 (Bartlett) | WP | 2.2 | 0.060 | 3700 | 4 | 14 | 0.36 0.82 | 0.1 0.26 | EP-PR-1064 |
| WA, 1987 (D'Anjou) | EC<br>EC | 3.9<br>2.2 | <br>0.060 | 190<br>3700 | 4<br>4 | 14<br>14 | 0.51 0.13<br>0.28 0.40 | <0.05 (2)<br><0.05 (2) | EP-PR-1068 |
| WA, 1987 (Bosc) | EC | 3.9 | | 94 | ( 4 | 14 | <0.05 (2) | <0.05 (2) | EP-PR-1069 |
| CA, 1988 (Bosc) | EC | 3.9 | | 190 | ( 4 | 14 | <0.05 0.06 | <0.05 (2) | EP-PR-1066 |
| CA, 1988 (Bartlett) | EC | 3.9 | | 280 | 4 | 14 | 0.18 0.05 | <0.05 (2) | EP-PR-1065 |
| CA, 1988 (Bartlett) | EC | | 0.060 | 3700 | 4 | 14 | 0.18 0.33 | 0.08 0.08 | EP-PR-1065 |

( aerial application

Table 25. Parathion residues in apricots from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| WA, 1988 (Tilton) | WP | 3.4 | | 470 | 4 | 14 | <0.05 (2) | <0.05 (2) | EP-AT-1162 |
| CA, 1988 (Modesto) | WP | 3.4 | | 750 | 4 | 14 | 0.18 <0.05 | <0.05 (2) | EP-AT-1157 |
| CA, 1988 (Blemheim) | WP | 3.4 | | 470 | 4 | 14 | <0.05 (2) | <0.05 (2) | EP-AT-1159 |
| WA, 1988 (Tilton) | EC<br>EC | 3.0<br>3.4 | 0.08 | 3700<br>470 | 4<br>4 | 14<br>14 | <0.05 (2)<br><0.05 0.06 | <0.05 (2)<br><0.05 (2) | EP-AT-1161 |
| CA, 1989 (Modesto) | EC | 3.4 | | 190 | ( 3 | 13 | <0.05 (2) | <0.05 (2) | EP-AT-1249 |
| CA, 1988 (Modesto) | EC<br>EC | 1.8<br>3.4 | 0.08 | 2200<br>770 | 4<br>4 | 14<br>14 | <0.05 (2)<br>0.10 0.19 | <0.05 (2)<br><0.05 (2) | EP-AT-1156 |
| CA, 1988 (Blemheim) | EC<br>EC | 1.5<br>3.4 | 0.08 | 1900<br>470 | 4<br>4 | 14<br>14 | <0.05 (2)<br>0.07 0.07 | <0.05 (2)<br><0.05 (2) | EP-AT-1158 |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| CA, 1988 (Blemheim) | EC | 3.4 | | 190 | ( 4 | 14 | <0.05 (2) | <0.05 (2) | EP-AT-1160 |

( aerial application

Table 26. Parathion residues in sour cherries from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| MI, 1988 (Montmorency) | WP<br>WP | 1.1<br>2.2 | 0.060<br>0.60 | 1900<br>370 | 6<br>6 | 14<br>14 | 0.1 0.09<br>0.65 0.37 | 0.18 0.18<br>0.46 0.28 | EP-CH-5149 |
| MI, 1988 (Napoleon) | WP<br>WP | 1.1<br>2.2 | 0.060<br>0.39 | 1900<br>570 | 4<br>4 | 14<br>14 | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CH-5152 |
| CA, 1989 (Bing) | WP<br>WP | 1.1<br>2.2 | 0.060 | 1900<br>470 | 4<br>4 | 14<br>14 | 0.10 0.08<br>0.09 0.46 | <0.05 (2)<br><0.05 0.05 | EP-CH-1217 |
| WA, 1988 (Bing) | WP<br>WP | 1.1<br>2.2 | 0.060 | 1900<br>470 | 4<br>4 | 14<br>14 | 1.6 1.3<br>2.0 3.0 | 0.22 0.21<br>0.22 0.28 | EP-CH-1215 |
| MI, 1988 (Montmorency) | EC | 1.1 | 0.060 | 1900 | 6 | 14 | 0.20 0.08 | 0.24 0.13 | EP-CH-5148 |
| MI, 1988 (Gold) | EC | 1.1 | 0.060 | 1900 | 4 | 14 | <0.05 (2) | <0.05 (2) | EP-CH-5151 |
| CA, 1988 (Bing) | EC<br>EC | 1.1<br>2.2 | 0.060 | 1900<br>470 | 4<br>4 | 14<br>14 | 0.06 0.08<br>0.29 0.25 | <0.05 (2)<br><0.05 (2) | EP-CH-1216 |
| WA, 1988 (Bing) | EC | 2.2 | 0.060 | 3700 | 4 | 14 | 1.0 0.72 | 0.16 0.11 | EP-CH-1214 |

Table 27. Parathion residues in fresh and dried plums or prunes from supervised residue trials and processing in the USA (Cañez, 1990e).

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| ID, 1988 (Empress) | WP | | 0.060 | | 4 | 14 | fresh<br>dried | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-PL-1073 |
| ID, 1988 (Empress) | EC | | 0.060 | | 4 | 14 | fresh<br>dried | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-PL-1074 |
| ID, 1988 (Empress) | EC | 4.5 | | | 4 | 14 | fresh<br>dried | <0.05 (2)<br>0.05 <0.05 | <0.05 (2)<br><0.05 (2) | EP-PL-1074 |
| OR, 1987 (Italian) | WP | | 0.060 | | 4 | 14 | fresh [1]<br>dried [1] | 0.28 0.22<br>0.17 0.13 | <0.05 (2)<br><0.05 (2) | EP-PL-1075 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| OR, 1987 (Italian) | 8F | | 0.060 | | 4 | 14 | fresh [1]<br>dried [1] | 0.30 0.24<br>0.13 0.12 | <0.05 (2)<br><0.05 (2) | EP-PL-1076 |
| OR, 1987 (Italian) | 8F | 4.5 | | | 4 | 14 | fresh [1]<br>dried [1] | 1.0 0.66<br>0.38 0.38 | 0.08 <0.05<br>0.05 0.05 | EP-PL-1076 |
| WA, 1987 (Late Italian) | EC | | 0.060 | | 4 | 14 | fresh [1]<br>dried [1] | 0.66 0.59<br>0.38 0.32 | <0.05 (2)<br><0.05 (2) | EP-PL-1077 |
| WA, 1987 (Late Italian) | EC | 4.5 | | | 4 | 14 | fresh [1]<br>dried [1] | 0.76 0.74<br>0.35 0.27 | <0.05 (2)<br>0.05 <0.05 | EP-PL-1077 |
| WA, 1987 (Late Italian) | WP | | 0.060 | | 4 | 14 | fresh [1]<br>dried [1] | 0.45 0.30<br>0.45 0.42 | <0.05 (2)<br>0.06 0.06 | EP-PL-1078 |
| WA, 1987 (Late Italian) | EC | 4.5 | | | ( 4 | 14 | fresh [1]<br>dried [1] | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-PL-1079 |
| CA, 1988 (French) | EC | | 0.060 | | 4 | 14 | fresh<br>dried | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-PL-1080 |
| CA, 1988 (French) | EC | 4.5 | | | 4 | 14 | fresh<br>dried | 0.28 0.23<br>0.21 0.20 | <0.05 (2)<br><0.05 (2) | EP-PL-1080 |
| CA, 1988 (French) | WP | | 0.060 | | 4 | 14 | fresh<br>dried | 0.05 0.08<br><0.05 (2) | <0.05 0.06<br><0.05 (2) | EP-PL-1081 |
| CA, 1988 (French) | EC | 4.5 | | | ( 4 | 14 | fresh<br>dried | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-PL-1082 |
| WA, 1987 (President) | WP | | 0.060 | | 4 | 14 | fresh [1] | 0.18 0.14 | <0.05 (2) | EP-PL-1139 |
| WA, 1987 (President) | EC | | 0.060 | | 4 | 14 | fresh [1] | 0.18 <0.05 | <0.05 (2) | EP-PL-1140 |
| WA, 1987 (President) | EC | 4.5 | | | 4 | 14 | fresh [1] | 0.07 0.86 | <0.05 0.06 | EP-PL-1140 |
| WA, 1988 (President) | EC | 4.5 | | | ( 4 | 14 | fresh | <0.05 (2) | <0.05 (2) | EP-PL-1141 |
| OR, 1988 (Italian) | WP | | 0.060 | | 4 | 14 | fresh | 0.30 0.25 | <0.05 (2) | EP-PL-1198 |
| OR, 1988 (Italian) | EC | | 0.060 | | 4 | 14 | fresh | 0.32 0.27 | <0.05 (2) | EP-PL-1199 |
| OR, 1988 (Italian) | EC | 4.5 | | | 4 | 14 | fresh | 2.4 2.1 | 0.09 0.07 | EP-PL-1199 |
| ID, 1988 (Simka) | WP | | 0.060 | | 4 | 14 | fresh | <0.05 (2) | <0.05 (2) | EP-PL-1200 |
| ID, 1988 (Simka) | EC | | 0.060 | | 4 | 14 | fresh | <0.05 (2) | <0.05 (2) | EP-PL-1201 |
| ID, 1988 (Simka) | EC | 4.5 | | | 4 | 14 | fresh | 0.06 <0.05 | <0.05 (2) | EP-PL-1201 |
| CA, 1988 (Angelina) | EC | 4.5 | | | ( 4 | 14 | fresh | <0.05 (2) | <0.05 (2) | EP-PL-1202 |

| State, year (variety) | Application | | | | | PHI, | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | days | | parathion | paraoxon | |
| CA, 1988 (Late Santa Rosa) | EC | | 0.060 | | 4 | 14 | fresh | 0.19 0.18 | <0.05 (2) | EP-PL-1203 |
| CA, 1988 (Late Santa Rosa) | EC | 4.5 | | | 4 | 14 | fresh | 0.31 0.47 | 0.05 0.07 | EP-PL-1203 |
| CA, 1988 (Late Santa Rosa) | WP | | 0.060 | | 4 | 14 | fresh | <0.05 0.08 | <0.05 (2) | EP-PL-1204 |
| CA, 1988 (French) | EC | | 0.060 | | 4 | 14 | fresh | <0.05 | <0.05 | EP-PL-2040 |
| CA, 1988 (French) | EC | | 0.30 | | 4 | 14 | fresh<br>dried | 0.07<br>0.08 | <0.05<br><0.05 | EP-PL-2040 |
| CA, 1988 (French) | EC | | 0.060 | | 4 | 14 | fresh | <0.05 | <0.05 | EP-PL-2041 |
| CA, 1988 (French) | EC | | 0.30 | | 4 | 14 | fresh<br>dried | 0.62<br>0.45 | <0.05<br>0.08 | EP-PL-2041 |

[1] stored for 800-820 days between sampling and analysis.
( aerial application

Table 28. Parathion residues in blackberries from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | days | parathion | paraoxon | |
| TX, 1988 (Cherokee) | EC<br>EC | 1.1<br>1.1 | 0.12 | 970<br>490 | 3<br>3 | 15<br>15 | 0.05 0.09<br>0.06 0.06 | <0.05 (2)<br><0.05 (2) | EP-BB-1165 |
| TX, 1988 (Cherokee) | WP | 1.1 | | 470 | 3 | 15 | 0.07 <0.05 | <0.05 (2) | EP-BB-1166 |
| OR, 1988 (Evergreen) | EC<br>EC | 2.8<br>1.1 | 0.12 | 2300<br>470 | 3<br>3 | 15<br>15 | 2.1 2.2<br>0.20 0.24 | 0.17 0.15<br><0.05 (2) | EP-BB-1167 |
| OR, 1988 (Evergreen) | WP | 1.1 | | 470 | 3 | 15 | 0.20 0.20 | <0.05 (2) | EP-BB-1168 |
| OR, 1988 (Evergreen) | EC | 1.1 | | 94 | ( 4 | 15 | 0.05 <0.05 | <0.05 (2) | EP-BB-1169 |
| CA, 1988 (Olallie) | EC<br>EC | 2.2<br>1.1 | 0.12 | 1900<br>470 | 3<br>3 | 15<br>15 | 0.72 0.60<br>0.25 0.24 | <0.05 (2)<br><0.05 (2) | EP-BB-1171 |
| CA, 1988 (Olallie) | WP | 1.1 | | 470 | 3 | 15 | 0.24 0.24 | <0.05 (2) | EP-BB-1172 |
| CA, 1988 (Olallie) | EC | 1.1 | | 190 | ( 3 | 15 | 0.06 0.06 | <0.05 (2) | EP-BB-1173 |
| MI, 1989 (Evergreen) | EC<br>EC | 2.2<br>1.1 | 0.12 | 1900<br>470 | 6<br>6 | 15<br>15 | 0.28 0.28<br>0.16 0.27 | 0.28 0.30<br>0.22 0.25 | EP-BB-1251 |
| MI, 1989 (Chester) | WP | 1.1 | | 470 | 6 | 15 | 0.46 0.12 | 0.32 0.20 | EP-BB-1252 |

( aerial application

Table 29. Parathion residues in wine grapes from supervised trials in France and the USA.

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| USA (CA), 1987 (Emperor) | EC | 1.7 | | 190 | ( 3 | 14 | <0.05 (2) | <0.05 (2) | EP-GR-1029 |
| USA (CA), 1987 (Emperor) | EC | 1.7 | | 470 | 3 | 14 | 0.30 0.25 | <0.05 (2) | EP-GR-1030 |
| USA (CA), 1987 (Emperor) | EC | 2.8 | 0.12 | 2300 | 3 | 14 | 0.63 0.39 | <0.05 (2) | EP-GR-1030 |
| USA (CA), 1987 (Emperor) | WP<br>WP | 2.8<br>1.7 | 0.12 | 2300<br>470 | 3<br>3 | 14<br>14 | 0.23 2.4<br>0.73 0.33 | <0.05 0.06<br>0.06 <0.05 | EP-GR-1031 |
| USA (WA), 1987 (Concord) | EC<br>EC | 1.7<br>2.2 | <br>0.12 | 190<br>1900 | 3<br>3 | 14<br>14 | <0.05 (2)<br>0.85 0.29 | <0.05 (2)<br><0.05 (2) | EP-GR-1032 |
| USA (WA), 1987 (Concord) | WP<br>WP | 1.7<br>2.2 | <br>0.12 | 190<br>1900 | 3<br>3 | 14<br>14 | 0.06 0.10<br>0.85 0.68 | <0.05 (2)<br><0.05 (2) | EP-GR-1033 |
| USA (WA), 1987 (Concord) | EC | 1.7 | | 93 | ( 3 | 14 | <0.05 (2) | <0.05 (2) | EP-GR-1035 |
| France, 1994 (Grenache) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.51<br>0.17<br>0.17<br>0.11<br>0.08 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV<br>50A/952129<br>Site II |
| France, 1994 (Chenin Blanc) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.16<br>0.11<br>0.15<br>0.07<br>0.13 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV<br>50A/952129<br>F1 |
| France, 1994 (Grenache) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.11<br>0.12<br>0.09<br>0.12<br>0.06 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV<br>50A/952129<br>Site I |
| France, 1994 (Cabernet) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.23<br>0.19<br>0.18<br>0.19<br>0.10 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV<br>50A/952129<br>F3 |
| France, 1994 (Cabernet) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.12<br>0.15<br>0.12<br>0.15<br>0.10 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV<br>50A/952129<br>F4 |
| France, 1994 (Grenache) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.31<br>0.14<br>0.09<br>0.05<br>0.02 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV<br>50C/952130<br>Site II |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| France, 1994 (Chenin blanc) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.10<br>0.06<br>0.05<br>0.03<br>0.02 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 50C/952130 F1 |
| France, 1994 (Grenache) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.06<br>0.06<br>0.03<br>0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 50C/952130 Site I |
| France, 1994 (Cabernet Franc) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.16<br>0.10<br>0.05<br>0.07<br>0.03 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | CHV 50C/952130 F3 |
| France, 1994 (Cabernet) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.11<br>0.13<br>0.08<br>0.07<br>0.05 | <0.01<br><0.01<br><0.01<br>0.01<br><0.01 | CHV 50C/952130 F4 |

( aerial application

Table 30. Parathion residues in strawberries from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| OR, 1988 (Shukson) | WP | 0.90 | | 190 | 4 | 14 | <0.05 (2) | <0.05 (2) | EP-ST-1134 |
| OR, 1988 (Shukson) | EC<br>EC | 1.3<br>0.90 | 0.095 | 1400<br>190 | 4<br>4 | 14<br>14 | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-ST-1135 |
| OR, 1988 (Totem) | EC | 0.90 | | 94 | ( 4 | 14 | <0.05 (2) | <0.05 (2) | EP-ST-1136 |
| CA, 1988 (Douglas) | EC<br>EC | 1.3<br>0.90 | 0.095 | 1400<br>280 | 4<br>4 | 14<br>14 | 0.20 0.20<br>0.29 0.06 | <0.05 (2)<br><0.05 (2) | EP-ST-1137 |
| CA, 1988 (Douglas) | WP | 0.90 | | 280 | 4 | 14 | 0.08 0.08 | <0.05 (2) | EP-ST-1138 |
| FL, 1988 (Salva) | EC<br>EC | 1.4<br>0.96 | 0.095 | 1460<br>210 | 6<br>6 | 14<br>14 | 1.1 1.2<br>0.35 0.27 | 0.08 0.09<br><0.05 (2) | EP-ST-5120 |
| FL, 1988 (Salva) | WP | 0.96 | | 210 | 6 | 14 | 0.42 0.21 | <0.05 (2) | EP-ST-5121 |
| FL, 1988 (Salva) | EC | 0.90 | | 72 | ( 6 | 14 | <0.05 (2) | <0.05 (2) | EP-ST-5122 |

( aerial application

Table 31. Parathion residues in olives from supervised trials in the USA.

| State year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| CA, 1988 (Manzanillo) | EC<br>EC | 1.8<br>5.6 | 0.096<br>1.2 | 1870<br>470 | 3<br>3 | 39<br>39 | <0.05 (2)<br>0.29 0.35 | <0.05 (2)<br><0.05 0.05 | EP-OL-1190 [1] |
| CA, 1988 (Manzanillo) | EC | 5.6 | | 190 | ( 3 | 42 | <0.05 (2) | <0.05 (2) | EP-OL-1191 [1] |
| CA, 1988 (Manzanillo) | EC<br>EC | 5.7<br>5.7 | 0.096 | 5950<br>470 | 3<br>3 | 42<br>42 | 7.3 9.1<br>0.34 0.17 | 0.16 0.19<br><0.05 (2) | EP-OL-1192 [1] |
| CA, 1988 (Manzanillo) | EC | 5.6 | | 94 | ( 3 | 42 | <0.05 (2) | <0.05 (2) | EP-OL-1193 [1] |

[1] unvalidated analytical data
( aerial application

Table 32. Parathion residues in garlic from supervised trials in the USA. Bulbs analysed.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| CA, 1988 (Rogers Virus Free) | EC | 0.56 | | 190 | ( 4 | 15 | <0.05 (2) | <0.05 (2) | EP-GA-1180 [1] |
| CA, 1988 (Rogers Virus Free) | EC | 0.56 | | 190 | 4 | 15 | <0.05 (2) | <0.05 (2) | EP-GA-1181 [1] |
| CA, 1988 (Rogers Virus Free) | WP | 0.56 | | 190 | 4 | 15 | <0.05 (2) | <0.05 (2) | EP-GA-1182 [1] |
| TX, 1989 (Crystal Pear) | EC | 0.56 | | 190 | 4 | 15<br>15 | 0.09 0.10<br>c 0.11 | <0.05 (2)<br>c <0.05 | EP-GA-1183 [1] |
| TX, 1989 (Crystal Pear) | WP | 0.56 | | 190 | 4 | 15<br>15 | 0.11 0.11<br>c 0.10 | <0.05 (2)<br>c <0.05 | EP-GA-1184 [1] |
| TX, 1989 (Crystal Pear) | EC | 0.56 | | 47 | ( 4 | 15<br>15 | <0.05 (2)<br>c 0.11 | <0.05 (2)<br>c <0.05 | EP-GA-1186 [1] |

[1] unvalidated analytical data
(aerial application c: sample from control plot

Table 33. Parathion residues in onions from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | sample | parathion | paraoxon | |
| CA, 1987 (Evergreen) | EC | 0.90 | | 370 | 6 | 15<br>15 | green | 0.16 0.11<br>c 0.16 | <0.05 (2)<br>c <0.05 | EP-ON-1058 [1] |
| CA, 1987 (Evergreen) | WP | 0.90 | | 370 | 6 | 15<br>15 | green | 0.34 0.33<br>c 0.13 | <0.05 (2)<br>c <0.05 | EP-ON-1059 [1] |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | sample | parathion | paraoxon | |
| CA, 1987 (Evergreen) | EC | 0.90 | | 190 | ( 6 | 15<br>15 | green | 0.24 0.18<br>c 0.08 | <0.05 (2)<br>c <0.05 | EP-ON-1060 [1] |
| CA, 1989 (K99) | EC | 0.90 | | 190 | ( 6 | 15 | green | <0.05 (2) | <0.05 (2) | EP-ON-1187 [1] |
| CA, 1989 (K99) | EC | 0.90 | | 280 | 6 | 15 | green | <0.05 (2) | <0.05 (2) | EP-ON-1188 [1] |
| CA, 1988 (K99) | WP | 0.90 | | 280 | 6 | 15 | green | <0.05 (2) | <0.05 (2) | EP-ON-1189 [1] |
| CA, 1988 (Southport Late) | EC | 0.90 | | 190 | 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | EP-ON-1224 [1] |
| CA, 1988 (Southport Late) | WP | 0.90 | | 190 | 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | EP-ON-1225 [1] |
| CA, 1988 (Southport Late) | EC | 0.90 | | 190 | ( 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | EP-ON-1226 [1] |
| WA, 1988 (Yellow Sweet Spanish) | EC | 0.90 | | 210 | 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | EP-ON-1227 [1] |
| WA, 1988 (Yellow Sweet Spanish) | WP | 0.90 | | 215 | 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | EP-ON-1228 [1] |
| WA, 1988 (Yellow Sweet Spanish) | EC | 0.90 | | 39 | ( 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | EP-ON-1229 [1] |
| CO, 1988 (Winters) | EC | 0.90 | | 200 | 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | EP-ON-5168 [1] |
| CO, 1988 (Winters) | WP | 0.90 | | 200 | 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | EP-ON-5169 [1] |
| NY, 1988 (Early Yellow Globe) | EC | 0.90 | | 190 | 4 | 15 | green | <0.05 (2) | <0.05 (2) | EP-ON-5170 [1] |
| NY, 1988 (Early Yellow Globe) | WP | 0.90 | | 190 | 4 | 15 | green | <0.05 (2) | <0.05 (2) | EP-ON-5171 [1] |

[1] unvalidated analytical data
( aerial application c: sample from control plot

Table 34. Parathion residues in broccoli from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| CA, 1989 (501 Green Valiant) | EC | 0.56 | | 190 | ( 6 | 7 | 0.22 0.22 | <0.05 (2) | EP-BR-1004 A [1] |
| CA, 1989 (501 Green Valiant) | EC | 0.84 | | 190 | ( 6 | 7 | 0.17 0.15 | <0.05 (2) | EP-BR-1004 B [1] |
| CA, 1989 (501 Green Valiant) | EC | 1.1 | | 190 | ( 6 | 7<br>14 | 0.24 0.17<br>0.11 0.11 | <0.05 (2)<br><0.05 (2) | EP-BR-1004 C [1] |
| CA, 1989 (501 Green Valiant) | EC | 0.56 | | 370 | 6 | 7 | 0.28 0.22 | <0.05 (2) | EP-BR-1005 A [1] |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| CA, 1989 (501 Green Valiant) | EC | 0.84 | | 370 | 6 | 7 | 0.27 0.33 | <0.05 (2) | EP-BR-1005 B [1] |
| CA, 1989 (501 Green Valiant) | EC | 1.1 | | 370 | 6 | 7<br>14 | 0.90 0.49<br>0.15 0.14 | <0.05 (2)<br><0.05 (2) | EP-BR-1005 C [1] |
| CA, 1988 (Green Duke) | EC | 0.56 | | 280 | 6 | 7 | 0.23 0.30 | <0.05 (2) | EP-BR-1006 A [1] |
| CA, 1988 (Green Duke) | EC | 1.7 | | 280 | 6 | 21 | <0.05 | <0.05 | EP-BR-1006 B [1] |
| CA, 1989 (501 Green Valiant) | WP | 0.56 | | 370 | 6 | 7 | 0.61 0.81 | <0.05 (2) | EP-BR-1007 [1] |
| CA, 1989 (Green Duke) | WP | 0.56 | | 280 | 6 | 7 | 0.11 0.11 | <0.05 (2) | EP-BR-1008 [1] |
| CA, 1988 (Shogun) | EC | 0.56 | | 200 | ( 6 | 7 | <0.05 (2) | <0.05 (2) | EP-BR-1009 A [1] |
| CA, 1988 (Shogun) | EC | 1.7 | | 200 | ( 6 | 21 | <0.05 (2) | <0.05 (2) | EP-BR-1009 B [1] |

[1] unvalidated analytical data
( aerial application

Table 35. Parathion residues in cabbage from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1989 (Headstart) | EC | 0.28 | | 190 | 6 | 7 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-1010 A [1] |
| CA, 1989 (Headstart) | EC | 0.28 | | 190 | 6 | 7 | including wrapper leaves | 0.11 0.17 | <0.05 (2) | EP-CB-1010 A [1] |
| CA, 1989 (Headstart) | EC | 0.56 | | 190 | 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-1010 B [1] |
| CA, 1989 (Headstart) | EC | 0.56 | | 190 | 6 | 10 | including wrapper leaves | 0.07 0.12 | <0.05 (2) | EP-CB-1010 B [1] |
| CA, 1989 (Headstart) | EC | 0.84 | | 190 | 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-1010 C [1] |
| CA, 1989 (Headstart) | EC | 0.84 | | 190 | 6 | 10 | including wrapper leaves | 0.35 0.44 | <0.05 (2) | EP-CB-1010 C [1] |
| CA, 1989 (Headstart) | EC | 1.1 | | 190 | 6 | 14 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-1010 D [1] |
| CA, 1989 (Headstart) | EC | 1.1 | | 190 | 6 | 14 | including wrapper leaves | 0.30 0.10 | <0.05 (2) | EP-CB-1010 D [1] |
| CA, 1989 (Headstart) | WP | 0.28 | | 190 | 6 | 7 | including wrapper leaves | 0.14 0.12 | <0.05 (2) | EP-CB-1011 [1] |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| TX, 1987 (Savoy Chieftain) | EC | 0.28 | | 150 | 6 | 7 | including wrapper leaves | 0.29 0.37 | <0.05 (2) | EP-CB-1012 A [1] |
| TX, 1987 (Savoy Chieftain) | EC | 0.56 | | 150 | 6 | 10 | including wrapper leaves | 0.76 1.4 | <0.05 (2) | EP-CB-1012 B [1] |
| TX, 1987 (Savoy Chieftain) | EC | 1.7 | | 150 | 6 | 21 | including wrapper leaves | 0.20 0.27 | <0.05 (2) | EP-CB-1012 C [1] |
| TX, 1987 (Savoy Chieftain) | WP | 0.28 | | 150 | 6 | 7 | including wrapper leaves | 0.61 0.38 | <0.05 (2) | EP-CB-1013 A [1] |
| TX, 1987 (Savoy Chieftain) | WP | 0.56 | | 150 | 6 | 10 | including wrapper leaves | 1.6 1.4 | 0.06 <0.05 | EP-CB-1013 B [1] |
| TX, 1987 (Savoy Chieftain) | WP | 1.7 | | 150 | 6 | 21 | including wrapper leaves | 0.40 0.62 | <0.05 (2) | EP-CB-1013 C [1] |
| CA, 1988 (Headstart) | EC | 0.28 | | 200 | ( 6 | 7 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-1014 A [1] |
| CA, 1988 (Headstart) | EC | 0.28 | | 200 | ( 6 | 7 | including wrapper leaves | 0.22 0.12 | 0.06 0.11 | EP-CB-1014 A [1] |
| CA, 1988 (Headstart) | EC | 0.56 | | 200 | ( 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-1014 B [1] |
| CA, 1988 (Headstart) | EC | 0.56 | | 200 | ( 6 | 10 | including wrapper leaves | 0.16 0.27 | <0.05 0.07 | EP-CB-1014 B [1] |
| CA, 1988 (Headstart) | EC | 0.84 | | 200 | ( 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-1014 C [1] |
| CA, 1988 (Headstart) | EC | 1.1 | | 200 | ( 6 | 14 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-1014 C [1] |
| CA, 1988 (Headstart) | EC | 0.84 | | 200 | ( 6 | 10 | including wrapper leaves | 0.25 0.23 | 0.13 0.06 | EP-CB-1014 C [1] |
| CA, 1988 (Headstart) | EC | 1.1 | | 200 | ( 6 | 14 | including wrapper leaves | 0.12 0.18 | 0.06 0.09 | EP-CB-1014 C [1] |
| FL, 1987 (Bravo) | EC | 0.28 | | 650 | 6 | 7 | including wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-5001 A [1] |
| FL, 1987 (Bravo) | EC | 0.56 | | 650 | 6 | 10<br>21 | including wrapper leaves | <0.05 0.08<br>0.20 <0.05 | <0.05 (2)<br><0.05 (2) | EP-CB-5001 B [1] |
| FL, 1987 (Bravo) | WP | 0.28 | | 650 | 6 | 7 | including wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-5002 [1] |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| FL, 1988 (Bravo) | EC | 0.28 | | 58 | ( 6 | 7 | including wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-5003 A [1] |
| FL, 1988 (Bravo) | EC | 0.56 | | 58 | ( 6 | 10 | including wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-5003 B [1] |
| FL, 1988 (Bravo) | EC | 1.7 | | 58 | ( 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-5003 C [1] |
| NY, 1987 (King Cole Hybrid) | EC | 0.28 | | 230 | 6 | 30 | including wrapper leaves | <0.05 (2) | <0.05 (2) | EP-CB-5005 A [1] |
| NY, 1987 (King Cole Hybrid) | EC | 0.56 | | 230 | 6 | 30 | including wrapper leaves | 0.19 <0.05 | <0.05 (2) | EP-CB-5005 B [1] |
| NY, 1987 (King Cole Hybrid) | EC | 1.7 | | 230 | 6 | 30 | including wrapper leaves | 0.28 <0.05 | <0.05 (2) | EP-CB-5005 C [1] |
| NY, 1987 (King Cole Hybrid) | WP | 0.28 | | 230 | 6 | 7 | including wrapper leaves | <0.05 0.07 | <0.05 (2) | EP-CB-5006 [1] |

[1] unvalidated analytical data
( aerial application

Table 36. Parathion residues in peppers from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| TX, 1988 (Grande Rio 66) | WP | 0.90 | | 150 | 6 | 15 | <0.05 (2) | <0.05 (2) | EP-PP-1120 |
| TX, 1988 (Grande Rio 66) | EC | 0.90 | | 150 | 6 | 15 | <0.05 (2) | <0.05 (2) | EP-PP-1121 |
| CA, 1987 (Jupiter) | EC | 0.90 | | 370 | 6 | 15 | <0.05 (2) | <0.05 (2) | EP-PP-1122 |
| CA, 1987 (Jupiter) | WP | 0.90 | | 370 | 6 | 15 | <0.05 (2) | <0.05 (2) | EP-PP-1123 |
| CA, 1987 (Jupiter) | EC | 0.90 | | 190 | ( 6 | 15 | <0.05 (2) | <0.05 (2) | EP-PP-1124 |
| FL, 1987 (Belle Captain) | EC | 0.90 | | 670 | 6 | 7 | 0.57 0.95 | <0.05 (2) | EP-PP-5065 |
| FL, 1988 (Belle Captain) | WP | 0.90 | | 670 | 6 | 7 | 0.94 0.72 | <0.05 (2) | EP-PP-5066 |
| FL, 1988 (Jupiter) | EC | 0.90 | | 58 | ( 6 | 7 | <0.05 (2) | <0.05 (2) | EP-PP-5067 |
| NJ, 1988 (Yolo Wonder) | EC | 0.90 | | 540 | 6 | 7 | 0.09 0.08 | <0.05 (2) | EP-PP-5069 |
| NJ, 1988 (Yolo Wonder) | WP | 0.90 | | 540 | 6 | 7 | 0.17 0.10 | <0.05 (2) | EP-PP-5070 |

( aerial application

Table 37. Parathion residues in sweet corn (ears) from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| WA, 1989 (Jubilee) | EC | 1.1 | | 94 | ( 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-1246 |
| WA, 1989 (Jubilee) | EC | 1.1 | | 190 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-1247 |
| WA, 1989 (Jubilee) | WP | 1.1 | | 190 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-1248 |
| FL, 1988 (Silver Queen) | EC | 1.1 | | 700 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5007 |
| FL, 1988 (Silver Queen) | WP | 1.1 | | 700 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5008 |
| FL, 1988 (Merrit) | EC | 1.1 | | 58 | ( 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5009 |
| NY, 1987 (Early Sunray) | EC | 1.1 | | 230 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5011 |
| NY, 1987 (Early Sunray) | WP | 1.1 | | 230 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5012 |
| WI, 1987 (Incredible) | EC | 1.1 | | 200 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5013 |
| WI, 1987 (Incredible) | WP | 1.1 | | 200 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5014 |

( aerial application

Table 38. Parathion residues in tomatoes from supervised trials in the USA.

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | parathion | paraoxon | |
| CA, 1987 (UC 7879) | EC | 1.1 | 6 | 10 | 0.42 0.52 | <0.05 (2) | EP-TO-1128 [1] |
| CA, 1988 (Roma) | EC | 1.1 | 6 | 10 | 0.14 | <0.05 | EP-TO-2009 [1] |
| CA, 1988 (Murietta) | EC | 1.1 | 5 | 10 | 0.19 | <0.05 | EP-TO-2010 [1] |
| FL, 1987 (Sunny) | EC | 1.1 | 6 | 10 | <0.05 0.10 | <0.05 (2) | EP-TO-5107 [1] |
| OH, 1988 (Heinz 1810) | EC | 1.1 | 6 | 10 | <0.05 (2) | <0.05 (2) | EP-TO-5109 [1] |
| CA, 1987 (UC 7879) | EC | 1.1 | ( 6 | 10 | 0.10 0.40 | <0.05 (2) | EP-TO-1129 [1] |
| OH, 1988 (Heinz 1810) | EC | 1.1 | ( 6 | 10 | <0.05 (2) | <0.05 (2) | EP-TO-5111 [1] |
| CA, 1987 (UC 7879) | WP | 1.1 | 6 | 10 | 0.50 1.2 | <0.05 (2) | EP-TO-1127 [1] |
| FL, 1987 (Sunny) | WP | 1.1 | 6 | 10 | <0.05 (2) | <0.05 (2) | EP-TO-5108 [1] |
| OH, 1988 (Heinz 1810) | WP | 1.1 | 6 | 10 | <0.05 (2) | <0.05 (2) | EP-TO-5110 [1] |

[1] unvalidated analytical data
( aerial application

Table 39. Parathion residues in field peas from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | Sample | parathion | paraoxon | |
| WA, 1989 (Dark Skin 49) | EC | 0.56 | | 187 | 6 | 10 | pod | 0.27 0.33 | 0.31 0.23 | EP-PE-1176 |
| WA, 1989 (Dark Skin 49) | WP | 0.56 | | 187 | 5 | 10 | pod | 0.15 0.11 | <0.05 (2) | EP-PE-1177 |
| WA, 1989 (Dark Skin 49) | EC | 0.56 | | 94 | ( 6 | 10 | pod | <0.05 (2) | <0.05 0.06 | EP-PE-1179 |
| WA, 1988 (Fraiser) | WP | 0.56 | | 140 | 6 | 10 | pod | <0.05 (2) | <0.05 (2) | EP-PE-1205 |
| WA, 1988 (Fraiser) | EC | 0.56 | | 140 | 6 | 10 | pod | 0.07 <0.05 | 0.06 <0.05 | EP-PE-1206 |
| WA, 1988 (Fraiser) | EC | 0.56 | | 47 | ( 6 | 10 | pod | 0.30 0.27 | 0.08 0.08 | EP-PE-1208 |
| WI, 1988 (Ego) | EC | 0.56 | | 240 | 5 | 9 | pod | <0.05 (2) | <0.05 (2) | EP-PE-5172 |
| WI, 1988 (Ego) | WP | 0.56 | | 240 | 5 | 9 | pod | <0.05 (2) | <0.05 (2) | EP-PE-5173 |
| WI, 1988 (9888F) | EC | 0.56 | | 38 | ( 5 | 10 | pod | <0.05 (2) | <0.05 (2) | EP-PE-5174 |

( aerial application

Table 40. Parathion residues in kale from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| TX, 1987 (Blue Scotch Curled) | EC | 0.28 | | 152 | 6 | 7 | 0.21 0.27 | <0.05 (2) | EP-KA-1039 [1] |
| TX, 1987 (Blue Scotch Curled) | EC | 0.56 | | 152 | 6 | 10 | 0.10 0.05 | <0.05 (2) | EP-KA-1039 [1] |
| TX, 1987 (Blue Scotch Curled) | WP | 0.28 | | 152 | 6 | 7 | 0.27 0.11 | <0.05 (2) | EP-KA-1040 [1] |
| TX, 1987 (Blue Scotch Curled) | WP | 0.56 | | 152 | 6 | 10<br>15 | 0.17 0.19<br>0.22 <0.05 | <0.05 (2)<br><0.05 (2) | EP-KA-1040 [1] |
| TX, 1987 (Blue Scotch Curled) | EC | 0.28 | | 47 | ( 6 | 7 | 0.11 0.15 | <0.05 (2) | EP-KA-1041 [1] |
| TX, 1987 (Blue Scotch Curled) | EC | 0.56 | | 47 | ( 6 | 10<br>15 | 0.11 0.15<br>0.06 0.06 | <0.05 (2)<br><0.05 (2) | EP-KA-1041 [1] |
| FL, 1987 (Blue Curly/Purple Curly) | EC | 0.30 | | 720 | 6 | 7 | 0.60 1.4 | <0.05 0.13 | EP-KA-5075 [1] |
| FL, 1987 (Blue Curly/Purple Curly) | EC | 0.61 | | 720 | 6 | 10<br>15 | 0.92 0.90<br>0.18 0.18 | <0.05 0.06<br><0.05 (2) | EP-KA-5075 [1] |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| FL, 1987 (Blue Curly/Purple Curly) | WP | 0.30 | | 720 | 6 | 7 | 0.65 0.22 | 0.06 <0.05 | EP-KA-5076 [1] |
| FL, 1987 (Blue Curly/Purple Curly) | WP | 0.61 | | 720 | 6 | 10<br>15 | 0.96 1.4<br>0.16 0.13 | 0.06 <0.05<br><0.05 (2) | EP-KA-5076 [1] |
| FL, 1988 (NA6060) | EC | 0.28 | | 58 | ( 6 | 7 | <0.05 (2) | <0.05 (2) | EP-KA-5077 [1] |
| FL, 1988 (NA6060) | EC | 0.56 | | 58 | ( 6 | 10<br>15 | 0.07 0.09<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-KA-5077 [1] |
| NJ, 1987 (Vates) | EC | 0.28 | | 360 | 6 | 7 | 0.11 0.08 | <0.05 (2) | EP-KA-5079 [1] |
| NJ, 1987 (Vates) | EC | 0.56 | | 360 | 6 | 10<br>15 | 0.21 0.10<br>0.10 0.05 | <0.05 (2)<br><0.05 (2) | EP-KA-5079 [1] |
| NJ, 1987 (Vates) | WP | 0.28 | | 360 | 6 | 7 | 0.13 0.10 | <0.05 (2) | EP-KA-5080 [1] |
| NJ, 1987 (Vates) | WP | 0.56 | | 360 | 6 | 10<br>15 | 0.16 0.12<br>0.10 0.06 | <0.05 (2)<br><0.05 (2) | EP-KA-5080 [1] |
| VA, 1987 (Dwarf Blue Curled Vates) | EC | 0.27 | | 143 | 6 | 7 | 0.53 0.73 | <0.05 0.10 | EP-KA-5081 [1] |
| VA, 1987 (Dwarf Blue Curled Vates) | EC | 0.54 | | 143 | 6 | 10<br>15 | 1.1 0.41<br>0.11 0.06 | 0.07 0.05<br><0.05 (2) | EP-KA-5081 [1] |
| VA, 1987 (Dwarf Blue Curled Vates) | WP | 0.27 | | 143 | 6 | 7 | 1.3 0.78 | 0.08 0.08 | EP-KA-5082 [1] |
| VA, 1987 (Dwarf Blue Curled Vates) | WP | 0.55 | | 143 | 6 | 10<br>15 | 0.40 0.68<br>0.15 0.20 | <0.05 0.06<br><0.05 (2) | EP-KA-5082 [1] |

[1] unvalidated analytical data
( aerial application

Table 41. Parathion residues in lettuce from supervised trials in the USA.

| Sample, State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| Bibb lettuce. CA, 1988 (Dark Green Boston) | WP<br>WP | 0.28<br>0.56 | | 370<br>370 | 6<br>6 | 14<br>21 | excluding wrapper leaves | 1.2 1.6<br>1.1 0.83 | <0.05 (2)<br><0.05 (2) | EP-LE-1049 |
| Bibb lettuce. CA, 1988 (Dark Green Boston) | WP<br>WP | 0.28<br>0.56 | | 370<br>370 | 6<br>6 | 14<br>21 | including wrapper leaves | 2.5 2.8<br>1.3 0.99 | <0.05 0.06<br><0.05 (2) | EP-LE-1049 |

| Sample, State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| Bibb lettuce. CA, 1988 (Dark Green Boston) | EC<br>EC | 0.28<br>0.56 | | 370<br>370 | 6<br>6 | 14<br>21 | excluding wrapper leaves | 1.2 1.3<br>0.96 0.76 | <0.05 (2)<br><0.05 (2) | EP-LE-1050 |
| Bibb lettuce. CA, 1988 (Dark Green Boston) | EC<br>EC | 0.28<br>0.56 | | 370<br>370 | 6<br>6 | 14<br>21 | including wrapper leaves | 1.7 2.4<br>2.1 1.6 | 0.05 <0.05<br><0.05 (2) | EP-LE-1050 |
| Bibb lettuce. CA, 1988 (Allermaran Butter) | EC | 0.56 | | 200 | ( 4 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-LE-1051 |
| Bibb lettuce. CA, 1988 (Allermaran Butter) | EC | 0.28 | | 200 | ( 4 | 14 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-LE-1051 |
| Bibb lettuce. CA, 1988 (Allermaran Butter) | EC<br>EC | 0.28<br>0.56 | | 200<br>200 | ( 4<br>( 4 | 14<br>21 | including wrapper leaves | <0.05 0.06<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-LE-1051 |
| Head lettuce. CA, 1988 (Salinas) | EC | 0.56 | | 370 | 6 | 7 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-LE-1052 |
| Head lettuce. CA, 1988 (Salinas) | EC | 0.56 | | 370 | 6 | 7 | including wrapper leaves | 0.90 0.90 | <0.05 (2) | EP-LE-1052 |
| Head lettuce. CA, 1988 (Salinas) | WP | 0.56 | | 370 | 6 | 7 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-LE-1053 |
| Head lettuce. CA, 1988 (Salinas) | WP | 0.56 | | 370 | 6 | 7 | including wrapper leaves | 0.85 0.60 | <0.05 (2) | EP-LE-1053 |
| Head lettuce. CA, 1988 (Salinas) | EC | 0.56 | | 200 | ( 6 | 7 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-LE-1054 |
| Head lettuce. CA, 1988 (Salinas) | EC | 0.56 | | 200 | ( 6 | 7 | including wrapper leaves | 0.49 0.53 | <0.05 (2) | EP-LE-1054 |
| Leaf lettuce. CA, 1988 (Green Leaf) | EC | 0.28 | | 200 | ( 4 | 14 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-LE-1055 |
| Leaf lettuce. CA, 1988 (Green Leaf) | EC | 0.56 | | 200 | ( 4 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | EP-LE-1055 |
| Leaf lettuce. CA, 1988 (Green Leaf) | EC<br>EC | 0.28<br>0.56 | | 200<br>200 | ( 4<br>( 4 | 14<br>21 | including wrapper leaves | 0.10 0.06<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-LE-1055 |
| Leaf lettuce. CA, 1989 (Royal Green Mountain) | WP | 0.28 | | 370 | 6 | 14 | excluding wrapper leaves | 1.4 0.75 | 0.06 <0.05 | EP-LE-1056 |
| Leaf lettuce. CA, 1989 (Royal Green Mountain) | WP | 0.56 | | 370 | 6 | 21 | excluding wrapper leaves | 0.25 0.25 | <0.05 (2) | EP-LE-1056 |

| Sample, State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| Leaf lettuce. CA, 1989 (Royal Green Mountain) | WP<br>WP | 0.28<br>0.56 | | 370<br>370 | 6<br>6 | 14<br>21 | including wrapper leaves | 2.0 2.8<br>0.72 0.97 | <0.05 0.09<br><0.05 (2) | EP-LE-1056 |
| Leaf lettuce. CA, 1988 (Royal Green Mountain) | EC<br>EC | 0.28<br>0.56 | | 370<br>370 | 6<br>6 | 14<br>21 | excluding wrapper leaves | 0.77 1.0<br>0.28 0.31 | <0.05 (2)<br><0.05 (2) | EP-LE-1057 |
| Leaf lettuce. CA, 1988 (Royal Green Mountain) | EC<br>EC | 0.28<br>0.56 | | 370<br>370 | 6<br>6 | 14<br>21 | including wrapper leaves | 1.1 0.81<br>0.98 1.2 | <0.05 (2)<br><0.05 (2) | EP-LE-1057 |
| Head lettuce. FL, 1987 (Ithaca) | EC | 0.61 | | 710 | 6 | 7<br>7 | including wrapper leaves | 0.60 0.06<br>0.65 0.07 | <0.05 (2)<br><0.05 (2) | EP-LE-5047 |
| Head lettuce. FL, 1987 (Ithaca) | WP | 0.61 | | 720 | 6 | 7<br>7 | including wrapper leaves | <0.05 0.62<br><0.05 0.28 | <0.05 (2)<br><0.05 (2) | EP-LE-5048 |
| Head lettuce. FL, 1987 (Ithaca) | EC | 0.56 | | 72 | ( 6 | 7<br>7 | including wrapper leaves | <0.05 (2)<br><0.05 0.16 | <0.05 (2)<br><0.05 (2) | EP-LE-5049 |
| Head lettuce. NY, 1987 (Ithaca) | EC | 1×6.7 preplant +6×0.56 | | 230 | 6 | 7<br>7 | including wrapper leaves | <0.05 1.1<br>c 1.1 | <0.05 (2)<br>c <0.05 | EP-LE-5051 |
| Head lettuce. NY, 1987 (Ithaca) | WP | 1×6.7EC preplant +6×0.56 | | 230 | 6 | 7 | including wrapper leaves | 1.3 1.6 | <0.05 (2) | EP-LE-5052 |
| Leaf lettuce. FL, 1987 (Salad Bowl) | EC<br>EC | 0.30<br>0.60 | | 720<br>720 | 6<br>6 | 14<br>21<br>14 | including wrapper leaves | 0.35 0.09<br>0.34 0.65<br>c 0.05 | <0.05 (2)<br><0.05 (2)<br>c <0.05 | EP-LE-5053 |
| Leaf lettuce. FL, 1987 (Salad Bowl) | WP<br>WP | 0.30<br>0.59 | | 720<br>720 | 6<br>6 | 14<br>21<br>14 | including wrapper leaves | 0.42 <0.05<br>0.48 0.90<br>c 1.3 | <0.05 (2)<br><0.05 (2)<br>c <0.05 | EP-LE-5054 |
| Leaf lettuce. FL, 1988 (Royal Red Leaf) | EC<br>EC | 0.28<br>0.56 | | 54<br>54 | ( 6<br>( 6 | 14<br>21 | including wrapper leaves | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-LE-5055 |
| Leaf lettuce. NY, 1987 (Black Seeded Simpson) | EC | 1×6.7 preplant +6×0.28 | | 230 | 6 | 14 | including wrapper leaves | 0.06 <0.05 | <0.05 (2) | EP-LE-5057-A |
| Leaf lettuce. NY, 1987 (Black Seeded Simpson) | EC | 1×6.7 preplant +6×0.56 | | 230 | 6 | 21 | including wrapper leaves | 0.05 0.07 | <0.05 (2) | EP-LE-5057-B |
| Leaf lettuce. NY, 1987 (Black Seeded Simpson) | WP | 1×6.7EC preplant +6×0.28 | | 230 | 6 | 14 | including wrapper leaves | 0.07 0.07 | <0.05 (2) | EP-LE-5058-A |
| Leaf lettuce. NY, 1987 (Black Seeded Simpson) | WP | 1×6.7EC preplant +6×0.56 | | 230 | 6 | 21 | including wrapper leaves | 0.11 0.14 | <0.05 (2) | EP-LE-5058-B |

| Sample, State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| Bibb lettuce. FL, 1987 (Bibb) | EC<br>EC | 0.30<br>0.60 | | 720<br>720 | 6<br>6 | 14<br>21 | including wrapper leaves | 0.57 0.32<br>0.14 <0.05 | <0.05 (2)<br><0.05 (2) | EP-LE-5059 |
| Bibb lettuce. FL, 1987 (Bibb) | WP | 0.30 | | 720 | 6 | 14<br>14 | including wrapper leaves | 0.56 <0.05<br>c 0.49 | <0.05 (2)<br>c <0.05 | EP-LE-5060 |
| Bibb lettuce. FL, 1987 (Flori-Bibb) | WP | 0.60 | | 720 | 6 | 21 | including wrapper leaves | 0.13 0.49 | <0.05 (2) | EP-LE-5060 |
| Bibb lettuce. FL, 1988 (Flori-Bibb) | EC<br>EC | 0.28<br>0.56 | | 54<br>54 | ( 6<br>( 6 | 14<br>21 | including wrapper leaves | <0.05 (2)<br>0.10 0.08 | <0.05 (2)<br><0.05 (2) | EP-LE-5061 |
| Bibb lettuce. NY, 1987 (Buttercrunch) | EC | 1×6.7 preplant +6×0.28 | | 230 | 6 | 14 | including wrapper leaves | 0.22 0.29 | <0.05 (2) | EP-LE-5063-A |
| Bibb lettuce. NY, 1987 (Buttercrunch) | EC | 1×6.7 preplant +6×0.56 | | 230 | 6 | 21 | including wrapper leaves | 0.28 0.31 | <0.05 (2) | EP-LE-5063-B |
| Bibb lettuce. NY, 1987 (Buttercrunch) | WP | 1×6.7EC preplant +6×0.28 | | 230 | 6 | 14 | including wrapper leaves | 0.27 0.35 | <0.05 (2) | EP-LE-5064-A |
| Bibb lettuce. NY, 1987 (Buttercrunch) | WP | 1×6.7EC preplant +6×0.56 | | 230 | 6 | 21 | including wrapper leaves | 0.44 0.32 | <0.05 (2) | EP-LE-5064-B |

( aerial application c: sample from control plot

Table 42. Parathion residues in spinach from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | Sample | parathion | paraoxon | |
| NJ, 1988 (America) | WP | 0.56 | | 540 | 6 | 14 | greens | 0.56 0.61 | 0.09 0.09 | EP-SP-5176 |
| CA, 1988 (St Helens) | WP | 0.56 | | 280 | 6 | 14 | greens | 0.12 0.15 | 0.06 0.06 | EP-SP-1093 |
| TX, 1987 (Dixie Market) | WP | 0.56 | | 150 | 6 | 14 | greens | 0.36 0.28 | 0.09 0.07 | EP-SP-1096 |
| CA, 1988 (St Helens) | EC | 0.56 | | 190 | ( 6 | 14 | greens | 0.12 0.12 | 0.09 0.16 | EP-SP-1094 |

( aerial application

Table 43. Parathion residues in snap beans from supervised trials in the USA. The samples were stored for 680-700 days before analysis, which is acceptable for parathion residues, but excessive for paraoxon in snap beans. The duplicate values are for duplicate field samples.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | Sample | parathion | paraoxon | |
| OR, 1987 (Roma II) | WP | 0.90 | | 110 | 3 | 15 | pods | 0.17 0.39 | <0.05 (2) | EP-LB-1130 |
| OR, 1987 (Roma II) | WP | 0.56 | | 110 | 3 | 7 | pods | 0.09 0.23 | 0.06 0.07 | EP-LB-1130 |
| OR, 1987 (Roma II) | EC<br>EC | 0.56<br>0.90 | | 120 | 3 | 7<br>15 | pods | 0.14 0.14<br>0.11 0.08 | <0.05 (2)<br>0.05 0.05 | EP-LB-1131 |
| OR, 1987 (OSU91) | EC<br>EC | 0.56<br>0.90 | | 110 | ( 3<br>( 3 | 7<br>15 | pods | <0.05 0.05<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-LB-1133 |
| NY, 1987 (Improved Tendergreen) | EC<br>EC | 0.56<br>0.90 | | 280 | 6 | 7<br>15 | pods | 0.06 <0.05<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-LB-5083 |
| NY, 1987 (Improved Tendergreen) | WP<br>WP | 0.56<br>0.90 | | 280 | 6<br>6 | 7<br>15 | pods | <0.05 0.05<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-LB-5084 |
| WI, 1987 (FLO) | EC<br>EC | 0.56<br>0.90 | | 60 | ( 6<br>( 6 | 7<br>15 | pods | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-LB-5085 |
| WI, 1987 (FLO) | EC<br>EC | 0.56<br>0.90 | | 260 | 6<br>6 | 7<br>15 | pods | <0.05 0.08<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-LB-5087 |
| WI, 1987 (FLO) | WP<br>WP | 0.56<br>0.90 | | 260 | 6<br>6 | 7<br>15 | pods | 0.07 <0.05<br><0.05 0.05 | <0.05 (2)<br><0.05 (2) | EP-LB-5088 |

( aerial application

Table 44. Parathion residues in dry beans from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1987 (Kidney) | WP<br>WP | 0.56<br>0.90 | | 280<br>280 | 6 | 7<br>14 | pods | 0.05 <0.05<br>0.17 0.13 | <0.05 (2)<br>0.06 <0.05 | EP-DB-1113 |
| CA, 1987 (Kidney) | EC<br>EC | 0.56<br>0.90 | | 280<br>280 | 6<br>6 | 7<br>14 | pods | 0.13 0.06<br>0.06 <0.05 | <0.05 (2)<br><0.05 (2) | EP-DB-1114 |
| CA, 1987 (Kidney) | EC<br>EC | 0.56<br>0.90 | | 94<br>94 | ( 6<br>( 6 | 7<br>14 | pods | <0.05 (2)<br><0.05 (2) | 0.05 <0.05<br><0.05 (2) | EP-DB-1115 |
| ID, 1988 (Pinto) | EC<br>EC | 0.56<br>0.90 | | 94<br>94 | ( 6<br>( 6 | 7<br>15 | pods | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-DB-1116 |
| ID, 1988 (Pinto) | WP<br>WP | 0.56<br>0.90 | | 190<br>190 | 6 | 7<br>15 | pods | <0.05 (2)<br><0.05 (2) | <0.05 0.05<br><0.05 (2) | EP-DB-1118 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| ID, 1988 (Pinto) | EC<br>EC | 0.56<br>0.90 | | 190<br>190 | 6<br>6 | 7<br>15 | pods | <0.05 (2)<br><0.05 0.26 | <0.05 (2)<br><0.05 0.27 | EP-DB-1119 |
| MI, 1988 (Seafarer Navy Bean) | EC<br>EC | 0.56<br>0.90 | | 215<br>215 | 6<br>6 | 15<br>15 | beans | <0.05<br><0.05 | <0.05<br><0.05 | EP-DB-5142 |
| MI, 1988 (Seafarer Navy Bean) | EC<br>EC | 0.56<br>0.90 | | 215<br>215 | 6<br>6 | 7<br>7 | pods | 0.11<br>0.12 | <0.05<br><0.05 | EP-DB-5142 |
| MI, 1988 (Seafarer Navy Bean) | WP<br>WP | 0.56<br>0.90 | | 215<br>215 | 6<br>6 | 15<br>15 | beans | <0.05<br><0.05 | <0.05<br><0.05 | EP-DB-5143 |
| MI, 1988 (Seafarer Navy Bean) | WP<br>WP | 0.56<br>0.90 | | 215<br>215 | 6<br>6 | 7<br>7 | pods | 0.07<br>0.095 | <0.05<br><0.05 | EP-DB-5143 |
| NE, 1988 (Pinto) | EC | 0.56 | | 190 | 7 | 7<br>15 | dry seed | <0.05<br><0.05 | <0.05<br><0.05 | EP-DB-5144 |
| NE, 1988 (Pinto) | EC | 0.90 | | 190 | 7 | 7<br>15 | dry seed | <0.05<br><0.05 | <0.05<br><0.05 | EP-DB-5144 |
| NE, 1988 (Pinto) | WP | 0.56 | | 190 | 7 | 7<br>15 | dry seed | <0.05<br><0.05 | <0.05<br><0.05 | EP-DB-5145 |
| NE, 1988 (Pinto) | WP | 0.90 | | 190 | 7 | 7<br>15 | dry seed | <0.05<br><0.05 | <0.05<br><0.05 | EP-DB-5145 |

( aerial application

Table 45. Parathion residues in soya beans from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| IL, 1988 (BSR 201) | EC | 0.90 | | 240 | 2 | 20 | dry seed | <0.05 (2) | <0.05 (2) | EP-SY-5197 |
| IL, 1988 (BSR 201) | WP | 0.90 | | 240 | 2 | 20 | dry seed | <0.05 (2) | <0.05 (2) | EP-SY-5198 |
| IL, 1988 (BSR 201) | EC | 0.90 | | 9 | ( 2 | 20 | dry seed | <0.05 (2) | <0.05 (2) | EP-SY-5199 |
| MN, 1988 (AP 1776) | EC | 0.90 | | 190 | 2 | 20 | dry seed | <0.05 (2) | <0.05 (2) | EP-SY-5200 |
| MN, 1988 (AP 1776) | WP | 0.90 | | 190 | 2 | 20 | dry seed | <0.05 (2) | <0.05 (2) | EP-SY-5201 |
| MN, 1988 (AP 1776) | EC | 0.90 | | 37 | ( 2 | 20 | dry seed | <0.05 (2) | <0.05 (2) | EP-SY-5202 |
| GA, 1988 (Coker 488) | EC | 0.90 | | 61 | 2 | 20 | dry seed | <0.05 (2) | <0.05 (2) | EP-SY-5203 |
| GA, 1988 (Coker 488) | WP | 0.90 | | 61 | 2 | 20 | dry seed | <0.05 (2) | <0.05 (2) | EP-SY-5204 |

( aerial application

Table 46. Parathion residues in carrots from supervised trials in the USA..

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| CA, 1988 (Champion) | EC | 1.1 | | 190 | 6 | 15 | 0.26 0.41 | <0.05 (2) | EP-CT-1015 [1] |
| CA, 1988 (Champion) | WP | 1.1 | | 190 | 6 | 15 | 0.35 0.47 | <0.05 (2) | EP-CT-1016 [1] |
| CA, 1988 (Champion) | EC | 1.1 | | 190 | ( 6 | 15 | 0.17 0.18 | <0.05 (2) | EP-CT-1017 [1] |
| TX, 1987 (Imperator #58) | WP | 1.1 | | 150 | 6 | 15 | 0.26 0.33 | <0.05 (2) | EP-CT-1018 [1] |
| TX, 1987 (Imperator #58) | EC | 1.1 | | 150 | 6 | 15 | 0.62 0.29 | <0.05 (2) | EP-CT-1019 [1] |
| TX, 1987 (Danver) | EC | 1.1 | | 47 | ( 6 | 15 | 0.05 0.08 | <0.05 (2) | EP-CT-1021 [1] |
| WA, 1988 (Montes Coulesa) | WP | 0.28 | | 280 | 6 | 15 | <0.05 0.07 | <0.05 (2) | EP-CT-1174 [1] |
| WA, 1988 (Montes Coulesa) | EC | 1.1 | | 280 | 6 | 15 | 0.06 0.23 | <0.05 (2) | EP-CT-1175 [1] |
| MI, 1987 (Scarlet Nantes) | EC | 1.1 | | 50 | 6 | 15 | 0.29 0.34 | <0.05 (2) | EP-CT-5099 [1] |
| MI, 1987 (Scarlet Nantes) | WP | 1.1 | | 50 | 6 | 15 | 0.17 0.19 | <0.05 (2) | EP-CT-5100 [1] |

[1] unvalidated analytical data
( aerial application

Table 47. Parathion residues in potatoes from supervised trials in the USA.

| State, year (variety) | Application | | | PHI, days | Commodity | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | | parathion | paraoxon | |
| ID, 1988 (Russet Burbank) | EC | 1.1 | ( 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | EP-PO-1194 [1] |
| ID, 1988 (Russet Burbank) | WP | 1.1 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | EP-PO-1196 [1] |
| ID, 1988 (Russet Burbank) | EC | 1.1 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | EP-PO-1197 [1] |
| ID, 1988 (Russet Burbank) | EC | 1.1 | 6 | 5 | tuber | <0.05 | <0.05 | EP-PO-2001 [1] |
| WA, 1988 (Russet Burbank) | EC | 1.1 | 6 | 5 | tuber | <0.05 | <0.05 | EP-PO-2002 [1] |
| ME, 1988 (Atlantic) | EC | 1.2 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | EP-PO-5039 [1] |
| ME, 1988 (Atlantic) | WP | 1.2 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | EP-PO-5040 [1] |
| ND, 1988 (Norchip) | EC | 1.1 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | EP-PO-5041 [1] |

| State, year (variety) | Application | | | PHI, days | Commodity | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | | parathion | paraoxon | |
| ND, 1988 (Norchip) | WP | 1.1 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | EP-PO-5042 [1] |
| WI, 1988 | EC | 1.1 | ( 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | EP-PO-5043 [1] |
| WI, 1987 (Norchip) | EC | 1.1 | 6 | 5 | tuber [2] | <0.05 (2) | <0.05 (2) | EP-PO-5045 [1] |
| WI, 1987 (Norchip) | WP | 1.1 | 6 | 5 | tuber [2] | 0.10 0.10 | <0.05 (2) | EP-PO-5046 [1] |

[1] unvalidated analytical data
[2] samples stored for 2 years before analysis.
( aerial application

Table 48. Parathion residues in radishes from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1988 (Comet) | WP | 0.56 | | 280 | 3 | 15 | roots | <0.05 0.11 | <0.05 (2) | EP-RD-1083 [1] |
| CA, 1988 (Comet) | WP | 0.56 | | 280 | 3 | 15 | roots and tops | 1.0 0.97 | <0.05 (2) | EP-RD-1083 [1] |
| CA, 1988 (Comet) | EC | 0.56 | | 280 | 3 | 15 | roots | 0.17 0.15 | <0.05 (2) | EP-RD-1084 [1] |
| CA, 1988 (Comet) | EC | 0.56 | | 280 | 3 | 15 | roots and tops | 1.1 1.2 | <0.05 (2) | EP-RD-1084 [1] |
| CA, 1988 (Comet) | EC | 0.56 | | 190 | ( 2 | 15 | roots | <0.05 (2) | <0.05 (2) | EP-RD-1085 [1] |
| CA, 1988 (Comet) | EC | 0.56 | | 190 | ( 2 | 15 | roots and tops | 0.21 0.33 | <0.05 (2) | EP-RD-1085 [1] |
| MN, 1987 (Red Scarlet Globe) | EC | 0.56 | | 47 | 3 | 15 | tops | 0.15 0.21 | <0.05 (2) | EP-RD-5095 [1] |
| MN, 1987 (Red Scarlet Globe) | EC | 0.56 | | 47 | 3 | 15 | roots | 0.05 0.07 | <0.05 (2) | EP-RD-5095 [1] |
| MN, 1987 (Red Scarlet Globe) | WP | 0.56 | | 47 | 3 | 15 | tops | 0.35 0.25 | <0.05 (2) | EP-RD-5096 [1] |
| MN, 1987 (Red Scarlet Globe) | WP | 0.56 | | 47 | 3 | 15 | roots | 0.09 0.07 | <0.05 (2) | EP-RD-5096 [1] |
| MI, 1987 (Cherry Belle) | EC | 0.56 | | 50 | 3 | 15 | tops | 3.6 2.8 | 0.11 0.06 | EP-RD-5097 [1] |
| MI, 1987 (Cherry Belle) | EC | 0.56 | | 50 | 3 | 15 | roots | 0.28 0.15 | <0.05 (2) | EP-RD-5097 [1] |
| MI, 1987 (Cherry Belle) | WP | 0.56 | | 50 | 3 | 15 | tops | 2.7 3.0 | 0.06 0.06 | EP-RD-5098 [1] |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| MI, 1987 (Cherry Belle) | WP | 0.56 | | 50 | 3 | 15 | roots | 0.15 0.22 | <0.05 (2) | EP-RD-5098 [1] |
| FL, 1987 (Red Globe) | EC | 0.56 | | 720 | 3 | 15 | tops | 0.64 1.6 2.6 | <0.05 (2) 0.07 | EP-RD-5124 [1] |
| FL, 1987 (Red Globe) | EC | 0.56 | | 720 | 3 | 15 | roots | <0.05 (3) | <0.05 (3) | EP-RD-5124 [1] |
| FL, 1987 (Red Globe) | WP | 0.56 | | 720 | 3 | 15 | tops | 3.0 0.48 2.9 | 0.08 <0.05 0.13 | EP-RD-5125 [1] |
| FL, 1987 (Red Globe) | WP | 0.56 | | 720 | 3 | 15 | roots | <0.05 (3) | <0.05 (3) | EP-RD-5125 [1] |
| FL, 1987 (Red Globe) | EC | 0.56 | | 72 | ( 3 | 15 | tops | 0.23 0.16 0.07 | <0.05 (3) | EP-RD-5126 [1] |
| FL, 1987 (Red Globe) | EC | 0.56 | | 72 | ( 3 | 15 | roots | <0.05 (3) | <0.05 (3) | EP-RD-5126 [1] |

[1] unvalidated analytical data
( aerial application

Table 49. Parathion residues in sugar beet roots from supervised trials in the USA.

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | parathion | paraoxon | |
| CA, 1988 (SS NB2) | EC | 0.90 | ( 6 | 15 | <0.05 (2) | <0.05 (2) | EP-SB-1097 [1] |
| CA, 1988 (SS NB2) | WP | 0.90 | 6 | 15 | <0.05 (2) | <0.05 (2) | EP-SB-1098 [1] |
| CA, 1988 (SS NB2) | EC | 0.90 | 6 | 15 | <0.05 (2) | <0.05 (2) | EP-SB-1099 [1] |
| ID, 1988 (WS 88) | EC | 0.90 | 6 | 15 | <0.05 (2) | <0.05 (2) | EP-SB-1125 [1] |
| ID, 1988 (WS 88) | WP | 0.90 | 6 | 15 | <0.05 (2) | <0.05 (2) | EP-SB-1126 [1] |
| CA, 1988 (SS NB2) | EC | 0.90 | 6 | 15 | <0.05 | <0.05 | EP-SB-2003 [1] |
| ND, 1988 (ACS ACH176) | EC | 0.90 | 6 | 15 | 0.06 | <0.05 | EP-SB-2004 [1] |
| MN, 1988 (Ultramono) | EC | 0.90 | 6 | 15 | 0.06 0.08 | <0.05 (2) | EP-SB-5089 [1] |
| MN, 1988 (Ultramono) | WP | 0.90 | 6 | 15 | 0.08 0.07 | <0.05 (2) | EP-SB-5090 [1] |
| ND, 1988 (ACS ACH176) | EC | 0.90 | 6 | 15 | 0.07 <0.05 | <0.05 (2) | EP-SB-5177 [1] |
| ND, 1988 (ACS ACH176) | WP | 0.90 | 6 | 15 | 0.07 0.05 | <0.05 (2) | EP-SB-5178 [1] |
| MN, 1988 (Ultramono) | EC | 0.90 | ( 6 | 15 | 0.05 <0.05 | <0.05 (2) | EP-SB-5179 [1] |

[1] unvalidated analytical data
( aerial application

Table 50. Parathion residues in Chinese turnips from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| TX, 1987 (Purple Top White Globe) | EC | 0.56 | | 47 | ( 6 | 21 | greens | 0.13 0.20 | <0.05 (2) | EP-TU-1103 [1] |
| TX, 1987 (Purple Top White Globe) | EC | 0.28 | | 47 | ( 6 | 7 | greens | 0.30 1.9 | <0.05 (2) | EP-TU-1103 [1] |
| TX, 1987 (Purple Top White Globe) | EC | 0.56 | | 150 | 6 | 21 | greens | 0.06 <0.05 | <0.05 (2) | EP-TU-1104 [1] |
| TX, 1987 (Purple Top White Globe) | EC | 0.28 | | 150 | 6 | 7 | greens | 0.47 0.26 | <0.05 (2) | EP-TU-1104 [1] |
| TX, 1987 (Purple Top White Globe) | WP | 0.56 | | 150 | 6 | 21 | greens | 0.06 <0.05 | <0.05 (2) | EP-TU-1105 [1] |
| TX, 1987 (Purple Top White Globe) | WP | 0.28 | | 150 | 6 | 7 | greens | 0.35 0.75 | <0.05 (2) | EP-TU-1105 [1] |
| CA, 1988 (Purple Top White Globe) | EC | 0.56 | | 190 | ( 6 | 21 | greens | 0.15 0.18 | <0.05 (2) | EP-TU-1106 [1] |
| CA, 1988 (Purple Top White Globe) | EC | 0.28 | | 190 | ( 6 | 7 | greens | 3.2 2.9 | 0.26 0.25 | EP-TU-1106 [1] |
| CA, 1988 (Purple Top White Globe) | WP | 0.56 | | 280 | 6 | 21 | greens | 0.10 0.08 | <0.05 (2) | EP-TU-1107 [1] |
| CA, 1988 (Purple Top White Globe) | WP | 0.28 | | 280 | 6 | 7 | greens | 0.46 0.77 | <0.05 (2) | EP-TU-1107 [1] |
| CA, 1988 (Purple Top White Globe) | EC | 0.56 | | 280 | 6 | 21 | greens | 0.12 0.14 | <0.05 (2) | EP-TU-1108 [1] |
| CA, 1988 (Purple Top White Globe) | EC | 0.28 | | 280 | 6 | 7 | greens | 0.63 0.73 | <0.05 (2) | EP-TU-1108 [1] |
| OH, 1988 (Purple Top White Globe) | EC | 0.28 | | 240 | 6 | 7 | greens | 0.34 0.23 | <0.05 (2) | EP-TU-5183 [1] |
| OH, 1988 (Purple Top White Globe) | EC | 0.56 | | 240 | 6 | 10 | greens | 0.12 0.20 | <0.05 (2) | EP-TU-5183 [1] |
| OH, 1988 (Purple Top White Globe) | WP | 0.28 | | 240 | 6 | 7 | greens | 0.08 0.07 | <0.05 (2) | EP-TU-5184 [1] |
| OH, 1988 (Purple Top White Globe) | WP | 0.56 | | 240 | 6 | 10 | greens | <0.05 0.10 | <0.05 (2) | EP-TU-5184 [1] |
| GA, 1988 (Purple Globe) | EC | 0.56 | | 290 | 6 | 10 | greens | 0.79 0.95 | <0.05 (2) | EP-TU-5185 [1] |
| GA, 1988 (Purple Globe) | EC | 0.28 | | 290 | 6 | 7 | greens | 0.52 0.53 | <0.05 (2) | EP-TU-5185 [1] |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| GA, 1988 (Purple Globe) | WP | 0.56 | | 290 | 6 | 10 | greens | 0.23 0.26 | <0.05 0.05 | EP-TU-5186 [1] |
| GA, 1988 (Purple Globe) | WP | 0.28 | | 290 | 6 | 7 | greens | 0.31 0.32 | <0.05 (2) | EP-TU-5186 [1] |
| NJ, 1988 (Purple Top (Agway)) | EC | 0.56 | | 137 | 6 | 7 | greens | 1.3 0.96 | <0.05 (2) | EP-TU-5187 [1] |
| NJ, 1988 (Purple Top (Agway)) | EC | 1.1 | | 137 | 6 | 10 | greens | 1.5 1.4 | <0.05 (2) | EP-TU-5187 [1] |
| NJ, 1988 (Purple Top (Agway)) | WP | 0.28 | | 137 | 6 | 7 | greens | 0.23 0.43 | <0.05 (2) | EP-TU-5188 [1] |
| NJ, 1988 (Purple Top (Agway)) | WP | 0.56 | | 137 | 6 | 10 | greens | 0.34 0.50 | <0.05 (2) | EP-TU-5188 [1] |
| OH, 1988 (Purple Top White Globe) | EC | 0.28 | | 240 | 6 | 7 | root | <0.05 (2) | <0.05 (2) | EP-TU-5183 [1] |
| OH, 1988 (Purple Top White Globe) | EC | 0.56 | | 240 | 6 | 10 | root | <0.05 (2) | <0.05 (2) | EP-TU-5183 [1] |
| OH, 1988 (Purple Top White Globe) | WP | 0.28 | | 240 | 6 | 7 | root | <0.05 (2) | <0.05 (2) | EP-TU-5184 [1] |
| OH, 1988 (Purple Top White Globe) | WP | 0.56 | | 240 | 6 | 10 | root | <0.05 (2) | <0.05 (2) | EP-TU-5184 [1] |
| GA, 1988 (Purple Globe) | EC | 0.56 | | 290 | 6 | 10 | root | <0.05 (2) | 0.06 0.05 | EP-TU-5185 [1] |
| GA, 1988 (Purple Globe) | EC | 0.28 | | 290 | 6 | 7 | root | <0.05 (2) | <0.05 (2) | EP-TU-5185 [1] |
| GA, 1988 (Purple Globe) | WP | 0.56 | | 290 | 6 | 10 | root | <0.05 (2) | <0.05 0.05 | EP-TU-5186 [1] |
| GA, 1988 (Purple Globe) | WP | 0.28 | | 290 | 6 | 7 | root | <0.05 (2) | <0.05 (2) | EP-TU-5186 [1] |
| NJ, 1988 (Purple Top (Agway)) | EC | 0.56 | | 137 | 6 | 7 | root | 0.08 0.08 | <0.05 (2) | EP-TU-5187 [1] |
| NJ, 1988 (Purple Top (Agway)) | EC | 1.1 | | 137 | 6 | 10 | root | <0.05 0.06 | <0.05 (2) | EP-TU-5187 [1] |
| NJ, 1988 (Purple Top (Agway)) | WP | 0.28 | | 137 | 6 | 7 | root | <0.05 (2) | <0.05 (2) | EP-TU-5188 [1] |
| NJ, 1988 (Purple Top (Agway)) | WP | 0.56 | | 137 | 6 | 10 | root | <0.05 (2) | <0.05 (2) | EP-TU-5188 [1] |

[1] unvalidated analytical data
( aerial application

Table 51. Parathion residues in celery from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1989 (5270R) | EC | 0.28 | | 190 | ( 2 | 15<br>22 | excluding foliage | 0.15 0.13<br>0.10 0.10 | <0.05 (2)<br><0.05 (2) | EP-CY-1232 (a) |
| CA, 1989 (5270R) | EC | 0.56 | | 190 | ( 2 | 21<br>28 | excluding foliage | 0.19 0.10<br>0.14 0.18 | <0.05 (2)<br><0.05 (2) | EP-CY-1232 (b) |
| CA, 1988 (Florida 683) | EC | 0.28 | | 370 | ( 2 | 15<br>22 | excluding foliage | 0.06 0.08<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-1233 (a) |
| CA, 1988 (Florida 683) | EC | 0.56 | | 370 | ( 2 | 21<br>28 | excluding foliage | 0.10 0.07<br>0.06 0.08 | <0.05 (2)<br><0.05 (2) | EP-CY-1233 (b) |
| CA, 1988 (Florida 683) | EC | 1.1 | | 370 | ( 2 | 30<br>37 | excluding foliage | 0.12 0.08<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-1233 (c) |
| CA, 1988 (Florida 683) | WP | 0.28 | | 370 | 2 | 15<br>22 | excluding foliage | 0.07 <0.05<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-1234 (a) |
| CA, 1988 (Florida 683) | WP | 0.56 | | 370 | 2 | 21<br>28 | excluding foliage | 0.07 0.06<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-1234 (b) |
| CA, 1988 (Florida 683) | WP | 1.1 | | 370 | 2 | 30<br>37 | excluding foliage | 0.11 0.18<br>0.08 0.12 | <0.05 (2)<br><0.05 (2) | EP-CY-1234 (c) |
| FL, 1988 (#683) | EC | 0.28 | | 71 | ( 2 | 15<br>22 | excluding foliage | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-5103 (a) |
| FL, 1988 (#683) | EC | 0.56 | | 71 | ( 2 | 21<br>28 | excluding foliage | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-5103 (b) |
| FL, 1988 (#683) | EC | 1.1 | | 71 | ( 2 | 30<br>37 | excluding foliage | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-5103 (c) |
| MI, 1988 (Utah Tall 52-70R1MP) | EC | 0.28 | | 215 | 2 | 15<br>22 | excluding foliage | 0.09 0.11<br>0.09 0.10 | 0.10 0.12<br>0.12 0.12 | EP-CY-5105 (a) |
| MI, 1988 (Utah Tall 52-70R1MP) | EC | 0.56 | | 215 | 2 | 21<br>28 | excluding foliage | 0.12 0.10<br>0.07 <0.05 | <0.05 (2)<br><0.05 (2) | EP-CY-5105 (b) |
| MI, 1988 (Utah Tall 52-70R1MP) | EC | 1.1 | | 215 | 2 | 30<br>37 | excluding foliage | 0.14 0.10<br>0.11 0.12 | <0.05 (2)<br><0.05 (2) | EP-CY-5105 (c) |
| MI, 1988 (Utah Tall 52-70R1MP) | WP | 0.28 | | 215 | 2 | 15<br>22 | excluding foliage | 0.06 <0.05<br><0.05 0.06 | <0.05 (2)<br><0.05 (2) | EP-CY-5106 (a) |
| MI, 1988 (Utah Tall 52-70R1MP) | WP | 0.56 | | 215 | 2 | 21<br>28 | excluding foliage | 0.09 0.10<br><0.05 0.06 | <0.05 (2)<br>0.07 <0.05 | EP-CY-5106 (b) |
| MI, 1988 (Utah Tall 52-70R1MP) | WP | 1.1 | | 215 | 2 | 30<br>37 | excluding foliage | 0.15 0.16<br>0.17 0.13 | <0.05 (2)<br><0.05 (2) | EP-CY-5106 (c) |
| FL, 1988 (#683) | EC | 0.28 | | 270 | 6 | 15<br>22 | excluding foliage | 0.08 0.09<br>0.05 0.08 | <0.05 (2)<br><0.05 (2) | EP-CY-5213 (a) |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| FL, 1988 (#683) | EC | 0.56 | | 270 | 6 | 21<br>28 | excluding foliage | 0.11 0.07<br>0.06 0.06 | <0.05 (2)<br><0.05 (2) | EP-CY-5213 (b) |
| FL, 1988 (#683) | EC | 1.1 | | 270 | 6 | 30<br>37 | excluding foliage | 0.17 0.28<br>0.14 <0.05 | <0.05 (2)<br><0.05 (2) | EP-CY-5213 (c) |
| FL, 1988 (#683) | WP | 0.28 | | 270 | 6 | 15<br>22 | excluding foliage | 0.06 <0.05<br>0.07 0.08 | <0.05 (2)<br><0.05 (2) | EP-CY-5214 (a) |
| FL, 1988 (#683) | WP | 0.56 | | 270 | 6 | 21<br>28 | excluding foliage | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-5214 (b) |
| FL, 1988 (#683) | WP | 1.1 | | 270 | 6 | 30<br>37 | excluding foliage | <0.05 0.08<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-5214 (c) |
| CA, 1987 (Tall Utah 5275) | WP | 0.28 | | 370 | 6 | 15 | including foliage | 0.20 0.38 | <0.05 (2) | EP-CY-1022 (a) |
| CA, 1987 (Tall Utah 5275) | WP | 0.56 | | 370 | 6 | 21 | including foliage | 0.58 0.51 | <0.05 (2) | EP-CY-1022 (b) |
| CA, 1987 (Tall Utah 5275) | WP | 1.1 | | 370 | 6 | 30 | including foliage | 2.4 2.5 | 0.05 0.06 | EP-CY-1022 (c) |
| CA, 1987 (Tall Utah 5275) | EC | 0.28 | | 370 | 6 | 15 | including foliage | 0.33 0.80 | <0.05 (2) | EP-CY-1023 (a) |
| CA, 1987 (Tall Utah 5275) | EC | 0.56 | | 370 | 6 | 21 | including foliage | 0.46 0.35 | <0.05 (2) | EP-CY-1023 (b) |
| CA, 1987 (Tall Utah 5275) | EC | 1.1 | | 370 | 6 | 30 | including foliage | 1.0 1.7 | <0.05 (2) | EP-CY-1023 (c) |
| CA, 1987 (Tall Utah 5275) | EC | 0.28 | | 190 | ( 6 | 15 | including foliage | 1.7 0.60 | 0.07 <0.05 | EP-CY-1024 (a) |
| CA, 1987 (Tall Utah 5275) | EC | 0.56 | | 190 | ( 6 | 21 | including foliage | 1.8 0.31 | 0.06 <0.05 | EP-CY-1024 (b) |
| CA, 1987 (Tall Utah 5275) | EC | 1.1 | | 190 | ( 6 | 30 | including foliage | 0.88 1.5 | <0.05 (2) | EP-CY-1024 (c) |
| CA, 1989 (5270R) | EC | 0.28 | | 190 | ( 2 | 15<br>22 | including foliage | 0.17 0.46<br>0.18 0.14 | <0.05 (2)<br><0.05 (2) | EP-CY-1232 (a) |
| CA, 1989 (5270R) | EC | 0.56 | | 190 | ( 2 | 21<br>28 | including foliage | 0.72 0.55<br>0.13 0.22 | <0.05 (2)<br><0.05 (2) | EP-CY-1232 (b) |
| CA, 1988 (Florida 683) | EC | 0.28 | | 370 | 2 | 15<br>22 | including foliage | 1.3 0.33<br>0.18 0.63 | <0.05 (2)<br><0.05 (2) | EP-CY-1233 (a) |
| CA, 1988 (Florida 683) | EC | 0.56 | | 370 | 2 | 21<br>28 | including foliage | 0.33 0.53<br>0.16 0.18 | <0.05 (2)<br><0.05 (2) | EP-CY-1233 (b) |
| CA, 1988 (Florida 683) | EC | 1.1 | | 370 | 2 | 30<br>37 | including foliage | 0.15 0.20<br>0.17 0.20 | <0.05 (2)<br><0.05 (2) | EP-CY-1233 (c) |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1988 (Florida 683) | WP | 0.28 | | 370 | 2 | 15<br>22 | including foliage | 0.13 0.74<br>0.06 0.12 | <0.05 (2)<br><0.05 (2) | EP-CY-1234 (a) |
| CA, 1988 (Florida 683) | WP | 0.56 | | 370 | 2 | 21<br>28 | including foliage | 0.11 0.49<br><0.05 0.08 | <0.05 (2)<br><0.05 (2) | EP-CY-1234 (b) |
| CA, 1988 (Florida 683) | WP | 1.1 | | 370 | 2 | 30<br>37 | including foliage | 0.23 0.26<br>0.07 0.13 | <0.05 (2)<br><0.05 (2) | EP-CY-1234 (c) |
| FL, 1988 (5270R) | EC | 0.30 | | 720 | 6 | 15 | including foliage | 12 12 | 0.16 0.16 | EP-CY-5101 (a) |
| FL, 1988 (5270R) | EC | 0.61 | | 720 | 6 | 21 | including foliage | 7.8 9.9 | 0.12 0.12 | EP-CY-5101 (b) |
| FL, 1988 (5270R) | EC | 1.2 | | 720 | 6 | 30 | including foliage | 8.9 5.9 | 0.14 0.11 | EP-CY-5101 (c) |
| FL, 1988 (5270R) | WP | 0.30 | | 720 | 6 | 15 | including foliage | 9.3 8.3 | 0.12 0.11 | EP-CY-5102 (a) |
| FL, 1988 (5270R) | WP | 0.61 | | 720 | 6 | 21 | including foliage | 8.6 9.0 | 0.11 0.12 | EP-CY-5102 (b) |
| FL, 1988 (5270R) | WP | 1.2 | | 720 | 6 | 30 | including foliage | 6.3 5.7 | 0.24 0.20 | EP-CY-5102 (c) |
| FL, 1988 (#683) | EC | 0.28 | | 71 | ( 2 | 15<br>22 | including foliage | 0.23 0.12<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-5103 (a) |
| FL, 1988 (#683) | EC | 0.56 | | 71 | ( 2 | 21<br>28 | including foliage | 0.16 0.07<br>0.05 <0.05 | <0.05 (2)<br><0.05 (2) | EP-CY-5103 (b) |
| FL, 1988 (#683) | EC | 1.1 | | 71 | ( 2 | 30<br>37 | including foliage | 0.09 0.11<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-CY-5103 (c) |
| MI, 1988 (Utah Tall 52-70R1MP) | EC | 0.28 | | 215 | 2 | 15<br>22 | including foliage | 1.0 0.92<br>1.5 1.2 | 1.1 1.6<br>1.3 0.68 | EP-CY-5105 (a) |
| MI, 1988 (Utah Tall 52-70R1MP) | EC | 0.56 | | 215 | 2 | 21<br>28 | including foliage | 2.2 1.4<br>1.5 1.2 | 0.58 0.30<br>0.36 0.23 | EP-CY-5105 (b) |
| MI, 1988 (Utah Tall 52-70R1MP) | EC | 1.1 | | 215 | 2 | 30<br>37 | including foliage | 2.1 1.3<br>1.4 0.78 | 0.09 0.05<br>0.06 <0.05 | EP-CY-5105 (c) |
| MI, 1988 (Utah Tall 52-70R1MP) | WP | 0.28 | | 215 | 2 | 15<br>22 | including foliage | 0.60 1.5<br>0.70 0.92 | 0.16 0.22<br>0.14 0.12 | EP-CY-5106 (a) |
| MI, 1988 (Utah Tall 52-70R1MP) | WP | 0.56 | | 215 | 2 | 21<br>28 | including foliage | 2.3 1.9<br>0.68 0.78 | 0.45 0.39<br>0.47 0.19 | EP-CY-5106 (b) |
| MI, 1988 (Utah Tall 52-70R1MP) | WP | 1.1 | | 215 | 2 | 30<br>37 | including foliage | 2.2 2.0<br>1.8 0.69 | 0.07 0.06<br>0.08 <0.05 | EP-CY-5106 (c) |
| FL, 1988 (#683) | EC | 0.28 | | 270 | 6 | 15<br>22 | including foliage | 0.26 0.12<br>0.34 0.37 | <0.05 (2)<br><0.05 (2) | EP-CY-5213 (a) |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| FL, 1988 (#683) | EC | 0.56 | | 270 | 6 | 21<br>28 | including foliage | 0.45 0.18<br>0.17 0.18 | <0.05 (2)<br><0.05 (2) | EP-CY-5213 (b) |
| FL, 1988 (#683) | EC | 1.1 | | 270 | 6 | 30<br>37 | including foliage | 1.0 0.96<br>0.46 0.64 | 0.08 <0.05<br><0.05 0.06 | EP-CY-5213 (c) |
| FL, 1988 (#683) | WP | 0.28 | | 270 | 6 | 15<br>22 | including foliage | 1.1 1.3<br>0.50 0.70 | 0.05 0.08<br><0.05 0.05 | EP-CY-5214 (a) |
| FL, 1988 (#683) | WP | 0.56 | | 270 | 6 | 21<br>28 | including foliage | 0.34 0.12<br><0.05 0.10 | 0.08 <0.05<br><0.05 (2) | EP-CY-5214 (b) |
| FL, 1988 (#683) | WP | 1.1 | | 270 | 6 | 30<br>37 | including foliage | 0.20 1.0<br>0.29 0.06 | <0.05 0.05<br><0.05 (2) | EP-CY-5214 (c) |

( aerial application

Table 52. Parathion residues in almonds from supervised trials in the USA. Kernels analysed.

| State, year (variety) | Application | | | | | Growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1988 (Non-Pareil) | WP | 1.4 | 0.060 | 2300 | 1 | dormant | 180 | <0.05 (2) | <0.05 (2) | EP-AL-1148 (a) |
| CA, 1988 (Non-Pareil) | WP | 2.8 | | 650 | 1 | dormant | 180 | <0.05 (2) | <0.05 (2) | EP-AL-1148 (b) |
| CA, 1988 (Non-Pareil) | WP | 1.4 | 0.060 | 2300 | 3 | 1-2% hull split | 14 | <0.05 0.06 | <0.05 0.33 | EP-AL-1148 (c) |
| CA, 1988 (Non-Pareil) | WP | 2.8 | | 650 | 3 | 1-2% hull split | 14 | 0.06 0.09 | <0.05 (2) | EP-AL-1148 (d) |
| CA, 1988 (Non-Pareil) | EC | 1.4 | 0.060 | 2300 | 1 | dormant | 180 | <0.05 (2) | <0.05 (2) | EP-AL-1149 (a) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 650 | 1 | dormant | 180 | <0.05 (2) | <0.05 (2) | EP-AL-1149 (b) |
| CA, 1988 (Non-Pareil) | EC | 1.4 | 0.060 | 2300 | 3 | 1-2% hull split | 14 | <0.05 0.06 | 0.16 0.19 | EP-AL-1149 (c) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 650 | 3 | 1-2% hull split | 14 | 0.06 0.06 | 0.07 0.05 | EP-AL-1149 (d) |
| CA, 1988 (Non-Pareil) | WP | 1.4 | 0.060 | 2300 | 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1150 (a) |
| CA, 1988 (Non-Pareil) | WP | 2.8 | | 650 | 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1150 (a) |
| CA, 1988 (Non-Pareil) | WP | 1.4 | 0.060 | 2300 | 3 | 1-2% hull split | 14 | 0.06 <0.05 | <0.05 (2) | EP-AL-1150 (c) |

| State, year (variety) | Application | | | | | Growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1988 (Non-Pareil) | WP | 2.8 | | 650 | 3 | 1-2% hull split | 14 | 0.07 0.08 | <0.05 (2) | EP-AL-1150 (d) |
| CA, 1988 (Non-Pareil) | EC | 1.4 | 0.060 | 2300 | 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1151 (a) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 650 | 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1151 (b) |
| CA, 1988 (Non-Pareil) | EC | 1.4 | 0.060 | 2300 | 3 | 1-2% hull split | 14 | 0.08 0.05 | 0.24 0.22 | EP-AL-1151 (c) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 650 | 3 | 1-2% hull split | 14 | 0.06 0.08 | 0.09 0.06 | EP-AL-1151 (d) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 190 | ( 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1152 (a) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 190 | ( 3 | 1-2% hull split | 14 | <0.05 (2) | <0.05 (2) | EP-AL-1152 (b) |
| CA, 1989 (Non-Pareil) | EC | 2.8 | | 190 | ( 3 | 1 week pre-hulls | 28 | <0.05 (2) | <0.05 (2) | EP-AL-1237 (a) |
| CA, 1989 (Non-Pareil) | EC | 2.8 | | 190 | ( 1 | dormant | 181 | <0.05 (2) | <0.05 (2) | EP-AL-1237 (b) |
| CA, 1989 (Non-Pareil) | EC | 2.5 | 0.060 | 4100 | 3 | 1 week pre-hulls | 28 | <0.05 (2) | <0.05 (2) | EP-AL-1238 (a) |
| CA, 1989 (Non-Pareil) | EC | 2.8 | | 420 | 3 | 1 week pre-hulls | 28 | <0.05 (2) | <0.05 (2) | EP-AL-1238 (b) |
| CA, 1989 (Non-Pareil) | EC | 2.5 | 0.060 | 4100 | 1 | dormant | 186 | <0.05 (2) | <0.05 (2) | EP-AL-1238 (c) |
| CA, 1989 (Non-Pareil) | EC | 2.8 | | 420 | 1 | dormant | 186 | <0.05 (2) | <0.05 (2) | EP-AL-1238 (d) |
| CA, 1989 (Non-Pareil) | WP | 2.5 | 0.060 | 4100 | 3 | 1 week pre-hulls | 28 | <0.05 (2) | <0.05 (2) | EP-AL-1239 (a) |
| CA, 1989 (Non-Pareil) | WP | 2.8 | | 420 | 3 | 1 week pre-hulls | 28 | <0.05 (2) | <0.05 (2) | EP-AL-1239 (b) |
| CA, 1989 (Non-Pareil) | WP | 2.5 | 0.060 | 4100 | 1 | dormant | 186 | <0.05 (2) | <0.05 (2) | EP-AL-1239 (c) |
| CA, 1989 (Non-Pareil) | WP | 2.8 | | 420 | 1 | dormant | 186 | <0.05 (2) | <0.05 (2) | EP-AL-1239 (d) |

( aerial application

Table 53. Parathion residues in pecans and walnuts from supervised trials in the USA. Walnut samples had been stored for approximately 970 days before analysis, which exceeds the 2 years storage stability demonstrated on almond kernels.

| State, year (variety) | Application | | | | | growth stage | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| Pecans | | | | | | | | | | | |
| TX, 1988 (Desirable) | WP | 4.0 | 0.11 | 3400 | 4 | full develop | 15 | nuts | <0.05 (2) | <0.05 (2) | EP-PC-1070 [1] |
| TX, 1988 (Desirable) | WP | 2.8 | 0.60 | 470 | 4 | full develop | 15 | nuts | <0.05 (2) | <0.05 (2) | EP-PC-1070 [1] |
| GA, 1987 (Stuart) | WP | 1.0 | 0.11 | 840 | 6 | shuck split | 15 | nuts and shells | <0.05 (2) | <0.05 (2) | EP-PC-5114 [1] |
| GA, 1987 (Stuart) | WP | 2.8 | 0.33 | 840 | 6 | shuck split | 15 | nuts and shells | <0.05 (2) | <0.05 (2) | EP-PC-5114 [1] |
| TX, 1988 (Desirable) | EC | 3.8 | 0.11 | 3400 | 4 | full develop | 15 | nuts | <0.05 (2) | <0.05 (2) | EP-PC-1071 [1] |
| GA, 1987 (Stuart) | EC | 1.0 | 0.12 | 840 | 6 | shuck split | 15 | nuts and shells | <0.05 (2) | <0.05 (2) | EP-PC-5113 [1] |
| Walnuts | | | | | | | | | | | |
| CA, 1987 (Sunland) | EC | 8.4 | | 2300 | 3 | mature nuts | 14 | nut in shell | <0.05 (2) | <0.05 (2) | EP-WA-1110 [1] |
| CA, 1987 (Sunland) | EC | 11.2 | | 470 | 3 | mature nuts | 14 | nut in shell | <0.05 (2) | <0.05 (2) | EP-WA-1110 [1] |
| CA, 1987 (Sunland) | EC | 11.2 | | 190 | ( 3 | mature nuts | 14 | nut in shell | <0.05 (2) | <0.05 (2) | EP-WA-1111 [1] |

[1] unvalidated analytical data
( aerial application

Table 54. Parathion residues in barley grain from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| MT, 1997 (Lewis) | EC | 0.84 | | 54 | ( 6 | 16 | 0.075 <u>0.15</u> | <0.01 <u>0.01</u> | MGB 97003.MT2 (b) |
| MT, 1997 (Pirolene) | EC | 0.84 | | 54 | ( 6 | 16 | 0.24 <u>0.25</u> | 0.02 <u>0.02</u> | MGB 97003.MT1 (b) |
| ND, 1997 (Robust) | EC | 0.84 | | 45 | ( 6 | 15 | <u>0.78</u> 0.59 | <u>0.03</u> 0.03 | MGB 97003.ND2 (b) |
| ND, 1997 (Stander) | EC | 0.84 | | 45 | ( 6 | 15 | <u>0.54</u> 0.47 | <u>0.07</u> 0.06 | MGB 97003.ND1 (b) |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| WI, 1997 (Robust) | EC | 0.84 | | 19 | ( 6 | 14 | 2.2 2.2 | 0.092 0.083 | MGB 97003.WI1 (c) |
| NY, 1997 | EC | 0.84 | | 28 | ( 6 | 15 | 1.6 1.4 | 0.095 0.075 | MGB 97003.NY1 (b) |
| MN, 1997 (Hazen) | EC | 0.81 | | 19 | ( 6 | 15 | 1.3 1.2 | 0.11 0.087 | MGB 97003.MN2 (b) |
| UT, 1997 (Idagold) | EC | 0.84 | | 32 | ( 6 | 14 | 1.9 2.2 | 0.11 0.12 | MGB 97003.UT1 (b) |
| ID, 1997 (Colter) | EC | 0.84 | | 32 | ( 6 | 15 | 3.5 4.9 | 0.15 0.19 | MGB 97003.ID1 (b) |
| MN, 1997 (Chilton) | EC | 0.81 | | 19 | ( 6 | 15 | 2.0 1.9 | 0.17 0.17 | MGB 97003.MN1 (b) |
| ID, 1997 (Russell) | EC | 0.84 | | 32 | ( 6 | 15 | 4.1 2.6 | 0.29 0.23 | MGB 97003.ID2 (b) |
| AZ, 1998 (Orea) | EC | 0.86 | | 48 | ( 6 | 14 | 3.3 3.0 | 0.29 0.25 | MGB 97003.AZ1 (b) |

( aerial application

Table 55. Parathion residues in maize from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| IA, 1989 (DeKalb 547) | EC | 1.1 | | 190 | 5 | 12 | 0.06 | <0.05 | EP-CN-2042 |
| MO, 1989 (Funks G-4500) | EC | 1.1 | | 190 | 6 | 12 | <0.05 | <0.05 | EP-CN-2043 |
| IL, 1987 (Asgrow 788) | EC | 1.1 | | 49 | ( 6 | 12 | <0.05 (2) [1] | <0.05 (2) [1] | EP-CN-5027 |
| IL, 1987 (Asgrow 788) | EC | 1.1 | | 190 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5029 |
| IL, 1987 (Asgrow 788) | WP | 1.1 | | 190 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5030 |
| MN, 1988 (Pioneer 3906) | EC | 1.1 | | 37 | ( 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5031 |
| MN, 1987 (Pioneer 3969) | EC | 1.1 | | 190 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5033 |
| MN, 1987 (Pioneer 3969) | WP | 1.1 | | 190 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5034 |
| NE, 1987 (Pioneer 3475) | EC | 1.1 | | 190 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5035 |
| NE, 1987 (Pioneer 3475) | WP | 1.1 | | 190 | 6 | 12 | <0.05 0.09 | <0.05 (2) | EP-CN-5036 |
| OH, 1987 (DeKalb 636) | EC | 1.1 | | 150 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5037 |
| OH, 1987 (DeKalb 636) | WP | 1.1 | | 150 | 6 | 12 | <0.05 (2) | <0.05 (2) | EP-CN-5038 |

( aerial application
[1] samples stored for 790 days between harvest and analysis.

Table 56. Parathion residues in rice grain from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| TX, 1987 (Lemont) | EC | 1.1 | | 206 | 3 | 1 | 4.0 3.2 | 0.72 0.69 | EP-RI-1088 |
| CA, 1988 (L202) | EC | 0.11 | | 75 | ( 3 | 1 | 0.28 0.33 | 0.07 0.05 | EP-RI-1235 |
| TX, 1987 (Lemont) | EC | 0.11 | | 47 | ( 3 | 1 | 0.064 0.059 | <0.05 (2) | EP-RI-1236 |
| LA, 1987 (Lemont) | EC | 1.1 | | 250 | 6 | 1 | 11 11 | 1.7 1.7 | EP-RI-5071 |
| LA, 1987 (Lemont) | EC | 1.1 | | 75 | ( 6 | 1 | 5.1 | 0.53 | EP-RI-5072 |
| LA, 1988 (Lemont) | EC | 0.11 | | 94 | ( 6 | 1 | 0.21 0.23 | <0.05 (2) | EP-RI-5215 |
| LA, 1988 (Lemont) | EC | 0.11 | | 240 | 6 | 1 | 0.90 0.90 | 0.42 0.39 | EP-RI-5216 |

( aerial application c: sample from control plot

Table 57. Parathion residues in sorghum grain from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| TX, 1994 | CS | 1.1 | | 44 | ( 2 | 12<br>21<br>28 | 0.29<br>0.17<br>0.14 | <0.01<br><0.01<br><0.01 | 94-TX-GS-989-01 (b) |
| TX, 1994 | EC | 1.1 | | 44 | ( 2 | 12<br>21<br>28 | 0.44<br>0.54<br>0.51 | 0.01<br>0.01<br>0.01 | 94-TX-GS-988-01 (b) |
| KS, 1992 (NK 2030) | EC | 0.56 | | 44 | ( 2 | 12<br>21<br>28 | 0.24 0.36<br>0.27 0.29<br>0.18 0.18 | 0.04 0.04<br>0.05 0.05<br>0.04 0.03 | 92150b-4 |
| MO, 1992 (Topaz) | EC | 1.1 | | 45 | ( 2 | 12<br>21<br>28 | 0.71 0.69<br>0.44 0.49<br>0.23 0.37 | 0.051 0.05<br>0.04 0.04<br>0.03 0.04 | 92150f-8 |
| NE, 1992 (NK 2030/Pioneer 8379) | EC | 1.1 | | 47 | ( 2 | 12<br>21<br>28 | 1.5 2.0<br>1.5 1.6<br>1.1 1.3 | 0.052 0.052<br>0.04 0.05<br>0.03 0.05 | 92150a-4 |
| TX, 1992 (Pioneer 8313) | EC | 1.1 | | 46 | ( 2 | 12<br>21<br>28 | 1.3 1.3<br>0.87 0.86<br>0.64 0.68 | 0.053 0.068<br>0.03 0.03<br>0.03 0.03 | 92150d-4 |
| OK, 1992 (NK 2030) | EC | 1.1 | | 47 | ( 2 | 12<br>21<br>28 | 0.69 0.60<br>0.19 0.23<br>0.11 0.09 | 0.06 0.06<br>0.03 0.04<br><0.05 (2) | 92150c-4 |
| TX, 1987 (PAG6670) | EC | 1.1 | | 28 | ( 6 | 12 | 0.61 0.59 | 0.12 0.13 | EP-SG-1090 |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| KS, 1987 (Paymaster 1022) | EC | 1.1 | | 47 | 6 | 12 | 1.6 1.3 | 0.17 0.16 | EP-SG-5091 |
| KS, 1987 (Paymaster 1022) | WP | 1.1 | | 47 | 6 | 12 | 1.7 1.7 | 0.18 0.19 | EP-SG-5092 |
| TX, 1987 (NK2660) | EC | 1.1 | | 47 | 6 | 12 | 0.85 0.85 | 0.20 0.16 | EP-SG-1092 |
| SD, 1992 (NK 1210) | EC | 1.1 | | 51 | ( 2 | 12<br>21<br>28 | 3.3 3.2<br>2.4 1.8<br>1.8 1.5 | 0.20 0.18<br>0.17 0.12<br>0.17 0.12 | 92150e-4 |
| TX, 1987 (NK2660) | WP | 1.1 | | 47 | 6 | 12 | 0.70 0.79 | 0.25 0.23 | EP-SG-1091 |
| KS, 1987 (Paymaster 1022) | EC | 1.1 | | 9 | ( 6 | 12 | 3.8 3.8 | 0.38 0.39 | EP-SG-5093 |

( aerial application

Table 58. Parathion residues in wheat grain from supervised trials in the USA. Residues are expressed on a fresh weight basis. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| WHEAT | | | | | | | | | |
| MN, 1994 (Krona) | EC | 0.84 | | 47 | ( 2 | 15<br>21<br>36 | 0.08<br>0.07<br>0.04 | <0.01<br><0.01<br><0.01 | 94-MN-WH-735-02 |
| MN, 1994 (Krona) | CS | 0.84 | | 47 | ( 2 | 15<br>21<br>36 | 0.11<br>0.10<br>0.07 | <0.01<br><0.01<br><0.01 | 94-MN-WH-736-02 |
| ND, 1994 (Pioneer 2375) | EC | 0.84 | | 47 | ( 2 | 15<br>21<br>36 | 0.02<br><0.01<br>0.02 | <0.01<br><0.01<br><0.01 | 94-ND-WH-735-01 |
| ND, 1994 (Pioneer 2375) | CS | 0.84 | | 47 | ( 2 | 16<br>21<br>36 | 0.05<br>0.04<br>0.02 | <0.01<br><0.01<br><0.01 | 94-ND-WH-736-01 |
| CA, 1988 (Anza) | EC | 1.1 | | 94 | ( 6 | 15 | 0.44 0.75 | <0.05 (2) | EP-WH-1219 [1] |
| CA, 1988 (Anza) | WP | 1.1 | | 190 | 6 | 15 | 0.29 0.29 | <0.05 (2) | EP-WH-1220 [1] |
| CA, 1988 (Anza) | EC | 1.1 | | 190 | 6 | 15 | 0.61 0.60 | <0.05 (2) | EP-WH-1221 [1] |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| WA, 1988 (Stephens) | WP | 1.1 | | 110 | 6 | 15 | 0.11 0.10 | <0.05 (2) | EP-WH-1222 [1] |
| WA, 1988 (Stephens) | EC | 1.1 | | 110 | 6 | 15 | 0.13 0.12 | <0.05 (2) | EP-WH-1223 [1] |
| MO, 1989 (Caldwell) | WP | 0.90 | | 190 | 6 | 15 | 0.68 0.80 | <0.05 (2) | EP-WH-1253 [1] |
| KS, 1988 (Pioneer 2157) | EC | 1.1 | | 120 | 6 | 15 | 1.0 0.94 | <0.05 (2) | EP-WH-5189 [1] |
| KS, 1988 (Pioneer 2157) | WP | 1.1 | | 120 | 6 | 15 | 0.86 0.78 | <0.05 (2) | EP-WH-5190 [1] |
| MO, 1988 (Caldwell) | EC | 1.1 | | 26 | ( 6 | 15 | <0.05 (2) | <0.05 (2) | EP-WH-5191 [1] |
| MO, 1988 (Caldwell) | EC | 1.1 | | 190 | 6 | 15 | 1.3 1.1 | <0.05 (2) | EP-WH-5193 [1] |
| ND, 1988 (Marshall) | EC | 1.1 | | 94 | 6 | 15 | 0.18 0.19 | <0.05 (2) | EP-WH-5195 [1] |
| ND, 1988 (Marshall) | WP | 1.1 | | 94 | 6 | 15 | 0.12 0.13 | <0.05 (2) | EP-WH-5196 [1] |
| SPRING WHEAT | | | | | | | | | |
| WA, 1992 (Penawawa) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | 0.32 0.32<br>0.25 0.28<br>0.28 0.24 | <0.02 (2)<br><0.02 ndr<br>ndr (2) | 92148a (a) |
| WA, 1992 (Penawawa) | EC | 0.85 | | 47 | ( 2 | 15<br>20<br>25 | 0.54 0.53<br>0.45 0.40<br>0.30 0.49 | <0.02 (2)<br><0.02 (2)<br>ndr <0.02 | 92148a (b) |
| ID, 1992 (Penawawa) | EC | 0.56 | | 49 | ( 2 | 15<br>20<br>25 | 0.48 0.46<br>0.51 0.51<br>0.49 0.56 | <0.02 (2)<br><0.02 (2)<br>0.02 0.02 | 92148b (a) |
| ID, 1992 (Penawawa) | EC | 0.85 | | 49 | ( 2 | 15<br>20<br>25 | 0.74 0.67<br>0.79 0.80<br>0.92 0.6 | 0.02 0.02<br>0.03 0.02<br>0.04 0.03 | 92148b (b) |
| MT, 1992 (Amadon) | EC | 0.56<br>-0.69 | | 48, 57 | ( 2 | 15<br>25 | 0.05 0.04<br>0.04 0.05 | ndr (2)<br>ndr (2) | 92148c (a) |
| MT, 1992 (Amadon) | EC | 0.90<br>-0.93 | | 51 | ( 2 | 15<br>25 | 0.05 0.059<br>0.05 0.04 | ndr (2)<br>ndr (2) | 92148c (b) |
| ND, 1992 (Gus) | EC | 0.56 | | 46 | ( 2 | 15<br>20<br>25 | 0.03 0.02<br>0.02 <0.02<br><0.02 (2) | <0.02 (2)<br>ndr (2)<br>ndr (2) | 92148d (a) |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| ND, 1992 (Gus) | EC | 0.84 | | 46 | ( 2 | 15<br>20<br>25 | 0.074 0.04<br>0.04 0.05<br>0.02 0.02 | <0.02 (2)<br><0.02 (2)<br>ndr (2) | 92148d (b) |
| MN, 1992 (Vance) | EC | 0.56 | | 46 | ( 2 | 15<br>20<br>25 | 0.075 0.062<br>0.02 0.02<br>0.04 0.04 | ndr (2)<br>ndr (2)<br>ndr (2) | 92148e (a) |
| MN, 1992 (Vance) | EC | 0.84 | | 46 | ( 2 | 15<br>20<br>25 | 0.16 0.15<br>0.062 0.12<br>0.12 0.11 | <0.05 (2)<br>ndr (2)<br>ndr (2) | 92148e (b) |
| SD, 1992 (Bute 86) | EC | 0.60<br>+0.48 | | 51<br>+40 | ( 2 | 15<br>20<br>25 | 0.29 0.30<br>0.18 0.19<br>0.18 0.17 | <0.02 (2)<br>ndr (2)<br>ndr (2) | 92148f (a) |
| SD, 1992 (Bute 86) | EC | 0.88<br>+0.80 | | 50<br>+44 | ( 2 | 15<br>20<br>25 | 0.57 0.63<br>0.50 0.48<br>0.43 0.45 | 0.02 0.02<br><0.02 (2)<br><0.02 (2) | 92148f (b) |
| WINTER WHEAT | | | | | | | | | |
| KS, 1993 (Karl) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | 0.03 0.03<br><0.02 (2)<br><0.02 (2) | <0.02 (2)<br>ndr (2)<br>ndr (2) | 93240a (c) |
| KS, 1993 (Karl) | EC | 0.83 | | 47 | ( 2 | 15<br>20<br>25 | 0.12 0.095<br>0.03 0.03<br>0.04 <0.02 | 0.03 0.03<br>ndr (2)<br>ndr (2) | 93240a (d) |
| TX, 1993 (DK 49 S) | EC | 0.61 | | 55 | ( 2 | 15<br>20<br>25 | 0.04 0.03<br>0.04 0.04<br>0.04 0.03 | ndr (2)<br>ndr (2)<br>ndr (2) | 93240b (c) |
| TX, 1993 (DK 49 S) | EC | 0.90 | | 54 | ( 2 | 15<br>20<br>25 | 0.12 0.11<br>0.13 0.12<br>0.13 0.14 | <0.02 (2)<br>ndr (2)<br>ndr (2) | 93240b (d) |
| OK, 1993 (McNair) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | 0.12 0.10<br>0.11 0.086<br>0.075 0.062 | ndr (2)<br>ndr (2)<br>ndr (2) | 93240c (c) |
| OK, 1993 (McNair) | EC | 0.84 | | 47 | ( 2 | 15<br>20<br>25 | 0.20 0.20<br>0.21 0.17<br>0.14 0.14 | <0.02 (2)<br><0.02 (2)<br>ndr (2) | 93240c (d) |
| CO, 1993 (Buckskin) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | 0.02 <0.02<br>ndr <0.02<br><0.02 (2) | <0.02 (2)<br>ndr (2)<br>ndr (2) | 93240d (c) |
| CO, 1993 (Buckskin) | EC | 0.84 | | 47 | ( 2 | 15<br>20<br>25 | 0.05 0.062<br><0.02 (2)<br><0.02 (2) | 0.02 0.02<br><0.02 (2)<br>ndr (2) | 93240d (d) |
| NE, 1993 (Buckskin) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | 0.03 <0.02<br><0.02 (2)<br><0.02 (2) | <0.02 (2)<br>ndr (2)<br>ndr (2) | 93240e (c) |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| NE, 1993 (Buckskin) | EC | 0.84 | | 47 | ( 2 | 15<br>20<br>25 | 0.11 0.12<br>0.02 <0.02<br>0.03 0.03 | 0.03 0.04<br><0.02 (2)<br>ndr (2) | 93240e (d) |

[1] unvalidated analytical data
( aerial application

Table 59. Parathion residues in canola from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | growth stage | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| MT, 1992 (Tobin) | EC | 0.56 | | 51 | ( 2 | early flowering | 28 | seed | 0.12 0.13 | <0.05 (2) | 92145b |
| MT, 1992 (Tobin) | EC | 1.1 | | 51 | ( 2 | early flowering | 28 | seed | 0.28 0.20 | <0.05 (2) | 92145b |
| WA, 1992 (Series) | EC | 0.56 | | 51 | ( 2 | post bloom | 28 | seed | <0.05 (2) | <0.05 (2) | 92145c |
| WA, 1992 (Series) | EC | 1.1 | | 51 | ( 2 | post bloom | 28 | seed | 0.23 0.19 | <0.05 (2) | 92145c |
| ID, 1992 (#104) | EC | 0.56 | | 42 | ( 2 | 5% bloom | 28 | seed | <0.05 (2) | <0.05 (2) | 92145d |
| ID, 1992 (#104) | EC | 1.1 | | 42 | ( 2 | 5% bloom | 28 | seed | <0.05 (2) | <0.05 (2) | 92145d |
| ND, 1992 (Legend) | EC | 2×0.56 +2×1.1 | | 47 | ( 4 | post flowering | 28 | seed | 0.17 0.17 | <0.05 (2) | 92145a |
| ND, 1992 (Legend) | EC | 2×0.28 +2×0.56 | | 47 | ( 4 | post flowering | 28 | seed | 0.073 0.088 | <0.05 (2) | 92145a |
| GA, 1994 (Iris) | EC | 0.50 | | 43 | ( 2 | mature seed | 28 | seed | 0.12 0.081 | | 94361a |

( aerial application

Table 60. Parathion residues in cotton seed from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | Growth stage | PHI, days | Sample | % moisture | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | | parathion | paraoxon | |
| AR, 1997 (PM 1220.BG,RR) | EC | 1.1 | | 37 | ( 6 | mature | 8 | seed | 13 | 0.15 0.10 | <0.01 (2) | MGB 97004.AR1 |
| AR, 1997 (NuCotn 33B) | EC | 1.1 | | 37 | ( 6 | mature | 8 | seed | 14 | 0.19 0.16 | <0.01 (2) | MGB 97004.AR2 |

| State, year (variety) | Application | | | | | Growth stage | PHI, days | Sample | % moisture | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | | parathion | paraoxon | |
| AZ, 1997 (Delta Pine 50) | EC | 1.1 | | 47 | ( 6 | bolls open | 7 | seed | 9 | 0.19 0.20 | 0.01 0.01 | MGB 97004.AZ1 |
| AZ, 1997 (DPL 33B) | EC | 1.1 | | 64 | ( 6 | top bolls open | 6 | seed | 11 | 0.20 0.17 | 0.01 0.02 | MGB 97004.AZ2 |
| AZ, 1997 (DPL BT35) | EC | 1.1 | | 45 | ( 6 | upper bolls open | 7 | seed | 13 | 0.65 0.61 | 0.02 0.02 | MGB 97004.AZ3 |
| GA, 1997 (BollGuard 33B) | EC | 1.1 | | 23 | ( 6 | open bolls | 7 | seed | 11 | 0.78 1.3 | 0.03 0.05 | MGB 97004.GA1 |
| MS, 1997 (Suregrow 125) | EC | 1.1 | | 19 | ( 6 | 80% open | 7 | seed | 11 | 0.40 0.29 | 0.04 0.03 | MGB 97004.MS1 |
| TX, 1997 (Suregrow 125) | EC | 1.1 | | 28 | ( 6 | | | seed | 9 | 2.0 1.4 | 0.12 0.074 | MGB 97004.TX2 |
| TX, 1997 (Explorer) | EC | 1.1 | | 28 | ( 6 | 2 nodes above ? | 7 | seed | 11 | 1.1 0.63 | 0.03 0.01 | MGB 97004.TX2 |
| TX, 1997 (HS-26) | EC | 1.1 | | 28 | ( 6 | 5 nodes above ? | 7 | seed | 11 | 0.20 0.30 | <0.01 0.01 | MGB 97004.TX3 |
| TX, 1997 (HS-26) | EC | 1.1 | | 28 | ( 6 | 4 nodes above ? | 7 | seed | 9 | 0.33 0.32 | 0.054 0.04 | MGB 97004.TX4 |
| TX, 1997 (M-1) | EC | 1.1 | | 28 | ( 6 | 6 nodes above ? | 7 | seed | 9 | 0.11 0.13 | <0.01 (2) | MGB 97004.TX5 |
| CA, 1987 (GO510) | WP | 1.1 | | 280 | 6 | bolls open | 7 | seed cotton | | 0.56 0.97 | 0.10 0.21 | EP-CS-1025 |
| CA, 1987 (GO510) | EC | 1.4 | | 280 | 6 | immature | 7 | seed cotton | | 0.25 0.66 | <0.05 0.087 | EP-CS-1026 |
| TX, 1987 (Stoneville 825) | WP | 1.1 | | 47 | 6 | bolls open | 7 | seed cotton | | 0.12 0.15 | <0.05 (2) | EP-CS-1027 |
| TX, 1987 (Stoneville 825) | EC | 1.4 | | 47 | 6 | bolls ½ open | 7 | seed cotton | | 0.26 0.14 | <0.05 (2) | EP-CS-1028 |
| MS, 1987 (DES 119) | EC | 1.4 | | 94 | 6 | full boll | 7 | seed cotton | | 0.16 0.21 | <0.05 (2) | EP-CS-5073 |
| MS, 1987 (DES 119) | WP | 1.1 | | 94 | 6 | full boll | 7 | seed cotton | | 0.48 0.43 | <0.05 (2) | EP-CS-5074 |

( aerial application
seed cotton: lint + seed

Table 61. Parathion residues in sunflower seed from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| ND, 1989 (Sigco Hybrid 465A) | EC | 1.1 | | 190 | 3 | post-flower | 30 | <0.05 (2) | <0.05 (2) | EP-SS-1240 |
| ND, 1989 (Sigco Hybrid 465A) | WP | 1.1 | | 190 | 3 | post-flower | 30 | <0.05 (2) | <0.05 (2) | EP-SS-1241 |
| ND, 1989 (Sigco Hybrid 465A) | EC | 1.1 | | 47 | ( 3 | post-flower | 30 | <0.05 (2) | <0.05 (2) | EP-SS-1242 |
| TX, 1989 (Sun Valley 230) | EC | 1.1 | | 190 | 3 | 10th true leaf | 30 | <0.05 (2) | <0.05 (2) | EP-SS-1243 |
| TX, 1989 (Sun Valley 230) | WP | 0.1 | | 190 | 3 | 10th true leaf | 30 | <0.05 (2) | <0.05 (2) | EP-SS-1244 |
| TX, 1989 (Sun Valley 230) | EC | 1.1 | | 59 | ( 3 | 10th true leaf | 30 | <0.05 (2) | <0.05 (2) | EP-SS-1245 |
| ND, 1988 (Sigco Hybrid 465A) | EC | 1.1 | | 190 | 3 | post-flower | 30 | <0.05 (2) | <0.05 (2) | EP-SS-5181 |

( aerial application

Table 62. Parathion residues in barley hay and straw from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Sample | % moisture | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| ND, 1997 (Stander) | EC | 0.84 | | 45 | ( 6 | 15 | hay | 37 | 0.096 0.073 | 0.01 0.01 | MGB 97003.ND1 (a) |
| ND, 1997 (Robust) | EC | 0.84 | | 45 | ( 6 | 15 | hay | 33 | 0.072 0.097 | 0.02 0.02 | MGB 97003.ND2 (a) |
| NY, 1997 | EC | 0.84 | | 28 | ( 6 | 15 | hay | 27 | 0.059 0.18 | 0.02 0.03 | MGB 97003.NY1 (a) |
| MT, 1997 (Pirolene) | EC | 0.81 | | 54 | ( 6 | 14 | hay | 38 | 0.056 0.16 | <0.01 0.05 | MGB 97003.MT1 (a) |
| WI, 1997 (Robust) | EC | 0.78 | | 19 | ( 6 | 14 | hay | 31 | 0.54 0.55 | 0.03 0.04 | MGB 97003.WI1 (a) |
| ID, 1997 (Colter) | EC | 0.84 | | 32 | ( 6 | 14 | hay | 25 | 0.21 0.20 | 0.04 0.03 | MGB 97003.ID1 (a) |

| State, year (variety) | Application | | | | | PHI, days | Sample | % moisture | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| UT, 1997 (Idagold) | EC | 0.84 | | 32 | ( 6 | 14 | hay | 36 | 0.70 0.39 | 0.051 0.04 | MGB 97003.UT1 (a) |
| MN, 1997 (Chilton) | EC | 0.78 | | 19 | ( 6 | 16 | hay | 32 | 0.72 1.1 | 0.057 0.071 | MGB 97003.MN1 (a) |
| MT, 1997 (Lewis) | EC | 0.81 | | 54 | ( 6 | 14 | hay | 27 | 0.17 0.19 | 0.059 0.078 | MGB 97003.MT2 (a) |
| MN, 1997 (Hazen) | EC | 0.78 | | 19 | ( 6 | 16 | hay | 39 | 2.5 3.6 | 0.17 0.17 | MGB 97003.MN2 (a) |
| ID, 1997 (Russell) | EC | 0.84 | | 32 | ( 6 | 14 | hay | 27 | 0.73 0.72 | 0.54 0.50 | MGB 97003.ID2 (a) |
| AZ, 1998 (Orea) | EC | 0.85 | | 48 | ( 6 | 14 | hay | 28 | 4.7 3.7 | 1.1 0.93 | MGB 97003.AZ1 (a) |
| MT, 1997 (Pirolene) | EC | 0.84 | | 54 | ( 6 | 16 | straw | 43 | 0.72 0.48 | 0.10 0.04 | MGB 97003.MT1 (b) |
| MN, 1997 (Hazen) | EC | 0.81 | | 19 | ( 6 | 15 | straw | 50 | 1.3 1.3 | 0.25 0.25 | MGB 97003.MN2 (b) |
| MT, 1997 (Lewis) | EC | 0.84 | | 54 | ( 6 | 16 | straw | 39 | 0.61 0.56 | 0.33 0.25 | MGB 97003.MT2 (b) |
| ND, 1997 (Stander) | EC | 0.84 | | 45 | ( 6 | 15 | straw | 54 | 2.8 1.6 | 0.53 0.40 | MGB 97003.ND1 (b) |
| WI, 1997 (Robust) | EC | 0.84 | | 19 | ( 6 | 14 | straw | 47 | 6.0 7.6 | 0.61 0.66 | MGB 97003.WI1 (b) |
| NY, 1997 | EC | 0.84 | | 28 | ( 6 | 15 | straw | 31 | 3.5 2.6 | 0.73 0.34 | MGB 97003.NY1 (b) |
| ID, 1997 (Russell) | EC | 0.84 | | 32 | ( 6 | 15 | straw | 55 | 2.9 2.0 | 0.75 0.67 | MGB 97003.ID2 (b) |
| ND, 1997 (Robust) | EC | 0.84 | | 45 | ( 6 | 15 | straw | 51 | 3.5 2.7 | 0.77 0.59 | MGB 97003.ND2 (b) |
| ID, 1997 (Colter) | EC | 0.84 | | 32 | ( 6 | 15 | straw | 38 | 2.0 2.0 | 0.89 0.85 | MGB 97003.ID1 (b) |
| MN, 1997 (Chilton) | EC | 0.81 | | 19 | ( 6 | 15 | straw | 59 | 5.0 8.0 | 1.0 1.9 | MGB 97003.MN1 (b) |
| UT, 1997 (Idagold) | EC | 0.84 | | 32 | ( 6 | 14 | straw | 22 | 9.6 6.1 | 1.5 1.2 | MGB 97003.UT1 (b) |
| AZ, 1998 (Orea) | EC | 0.86 | | 48 | ( 6 | 14 | straw | 20 | 8.5 13 | 1.9 2.6 | MGB 97003.AZ1 (b) |

( aerial application

Table 63. Parathion residues in maize forage, fodder and silage from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| WA, 1989 (Jubilee) | EC | 1.1 | | 94 | ( 6 | 12 | fodder | <0.05 (2) | <0.05 (2) | EP-CN-1246 |
| WA, 1989 (Jubilee) | EC | 1.1 | | 190 | 6 | 12 | fodder | 0.26 0.74 | <0.05 0.06 | EP-CN-1247 |
| WA, 1989 (Jubilee) | WP | 1.1 | | 190 | 6 | 12 | fodder | 0.92 0.82 | 0.06 0.06 | EP-CN-1248 |
| FL, 1988 (Silver Queen) | EC | 1.1 | | 700 | 6 | 12 | fodder | 0.05 0.10 | 0.07 0.07 | EP-CN-5007 |
| FL, 1989 (Silver Queen) | WP | 1.1 | | 700 | 6 | 12 | fodder | <0.05 0.06 | 0.06 0.06 | EP-CN-5008 |
| FL, 1988 (Merrit) | EC | 1.1 | | 59 | ( 6 | 12 | fodder | 0.12 0.12 | 0.12 0.10 | EP-CN-5009 |
| NY, 1987 (Early Sunray) | EC | 1.1 | | 230 | 6 | 12 | fodder | 0.30 0.39 | 0.12 0.11 | EP-CN-5011 |
| NY, 1987 (Early Sunray) | WP | 1.1 | | 230 | 6 | 12 | fodder | 0.35 0.45 | 0.10 0.12 | EP-CN-5012 |
| WI, 1987 (Incredible) | EC | 1.1 | | 200 | 6 | 12 | fodder | 1.6 0.76 | 0.35 0.12 | EP-CN-5013 |
| WI, 1987 (Incredible) | WP | 1.1 | | 200 | 6 | 12 | fodder | 1.4 1.2 | 0.16 0.21 | EP-CN-5014 |
| IL, 1987 (Asgrow 788) | EC | 1.1 | | 49 | ( 6 | 12 | fodder [1] | 0.39 0.86 | <0.05 (2) | EP-CN-5027 |
| IL, 1987 (Asgrow 788) | EC | 1.1 | | 190 | ( 6 | 12 | fodder | 0.71 2.3 | 0.05 0.05 | EP-CN-5029 |
| IL, 1987 (Asgrow 788) | WP | 1.1 | | 190 | 6 | 12 | fodder | 5.7 6.3 | 0.56 0.43 | EP-CN-5030 |
| MN, 1988 (Pioneer 3906) | EC | 1.1 | | 37 | ( 6 | 12 | fodder | 6.5 8.0 | 1.5 1.2 | EP-CN-5031 |
| MN, 1988 (Pioneer 3969) | EC | 1.1 | | 190 | 6 | 12 | fodder | 2.2 2.6 | 0.17 0.18 | EP-CN-5033 |
| MN, 1987 (Pioneer 3969) | WP | 1.1 | | 190 | 6 | 12 | fodder | 2.7 1.8 | 0.24 0.16 | EP-CN-5034 |
| NE, 1987 (Pioneer 3475) | EC | 1.1 | | 190 | 6 | 12 | fodder | 5.5 0.07 | 0.40 0.07 | EP-CN-5035 |
| NE, 1987 (Pioneer 3475) | WP | 1.1 | | 190 | 6 | 12 | fodder | 8.4 7.2 | 0.64 0.74 | EP-CN-5036 |
| OH, 1987 (DeKalb 636) | EC | 1.1 | | 150 | 6 | 12 | fodder | 19 15 | 2.9 1.2 | EP-CN-5037 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| OH, 1987 (DeKalb 636) | WP | 1.1 | | 150 | 6 | 12<br>12 | fodder | 7.8 13<br>c 0.05 | 0.47 0.71<br>c <0.05 | EP-CN-5038 |
| WA, 1989 (Jubilee) | EC | 1.1 | | 94 | ( 6 | 0<br>6<br>12<br>18<br>24 | forage | 0.15 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-CN-1246 |
| WA, 1989 (Jubilee) | EC | 1.1 | | 190 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 14 9.0<br>2.6 1.8<br>1.1 1.0<br>0.10 0.27<br>0.39 0.35<br>c 0.11 | 1.7 0.24<br>0.11 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | EP-CN-1247 |
| WA, 1989 (Jubilee) | WP | 1.1 | | 190 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 11 14<br>1.5 1.4<br>1.5 1.3<br>0.23 0.37<br>0.19 0.21 | 2.2 <0.05<br>0.19 0.12<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-CN-1248 |
| FL, 1988 (Silver Queen) | EC | 1.1 | | 700 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 5.6 4.8<br>0.18 0.19<br>0.10 0.10<br><0.05 (2)<br><0.05 (2) | 0.71 0.61<br>0.09 0.12<br>0.06 0.06<br>0.11 0.10<br><0.05 (2) | EP-CN-5007 |
| FL, 1989 (Silver Queen) | WP | 1.1 | | 700 | 6 | 0<br>6<br>12<br>18<br>24<br>18 | forage | 8.3 5.9<br>0.19 0.13<br><0.05 0.05<br>0.05 (2)<br><0.05 (2)<br>c <0.05 | 1.4 0.66<br>0.09 0.09<br><0.05 0.07<br>0.08 0.08<br><0.05 (2)<br>c 0.09 | EP-CN-5008 |
| FL, 1988 (Merrit) | EC | 1.1 | | 58 | ( 6 | 0<br>6<br>12<br>18<br>24 | forage | 5.8 5.4<br>0.16 0.26<br><0.05 0.09<br><0.05 (2)<br><0.05 (2) | 0.30 0.21<br><0.05 0.11<br><0.05 0.06<br><0.05 (2)<br>0.06 0.06 | EP-CN-5009 |
| NY, 1987 (Early Sunray) | EC | 1.1 | | 230 | 6 | 0<br>12<br>18<br>24 | forage | 33<br>0.56 0.46<br>0.18 0.10<br>0.17 0.15 | 0.48<br>0.16 0.09<br>0.07 <0.05<br>0.07 0.07 | EP-CN-5011 |
| NY, 1987 (Early Sunray) | WP | 1.1 | | 230 | 6 | 0<br>12<br>18<br>24 | forage | 22<br>1.3 0.95<br>0.12 0.21<br>0.17 0.15 | 0.24<br>0.19 0.17<br>0.08 0.08<br>0.11 0.11 | EP-CN-5012 |
| WI, 1987 (Incredible) | EC | 1.1 | | 200 | 6 | 0<br>7<br>12<br>18<br>7 | forage | 26 20<br>0.98 2.0<br>0.64 1.4<br>0.37 0.57<br>c 0.13 | 2.4 2.9<br>0.11 0.13<br>0.10 0.15<br>0.14 0.17<br>c <0.05 | EP-CN-5013 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| WI, 1987 (Incredible) | WP | 1.1 | | 200 | 6 | 0<br>7<br>12<br>18<br>7 | forage | 30 20<br>1.2 1.1<br>2.1 0.96<br>0.31 0.33<br>c 0.10 | 2.9 0.29<br>0.11 0.15<br>0.19 0.21<br>0.12 0.12<br>c <0.05 | EP-CN-5014 |
| IL, 1987 (Asgrow 788) | EC | 1.1 | | 48 | ( 6 | 0<br>6<br>18<br>24 | forage | 2.2 5.6<br>1.1 0.53<br>0.25 0.28<br>0.36 0.21 | 0.10 0.11<br>0.08 0.05<br><0.05 (2)<br>0.06 <0.05 | EP-CN-5015 |
| IL, 1987 (Asgrow 788) | EC | 1.1 | | 190 | 6 | 0<br>6<br>18<br>24<br>6<br>24 | forage [2] | 9.6 29<br>0.43 3.3<br>1.4 1.0<br>0.62 0.62<br>c 0.07<br>c 0.07 | 0.40 0.92<br>0.51 0.18<br>0.13 0.11<br>0.08 0.09<br>c <0.05<br>c <0.05 | EP-CN-5017 |
| IL, 1987 (Asgrow 788) | WP | 1.1 | | 190 | 6 | 0<br>6<br>18<br>24 | forage | 30 20<br>2.3 3.3<br>1.2 0.98<br>0.95 1.6 | 1.1 0.73<br>0.20 0.26<br>0.13 0.12<br>0.11 0.18 | EP-CN-5018 |
| MN, 1988 (Pioneer 3732) | EC | 1.1 | | 230 | 6 | 0<br>6<br>18<br>25 | forage | 8.4 12<br>2.0 2.9<br>0.71 0.78<br>0.62 0.78 | 0.31 0.45<br>0.10 0.15<br>0.10 0.07<br>0.06 0.09 | EP-CN-5019 |
| MN, 1987 (Pioneer 3732) | WP | 1.1 | | 230 | 6 | 0<br>6<br>18<br>25 | forage | 11 9.7<br>1.2 1.8<br>0.90 0.86<br>0.48 0.44 | 0.56 0.47<br>0.07 0.12<br>0.09 0.09<br><0.05 0.07 | EP-CN-5020 |
| MN, 1988 (Pioneer 3906) | EC | 1.1 | | 37 | ( 6 | 0<br>8<br>18<br>24 | forage | 11 11<br>1.7 2.1<br>1.2 1.0<br>1.2 0.97 | 0.07 0.08<br>0.15 0.14<br>0.16 0.14<br>0.15 0.16 | EP-CN-5021 |
| NE, 1987 (Funk's 4500) | EC | 1.1 | | 190 | 6 | 0<br>6<br>18<br>24 | forage | 20 10<br>1.5 1.5<br>1.0 1.2<br>0.48 0.72 | 0.09 0.05<br>0.33 0.22<br>0.10 0.12<br>0.06 0.07 | EP-CN-5023 |
| NE, 1987 (Funk's 4500) | WP | 1.1 | | 190 | 6 | 0<br>0<br>6<br>18<br>24 | forage | 12 6.6<br>0.52<br>0.24 1.3<br>1.1 1.4<br>0.60 | 0.32 0.16<br><0.05<br>0.18 0.11<br><0.05 (2)<br><0.05 | EP-CN-5024 |
| OH, 1987 (Pioneer 3352) | EC | 1.1 | | 150 | 6 | 0<br>6<br>18<br>24<br>0<br>18<br>24 | forage | 23 19<br>6.3 8.1<br>0.26<br>0.30 0.31<br>c 0.21<br>c 0.14<br>c 0.20 | 1.0 1.2<br>0.32 0.35<br>0.28<br>0.25 0.28<br>c <0.05<br>c <0.05<br>c <0.05 | EP-CN-5025 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| OH, 1987 (DeKalb 636) | WP | 1.1 | | 150 | 6 | 0<br>6<br>18<br>24<br>0<br>6<br>18<br>24 | forage | 5.5 15<br>7.2 8.1<br>6.0 4.1<br>3.9 4.3<br>c 0.21<br>c 0.21<br>c 0.18<br>c 0.17 | 0.21 0.89<br>0.40 0.45<br>0.43 0.33<br>0.28 0.29<br>c <0.05<br>c <0.05<br>c <0.05<br>c <0.05 | EP-CN-5026 |
| IL, 1987 (Asgrow 788) | EC | 1.1 | | 48 | ( 6 | 12 | silage | 0.20 <u>0.34</u> | <<u>0.05</u> (2) | EP-CN-5015 |
| IL, 1987 (Asgrow 788) | EC | 1.1 | | 190 | 6 | 12 | silage [2] | <u>2.4</u> 0.78 | <u>0.28</u> 0.07 | EP-CN-5017 |
| IL, 1987 (Asgrow 788) | WP | 1.1 | | 190 | 6 | 12 | silage | 0.54 <u>0.78</u> | 0.097 <u>0.11</u> | EP-CN-5018 |
| MN, 1988 (Pioneer 3732) | EC | 1.1 | | 190 | 6 | 12 | silage | 1.4 <u>2.6</u> | 0.13 <u>0.24</u> | EP-CN-5019 |
| MN, 1987 (Pioneer 3732) | WP | 1.1 | | 230 | 6 | 12 | silage | 0.90 <u>1.2</u> | 0.09 <u>0.10</u> | EP-CN-5020 |
| MN, 1988 (Pioneer 3906) | EC | 1.1 | | 37 | ( 6 | 12 | silage | <u>1.8</u> 1.2 | <u>0.24</u> 0.20 | EP-CN-5021 |
| NE, 1987 (Funk's 4500) | EC | 1.1 | | 190 | 6 | 12 | silage | <u>1.3</u> 0.81 | <u>0.13</u> 0.11 | EP-CN-5023 |
| NE, 1987 (Funk's 4500) | WP | 1.1 | | 190 | 6 | 12 | silage | 0.90 <u>1.1</u> | <<u>0.05</u> (2) | EP-CN-5024 |

( aerial application c: sample from control plot
[1] samples stored for 790 days before analysis
[2] samples stored for 2 years before analysis.

Table 64. Parathion residues in rice straw from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| TX, 1987 (Lemont) | EC | 1.1 | | 206 | 3 | 1 | straw | 19 20 | 1.3 1.1 | EP-RI-1088 |
| CA, 1988 (L202) | EC | 0.11 | | 75 | ( 3 | 1 | straw | 0.87 0.68 | <0.05 (2) | EP-RI-1235 |
| TX, 1987 (Lemont) | EC | 0.11 | | 47 | ( 3 | 1 | straw | 0.44 0.38 | <0.05 (2) | EP-RI-1236 |
| LA, 1987 (Lemont) | EC | 1.1 | | 250 | 6 | 1 | straw | 163 45 | 10 3.7 | EP-RI-5071 |
| LA, 1987 (Lemont) | EC | 1.1 | | 75 | ( 6 | 1 | straw | 15 19 | 1.3 1.3 | EP-RI-5072 |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| LA, 1988 (Lemont) | EC | 0.11 | | 94 | ( 6 | 1 | straw | 1.3 0.94 | 0.13 0.07 | EP-RI-5215 |
| LA, 1988 (Lemont) | EC | 0.11 | | 240 | 6 | 1 | straw | 3.8 3.6 | 0.47 0.38 | EP-RI-5216 |

( aerial application

Table 65. Parathion residues in sorghum hay, forage and stover from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Sample | % dry matter | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| TX, 1994 | CS | 1.1 | | 44 | ( 2 | 12<br>21<br>28 | fodder | 29<br>26<br>29 | 0.87<br>0.44<br>0.28 | 0.05<br><0.05<br><0.05 | 94-TX-GS-989-01 (b) 41988 |
| TX, 1994 | EC | 1.1 | | 44 | ( 2 | 12<br>21<br>28 | fodder | 28<br>27<br>29 | 1.2<br>0.76<br>0.24 | 0.11<br>0.06<br><0.05 | 94-TX-GS-988-01 (b) 41989 |
| NE, 1992 (NK 2030/Pioneer 8379) | EC | 0.55 | | 47 | ( 2 | 28 | forage | 26 | <0.05 (2) | <0.05 (2) | 92150a-1 |
| KS, 1992 (NK 2030) | EC | 0.56 | | 44 | ( 2 | 28 | forage | 24 | <0.05 (2) | <0.05 (2) | 92150b-1 |
| OK, 1992 (NK 2030) | EC | 0.56 | | 47 | ( 2 | 28 | forage | 25 | <0.05 (2) | <0.05 (2) | 92150c-1 |
| TX, 1992 (Pioneer 8313) | EC | 0.55 | | 45 | ( 2 | 28 | forage | 21 | <0.05 (2) | <0.05 (2) | 92150d-1 |
| SD, 1992 (NK 1210) | EC | 0.58 | | 51 | ( 2 | 28 | forage | 25 | <0.05 (2) | <0.05 (2) | 92150e-1 |
| MO, 1992 (Funks - G1506) | EC | 0.55 | | 44 | ( 2 | 28 | forage | 16 | <0.05 (2) | <0.05 (2) | 92150f-1 |
| NE, 1992 (NK 2030/Pioneer 8379) | EC | 1.1 | | 47 | ( 2 | 12<br>28 | forage | 25<br>28 | 0.087 0.082<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | 92150a-2 |
| KS, 1992 (NK 2030) | EC | 1.1 | | 44 | ( 2 | 12<br>28 | forage | 23<br>25 | 0.59 0.72<br>0.11 0.18 | <0.05 (2)<br><0.05 (2) | 92150b-2 |
| OK, 1992 (NK 2030) | EC | 1.1 | | 47 | ( 2 | 12<br>28 | forage | 15<br>23 | 0.31 0.40<br><0.05 0.08 | <0.05 (2)<br><0.05 (2) | 92150c-2 |
| TX, 1992 (Pioneer 8313) | EC | 1.1 | | 46 | ( 2 | 28 | forage | 17<br>18 | 0.13 0.12<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | 92150d-2 |
| SD, 1992 (NK 1210) | EC | 1.3 | | 51 | ( 2 | 12<br>28 | forage | 20<br>26 | 1.7 1.0<br>0.40 0.35 | <0.05 (2)<br><0.05 (2) | 92150e-2 |
| MO, 1992 (Funks - G1506) | EC | 1.1 | | 45 | ( 2 | 12<br>28 | forage | 16<br>16 | 0.56 0.30<br><0.05 0.07 | <0.05 (2)<br><0.05 (2) | 92150f-2 |

| State, year (variety) | Application | | | | | PHI, days | Sample | % dry matter | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| TX, 1994 | EC | 1.1 | | 44 | ( 2 | 12<br>21<br>28 | forage | 30<br>35<br>37 | 0.34 0.25<br><0.05 0.14<br>0.10 0.07 | <0.05 (2)<br><0.05 (2)<br><0.05 (2) | 94-TX-GS-988-01 (a) 41988 |
| TX, 1994 | CS | 1.1 | | 44 | ( 2 | 12<br>21<br>28 | forage | 29<br>36<br>40 | 0.87 1.1<br>0.50 0.40<br>0.34 0.52 | <0.05 (2)<br><0.05 (2)<br><0.05 (2) | 94-TX-GS-989-01 (a) |
| NE, 1992 (NK 2030/Pioneer 8379) | EC | 0.55 | | 47 | ( 2 | 28 | hay | 48 | <0.05 (2) | <0.05 (2) | 92150a-1 |
| KS, 1992 (NK 2030) | EC | 0.56 | | 44 | ( 2 | 28 | hay | 43 | <0.05 (2) | <0.05 (2) | 92150b-1 |
| OK, 1992 (NK 2030) | EC | 0.56 | | 47 | ( 2 | 28 | hay | 42 | <0.05 (2) | <0.05 (2) | 92150c-1 |
| TX, 1992 (Pioneer 8313) | EC | 0.55 | | 45 | ( 2 | 28 | hay | 34 | <0.05 (2) | <0.05 (2) | 92150d-1 |
| SD, 1992 (NK 1210) | EC | 0.58 | | 51 | ( 2 | 28 | hay | 42 | 0.07 0.10 | <0.05 (2) | 92150e-1 |
| MO, 1992 (Funks - G1506) | EC | 0.55 | | 44 | ( 2 | 28 | hay | 43 | <0.05 (2) | <0.05 (2) | 92150f-1 |
| NE, 1992 (NK 2030/Pioneer 8379) | EC | 1.1 | | 47 | ( 2 | 12<br>28 | hay | 47<br>47 | 0.18 0.13<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | 92150a-2 |
| KS, 1992 (NK 2030) | EC | 1.1 | | 44 | ( 2 | 12<br>28 | hay | 41<br>43 | 1.5 1.6<br>0.25 0.22 | <0.05 (2)<br><0.05 (2) | 92150b-2 |
| OK, 1992 (NK 2030) | EC | 1.1 | | 47 | ( 2 | 12<br>28 | hay | 25<br>42 | 0.15 0.34<br>0.05 <0.05 | <0.05 (2)<br><0.05 (2) | 92150c-2 |
| TX, 1992 (Pioneer 8313) | EC | 1.1 | | 46 | ( 2 | 12<br>28 | hay | 32<br>36 | 0.23 0.25<br>0.065 0.086 | <0.05 (2)<br><0.05 (2) | 92150d-2 |
| SD, 1992 (NK 1210) | EC | 1.3 | | 51 | ( 2 | 12<br>28 | hay | 43<br>43 | 4.3 3.4<br>0.37 0.71 | <0.05 (2)<br><0.05 (2) | 92150e-2 |
| MO, 1992 (Funks - G1506) | EC | 1.1 | | 45 | ( 2 | 12<br>28 | hay | 22<br>36 | 0.33 0.52<br>0.08 0.08 | <0.05 (2)<br><0.05 (2) | 92150f-2 |
| OK, 1992 (NK 2030) | EC | 1.1 | | 47 | ( 2 | 28 | stover | 25 | 0.67 0.50 | 0.02 0.02 | 92150c-4 |
| KS, 1992 (NK 2030) | EC | 1.1 | | 44 | ( 2 | 28 | stover | 32 | 0.44 0.39 | 0.04 0.03 | 92150b-4 |
| MO, 1992 (Topaz) | EC | 1.1 | | 45 | ( 2 | 28 | stover | 31 | 0.96 0.78 | 0.04 0.04 | 92150f-8 |
| NE, 1992 (NK 2030/Pioneer 8379) | EC | 1.1 | | 47 | ( 2 | 28 | stover | 25 | 3.5 3.1 | 0.14 0.11 | 92150a-4 |
| SD, 1992 (NK 1210) | EC | 1.1 | | 51 | ( 2 | 28 | stover | 39 | 1.3 1.1 | 0.15 0.12 | 92150e-4 |
| TX, 1992 (Pioneer 8313) | EC | 1.1 | | 46 | ( 2 | 28 | stover | 33 | 4.0 0.83 | 0.18 0.071 | 92150d-4 |

( aerial application

Table 66. Parathion residues in wheat hay, straw and forage from supervised trials in the USA. Residues are expressed on a fresh weight basis. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Sample | % dry matter | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| MN, 1994 (Krona) | EC | 0.84 | | 47 | ( 2 | 15<br>21<br>35 | forage | [2] | 0.12<br>0.08<br><0.05 | <0.05<br><0.05<br><0.05 | 94-MN-WH-735-02 |
| MN, 1994 (Krona) | CS | 0.84 | | 47 | ( 2 | 15<br>21<br>35 | forage | [2] | <0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05 | 94-MN-WH-736-02 |
| ND, 1994 (Pioneer 2375) | EC | 0.84 | | 47 | ( 2 | 15<br>21<br>35 | forage | [2]<br>20<br>32 | 0.09<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05 | 94-ND-WH-735-01 |
| ND, 1994 (Pioneer 2375) | CS | 0.84 | | 47 | ( 2 | 15<br>21<br>35 | forage | [2]<br><br>32 | 0.10<br><0.05<br>0.10 | <0.05<br><0.05<br><0.05 | 94-ND-WH-736-01 |
| CA, 1988 (Anza) | EC | 1.1 | | 94 | ( 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | | 8.3 14<br>9.0 10<br>5.9 7.0<br>5.1 7.5<br>5.6 6.1<br>4.2 6.9 | 0.36 1.1<br>1.2 1.4<br>1.0 1.3<br>0.82 1.4<br>1.2 1.4<br>1.0 1.4 | EP-WH-1219 [1] |
| CA, 1988 (Anza) | WP | 1.1 | | 190 | 6 | 0<br>5<br>9<br>15<br>20<br>25 | forage | | 5.4 17<br>6.1 6.6<br>8.9 4.7<br>3.7 4.8<br>3.1 3.3<br>3.6 3.4 | 0.49 1.2<br>0.64 0.72<br>0.92 0.48<br>0.54 0.66<br>0.45 0.49<br>0.63 0.59 | EP-WH-1220 [1] |
| CA, 1988 (Anza) | EC | 1.1 | | 190 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | | 19 5.9<br>5.1 6.4<br>4.3 7.0<br>2.3 6.5<br>6.7 7.3<br>5.5 15 | 1.1 0.57<br>0.52 0.77<br>0.53 0.85<br>0.10 0.92<br>1.0 0.80<br>0.73 1.3 | EP-WH-1221 [1] |
| WA, 1988 (Stephens) | WP | 1.1 | | 110 | 6 | 0<br>5<br>10<br>15<br>20<br>25<br>25 | forage | | 20 31<br>2.9 1.8<br>0.91 0.46<br>0.54 0.74<br>0.84 0.95<br>0.73 0.62<br>0.05 | 0.53 0.85<br>0.62 0.37<br>0.32 0.22<br>0.22 0.22<br>0.24 0.31<br>0.26 0.21 | EP-WH-1222 [1] |

| State, year (variety) | Application | | | | | PHI, days | Sample | % dry matter | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| WA, 1988 (Stephens) | EC | 1.1 | | 110 | 6 | 0<br>5<br>10<br>15<br>20<br>25<br>25 | forage | | 17 14<br>1.6 1.6<br>1.5 1.4<br>0.58 0.56<br>0.75 0.84<br>1.1 0.79<br>c 0.06 | 0.52 0.59<br>0.47 0.45<br>0.43 0.39<br>0.20 0.18<br>0.28 0.35<br>0.54 0.35 | EP-WH-1223 [1] |
| MO, 1989 (Caldwell) | WP | 0.90 | | 190 | 6 | 0<br>7<br>10<br>15<br>20<br>25 | forage | | 8.6 9.3<br>2.6 3.6<br>1.5 2.0<br>2.3 1.2<br>2.3 2.3<br>1.4 1.5 | 0.64 0.95<br>0.36 0.50<br>0.19 0.25<br>0.40 0.19<br>0.39 0.36<br>0.20 0.26 | EP-WH-1253 [1] |
| KS, 1988 (Pioneer 2157) | EC | 1.1 | | 120 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | | 34 24<br>14 11<br>10 6.4<br>5.8 5.6<br>6.2 4.8<br>1.7 1.4<br>c 0.13 0.07 | 1.3 1.1<br>1.4 1.1<br>1.1 0.62<br>0.63 0.63<br>0.49 0.56<br>0.13 0.11 | EP-WH-5189 [1] |
| KS, 1988 (Pioneer 2157) | WP | 1.1 | | 120 | 6 | 0<br>5<br>10<br>15<br>20<br>25<br>0<br>5 | forage | | 28 30<br>11 14<br>8.0 10<br>6.6 5.4<br>3.9 4.3<br>1.3 1.6<br>c 0.12<br>c 0.12 | 0.92 1.1<br>0.92 1.3<br>0.58 0.75<br>0.54 0.42<br>0.41 0.43<br>0.09 0.15 | EP-WH-5190 [1] |
| MO, 1988 (Caldwell) | EC | 1.1 | | 26 | ( 6 | 0<br>5<br>10<br>15<br>20<br>25<br>0<br>15<br>20 | forage | | 0.19 0.16<br>0.12 0.11<br>0.14 0.13<br>0.08 0.12<br>0.12 0.07<br>0.05 0.09<br>c 0.12<br>c 0.06<br>c 0.06 | 0.13 0.10<br>0.09 0.12<br>0.15 0.16<br>0.09 0.12<br>0.09 0.12<br>0.08 <0.05<br><br>c 0.05 | EP-WH-5191 [1] |
| MO, 1988 (Caldwell) | EC | 1.1 | | 190 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | | 26 30<br>9.2 13<br>7.4 8.8<br>5.5 5.7<br>2.8 5.7<br>4.6 4.2<br>c 0.05 | 1.3 1.3<br>0.99 1.5<br>1.2 1.2<br>1.1 1.0<br>0.71 1.1<br>0.96 0.73 | EP-WH-5193 [1] |
| ND, 1988 (Marshall) | EC | 1.1 | | 94 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | | 11 7.8<br>6.4 5.0<br>4.9 4.7<br>2.7 2.1<br>2.4 2.5<br>1.2 1.0 | 0.67 0.61<br>0.73 0.72<br>0.65 0.70<br>0.32 0.33<br>0.31 0.35<br>0.21 0.17 | EP-WH-5195 [1] |

| State, year (variety) | Application | | | | | PHI, days | Sample | % dry matter | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| ND, 1988 (Marshall) | WP | 1.1 | | 94 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | | 12 18<br>3.0 3.2<br>2.9 3.1<br>1.9 2.1<br>1.1 1.2<br>0.58 0.69 | 0.79 0.89<br>0.57 0.44<br>0.60 0.64<br>0.28 0.26<br>0.17 0.25<br>0.12 0.11 | EP-WH-5196 [1] |
| MN, 1994 (Krona) | EC | 0.84 | | 47 | ( 2 | 42<br>48<br>62 | hay | [3]<br><br>62 | 0.10<br>0.05<br><0.05 | <0.05<br><0.05<br><0.05 | 94-MN-WH-735-02 |
| MN, 1994 (Krona) | CS | 0.84 | | 47 | ( 2 | 42<br>48<br>62 | hay | [3]<br><br>67 | <0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05 | 94-MN-WH-736-02 |
| ND, 1994 (Pioneer 2375) | EC | 0.84 | | 47 | ( 2 | 42<br>48<br>62 | hay | 67<br>72<br>63<br>[3] | <0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05 | 94-ND-WH-735-01 |
| ND, 1994 (Pioneer 2375) | CS | 0.84 | | 47 | ( 2 | 42<br>48<br>62 | hay | [3]<br><br>67 | 0.07<br><0.05<br>0.05 | <0.05<br><0.05<br><0.05 | 94-ND-WH-736-01 |
| MN, 1994 (Krona) | EC | 0.84 | | 47 | ( 2 | 15<br>21<br>36 | straw | [4] | 3.1<br>2.1<br>1.4 | 0.24<br>0.16<br>0.09 | 94-MN-WH-735-02 |
| MN, 1994 (Krona) | CS | 0.84 | | 47 | ( 2 | 15<br>21<br>36 | straw | [4] | 3.1<br>1.5<br>0.65 | 0.15<br>0.10<br><0.05 | 94-MN-WH-736-02 |
| ND, 1994 (Pioneer 2375) | EC | 0.84 | | 47 | ( 2 | 15<br>21<br>36 | straw | [4]<br><br>65 | 0.94<br>0.54<br>0.98 | 0.07<br><0.05<br>0.06 | 94-ND-WH-735-01 |
| ND, 1994 (Pioneer 2375) | CS | 0.84 | | 47 | ( 2 | 16<br>21<br>36 | straw | [4]<br><br>67 | 0.67<br>0.49<br>0.36 | <0.05<br><0.05<br><0.05 | 94-ND-WH-736-01 |
| CA, 1988 (Anza) | EC | 1.1 | | 94 | ( 6 | 15 | straw | | 6.9 7.7 | 1.0 1.1 | EP-WH-1219 [1] |
| CA, 1988 (Anza) | WP | 1.1 | | 190 | 6 | 15 | straw | | 3.4 3.6 | 0.53 0.51 | EP-WH-1220 [1] |
| CA, 1988 (Anza) | EC | 1.1 | | 190 | 6 | 15 | straw | | 3.9 5.0 | 0.44 0.58 | EP-WH-1221 [1] |
| WA, 1988 (Stephens) | WP | 1.1 | | 110 | 6 | 15 | straw | | 1.2 1.1 | 0.12 0.13 | EP-WH-1222 [1] |
| WA, 1988 (Stephens) | EC | 1.1 | | 110 | 6 | 15 | straw | | 1.4 0.51 | 0.23 0.10 | EP-WH-1223 [1] |
| MO, 1989 (Caldwell) | WP | 0.90 | | 190 | 6 | 15 | straw | | 3.9 3.7 | 0.52 0.52 | EP-WH-1253 [1] |

| State, year (variety) | Application | | | | | PHI, days | Sample | % dry matter | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| KS, 1988 (Pioneer 2157) | EC | 1.1 | | 120 | 6 | 15 | straw | | 8.7 11 | 0.82 0.92 | EP-WH-5189 [1] |
| KS, 1988 (Pioneer 2157) | WP | 1.1 | | 120 | 6 | 15 | straw | | 9.4 8.5 | 0.74 0.83 | EP-WH-5190 [1] |
| MO, 1988 (Caldwell) | EC | 1.1 | | 26 | ( 6 | 15 | straw | | 0.11 0.12 | 0.11 0.10 | EP-WH-5191 [1] |
| MO, 1988 (Caldwell) | EC | 1.1 | | 190 | 6 | 15 | straw | | 6.0 6.9<br>c 0.08 | 1.1 1.2 | EP-WH-5193 [1] |
| ND, 1988 (Marshall) | EC | 1.1 | | 94 | 6 | 15 | straw | | 2.8 4.3 | 0.24 0.33 | EP-WH-5195 [1] |
| ND, 1988 (Marshall) | WP | 1.1 | | 94 | 6 | 15 | straw | | 2.7 2.7 | 0.21 0.22 | EP-WH-5196 [1] |
| WA, 1992 (Penawawa) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | straw | 87<br>92<br>86 | 3.6 3.4<br>3.1 2.8<br>2.0 2.1 | 0.25 0.26<br>0.27 0.26<br>0.20 0.23 | 92148a (a) |
| WA, 1992 (Penawawa) | EC | 0.85 | | 47 | ( 2 | 15<br>20<br>25 | straw | 91<br>93<br>90 | 6.7 7.5<br>5.9 6.6<br>5.6 4.9 | 0.47 0.53<br>0.51 0.52<br>0.51 0.43 | 92148a (b) |
| ID, 1992 (Penawawa) | EC | 0.56 | | 49 | ( 2 | 15<br>20<br>25<br>15 | straw | 92<br>91<br>62 | 1.4 1.4<br>1.9 1.8<br>1.2 1.1<br>c 0.05 | 0.19 0.19<br>0.29 0.32<br>0.15 0.13 | 92148b (a) |
| ID, 1992 (Penawawa) | EC | 0.85 | | 49 | ( 2 | 15<br>20<br>25 | straw | 91<br>90<br>69 | 3.2 3.2<br>2.9 3.8<br>2.7 2.8 | 0.37 0.32<br>0.36 0.45<br>0.27 0.29 | 92148b (b) |
| MT, 1992 (Amadon) | EC | 0.56<br>-0.69 | | 48, 57 | ( 2 | 15<br>25 | straw | 77<br>91 | 0.50 0.55<br>1.4 0.73 | 0.11 0.13<br>0.28 0.15 | 92148c (a) |
| MT, 1992 (Amadon) | EC | 0.90<br>-0.93 | | 51 | ( 2 | 15<br>25 | straw | 69<br>86 | 0.63 0.86<br>0.86 1.0 | 0.16 0.20<br>0.26 0.31 | 92148c (b) |
| ND, 1992 (Gus) | EC | 0.56 | | 46 | ( 2 | 15<br>20<br>25 | straw | 39<br>38<br>36 | 0.44 0.35<br>0.32 0.36<br>0.48 0.39 | 0.053 0.03<br>0.03 0.03<br>0.03 0.03 | 92148d (a) |
| ND, 1992 (Gus) | EC | 0.84 | | 46 | ( 2 | 15<br>20<br>25 | straw | 35<br>34<br>39 | 0.91 1.2<br>1.1 0.92<br>0.99 0.90 | 0.055 0.084<br>0.052 0.05<br>0.05 0.051 | 92148d (b) |
| MN, 1992 (Vance) | EC | 0.56 | | 46 | ( 2 | 15<br>20<br>25 | straw | 41<br>52<br>59 | 1.7 2.6<br>1.3 1.5<br>1.5 1.3 | 0.17 0.21<br>0.085 0.097<br>0.074 0.065 | 92148e (a) |
| MN, 1992 (Vance) | EC | 0.84 | | 46 | ( 2 | 15<br>20<br>25 | straw | 40<br>59<br>60 | 5.2 7.3<br>5.4 2.9<br>4.2 6.1 | 0.34 0.43<br>0.25 0.12<br>0.19 0.27 | 92148e (b) |

| State, year (variety) | Application | | | | | PHI, days | Sample | % dry matter | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| SD, 1992 (Bute 86) | EC | 0.88<br>+0.80 | | 50<br>+44 | ( 2 | 15<br>20<br>25 | straw | 87<br>91<br>63 | 9.5 8.7<br>6.8 6.5<br>7.6 4.9 | 0.81 0.53<br>0.48 0.56<br>0.45 0.45 | 92148f (b) |
| SD, 1992 (Bute 86) | EC | 0.60<br>+0.48 | | 51<br>+40 | ( 2 | 15<br>20<br>25 | straw | 83<br>90<br>87 | 3.4 4.5<br>4.3 3.7<br>2.6 2.3 | 0.39 0.35<br>0.37 0.30<br>0.17 0.19 | 92148f (a) |
| KS, 1993 (Karl) | EC | 0.56 | | 47 | ( 2 | 15 | forage | 18 | 0.073 0.061 | 0.02 <0.02 | 93240a (a) |
| KS, 1993 (Karl) | EC | 0.84 | | 47 | ( 2 | 15 | forage | 19 | 0.087 0.051 | 0.02 <0.02 | 93240a (b) |
| TX, 1993 (DK 49 S) | EC | 0.56 | | 52 | ( 2 | 15 | forage | 33 | 0.098 0.14 | 0.05 0.060 | 93240b (a) |
| TX, 1993 (DK 49 S) | EC | 0.84 | | 51 | ( 2 | 15 | forage | 32 | 0.13 0.15 | 0.05 0.052 | 93240b (b) |
| OK, 1993 (McNair) | EC | 0.55 | | 45 | ( 2 | 15 | forage | 19 | 0.32 0.34 | 0.092 0.079 | 93240c (a) |
| OK, 1993 (McNair) | EC | 0.83 | | 45 | ( 2 | 15 | forage | 22 | 0.37 0.48 | 0.097 0.13 | 93240c (b) |
| CO, 1993 (Buckskin) | EC | 0.56 | | 46 | ( 2 | 15 | forage | 26 | 0.057 0.15 | <0.02 (2) | 93240d (a) |
| CO, 1993 (Buckskin) | EC | 0.83 | | 46 | ( 2 | 15 | forage | 25 | 0.31 0.79 | 0.073 0.091 | 93240d (b) |
| NE, 1993 (Buckskin) | EC | 0.56 | | 46 | ( 2 | 15 | forage | 29 | 0.72 0.43 | 0.11 0.072 | 93240e (a) |
| NE, 1993 (Buckskin) | EC | 0.83 | | 46 | ( 2 | 15 | forage | 27 | 0.52 0.48 | 0.13 0.11 | 93240e (b) |
| KS, 1993 (Karl) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | straw | 60<br>69<br>87 | 0.84 0.92<br>0.42 0.44<br>0.44 0.35 | 0.092 0.083<br>0.02 0.03<br>0.02 <0.02 | 93240a (c) |
| KS, 1993 (Karl) | EC | 0.83 | | 47 | ( 2 | 15<br>20<br>25 | straw | 55<br>65<br>87 | 1.8 1.3<br>0.79 0.91<br>1.2 0.67 | 0.17 0.087<br>0.04 0.055<br>0.04 0.03 | 93240a (d) |
| TX, 1993 (DK 49 S) | EC | 0.61 | | 55 | ( 2 | 15<br>20<br>25 | straw | 43<br>51<br>65 | 0.78 0.51<br>1.5 0.64<br>0.41 0.69 | 0.051 0.04<br>0.10 0.05<br>0.02 0.04 | 93240b (c) |
| TX, 1993 (DK 49 S) | EC | 0.90 | | 54 | ( 2 | 15<br>20<br>25 | straw | 51<br>65<br>70 | 2.1 2.4<br>2.6 3.1<br>2.7 3.5 | 0.15 0.16<br>0.15 0.20<br>0.14 0.15 | 93240b (d) |
| OK, 1993 (McNair) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | straw | 65<br>77<br>87 | 0.80 3.3[5]<br>0.53 0.59<br>0.41 0.34 | 0.04 0.14<br>0.02 <0.02<br><0.02 (2) | 93240c (c) |
| OK, 1993 (McNair) | EC | 0.84 | | 47 | ( 2 | 15<br>20<br>25 | straw | 56<br>76<br>87 | 1.0 1.9<br>1.5 1.1<br>0.63 0.44 | 0.04 0.063<br>0.03 0.03<br><0.02 (2) | 93240c (d) |

| State, year (variety) | Application | | | | | PHI, days | Sample | % dry matter | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| CO, 1993<br>(Buckskin) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | straw | 63<br>76<br>79 | 0.21 0.22<br>0.13 0.29<br>0.16 0.27 | 0.058 0.066<br>0.04 0.089<br>0.03 0.05 | 93240d (c) |
| CO, 1993<br>(Buckskin) | EC | 0.84 | | 47 | ( 2 | 15<br>20<br>25 | straw | 64<br>71<br>80 | 0.25 0.47<br>0.39 0.50<br>0.38 0.33 | 0.066 0.13<br>0.12 0.14<br>0.062 0.055 | 93240d (d) |
| NE, 1993<br>(Buckskin) | EC | 0.56 | | 47 | ( 2 | 15<br>20<br>25 | straw | 55<br>63<br>70 | 0.20 0.32<br>0.26 0.27<br>0.12 0.13 | 0.058 0.11<br>0.094 0.082<br>0.03 0.02 | 93240e (c) |
| NE, 1993<br>(Buckskin) | EC | 0.84 | | 47 | ( 2 | 15<br>20<br>25 | straw | 57<br>68<br>77 | 1.4 1.8<br>0.92 0.92<br>1.4 1.5 | 0.22 0.30<br>0.16 0.20<br>0.22 0.24 | 93240e (d) |

[1] unvalidated analytical data
[2] % dry matter was measured for selected forage samples in trials 94-xx-WH-735 and -736. Mean 30%
[3] % dry matter was measured for selected hay samples in trials 94-xx-WH-735 and -736. Mean 66%
[4] % dry matter was measured for selected straw samples in trials 94-xx-WH-735 and -736. Mean 64%
[5] Results on day 15 verified by re-extraction and analysis.
( aerial application c: sample from control plot

Table 67. Parathion residues in alfalfa from supervised trials in the USA.

| State, year (variety) | Application | | | | | PHI, days | Sample[1] | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | % water | parathion | paraoxon | |
| IA, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>49 | forage | 69<br>75<br>71<br>75 | 7.5<br>2.5<br>0.81<br><0.05 | 0.50<br>0.29<br><0.05<br><0.05 | 94-IA-AL-873-01 |
| IA, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>49 | hay | 63<br>47<br>20<br>54 | 6.0<br>4.2<br>2.3<br>0.23 | 0.47<br>0.38<br>0.14<br><0.05 | 94-IA-AL-873-01 |
| ID, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>26<br>51 | forage | 83<br>81<br>77<br>79 | 4.6<br>2.5<br>1.4<br><0.05 | 0.23<br>0.17<br>0.09<br><0.05 | 94-ID-AL-873-04 |
| ID, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>26<br>51 | hay | 24<br>21<br>26<br>57 | 15<br>5.9<br>2.9<br>0.11 | 0.64<br>0.63<br>0.20<br><0.05 | 94-ID-AL-873-04 |
| KS, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | forage | 69<br>68<br>65<br>72 | 17<br>3.9<br>0.70<br>0.23 | 1.1<br>0.42<br><0.05<br><0.05 | 94-KS-AL-873-12 |

| State, year (variety) | Application | | | | | PHI, days | Sample[1] | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | % water | parathion | paraoxon | |
| KS, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | hay | 30<br>17<br>22<br>42 | 9.9<br>5.2<br>1.6<br>0.20 | 0.80<br>0.48<br>0.09<br><0.05 | 94-KS-AL-873-12 |
| MN, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>57 | forage | 78<br>74<br>72<br>76 | 4.4<br>2.0<br>0.56<br>0.28 | 0.51<br>0.30<br>0.14<br><0.05 | 94-MN-AL-873-08 |
| MN, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>57 | hay | 41<br>49<br>32<br>29 | 9.0<br>3.9<br>1.8<br>0.37 | 0.86<br>0.63<br><0.05<br><0.05 | 94-MN-AL-873-08 |
| MO, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | forage | 75<br>73<br>74<br>74 | 8.8<br>2.4<br>0.28<br>0.06 | 0.34<br>0.10<br><0.05<br><0.05 | 94-MO-AL-873-13 |
| MO, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | hay | 34<br>43<br>39<br>42 | 18<br>3.6<br>0.82<br>0.08 | 0.84<br>0.22<br><0.05<br><0.05 | 94-MO-AL-873-13 |
| MT, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | forage | 76<br>75<br>72<br>70 | 13<br>7.3<br>5.7<br>1.9 | 0.46<br>0.38<br>0.28<br><0.05 | 94-MT-AL-873-06 |
| MT, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | hay | 47<br>42<br>37<br>46 | 36<br>16<br>10<br>3.7 | 0.77<br>0.85<br>0.51<br>0.07 | 94-MT-AL-873-06 |
| ND, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>56 | forage | 81<br>80<br>77<br>77 | 2.0<br>0.64<br>0.65<br>0.25 | 0.26<br>0.10<br><0.05<br><0.05 | 94-ND-AL-873-07 |
| ND, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>56 | hay | 52<br>61<br>51<br>31 | 4.2<br>0.97<br>0.79<br>0.42 | 0.57<br>0.20<br><0.05<br><0.05 | 94-ND-AL-873-07 |
| NE, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | forage | 80<br>80<br>78<br>83 | 3.3<br>0.95<br>0.23<br><0.05 | 0.29<br>0.09<br><0.05<br><0.05 | 94-NE-AL-873-09 |
| NE, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | hay | 33<br>48<br>27<br>45 | 7.5<br>1.4<br>1.0<br><0.05 | 0.67<br>0.14<br><0.05<br><0.05 | 94-NE-AL-873-09 |

| State, year (variety) | Application | | | | | PHI, days | Sample[1] | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | % water | parathion | paraoxon | |
| NY, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | forage | 74<br>79<br>75<br>81 | 6.6<br>2.5<br>2.3<br>0.06 | 0.40<br>0.10<br>0.08<br><0.05 | 94-NY-AL-873-03 |
| NY, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | hay | 55<br>28<br>36<br>45 | 12<br>7.3<br>5.7<br>0.43 | 0.73<br>0.38<br>0.23<br><0.05 | 94-NY-AL-873-03 |
| OK, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>49 | forage | 76<br>73<br>76<br>80 | 24<br>8.2<br>1.2<br><0.05 | 0.67<br>0.29<br>0.06<br><0.05 | 94-OK-AL-873-11 |
| OK, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>49 | hay | 32<br>14<br>34<br>25 | 30<br>25<br>3.4<br><0.05 | 1.0<br>0.91<br>0.27<br><0.05 | 94-OK-AL-873-11 |
| SD, 1994 | EC | 1.1 | | 47 | (<br>2 | 7<br>15<br>25<br>50 | forage | 77[3] | 15<br>6.6<br>1.9<br>0.10 | 1.0<br>0.53<br>0.11<br><0.05 | 94-SD-AL-737-02<br>41737 |
| SD, 1994 | EC | 1.1 | | 47 | (<br>2 | 7<br>15<br>25<br>50 | hay | 28[4] | 31<br>9.5<br>1.5<br>0.22 | 1.9<br>0.49<br>0.09<br><0.05 | 94-SD-AL-737-02 |
| SD, 1994 | CS | 1.1 | | 47 | (<br>2 | 7<br>15<br>25<br>50 | forage | 77[3] | 12<br>3.4<br>1.1<br>0.11 | 0.59<br>0.33<br>0.08<br><0.05 | 94-SD-AL-763-02 |
| SD, 1994 | CS | 1.1 | | 47 | (<br>2 | 7<br>15<br>25<br>50 | hay | 28[4] | 35<br>5.3<br>2.9<br>0.31 | 1.5<br>0.28<br>0.13<br><0.05 | 94-SD-AL-763-02 |
| UT, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>26<br>56 | forage | 80<br>76<br>72<br>76 | 3.9<br>2.2<br>1.1<br><0.05 | 0.19<br>0.16<br>0.11<br><0.05 | 94-UT-AL-873-05 |
| UT, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>26 | hay | 25<br>17<br>27 | 13<br>5.4<br>3.2 | 0.85<br>0.54<br>0.35 | 94-UT-AL-873-05 |
| WA, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | forage | 84<br>83<br>79<br>83 | 1.9<br>0.44<br>0.33<br><0.05 | 0.16<br><0.05<br><0.05<br><0.05 | 94-WA-AL-873-02 |
| WA, 1994 | CS | 1.1 | | 47 | ( 2 | 7<br>15<br>25<br>50 | hay | 54<br>62<br>70<br>61 | 5.8<br>1.2<br>1.3<br><0.05 | 0.54<br>0.17<br>0.10<br><0.05 | 94-WA-AL-873-02 |

| State, year (variety) | Application | | | | | PHI, days | Sample[1] | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | % water | parathion | paraoxon | |
| WI, 1994 | EC | 1.1 | | 47 | (<br>2 | 7<br>15<br>25<br>67 | forage | 77[3] | 11<br>4.5<br>1.5<br><0.05 | 1.0<br>0.48<br>0.12<br><0.05 | 94-WI-AL-737-01 |
| WI, 1994 | EC | 1.1 | | 47 | (<br>2 | 7<br>15<br>25<br>67 | hay | 28[4] | 33<br>9.9<br>2.2<br><0.05 | 2.2<br>1.1<br>0.24<br><0.05 | 94-WI-AL-737-01 |
| WI, 1994 | CS | 1.1 | | 47 | (<br>2 | 7<br>15<br>25<br>67 | forage | 77[3] | 6.4<br>2.8<br>0.68<br><0.05 (2) | 0.39<br>0.23<br><0.05<br><0.05 (2) | 94-WI-AL-763-01 |
| WI, 1994 | CS | 1.1 | | 47 | (<br>2 | 7<br>15<br>25<br>67 | hay | 28[4] | 19<br>7.0<br>1.7<br><0.05 | 1.0<br>0.52<br>0.12<br><0.05 | 94-WI-AL-763-01 |
| CA, 1987 (Millet Mix) | EC | 0.90 | | 94 | ( 4 | 15<br>15<br>37<br>15<br>15<br>37 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 1.5 1.2<br>0.63 0.38<br><0.05 (2)<br>0.63 0.96<br>0.96 1.4<br>0.11 <0.05 | 0.29 0.24<br>0.08 0.06<br><0.05 (2)<br>0.08 0.07<br><0.05 0.06<br><0.05 (2) | EP-AF-1001 [2] |
| CA, 1987 (Millet Mix) | EC | 0.90 | | 190 | 4 | 15<br>15<br>37<br>15<br>15<br>63 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 12 21<br>1.2 0.69<br><0.05 (2)<br>32 34<br>2.1 6.1<br>0.05 <0.05 | 1.6 2.6<br>0.12 0.09<br><0.05 (2)<br>2.3 3.9<br>0.24 0.40<br><0.05 (2) | EP-AF-1002 [2] |
| CA, 1987 (Millet Mix) | WP | 0.90 | | 190 | 4 | 15<br>15<br>37<br>15<br>15<br>37 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 3.9 5.9<br>0.63 0.36<br><0.05 0.06<br>1.7 2.5<br>1.8 2.5<br><0.05 0.06 | 0.64 1.1<br>0.06 0.06<br><0.05 (2)<br>0.23 0.29<br>0.40 0.36<br><0.05 (2) | EP-AF-1003 [2] |
| CA, 1988 (Cuff 101) | EC | 0.90 | | 190 | 2 | 15 | hay 1 | | 1.9 | 0.20 | EP-AF-2032 [2] |
| CA, 1988 (Cuff 101) | EC | 4.5 | | 190 | 2 | 15 | hay 1<br>meal 1 | | 11<br>12 | 0.56<br>1.4 | EP-AF-2032 [2] |
| CA, 1988 (Cuff 101) | EC | 0.90 | | 190 | 2 | 15 | hay 1 | | <0.05<br>c 0.11 0.39 | <0.05<br>c <0.05 (2) | EP-AF-2033 [2] |
| CA, 1988 (Cuff 101) | EC | 4.5 | | 190 | 2 | 15 | hay 1<br>meal 1 | | 0.09<br>0.08 | <0.05<br><0.05 | EP-AF-2033 [2] |

| State, year (variety) | Application | | | | | PHI, days | Sample[1] | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | % water | parathion | paraoxon | |
| IA, 1988 | EC | 0.90 | | 170 | 4 | 13<br>14<br>46<br>13<br>14<br>46 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 3.4 1.4<br>1.1 1.6<br><0.05 (2)<br>3.6 1.2<br>2.6 2.6<br>0.09 0.11 | 0.26 0.11<br>0.09 0.13<br><0.05 (2)<br>0.28 0.10<br>0.27 0.23<br><0.05 (2) | EP-AF-5128 [2] |
| IA, 1988 | WP | 0.90 | | 170 | 4 | 13<br>14<br>46<br>13<br>14<br>46 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 3.5 2.9<br>0.43 0.42<br><0.05 (2)<br>5.1 4.3<br>0.98 0.63<br>0.14 0.10 | 0.33 0.25<br><0.05 (2)<br><0.05 (2)<br>0.59 0.53<br>0.11 0.09<br><0.05 (2) | EP-AF-5129 [2] |
| WI, 1988 (True Blue (Renk)) | EC | 0.90 | | 240 | 5 | 15<br>15<br>21<br>15<br>15<br>21 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 3.5 2.6<br>1.0 1.3<br>0.23 0.25<br>6.2 6.2<br>1.2 1.2<br>1.0 0.51 | 0.53 0.38<br>0.19 0.25<br><0.05 (2)<br>0.77 0.77<br>0.16 0.17<br>0.08 <0.05 | EP-AF-5130 [2] |
| WI, 1988 (True Blue (Renk)) | WP | 0.90 | | 240 | 5 | 15<br>15<br>21<br>15<br>15<br>21 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 1.3 2.0<br>0.42 0.38<br>0.22 0.21<br>2.0 2.3<br>1.5 0.74<br>3.4 0.70 | 0.19 0.23<br>0.09 0.07<br><0.05 (2)<br>0.33 0.43<br>0.16 0.10<br>0.21 <0.05 | EP-AF-5131 [2] |
| IA, 1988 | EC | 0.90 | | 19 | ( 4 | 15<br>13<br>46<br>15<br>13<br>46 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 0.86 0.78<br>0.14 0.16<br><0.05 (2)<br>1.9 2.7<br>0.43 0.63<br><0.05 (2) | 0.05 0.05<br><0.05 (2)<br><0.05 (2)<br>0.17 0.21<br><0.05 (2)<br><0.05 (2) | EP-AF-5132 [2] |
| SD, 1988 (Coyote 990) | EC | 0.90 | | 75 | 4 | 14<br>15<br>63<br>14<br>15<br>63 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 0.79 4.6<br>1.7 1.8<br><0.05 (2)<br>2.4 5.5<br>2.9 2.7<br><0.05 (2) | 0.11 0.06<br>0.19 0.18<br><0.05 (2)<br>0.25 0.60<br>0.44 0.40<br><0.05 (2) | EP-AF-5134 [2] |
| SD, 1988 (Coyote 990) | WP | 0.90 | | 75 | 4 | 14<br>15<br>63<br>14<br>15<br>63 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 2.5 1.2<br>2.5 0.99<br><0.05 (2)<br>3.0 2.8<br>2.4 0.47<br><0.05 (2) | 0.23 0.10<br>0.15 0.11<br><0.05 (2)<br>0.31 0.26<br>0.26 0.06<br><0.05 (2) | EP-AF-5135 [2] |
| NE, 1988 (Wrangler) | EC | 0.90 | | 190 | 4 | 15<br>15<br>49<br>15<br>15<br>49 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 3.0 5.8<br>0.22 0.14<br><0.05 (2)<br>6.2 5.6<br>0.86 1.0<br><0.05 (2) | 0.17 0.32<br><0.05 (2)<br><0.05 (2)<br>0.31 0.32<br>0.15 0.15<br><0.05 (2) | EP-AF-5136 [2] |

| State, year (variety) | Application | | | | | PHI, days | Sample[1] | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | % water | parathion | paraoxon | |
| NE, 1988 (Wrangler) | WP | 0.90 | | 190 | 4 | 15<br>15<br>49<br>15<br>15<br>49 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 3.0 1.9<br>0.10 0.38<br><0.05 (2)<br>3.0 6.0<br>0.39 0.48<br><0.05 (2) | 0.16 0.10<br><0.05 0.06<br><0.05 (2)<br>0.09 0.34<br>0.07 0.10<br><0.05 (2) | EP-AF-5137 [2] |
| MN, 1988 (Iroquois) | EC | 0.90 | | 190 | 4 | 15<br>15<br>63<br>15<br>15<br>63 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 0.92 1.1<br>0.51 0.62<br><0.05 (2)<br>1.9 1.2<br>1.1 0.82<br><0.05 (2) | 0.08 0.07<br>0.06 0.08<br><0.05 (2)<br>0.19 0.12<br>0.14 0.20<br><0.05 (2) | EP-AF-5138 [2] |
| MN, 1988 (Iroquois) | WP | 0.90 | | 190 | 4 | 15<br>15<br>63<br>15<br>15<br>63 | forage 1<br>forage 2<br>forage 3<br>hay 1<br>hay 2<br>hay 3 | | 1.1 0.64<br>0.33 0.35<br><0.05 (2)<br>1.4 1.8<br>0.62 0.49<br><0.05 (2) | 0.06 <0.05<br><0.05 (2)<br><0.05 (2)<br>0.09 0.20<br>0.09 0.05<br><0.05 (2) | EP-AF-5139 [2] |

[1] forage 1, forage 2, hay 1, etc refer to forage 1st cut, forage 2nd cut, hay 1st cut, etc
[2] unvalidated analytical data
[3] % moisture was measured for selected forage samples in trials 94-xx-AL-737 and -763. Mean 77%
[4] % moisture was measured for selected hay samples in trials 94-xx-AL-737 and -763. Mean 28%
( aerial application

Table 68. Parathion residues in red clover from supervised trials in the USA.

| State, year (variety) | Application | | | | | growth stage | PHI, days | Sample[1] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| MO, 1988 | EC | 0.90 | | 187 | 3 | flowering | 15 | forage 2 | 1.4 1.4 | 0.07 0.07 | EP-CL-5154 |
| MO, 1988 | EC | 0.90 | | 187 | 3 | flowering | 15 | hay 2 | 3.0 2.8 | 0.16 0.16 | EP-CL-5154 |
| MO, 1988 | WP | 0.90 | | 187 | 4 | flowering | 15 | forage 1 | 0.12 1.7 | <0.05 0.14 | EP-CL-5155 |
| MO, 1988 | WP | 0.90 | | 187 | 4 | flowering | 15 | forage 2 | 1.6 1.0 | 0.07 0.06 | EP-CL-5155 |
| MO, 1988 | WP | 0.90 | | 187 | 4 | flowering | 15 | hay 1 | 1.1 1.5 | 0.09 0.11 | EP-CL-5155 |
| MO, 1988 | WP | 0.90 | | 187 | 4 | flowering | 15 | hay 2 | 2.1 2.1 | 0.08 0.09 | EP-CL-5155 |
| MO, 1988 | EC | 0.90 | | 24 | ( 4 | 80% flowering | 15 | forage 1 | 0.13 0.27 | <0.05 (2) | EP-CL-5156 |
| MO, 1988 | EC | 0.90 | | 24 | ( 4 | 80% flowering | 15 | forage 2 | 0.61 1.0 | <0.05 (2) | EP-CL-5156 |

| State, year (variety) | Application | | | | | growth stage | PHI, days | Sample[1] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| MO, 1988 | EC | 0.90 | | 24 | ( 4 | 80% flowering | 15 | hay 1 | <0.05 0.08 | 0.10 <0.05 | EP-CL-5156 |
| MO, 1988 | EC | 0.90 | | 24 | ( 4 | 80% flowering | 15 | hay 2 | 1.2 1.8 | 0.06 0.09 | EP-CL-5156 |
| NY, 1988 | EC | 0.90 | | 260 | 4 | bud stage | 15 | forage 1 | 3.2 1.0 | 0.19 0.06 | EP-CL-5158 |
| NY, 1988 | EC | 0.90 | | 260 | 4 | bud stage | 15 | forage 2 | 1.8 1.8 | 0.06 0.08 | EP-CL-5158 |
| NY, 1988 | EC | 0.90 | | 260 | 4 | bud stage | 15 | hay 1 | 2.1 5.8 | 0.08 0.38 | EP-CL-5158 |
| NY, 1988 | EC | 0.90 | | 260 | 4 | bud stage | 15 | hay 2 | 1.4 2.8 | 0.05 0.11 | EP-CL-5158 |
| NY, 1988 | WP | 0.90 | | 260 | 4 | bud stage | 15 | forage 1 | 3.0 1.6 | 0.26 0.10 | EP-CL-5159 |
| NY, 1988 | WP | 0.90 | | 260 | 4 | bud stage | 15 | forage 2 | 1.7 1.6 | 0.12 0.09 | EP-CL-5159 |
| NY, 1988 | WP | 0.90 | | 260 | 4 | bud stage | 15 | hay 1 | 2.6 3.9 | 0.18 0.26 | EP-CL-5159 |
| NY, 1988 | WP | 0.90 | | 260 | 4 | bud stage | 15 | hay 2 | 4.0 2.1 | 0.28 0.12 | EP-CL-5159 |
| OH, 1988 (Medium) | EC | 0.90 | | 190 | 4 | pre-bloom | 14 | forage 1 | 1.5 1.6 | 0.12 0.14 | EP-CL-5160 |
| OH, 1988 (Medium) | EC | 0.90 | | 190 | 4 | pre-bloom | 15 | forage 2 | 0.57 0.78 | <0.05 (2) | EP-CL-5160 |
| OH, 1988 (Medium) | EC | 0.90 | | 190 | 4 | pre-bloom | 14 | hay 1 | 4.8 2.8 | 0.36 0.20 | EP-CL-5160 |
| OH, 1988 (Medium) | EC | 0.90 | | 190 | 4 | pre-bloom | 15 | hay 2 | 1.4 1.5 | 0.06 0.06 | EP-CL-5160 |
| OH, 1988 (Medium) | WP | 0.90 | | 190 | 4 | pre-bloom | 14 | forage 1 | 1.6 1.6 | 0.12 0.14 | EP-CL-5161 |
| OH, 1988 (Medium) | WP | 0.90 | | 190 | 4 | pre-bloom | 15 | forage 2 | 1.2 1.6 | <0.05 0.08 | EP-CL-5161 |
| OH, 1988 (Medium) | WP | 0.90 | | 190 | 4 | pre-bloom | 14 14 | hay 1 | 5.2 5.2 c 0.54 | 0.38 0.40 c <0.05 | EP-CL-5161 |
| OH, 1988 (Medium) | WP | 0.90 | | 190 | 4 | pre-bloom | 15 | hay 2 | 5.7 3.2 | 0.14 0.10 | EP-CL-5161 |
| PA, 1988 | EC | 0.90 | | 260 | 4 | bloom | 15 | forage 1 | 1.6 1.5 | 0.06 0.08 | EP-CL-5162 |

| State, year (variety) | Application | | | | | growth stage | PHI, days | Sample[1] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | | parathion | paraoxon | |
| PA, 1988 | EC | 0.90 | | 260 | 4 | bloom | 15 | forage 2 | 1.2 0.85 | 0.06 0.05 | EP-CL-5162 |
| PA, 1988 | EC | 0.90 | | 260 | 4 | bloom | 15 | hay 1 | 3.5 5.0 | 0.22 0.31 | EP-CL-5162 |
| PA, 1988 | EC | 0.90 | | 260 | 4 | bloom | 15 | hay 2 | 1.6 2.2 | 0.24 0.16 | EP-CL-5162 |
| PA, 1988 | WP | 0.90 | | 260 | 4 | bloom | 15 | forage 1 | 2.3 1.5 | 0.18 0.08 | EP-CL-5163 |
| PA, 1988 | WP | 0.90 | | 260 | 4 | bloom | 15 | forage 2 | 0.75 0.85 | <0.05 (2) | EP-CL-5163 |
| PA, 1988 | WP | 0.90 | | 260 | 4 | bloom | 15 | hay 1 | 3.4 5.3 | 0.22 0.33 | EP-CL-5163 |
| PA, 1988 | WP | 0.90 | | 260 | 4 | bloom | 15 | hay 2 | 1.8 3.1 | 0.10 0.16 | EP-CL-5163 |
| WI, 1988 (Medium) | EC | 0.90 | | 240 | 4 | | 15 | forage 1 | 0.30 0.22 | <0.05 (2) | EP-CL-5164 |
| WI, 1988 (Medium) | EC | 0.90 | | 240 | 4 | | 15 | forage 2 | 4.4 6.0 | 0.14 0.20 | EP-CL-5164 |
| WI, 1988 (Medium) | EC | 0.90 | | 240 | 4 | | 15<br>15 | hay 1 | 0.44 0.64<br>c 0.12 | <0.05 0.08 | EP-CL-5164 |
| WI, 1988 (Medium) | EC | 0.90 | | 240 | 4 | | 15 | hay 2 | 23 21 | 0.75 0.61 | EP-CL-5164 |
| WI, 1988 (Medium) | WP | 0.90 | | 240 | 4 | | 15 | forage 1 | 0.34 0.41 | <0.05 0.05 | EP-CL-5165 |
| WI, 1988 (Medium) | WP | 0.90 | | 240 | 4 | | 15 | forage 2 | 5.4 12 | 0.18 0.34 | EP-CL-5165 |
| WI, 1988 (Medium) | WP | 0.90 | | 240 | 4 | | 15<br>15 | hay 1 | 0.32 0.50<br>c 2.9 | <0.05 0.06<br>c 0.10 | EP-CL-5165 |
| WI, 1988 (Medium) | WP | 0.90 | | 240 | 4 | | 15<br>15 | hay 2 | 28 25<br>c 0.05 | 1.0 0.80<br>c <0.05 | EP-CL-5165 |
| OH, 1988 (Medium) | EC | 0.90 | | 47 | ( 4 | pre-bloom | 14 | forage 1 | 0.35 0.68 | <0.05 0.11 | EP-CL-5166 |
| OH, 1988 (Medium) | EC | 0.90 | | 47 | ( 4 | pre-bloom | 15 | forage 2 | 0.90 1.3 | <0.05 0.05 | EP-CL-5166 |
| OH, 1988 (Medium) | EC | 0.90 | | 47 | ( 4 | pre-bloom | 14 | hay 1 | 0.86 2.4 | 0.06 0.18 | EP-CL-5166 |
| OH, 1988 (Medium) | EC | 0.90 | | 47 | ( 4 | pre-bloom | 15 | hay 2 | 2.5 3.2 | 0.14 0.14 | EP-CL-5166 |

[1] forage 1, forage 2, hay 1, etc refer to forage 1st cut, forage 2nd cut, hay 1st cut, etc
( aerial application

Table 69. Parathion residues in bean forage and vines from supervised trials in the USA. The duplicate values reported are for duplicate field samples.

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| NY, 1987 (Improved Tendergreen) | EC<br>EC | 0.56<br>0.90 | | 280 | 6 | 7<br>15 | vines | 0.38 0.34<br>0.35 0.26 | 0.12 0.11<br>0.23 0.23 | EP-LB-5083 |
| NY, 1987 (Improved Tendergreen) | WP<br>WP | 0.56<br>0.90 | | 280 | 6<br>6 | 7<br>15 | vines | 0.89 0.50<br>0.26 0.35 | 0.06 0.09<br>0.09 0.12 | EP-LB-5084 |
| WI, 1987 (FLO) | EC<br>EC | 0.56<br>0.90 | | 60 | ( 6<br>( 6 | 7<br>15 | vines | 0.14 0.20<br>0.30 0.19 | <0.05 (2)<br>0.06 <0.05 | EP-LB-5085 |
| WI, 1987 (FLO) | EC<br>EC | 0.56<br>0.90 | | 260 | 6<br>6 | 7<br>15 | vines | 0.38 0.31<br>0.52 0.74 | <0.05 (2)<br><0.05 (2) | EP-LB-5087 |
| WI, 1987 (FLO) | WP<br>WP | 0.56<br>0.90 | | 260 | 6<br>6 | 7<br>15 | vines | 0.44 0.75<br>0.36 0.49 | <0.05 0.13<br><0.05 (2) | EP-LB-5088 |
| OR, 1987 (Roma II) | WP | 0.90 | | 110 | 3 | 0<br>7<br>15<br>21<br>28 | forage | 28 18<br>1.9 1.9<br>0.52 1.2<br>2.2 1.2<br>0.40 0.96 | 2.4 0.40<br>0.83 1.0<br>0.34 0.33<br>0.40 0.31<br>0.25 0.43 | EP-LB-1130 |
| OR, 1987 (Roma II) | WP | 0.56 | | 110 | 3 | 0<br>7<br>15<br>21<br>28 | forage | 17 12<br>0.92 1.3<br>0.34 0.50<br>0.21 0.15<br>0.13 0.27 | 0.38 0.33<br>0.46 0.66<br>0.33 0.35<br>0.11 0.11<br>0.09 0.23 | EP-LB-1130 |
| OR, 1987 (Roma II) | EC | 0.56 | | 120 | 3 | 0<br>7<br>15<br>21<br>28 | forage | 19 12<br>0.56 0.84<br>0.08 0.10<br>0.23 0.24<br>0.39 0.30 | 1.0 0.75<br>0.09 0.13<br><0.05 (2)<br><0.05 0.07<br>0.29 0.17 | EP-LB-1131 |
| OR, 1987 (Roma II) | EC | 0.90 | | 120 | 3 | 0<br>7<br>15<br>21<br>28 | forage | 43 57<br>3.3 2.3<br>7.5 3.8<br>0.32 0.75<br>1.9 1.9 | 2.9 3.8<br>0.19 0.16<br>0.46 0.28<br>0.08 0.07<br>0.24 0.24 | EP-LB-1131 |
| OR, 1987 (OSU91) | EC | 0.56 | | 110 | ( 3 | 0<br>7<br>15<br>21<br>28<br>21 | forage | 17 16<br>0.28 0.25<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c 0.07 | 0.21 0.20<br>0.11 0.09<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-LB-1133 |
| OR, 1987 (OSU91) | EC | 0.90 | | | ( 3 | 0<br>7<br>15<br>21<br>28 | forage | 25 17<br>1.1 1.5<br><0.05 0.07<br>0.07 0.08<br><0.05 (2) | 0.29 0.21<br>0.21 0.24<br>0.06 0.07<br><0.05 (2)<br><0.05 (2) | EP-LB-1133 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| NY, 1987 (Improved Tendergreen) | EC | 0.56 | | 280 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 19 24<br>0.28 0.15<br>0.08 0.11<br>0.10 0.08<br>0.06 0.05 | 0.20 0.23<br>0.05 0.05<br>0.08 0.10<br><0.05 (2)<br><0.05 (2) | EP-LB-5083 |
| NY, 1987 (Improved Tendergreen) | EC | 0.90 | | | 6 | 0<br>7<br>15<br>21<br>28 | forage | 41 27<br>0.41 0.47<br>0.14 0.25<br>0.19 0.12<br>0.17 0.14 | <0.05 0.31<br>0.11 0.09<br>0.12 0.13<br>0.09 0.06<br>0.09 0.09 | EP-LB-5083 |
| NY, 1987 (Improved Tendergreen) | WP | 0.56 | | 280 | 6 | 0<br>7<br>15<br>21<br>28<br>0<br>28 | forage | 31 30<br>0.37 0.49<br>0.09 0.06<br>0.09 0.10<br>0.06 0.17<br>c 0.24<br>c 0.06 | 0.35 0.28<br><0.05 0.05<br><0.05 (2)<br>0.08 0.10<br><0.05 (2) | EP-LB-5084 |
| NY, 1987 (Improved Tendergreen) | WP | 0.90 | | | 6 | 0<br>7<br>15<br>21<br>28<br>0<br>28 | forage | 53 52<br>0.49 0.48<br>0.14 0.15<br>0.08 0.09<br>0.11 0.11<br>c 0.24<br>c 0.06 | 0.51 0.51<br>0.09 0.06<br>0.09 0.07<br>0.11 0.08<br>0.06 0.09 | EP-LB-5084 |
| WI, 1987 (FLO) | EC | 0.56 | | 60 | ( 6 | 0<br>7<br>15<br>21 | forage | 4.6 5.6<br>0.11 0.20<br>0.05 0.07<br><0.05 (2) | 0.05 0.07<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-LB-5085 |
| WI, 1987 (FLO) | EC | 0.90 | | | ( 6 | 0<br>7<br>15<br>21 | forage | 8.8 12<br>0.33 0.22<br>0.10 0.13<br><0.05 (2) | 0.41 0.11<br>0.10 <0.05<br><0.05 (2)<br><0.05 (2) | EP-LB-5085 |
| WI, 1987 (FLO) | EC | 0.56 | | 260 | 6 | 0<br>7<br>15<br>21 | forage | 18 14<br>0.41 0.30<br>0.18 0.10<br>0.06 0.06 | 0.30- 0.33<br>0.07 0.05<br><0.05 (2)<br><0.05 (2) | EP-LB-5087 |
| WI, 1987 (FLO) | EC | 0.90 | | | 6 | 0<br>7<br>15<br>21<br>0 | forage | 16 17<br>0.52 0.55<br>0.24 0.28<br>0.13 0.11<br>c 0.46 | 0.25 0.30<br>0.06 0.08<br><0.05 0.05<br><0.05 (2) | EP-LB-5087 |
| WI, 1987 (FLO) | WP | 0.56 | | 260 | 6 | 0<br>7<br>15<br>21 | forage | 18 11<br>0.34 0.41<br>0.13 0.12<br><0.05 0.06 | 0.15 0.15<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-LB-5088 |
| WI, 1987 (FLO) | WP | 0.90 | | 260 | 6 | 0<br>7<br>15<br>21 | forage | 32 31<br>0.67 0.63<br>0.21 0.23<br>0.15 0.08 | 0.22 0.19<br>0.07 0.10<br>0.07 <0.05<br><0.05 (2) | EP-LB-5088 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1987 (Kidney) | WP<br>WP | 0.56<br>0.90 | | 280<br>280 | 6 | 7<br>14 | vines | 0.54 0.57<br>0.43 0.47 | 0.07 0.08<br>0.09 0.12 | EP-DB-1113 |
| CA, 1987 (Kidney) | EC<br>EC | 0.56<br>0.90 | | 280<br>280 | 6<br>6 | 7<br>14<br>7 | vines | 0.69 0.88<br>0.13 0.52<br>c <0.05 | 0.23 0.21<br>0.21 0.33<br>c 0.05 | EP-DB-1114 |
| CA, 1987 (Kidney) | EC<br>EC | 0.56<br>0.90 | | 94<br>94 | ( 6<br>( 6 | 7<br>14<br>14 | vines | 0.07 0.33<br>0.14 <0.05<br>c 0.11 | <0.05 (2)<br><0.05 (2)<br>c <0.05 | EP-DB-1115 |
| ID, 1988 (Pinto) | EC<br>EC | 0.56<br>0.90 | | 94<br>94 | ( 6<br>( 6 | 7<br>15 | vines | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | EP-DB-1116 |
| ID, 1988 (Pinto) | WP<br>WP | 0.56<br>0.90 | | 190<br>190 | 6 | 7<br>15 | vine | 1.1 1.9<br>0.39 0.36 | 0.12 0.09<br>0.05 <.05 | EP-DB-1118 |
| ID, 1988 (Pinto) | EC<br>EC | 0.56<br>0.90 | | 190<br>190 | 6<br>6 | 7<br>15 | vines | 0.78 0.49<br>0.29 0.37 | 0.09 0.06<br><0.05 (2) | EP-DB-1119 |
| MI, 1988 (Seafarer Navy Bean) | EC | 0.56 | | 215 | 6 | 7<br>15 | vines | 0.80<br>0.77 | 0.10<br><0.05 | EP-DB-5142 |
| MI, 1988 (Seafarer Navy Bean) | EC | 0.90 | | 215 | 6 | 7<br>15 | vines | 0.93<br>0.66 | 0.10<br><0.05 | EP-DB-5142 |
| MI, 1988 (Seafarer Navy Bean) | WP | 0.56 | | 215 | 6 | 7<br>15 | vine | 0.51<br>0.40 | <0.05<br>0.11 | EP-DB-5143 |
| MI, 1988 (Seafarer Navy Bean) | WP | 0.90 | | 215 | 6 | 7<br>15 | vine | 0.40<br>0.47 | <0.05<br>0.10 | EP-DB-5143 |
| NE, 1988 (Pinto) | EC | 0.56 | | 190 | 7 | 7<br>15 | vines - dried | 0.16<br>0.11 | <0.05<br><0.05 | EP-DB-5144 |
| NE, 1988 (Pinto) | EC | 0.90 | | 190 | 7 | 7<br>15 | vines - dried | 0.36<br>0.30 | <0.05<br><0.05 | EP-DB-5144 |
| NE, 1988 (Pinto) | WP | 0.56 | | 190 | 7 | 7<br>15 | vines - dried | 0.08<br>0.09 | <0.05<br><0.05 | EP-DB-5145 |
| NE, 1988 (Pinto) | WP | 0.90 | | 190 | 7 | 7<br>15 | vines - dried | 0.27<br>0.27 | <0.05<br>0.14 | EP-DB-5145 |
| CA, 1987 (Kidney) | WP | 0.56 | | 280 | 6 | 0<br>7<br>14<br>21<br>28 | forage | 5.4 3.5<br>0.17 0.37<br>0.09 0.09<br>0.12 0.09<br>0.06 0.09 | 0.31 0.24<br>0.05 0.09<br><0.05 (2)<br>0.07 0.06<br><0.05 (2) | EP-DB-1113 |
| CA, 1987 (Kidney) | WP | 0.90 | | 280 | 6 | 0<br>7<br>14<br>21<br>28 | forage | 15 6.4<br>0.47 0.93<br>0.22 0.13<br>0.43 0.21<br>0.35 0.35 | 0.82 0.50<br>0.08 0.10<br>0.06 <0.05<br>0.12 0.11<br>0.07 <0.05 | EP-DB-1113 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1987 (Kidney) | EC | 0.56 | | 280 | 6 | 0<br>7<br>14<br>21<br>28 | forage | 6.8 9.0<br>0.20 0.45<br>0.15 0.12<br>0.09 0.09<br>0.09 0.11 | 0.24 0.32<br>0.12 0.20<br>0.07 0.07<br><0.05 (2)<br><0.05 (2) | EP-DB-1114 |
| CA, 1987 (Kidney) | EC | 0.09 | | 280 | 6 | 0<br>7<br>14<br>21<br>28 | forage | 13 14<br>0.45 0.49<br>0.14 0.18<br>0.13 0.18<br>0.14 0.14 | 0.36 0.41<br>0.18 0.12<br>0.07 0.19<br>0.07 0.09<br>0.08 0.08 | EP-DB-1114 |
| CA, 1987 (Kidney) | EC | 0.56 | | 94 | ( 6 | 0<br>7<br>14<br>21<br>28<br>0 | forage | 8.6 4.3<br>0.07 0.06<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c 0.07 | 0.18 0.13<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | EP-DB-1115 |
| CA, 1987 (Kidney) | EC | 0.90 | | 94 | ( 6 | 0<br>7<br>14<br>21<br>28 | forage | 11 6.2<br>0.07 0.19<br>0.05 <0.05<br>0.06 0.07<br><0.05 (2) | 0.21 0.17<br><0.05 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-DB-1115 |
| ID, 1988 (Pinto) | EC | 0.56 | | 94 | ( 6 | 0<br>7<br>15<br>21<br>28 | forage | 0.48 3.4<br>0.07 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | <0.05 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-DB-1116 |
| ID, 1988 (Pinto) | EC | 0.90 | | 94 | ( 6 | 0<br>7<br>15<br>21<br>28 | forage | 5.7 3.0<br><0.05 (2)<br>0.06 0.07<br><0.05 (2)<br><0.05 (2) | 0.16 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-DB-1116 |
| ID, 1988 (Pinto) | WP | 0.56 | | 190 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 15 31<br>1.1 1.2<br>0.21 0.13<br>0.16 0.21<br><0.05 0.06 | 0.15 0.28<br><0.05 0.14<br>0.06 <0.05<br><0.05 (2)<br>0.06 0.07 | EP-DB-1118 |
| ID, 1988 (Pinto) | WP | 0.90 | | 190 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 25 31<br>1.6 2.8<br>0.28 0.25<br>0.08 0.07<br>0.05 0.30 | 0.26 0.32<br>0.19 0.21<br>0.07 0.06<br><0.05 (2)<br>0.13 0.08 | EP-DB-1118 |
| ID, 1988 (Pinto) | EC | 0.56 | | 190 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 14 19<br>0.86 0.93<br><0.05 0.13<br>0.07 0.09<br><0.05 (2) | 0.07 0.08<br>0.06 0.11<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-DB-1119 |

| State, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| ID, 1988 (Pinto) | EC | 0.90 | | 190 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 37 21<br>2.0 1.3<br>0.13 0.22<br>0.08 0.09<br>0.06 0.06 | 0.17 0.11<br>0.14 0.10<br>0.08 0.08<br><0.05 (2)<br><0.05 (2) | EP-DB-1119 |
| MI, 1988 (Seafarer Navy Bean) | EC | 0.56 | | 215 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 6.2 4.1<br>0.30 0.25<br>0.32 0.25<br>0.22 0.17<br>0.09 0.09 | 0.18 0.12<br>0.10 0.11<br>0.05 <0.05<br><0.05 (2)<br><0.05 (2) | EP-DB-5142 |
| MI, 1988 (Seafarer Navy Bean) | EC | 0.90 | | 215 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 11<br>0.56 0.67<br>0.37 0.58<br>0.59 0.35<br>0.07 0.11 | 0.31<br>0.17 0.17<br><0.05 0.07<br>0.08 0.07<br><0.05 (2) | EP-DB-5142 |
| MI, 1988 (Seafarer Navy Bean) | WP | 0.56 | | 215 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 5.1 7.3<br>0.47 0.47<br>0.06 0.10<br>0.10 0.15<br>0.12 0.16 | 0.21 0.25<br>0.06 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-DB-5143 |
| MI, 1988 (Seafarer Navy Bean) | WP | 0.90 | | 215 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 7.3 7.6<br>1.0 0.82<br>0.16 0.29<br>0.16 0.16<br>0.26 0.24 | 0.29 0.36<br>0.08 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-DB-5143 |
| NE, 1988 (Pinto) | EC | 0.56 | | 190 | 7 | 0<br>7<br>15<br>21<br>28 | forage | 11 10<br>0.18 0.24<br>0.08 0.08<br><0.05 (2)<br><0.05 (2) | 0.09 0.11<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-DB-5144 |
| NE, 1988 (Pinto) | EC | 0.90 | | 190 | 7 | 0<br>7<br>15<br>21<br>28 | forage | 12 16<br>0.62 0.88<br>0.18 0.20<br>0.09 0.08<br>0.13 0.09 | 0.12 0.25<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-DB-5144 |
| NE, 1988 (Pinto) | WP | 0.56 | | 190 | 7 | 0<br>7<br>15<br>21<br>28<br>28 | forage | 8.8 7.3<br>0.12 0.09<br>0.05 0.13<br><0.05 (2)<br><0.05 (2)<br>c 0.06 | 0.29 0.24<br>0.06 0.06<br><0.05 0.06<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | EP-DB-5145 |
| NE, 1988 (Pinto) | WP | 0.90 | | 190 | 7 | 0<br>7<br>15<br>21<br>28 | forage | 18 18<br>0.47 0.45<br>0.13 0.23<br><0.05 (2)<br>0.05 <0.05 | 0.47 0.53<br>0.11 0.12<br>0.09 0.09<br><0.05 (2)<br><0.05 (2) | EP-DB-5145 |

( aerial application c: sample from control plot

Table 70. Parathion residues in field pea forage and vines from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | Sample | parathion | paraoxon | |
| WA, 1989 (Dark Skin 49) | EC | 0.56 | | 187 | 6 | 0<br>5<br>10<br>15 | forage | 5.1 8.1<br>2.4 2.5<br>1.7 1.4<br>0.89 0.69 | 0.38 1.1<br>0.65 0.53<br>0.93 0.42<br>0.60 0.35 | EP-PE-1176 |
| WA, 1989 (Dark Skin 49) | WP | 0.56 | | 187 | 5 | 0<br>5<br>10<br>15 | forage | 2.3 12<br>1.9 3.5<br>2.0 3.0<br>0.19 0.36 | 0.41 1.2<br>0.81 1.1<br>1.1 0.91<br><0.05 0.14 | EP-PE-1177 |
| WA, 1989 (Dark Skin 49) | EC | 0.56 | | 94 | ( 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>0.11 0.11<br>0.08 0.05 | 0.44 <0.05<br><0.05 (2)<br>0.19 0.25<br>0.28 0.36<br><0.05 (2)<br><0.05 (2) | EP-PE-1179 |
| WA, 1988 (Fraiser) | WP | 0.56 | | 140 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | 6.8 7.1<br>0.52 0.67<br>0.20 0.15<br>0.05 0.11<br>0.05 <0.05<br><0.05 0.12 | 0.23 0.23<br>0.11 0.13<br>0.08 0.06<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-PE-1205 |
| WA, 1988 (Fraiser) | EC | 0.56 | | 140 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | 6.2 7.9<br>1.1 0.93<br>0.13 0.11<br>0.32 0.14<br>0.17 0.10<br>0.10 0.10 | 0.24 0.30<br>0.22 0.20<br>0.06 0.06<br>0.08 <0.05<br><0.05 (2)<br><0.05 (2) | EP-PE-1206 |
| WA, 1988 (Fraiser) | EC | 0.56 | | 47 | ( 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage | 7.4 6.4<br>1.3 1.0<br><0.05 (2)<br>0.07 0.12<br>0.07 0.08<br>0.08 <0.05 | 0.23 0.18<br>0.22 0.19<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-PE-1208 |
| WI, 1988 (Ego) | EC | 0.56 | | 240 | 5 | 0<br>5<br>9<br>14<br>19<br>24 | forage | 2.1 2.1<br>0.10 0.07<br><0.05 (2)<br><0.05 (2)<br>0.05 <0.05<br><0.05 0.05 | 0.17 0.15<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-PE-5172 |
| WI, 1988 (Ego) | WP | 0.56 | | 240 | 5 | 0<br>5<br>9<br>14<br>19<br>24 | forage | 3.1 4.1<br>0.09 0.11<br>0.08 <0.05<br><0.05 (2)<br><0.05 (2)<br>0.05 <0.05 | 0.16 0.18<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-PE-5173 |

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | Sample | parathion | paraoxon | |
| WI, 1988 (9888F) | EC | 0.56 | | 38 | ( 5 | 0<br>6<br>10<br>15<br>20<br>25 | forage | <0.05 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-PE-5174 |
| WA, 1988 (Fraiser) | WP | 0.56 | | 140 | 6 | 10 | vine | 0.29 0.51 | 0.15 0.21 | EP-PE-1205 |
| WA, 1988 (Fraiser) | EC | 0.56 | | 140 | 6 | 10 | vine | 0.47 0.61 | 0.12 0.20 | EP-PE-1206 |
| WA, 1988 (Fraiser) | EC | 0.56 | | 47 | ( 6 | 10 | vine | 0.45 0.37 | 0.19 0.11 | EP-PE-1208 |
| WI, 1988 (Ego) | EC | 0.56 | | 240 | 5 | 9 | vine | 0.08 0.05 | <0.05 (2) | EP-PE-5172 |
| WI, 1988 (Ego) | WP | 0.56 | | 240 | 5 | 9 | vine | 0.08 0.07 | <0.05 (2) | EP-PE-5173 |
| WI, 1988 (9888F) | EC | 0.56 | | 38 | ( 5 | 10 | vine | <0.05 (2) | <0.05 (2) | EP-PE-5174 |

( aerial application

Table 71. Parathion residues in soya bean hay from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion | paraoxon | |
| IL, 1988 (BSR 201) | EC | 0.90 | | 240 | 2 | 20 | 0.23 0.25 | <0.05 (2) | EP-SY-5205 |
| IL, 1988 (BSR 201) | WP | 0.90 | | 240 | 2 | 20 | 0.13 0.08 | <0.05 (2) | EP-SY-5206 |
| IL, 1988 (BSR 201) | EC | 0.90 | | 9 | ( 2 | 20 | 0.46 0.36 | <0.05 (2) | EP-SY-5207 |
| MN, 1988 (Evans) | EC | 0.90 | | 190 | 2 | 20 | 0.21 0.57 | 0.12 0.23 | EP-SY-5208 |
| MN, 1988 (Evans) | WP | 0.90 | | 190 | 2 | 20 | 0.31 0.50 | 0.17 0.17 | EP-SY-5209 |
| MN, 1988 (Evans) | EC | 0.90 | | 37 | ( 2 | 20 | 0.23 0.32 | 0.12 0.10 | EP-SY-5210 |
| GA, 1988 (Coker 488) | EC | 0.90 | | 61 | 2 | 20 | 0.58 0.61 | <0.05 (2) | EP-SY-5211 |
| GA, 1988 (Coker 488) | WP | 0.90 | | 61 | 2 | 20 | 0.27 0.62 | <0.05 (2) | EP-SY-5212 |

( aerial application

Table 72. Parathion residues in almond hulls from supervised trials in the USA.

| State, year (variety) | Application | | | | | Growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1988 (Non-Pareil) | WP | 1.4 | 0.060 | 2300 | 1 | dormant | 180 | <0.05 (2) | <0.05 (2) | EP-AL-1148 (a) |
| CA, 1988 (Non-Pareil) | WP | 2.8 | | 650 | 1 | dormant | 180 | <0.05 (2) | <0.05 (2) | EP-AL-1148 (b) |
| CA, 1988 (Non-Pareil) | WP | 1.4 | 0.060 | 2300 | 3 | 1-2% hull split | 14 | 1.1 0.78 | 0.07 0.07 | EP-AL-1148 (c) |
| CA, 1988 (Non-Pareil) | WP | 2.8 | | 650 | 3 | 1-2% hull split | 14 | 1.5 1.1 | 0.06 <0.05 | EP-AL-1148 (d) |
| CA, 1988 (Non-Pareil) | EC | 1.4 | 0.060 | 2300 | 1 | dormant | 180 | <0.05 (2) | <0.05 (2) | EP-AL-1149 (a) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 650 | 1 | dormant | 180 | <0.05 (2) | <0.05 (2) | EP-AL-1149 (b) |
| CA, 1988 (Non-Pareil) | EC | 1.4 | 0.060 | 2300 | 3 | 1-2% hull split | 14 | 0.18 0.18 | 0.62 0.50 | EP-AL-1149 (c) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 650 | 3 | 1-2% hull split | 14 | 0.09 0.06 | 0.20 0.17 | EP-AL-1149 (d) |
| CA, 1988 (Non-Pareil) | WP | 1.4 | 0.060 | 2300 | 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1150 (a) |
| CA, 1988 (Non-Pareil) | WP | 2.8 | | 650 | 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1150 (a) |
| CA, 1988 (Non-Pareil) | WP | 1.4 | 0.060 | 2300 | 3 | 1-2% hull split | 14 | 0.90 0.90 | 0.07 0.10 | EP-AL-1150 (c) |
| CA, 1988 (Non-Pareil) | WP | 2.8 | | 650 | 3 | 1-2% hull split | 14 | 1.1 0.95 | <0.05 (2) | EP-AL-1150 (d) |
| CA, 1988 (Non-Pareil) | EC | 1.4 | 0.060 | 2300 | 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1151 (a) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 650 | 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1151 (b) |
| CA, 1988 (Non-Pareil) | EC | 1.4 | 0.060 | 2300 | 3 | 1-2% hull split | 14 | 0.20 0.21 | 0.64 0.58 | EP-AL-1151 (c) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 650 | 3 | 1-2% hull split | 14 | 0.05 0.11 | 0.16 0.15 | EP-AL-1151 (d) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 190 | ( 1 | dormant | 178 | <0.05 (2) | <0.05 (2) | EP-AL-1152 (a) |
| CA, 1988 (Non-Pareil) | EC | 2.8 | | 190 | ( 3 | 1-2% hull split | 14 | 0.21 0.20 | <0.05 (2) | EP-AL-1152 (b) |

| State, year (variety) | Application | | | | | Growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion | paraoxon | |
| CA, 1989 (Non-Pareil) | EC | 2.8 | | 190 | ( 3 | 1 week pre-hulls | 28 | <0.05 (2) | <0.05 (2) | EP-AL-1237 (a) |
| CA, 1989 (Non-Pareil) | EC | 2.8 | | 190 | ( 1 | dormant | 181 | 0.41 0.19 | <0.05 (2) | EP-AL-1237 (b) |
| CA, 1989 (Non-Pareil) | EC | 2.5 | 0.060 | 4100 | 3 | 1 week pre-hulls | 28 | 0.06 0.16 | <0.05 (2) | EP-AL-1238 (a) |
| CA, 1989 (Non-Pareil) | EC | 2.8 | | 420 | 3 | 1 week pre-hulls | 28 | 0.47 0.47 | <0.05 (2) | EP-AL-1238 (b) |
| CA, 1989 (Non-Pareil) | EC | 2.5 | 0.060 | 4100 | 1 | dormant | 186 | 0.08 0.08 | <0.05 (2) | EP-AL-1238 (c) |
| CA, 1989 (Non-Pareil) | EC | 2.8 | | 420 | 1 | dormant | 186 | 0.08 0.10 | <0.05 (2) | EP-AL-1238 (d) |
| CA, 1989 (Non-Pareil) | WP | 2.5 | 0.060 | 4100 | 3 | 1 week pre-hulls | 28 | 0.30 0.18 | <0.05 (2) | EP-AL-1239 (a) |
| CA, 1989 (Non-Pareil) | WP | 2.8 | | 420 | 3 | 1 week pre-hulls | 28 | 0.81 0.70 | <0.05 (2) | EP-AL-1239 (b) |
| CA, 1989 (Non-Pareil) | WP | 2.5 | 0.060 | 4100 | 1 | dormant | 186 | 0.10 0.13 | <0.05 (2) | EP-AL-1239 (c) |
| CA, 1989 (Non-Pareil) | WP | 2.8 | | 420 | 1 | dormant | 186 | 0.10 0.08 | <0.05 (2) | EP-AL-1239 (d) |

( aerial application

Table 73. Parathion residues in cotton gin trash from supervised trials in the USA. All EC formulations.

| State, year (variety) | Application | | | Growth stage | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | | % moisture | parathion | paraoxon | |
| AR, 1997 (PM 1220.BG,RR) | 1.1 | 37 | ( 6 | mature | 8 | 45 | 2.2 2.4 | 0.080 0.075 | MGB 97004.AR1 |
| MS, 1997 (Suregrow 125) | 1.1 | 19 | ( 6 | 80% open | 7 | 23 | 13 25 | 0.49 0.86 | MGB 97004.MS1 |
| TX, 1997 (Explorer) | 1.1 | 28 | ( 6 | 2 nodes above ? | 7 | 25 | 3.6 4.8 | 0.13 0.16 | MGB 97004.TX2 |
| TX, 1997 (HS-26) | 1.1 | 28 | ( 6 | 5 nodes above ? | 7 | 34 | 2.7 1.6 | 0.04 0.07 | MGB 97004.TX3 |
| TX, 1997 (HS-26) | 1.1 | 28 | ( 6 | 4 nodes above ? | 7 | 10 | 5.2 5.7 | 0.26 0.38 | MGB 97004.TX4 |

( aerial application

Table 74. Parathion residues in sugar beet fodder from supervised trials in the USA in 1988.

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | parathion | paraoxon | |
| CA (SS NB2) | EC | 0.90 | ( 6 | 21 | 0.68 0.67 | 0.08 0.06 | EP-SB-1097 [1] |
| CA (SS NB2) | WP | 0.90 | 6 | 21 | 1.3 0.76 | <0.05 (2) | EP-SB-1098 [1] |
| CA (SS NB2) | EC | 0.90 | 6 | 21 | 0.71 0.57 | <0.05 (2) | EP-SB-1099 [1] |
| ID (WS 88) | EC | 0.90 | 6 | 21 | 0.27 0.29 | <0.05 0.06 | EP-SB-1125 [1] |
| ID (WS 88) | WP | 0.90 | 6 | 21 | 0.38 0.71 | 0.06 0.10 | EP-SB-1126 [1] |
| MN (Ultramono) | EC | 0.90 | 6 | 21 | 0.79 0.71 | <0.05 0.07 | EP-SB-5089 [1] |
| MN (Ultramono) | WP | 0.90 | 6 | 21 | 1.1 0.91 | 0.06 0.05 | EP-SB-5090 [1] |
| ND (ACS ACH176) | EC | 0.90 | 6 | 21 | 0.66 0.62 | <0.05 (2) | EP-SB-5177 [1] |
| ND (ACS ACH176) | WP | 0.90 | 6 | 21 | 0.79 0.80 | <0.05 (2) | EP-SB-5178 [1] |
| MN (Ultramono) | EC | 0.90 | ( 6 | 21 | 0.13 0.23 | <0.05 (2) | EP-SB-5179 [1] |

[1] unvalidated analytical data
( aerial application

Table 75. Parathion residues in sunflower forage from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimation of maximum residue levels.

| State, year (variety) | Application | | | | growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion | paraoxon | |
| ND, 1989 (Sigco Hybrid 465A) | EC | 1.1 | 190 | 3 | post-flower | 0<br>10<br>20<br>30<br>40 | 21 26<br>4.4 3.8<br>1.6 1.9<br>3.2 5.5<br>2.0 2.8 | 0.14 0.20<br>0.13 0.11<br>0.09 0.14<br>0.16 0.22<br>0.10 0.12 | EP-SS-1240 |
| ND, 1989 (Sigco Hybrid 465A) | WP | 1.1 | 190 | 3 | post-flower | 0<br>10<br>20<br>30<br>40 | 18 17<br>4.4 1.6<br>0.60 0.73<br>4.6 3.3<br>5.5 2.2 | 0.22 0.13<br>0.12 0.06<br><0.05 (2)<br>0.20 0.11<br>0.16 0.10 | EP-SS-1241 |
| ND, 1989 (Sigco Hybrid 465A) | EC | 1.1 | 47 | ( 3 | post-flower | 0<br>10<br>20<br>30<br>40 | 4.2 4.2<br>1.6 2.4<br>1.2 1.2<br>0.76 1.3<br>2.2 0.47 | <0.05 (2)<br>0.08 0.11<br>0.06 <0.05<br>0.05 0.08<br>0.06 <0.05 | EP-SS-1242 |
| TX, 1989 (Sun Valley 230) | EC | 1.1 | 190 | 3 | 10th true leaf | 0<br>10<br>20<br>30<br>40 | 32 67<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 0.06 0.13<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-SS-1243 |

| State, year (variety) | Application | | | | growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion | paraoxon | |
| TX, 1989 (Sun Valley 230) | WP | 0.1 | 190 | 3 | 10th true leaf | 0<br>10<br>20<br>30<br>40 | 42 32<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 0.10 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-SS-1244 |
| TX, 1989 (Sun Valley 230) | EC | 1.1 | 59 | (<br>3 | 10th true leaf | 0<br>10<br>20<br>30<br>40 | 62 67<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 0.12 0.22<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | EP-SS-1245 |

( aerial application

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### In processing

The Meeting received information on parathion residues in processed lemons, grapefruit, oranges, apples, grapes, olives, tomatoes, potatoes, sugar beet, oats, maize, rice, sorghum, wheat sunflower seed, cotton seed and canola. Trials on olives, tomatoes, potatoes and sugar beet were based on unvalidated analytical data from Craven Laboratories and could not be used.

Lemons. In 2 trials in 1988 in California, USA, lemons treated with parathion applied with a commercial sprayer in the first trial and with a handgun sprayer in the second were harvested 30 days after the last application (LeRoy, 1990i). An EC formulation of 16 kg ai/ha, spray concentration 0.72 kg ai/hl and volume 22 hl/ha, was used at 3 times the label rate. Residues decreased in the juice during processing but increased in the cold pressed oil (Table 76). The process is outlined in Figure 4.

Table 76. Parathion residues in lemons and their processed commodities from supervised trials and processing in 1988 in the USA (LeRoy, 1990i).

| Variety | Application | | | PHI days | Residues, mg/kg | | | Ref |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/hl | no. | | commodity | parathion | paraoxon | |
| Lisbon | EC | 0.72 | 3 | 30 | fruit, unwashed<br>juice<br>wet peel<br>dried peel<br>molasses<br>cold pressed oil | 12<br>0.29<br>5.7<br>14<br>0.40<br>sample lost | 0.49<br><0.05<br>0.08<br>0.18<br><0.05 | EP-LM-2013 |
| Lisbon | EC | 0.72 | 3 | 30 | fruit, unwashed<br><br>juice<br>wet peel<br>dried peel<br>molasses<br>cold pressed oil | 0.15 7.2<br>9.4 3.2<br>0.14<br>7.9<br>17<br>1.6<br>495 | <0.05 0.09<br>0.11 0.07<br><0.05<br>0.07<br>0.15<br><0.05<br>4.1 | EP-LM-2014 |

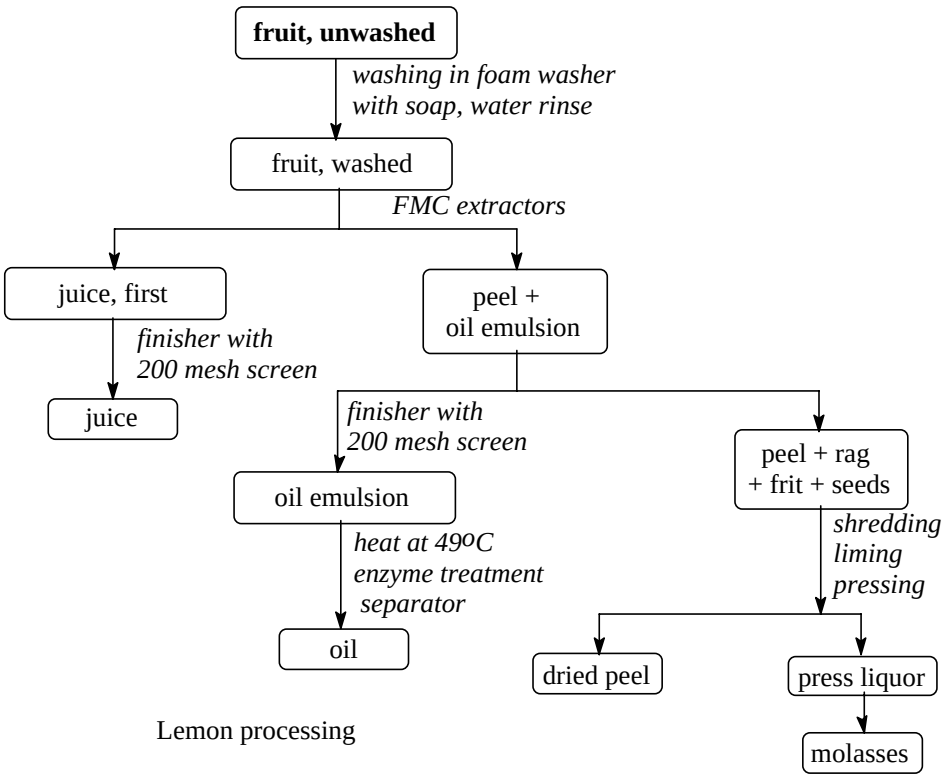



Figure 4. Processing of lemons (LeRoy, 1990i).

Grapefruit. In a trial in 1988 in Florida, USA, grapefruit were sprayed with parathion by commercial airblast equipment and harvested 31 days after the last application (LeRoy, 1990g). The application rate of 34 kg ai/ha of an EC formulation was 3 times the label concentration. Residue levels decreased in the juice during processing but increased in cold pressed oil (Table 77). The process was the same as that described for oranges below (Figure 5).

Table 77. Parathion residues in red grapefruit and its processed commodities from a supervised trial and processing in the USA (LeRoy, 1990h).

| State, year | Application | | | PHI, days | Residues, mg/kg | | | Ref |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | commodity | parathion | paraoxon | |
| FL, 1988 | EC | 34 | 3 | 31 | fruit, unwashed | 5.4 | 0.43 | EP-GF-2015 |
| | | | | | juice | <0.05 | <0.05 | |
| | | | | | wet pulp | 8.7 | 0.71 | |
| | | | | | dried pulp | 32 | 1.7 | |
| | | | | | molasses | 1.2 | 1.3 | |
| | | | | | cold pressed oil | 1650 | 49 | |

Oranges. In one US trial in Florida in 1988 an orange orchard was sprayed with parathion by commercial airblast equipment at 0.72 kg ai/hl (34 kg ai/ha) of an EC formulation (3 times the label concentration) and the oranges were harvested 31 days after the last application (LeRoy, 1990g). “Wet” pulp is the residual juice sacs after juice separation and “dried” pulp is the residual chopped and dried peel after oil extraction (Figure 5). Residues decreased in the juice during processing but increased substantially in cold pressed oil (Table 78).

Table 78. Parathion residues in oranges and their processed commodities from a supervised trial in the USA (LeRoy, 1990g).

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | commodity | parathion | paraoxon | |
| FL, 1988 (Hamlin) | EC | 34 | 3 | 31 | fruit, unwashed | 7.0 | 0.40 | EP-OR-2011 |
| | | | | | fruit, washed | 6.3 | 0.38 | |
| | | | | | juice | 0.36 | <0.05 | |
| | | | | | wet pulp | 4.9 | 0.40 | |
| | | | | | dried pulp | 34 | 1.4 | |
| | | | | | molasses | 4.6 | 0.08 | |
| | | | | | cold pressed oil | 1674 | 33 | |

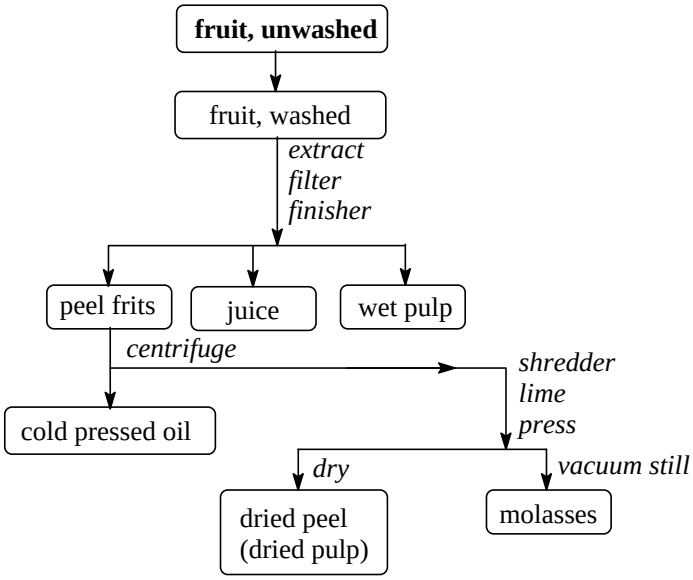
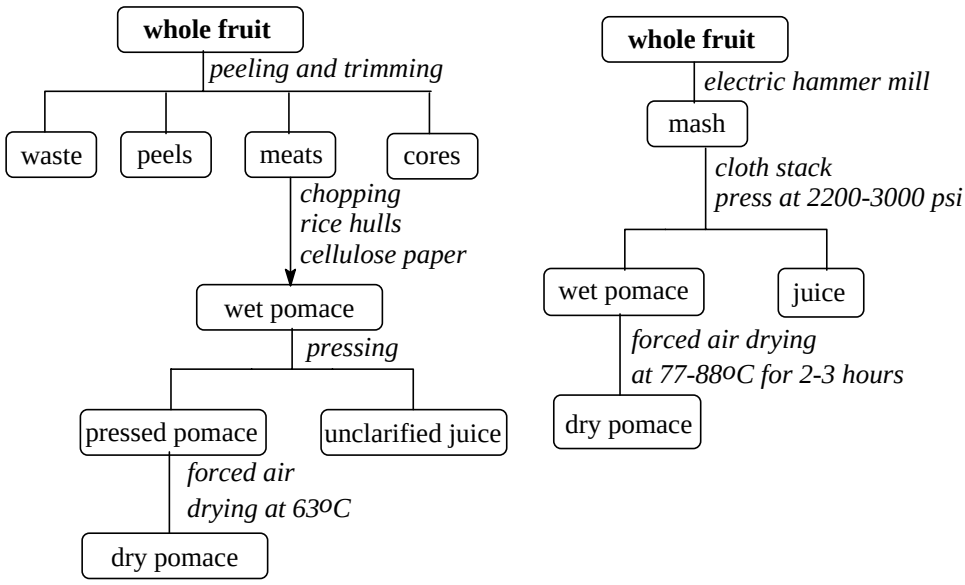



Figure 5. Processing of oranges (LeRoy, 1990g).

Apples. In a processing trial (EP-AP-2018) in 1988 in the USA (NY) apples were treated by tractor-mounted airblast equipment with parathion at 1.2 kg ai/hl (22 kg ai/ha) and harvested 31 days after the last application (Cañez, 1990f). In a second trial (EP-AP-2017) in Oregon, apples were treated by $CO_2$ backpack sprayer with parathion at 0.48 kg ai/hl (7.8 kg ai/ha) and processed into juice and pomace. The processing procedures were quite different (see Figure 6, e.g. peels were included or excluded in juice production). “Wet” pomace in one trial is before juice production and in the other is the residue after juice removal.

Table 79. Parathion residues in apples and processed commodities from supervised trials and processing in the USA (Cañez, 1990f).

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/hl | no. | | commodity | parathion | paraoxon | |
| NY, 1988 (Romes) | WP | 1.2 | 5 | 14 | apples, whole | 2.6 9.6 | <0.05 0.20 | EP-AP-2018 |
| | | | | | juice, unclarified | 0.40 | 0.05 | |
| | | | | | wet pomace | 19 | 0.45 | |
| | | | | | dry pomace | 12.5 | 0.32 | |
| OR, 1988 (Red Delicious) | WP | 0.48 | 6 | 14 | apples, whole | 2.7 | 0.13 | EP-AP-2017 |
| | | | | | juice, unclarified | <0.05 | <0.05 | |
| | | | | | wet pomace | 0.40 | 0.06 | |
| | | | | | dry pomace | 1.1 | 0.25 | |




Apple processing, EP-AP-2017

Apple processing, EP-AP-2018

Figure 6. Processing of apples (Cañez, 1990f).

Grapes. In 3 trials in the USA in 1988 grapes were treated by commercial spray equipment (California) and by $CO_2$ backpack sprayer (Oregon) with parathion at a concentration of 0.60 kg ai/hl (application rates 11, 11 and 8.4 kg ai/ha) of an EC formulation at each site at 5 times the label concentration. and harvested 14 days after the last application for juicing and drying (LeRoy, 1990f). About 23 kg of grapes were harvested for processing into juice and pomace and 55 kg for drying (Table 890).

Table 80. Parathion residues in grapes and processed commodities from supervised trials and processing in the USA (LeRoy, 1990f).

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/hl | no. | | commodity | parathion | paraoxon | |
| CA, 1988 (Thompson Seedless) | EC | 0.60 | 3 | 14 | grapes<br>juice<br>wet pomace<br>dried pomace<br>raisins<br>raisin waste | 2.1<br>0.31 0.24<br>8.0 10<br>22 19<br>0.58<br>1.4 | <0.05<br><0.05 (2)<br>0.11 0.07<br>0.14 0.21<br>0.06<br>0.12 | EP-GR-2019 |
| CA, 1988 (Thompson Seedless) | EC | 0.60 | 3 | 14 | grapes<br>juice<br>wet pomace<br>dried pomace<br>raisins<br>raisin waste | 2.7<br>0.36<br>15<br>15<br>0.90<br>3.8 | <0.05<br><0.05<br>0.18<br>0.20<br>0.13<br>0.75 | EP-GR-2020 |
| OR, 1988 (Pinot Noir) | EC | 0.60 | 3 | 14 | grapes<br>juice<br>wet pomace<br>dried pomace | 9.2<br>0.05<br>19<br>36 | 0.17<br><0.05<br>0.36<br>0.36 | EP-GR-2021 |

Olives. In 2 trials in the USA in 1988 olives were sprayed with parathion at a concentration of 0.48 kg ai/hl with an EC formulation at each site (5 times the label concentration) by commercial spray equipment and harvested 42 days after the last application for oil production (Cañez, 1990d). The olives (about 450 kg) were first washed and then ground. Oil and water were separated by centrifugation.

Table 81. Parathion residues in olives and olive oil from supervised trials and processing in the USA (Cañez, 1990d).

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/hl | no. | | commodity | parathion | paraoxon | |
| CA, 1988 (Manzinella) | EC | 0.48 | 3 | 42 | fruit<br>oil | 0.37<br>4.2 | <0.05<br>0.08 | EP-OL-2036 [1] |
| CA, 1988 (Manzinella) | EC | 0.48 | 3 | 42 | fruit<br>oil | 4.1<br>23 | 0.11<br>0.31 | EP-OL-2037 [1] |
| CA, 1988 (Manzinella) | EC | 0.096 | 3 | 42 | fruit | <0.05 | <0.05 | EP-OL-2036 [1] |
| CA, 1988 (Manzinella) | EC | 0.096 | 3 | 42 | fruit | 0.11 | <0.05 | EP-OL-2037 [1] |

[1] unvalidated analytical data

Tomatoes. In 2 US trials in 1988 crops were treated 5 or 6 times by ground equipment ($CO_2$ backpack sprayers) with parathion at 5.6 kg ai/ha with an EC formulation at each site at 5 times the label rate. and harvested 10 days after the last application (Cañez, 1990c). About 90 kg of tomatoes were sent for processing (Table 82).

Table 82. Parathion residues in tomatoes and processed commodities from supervised trials and processing in the USA.

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | commodity | parathion | paraoxon | |
| CA, 1988 (Roma) | EC | 5.6 | 6 | 10 | fruit | 0.45 | <0.05 | EP-TO-2009 [1] |
| | | | | | juice | 0.85 | <0.05 | |
| | | | | | puree | 1.4 | <0.05 | |
| | | | | | wet pomace | 11 | 0.11 | |
| | | | | | dry pomace | 28 | 1.1 | |
| | | | | | catsup | 0.80 | <0.05 | |
| CA, 1988 (Murietta) | EC | 5.6 | 5 | 10 | fruit | 0.55 | <0.05 | EP-TO-2010 [1] |
| | | | | | juice | 0.55 | <0.05 | |
| | | | | | puree | 1.1 | <0.05 | |
| | | | | | wet pomace | 9.5 | 0.09 | |
| | | | | | dry pomace | 25 | 2.0 | |
| | | | | | catsup | 0.58 | <0.05 | |

[1] unvalidated analytical data

Potatoes. In 2 US trials in 1988 in Washington and Idaho crops were sprayed by ground self-propelled research sprayers with 6 applications of parathion at 7-day intervals at 5.6 kg ai/ha with an EC formulation at both sites (i.e. at 5 times the label rate) and harvested 5 days after the last application for processing (Cañez, 1990a). The tubers (about 70 and 90 kg) were sent for milling to produce chips, flakes and granules (Table 83). Unfortunately residue levels were too low in both trials to provide information on the fate of parathion during processing.

Table 83. Parathion residues in potatoes and processed commodities from supervised trials and processing in the USA.

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | commodity | parathion | paraoxon | |
| WA, 1988 (Russet Burbank) | EC | 5.6 | 6 | 5 | tuber | <0.05 | <0.05 | EP-PO-2002 [1] |
| | | | | | wet peel chip | <0.05 | <0.05 | |
| | | | | | chips | <0.05 | <0.05 | |
| | | | | | dried peel chip | <0.05 | <0.05 | |
| | | | | | wet peel flake | <0.05 | <0.05 | |
| | | | | | dried peel flake | 0.08 | <0.05 | |
| | | | | | flakes | <0.05 | <0.05 | |
| | | | | | granules | <0.05 | <0.05 | |
| ID, 1988 (Russet Burbank) | EC | 5.6 | 6 | 6 | tuber | <0.05 | <0.05 | EP-PO-2001 [1] |
| | | | | | wet peel chip | <0.05 | <0.05 | |
| | | | | | chips | <0.05 | <0.05 | |
| | | | | | dried peel chip | 0.11 | <0.05 | |
| | | | | | wet peel flake | <0.05 | <0.05 | |
| | | | | | dried peel flake | 0.14 | <0.05 | |
| | | | | | flakes | <0.05 | <0.05 | |
| | | | | | granules | <0.05 | <0.05 | |

[1] unvalidated analytical data

Sugar beet. In 2 trials in the USA in 1988 in California and North Dakota crops were sprayed (by a $CO_2$ backpack sprayer in trial EP-SB-2003 and a tractor mounted sprayer in trial EP-SB-2004) with 6 applications of parathion at 4.5 kg ai/ha with an EC formulation at 5 times the label rate at 7-day

intervals and harvested 15 days after the last application (Cañez, 1990b). The roots (about 115 kg) were sent for processing (Table 84).

The roots were washed, sliced, and then extracted counter-current with water at c. 80°C to produce “diffusion juice”. The pulp was pressed and dried to less than 3% moisture. The press water was returned to the diffusion juice, which was then treated with approximately 1.5% lime (CaO) at 80°C for 15 min and brought to a pH of about 9.5 with $CO_2$. After the addition of a settling aid the “thin juice” was decanted and concentrated to “thick juice” (about 70% solids) under vacuum at 65°C. After decantation from the sludge, refined sugar was produced by about 3 crystallizations; the collected and concentrated mother liquors constituted the molasses. The contact surfaces used in the processes were stainless steel, teflon, food-grade plastics and glass.

Table 84. Parathion residues in sugar beet and processed commodities from supervised trials and processing in the USA.

| State, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | commodity | parathion | paraoxon | |
| CA, 1988 (SS NB2) | EC | 4.5 | 6 | 15 | root<br>dehydrated pulp<br>molasses<br>refined sugar | 0.07<br>0.28<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05<br><0.05 | EP-SB-2003 [1] |
| ND, 1988 (ACS ACH176) | EC | 4.5 | 6 | 15 | root<br>dehydrated pulp<br>molasses<br>refined sugar | 0.19<br>1.2<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05<br><0.05 | EP-SB-2004 [1] |

[1] unvalidated analytical data

Oats. In 2 US trials in Missouri and North Dakota in 1989 crops were sprayed with 6 applications at 7-day intervals of parathion at 4.2 kg ai/ha with an EC formulation (5 times the label rate) with a ground $CO_2$ backpack sprayer in trial EP-OT-2025, and with a commercial tractor sprayer in trial EP-OT-2024 and harvested 15 days after the last application for processing (LeRoy, 1990d). Oats (about 36 kg) were sent for milling (16 kg milled) to produce hulls, rolled oats, bran and flour

Table 85).

Table 85. Residues of parathion and paraoxon in oats and processed commodities in the USA from the milling of oats treated 6 times in the field at 4.2 kg ai/ha and harvested 15 days after the last treatment (LeRoy, 1990d).

| Location, variety | Applic. rate, kg ai/ha | Commodity | Residues, mg/kg | | Reference |
|---|---|---|---|---|---|
| | | | parathion | paraoxon | |
| Missouri, Otee | 4.2 | grain<br>bran<br>flour<br>rolled oats<br>hulls | 0.96<br>0.39<br>0.40<br>0.38<br>2.5 | 0.23<br><0.05<br>0.06<br><0.05<br>0.68 | EP-OT-2025 |
| North Dakota, Dumont | 4.2 | grain<br>bran<br>flour<br>rolled oats<br>hulls | 3.2<br>1.3<br>2.2<br>0.93<br>- | 0.50<br>0.19<br>0.12<br>0.12<br><0.05<br>c 1.5 [1] | EP-OT-2024 |

[1] c: sample from control plot. Possible mislabelling of treated and control samples.

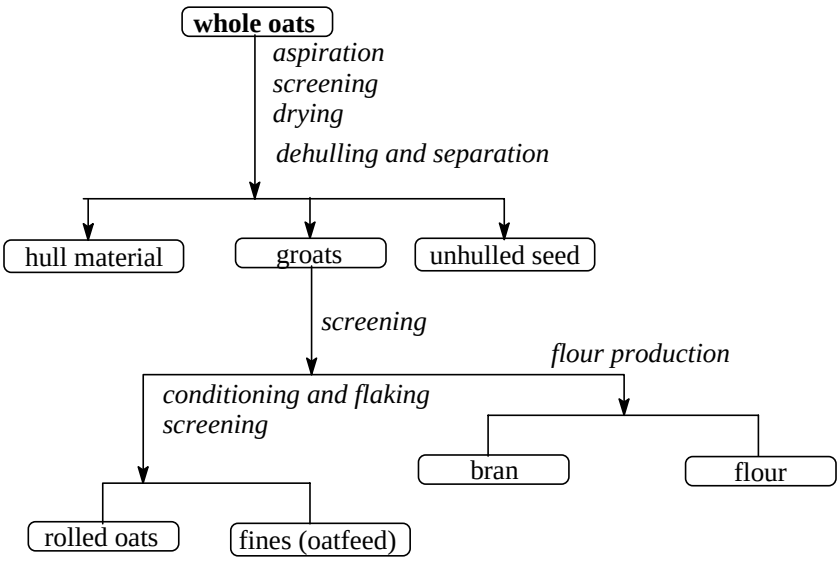



Figure 7. Milling of oats (LeRoy, 1990d).

Maize. Bookbinder (1998a) measured the residues in aspirated grain fractions (AGF or grain dust) of maize harvested from treated plots. In Wisconsin, USA, in 1997-98 maize was treated 6 times at 0.84 kg ai/ha by aerial application at approximately 5-day intervals and harvested 12 days after the last treatment. The grain (225 kg) was oven-dried at 61°C and transported on a simulated commercial grain bucket elevator and drag conveyor system while an aspiration system collected and filtered the air from various points. The AGF were collected in sieves and an initial 220.7 kg produced 410 g. Initial residues in the grain were 0.072 mg/kg parathion and <0.01 mg/kg paraoxon, and in the AGF 0.43 mg/kg parathion and 0.027 mg/kg paraoxon.

In 2 US trials in Missouri and Iowa in 1989 crops were sprayed by a research sprayer for trial EP-CN-2042 and by a $CO_2$ backpack sprayer for trial EP-CN-2043 with 6 applications at 5-day intervals of parathion at 5.6 kg ai/ha with an EC formulation at 5 times the label rate, and harvested 12 days after the last application for processing (LeRoy, 1990e). The maize (about 160 kg) was sent for milling to produce flour, grits, meal, starch, crude oil and refined oil (Table 886).

Table 86. Residues of parathion and paraoxon in maize and processed commodities in the USA from the milling of maize treated 6 times in the field at 5.6 kg ai/ha and harvested 12 days after the last treatment (LeRoy, 1990e).

| Location, variety | Applic. rate kg ai/ha | Commodity | Residues, mg/kg | | Reference |
|---|---|---|---|---|---|
| | | | parathion | paraoxon | |
| Iowa, DeKalb 547 | 5.6 | grain | 0.137 | <0.05 | EP-CN-2042 |
| | | meal | 0.082 | <0.05 | |
| | | grits | <0.05 | <0.05 | |
| | | flour | 0.065 | <0.05 | |
| | | starch | <0.05 | <0.05 | |
| | | crude oil, dry milled | 0.065 | <0.05 | |
| | | crude oil, wet milled | 0.18 | <0.05 | |
| | | refined oil, dry milled | 0.11 | <0.05 | |
| | | refined oil, wet milled | 0.18 | <0.05 | |
| Missouri, Funks G-4500 | 5.6 | grain | 0.181 | <0.05 | EP-CN-2043 |
| | | meal | 0.16 | <0.05 | |
| | | grits | 0.18 | <0.05 | |
| | | flour | 0.16 | <0.05 | |
| | | starch | <0.05 | <0.05 | |

| Location, variety | Applic. rate kg ai/ha | Commodity | Residues, mg/kg | | Reference |
|---|---|---|---|---|---|
| | | | parathion | paraoxon | |
| | | crude oil, dry milled | 0.12 | <0.05 | |
| | | crude oil, wet milled | 0.61 | <0.05 | |
| | | refined oil, dry milled | 0.37 | <0.05 | |
| | | refined oil, wet milled | 0.63 | <0.05 | |

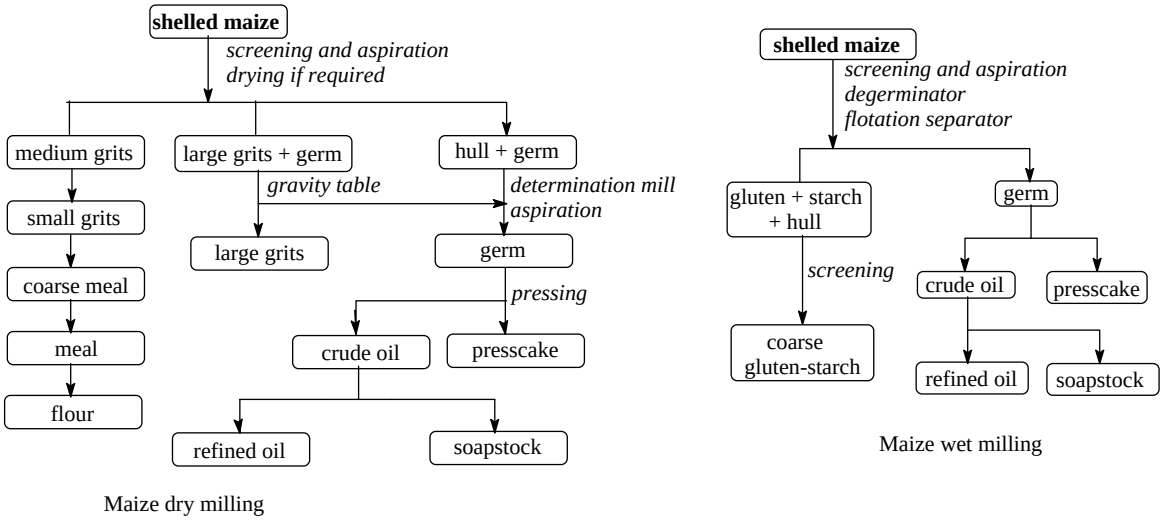



Figure 8. Milling of maize (LeRoy, 1990e).

Rice. In 2 trials in the USA (California and Texas) in 1988 rice was treated by ground equipment with parathion (3 applications at 7-day intervals) and harvested 1 day after the last application for processing (LeRoy, 1990a). An application rate of 0.56 kg ai/ha of an EC formulation at each site was 5 times the label rate. Harvested rice (about 14 kg in trial EP-RI-2026 and 23 kg in trial EP-RI-2027) was milled to produce brown rice, hulls, bran and polished rice (Table 87).

Table 87. Residues of parathion and paraoxon in milled rice grain and its processed commodities treated 3 times in the field at 0.56 kg ai/ha and harvested 1 day after the last treatment (LeRoy, 1990a).

| Location, variety | Applic. rate kg ai/ha | Commodity | Residues, mg/kg | | Reference |
|---|---|---|---|---|---|
| | | | parathion | paraoxon | |
| California, M201 | 0.56 | grain | 1.6 | 0.15 | EP-RI-2026 |
| | | brown rice | 0.15 | <0.05 | |
| | | bran | 0.59 | <0.05 | |
| | | hulls | 6.3 | 0.66 | |
| | | polished rice | <0.05 | <0.05 | |
| Texas, Gulfmont | 0.56 | grain | 0.72 | 0.23 | EP-RI-2027 |
| | | brown rice | 0.23 | <0.05 | |
| | | bran | 0.68 | 0.19 | |
| | | hulls | 2.4 | 0.50 | |
| | | polished rice | <0.05 | <0.05 | |

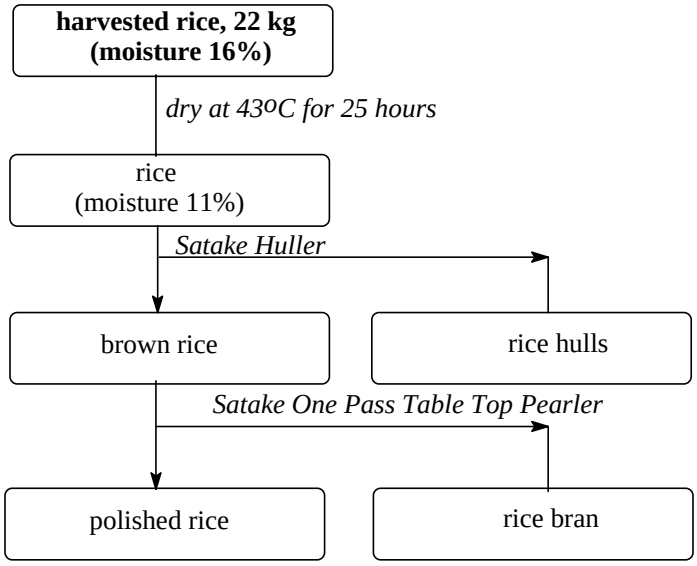



Figure 9. Processing of rice in trial EP-RI-2027 (LeRoy, 1990a).

Sorghum. In 2 US trials in Missouri and Texas in 1988 sorghum was treated 6 times at 5.6 kg ai/ha with an EC formulation (5 times the label rate) by ground equipment at 7-day intervals and harvested 12 days after the last application (LeRoy, 1990b). Harvested sorghum (minimum 27 kg) was sent for processing by wet and dry milling to produce decorticated grain, flour, grits and starch (Table 88).

Table 88. Residues of parathion and paraoxon in sorghum grain and processed commodities from the milling of sorghum treated 6 times in the field at 5.6 kg ai/ha and harvested 12 days after the last treatment (LeRoy, 1990b).

| Trial, variety | Applic. rate kg ai/ha | Commodity | Residues, mg/kg | | Reference |
|---|---|---|---|---|---|
| | | | parathion | paraoxon | |
| Missouri, | 5.6 | Grain | 20.2 | 0.56 | EP-SG-2028 |
| Funk's G-623 | | Decortication grain | 7.2 | 0.35 | |
| | | Coarse bran | 31 | 1.3 | |
| | | Fine bran | 23 | 0.86 | |
| | | Grits | 6.7 | 0.35 | |
| | | Flour | 4.6 | 0.23 | |
| | | Starch | 0.19 | 0.067 | |
| Texas, | 5.6 | Grain | 8.5 | 0.70 | EP-SG-2029 |
| Funk 522 DR | | Decortication grain | 5.2 | 0.40 | |
| | | Coarse bran | 32 | 1.8 | |
| | | Fine bran | 8.2 | 1.1 | |
| | | Grits | 4.8 | 0.48 | |
| | | Flour | 4.8 | 0.43 | |
| | | Starch | 0.10 | 0.063 | |

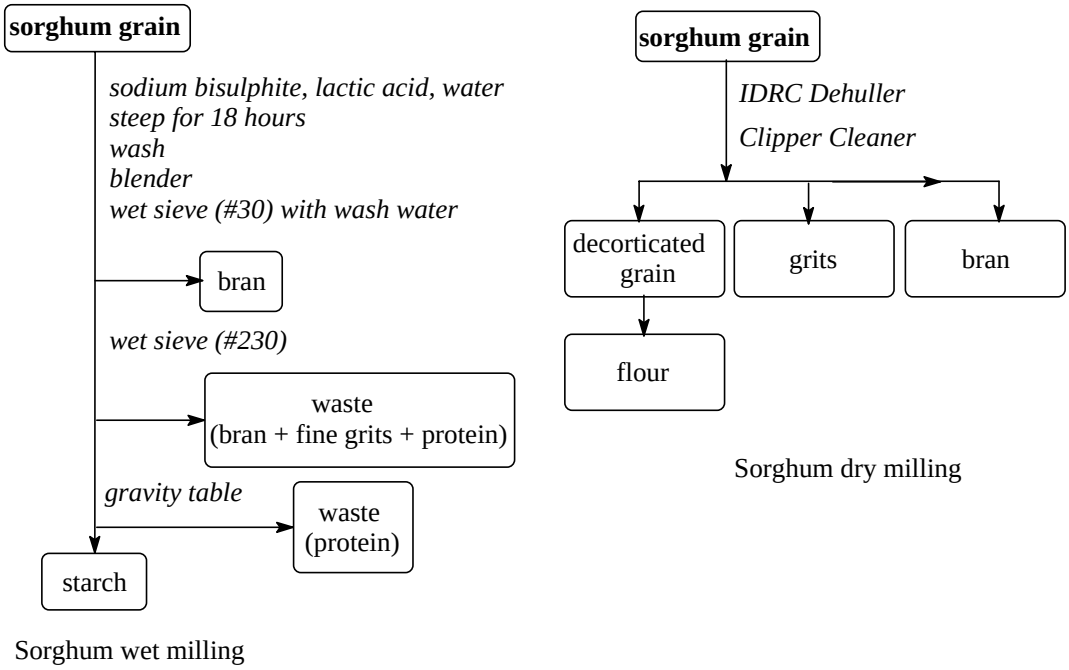



Figure 10. Processing of sorghum in Trial EP-SG-2028 (LeRoy, 1990b).

Bookbinder (1998b) measured the residues in aspirated grain (grain dust) after movement and aspiration of field-treated sorghum. In Wisconsin, USA, in 1997, the crop was sprayed 6 times from the air at 7-day intervals with parathion at 1.1 kg ai/ha and harvested 12 days after the last treatment. The harvested grain (225 kg) was oven-dried at 61°C and transported in a simulated commercial grain bucket elevator and drag conveyor system while an aspiration system collected and filtered the air from various points. The AGF were collected from the air in sieves. The grain (216 kg) produced 752 g of AGF. Initial residues in the grain were 2.2 mg/kg parathion and 0.057 mg/kg paraoxon, producing residues in the AGF of 4.2 mg/kg parathion and 0.15 mg/kg paraoxon.

Wheat. Spring wheat in North Dakota, USA, in 1992 was sprayed twice with parathion EC at 4.2 kg ai/ha by a spray boom fitted to a tractor with a 14-day interval and harvested 15 days after the last application (Belcher and Norby, 1994b). The grain (67 kg) was dried and cleaned up by aspiration and screening and then, after moisture adjustment, milled.

Table 89. Parathion and paraoxon residues in wheat grown in the USA and harvested 15 days after 2 applications of parathion at 4.2 kg ai/ha and its milled commodities (Belcher and Norby, 1994b).

| Commodity | Definition | Milling yield | Residues, mg/kg | |
|---|---|---|---|---|
| | | | parathion | paraoxon |
| Grain | | | 1.21 | 0.035 |
| Bran | broken coats of individual cleaned grain | 11.8% | 5.6 | 0.13 |
| Shorts | low-grade mill product, principally germ and fine bran particles - an animal feed | 49.9% | 0.97 | 0.021 |
| Flour | | 27.6% | 0.44 | <0.02 |
| Middlings | larger particles from the endosperm of the wheat grain during milling | 5.4% | 1.2 | 0.021 |
| Chaff and grain dust | light impurities obtained by aspiration of whole wheat | | 5.4 | 1.70 |

| Commodity | Definition | Milling yield | Residues, mg/kg | |
|---|---|---|---|---|
| | | | parathion | paraoxon |
| TOTAL | | 94.7% | | |

Bookbinder (1998c) measured the residues in aspirated grain fractions (AGF or grain dust) resulting from the movement and aspiration of field-treated spring wheat. In Wisconsin, USA, in 1997 wheat was treated 6 times by aerial application at approximately 7-day intervals with parathion at 0.84 kg ai/ha and harvested 15 days after the last treatment. The harvested grain (225 kg) was oven-dried at 63°C and transported on a simulated commercial grain bucket elevator and drag conveyor system while an aspiration system collected and filtered the air from various points. The AGF were collected from the air in sieves. Grain (213 kg) produced 161 g of AGF. Residue levels in the grain were 1.1 mg/kg parathion and 0.019 mg/kg paraoxon, which produced residue levels in the AGF of 4.4 mg/kg parathion and 0.15 mg/kg paraoxon.

Sunflower seed. Sunflowers in Wisconsin, USA, in 1997 were treated by aerial application three times at 5- or 6-day intervals with parathion EC at 5.6 kg ai/ha (5 times the label rate) and the seed (41 kg) was harvested 30 days after the last application (Bookbinder 1998d). The seed was dried to less than 10% moisture, cleaned up by aspiration and screening, and hulled in a Bauer disc mill. The kernels, after moisture adjustment and heating, were pressed and extracted with hexane to produce crude oil and seed meal. An aliquot of crude oil was mixed with sodium hydroxide and heated to produce refined oil and soapstock.

Table 90. Parathion and paraoxon residues in sunflower seed and processed commodities from sunflowers grown in the USA and harvested 30 days after 3 applications of parathion at 5.6 kg ai/ha (Bookbinder 1998d).

| Commodity | parathion, mg/kg | paraoxon, mg/kg |
|---|---|---|
| Sunflower seed | 0.76 | 0.021 |
| Sunflower seed meal | 0.056 | <0.01 |
| Sunflower seed oil, refined | 0.33 | <0.01 |

Cotton seed. In 2 US trials in Texas and California in 1988 crops were sprayed by a self-propelled research sprayer (TX) and a $CO_2$ backpack sprayer (CA) with 6 applications of parathion at 7.0 kg ai/ha with an EC formulation (5 times the label rate) at 7-day intervals and harvested 7 days after the last application (LeRoy, 1990c). Harvested seed cotton (18 kg) was sent for processing to produce hulls, meal, crude oil, refined oil and soapstock (Table 91).

Table 91. Residues of parathion and paraoxon in seed cotton and commodities from the processing of cotton treated 6 times in the field at 7.0 kg ai/ha and harvested 7 days after the last treatment (LeRoy, 1990c).

| Trial, variety | Applic. rate kg ai/ha | Commodity | Residues, mg/kg | | Reference |
|---|---|---|---|---|---|
| | | | parathion | paraoxon | |
| Texas | 7.0 | Seed cotton | 13.8 | 7.2 | EP-CS-2034 |
| DPL-41 | | Hulls | 1.9 | 0.18 | |
| | | Cotton seed meal | 0.47 | <0.05 | |
| | | Cotton seed oil crude | 0.24 | <0.05 | |
| | | Cotton seed oil refined | 0.29 | <0.05 | |
| | | Soapstock | <0.05 | <0.05 | |
| California | | Seed cotton | 9.0 | 8.2 | EP-CS-2035 |
| SJ-1 | | Hulls | 1.4 | 0.15 | |

| Trial, variety | Applic. rate kg ai/ha | Commodity | Residues, mg/kg | | Reference |
|---|---|---|---|---|---|
| | | | parathion | paraoxon | |
| | | Cotton seed meal | <0.05 | <0.05 | |
| | | Cotton seed oil crude | 0.73 | <0.05 | |
| | | Cotton seed oil refined | <0.05<br>c 0.81 [1] | <0.05<br>c <0.05 | |
| | | Soapstock | <0.05 | <0.05 | |

[1] c: sample from control plot

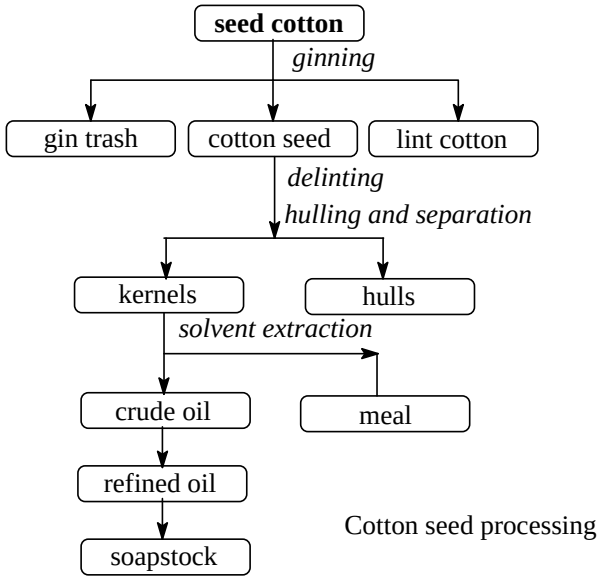



Figure 11. Processing of seed cotton (LeRoy, 1990c).

Canola. A canola crop (variety Series) in Washington State, USA, in 1992, was sprayed twice with a tractor-mounted spray boom at a 7-day interval at post-bloom stage with parathion EC at 5.6 kg ai/ha (5 times the label rate) and the seed (90 kg) was harvested 28 days after the last application (Belcher, 1993). Residues of parathion and paraoxon were determined in the seed, meal, and crude and refined oil (Table 92).

Table 92. Residues of parathion and paraoxon in canola seed harvested 28 days after 2 applications of parathion at 5.6 kg ai/ha and its processed commodities (Belcher, 1993).

| Canola | parathion, mg/kg | paraoxon, mg/kg |
|---|---|---|
| Seed | 1.1 | <0.05 |
| Meal | 0.20 | <0.05 |
| Crude oil | 1.72 | <0.05 |
| Refined oil | 1.66 | <0.05 |

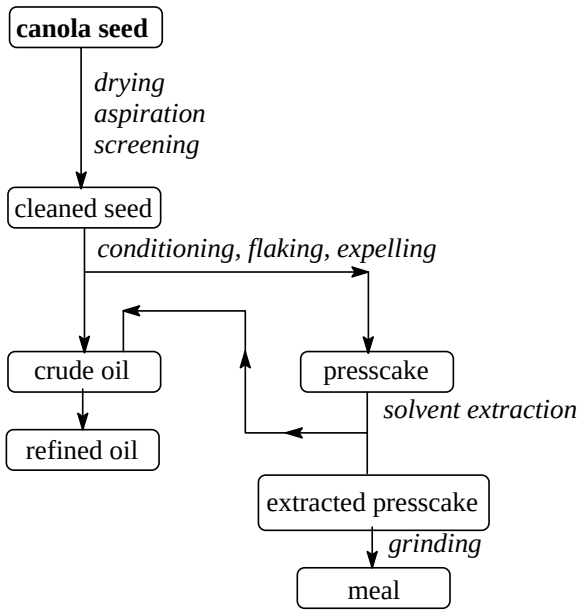



Figure 12. Processing of canola seed (Belcher, 1993).

**Residues in the edible portion of food commodities**

Processing factors from the results of the processing trials for parathion only and for parathion + 1.058 × paraoxon, i.e. sum of parathion and paraoxon expressed as parathion. The factors are shown in Table 93.

In the parathion-only calculation where the residue in the processed commodity was below the LOQ, the processing factor was calculated from the LOQ and is reported as less than the calculated value.

In the total-residue calculation where the residue of paraoxon was below the LOQ it was assumed to be zero except when both parathion and paraoxon residues were below the LOQ, in which case the processing factor was calculated from the LOQ and reported with a 'less than' symbol. For example:

| parathion | paraoxon | total residue | processing factor |
|---|---|---|---|
| 0.84 | 0.13 | 0.98 | [0.95 ÷ x] |
| 0.84 | <0.05 | 0.84 | [0.84 ÷ x] |
| <0.05 | <0.05 | <0.05 | [0.05 ÷ x] |

Where x is the total residue in the RAC

Because parathion is usually the dominant residue component the processing factors for parathion only and for the total residue are usually much the same.

Table 93. Calculated processing factors from the reported processing trials. Factors are calculated for parathion residues only and for the sum of parathion and paraoxon expressed as parathion.

| Commodity | Residues, mg/kg | | | | Processing factors | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | parathion | | paraoxon | | parathion | | | parathion + 1.058 × paraoxon | | |
| | Trial A | Trial B | Trial A | Trial B | A | B | Mean | A | B | Mean |
| Lemons, unwashed fruit | 12 | 6.6 | 0.49 | 0.09 | | | | | | |
| juice | 0.29 | 0.14 | <0.05 | <0.05 | 0.024 | 0.021 | 0.023 | 0.023 | 0.021 | 0.022 |
| wet peel | 5.7 | 7.9 | 0.08 | 0.07 | 0.48 | 1.20 | 0.84 | 0.46 | 1.19 | 0.83 |
| dried peel | 14 | 17 | 0.18 | 0.15 | 1.2 | 2.6 | 1.9 | 1.1 | 2.6 | 1.8 |
| molasses | 0.40 | 1.6 | <0.05 | <0.05 | 0.033 | 0.242 | 0.14 | 0.03 | 0.24 | 0.14 |
| cold pressed oil | | 495 | | 4.1 | | 75 | 75 | | 75 | 75 |
| | | | | | | | | | | |
| Grapefruit, unwashed fruit | 5.4 | | 0.43 | | | | | | | |
| juice | <0.05 | | <0.05 | | <0.009 | | | <0.009 | | |
| wet pulp | 8.7 | | 0.71 | | 1.6 | | 1.6 | 1.6 | | 1.6 |
| dried pulp | 32 | | 1.7 | | 5.9 | | 5.9 | 5.8 | | 5.8 |
| molasses | 1.2 | | 1.3 | | 0.22 | | 0.22 | 0.44 | | 0.44 |
| cold pressed oil | 1650 | | 49 | | 306 | | 306 | 290 | | 290 |
| | | | | | | | | | | |
| Oranges, unwashed fruit | 7.0 | | 0.40 | | | | | | | |
| oranges, washed | 6.3 | | 0.38 | | 0.90 | | 0.90 | 0.90 | | 0.90 |
| juice | 0.36 | | <0.05 | | 0.051 | | 0.051 | 0.048 | | 0.048 |
| wet pulp | 4.9 | | 0.40 | | 0.70 | | 0.70 | 0.72 | | 0.72 |
| dried pulp | 34 | | 1.4 | | 4.9 | | 4.9 | 4.8 | | 4.8 |
| molasses | 4.6 | | 0.08 | | 0.66 | | 0.66 | 0.63 | | 0.63 |
| cold pressed oil | 1674 | | 33 | | 239 | | 239 | 230 | | 230 |
| | | | | | | | | | | |
| Apple, whole | 6.1 | 2.7 | 0.20 | 0.13 | | | | | | |
| juice, unclarified | 0.4 | <0.05 | 0.05 | <0.05 | 0.066 | <0.019 | 0.066 | 0.072 | <0.018 | 0.072 |
| wet pomace | 19 | 0.40 | 0.45 | 0.06 | 3.1 | 0.15 | 1.6 | 3.1 | 0.16 | 1.6 |
| dry pomace | 12.5 | 1.1 | 0.32 | 0.25 | 2.0 | 0.41 | 1.2 | 2.0 | 0.48 | 1.3 |
| | | | | | | | | | | |
| Oats, grain | 0.96 | 3.2 | 0.23 | 0.5 | | | | | | |
| bran | 0.39 | 1.3 | <0.05 | 0.19 | 0.41 | 0.41 | 0.41 | 0.32 | 0.40 | 0.36 |
| flour | 0.40 | 2.2 | 0.06 | 0.12 | 0.42 | 0.69 | 0.55 | 0.39 | 0.62 | 0.50 |
| rolled oats | 0.38 | 0.93 | <0.05 | 0.12 | 0.40 | 0.29 | 0.34 | 0.32 | 0.28 | 0.30 |
| hulls | 2.5 | | 0.68 | | 2.6 | | 2.6 | 2.7 | | 2.7 |
| | | | | | | | | | | |
| Maize, grain | 0.137 | 0.181 | <0.05 | <0.05 | | | | | | |
| meal | 0.082 | 0.16 | <0.05 | <0.05 | 0.60 | 0.88 | 0.74 | | | |
| grits | <0.05 | 0.18 | <0.05 | <0.05 | <0.36 | 0.99 | 0.99 | | | |
| flour | 0.065 | 0.16 | <0.05 | <0.05 | 0.47 | 0.88 | 0.68 | | | |
| starch | <0.05 | <0.05 | <0.05 | <0.05 | <0.36 | <0.28 | | | | |
| crude oil, dry milled | 0.065 | 0.12 | <0.05 | <0.05 | 0.47 | 0.66 | 0.57 | | | |
| crude oil, wet milled | 0.18 | 0.61 | <0.05 | <0.05 | 1.3 | 3.4 | 2.3 | | | |
| refined oil, dry milled | 0.11 | 0.37 | <0.05 | <0.05 | 0.80 | 2.0 | 1.4 | | | |
| refined oil, wet milled | 0.18 | 0.63 | <0.05 | <0.05 | 1.3 | 3.5 | 2.4 | | | |
| | | | | | | | | | | |
| Maize, grain | 0.072 | | <0.01 | | | | | | | |
| grain dust | 0.43 | | 0.027 | | 6.0 | | 6.0 | 6.4 | | 6.4 |
| | | | | | | | | | | |
| Rice, grain | 1.6 | 0.72 | 0.15 | 0.23 | | | | | | |
| brown rice | 0.15 | 0.23 | <0.05 | <0.05 | 0.094 | 0.32 | 0.21 | 0.085 | 0.24 | 0.16 |
| bran | 0.59 | 0.68 | <0.05 | 0.19 | 0.37 | 0.94 | 0.66 | 0.34 | 0.91 | 0.63 |
| hulls | 6.3 | 2.4 | 0.66 | 0.5 | 3.9 | 3.3 | 3.6 | 4.0 | 3.0 | 3.5 |
| polished rice | <0.05 | <0.05 | <0.05 | <0.05 | <0.031 | <0.069 | | <0.029 | <0.053 | |
| | | | | | | | | | | |
| Sorghum, grain | 20.2 | 8.5 | 0.56 | 0.7 | | | | | | |
| decortication grain | 7.2 | 5.2 | 0.35 | 0.40 | 0.36 | 0.61 | 0.48 | 0.36 | 0.61 | 0.49 |

| Commodity | Residues, mg/kg | | | | Processing factors | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | parathion | | paraoxon | | parathion | | | parathion + 1.058 × paraoxon | | |
| | Trial A | Trial B | Trial A | Trial B | A | B | Mean | A | B | Mean |
| coarse bran | 31 | 32 | 1.3 | 1.8 | 1.5 | 3.8 | 2.6 | 1.6 | 3.7 | 2.6 |
| fine bran | 23 | 8.2 | 0.86 | 1.1 | 1.14 | 0.96 | 1.05 | 1.15 | 1.01 | 1.08 |
| grits | 6.7 | 4.8 | 0.35 | 0.48 | 0.33 | 0.56 | 0.45 | 0.34 | 0.57 | 0.46 |
| flour | 4.6 | 4.8 | 0.23 | 0.43 | 0.23 | 0.56 | 0.40 | 0.23 | 0.57 | 0.40 |
| starch | 0.19 | 0.10 | 0.067 | 0.063 | 0.009 | 0.012 | 0.011 | 0.013 | 0.018 | 0.015 |
| | | | | | | | | | | |
| Sorghum, grain | 2.2 | | 0.057 | | | | | | | |
| grain dust | 4.2 | | 0.15 | | 1.9 | | 1.9 | 1.9 | | 1.9 |
| | | | | | | | | | | |
| Wheat, grain | 1.21 | | 0.035 | | | | | | | |
| bran | 5.6 | | 0.13 | | 4.6 | | 4.6 | 4.6 | | 4.6 |
| shorts | 0.97 | | 0.021 | | 0.80 | | 0.80 | 0.80 | | 0.80 |
| flour | 0.44 | | <0.02 | | 0.36 | | 0.36 | 0.35 | | 0.35 |
| middlings | 1.2 | | 0.021 | | 0.99 | | 0.99 | 0.98 | | 0.98 |
| chaff and grain dust | 5.4 | | 1.7 | | 4.5 | | 4.5 | 5.8 | | 5.8 |
| | | | | | | | | | | |
| Wheat, grain | 1.1 | | 0.019 | | | | | | | |
| grain dust | 4.4 | | 0.15 | | 4.0 | | 4.0 | 4.1 | | 4.1 |
| | | | | | | | | | | |
| Sunflower seed | 0.76 | | 0.021 | | | | | | | |
| Sunflower seed meal | 0.056 | | <0.01 | | 0.074 | | 0.074 | 0.072 | | 0.072 |
| Sunflower seed oil, refined | 0.33 | | <0.01 | | 0.43 | | 0.43 | 0.42 | | 0.42 |
| | | | | | | | | | | |
| Seed cotton | 13.8 | 9.0 | 7.2 | 8.2 | | | | | | |
| hulls | 1.9 | 1.4 | 0.18 | 0.15 | 0.14 | 0.16 | 0.15 | 0.098 | 0.088 | 0.093 |
| cotton seed meal | 0.47 | <0.05 | <0.05 | <0.05 | 0.0341 | <0.006 | 0.034 | 0.022 | <0.003 | 0.022 |
| cotton seed oil, crude | 0.24 | 0.73 | <0.05 | <0.05 | 0.017 | 0.081 | 0.049 | 0.011 | 0.041 | 0.026 |
| cotton seed oil, refined | 0.29 | <0.05 | <0.05 | <0.05 | 0.021 | <0.006 | 0.021 | 0.014 | <0.003 | 0.014 |
| soapstock | <0.05 | <0.05 | <0.05 | <0.05 | <0.004 | <0.006 | | <0.002 | <0.003 | |
| | | | | | | | | | | |
| Canola seed | 1.1 | | <0.05 | | | | | | | |
| canola meal | 0.20 | | <0.05 | | 0.18 | | 0.18 | | | |
| canola crude oil | 1.7 | | <0.05 | | 1.55 | | 1.55 | | | |
| canola refined oil | 1.66 | | <0.05 | | 1.51 | | 1.51 | | | |
| | | | | | | | | | | |
| Grapes (3 trials) | 2.1 2.7 9.2 | | <0.05 <0.05 0.17 | | | | | | | |
| juice | 0.275 0.36 0.05 | | <0.05 <0.05 <0.05 | | 0.13 0.13 0.005 | | 0.090 | 0.13 0.13 0.005 | | 0.090 |
| wet pomace | 9.0 15 19 | | 0.09 0.18 0.36 | | 4.3 5.6 2.1 | | 4.0 | 4.3 5.6 2.1 | | 4.0 |
| dried pomace | 20.5 15 36 | | 0.175 0.20 0.36 | | 9.8 5.6 3.9 | | 6.4 | 9.8 5.6 3.9 | | 6.5 |
| raisins | 0.58 0.90 | | 0.060 0.13 | | 0.28 0.33 | | 0.30 | 0.31 0.38 | | 0.35 |
| raisin waste | 1.4 3.8 | | 0.12 0.75 | | 0.67 1.4 | | 1.0 | 0.73 1.7 | | 1.2 |

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Information was provided by the governments of Australia and The Netherlands.

Information on monitoring for parathion residues was provided by the government of The Netherlands (Table 94). Very few residues exceeded the MRLs (5 samples of lettuce, 1 endive, 1 parsley, 1 other herbs, 4 celery, 2 other stem vegetables, 1 maize, 1 celeriac and 3 spinach).

Table 94. Netherlands monitoring data for parathion residues for 1994-1998. The LOQ was 0.02 mg/kg.

| Commodity | No. of samples | | Commodity | No. of samples | |
|---|---|---|---|---|---|
| | analysed | containing residues | | analysed | residues detected |
| Apples | 1495 | 3 | Maize | 37 | 1 |
| Beans, fresh (in pods) | 750 | 4 | Melons | 390 | 3 |
| Billberries, cowberries, cranberries | 39 | 1 | Nectarines | 221 | 6 |
| Brussels sprouts | 176 | 2 | Onions | 194 | 2 |
| Carrots | 424 | 6 | Oranges | 1237 | 27 |
| Celeriac | 183 | 4 | Other arable products | 699 | 3 |
| Celery | 525 | 24 | Other fruits and fruit products | 385 | 2 |
| Chinese cabbage | 413 | 9 | Other herbs | 148 | 3 |
| Chives | 21 | 1 | Other leafy vegetables | 292 | 3 |
| Currants | 576 | 5 | Other root and tuber vegetables | 87 | 6 |
| Endive | 1737 | 19 | Other stem vegetables | 341 | 25 |
| Fennel | 95 | 1 | Parsley | 426 | 8 |
| Figs | 48 | 1 | Peaches | 252 | 1 |
| Grapefruit | 301 | 4 | Peppers | 2132 | 6 |
| Grapes | 962 | 66 | Plums | 437 | |
| Iceberg lettuce | 471 | 4 | Radishes | 1010 | 8 |
| Kiwifruit | 223 | 2 | Raspberries | 315 | 2 |
| Lamb's lettuce | 268 | 2 | Spinach | 610 | 8 |
| Leeks | 441 | 14 | Strawberries | 2378 | 3 |
| Lemons | 243 | 5 | Tangerines | 672 | 19 |
| Lettuce | 4560 | 78 | | | |

In the 1994 Australian Market Basket Survey 77 foods were analysed for pesticide residues (Marro, 1996). The estimated daily intakes of parathion residues in food for mean energy diets expressed as a percentage of the ADI (0.005 mg/kg bw) were for adult males 0.03%, adult females 0.03%, boys aged 12 0.04%, girls aged 12 0.05%, toddlers 0.14% and infants 0.11%.

Parathion was included in the targeted enforcement monitoring programmes in Australian States for 1995-1999. In Queensland no parathion residues (LOQ 0.02-0.1 mg/kg) were detected in 413 samples of fruits, vegetables and cereals. In Victoria parathion was detected (LOQ 0.01 mg/kg) in 1 of 20 apple samples and 6 of 20 pear samples with the highest residue at 0.1 mg/kg; no parathion residues were detected (LOQ 0.01 mg/kg) in 111 cereal or vegetable samples.

## NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported to the Meeting.

| Country | MRL, mg/kg | Commodity |
|---|---|---|
| Australia[1] | T 1 | Apricot, cotton seed, peach |
| | T 0.7 | Vegetables except carrot |
| | T 0.5 | Carrot, cereal grains, cotton seed oil crude, fruits except apricot and peach, |
| | T 0.05* | Edible offal mammalian, meat mammalian, milks |
| Netherlands[2] | 1 | Citrus fruit, apricots, peaches |
| | 0.5 | Other fruit, vegetables |
| | 0.1 | Tea |
| | 0.05 | Other vegetable products |
| | 0.02* | Other food commodities |
| USA[3] | 5 | Alfalfa hay |
| | 3 | Almond hulls, sorghum fodder, sorghum forage |

| Country | MRL, mg/kg | Commodity |
|---|---|---|
| | 1.25 | Alfalfa (fresh) |
| | 1 | Apples, apricots, artichokes, avocados, barley, beans, beet greens, beets, blackberries, blueberries, boysenberries, broccoli, Brussels sprouts, cabbage, carrots, cauliflower, celery, cherries, clover, collards, corn, corn forage, cranberries, cucumbers, currants, dates, dewberries, eggplants, endive, figs, garlic, gooseberries, grapes, grass forage, guavas, hops, kale, kohlrabi, lettuce, loganberries, mangoes, melons, mustard greens, nectarines, oats, okra, olives, onions, parsnips, parsnip greens, peaches, peanuts, pears, peas, pea forage, peppers, pineapples, plums, pumpkins, quinces, radishes, radish tops, raspberries, rice, rutabagas, rutabaga tops, soybean hay, spinach, squash, strawberries, summer squash, Swiss chard, tomatoes, turnips, turnip greens, vetch, wheat, youngberries |
| | 0.75 | Cotton seed |
| | 0.2 | Mustard seed, rape seed, sunflower seed |
| | 0.1 | Almonds, sugar beets, sugar beet tops, filberts, pecans, potatoes, safflower seed, sorghum, soybeans, sweet potatoes, walnuts |

* indicates MRL set at or about limit of quantitation.
[1] Residue definition: parathion. MRLs temporary; parathion is no longer registered for use in Australia, the last products were withdrawn in 1999.
[2] Residue definition: parent compound, expressed as parathion.
[3] US tolerances are established for residues of parathion or its methyl homologue (parathion-methyl).

## APPRAISAL

Parathion was first evaluated by the Joint Meeting in 1965 and has been reviewed several times since. At its thirtieth session in 1998, the CCPR (ALINORM 99/24, Appendix VII) listed parathion for periodic review for residues by the 2000 JMPR. The Meeting received information on physical and chemical properties, metabolism and environmental fate, analytical methods, stability in frozen storage, registered uses, the results of supervised trials on fruits, vegetables, and field crops, and studies on processing.

### *Metabolism*

*Animals*

Parathion is metabolized to paraoxon and diethyl phosphorothioate. In cattle, ruminal microorganisms are believed to be responsible for the production of aminoparathion and aminoparaoxon (Annex 6, reference 74).

When lactating goats, initially weighing 57 and 42 kg, were dosed with [$^{14}$C-phenyl]parathion at 188 mg/day (equivalent to 97 ppm in the diet) for 5 days, parathion was detected at 0.019 mg/kg in milk, 0.56 mg/kg in liver, 0.48 mg/kg in kidney, 0.15 mg/kg in renal fat, and 0.019 mg/kg in muscle. The major component of the residue was *para*-acetamido-paraoxon. Paraoxon itself was not detected. Approximately 40% of the administered radiolabel was recovered, leaving a large part unaccounted for.

When laying hens weighing 1.3-2.1 kg were dosed orally with [$^{14}$C-phenyl]parathion six times at daily intervals at a dose of 1.5 mg/day, equivalent to 16.5 ppm in the diet, parathion was detected at 0.001 mg/kg in eggs, 0.001 mg/kg in liver, 0.004 mg/kg in kidney, and 0.002 mg/kg in skin with fat. The total amounts of radiolabel in muscle were very low (<0.01% of the dose). Paraoxon was detected at 0.001 mg/kg in liver and kidney. The major identified components of the residue were *para*-nitrophenyl phosphate and *para*-acetamidophenol. The proportion of radiolabel accounted for in this study was 83%.

The studies showed that parathion is degraded by de-ethylation, oxidation, hydrolysis of the phosphate ester, reduction of the nitro group to an amine, and conjugation.

*Plants*

The Meeting received information on the fate of parathion in wheat, cotton, and potatoes.

The main component (51-65% of the radiolabel) of the residue in wheat straw, chaff, and grain sampled from wheat plants 7 days after a second treatment with [$^{14}$C-phenyl]parathion at 1.3 kg ai /ha was parathion itself. The concentrations found were parathion, 66 mg/kg, and paraoxon, 4.2 mg/kg in straw; parathion, 197 mg/kg, paraoxon, 12 mg/kg in chaff; and parathion, 6.7 mg/kg, paraoxon, 0.13 mg/kg in grain. Other metabolites included *para*-nitrophenol, *S*-phenyl parathion, and *O*-desethyl parathion.

The concentration of parathion (0.019 mg/kg) in cotton seed was too low for identification 14 days after the plants were treated twice with [$^{14}$C-phenyl]parathion at 1.7 kg ai/ha. Parathion was the major residue component in calyx and leaf, but paraoxon, *para*-nitrophenol and other metabolites were also identified.

When potato plants were given two foliar treatments with [$^{14}$C-phenyl]parathion at 3.0 kg ai/ha and harvested 15 days after the second treatment, most of the radiolabel (20-31 mg/kg) remained in the stems and foliage, although small amounts (0.093-0.14 mg/kg) reached the tubers. Approximately 1% of the radiolabel in the tubers was identified by thin-layer chromatography as parathion and 10% as *para*-nitrophenol.

The plant metabolites identified indicate that hydrolysis of parathion to nitrophenol is the major pathway, but oxidation to paraoxon, some rearrangements of the thiophosphate ester, and *O*-de-ethylation also occur. Nitrophenol readily forms conjugates.

***Environmental fate***

*Degradation in soil*

Parathion was the major component of the residue in a 1-year study of degradation in aerobic soil. The half-life for disappearance of the parent parathion was 58 days. After 1 month and 1 year, 9.8% and 44%, respectively, of the dose had been mineralized. Paraoxon, nitrophenol, and *O*,*O*-bis(4-nitrophenyl) ethyl phosphate were identified as products.

In a study of the degradation of [$^{14}$C-phenyl]parathion in anaerobic soil under flooded conditions, the initial half-life for loss of parent parathion was 13 h, but the rate declined after 24 h, suggesting that some of the parathion became bound or was less readily available for microbial attack. Much of the dose (89% after 3 months) was converted to an unextractable residue. A considerable portion of the unextractable residues was incorporated into the biomass of the soil.

*Fate in water and sediment systems*

Parathion disappeared quickly, with an initial half-life of 2.4 days, during aerobic degradation in a water-sediment system. After 1 month, parent parathion accounted for 2.5% of the dose, while 60% was unextractable. Very little (3%) had been mineralized.

***Methods of analysis***

The Meeting received information on analytical methods for residues of parathion and paraoxon in supervised trials and for enforcement.

The analytical method used in supervised trials in the USA, most of which were carried out in 1988-90, were based on gas-liquid chromatography with flame photometry after solvent extraction and simple clean-up with solvent partition. A 30-min acid reflux was introduced at the extraction step because the studies of metabolism in wheat straw and grain had shown that the acid releases additional parathion and paraoxon residues. However, reflux acid extraction and extraction at room temperature with water and methanol of peppers and celery gave comparable results. The LOQ of the method was generally 0.05 mg/kg, and the analytical recovery was 80-90%. The method was tested and used on 39 substrates, including vegetables, nuts, forage, hay, olives, processed commodities, and wheat, and was tested for interference from 230 pesticides.

The method was modified by use of capillary gas-liquid chromatography to achieve an LOQ of 0.02 mg/kg for parathion and paraoxon in wheat, forage, and processed commodities. The recovery was generally 80-110%, that of paraoxon at 0.02 mg/kg tending to be higher. The method was tested on sorghum, rape seed, and their processed commodities, with an LOQ of 0.02 mg/kg for some commodities and 0.05 mg/kg for others. The recovery after fortification at 0.02 mg/kg was unacceptably high for some commodities.

An LOQ of 0.01 mg/kg was achieved for parathion and paraoxon in apples and grapes in a method based on gas-liquid chromatography with flame photometry after acetone-water extraction, solvent partition, and $C_{18}$ column clean-up.

A method for analysis of residues in animal commodities is based on capillary gas-liquid chromatography with flame photometry after acetone extraction of the sample and clean-up by solvent partition and on carbon Celite and $C_{18}$ columns. The LOQ for parathion and paraoxon in liver and fat was 0.05 mg/kg. The concentration of parathion residues was similar after analysis of hen fat by this method (0.12 mg/kg) and by the $^{14}C$ method (0.14 mg/kg). LOQs of 0.001 mg/kg and 0.01 mg/kg were achieved for milk and kidney, respectively, in a similar method.

***Stability of residues in stored analytical samples***

Parathion residues were stable in frozen storage for 2 years in almond kernels, apples, clover, cotton seed, green peppers, kidney beans, oranges, plums, snap beans, spinach, strawberries, and sunflower seeds; for 14 months in rape seed, crude rape seed oil, and rape seed meal; for 19 months in sorghum flour; and for 4 months in maize grain, flour, starch, oil, meal, and corn grits. Because of the reproducibility of the analytical method, a decrease of less than 30% would not be distinguishable from variability. The concentrations of parathion residues in almond kernels and oranges appeared to have decreased by an estimated 30% within 2 years. Paraoxon residues were also stable in frozen storage, except in snap beans (in which a substantial decline was seen after 12 months), spinach (borderline 30% decrease), and rape seed.

***Definition of the residue***

Parathion and paraoxon are the predominant components of the residue. Parathion represents the major portion of the residue, whereas paraoxon is a minor component when the residues are fresh and occur at higher concentrations. At very low concentrations in some circumstances, paraoxon may constitute a significant part of the residue. In residue trials that complied with GAP, 227 samples of food and feed commodities contained both parathion and paraoxon at concentrations that exceeded the LOQ. There was generally good agreement between the concentration of combined residues and that of parathion.

The Meeting recommended that the residue definition for compliance with MRLs continue to be parathion, and the definition for estimating dietary intake should be the sum of parathion and paraoxon expressed as parathion (parathion + 1.058 $\infty$ paraoxon).

The log $P_{ow}$ of 3.2 and the results of studies of animal metabolism suggest that parathion is of borderline solubility in fat. In goats, the concentration of parathion residues in renal fat (0.15 mg/kg) was substantially higher than that in muscle tissue (0.019 mg/kg), although those in liver (0.56 mg/kg) and kidney (0.48 mg/kg) were both higher than that in fat. Paraoxon was not detected in the tissues or milk of the goat, but it was present at very low concentrations in liver and kidney of laying hens. The Meeting agreed that the residue definition for animal commodities should be reconsidered when the MRLs for animal commodities are recommended.

***Results of supervised trials***

Extensive data were provided from supervised trials on many crops: grapefruit, lemon, orange, apple, pear, apricot, cherry, plum and prune, blackberry, grape, strawberry, olive, garlic, onion, broccoli, cabbage, pepper, sweet corn, tomato, field pea, kale, lettuce, spinach, snap bean, dry bean, soya bean, carrot, potato, radish, sugar beet, turnip, celery, almond, pecan, walnut, barley, maize, rice, sorghum, wheat, rape seed, cotton seed, and sunflower seed. Supervised trials based on unvalidated analytical data (from Craven Laboratories) could not be considered further for the following crops: alfalfa, broccoli, cabbage, carrot, garlic, kale, olive, processed olive, onion, pecan, potato, radish, sugar beet, tomato, turnip, walnut, and wheat.

No relevant GAP was available to evaluate data for grapefruit, lemon, orange, pear, apricot, cherry, plum and prune, blackberry, grape, strawberry, pepper, field pea, lettuce, spinach, snap bean, dry bean, celery, almond, rice, alfalfa, and clover.

The Meeting agreed to withdraw the current recommendations for apricot (1 mg/kg), leek (0.05 mg/kg), lemon (0.5 mg/kg), mandarin (0.5 mg/kg), virgin olive oil (2 mg/kg), olive (0.5 mg/kg), sweet and sour orange (0.5 mg/kg), peach (1 mg/kg), and potato (0.05* mg/kg), as the MRLs are not supported by current GAP or in supervised trials evaluated against current GAP.

The residue definition for dietary intake requires the addition of parathion and paraoxon residues expressed as parathion. In this calculation, concentrations of residues of paraoxon that were <LOQ were assumed to be 0, except when the concentrations of both parathion and paraoxon residues were <LOQ. In the latter case, the total was taken to be <LOQ, which is a reasonable assumption because the concentration of paraoxon is usually much lower than that of parathion. For example:

| Parathion | Paraoxon | Total residue (parathion + 1.058 x paraoxon) |
|---|---|---|
| 3.20 | 0.34 | 3.56 |
| 0.42 | <0.05 | 0.42 |
| <0.05 | <0.05 | <0.05 |

Trials on *apple* in the USA were not evaluated because there was no matching GAP. In Italy, parathion is registered for use on pome fruits at a spray concentration of 0.02-0.04 kg ai/hl with a PHI of 20 days. Twelve trials conducted in France in 1994 at 0.036 kg ai/hl with a 21-day PHI were evaluated against the Italian GAP. The concentrations of parathion residues in rank order (median in italics) were <0.01, 0.01 (2 trials), 0.02 (2 trials), ***0.02***, ***0.03***, 0.05, 0.08 (2 trials), 0.14, and 0.16 mg/kg. As the values for paraoxon were all <LOQ (0.01 mg/kg), the concentration of total residue is the same as that for parathion.

The Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR value of 0.025 mg/kg, and a HR value of 0.16 mg/kg for parathion in apples. The estimated maximum residue level replaces the current recommendation of 0.05* mg/kg.

Parathion is registered in the USA for use on *sweet corn* at a rate of 0.28-0.84 kg ai/ha with a PHI of 12 days. Ten trials in four states in 1987-89 involving six applications of 1.1 kg ai/ha and

harvesting 12 days after the final treatment showed no residues of parathion or paraoxon in sweet corn ears that exceeded the LOQ (0.05 mg/kg). Although no residues were detected, there was no evidence that residues were not present, and the STMR value should be equivalent to the LOQ.

The Meeting estimated a maximum residue level of 0.05* mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.05 mg/kg for parathion in sweet corn.

Parathion is registered in the USA for use on *soya bean* at 0.28-0.84 kg ai/ha with a PHI of 20 days for harvesting, cutting, or use as forage. Eight trials in three states in 1988 with two applications of 0.90 kg ai/ha (PHI, 20 days) showed no residue (<0.05 mg/kg) of parathion or paraoxon in harvested soya beans. Although no residues were detected, there was no evidence that residues were not present, and the STMR value should be equivalent to the LOQ.

The Meeting estimated a maximum residue level of 0.05* mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.05 mg/kg for parathion in dry soya beans. The estimated maximum residue level confirms the current recommendation for dry soya beans of 0.05* mg/kg.

Parathion is registered in the USA for use on *barley* at 0.28-0.84 kg ai/ha with a PHI of 15 days for harvesting, cutting, or use as forage. Twelve trials in eight states in 1997 and 1998 with six aerial applications of 0.81-0.84 kg ai/ha and harvesting 14 or 15 days after the final treatment resulted in the following concentrations of parathion residues in barley grain: 0.15, 0.25, 0.54, 0.78, 1.3, 1.6, 2.0, 2.2 (2 trials), 3.3, 4.1, and 4.9 mg/kg, and those of the combined residues of parathion and paraoxon in rank order were 0.16, 0.27, 0.61, 0.81, 1.4, ***1.7***, ***2.2***, 2.3, 2.3, 3.6, 4.4, and 5.1 mg/kg.

The Meeting estimated a maximum residue level of 7 mg/kg, an STMR value of 1.95 mg/kg, and a HR value of 5.1 mg/kg for parathion in barley.

Parathion is registered in the USA for use on *maize* at 0.28-0.84 kg ai/ha with a PHI of 12 days for harvesting, cutting, or use as forage. Twelve trials in six states in 1987-89 with five or six applications of 1.1 kg ai/ha and harvesting 12 days after the final treatment resulted in the following concentrations of parathion residues in maize grain: <0.05 (10 trials), 0.06, and 0.09 mg/kg. The concentrations of paraoxon residues were all <LOQ (0.05 mg/kg).

The Meeting estimated a maximum residue level of 0.1 mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.09 mg/kg for parathion in maize. The estimated maximum residue level confirms the current recommendation for maize of 0.1 mg/kg.

Parathion is registered in the USA for use on *sorghum* at 0.28-1.1 kg ai/ha with a PHI of 12 days for harvesting, cutting, or use as forage. Six trials in 1987, five in 1992, and two in 1994 in six states with two or six applications of 1.1 kg ai/ha and harvesting 12 days after the final treatment resulted in the following concentrations of parathion residues in sorghum grain: 0.29, 0.54, 0.61, 0.69, 0.71, 0.79, 0.85, 1.3, 1.6, 1.7, 2.0, 3.3, and 3.8 mg/kg, and concentrations of combined parathion and paraoxon residues in rank order of: 0.29, 0.55, 0.74, 0.75, 0.76, 1.03, ***1.06***, 1.4, 1.8, 1.9, 2.1, 3.5, and 4.2 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg, an STMR value of 1.06 mg/kg, and a HR value of 4.2 mg/kg for parathion in sorghum. The estimated maximum residue level confirms the current recommendation for sorghum of 5 mg/kg.

Parathion is registered in the USA for use on *wheat* at 0.28-0.84 kg ai/ha with a PHI of 15 days for harvesting, cutting, or use as forage. Seven trials in 1992, five in 1993, and four in 1994 in 11 states, with two aerial applications of 0.69-0.93 kg ai/ha (most trials at 0.84 kg ai/ha) and harvesting 15 days (or longer if the residue concentration was higher than at 15 days) after the final treatment resulted in the following concentrations of parathion residues in wheat grain: 0.02, 0.05 (2 trials), 0.06

(2 trials), 0.07, 0.08, 0.11, 0.12 (2 trials), 0.14, 0.16, 0.21, 0.54, 0.63, and 0.92 mg/kg, and those of combined parathion and paraoxon residues in rank order were 0.02, 0.05 (2 trials), 0.06, 0.07, 0.08 (2 trials), ***0.11***, ***0.14***, 0.15, 0.16 (2 trials), 0.21, 0.54, 0.65, and 0.96 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg, an STMR value of 0.125 mg/kg, and a HR value of 0.96 mg/kg for parathion in wheat.

Parathion is registered in the USA for use on *oilseed rape* at 0.56 kg ai/ha with a PHI of 28 days. Five trials in five states in 1992 and 1994, with two aerial applications of 0.56 kg ai/ha (0.50 kg ai/ha in one trial) and harvesting 28 days after the final treatment resulted in the following concentrations of parathion residues in rape seed: <0.05 (2 trials), 0.09, 0.12, and 0.13 mg/kg. The concentrations of paraoxon residues were <LOQ (0.05 mg/kg) in all trials.

The Meeting decided that five trials were too few to allow estimation of a maximum residue level.

Parathion is registered in the USA for use on *cotton* at 0.29-1.1 kg ai/ha with a PHI of 7 days. Six trials on cotton seed in 1987 and 12 in 1997 in six states, with six applications of 1.1 kg ai/ha (1.4 kg ai/ha in three trials, still considered to comply with GAP) and harvesting 7 days after the final treatment resulted in the following concentrations of parathion residues: 0.13, 0.15 (2 trials), 0.19, 0.20 (2 trials), 0.21, 0.26, 0.30, 0.33, 0.40, 0.48, 0.65, 0.66, 0.97, 1.1, 1.3, and 2.0 mg/kg, and those of the combined parathion and paraoxon residues in rank order were 0.13, 0.15 (2 trials), 0.19, 0.21 (3 trials), 0.26, ***0.31***, ***0.39***, 0.44, 0.48, 0.67, 0.75, 1.1, 1.2, 1.4, and 2.1 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg, an STMR value of 0.35 mg/kg, and a HR value of 2.1 mg/kg for parathion in cotton seed. The estimated maximum residue level replaces the current recommendation (1 mg/kg) for cotton seed.

Parathion is registered in the USA for use on *sunflower* at 0.56-1.1 kg ai/ha with a PHI of 30 days. Seven trials in 1988 and 1999 in two states, with three applications of 1.1 kg ai/ha and harvesting 30 days after the final treatment resulted in no residues of parathion or paraoxon >LOQ (0.05 mg/kg). Residues of both compounds were detected in sunflower seed in a processing study after treatment at five times the label rate, however, indicating that, even though no residues were found at >LOQ in the supervised trials, the concentration is not effectively 0. The STMR value should therefore be equivalent to the LOQ.

The Meeting estimated a maximum residue level of 0.05* mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.05 mg/kg for parathion in sunflower seed. The estimated maximum residue level confirms the current recommendation for sunflower seed of 0.05* mg/kg.

As noted above, parathion is registered in the USA for use on barley. Twelve trials in eight states in 1997 and 1998, with six aerial applications of 0.78-0.84 kg ai/ha and cutting or harvesting 14-16 days after the final treatment resulted in residues in *barley hay and straw*. As the moisture was measured, the residues could be expressed on a dry weight basis. The concentrations of parathion residues in barley hay were 0.10 (2 trials), 0.16, 0.18, 0.19, 0.21, 0.55, 0.70, 0.73, 1.1, 3.6, and 4.7 mg/kg (fresh weight) and 0.14, 0.15, 0.25, 0.26 (2 trials), 0.28, 0.80, 1.0, 1.1, 1.6, 5.9, and 6.5 mg/kg (dry weight). The concentrations of combined parathion and paraoxon residues in barley hay were 0.17, 0.18, 0.29, 0.34 (2 trials), ***0.37***, ***0.86***, 1.2, 1.7, 1.8, 6.2, and 8.1 mg/kg (dry weight). The concentrations of parathion residues in barley straw were 0.6, 0.7, 1.3, 2.0, 2.8, 2.9, 3.5 (2 trials), 7.6, 8.0, 9.6, and 13 mg/kg (fresh weight) and 1.0, 1.3, 2.6, 3.2, 5.1, 6.1, 6.4, 7.1, 12, 14, 16, and 20 mg/kg (dry weight). The concentrations of combined parathion and paraoxon residues in barley straw were 1.4, 1.6, 3.1, 4.7, 6.2, ***7.3***, ***8.2***, 8.8, 14, 16, 20, and 24 mg/kg (dry weight).

The data for barley hay and straw were combined to propose an MRL for barley straw and fodder. The concentrations of residues in straw were usually higher than those in hay in the same trial (both expressed as dry weight). The higher value ( for hay or straw on a dry weight basis) in each trial was taken to represent that for the residue in barley straw and fodder in that trial. The concentrations of parathion residues in barley straw and fodder were thus: 1.0, 1.3, 3.2, 5.1, 5.9, 6.1, 6.4, 7.1, 12, 14, 16, and 20 mg/kg (dry weight), and those of the combined residues of parathion and paraoxon were 1.4, 1.6, 4.7, 6.2 (2 trials), ***7.3***, ***8.2***, 8.8, 14, 16, 20, and 24 mg/kg (dry weight).

The Meeting estimated a maximum residue level of 30 mg/kg and an STMR value of 7.75 mg/kg for parathion in barley straw and fodder (dry weight).

As noted above, parathion is registered in the USA for use on maize. A series of 27 trials in eight states in 1987-89, with six applications of 1.1 kg ai/ha and harvesting or cutting 12 days after the final treatment resulted in residues in *maize fodder, forage, and silage*. The application rate of 1.1 kg ai/ha used in the trials is 33% higher than the recommended rate (1 pint per acre in trials, 0.75 pint per acre according to GAP), but the data were considered adequate to represent residues resulting from GAP. Data were not available on moisture levels or percent dry matter.

The concentrations of the resulting parathion residues in *maize fodder* were <0.05, 0.06, 0.10, 0.12, 0.39, 0.45, 0.74, 0.86, 0.92, 1.4, 1.6, 2.3, 2.6, 2.7, 5.5, 6.3, 8.0, 8.4, 13, and 19 mg/kg (fresh weight), and those of the combined parathion and paraoxon residues were <0.05, 0.12, 0.17, 0.25, 0.51, 0.58, 0.80, 0.86, 0.98, ***1.6***, ***2.0***, 2.4, 2.8, 3.0, 5.9, 6.8, 9.3, 9.1, 14, and 22 mg/kg (fresh weight). Allowing for the standard 83% of dry matter in maize fodder (FAO, 1997, p. 123, corn stover = maize fodder), the Meeting estimated a maximum residue level of 30 mg/kg and an STMR value of 2.13 mg/kg for parathion in maize fodder (dry weight). The highest value of dry weight = 22/0.83 = 26.5.

The concentrations of parathion residues in *maize forage* were <0.05, 0.05, 0.09, 0.10, 0.56, 1.1, 1.3, 1.4, 1.5, and 2.1 mg/kg (fresh weight), and those of the combined parathion and paraoxon residues were <0.05, 0.12, 0.15, 0.16, ***0.73***, ***1.1***, 1.5 (2 trials), 1.6, and 2.3 mg/kg (fresh weight). Allowing for the standard 40% of dry matter in maize forage (FAO, 1997, p. 123), the Meeting estimated a maximum residue level of 10 mg/kg and an STMR value of 2.28 mg/kg for parathion in maize forage (dry weight). The highest value of dry weight = 2.3/0.40 = 5.75 and the STMR dry weight = 0.5 ∞ (0.73 + 1.1)/0.40 = 2.28.

The concentrations of parathion residues in *maize silage* were 0.34, 0.78, 1.1, 1.2, 1.3, 1.8, 2.4, and 2.6 mg/kg (fresh weight). No information on the percent dry matter in the silage was available, but the residues in silage should be covered by the estimated maximum residue level for fodder.

As noted above, parathion is registered in the USA for use on sorghum. Eight trials in six states in 1992 and 1994, with two aerial applications of 1.1 kg ai/ha and harvesting or cutting 12 days after the second application resulted in residues in *sorghum forage, fodder, and hay*. The percent dry matter was available for all samples.

The resulting concentrations of residues of parathion in *sorghum fodder and hay* were 0.18, 0.25, 0.34, 0.52, 0.87, 1.6, 1.2, and 4.3 mg/kg (fresh weight) or 0.38, 0.78, 1.4, 2.4, 3.0, 3.9, 4.3, and 10 mg/kg (dry weight), and those of the combined parathion and paraoxon residues were 0.18, 0.25, 0.34, ***0.52***, ***0.92***, 1.6, 1.3, and 4.3 mg/kg (fresh weight) or 0.38, 0.78, 1.4, ***2.4***, ***3.2***, 3.9, 4.7, and 10 mg/kg (dry weight). The Meeting estimated a maximum residue level of 15 mg/kg and an STMR value of 2.8 mg/kg for parathion in sorghum straw and fodder (dry weight).

The concentrations of residues of parathion in *sorghum forage* were 0.09, 0.34, 0.40, ***0.56***, 0.72, 1.1, and 1.7 mg/kg (fresh weight) or 0.35, 1.1, 2.7, ***3.1***, 3.5, 3.8 and 8.5 mg/kg (dry weight). The concentration of paraoxon residues did not exceed the LOQ (0.05 mg/kg) in any sample. The Meeting

estimated a maximum residue level of 10 mg/kg and an STMR value of 3.1 mg/kg for parathion in sorghum forage (dry weight).

As noted above, parathion is registered in the USA for use on wheat. Trials in 10 states in 1992-94, with two aerial applications of 0.84 kg ai/ha and harvesting or cutting 15 days after the second application resulted in residues in *wheat forage and straw*. The percent dry matter was available for all samples in some trials and for representative samples in others.

The concentrations of residues of parathion in *wheat forage* were <0.05, 0.09 (2 trials), 0.10, 0.12, 0.15, 0.48, 0.52, and 0.79 mg/kg (fresh weight) or <0.05, 0.30, 0.33, 0.40, 0.46, 0.47, 1.9, 2.2, and 3.2 mg/kg (dry weight), and those of the combined parathion and paraoxon residues were <0.05, 0.09, 0.10, 0.11, ***0.12***, 0.21, 0.62, 0.66, and 0.89 mg/kg (fresh weight) or <0.05, 0.30, 0.33, 0.40, ***0.57***, 0.64, 2.4, 2.8, and 3.5 mg/kg (dry weight). Residues in wheat forage are covered by the recommendations for wheat straw and fodder.

The concentrations of residues of parathion in *wheat straw* were 0.50, 0.67, 0.98, 1.0, 1.2, 1.4, 1.5, 1.8 (2 trials), 1.9, 3.1 (2 trials), 3.5, 3.8, 7.3, 7.5, and 9.5 mg/kg (fresh weight) or 0.70, 0.91, 1.2, 1.5 (2 trials), 2.9, 3.2, 3.3, 3.4 (2 trials), 4.2 (3 trials), 5.0, 8.2, 11, and 18 mg/kg (dry weight). The concentrations of the combined parathion and paraoxon residues were 0.65, 0.67, 1.0, 1.3 (2 trials), 1.6, 1.7, ***2.0*** (2 trials), 2.1, 3.3, 3.4, 3.7, 4.3, 7.8, 8.1, and 10 mg/kg (fresh weight) or 0.91 (2 trials), 1.5, 1.6, 1.9, 3.1, 3.5, 3.6, ***3.7*** (2 trials), 4.4, 4.5, 4.8, 5.2, 8.9, 12, and 19 mg/kg (dry weight). The Meeting estimated a maximum residue level of 20 mg/kg and an STMR value of 3.7 mg/kg for parathion in wheat straw and fodder (dry weight).

As noted above, parathion is registered in the USA for use on soya beans. Eight trials in three states in 1988, with two aerial applications of 0.90 kg ai/ha and a PHI of 20 days resulted in the following concentrations of parathion residues in *soya bean hay*: 0.13, 0.25, 0.32, 0.46, 0.50, 0.57, 0.61, and 0.62 mg/kg (fresh weight), and those of the combined parathion and paraoxon residues were 0.13, 0.25, 0.43, ***0.46***, ***0.61***, 0.62, 0.68, and 0.81 mg/kg (fresh weight). Allowing for the standard 85% of dry matter in soya bean hay (FAO, 1997, p. 126), the Meeting estimated a maximum residue level of 2 mg/kg and an STMR value of 0.63 mg/kg for parathion in soya bean fodder (dry weight).

***Fate of residues during processing***

The Meeting received information on the fate of incurred residues of parathion and paraoxon during the processing of lemons, grapefruit, oranges, apples, grapes, oats, maize, rice, sorghum, wheat, sunflower seed, cotton seed, and rape seed, and processing factors were calculated for processed commodities derived from these raw agricultural commodities. The studies on apples, cotton seed, maize, sorghum, sunflower seed, and wheat are summarized below because maximum residue levels are estimated for these raw agricultural commodities.

Processing factors were calculated for parathion residues and for combined parathion and paraoxon residues. As parathion is the dominant component of the residue, the processing factor is similar with the two calculations. Nevertheless, since these factors are used in calculating the concentrations of residues in processed foods for the purpose of estimating dietary intake, that for the combined residue was used when available. When the concentration of residues in the processed commodity did not exceed the LOQ, the processing factor was calculated from the LOQ and was prefixed with a ‘less than’ symbol (<).

The factors for estimating parathion after the processing of *apples* to dry pomace were divergent, 3.1 and 0.16, reflecting the results of two processes. The Meeting decided to use the conservative value of 3.1 rather than the mean, which would represent neither process. Residues were detected in apple juice with one process but not the other, leading to processing factors of <0.018 and 0.072, and the conservative value 0.072 was chosen. Application of these factors to the STMR value

and MRL for apples provides an STMR-P value of 0.078 mg/kg and a HR-P value of 0.62 mg/kg for dry apple pomace and an STMR value for apple juice of 0.0018 mg/kg.

The processing factors for dry milling of *maize* were grits (<0.36, 0.99; best estimate, 0.99), meal (0.69, 0.88; mean, 0.74), flour (0.47, 0.88; mean, 0.68), crude oil (0.47, 0.66; mean, 0.57), and refined oil (0.80, 2.0; mean, 1.4). The processing factors for wet milling of maize were starch (<0.36, <0.28; best estimate, <0.28), crude oil (1.3, 3.4; mean, 2.3), and refined oil (1.3, 3.5; mean, 2.4). Application of the factors to the STMR value and MRL for maize provides an STMR-P value of 0.037 mg/kg and an HR-P value of 0.074 mg/kg for maize meal and STMR-P values of 0.05 mg/kg for grits, 0.034 mg/kg for maize flour, and 0.014 mg/kg for maize starch. Application of the factor for maize flour (0.68) to the MRL for maize results in a calculated highest residue of 0.068 mg/kg in maize flour. The Meeting estimated a maximum residue level of 0.1 mg/kg for parathion in maize flour.

The two processes resulted in different concentrations of residues in maize oil. The processing factors for oils were 0.57 and 1.4 with the dry process and 2.3 and 2.4 with the wet process. The Meeting agreed to use the values for the wet process, which, when applied to the STMR value for maize, provide STMR-P values of 0.12 mg/kg for both crude oil and refined oil. Application of the processing factors to the MRL for maize results in calculated highest residues of 0.23 and 0.24 mg/kg in crude and refined oils, respectively. The Meeting estimated a maximum residue level of 0.3 mg/kg for parathion in both crude and refined maize oil.

The processing factors for parathion after milling of *sorghum* were 1.6, 3.7, 1.16, and 1.01 (mean, 1.9) for bran; 0.34 and 0.57 (mean, 0.46) for grits; 0.23 and 0.57 (mean, 0.40) for flour; and 0.012 and 0.018 (mean, 0.015) for starch. Application of the factors to the STMR value for sorghum provides STMR-P values of 2.0 mg/kg for bran, 0.49 mg/kg for grits, 0.42 mg/kg for flour, and 0.016 mg/kg for starch.

The processing factors for parathion after milling of *wheat* were 4.6 for bran, 0.80 for shorts, and 0.35 for flour. Application of the factors to the STMR value and MRL for wheat provides STMR-P and HR-P values of 0.10 and 0.80 mg/kg for wheat shorts and STMR-P values of 0.044 mg/kg for wheat flour and 0.58 mg/kg for wheat bran. Only one milling trial was available for wheat, a major commodity, and this was considered insufficient to allow estimation of maximum residue levels for wheat bran and flour.

The processing factors for *sunflower seed* were 0.072 for meal and 0.42 for refined sunflower seed oil. Application of the factors to the STMR value and MRL for sunflower seed provides STMR-P and HR-P values for sunflower seed meal of 0.0025 mg/kg and an STMR-P value for refined sunflower seed oil of 0.021 mg/kg. The Meeting noted that parathion and paraoxon residues in refined oil were depleted below the concentrations in the seed and estimated a maximum residue level of 0.05* mg/kg for edible sunflower seed oil, on the basis of the LOQ of the method in trials on sunflower seeds.

The processing studies on *cotton seed* could not be used because no data on residues were provided.

***Residues in animal and poultry commodities***

The Meeting estimated the dietary burden of parathion residues in farm animals on the basis of the diets listed in Appendix IX of the *FAO Manual* (FAO, 1997). Calculation from MRLs (or highest residues) provides concentrations in feed suitable for estimating maximum residue levels for animal commodities, while calculation from STMR values for feed is suitable for estimating STMR values for animal commodities. The percent dry matter is considered to be 100% for MRLs and STMR values expressed in dry weight.

| Commodity | MRL or HR, mg/kg | Group | % dry matter | MRL/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |
| Maize forage | 10 | AF | 100 | 10 | 15 | | | 1.50 | | |
| Sorghum forage | 10 | AF | 100 | 10 | | | | | | |
| Barley straw and fodder, dry | 30 | AS | 100 | 30 | 25 | 60 | | 7.50 | 18.00 | |
| Maize fodder | 30 | AS | 100 | 30 | | | | | | |
| Sorghum straw and fodder, dry | 15 | AS | 100 | 15 | | | | | | |
| Wheat straw and fodder, dry | 20 | AS | 100 | 30 | | | | | | |
| Soya bean fodder | 2 | AL | 100 | 2 | 10 | | | | 0.20 | |
| Maize meal | 0.074 | CF | 85 | 0.087 | | | | | | |
| Wheat shorts | 0.80 | CM | 88 | 0.91 | | | | | | |
| Barley | 7 | GC | 88 | 8.0 | 50 | 40 | 75 | 3.98 | 3.18 | 5.97 |
| Maize | 0.1 | GC | 88 | 0.11 | | | | | | |
| Sorghum | 5 | GC | 86 | 5.81 | | | 5 | | | 0.29 |
| Wheat | 1 | GC | 89 | 1.12 | | | | | | |
| Apple pomace, dry | 0.62 | AB | 100 | 0.62 | | | | | | |
| Sunflower seed meal | 0.0025 | | 92 | 0.003 | | | 20 | | | 0.00 |
| Total | | | | | | | | 13.2 | 21.2 | 6.26 |

| Commodity | STMR | Group | % dry matter | STMR/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |
| Maize forage | 2.28 | AF | 100 | 2.28 | | | | | | |
| Sorghum forage | 3.1 | AF | 100 | 3.10 | 15 | | | 0.47 | | |
| Barley straw and fodder, dry | 7.75 | AS | 100 | 7.75 | 25 | 60 | | 1.94 | 4.65 | |
| Maize fodder | 2.13 | AS | 100 | 2.13 | | | | | | |
| Sorghum straw and fodder, dry | 2.8 | AS | 100 | 2.80 | | | | | | |
| Wheat straw and fodder, dry | 3.7 | AS | 100 | 3.70 | | | | | | |
| Soya bean fodder | 0.63 | AL | 100 | 0.63 | 10 | | | 0.06 | | |
| Maize meal | 0.037 | CF | 85 | 0.044 | | | | | | |
| Wheat shorts | 0.10 | CM | 88 | 0.11 | | | | | | |
| Barley | 1.95 | GC | 88 | 2.22 | 50 | 40 | 75 | 1.11 | 0.89 | 1.66 |
| Maize | 0.05 | GC | 88 | 0.06 | | | | | | |
| Sorghum | 1.06 | GC | 86 | 1.23 | | | 5 | | | 0.06 |
| Wheat | 0.125 | GC | 89 | 0.14 | | | | | | |
| Apple pomace, dry | 0.078 | AB | 100 | 0.078 | | | | | | |
| Sunflower seed meal | 0.0025 | | 92 | 0.003 | | | 20 | | | 0.00 |
| Total | | | | | | | | 3.6 | 5.5 | 1.72 |

The dietary burdens of parathion for estimating MRLs and STMR values (concentrations of residue in animal feeds expressed in dry weight) are 13 and 3.6 ppm in beef cattle, 21 and 5.5 ppm in dairy cows, and 6.3 and 1.7 ppm in poultry. The studies of metabolism in goats fed diets containing 97 ppm and laying hens fed diets containing 16.5 ppm provide evidence that the concentration of parathion is likely to be low in meat, milk, and eggs. However, the duration of feeding in these studies was only 5 or 6 days, only one dietary concentration was tested making interpolation or extrapolation to other concentrations difficult, and the concentration in eggs may not have reached a plateau by the end of the study.

The Meeting decided that studies of farm animal feeding were needed for estimation of MRLs and STMR values for animal and poultry commodities. The Meeting was informed that a study in dairy cows and one in laying hens were available.

## RECOMMENDATIONS

The Meeting estimated the maximum residue levels and STMRs shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue
For compliance with MRLs: parathion
For estimation of dietary intake: sum of parathion and paraoxon expressed as parathion

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| FP 0226 | Apple[1] | 0.2 | 0.05* | 0.025 | 0.16 |
| JF 0226 | Apple juice | | | 0.0018 | |
| AB 0226 | Apple pomace, dry | | | 0.078 | 0.62 |
| FS 0240 | Apricot | W | 1 | | |
| GC 0640 | Barley[2] | 7 | - | 1.95 | 5.1 |
| AS 0640 | Barley straw and fodder, dry | 30 | - | 7.75 | |
| SO 0691 | Cotton seed | 3 | 1 | 0.35 | 2.1 |
| VA 0384 | Leek | W | 0.05 | | |
| FC 0204 | Lemon | W | 0.5 | | |
| GC 0645 | Maize | 0.1 | 0.1 | 0.05 | 0.09 |
| CF1255 | Maize flour | 0.1 | - | 0.034 | |
| AS 0645 | Maize fodder | 30 | - | 2.13 | |
| AF 0645 | Maize forage | 10 (dry wt) | - | 2.28 (dry wt) | |
| | Maize grits | | | 0.05 | |
| CF 0645 | Maize meal | | | 0.037 | 0.074 |
| | Maize starch | | | 0.014 | |
| OC 0645 | Maize oil, crude | 0.3 | - | 0.12 | |
| OR 0645 | Maize oil, edible | 0.3 | - | 0.12 | |
| FC 0206 | Mandarin | W | 0.5 | | |
| OC 0305 | Olive oil, virgin | W | 2 | | |
| FT 0305 | Olives | W | 0.5 | | |
| FC 0004 | Oranges, Sweet, Sour | W | 0.5 | | |
| FS 0247 | Peach | W | 1 | | |
| VR 0589 | Potato | W | 0.05* | | |
| GC 0651 | Sorghum | 5 | 5 | 1.06 | 4.2 |
| | Sorghum bran | | | 2.0 | |
| | Sorghum flour | | | 0.42 | |
| | Sorghum grits | | | 0.49 | |
| | Sorghum starch | | | 0.016 | |
| AF 0651 | Sorghum forage (green) | 10 (dry wt) | - | 3.1 (dry wt) | |
| AS 0651 | Sorghum straw and fodder, dry | 15 | - | 2.8 | |
| VD 0541 | Soya bean (dry) | 0.05* | 0.05* | 0.05 | 0.05 |
| AL 0541 | Soya bean fodder | 2 | - | 0.63 | |
| SO 0702 | Sunflower seed | 0.05* | 0.05* | 0.05 | 0.05 |
| | Sunflower seed meal | | | 0.0025 | 0.0025 |
| OR 0702 | Sunflower seed oil, edible | 0.05* | - | 0.021 | |
| VO 0447 | Sweet corn (corn-on-the-cob) | 0.05* | - | 0.05 | 0.05 |
| GC 0654 | Wheat | 1 | - | 0.125 | 0.96 |
| CM 0654 | Wheat bran, unprocessed | | | 0.58 | |
| CF 1211 | Wheat flour | | | 0.044 | |
| | Wheat shorts - animal feed | | | 0.10 | 0.80 |
| AS 0654 | Wheat straw and fodder, dry | 20 | - | 3.7 | |

[1]The information provided to the JMPR precludes an estimate that the acute dietary intake for children would be below the acute reference dose
[2]The information provided to the JMPR precludes an estimate that the acute dietary intake for the general population would be below the acute reference dose

## Further work or information

*Desirable*

1. An additional trial on milling of wheat for estimation of maximum residue levels in flour and bran.

2. Information on the fate of parathion during malting and brewing of barley.

3. Studies of farm animal feeding to permit estimation of maximum residue levels and STMR values for animal commodities. The Meeting was informed that studies in dairy cows and laying hens were available.

## Dietary risk assessment

### *Chronic intake*

The periodic review of parathion resulted in recommendations for new and revised MRLs and new STMR values for raw and processed commodities. Data on consumption were available for 10 food commodities and were used in calculating dietary intake. The results are shown in Annex 3.

The international estimated daily intakes from the five GEMS/Food regional diets, based on estimated STMR values, represented 7-20% of the ADI. The Meeting concluded that long-term intake of residues of parathion from uses that have been considered by the JMPR is unlikely to present a public health concern.

### *Short-term intake*

The IESTI of parathion was calculated for the food commodities (and their processing fractions) for which maximum residue levels and STMR values have been estimated and for which data on consumption were available. The results are shown in Annex 4. The IESTI represented 0-400% of the acute RfD for the general population. That representing 400% results from a direct calculation based on the residues in barley because no data were available on the fate of parathion during brewing. The IESTI represented 0-140% of the acute RfD for children. The value of 140% represents the estimated short-term intake of residues in apples, but the Meeting was informed that the large portion size (679 g) of apple consumption by children may represent total apple consumption (including apple juice) rather than consumption of whole apples only.

The Meeting concluded that the acute intake of residues of parathion from uses, other than on barley and apples, that have been considered by the JMPR is unlikely to present a public health concern.

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# PARATHION-METHYL (059)

## EXPLANATION

Parathion-methyl was first evaluated in 1965 and has been reviewed recently in 1991, 1992, 1994 and 1995. It was listed by the 1998 CCPR (30th Session, ALINORM 99/24, Appendix VII) for periodic re-evaluation by the 2000 JMPR. Comprehensive data were provided by the basic manufacturer. Information was also provided by the governments of Australia, Germany, The Netherlands, Poland and Thailand.

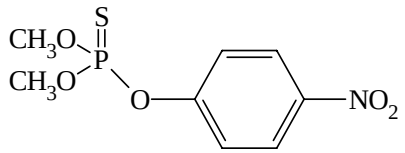
## IDENTITY

ISO common name: parathion-methyl

Chemical name
IUPAC: *O*,*O*-dimethyl *O*-4-nitrophenyl phosphorothioate
CA: *O*,*O*-dimethyl *O*-(4-nitrophenyl) phosphorothioate

CAS No.: 298-00-0

Structural formula:




Molecular formula: $C_8H_{10}NO_5PS$

Molecular weight: 263.2

### Physical and chemical properties

Pure active ingredient

Appearance: colourless, crystalline solid, with garlic-like odour.

Vapour pressure: 1.72 (± 0.09) × $10^{-5}$ mm Hg at 25°C

Melting point: 35-36°C

Octanol/water partition coefficient: $P_{ow}$ = 629, log $P_{ow}$ = 2.8

Solubility:

in water: 70.3 ± 2.73 mg/l at 25°C.
in solvents: readily soluble in most aromatic solvents such as hydrocarbons, alcohols, esters and ketones. Slightly soluble in petroleum and some mineral oils.

Specific gravity: 1.358 ($d^{20}_4$)

Hydrolysis:

hydrolysis half-life at 25°C in the dark under sterile conditions at initial concentration of 4 mg/l
pH 5: 68 days
pH 7: 40 days
pH 9: 33 days.

The dominant product of acid hydrolysis is demethyl parathion-methyl and of alkaline hydrolysis 4-nitrophenol. In neutral conditions nearly equal amounts of both products are formed.

Photolysis

Parathion-methyl had a half-life of approximately 49 hours in water in a photodegradation study (FIFRA Guideline N161-2).

Thermal stability

Heating at 150°C for 6.5 hours resulted in 91% isomerization to *O,S*-dimethyl-parathion-methyl, followed by generation of dimethyl sulfide, sulfur dioxide and other substances. Parathion-methyl should not be heated above 55ºC.

Technical material

Purity: minimum 80% (technical concentrate, 80% parathion-methyl in xylene).

Melting point: 17ºC

Main impurity: 4-nitrophenol, less than 0.2%.

Stability: Samples of parathion-methyl technical concentrate were stored in sealed glass bottles in the dark at 20ºC, 40ºC and 55ºC. The initial parathion-methyl content was 77.7%, which decreased marginally to 76.6% and 75.0% after two years at 20ºC and 40ºC respectively. After storage at 55ºC for 20 weeks the parathion-methyl content was 73.2%, and after one year at this temperature the material had polymerized.

**Formulations**

Danatox 50 EC, Novafos M 20, Novafos M 40, Parathion-methyl 4 EC, Parashoot 450 CS, Danacap 450 CS.

## METABOLISM AND ENVIRONMENTAL FATE

### Animal metabolism

The Meeting received information on the metabolism of parathion-methyl in lactating goats and laying hens.

Goats. A lactating goat weighing 60 kg was dosed orally by intubation with 35 mg per day of [*phenyl*-$^{14}$C]parathion-methyl, equivalent to 6.25 ppm in the diet, for 3 days (Van Dijk, 1988a). The goat produced 1.15 kg milk on day 2, the only full day of the experiment. Milk and excreta were collected throughout, and the animal was slaughtered 1 hour after the final dose. Residues were determined in the milk, blood, excreta and round, loin and flank muscle, subcutaneous, omental and perirenal fat, liver and kidneys. The total recovery of $^{14}$C was 35.5%. No residues of parathion-methyl or paraoxon-methyl were detected in tissues or milk. The results are shown in Tables 1 and 2.

Table 1. Residues in the tissues, milk, blood and excreta of a lactating goat (Van Dijk, 1988a).

| Sample | Recovered $^{14}$C | |
|---|---|---|
| | % of dose | mg/kg as parathion-methyl |
| Kidney | 0.3 | 1.6 |
| Liver | 0.5 | 0.59 |
| Muscle | 1.3 | 0.055 |

| Sample | Recovered $^{14}C$ | |
|---|---|---|
| | % of dose | mg/kg as parathion-methyl |
| Fat | 0.1 | 0.014 |
| Tissues + organs + blood | 2.8 | |
| Milk | <0.1 | 0.008-0.036 |
| Urine + pan rinse | 19.5 | |
| Faeces | 13.2 | |
| Total | 35.5 | |

Table 2. Identified metabolites in tissues and milk of a lactating goat (Van Dijk, 1988a).

| Compound | Concentration, mg/kg, as parathion-methyl | | | | |
|---|---|---|---|---|---|
| | milk, 8 hours after 2nd dose | kidney | liver | muscle | fat |
| *O*-demethyl-paraoxon-methyl | 0.013 | 0.17 | 0.14 | ndr | ndr |
| *N*-acetylaminophenol | ndr | ndr | 0.14 | ndr | ndr |
| *N*-acetylaminophenyl glucuronide | ndr | 0.21 | 0.037 | 0.001 | ndr |
| amino-paraoxon-methyl | 0.012 | 0.15 | ndr | 0.032 | 0.004 |
| *O*-demethyl-amino-parathion-methyl | ndr | 0.29 | ndr | ndr | ndr |
| *O*-demethyl-amino-paraoxon-methyl | ndr | 0.24 | 0.13 | 0.014 | ndr |
| *p*-aminophenyl glucuronide | 0.004 | ndr | ndr | 0.002 | ndr |
| amino-parathion-methyl | ndr | 0.029 | ndr | ndr | <0.001 |

ndr: no detected residue

Hens. Five white Leghorn-hybrid laying hens weighing 1.5-2.0 kg each were dosed 3 times at daily intervals by intubation with 0.5 mg/kg/bw of [*phenyl*-$^{14}$C]parathion-methyl equivalent to 6.25 ppm parathion-methyl in the feed (Van Dijk, 1988b). Eggs and excreta were collected throughout, and the birds killed 3 hours after the final dose.

The total recovery of $^{14}$C was 54%, with 52% of the dose in the excreta and 2% in the edible tissues, organs, eggs and blood. $^{14}$C in the digestive tract was not measured, but may have accounted for some of the missing $^{14}$C.

$^{14}$C levels expressed as parathion-methyl in eggs were in the range 0.001-0.031 mg/kg, with the highest level in an egg laid 3 hours after the third dose. Nitrophenol and nitrophenyl sulfate were the only metabolites identified in eggs.

The total $^{14}$C level was higher in kidneys than in the other tissues, but parathion-methyl was a minor component of the residue, the main compound being a glucuronide conjugate. Except for skin, fat was the tissue with the highest residue of parathion-methyl itself, suggesting a degree of fat solubility. Paraoxon-methyl was not detected in eggs or any of the tissues. The levels of identified compounds are shown in Table 3.

Table 3. Identified compounds in the tissues and eggs of laying hens dosed orally daily for 3 consecutive days with 0.5 mg/kg/bw [*phenyl*-$^{14}$C]parathion-methyl (Van Dijk, 1988b).

| Compounds | $^{14}$C, mg/kg as parathion-methyl | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | eggs | liver | kidney | muscle | gizzard | heart | fat | skin |
| parathion-methyl | nd | nd | 0.001 | nd | 0.004 | 0.001 | 0.008 | 0.011 |
| *O*-demethyl-paraoxon-methyl | nd | nd | 0.014 | 0.010 | nd | nd | nd | nd |
| e-nitrophenyl phosphorothioate | nd | nd | 0.008 | nd | nd | nd | nd | 0.002 |
| *p*-nitrophenol | 0.008 | 0.007 | 0.005 | nd | 0.011 | 0.005 | 0.008 | 0.064 |
| *p*-nitrophenyl sulfate | 0.021 | nd | nd | nd | nd | nd | nd | 0.003 |

| Compounds | $^{14}C$, mg/kg as parathion-methyl | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | eggs | liver | kidney | muscle | gizzard | heart | fat | skin |
| *N*-acetylaminophenol | nd | 0.048 | nd | nd | 0.002 | 0.012 | nd | nd |
| *N*-acetylaminophenyl glucuronide | nd | 0.017 | 0.11 | nd | nd | 0.013 | nd | 0.012 |
| *O*-demethyl-amino-parathion-methyl | nd | nd | nd | nd | nd | 0.003 | nd | 0.006 |
| *p*-aminophenol | nd | nd | 0.014 | nd | nd | 0.002 | nd | nd |
| *p*-aminophenyl glucuronide | nd | nd | 0.020 | nd | nd | 0.001 | nd | nd |
| Total $^{14}C$ as parathion-methyl | | 0.076 | 0.24 | 0.017 | 0.029 | 0.051 | 0.021 | 0.11 |

nd: not detected

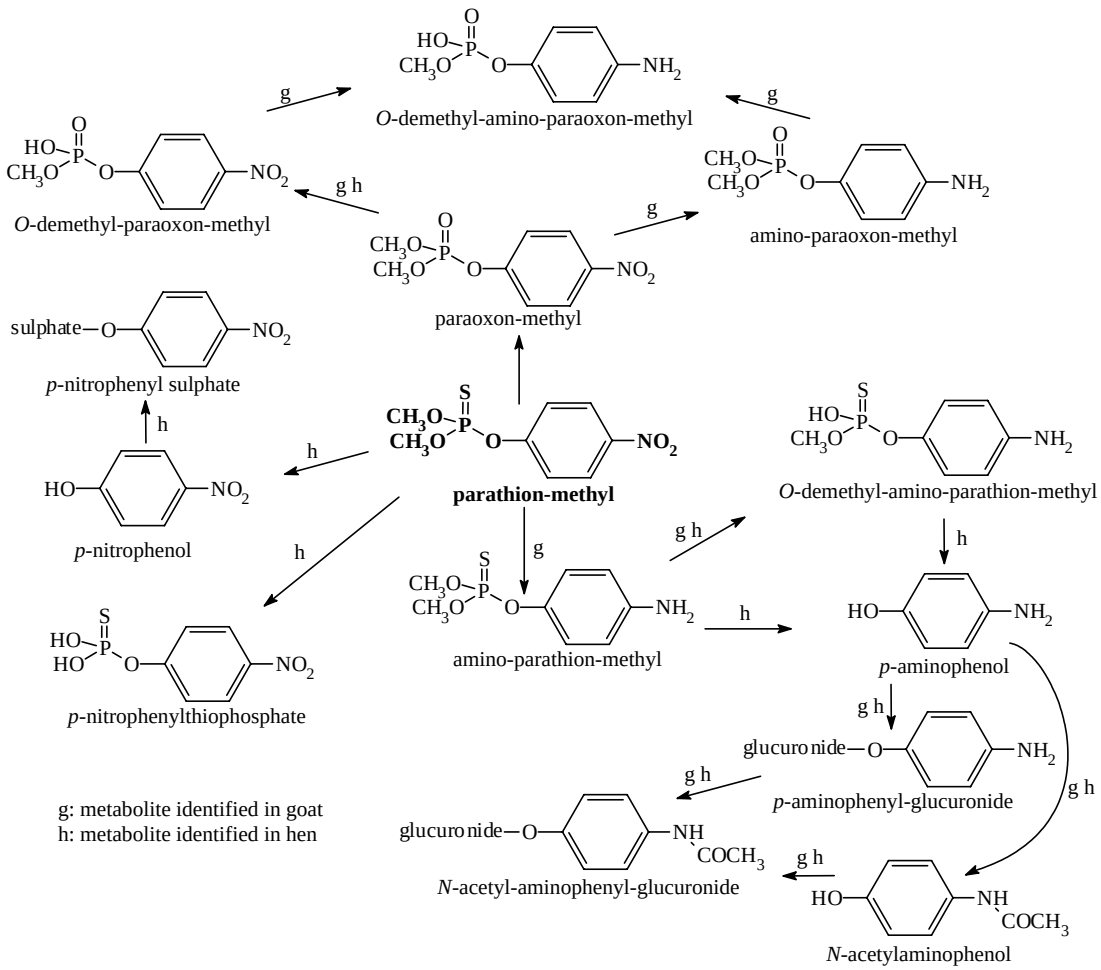



Figure 1. Animal metabolism of parathion-methyl.

## Plant metabolism

The Meeting received information on the metabolism of parathion-methyl in potatoes, cotton and lettuce.

Potatoes. Linke and Brauner (1988) applied [*phenyl*-$^{14}C$]parathion-methyl once as a foliar spray to 3-month old plants at a rate equivalent to 4.7 kg ai/ha and harvested them 5 days and 21 days after treatment. In the plants harvested on day 5, the distribution of $^{14}C$ was 0.01-0.02% in tubers and 99.98-99.99% in foliage, and on day 21 0.13-0.14% and 99.86-99.87% respectively. The components

of the residue identified in the tubers are shown in Table 4. Paraoxon-methyl was a tentative identification.

Table 4. Compounds in tubers harvested 5 and 21 days after a single foliar treatment with [*phenyl*-$^{14}$C]parathion-methyl at a rate equivalent to 4.7 kg ai/ha.

| Compound | $^{14}$C as parathion-methyl, mg/kg | |
|---|---|---|
| | Tubers harvested 5 days after crop treatment | Tubers harvested 21 days after crop treatment |
| parathion-methyl | 0.001 | <0.001 |
| paraoxon-methyl | | 0.002 |
| *p*-nitrophenyl conjugate | 0.002 | 0.003 |
| *p*-nitrophenyl glucoside | | <0.001 |
| demethyl-parathion-methyl | 0.002 | 0.002 |
| After hydrolysis | | |
| parathion-methyl | <0.001 | |
| *p*-nitrophenol | <0.001 | 0.011 |
| Total $^{14}$C | 0.024 | 0.068 |

Cotton. A plant was foliar-sprayed with [*phenyl*-$^{14}$C]parathion-methyl at a rate equivalent to 0.376 kg ai/ha (Linke *et al.*, 1988). Ten days later the $^{14}$C level in the seed was 0.08 mg/kg expressed as parathion-methyl. Parathion-methyl (0.008 mg/kg), *p*-nitrophenol (0.009 mg/kg), *p*-nitrophenyl glucopyranoside (0.007 mg/kg) and demethyl-parathion-methyl (0.004 mg/kg) were identified as components of the residue.

In a second experiment a solution of [*phenyl*-$^{14}$C]parathion-methyl was applied directly to cotton leaves with a syringe (Linke *et al.*, 1988). The leaves were analysed fifteen days later. The total $^{14}$C expressed as parathion-methyl in the plants was 484 mg/kg; identified components of the residue were parathion-methyl (168 mg/kg), *p*-nitrophenol (48 mg/kg), *p*-nitrophenyl glucopyranoside (55 mg/kg), demethyl-parathion-methyl (55 mg/kg), and demethyl-paraoxon-methyl (15 mg/kg), all concentrations expressed as parathion-methyl. Paraoxon-methyl was not detected.

Lettuce. In a separate trial lettuces were sprayed with an emulsion of [*phenyl*-$^{14}$C]parathion-methyl at a rate equivalent to 1.23 kg ai/ha and sampled 14 and 21 days later (Ritter, 1988). Identified components of the residue are shown in Table 5. No P=O metabolites were detected. At least 7 other metabolites were observed, but none could be hydrolysed to *p*-nitrophenol, suggesting significant metabolism of the parent molecule.

Table 5. Identified components of the $^{14}$C residue in lettuce treated with [*phenyl*-$^{14}$C]parathion-methyl (Ritter, 1988).

| Compound | $^{14}$C, mg/kg as parathion-methyl | |
|---|---|---|
| | Day 14 | Day 21 |
| parathion-methyl | 2.2 | 0.97 |
| *p*-nitrophenol | 2.5 | 2.07 |
| *p*-nitrophenyl glucopyranoside | 0.30 | 0.33 |
| demethyl-parathion-methyl | 0.25 | 0.41 |
| Total $^{14}$C | 11.4 | 9.7 |

Linke (1988) further examined the unextractable $^{14}$C in plants treated with [*phenyl*-$^{14}$C]parathion-methyl (Ritter, 1988), which accounted for 38% of the total $^{14}$C on day 14 and 45% on day 21. Vigorous cid and alkaline hydrolysis released *p*-nitrophenol accounting for 12% of the $^{14}$C in

the leaves. Attempts were made to determine whether part of the unextractable $^{14}C$ in the leaves resulted from incorporation of $^{14}CO_2$ produced by degradation of parathion-methyl in the soil. Very small amounts of $^{14}C$-labelled glucose were identified, but a quantitative estimate was not possible.

Boner (1998) treated greenhouse plants at about 72 days with [*phenyl*-$^{14}C$]parathion-methyl at a rate equivalent to 1.1 kg ai/ha and harvested the lettuce 21 days later (Table 6). The major metabolite was 4-nitrophenyl 6-*O*-malonyl-ß-D-glucopyranoside. The remaining unidentified metabolites were very polar. Metabolism of parathion-methyl proceeds via cleavage to *p*-nitrophenol which is conjugated with glucose or further with a malonyl group.

Table 6. Identified components of the $^{14}C$ residue in lettuce treated with [*phenyl*-$^{14}C$]parathion-methyl (Boner, 1998).

| Compound | % of total $^{14}C$ | mg/kg as parathion-methyl |
|---|---|---|
| parathion-methyl | 36.2 | 2.1 |
| *p*-nitrophenol | 5.2 | 0.30 |
| *p*-nitrophenyl glucoside | 9.9 | 0.58 |
| demethyl-parathion-methyl | 3.7 | 0.22 |
| 4-nitrophenyl 6-*O*-malonyl-ß-D-glucopyranoside | 25.5 | 1.49 |
| Total $^{14}C$ | 100 | 5.9 |

Proposed routes of metabolism are shown in Figure 2.

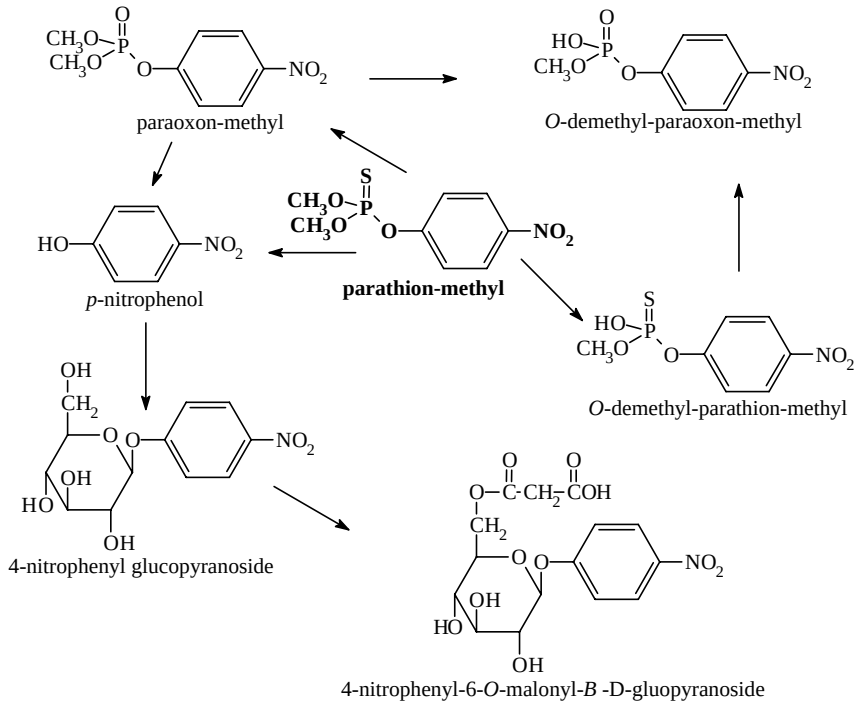



Figure 2. Proposed metabolic pathways of parathion-methyl in plants.

**Environmental fate in soil**

The Meeting received information on the adsorption, desorption, mobility, dissipation, aerobic and anaerobic degradation, and photolysis of parathion-methyl in soil.

Adsorption. Daly (1989) measured the adsorption-desorption of parathion-methyl in 4 soils equilibrated with 0.01M $CaCl_2$ solutions. One or 3 g samples of the soils were sterilized by autoclave and equilibrated with 10-ml aliquots of [*phenyl*-$^{14}$C]parathion-methyl at 2, 10, 15 and 20 µg/ml in aqueous 0.01M $CaCl_2$ by shaking at 25°C for 24 hours. The concentration of $^{14}$C was measured in the decanted supernatant after centrifugation. The stability of the [$^{14}$C]parathion-methyl was examined by TLC, and desorption was determined by equilibrating the residual soil sediment with 0.01M $CaCl_2$ solution and measuring the $^{14}$C released.

Table 7. $K_d$ and $K_{oc}$ values for parathion-methyl in 4 soils (Daly, 1989). ($K_d$ is ratio of pesticide concentration in soil and $K_{oc}$ that of pesticide concentration in soil organic matter, to that in the aqueous phase at equilibrium.)

| Soil | pH | % organic carbon | Adsorption | | Desorption | | Mobility |
|---|---|---|---|---|---|---|---|
| | | | $K_d$ | $K_{oc}$ | $K_d$ | $K_{oc}$ | |
| Silt loam | 7.8 | 1.2 | 7.09 | 591 | 11.1 | 923 | Low |
| Clay loam | 7.5 | 1.3 | 8.71 | 670 | 12.3 | 947 | Low |
| Sandy loam | 6.5 | 0.40 | 1.82 | 456 | 2.71 | 677 | Medium-low |
| Sand | 7.4 | 0.25 | 0.57 | 230 | 0.89 | 357 | Medium |

Degradation. Patterson and Bielefeld (1990) incubated [*phenyl*-$^{14}$C]parathion-methyl in a soil consisting of 56% sand, 26% silt, and 18% clay, with 1.6% organic matter (pH 6.8) at a nominal 10 mg/kg under aerobic conditions at 25°C in the dark for 6 months. Moisture levels were maintained at approximately 75% of field capacity. 92.5-101% of the applied $^{14}$C, including volatiles, was accounted for.

Levels of parathion-methyl and degradation products are shown in Table 8. The estimated half-life of parathion-methyl in the first 14 days was 3.9 days, and the mineralization half-life 16 days. Once most of the parathion-methyl had disappeared, the mineralization half-life was approximately 400 days calculated for the 28-181 days period. The main component of the identified residue was parathion-methyl, and the identified products were *p*-nitrophenol and *O*,*O*-bis(4-nitrophenyl) *O*-methyl phosphorothioate. The unextractable residue reached a maximum after 1 month and then decreased at approximately the same rate as the long-term mineralization. The degradation is shown in Figure 3.

Table 8. Dissipation of parathion-methyl and generation of products during aerobic degradation of labelled parathion-methyl in a sandy loam soil (Patterson and Bielefeld, 1990).

| Time | $^{14}$C, % of applied | | | | | |
|---|---|---|---|---|---|---|
| | Total | $CO_2$ | unextractable | parathion-methyl | 4-nitrophenol | $C_{13}H_{11}N_2O_7S$[1] |
| day 0 | 101 | 0 | 0.34 | 98 | 0 | 2.3 |
| day 1 | 97.4 | 0.22 | 2.5 | 79 | 7.7 | 1.8 |
| day 3 | 94.1 | 10.8 | 15.1 | 54 | 5.6 | 1.7 |
| day 7 | 91.3 | 30 | 27 | 27 | 0.13 | 1.38 |
| day 14 | 93.5 | 43 | 37 | 7.9 | 0 | 1.06 |
| 1 month | 95.9 | 51 | 39 | 1.6 | 0.27 | 0.37 |
| 2 months | 92.9 | 58 | 31 | 0.69 | 0.06 | 0.28 |
| 3 months | 96.9 | 60 | 33 | 0.32 | 0 | 0.27 |

| Time | $^{14}C$, % of applied | | | | | |
|---|---|---|---|---|---|---|
| | Total | $CO_2$ | unextractable | parathion-methyl | 4-nitrophenol | $C_{13}H_{11}N_2O_7S$[1] |
| 4 months | 94.7 | 61 | 30 | 0.33 | 0 | 0.19 |
| 6 months | 92.5 | 63 | 27 | 0.16 | 0 | 0.15 |

[1]*O*,*O*-bis(4-nitrophenyl) *O*-methyl phosphorothioate

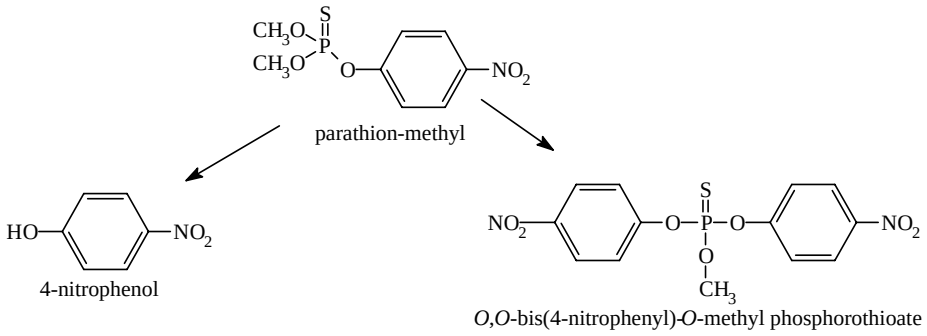



Figure 3. Aerobic degradation of parathion-methyl.

Patterson (1990) repeated the previous experiment, but under anaerobic conditions for 12 months. The soil was flooded with water. The recovery of $^{14}C$, including volatiles, was in the range 90.8-110%. Parathion-methyl had an estimated half-life of 10.5 hours calculated for the first 24 hours (Figure 4). A number of products were identified, but mostly as minor components of the residue (Table 9). Nitrophenol was the main identified degradation product at most samplings. Very little mineralization had occurred after 1 year, and most of the $^{14}C$ had been incorporated into the unextractable fraction. The degradation pathways are shown in Figure 5.

Table 9. Disappearance of parathion-methyl and generation of products during anaerobic degradation of labelled parathion-methyl in a sandy loam soil (Patterson, 1990).

| Time | $^{14}C$, % of applied | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | Total | $CO_2$ | Unex-tractable | parathion-methyl | *O*,*O*-bis(4-nitrophenyl) *O*-methyl phosphorothioate | *S*-methyl-parathion-methyl | *S*-nitrophenyl-parathion-methyl | paraoxon-methyl | 4-nitrophenol | amino-parathion-methyl |
| 0 h | 99.9 | 0 | 1.4 | 78 | 2.3 | 0 | 0 | 0 | 4.7 | 0 |
| 1 h | 110 | 0 | 3.5 | 50 | 1.6 | 0 | 0 | 0 | 17 | 0 |
| 2 h | 97.4 | 0 | 4.8 | 39 | 1.2 | 0 | 0 | 0 | 3.7 | 0 |
| 4 h | 96.5 | 0 | 5.6 | 23 | 0 | 0.6 | 1.1 | 0.7 | 17 | 0 |
| 6 h | 100 | 0 | 7.3 | 41 | 0 | 0 | 0 | 0 | 17 | 0 |
| 12 h | 93.8 | 0 | 9.0 | 25 | 0 | 1.7 | 0.9 | 1.7 | 18 | 0 |
| 1 d | 94.1 | 0 | 15 | 11 | 0 | 2.7 | 1.1 | 2.1 | 19 | 0 |
| 3 d | 92.3 | 0 | 17 | 0.9 | 0 | 0.8 | 0 | 0 | 17 | 0 |
| 7 d | 93.2 | 0 | 33 | 0.7 | 0 | 0.5 | 0 | 0 | 14 | 1.5 |
| 14 d | 92.9 | 0.01 | 52 | 0 | 0 | 0.9 | 0 | 0.2 | 1.7 | 0.6 |
| 30 d | 93.1 | 0.02 | 59 | 0.2 | 0 | 0.6 | 0 | 0 | 0.7 | 0.8 |
| 61 d | 99.4 | 1.75 | 71 | 0.1 | 0 | 0 | 0 | 0 | 0 | 1.1 |
| 91 d | 93.5 | 1.8 | 70 | 0 | 0 | 0.6 | 1.1 | 0 | 0.9 | 0.7 |
| 121 d | 92.2 | 2.21 | 68 | 0.1 | 0 | 1.2 | 0 | 0 | 0.9 | 0.3 |
| 182 d | 90.8 | 2.33 | 79 | 0 | 0 | 0.1 | 0.1 | 0.1 | 0.4 | 0.3 |
| 273 d | 98.4 | 2.53 | 78 | 0 | 0 | 0.2 | 0.2 | 0.2 | 0.7 | 0.2 |
| 365 d | 95.5 | 2.77 | 75 | 0 | 0 | 0.1 | 0 | 0.1 | 0.6 | 0.1 |

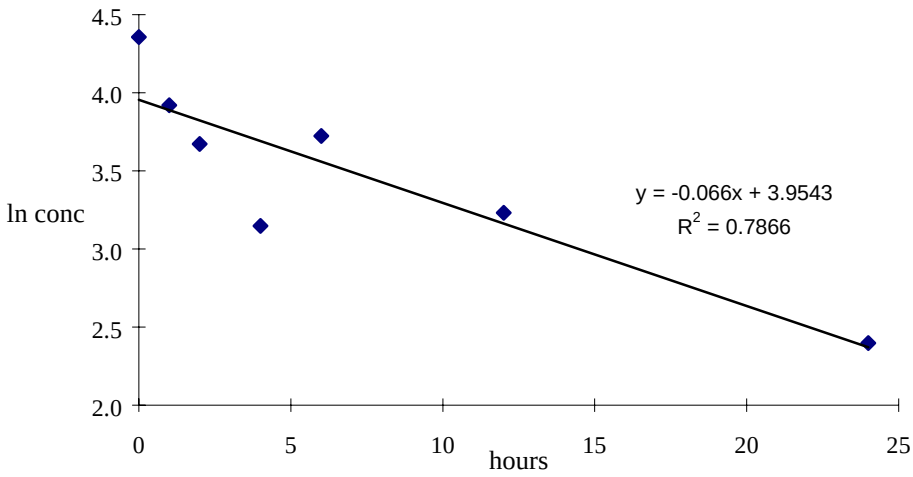



Figure 4. Degradation of parathion-methyl during anaerobic incubation of a sandy loam soil (Patterson, 1990).

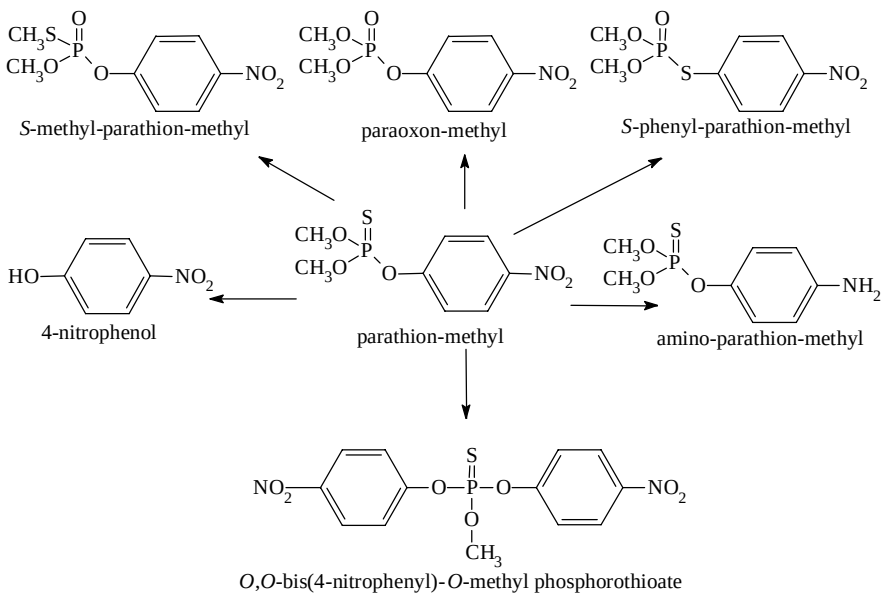



Figure 5. Anaerobic degradation of parathion-methyl.

Mobility. Pors (1995) compared the mobility of parathion-methyl in soil when formulated as an EC (emulsifiable concentrate) and as a CS (capsule suspension). The test soils were a sand (89.3% sand, 1.4% organic carbon) and a sandy loam (69.8% sand, 1.7% organic carbon). The formulations were applied at a rate equivalent to 200 µg ai for the EC and 55 µg for the CS to stainless steel columns of the soils (h 300 mm, diam 50 mm). Simulated rainwater was applied to the columns 18 times during 48 hours at volumes ranging from 18 to 25 ml. Parathion-methyl was not detected (<0.002 mg/l) in the percolates (volume 395 ml). There was no detectable mobility of parathion-methyl in soil from either formulation.

Photolysis. In two tests Wilmes (1987a) used a xenon lamp to simulate sunlight irradiating [*phenyl*-$^{14}$C]parathion-methyl (about 0.3 mg/cm$^2$) on a sandy loam soil at 27°C for 11 days. In one the soil was dampened but maintained below its maximum water capacity, and the initial half-life of parathion-methyl was 3.9 days with the longer-term half-life estimated as 8.6 days. In the other water was minimized and the initial and later half-lives were 4.5 and 24 days respectively. The moisture appeared to assist mobility, allowing more exposure to the irradiation.

The estimated half-lives in midsummer at 40° latitude were 17-19 days for a fresh residue and 37-104 days for longer-term residues.

Dissipation. In a trial in California, USA, an EC formulation was applied to a cotton crop 6 times at weekly intervals at 1.1 kg ai/ha (Rice *et al.*, 1990a). The soil was a sandy loam (surface soil 0.2% organic matter, pH 6.2, 77% sand, 17% silt, 6% clay). Soil cores to a depth of 1.2 m were taken before and after each application and then 1, 7, 14, 21 and 28 days after the final application. Cores were sectioned down the profile into 0-10 cm, 10-20 cm, 20-30 cm, 30-45 cm, 45-60 cm, 60-90 cm and 90-120 cm for analysis.

Residues of parathion-methyl were detected (LOQ 0.05 mg/kg) only in 0-10 cm samples taken immediately after spraying and 1 day after the final application, where the means of duplicate field samples were 0.37, 0.32, 0.25, 0.16, 0.16, 0.085 and 0.033 mg/kg. The identity of parathion-methyl in one duplicate of each of the first two samples was confirmed by GC-MS (Jacobsen and Fieser, 1990a). No residues of paraoxon-methyl were detected in any sample. The dissipation of parathion-methyl was too rapid to be measured accurately and no movement down the soil profile was detected. The study suggests that parathion-methyl disappears quickly by degradation and residues are unlikely to move down the soil.

Samples had been stored in a freezer for 40-289 days before analysis. A freezer storage study on the same soil showed that parathion-methyl residues would not have changed, but paraoxon-methyl residues would have decreased by approximately 30% at the longer intervals.

A second trial (Rice *et al.*, 1990b) was identical except that the EC formulation was applied to a cotton crop in Missouri and the soil was a loam (surface soil 0.7% organic matter, pH 5.4, 30% sand, 46% silt, 24% clay).

Again residues of parathion-methyl were only at 0-10 cm. Duplicate samples taken immediately after spraying and 1 day after the final application contained mean residues of 0.039, 0.030, 0.059, 0.087, 0.022 and 0.052 mg/kg. The identity of parathion-methyl in one duplicate of each of two of the samples (0.059 and 0.087 mg/kg) was confirmed by GC-MS (Jacobsen and Fieser, 1990b). No residues of paraoxon-methyl were detected in any sample, as in the California study. The samples were stored in a freezer for 19-182 days before analysis, which would not have affected parathion-methyl levels but would be likely to have reduced paraoxon-methyl residues at the longer intervals. However, 5 surface soil samples taken soon after spraying were analysed after 19-47 days of storage.

**Environmental fate in water/sediment systems**

The Meeting received information on parathion-methyl photolysis in aqueous solution and on field dissipation in rice paddies.

In a photolysis experiment Wilmes (1987b) used a xenon lamp to irradiate [*phenyl*-$^{14}$C]parathion-methyl dissolved in a sterile aqueous buffer (pH 5) at a concentration of 5 mg/l at 25°C. The half-life was 48 hours, equivalent to an environmental half-life of about 9 days for 40 degrees latitude with no cloud and in the water layer. The main photoproduct was *p*-nitrophenol. Paraoxon-methyl accounting for up to 2% of the applied $^{14}$C was detected in the water. In a second experiment where volatiles were collected, the distribution of $^{14}$C after 212 hours irradiation was 5.9%

parathion-methyl, 3.4% demethyl-parathion-methyl, 1.6% *p*-nitrophenol, 41.7% polar compounds and 24.7% $CO_2$. The demethyl-parathion-methyl was probably a product of hydrolysis rather than photolysis because it was also detected in the dark controls.

Hellpointer (1992), in a laboratory study, estimated the very low quantum yield of 0.0001 for the direct photodegradation of parathion-methyl dissolved in water and irradiated with polychromatic light. Modelling with this value suggests that direct photodegradation in water is a very minor contributor to the overall elimination of parathion-methyl in the environment.

Wilmes (1988) reported that the hydrolysis half-life of parathion-methyl in a sterile aqueous buffer solution at pH 5 and 25°C was 68 days.

In an aquatic field dissipation study an EC formulation was applied 6 times at weekly intervals at 0.84 kg ai/ha to rice paddy in California, USA (Rice *et al.*, 1990c). The soil was a sandy loam (surface soil 0.2% organic matter, pH 6.2, 77% sand, 17% silt, 6% clay). Soil cores to a depth of 0.6 m were taken after each application and then at 1, 7, 14, 21 and 28 days after the final application. Cores sectioned into 0-10 cm, 10-20 cm, 20-30 cm, 30-45 cm and 45-60 cm were analysed for parathion-methyl and paraoxon-methyl, but none was detected (<0.05 mg/kg) in any sample. The soil samples were stored in a freezer for 30-192 days before analysis, which would not have influenced the residue levels of either compound. The soil was the same as in the dissipation trial with a cotton crop (Rice *et al.*, 1990a).

Parathion-methyl was detected (LOQ 0.01 mg/l) in water samples, but only on the days of treatment at 0.21, 0.17, 0.30, 0.28, 0.30 and 0.13 mg/l and the day after the final treatment at 0.070 mg/l. The identity of parathion-methyl in one of each of two duplicate field samples (0.30 and 0.30 mg/kg) was confirmed by GC-MS (Jacobsen and Fieser, 1990c). Paraoxon-methyl was not detected (<0.01 mg/l) in any water sample. The water samples were stored frozen for 12-47 days before analysis.

The study suggests rapid degradation of parathion-methyl in an aquatic field environment with negligible movement down the soil profile.

In a second trial an EC formulation was applied at 6 weekly intervals at 0.84 kg ai/ha to rice paddy in Missouri, USA (Rice *et al.*, 1990d). The soil was the sandy loam (0.7% organic matter, pH 5.4, 30% sand, 46% silt, 24% clay) used previously by Rice *et al.* (1990b). The procedure was the same as in the California trial (Rice *et al.*, 1990c). Parathion-methyl was detected (LOQ 0.01 mg/l) in water samples on the days of and after treatment, and paraoxon-methyl was also detected twice (Table 10). Water samples were stored frozen for 22-53 days before analysis.

Soil cores were analysed for parathion-methyl and paraoxon-methyl, but neither was detected in any sample. Soil samples were stored in a freezer for 112-222 days before analysis, which would not have influenced the residue levels of parathion-methyl, but paraoxon-methyl residues, if present initially, would have been substantially degraded.

Table 10. Residues in the water from a rice plot treated at 6 weekly intervals with parathion-methyl at 0.84 kg ai/ha in Missouri, USA (Rice *et al.*, 1990d).

| Days after initial treatment | Days after most recent treatment | parathion-methyl, mg/l | paraoxon-methyl, mg/l | Comments |
|---|---|---|---|---|
| -1 | | <0.01 (2) | <0.01 (2) | |
| 0 | | <0.01 (2) | <0.01 (2) | |
| 0 treatment 1 | 0 | <u>0.40</u> <u>0.41</u> 0.12 | <0.01 (3) | Parathion-methyl confirmed by GC-MS in underlined samples (Jacobsen and Fieser, 1990d) |
| 7 | 7 | <0.01 (2) | <0.01 (2) | |

| Days after initial treatment | Days after most recent treatment | parathion-methyl, mg/l | paraoxon-methyl, mg/l | Comments |
|---|---|---|---|---|
| 7 treatment 2 | 0 | 0.14 0.14 0.18 | <0.01 (3) | |
| 14 | 7 | <0.01 (2) | <0.01 (2) | |
| 14 treatment 3 | 0 | 0.27 0.067 | <0.01 (2) | |
| 21 | 7 | <0.01 (2) | <0.01 (2) | |
| 21 treatment 4 | 0 | 0.24 0.22 | <0.01 0.011 | |
| 28 | 7 | <0.01 (3) | <0.01 (3) | |
| 28 treatment 5 | 0 | 0.13 <u>0.31</u> | <0.01 0.012 | Parathion-methyl confirmed by GC-MS in underlined sample. Paraoxon-methyl was too low for GC-MS confirmation. Sample may have been stored for 15 months before analysis, which could have allowed some loss of paraoxon-methyl if present. |
| 35 | 7 | <0.01 (2) | <0.01 (2) | |
| 35 treatment 6 | 0 | <0.01 (2) | <0.01 (2) | |
| 36 | 1 | 0.027 0.030 | <0.01 (2) | |
| 42 | 7 | <0.01 (2) | <0.01 (2) | |
| 49 | 14 | <0.01 (2) | <0.01 (2) | |
| 56 | 21 | <0.01 (3) | <0.01 (3) | |
| 63 | 28 | <0.01 (3) | <0.01 (3) | |

## METHODS OF RESIDUE ANALYSIS

### Analytical methods

The Meeting received information on analytical methods for residues of parathion-methyl and paraoxon-methyl in a variety of crops and processed commodities.

Gilbert (1996e) described a method for the determination of parathion-methyl and paraoxon-methyl in peaches and peach juice. A 20 g subsample is homogenised with 100 ml of acetone/water, the homogenate vacuum filtered, and the acetone removed by rotary film evaporation. The resulting aqueous solution is mixed with saturated sodium chloride and extracted with dichloromethane. The extract is dried with sodium sulfate and evaporated to dryness, and the residue taken up in methanol and cleaned up on a C-18 column. The eluate is evaporated and the residue taken up in ethyl acetate for GLC analysis with FP detection.

Recoveries of parathion-methyl were 83-95% and of paraoxon-methyl 84-99% at fortifications of 0.01, 0.2 and 2.0 mg/kg (n=18) for peaches and juice. The LOQ was 0.01 mg/kg.

Gilbert (1996f) used the same method on raisins, wine and grape juice. Recoveries were 77-96% for parathion-methyl and 84-108% for paraoxon-methyl at fortifications of 0.01, 0.2 and 2.0 mg/kg (n=27). The LOQ was 0.01 mg/kg.

Gilbert (1997a) used essentially the same method on apple juice and dry pomace. Juice samples did not require a C-18 column clean-up and were ready for GLC analysis after extraction with dichloromethane. Recoveries of parathion-methyl were 76-91% from pomace and 91-114% from juice and of paraoxon-methyl 65-89% from pomace and 92-116% from juice, at fortifications of 0.01, 0.2 and 2.0 mg/kg, n=9, for each commodity. The LOQ was 0.01 mg/kg.

Bower (1995) validated a GLC analytical method for apples and grapes. Samples were extracted with an acetone/water mixture (80:20) and the filtered extract treated with saturated sodium chloride and extracted with dichloromethane. The dichloromethane was evaporated and the residue dissolved in methanol and cleaned up on a small C-18 column. Analysis was by GLC with an FPD. Recoveries of parathion-methyl were 73-95% (mean 86%, n=18) and of paraoxon-methyl 84-112%

(mean 98%, n=18) from apples and grapes fortified at 0.01, 0.2 and 2.0 mg/kg. Recoveries of paraoxon-methyl tended to be higher at 0.01 mg/kg. The LOQ was 0.01 mg/kg.

Norby (1993) described a method for parathion-methyl and paraoxon-methyl (also parathion and paraoxon) in canola and its processed commodities. The extraction solution (100 ml of methanol + water + HCl) is blended with canola meal (25 g) and the whole mixture with washings and rinsings is refluxed gently for 30 minutes, cooled and filtered, and the methanol evaporated. The residual aqueous solution is treated with saturated sodium chloride and the residues are extracted into ethyl acetate, which is dried with sodium sulfate and evaporated to 5-10 ml for analysis by GLC with an FPD. For canola seed, oil and processing waste an additional clean-up with hexane-acetonitrile partition was introduced after the reflux step. Recoveries were more variable for paraoxon-methyl than for parathion-methyl (Table 11). Some values fell outside the acceptable 70-120% range with excessive variation at a fortification level of 0.02 mg/kg, so the LOQ was determined to be 0.05 mg/kg.

Table 11. Recoveries of parathion-methyl and paraoxon-methyl from canola and its processed commodities fortified at four levels (Norby, 1993).

| Fortification, mg/kg | Recovery, % | | | | Mean recovery | SD |
|---|---|---|---|---|---|---|
| | Seed | Meal | Crude oil | Refined oil | | |
| parathion-methyl | | | | | | |
| 0.02 | 103 122 | 79.3 96.5 | 99.5 119 | 103 98.5 | 102.6 | 13.4 |
| 0.05 | 97.0 99.8 | 81.9 89.1 | 90.1 98.4 | 86.5 84.7 | 90.9 | 6.7 |
| 0.5 | 94.6 95.8 | 85.7 88.3 | 92.9 95.8 | 78.8 79.8 | 89.0 | 7.0 |
| 5 | 81.3 86.3 87.3 | 83.1 87.7 84.3 | 83.1 92.9 | 79.6 77.8 73.2 | 83.3 | 5.4 |
| paraoxon-methyl | | | | | | |
| 0.02 | 126 126 | 94.5 115 | 116 128 | 117 64 | 110.8 | 21.7 |
| 0.05 | 119 102 | 93.8 91 | 114 96.2 | 72.8 113 | 100.2 | 15.1 |
| 0.5 | 108 105 | 97.4 89.2 | 104 99 | 72 76.4 | 93.9 | 13.5 |
| 5 | 109 117 114 | 92.4 102 97.4 | 95.4 106 | 82.2 79.2 81.6 | 97.8 | 13.1 |

Deyrup and Cassidy (1992) described a general GLC method for determining parathion-methyl and paraoxon-methyl in numerous substrates which requires reflux in acidic aqueous methanol for one hour, rotary evaporation of the methanol and partition clean-up with ethyl acetate followed by GLC analysis of the concentrated extract. Some changes to the method are required for certain substrates, e.g. a gel permeation chromatography clean-up step is necessary for oily samples. The method was applied to the analysis of numerous substrates in three laboratories. Recoveries were generally 70-120%, with typical LOQs of 0.05 mg/kg. The most difficult samples were soya bean soapstock, onions, clover and grass.

The method was tested on alfalfa seed, artichoke heads, bluegrass hay, broccoli, brown rice, bulb onion, cabbage heads, carrots, celery stalks, clover forage, cotton seed, cotton seed crude oil, cotton seed hulls, cotton seed meal, cotton seed refined oil, cotton seed soapstock, dry bean forage, dry bean hay, dry bean seeds, dry pea forage, dry pea seed, dry pea straw, fescue grass hay, field corn expeller oil, field corn flour, field corn fodder, field corn forage, field corn grain, field corn grits, field corn meal, field corn refined oil, field corn silage, field corn starch, green onion, head lettuce, leaf lettuce, lima bean pods, low grade flour, mustard greens, polished rice, potato chips, potato dried peel, potato granules, potato tubers, potato wet peel, rice bran, rice grain, rice hulls, rice straw, snap bean cannery waste, snapbean pods, sorghum fodder, sorghum forage, sorghum hay, sorghum grain, sorghum silage, soya bean crude oil, soya bean hulls, soya bean meal, soya bean refined oil, soya bean seed, soya bean soapstock, spinach greens, succulent pea forage, succulent pea vine, sugar beet dehydrated pulp, sugar beet fodder, sugar beet molasses, sugar beet refined sugar, sugar beet roots, sunflower seed, sweet corn ears, sweet corn fodder, sweet corn forage, turnip root, wheat bran, wheat

forage, wheat grain, wheat hay, wheat middlings, wheat patent flour, wheat red dog, wheat rough, wheat shorts and wheat straw.

Parathion-methyl is included in the multiresidue GLC analytical method for non-fatty and fatty foods published in the Official Methods of Analysis in The Netherlands (Netherlands, 1996). Parathion-methyl is detected with an ion-trap or nitrogen-phosphorus detector. The LOQ is 0.05 mg/kg.

**Stability of pesticide residues in stored analytical samples**

The Meeting received information on the freezer storage stability of parathion-methyl and paraoxon-methyl in a range of crops, processed commodities and soils.

Wassell and Gilles (1991) tested storage stability in a range of homogenized commodities which were weighed into individual glass jars, fortified and stored at -20°C for up to 2 years. At intervals samples were withdrawn for analysis and determination of procedural recoveries (Table 12).

Table 12. Stability at -20°C of parathion-methyl and paraoxon-methyl in samples fortified at 1 mg/kg (Wassell and Gilles, 1991).

| Storage, months | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | paraoxon-methyl | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov |
| | snap bean seed and pod | | | | dry bean seed | | | | celery | | | |
| 0 | 84 82 | 79 96 | 83 74 | 85 93 | 94 93 | 92 94 | 95 97 | 101 93 | 100 101 | 102 93 | 100 98 | 100 93 |
| 1 | 103 106 | 97 106 | 96 101 | 93 101 | 98 79 | 85 67 | 76 61 | 77 56 | 83 86 | 107 | 89 93 | 83 |
| 2 | 84 77 | 68 74 | 74 76 | 69 76 | 85 98 | 85 65 | 83 94 | 86 42 | 96 107 | 94 74 | 95 97 | 88 88 |
| 3 | 80 84 | 67 91 | 77 82 | 81 90 | | | | | 99 78 | 99 85 | 97 76 | 98 88 |
| 4 | 86 92 | 93 99 | 91 89 | 90 98 | 75 84 | 64 85 | 76 81 | 63 79 | 102 106 | 105 109 | 102 105 | 92 98 |
| 6 | 81 86 | 94 97 | 90 94 | 97 107 | 73 77 | 81 78 | 75 77 | 84 85 | 80 80 | 86 87 | 81 85 | 102 95 |
| 12 | 85 93 | 95 91 | 85 91 | 94 96 | 96 98 | 90 93 | 91 89 | 91 91 | 77 77 | 75 86 | 96 93 | 92 110 |
| 18 | 91 90 | 82 73 | 95 97 | 91 82 | 94 109 | 102 101 | 84 93 | 91 88 | 100 104 | 98 105 | 93 95 | 90 101 |
| 24 | 92 95 | 101 89 | 96 97 | 112 94 | 90 100 | 99 101 | 92 100 | 112 113 | 104 119 | 98 113 | 98 115 | 104 116 |
| | maize forage | | | | maize fodder | | | | maize grain | | | |
| 0 | 101 95 | 101 98 | 78 91 | 90 91 | 86 77 | 89 94 | 73 66 | 84 76 | 85 65 | 78 72 | 106 96 | 110 102 |
| 1 | 91 99 | 85 96 | 94 92 | 81 90 | 110 106 | 108 117 | 84 85 | 86 84 | 88 82 | 81 96 | 98 87 | 93 101 |
| 2 | 80 121 | 82 88 | 82 112 | 88 92 | 78 92 | 56 82 | 82 90 | 58 88 | 94 112 | 101 128 | 106 107 | 97 117 |
| 3 | | | | | 90 91 | 81 80 | 87 82 | 81 85 | 79 62 | 89 86 | 89 89 | 103 98 |
| 4 | 78 82 | 80 81 | 77 80 | 75 76 | 87 83 | 85 89 | 92 86 | 85 86 | | | | |
| 6 | 82 80 | 97 95 | 91 93 | 101 99 | 78 68 | 82 96 | 80 71 | 84 102 | 72 74 | 69 83 | 100 96 | 91 104 |
| 12 | 98 88 | 103 86 | 101 94 | 104 85 | 82 85 | 81 100 | 75 80 | 85 82 | 64 62 | 62 71 | 90 91 | 86 100 |
| 18 | 101 103 | 97 83 | 90 98 | 91 81 | 91 95 | 94 96 | 91 99 | 90 92 | 69 83 | 94 89 | 87 103 | 97 100 |
| 24 | 91 81 | 91 91 | 86 75 | 89 97 | 93 93 | 93 80 | 91 89 | 91 81 | 122 104 | 116 107 | 128 115 | 130 130 |
| | turnip root | | | | turnip top | | | | | | | |
| 0 | 85 90 | 92 91 | 84 88 | 91 90 | 82 87 | 88 87 | 88 91 | 95 95 | | | | |
| 1 | 89 76 | 89 87 | 79 73 | 81 79 | 92 90 | 94 93 | 88 81 | 84 90 | | | | |
| 2 | 96 93 | 87 89 | 95 80 | 84 78 | 91 94 | 86 74 | 88 77 | 79 71 | | | | |
| 3 | 96 94 | 94 74 | 79 84 | 91 76 | 68 79 | 73 84 | 68 78 | 73 81 | | | | |
| 4 | 96 93 | 94 92 | 71 80 | 91 90 | 115 87 | 81 86 | 119 96 | 89 98 | | | | |
| 6 | 79 87 | 88 102 | 79 91 | 86 100 | 77 84 | 85 60 | 83 92 | 88 92 | | | | |
| 12 | 84 84 | 82 93 | 79 79 | 84 97 | 83 84 | 89 87 | 92 89 | 97 99 | | | | |
| 18 | 88 80 | 65 79 | 100 76 | 66 75 | 82 98 | 71 73 | 79 92 | 69 70 | | | | |
| 24 | 92 102 | 109 109 | 83 91 | 114 114 | 95 99 | 114 102 | 90 94 | 123 110 | | | | |

% rem: % remaining after storage% recov: % procedural recovery

Gillard (1992) tested stability in other commodities using similar methods (Table 13).

Table 13. Stability at -20°C of parathion-methyl and paraoxon-methyl in homogenized samples fortified at 1 mg/kg (Gillard, 1992).

| Storage, months | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | paraoxon-methyl | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov |
| | lettuce | | | | wheat straw | | | | mustard | | | |
| 0 | 98 97 | | 118 123 | | 112 107 | | 132 121 | | 110 106 | | 131 125 | |
| 1 | 109 110 | 109 110 | 100 101 | 117 120 | 92 92 | 101 96 | 116 114 | 128 122 | 105 101 | 102 115 | 109 108 | 109 123 |
| 2 | 106 104 | 106 104 | 93 88 | 113 112 | 104 108 | 100 92 | 91 95 | 93 88 | 92 96 | 103 100 | 86 89 | 102 98 |
| 3 | 90 86 | 89 88 | 78 72 | 112 108 | 112 98 | 112 108 | 133 114 | 137 131 | 91 91 | 85 89 | 97 98 | 99 104 |
| 4 | 96 96 | 97 96 | 81 81 | 110 110 | 99 98 | 104 87 | 109 108 | 122 103 | 84 85 | 82 89 | 85 83 | 88 90 |
| 6 | 91 78 | 76 98 | 90 76 | 97 123 | 92 94 | 92 89 | 104 109 | 110 105 | 82 86 | 73 91 | 86 89 | 82 92 |
| 12 | 83 87 | 88 91 | 60 62 | 103 108 | 87 101 | 95 93 | 82 95 | 103 99 | 78 76 | 78 85 | 81 78 | 93 99 |
| 18 | 92 88 | 92 95 | 51 45 | 107 110 | 93 92 | 106 107 | 77 78 | 104 107 | 80 88 | 100 100 | 70 78 | 107 109 |
| 25 | 82 83 | 86 93 | 58 62 | 103 145 | 71 84 | 81 93 | 72 93 | 108 123 | 82 77 | 95 95 | 83 82 | 131 119 |
| | wheat forage | | | | wheat grain | | | | sunflower seed | | | |
| 0 | 108 107 | | 127 127 | | 96 91 | | 97 97 | | 100 91 | | 117 108 | |
| 1 | 96 95 | 91 94 | 122 121 | 117 122 | 87 77 | 90 70 | 117 114 | 126 119 | 86 84 | 91 116 | 85 82 | 101 120 |
| 2 | 98 114 | 113 107 | 89 106 | 109 104 | 90 95 | 102 101 | 78 86 | 98 98 | 84 76 | 78 86 | 88 82 | 91 96 |
| 3 | 88 102 | 104 113 | 110 120 | 129 134 | 65 77 | 101 110 | 99 108 | 117 126 | 96 96 | 100 99 | 106 105 | 124 118 |
| 4 | 87 92 | 93 102 | 105 99 | 108 119 | 65 62 | 91 74 | 92 99 | 116 104 | 100 103 | 104 97 | 87 103 | 92 100 |
| 6 | 96 | 96 94 | 110 | 118 120 | 67 73 | 77 76 | 98 105 | 119 114 | 85 99 | 91 92 | 88 94 | 106 114 |
| 12 | 95 98 | 110 105 | 88 95 | 110 108 | 77 95 | 100 80 | 76 85 | 108 110 | 84 83 | 100 89 | 78 77 | 127 112 |
| 18 | 101 96 | 105 98 | 94 92 | 115 110 | 84 92 | 83 104 | 72 74 | 89 114 | 79 86 | 75 73 | 103 102 | 104 105 |
| 24 | 96 87 | 91 84 | 125 118 | 118 108 | 91 90 | 88 84 | 89 110 | 108 77 | 54 60 | 90 89 | 68 58 | 121 124 |
| | onion | | | | | | | | | | | |
| 0 | 117 117 | | 122 122 | | | | | | | | | |
| 1 | 100 103 | 104 106 | 98 101 | 106 107 | | | | | | | | |
| 2 | 93 81 | 83 67 | 102 99 | 104 88 | | | | | | | | |
| 3 | 94 90 | 94 102 | 92 87 | 97 109 | | | | | | | | |
| 4 | 86 87 | 88 93 | 74 78 | 84 88 | | | | | | | | |
| 6 | 98 94 | 94 93 | 107 101 | 108 113 | | | | | | | | |
| 12 | 84 82 | 86 90 | 86 83 | 95 99 | | | | | | | | |
| 18 | 88 84 | 90 87 | 100 93 | 121 128 | | | | | | | | |
| 24 | 74 70 | 83 81 | 61 57 | 84 83 | | | | | | | | |

Owen (1995) tested the stability of parathion-methyl and paraoxon-methyl in canola seed, oil and meal (Table 14). 10 g subsamples were fortified with the compounds at 1 mg/kg in 250 ml bottles and stored in a freezer at a temperature below -5°C. Bottles were withdrawn periodically for analysis and procedural recoveries were determined. There was no evidence of the conversion of parathion-methyl to paraoxon-methyl during storage. Parathion-methyl was stable in the three substrates. Paraoxon-methyl was stable in the oil, but the estimated times for a 30% decrease were 5 months in the seed and 7 in the meal.

Table 14. Stability at -5°C of parathion-methyl and paraoxon-methyl in canola and its processed commodities fortified at 1 mg/kg (Owen, 1995).

| Storage, months | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | paraoxon-methyl | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov |
| | seed | | | | crude oil | | | | meal | | | |
| 0 | 80 89 | 86 93 | 74 79 | 78 83 | 97 98 | 96 98 | 101 103 | 99 101 | 81 84 | 73 87 | 83 84 | 75 86 |
| 1 | 105 99 | 99 103 | 81 74 | 102 102 | 97 96 | 100 98 | 101 100 | 103 99 | 100 86 | 96 94 | 99 89 | 104 102 |
| 3 | 99 101 | 108 101 | 63 75 | 109 104 | 82 88 | 94 92 | 67 85 | 91 77 | 79 80 | 89 83 | 62 63 | 80 73 |
| 5 | 91 95 | 91 99 | 53 58 | 83 87 | 100 99 | 100 110 | 87 82 | 88 97 | 100 97 | 95 93 | 85 81 | 97 96 |
| 6 | 88 87 | 89 91 | 49 47 | 76 79 | 85 84 | 90 5 | 67 65 | 72 74 | 71 82 | 85 86 | 54 61 | 79 77 |
| 14 | 69 69 | 78 71 | 28 34 | 80 71 | 82 99 | 94 81 | 48 96 | 65 64 | 67 64 | 87 87 | 43 41 | 78 80 |

Davis (1992) tested the stability of parathion-methyl and paraoxon-methyl in vegetables and forages (Table 15). 25 g subsamples were weighed into individual glass jars, fortified with the analytes at a nominal 1 mg/kg and stored in a freezer between -25°C and -20°C for up to 2 years. Jars were periodically removed for analysis and procedural recoveries checked. Parathion-methyl was stable in all samples for 2 years, and paraoxon-methyl also in all samples except succulent pea pods where stability was marginal: the results suggested a 30% decrease over about 20 months.

Table 15. Stability at -25°C to -20°C of parathion-methyl and paraoxon-methyl in homogenized samples fortified at 1 mg/kg (Davis, 1992).

| Storage, months | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | paraoxon-methyl | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov | % rem | % recov |
| | head cabbage | | | | bluegrass hay | | | | dry pea seed | | | |
| 0 | 87 87 | 88 89 | 90 90 | 91 92 | 97 87 | 83 100 | 97 87 | 83 102 | 92 90 | 89 91 | 82 79 | 78 78 |
| 1 | 77 92 | 75 92 | 77 88 | 75 93 | 72 70 | 71 76 | 69 67 | 70 73 | 89 90 | 94 80 | 79 80 | 95 80 |
| 2 | 98 94 | 119 116 | 95 93 | 122 119 | 81 79 | 94 90 | 77 75 | 96 91 | 78 74 | 94 94 | 80 73 | 101 100 |
| 3 | 65 68 | 88 87 | 66 68 | 91 92 | 93 91 | 75 82 | 91 91 | 77 85 | 71 75 | 77 82 | 68 70 | 79 81 |
| 4 | 87 87 | 88 88 | 85 82 | 89 88 | 95 98 | 90 89 | 108 108 | 102 102 | 71 80 | 82 77 | 70 75 | 81 80 |
| 6 | 92 81 | 95 91 | 94 87 | 103 101 | 88 86 | 93 93 | 88 87 | 99 96 | 72 75 | 92 95 | 70 73 | 92 96 |
| 12 | 94 86 | 89 93 | 98 90 | 110 107 | 100 96 | 87 85 | 98 101 | 97 97 | 81 75 | 78 79 | 76 70 | 88 84 |
| 18 | 108 83 | 87 93 | 105 84 | 102 98 | 92 96 | 91 90 | 103 103 | 95 99 | 84 88 | 84 90 | 77 86 | 93 102 |
| 24 | 89 88 | 88 92 | 87 77 | 88 97 | 85 95 | 73 71 | 78 86 | 82 76 | 80 86 | 95 90 | 77 83 | 98 101 |
| | dry pea straw | | | | succulent pea pod | | | | succulent pea forage | | | |
| 0 | 85 81 | 80 85 | 86 82 | 80 88 | 79 73 | 75 76 | 84 81 | 82 81 | 86 87 | 84 82 | 90 90 | 85 84 |
| 1 | 87 85 | 92 92 | 87 85 | 92 94 | 80 77 | 84 87 | 78 86 | 97 102 | 85 75 | 85 95 | 94 83 | 104 115 |
| 2 | 85 83 | 93 96 | 95 94 | 103 107 | | | | | | | | |
| 3 | 75 79 | 81 80 | 73 77 | 80 79 | 80 80 | 80 87 | 69 72 | 94 94 | 79 81 | 86 89 | 81 83 | 89 95 |
| 4 | 67 67 | 80 86 | 71 71 | 81 87 | 76 73 | 86 85 | 66 66 | 82 83 | 81 81 | 90 88 | 80 78 | 88 87 |
| 6 | 81 79 | 102 115 | 85 84 | 106 119 | 77 76 | 81 80 | 64 62 | 84 84 | 85 89 | 84 85 | 80 85 | 91 96 |
| 12 | 86 81 | 92 92 | 83 77 | 92 89 | 75 75 | 80 81 | 60 56 | 84 83 | 84 82 | 73 83 | 72 75 | 75 88 |
| 18 | 88 77 | 93 102 | 84 75 | 98 102 | 83 82 | 90 89 | 70 68 | 104 102 | 85 89 | 93 94 | 84 85 | 104 102 |
| 24 | 87 94 | 98 97 | 76 87 | 95 97 | 68 76 | 87 84 | 47 44 | 87 78 | 88 89 | 93 87 | 76 80 | 105 99 |
| | clover forage | | | | soya bean seed | | | | | | | |
| 0 | 91 91 | 92 89 | 95 95 | 97 94 | 63 69 | 64 78 | 89 86 | 89 87 | | | | |
| 1 | 83 81 | 88 78 | 93 90 | 106 92 | 71 71 | 64 78 | 77 72 | 74 84 | | | | |
| 2 | 86 87 | 96 99 | 81 81 | 96 101 | 71 67 | 70 71 | 61 57 | 67 66 | | | | |
| 3 | 89 85 | 93 97 | 89 86 | 102 105 | 70 73 | 80 80 | 60 64 | 80 82 | | | | |
| 4 | 85 87 | 103 99 | 80 82 | 107 102 | 59 69 | 79 75 | 49 54 | 76 73 | | | | |
| 6 | 97 87 | 98 100 | 93 86 | 116 118 | 82 85 | 82 80 | 63 62 | 78 83 | | | | |
| 12 | 83 91 | 82 89 | 70 74 | 87 91 | 72 63 | 64 67 | 53 52 | 69 73 | | | | |
| 18 | 107 85 | 93 106 | 104 77 | 107 118 | 98 84 | 82 93 | 61 54 | 89 101 | | | | |
| 24 | 100 117 | 115 115 | 68 85 | 117 116 | 98 100 | 96 108 | 56 61 | 93 96 | | | | |

Rice *et al* (1990a) tested the stability of parathion-methyl and paraoxon-methyl in a fortified sandy loam soil (0.2% organic matter, pH 6.2, 77% sand, 17% silt, 6% clay). 20 g samples in polyethylene bottles were fortified with 0.20 mg/kg of either parathion-methyl or paraoxon-methyl and stored at -20°C for 12 months. Samples were withdrawn at intervals and analysed, and procedural recoveries were determined. The results are shown in Table 16. Parathion-methyl was stable under these conditions, and no paraoxon-methyl (LOQ 0.05 mg/kg) was detected in the parathion-methyl samples. Paraoxon-methyl showed a half-life of about 485 days, equivalent to a 30% decrease in 250 days.

Table 16. Stability of parathion-methyl and paraoxon-methyl in fortified sandy loam soil stored for 12 months at -20°C (Rice *et al*, 1990a).

| Day | Parathion-methyl, mg/kg | | | | Paraoxon-methyl, mg/kg | | | |
|---|---|---|---|---|---|---|---|---|
| | stored | | procedural recoveries | | stored | | procedural recoveries | |
| 0 | 0.200 | 0.210 | 0.223 | 0.216 | 0.200 | 0.181 | 0.213 | 0.208 |
| 7 | 0.199 | 0.208 | 0.194 | 0.203 | 0.214 | 0.203 | 0.196 | 0.212 |
| 14 | 0.210 | 0.204 | 0.212 | 0.209 | 0.204 | 0.207 | 0.212 | 0.214 |
| 30 | 0.234 | 0.202 | 0.234 | 0.202 | 0.180 | 0.179 | 0.194 | 0.159 |
| 61 | 0.203 | 0.164 | 0.203 | 0.18 | 0.191 | 0.199 | 0.209 | 0.185 |
| 91 | 0.171 | 0.155 | 0.164 | 0.165 | 0.156 | 0.151 | 0.153 | 0.168 |
| 182 | 0.189 | 0.193 | 0.224 | 0.205 | 0.151 | 0.167 | 0.215 | 0.205 |
| 273 | 0.182 | 0.195 | 0.194 | 0.185 | 0.144 | 0.146 | 0.185 | 0.182 |
| 365 | 0.180 | 0.181 | 0.186 | 0.165 | 0.116 | 0.109 | 0.186 | 0.178 |

Rice *et al* (1990b) also tested the freezer storage stability of parathion-methyl and paraoxon-methyl in a fortified loam soil (0.7% organic matter, pH 5.4, 30% sand, 46% silt, 24% clay) using the same procedures. Parathion-methyl was stable during the 12 months, but paraoxon-methyl disappeared with an estimated half-life of 42 days or a time of 22 days for a 30% decrease (Table 17).

Table 17. Stability of parathion-methyl and paraoxon-methyl in fortified loam soil stored for 12 months at -20°C (Rice *et al.*, 1990b).

| Day | Parathion-methyl, mg/kg | | | | Paraoxon-methyl, mg/kg | | | |
|---|---|---|---|---|---|---|---|---|
| | stored | | procedural recoveries | | stored | | procedural recoveries | |
| 0 | 0.238 | 0.235 | 0.236 | 0.239 | 0.225 | 0.224 | 0.229 | 0.239 |
| 7 | 0.169 | 0.185 | 0.189 | 0.187 | 0.142 | 0.118 | 0.187 | 0.188 |
| 14 | 0.206 | 0.207 | 0.227 | 0.218 | 0.166 | 0.142 | 0.221 | 0.214 |
| 30 | 0.183 | 0.177 | 0.216 | 0.192 | 0.101 | 0.086 | 0.214 | 0.189 |
| 61 | 0.150 | 0.175 | 0.171 | 0.152 | 0.059 | 0.048 | 0.174 | 0.159 |
| 91 | 0.179 | 0.175 | 0.173 | 0.167 | 0.049 | 0.045 | 0.172 | 0.171 |
| 182 | 0.183 | 0.166 | 0.158 | 0.195 | <0.05 | <0.05 | 0.167 | 0.209 |
| 273 | 0.182 | 0.161 | 0.183 | 0.195 | <0.05 | <0.05 | 0.181 | 0.197 |
| 365 | 0.160 | 0.159 | 0.15 | 0.169 | <0.05 | <0.05 | 0.159 | 0.185 |

**Definition of the residue**

Parathion-methyl and paraoxon-methyl are important components of the residue. In trials according to GAP to determine residues of parathion-methyl and paraoxon-methyl in food and feed commodities there were 54 and 155 cases respectively where the levels of both compounds exceeded the LOQ. The total residues were closely related to parathion-methyl levels in food and feed commodities, but with more spread in the relation at the lower concentrations.

The Meeting recommended that the definition of the residue for compliance with MRLs should continue to be parathion-methyl and for the estimation of dietary intake should be the sum of parathion-methyl and paraoxon-methyl expressed as parathion-methyl (parathion-methyl + 1.065 × paraoxon-methyl).

The log $P_{ow}$ of 2.8 suggests that parathion-methyl should not be classified as fat-soluble. The animal metabolism studies provide limited information on the fat-solubility of parathion-methyl

because it is metabolized quickly. Parathion-methyl was not identified as a residue in tissues or milk in the goat metabolism study. In the laying-hen study parathion-methyl residues in fat were higher than in the other tissues, except skin, suggesting some fat-solubility. The Meeting decided that the evidence of fat-solubility was not sufficiently clear to describe parathion-methyl as fat-soluble.

## USE PATTERN

Parathion-methyl is registered in many countries for the control of insects on fruit, vegetables, cereals, oilseeds and forage crops. The information available to the Meeting on registered uses is summarized in Table 18.

Table 18. Registered uses of parathion-methyl.

| Crop | Country | Form | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray conc., kg ai/hl | No. | |
| Agric and horti crops | Netherlands | EC | soil treatment | 2.6 | | 1 | |
| Alfalfa | Hungary | CS 450 g/l | foliar | 0.45 | | | 14 |
| Alfalfa | Hungary | EC 480 g/l | foliar | 0.24-0.34 | | | 14 |
| Alfalfa | USA | EC 480 g/l | foliar | 0.28-1.1 | | | 15 |
| Apple | Australia | ME 240 | foliar | - | 0.03 | note [1] | 14 |
| Apple | Netherlands | EC | foliar | 0.24-0.36 | | | 21 |
| Apple | Poland | CS 450 g/l | foliar high volume | 0.32-0.58 | 0.032-0.12 | 2 | 42 |
| Banana | Spain | CS 450 g/l | foliar | 0.68-1.76 | 0.045-0.059 | | 21 |
| Barley | Germany | WP 405 g/kg | bait | 0.18 | | 1 | |
| Barley | USA | EC 480 g/l | foliar | 0.28-0.84 | | | 15 |
| Beans | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Beans | Italy | EC 150 g/l | foliar | | 0.023-0.030 | | 20 |
| Beans, dry | USA | EC 480 g/l | foliar | 0.56-1.7 | | | 21[3] |
| Berries | Hungary | CS 450 g/l | foliar | 0.45 | | | 14 |
| Berries | Hungary | EC 480 g/l | foliar | 0.48 | | | 14 |
| Cabbage | France | EC 400 g/l | foliar | 0.3 | | | 15 |
| Cabbage | Italy | CS 170 g/l | foliar | 0.32-0.41 | | | 20 |
| Cabbage | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.075-0.23 | 2 | 35 |
| Cabbage | USA | EC 480 g/l | foliar | 0.56-1.7 | | | 21[4] |
| Capsicums (Sweet peppers) | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Carrots | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Cereals | Hungary | CS 450 g/l | foliar | 0.45 | | | 14 |
| Cereals | Hungary | EC 480 g/l | foliar | 0.24-0.34 | | | 14 |
| Cereals | Netherlands | EC | foliar | 0.36 | | | 21 |
| Citrus | Australia | ME 240 | foliar | - | 0.03 | note [5] | 14 |
| Citrus fruits | Guatemala | | foliar | | 0.09 | | 14 |
| Citrus fruits | Italy | CS 170 g/l | foliar | | 0.041-0.049 | | 20 |
| Citrus fruits | Italy | EC 150 g/l | foliar | | 0.023-0.038 | | 20 |
| Clover seed crops | Australia | EC 500 g/l | foliar | 0.4 | | | 14 |
| Cotton | Australia | EC 500 g/l | foliar | 0.28-1.1 | | | 14 |
| Cotton | Australia | ME 450 | foliar | 0.34-1.4 | - | | 14 |
| Cotton | Guatemala | CS 450 g/l | foliar | 0.45 | | | 7 |

[1] Apples and pears - apply as determined by trap counts at minimum interval of 2 weeks.

[2] Apply as pest appears or at 10-14 days intervals.

[3] Dry beans, USA. PHI 15 days for rates up to 0.56 kg ai/ha, 21 days for higher rates.

[4] Cabbage USA. PHI 10 days for rates up to 0.56 kg ai/ha, 21 days for higher rates.

[5] Apply at minimum intervals of 3 weeks.

| Crop | Country | Form | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray conc., kg ai/hl | No. | |
| Cotton | Pakistan | EC 500 g/l | foliar | 0.43-1.2 | | | 14-21 |
| Cotton | Spain | 2% dust | foliar | 0.40-0.60 | | 2 | 21 |
| Cotton | Spain | CS 450 g/l | foliar | 0.13-0.29 | 0.045-0.059 | | 21 |
| Cotton | Spain | EC 200 g/l | foliar | 0.048-0.18 | 0.016-0.036 | | 21 |
| Cotton | Thailand | EC 500 g/l | foliar high volume | 0.13-0.25 | 0.025-0.05 | | 14 |
| Cotton | USA | EC 480 g/l | foliar | 0.15-3.4 | | | 0 or 5[6] |
| Cruciferous crops | Guatemala | CS 450 g/l | foliar | 0.34-0.45 | | | 21 |
| Crucifers (Forage) | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Cucurbits | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Cucurbits | Guatemala | CS 450 g/l | foliar | 0.45 | | | |
| Egg plant | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Fibrous flax | Netherlands | EC | foliar | 0.36 | | | 21 |
| Field crops | Lebanon | CS 450 g/l | foliar | | 0.045-0.068 | | |
| Fodder beet | Netherlands | EC | foliar | 0.15-0.48 | | | 21 |
| Fruit trees | Lebanon | CS 450 g/l | foliar | | 0.045-0.068 | | |
| Fruiting vegetables | Guatemala | CS 450 g/l | foliar | 0.45 | | | 15 |
| Fruiting vegetables | Italy | CS 170 g/l | foliar | 0.32-0.41 | | | 20 |
| Grapefruit | Australia | EC 500 | foliar | - | 0.01-0.05 | | 14 |
| Grapefruit | Australia | ME 450 | foliar | - | 0.01-0.05 | note [5] | 14 |
| Grapes | Australia | EC 500 | foliar | - | 0.03 | | 14 |
| Grapes | Australia | ME 240 | foliar | - | 0.03 | note [5] | 14 |
| Grapes | Italy | CS 170 g/l | foliar | | 0.032-0.041 | | 20 |
| Grapes | Italy | EC 150 g/l | foliar | | 0.023-0.030 | | 20 |
| Grapes, wine and table | Spain | CS 450 g/l | foliar | | 0.045-0.059 | | 21 |
| Grass (forage) | USA | EC 480 g/l | foliar | 0.56-0.84 | | | 15[7] |
| Horse radish | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.075-0.23 | 2 | 35 |
| Leafy vegetables | Italy | CS 170 g/l | foliar | 0.32-0.41 | | | 20 |
| Leek | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.075-0.23 | 2 | 14 |
| Legume vegetables | Guatemala | CS 450 g/l | foliar | 0.45 | | | 15 |
| Lemons | Australia | EC 500 | foliar | - | 0.01-0.05 | | 14 |
| Lemons | Australia | ME 450 | foliar | - | 0.01-0.05 | note [5] | 14 |
| Lettuce | France | EC 400 g/l | foliar | 0.3 | | | 15 |
| Linseed | Netherlands | EC | foliar | 0.36 | | | 21 |
| Maize | Guatemala | CS 450 g/l | foliar | 0.34 | | | 12 |
| Maize | Hungary | CS 450 g/l | foliar | 0.45-0.68 | | | 14 |
| Maize | Hungary | EC 480 g/l | foliar | 0.72 | | | 14 |
| Maize | Netherlands | EC | foliar | 0.48 | | | 21 |
| Maize | Spain | CS 450 g/l | foliar | 0.13-0.29 | 0.045-0.059 | | 21 |
| Maize | USA | EC 480 g/l | foliar | 0.28-0.56 | | | 12 |
| Mandarins | Australia | EC 500 | foliar | - | 0.01-0.05 | | 14 |
| Mandarins | Australia | ME 450 | foliar | - | 0.01-0.05 | note [5] | 14 |
| Oats | Germany | WP 405 g/kg | bait | 0.18 | | 1 | |
| Oats | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.11-0.30 | 2 | 21 |
| Oats | USA | EC 480 g/l | foliar | 0.28-0.84 | | | 15 |
| Oilseed rape | France | EC 400 g/l | foliar | 0.2-0.3 | | | 21 |
| Oilseed rape | France | EC 400 g/l | foliar | 0.2-0.3 | | | 15 |
| Oilseed rape | Poland | CS 450 g/l | foliar high volume | 0.22-0.45 | 0.055-0.45 | 2 | 42 |
| Olives | Italy | CS 170 g/l | foliar | | 0.041-0.049 | | 20 |
| Onion | Hungary | CS 450 g/l | foliar | 0.45 | | | 14 |

[6] Cotton, USA. PHI 5 days for hand picking, 0 days for mechanical picking.

[7] Grass, USA. PHI 15 days applies to cutting or grazing.

| Crop | Country | Form | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray conc., kg ai/hl | No. | |
| Onion | Hungary | EC 480 g/l | foliar | 0.72 | | | 14 |
| Onion | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.075-0.23 | 2 | 14 |
| Onion | USA | EC 480 g/l | foliar | 0.56 | | | 15 |
| Onions | Netherlands | EC | foliar | 0.36 | | | 21 |
| Oranges | Australia | EC 500 | foliar | - | 0.01-0.05 | | 14 |
| Oranges | Australia | ME 450 | foliar | - | 0.01-0.05 | note [5] | 14 |
| Orchards | Pakistan | EC 500 g/l | foliar | | 0.05-0.075 | | 14-21 |
| Paprika | Hungary | CS 450 g/l | foliar | 0.45 | | | 14 |
| Paprika | Hungary | EC 480 g/l | foliar | 0.72 | | | 14 |
| Peach | France | EC 400 g/l | foliar | | 0.03 | | 15 |
| Peanut | Spain | CS 450 g/l | foliar | 0.13-0.29 | 0.045-0.059 | | 21 |
| Peanut | Thailand | EC 500 g/l | foliar high volume | 0.63-0.78 | 0.1-0.13 | | 14 |
| Pear | Netherlands | EC | foliar | 0.24-0.36 | | | 21 |
| Pear | Poland | CS 450 g/l | foliar high volume | 0.32-0.58 | 0.032-0.12 | 2 | 42 |
| Pears | Australia | ME 240 | foliar | - | 0.03 | note [1] | 14 |
| Peas | France | EC 400 g/l | foliar | 0.3 | | | 15 |
| Peas | Hungary | CS 450 g/l | foliar | 0.45 | | | 14 |
| Peas | Hungary | EC 480 g/l | foliar | 0.34 | | | 14 |
| Peas | Netherlands | EC | foliar | 0.24-0.36 | | | 21 |
| Peas (Green/Garden) | Australia | EC 500 | foliar | 0.35-0.55 | - | | 14 |
| Peas, dry | USA | EC 480 g/l | foliar | 0.56-1.1 | | | 15[8] |
| Pome fruits | Australia | EC 500 g/l | dormant spray | | 0.05 | | |
| Pome fruits | Australia | EC 500 g/l | foliar | | 0.02-0.033 | | 14 |
| Pome fruits | Australia | ME 450 | foliar | - | 0.02-0.05 | | 14 |
| Pome fruits | France | EC 400 g/l | foliar | | 0.03 | | 15 |
| Pome fruits | Germany | WP 405 g/kg | foliar | note[9] | 0.02 | 1 | 28 |
| Pome fruits | Hungary | CS 450 g/l | foliar | 0.45 | | | 14 |
| Pome fruits | Hungary | EC 480 g/l | foliar | 0.48-0.86 | | | 14 |
| Pome fruits | Italy | CS 170 g/l | foliar | | 0.032-0.041 | | 20 |
| Pome fruits | Italy | EC 150 g/l | foliar + mineral oil | | 0.023- 0.045 | | 20 |
| Pome fruits | Spain | CS 450 g/l | foliar | | 0.045-0.059 | | 21 |
| Pome fruits | Spain | EC 200 g/l | foliar | 0.16-0.54 | 0.016-0.036 | | 21 |
| Pome fruits | Spain | 3% EC + oil | foliar or dormant | 0.48-1.35 | 0.060-0.090 | 1 | 21 |
| Potato | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Potato | Lebanon | CS 450 g/l | foliar | | 0.045-0.068 | | |
| Potato | Poland | CS 450 g/l | foliar high volume | 0.45-0.68 | 0.11-0.45 | 2 | 48 |
| Potato | USA | EC 480 g/l | foliar | 1.7 | | | 6 |
| Rape seed | Netherlands | EC | foliar | 0.48 | | | 21 |
| Rape seed, canola | USA | EC 480 g/l | foliar | 0.56 | | 2 | 28 |
| Rice | USA | EC 480 g/l | foliar | 0.56-0.84 | | | 15 |
| Rye | Germany | WP 405 g/kg | bait | 0.18 | | 1 | |
| Rye | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.11-0.30 | 2 | 21 |
| Rye | USA | EC 480 g/l | foliar | 0.28-0.84 | | | 15 |
| Soya bean | Thailand | EC 500 g/l | foliar high volume | 0.63-0.78 | 0.1-0.13 | | 14 |
| Soya bean | USA | EC 480 g/l | foliar | 0.43-1.1 | | | 20[10] |
| Spring barley | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.11-0.30 | 2 | 21 |
| Spring triticale | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.11-0.30 | 2 | 21 |
| Spring wheat | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.11-0.30 | 2 | 21 |
| Stone fruits | Australia | EC 500 g/l | dormant spray | | 0.05 | | |

[8] Dry peas, USA. PHI 10 days for rates up to 0.56 kg ai/ha, 15 days for higher rates.

[9] Pome fruit. Application 0.1 kg ai/ha per m height of tree crown, i.e. 0.3 kg ai/ha for standard tree of 3 m and 1500 l water/ha.

[10] Soya beans, USA. PHI 20 days also applies to grazing.

| Crop | Country | Form | Application | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Rate, kg ai/ha | Spray conc., kg ai/hl | No. | |
| Stone fruits | Australia | EC 500 g/l | foliar | | 0.02-0.033 | | 14 |
| Stone fruits | Australia | ME 450 | foliar | - | 0.02-0.05 | | 14 |
| Stone fruits | Hungary | CS 450 g/l | foliar | 0.45 | | | 14 |
| Stone fruits | Hungary | EC 480 g/l | foliar | 0.48-0.86 | | | 14 |
| Stone fruits | Italy | CS 170 g/l | foliar | | 0.032-0.041 | | 20 |
| Stone fruits | Italy | EC 150 g/l | foliar + mineral oil | | 0.023- 0.045 | | 20 |
| Stone fruits | Spain | CS 450 g/l | foliar | | 0.045-0.059 | | 21 |
| Stone fruits | Spain | EC 200 g/l | foliar | 0.16-0.54 | 0.016-0.036 | | 21 |
| Stone fruits | Spain | 3% EC + oil | foliar or dormant | 0.48-1.35 | 0.060-0.090 | 1 | 21 |
| Sugar beet | France | EC 400 g/l | foliar | 0.15-0.3 | | | 21 |
| Sugar beet | France | EC 400 g/l | foliar | 0.2-0.3 | | | 15 |
| Sugar beet | Hungary | CS 450 g/l | foliar | 0.45-0.68 | | | 14 |
| Sugar beet | Hungary | EC 480 g/l | foliar | 0.34-0.67 | | | 14 |
| Sugar beet | Italy | CS 170 g/l | foliar | 0.32-0.41 | | | 20 |
| Sugar beet | Netherlands | EC | foliar | 0.15-0.48 | | | 21 |
| Sugar beet | Spain | 2% dust | foliar | 0.40-0.60 | | 2 | 21 |
| Sugar beet | Spain | CS 450 g/l | foliar | 0.13-0.59 | 0.045-0.059 | | 21 |
| Sugar beet | Spain | EC 200 g/l | foliar | 0.048-0.36 | 0.016-0.036 | | 21 |
| Sugar beet | USA | EC 480 g/l | foliar | 0.28-0.43 | | | 20[11] |
| Sugar cane | Pakistan | EC 500 g/l | foliar | 0.43-0.62 | | | 14-21 |
| Sunflowers | USA | EC 480 g/l | foliar | 1.1 | | | 30 |
| Tomatoes | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Triticale | Germany | WP 405 g/kg | bait | 0.18 | | 1 | |
| Vegetables | Australia | EC 500 | foliar | 0.35 | 0.033 | note [2] | 14 |
| Vegetables | Lebanon | CS 450 g/l | foliar | | 0.045-0.068 | | |
| Vines | France | EC 400 g/l | foliar | 0.3 | | | 21 |
| Vines | Spain | 3% EC + oil | foliar or dormant | 0.18-0.45 | 0.060-0.090 | 1 | 21 |
| Watermelon | Spain | CS 450 g/l | foliar | 0.13-0.59 | 0.045-0.059 | | 21 |
| Wheat | Germany | WP 405 g/kg | bait | 0.18 | | 1 | |
| Wheat | USA | EC 480 g/l | foliar | 0.28-0.84 | | | 15 |
| Wine grapes | Germany | WP 405 g/kg | foliar | 0.081-0.16 | 0.02 | 1 | note[12] |
| Wine grapes | Germany | WP 405 g/kg | foliar | 0.081-0.32 | 0.02 | 2 note[13] | 35 |
| Winter barley | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.11-0.30 | 2 | 21 |
| Winter triticale | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.11-0.30 | 2 | 21 |
| Winter wheat | Poland | CS 450 g/l | foliar high volume | 0.45 | 0.11-0.30 | 2 | 21 |

## RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials on

| | | |
|---|---|---|
| Fruits | Table 20. | Apple, pear |
| | Table 21. | Peach |
| | Table 22. | Grapes |
| Vegetables | Table 23. | Onions |
| | Table 24. | Broccoli |
| | Table 25. | Cabbage |
| | Table 26. | Sweet corn |
| | Table 27. | Mustard greens |

[11] Sugar beet, USA. PHI of 60 days applies to tops used as animal fodder.

[12] Wine grapes. Use at beginning of infestation (vine leaf-roller, leaf gall miner), stage 12-57.

[13] Wine grapes. Not more than 1 application between flowering and harvest.

| | | |
|---|---|---|
| | Table 28. | Lettuce |
| | Table 29. | Spinach |
| | Table 30. | Lima beans, snap beans |
| | Table 31. | Soya beans |
| | Table 32. | Field peas |
| | Table 33. | Dried beans |
| | Table 34. | Carrots |
| | Table 35. | Potatoes |
| | Table 36. | Sugar beet |
| | Table 37. | Turnips |
| | Table 38. | Celery |
| | Table 39. | Artichokes |
| Cereals | Table 40. | Maize |
| | Table 41. | Rice |
| | Table 42. | Sorghum |
| | Table 43. | Wheat |
| Oilseeds | Table 44. | Cotton seed |
| | Table 45. | Canola, sunflower seed |
| Legume animal feeds | Table 46. | Alfalfa |
| | Table 47. | Clover |
| | Table 48. | Field pea forage, straw, vine |
| | Table 49. | Bean forage, hay |
| Cereal animal feeds | Table 50. | Sweet corn and maize fodder, forage, silage |
| | Table 51. | Sorghum fodder, forage, hay, silage |
| | Table 52. | Wheat forage, hay, straw |
| Grasses | Table 53. | Pasture grasses |
| Miscellaneous fodder | Table 54. | Sugar beet fodder, cotton gin trash |
| Dried hops | Table 55. | Hops |

Trials were generally well documented with full laboratory and field reports. Laboratory reports included method validation including batch recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses were also provided. Field reports provided data on the sprayers used and their calibration, plot size, residue sample size and sampling dates.

Where residues were not detected, data are recorded in the Tables as below the limit of quantification (LOQ), e.g. <0.01 mg/kg. Residue data, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOQ, to 1 significant figure. Although trials included control plots, no control data are recorded except where residues in control samples exceeded the LOQ. Residues are recorded unadjusted for procedural recoveries.

Conditions of the supervised trials are summarized in Table 19. Most trials were unreplicated or single replicate split plots. Where replicate residues are shown they represent samples from the split plots or replicate field samples from the same treated plot.

Periods of freezer storage between sampling and analysis were recorded for all trials and were within the acceptable determined stability period of 14 months or 2 years except in a few cases.

Table 19. Sprayers, plot sizes and field sample sizes in the supervised trials.

| Crop | Country | Year | Sprayer | Plot size | Sample size |
|---|---|---|---|---|---|
| Apple, pear | Germany | 1994 | knapsack | 6-10 trees | 1.8-7 kg |

| Crop | Country | Year | Sprayer | Plot size | Sample size |
|---|---|---|---|---|---|
| Apple | France | 1994-95 | motorised knapsack | 14 trees, 75-150 $m^2$ | 36 fruit, 1-4 kg |
| Peach | Italy | 1994 | knapsack or wheel barrow sprayer with boom | 4-5 trees, 58-300 $m^2$ | 1-6.5 kg |
| Peach | Italy | 1995 | hand gun motor pump sprayer | 136-224 $m^2$ | 2 kg |
| Grapes | France, Spain | 1995 | motorised knapsack | 4-8 rows of 25-30 m | min 1 kg |
| Grapes | Germany | 1994 | knapsack | 126-300 $m^2$ | 4-4.5 kg |
| Onion | USA | 1988-89 | backpack $CO_2$ sprayer, aerial fixed wing, helicopter | 19 $m^2$-1.2 ha | 12 bulbs, 1 kg |
| Broccoli | USA | 1988-89 | backpack $CO_2$ sprayer, aerial fixed wing, helicopter | 60 $m^2$-0.11 ha | 12 heads |
| Cabbage | USA | 1988-89 | backpack $CO_2$ sprayer, aerial fixed wing, helicopter | 23 $m^2$-0.33 ha | 12 heads or half-heads |
| Sweet corn | USA | 1988-89 | backpack $CO_2$ sprayer, aerial fixed wing, helicopter | 37 $m^2$-0.33 ha | 12 ears, 1 kg fodder, 2 kg forage |
| Mustard greens | USA | 1988-89 | backpack $CO_2$ sprayer, self-propelled research sprayer, aerial fixed wing | 28 $m^2$-0.20 ha | 1-3 kg |
| Lettuce | USA | 1988-89 | backpack $CO_2$ sprayer, aerial fixed wing, helicopter | 23 $m^2$-0.20 ha | 12 heads, 1-3 kg |
| Spinach | USA | 1988-89 | backpack $CO_2$ sprayer, aerial fixed wing, helicopter | 28 $m^2$-0.11 ha | 1-2 kg |
| Lima beans | USA | 1989 | backpack $CO_2$ sprayer, self-propelled research sprayer, aerial fixed wing, helicopter | 98 $m^2$-0.16 ha | 1 kg |
| Snap beans | USA | 1988 | backpack $CO_2$ sprayer, self-propelled research sprayer, aerial fixed wing | 37 $m^2$c-0.16 ha | 1 kg |
| Soya beans | USA | 1988-90 | backpack $CO_2$ sprayer, tractor mounted sprayer | 56 $m^2$-465 $m^2$ | 1 kg |
| Peas, dried | USA | 1989 | backpack $CO_2$ sprayer, tractor mounted sprayer, helicopter, aerial fixed wing | 31 $m^2$-0.26 ha | 0.25-2 kg |
| Beans, dry | USA | 1989 | backpack $CO_2$ sprayer, tractor mounted sprayer, aerial fixed wing | 88 $m^2$-0.16 ha | 0.5-1 kg |
| Carrot | USA | 1989 | backpack $CO_2$ sprayer, self-propelled research sprayer, aerial fixed wing | 29 $m^2$-1.2 ha | 1-4 kg |
| Potatoes | USA | 1988-89 | backpack $CO_2$ sprayer, self-propelled research sprayer, aerial fixed wing | 53 $m^2$ -0.14 ha | 16 tubers |
| Sugar beet | USA | 1988-89 | backpack $CO_2$ sprayer, self-propelled research sprayer, aerial fixed wing | 56 $m^2$-1.6 ha | 2 kg |
| Turnip | USA | 1988-89 | backpack $CO_2$ sprayer, self-propelled research sprayer, aerial fixed wing | 37 $m^2$-0.14 ha | 1-4 kg |
| Celery | USA | 1988-89 | backpack $CO_2$ sprayer, aerial fixed wing, helicopter | 29 $m^2$-0.33 ha | 12 plants |
| Maize | USA | 1988-90 | backpack $CO_2$ sprayer, self-propelled research sprayer, tractor mounted sprayer, aerial fixed wing, | 46 $m^2$-0.17 ha | 1-2.5 kg |
| Rice | USA | 1988 | aerial fixed wing | 0.04-0.84 ha | 1-1.5 kg |
| Sorghum | USA | 1988-89 | tractor mounted sprayer, self-propelled research sprayer, aerial fixed wing | 60 $m^2$-0.20 ha | 0.5-1.5 kg |
| Wheat | USA | 1989 | backpack $CO_2$ sprayer, self-propelled research sprayer, tractor mounted sprayer, aerial fixed wing, helicopter | 46 $m^2$-0.37 ha | 0.5-1 kg |

| Crop | Country | Year | Sprayer | Plot size | Sample size |
|---|---|---|---|---|---|
| Cotton | USA | 1988, 1998 | backpack $CO_2$ sprayer, self-propelled research sprayer, tractor mounted sprayer, aerial fixed wing | 56 $m^2$-0.84 ha | 1-2 kg. 40 kg seed cotton to produce 1 kg seed |
| Canola | USA | 1992 | aerial fixed wing | 0.4-1.8 ha | 1-1.5 kg |
| Sunflower | USA | 1988 | tractor mounted sprayer, aerial fixed wing | 170-900 $m^2$ | 1 kg |
| Alfalfa | USA | 1989, 1998 | backpack $CO_2$ sprayer, tractor mounted sprayer, hand held boom, aerial fixed wing | 74 $m^2$-1.2 ha | 0.5-4 kg |
| Clover | USA | 1989 | backpack $CO_2$ sprayer, self-propelled research sprayer, tractor mounted sprayer, aerial fixed wing, helicopter | 74 $m^2$-0.14 ha | 0.5-1 kg |
| Pasture grasses | USA | 1988, 1998 | backpack $CO_2$ sprayer, tractor mounted sprayer, self-propelled research sprayer, hand held boom, aerial fixed wing, helicopter | 42 $m^2$-0.45 ha | 0.5-3 kg |
| Hops | USA | 1990 | hand held sprayer, backpack mist blower | 49-125 $m^2$ | 0.5-1 kg |

Table 20. Residues in apples and pears from supervised trials in Germany and France. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| APPLE | | | | | | | | | |
| Germany, 1994 (James Grieve) | CS | 1.0 | | 1000 | 1 | 28 | <0.01 | | 407739 |
| Germany, 1994 (James Grieve) | CS | 0.45 | | 1000 | 1 | 28 | <0.01 | | 407720 |
| Germany, 1994 (James Grieve) | WP | 0.50 | | 1000 | 1 | 28 | <0.01 (2) | | 407704 |
| Germany, 1994 (James Grieve) | WP | 0.50 | | 1000 | 1 | 28 | <0.01 (2) | | 407690 |
| France, 1994 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.24<br>0.05<br>0.03<br>0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Site I<br>51D/951553 |
| France, 1994 (Red Chief) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.16<br>0.10<br>0.07<br>0.04<br>0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Site II<br>51D/951553 |
| France, 1994 (Melrose) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.07<br>0.06<br>0.03<br>0.02<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Olivet F1<br>51D/951553 |
| France, 1994 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.22<br>0.05<br>0.02<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Olivet F2<br>51D/951553 |
| France, 1995 (Canada Gris) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.40<br>0.51<br>0.26<br>0.12<br>0.09<br>0.04 | <0.01<br>0.02<br><0.01<br>0.02<br>0.01<br><0.01 | Cheille F3<br>961451 |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| France, 1995 (Jonagold) | CS | 0.36 | 0.036 | 1000 | 2 | 0 | 0.22 | <0.01 | Loiret F1 |
| | | | | | | 3 | 0.10 | <0.01 | 961451 |
| | | | | | | 7 | 0.06 | <0.01 | |
| | | | | | | 14 | 0.04 | <0.01 | |
| | | | | | | 21 | 0.02 | <0.01 | |
| | | | | | | 28 | 0.02 | <0.01 | |
| France, 1995 (Golden Delicious | CS | 0.36 | 0.036 | 1000 | 2 | 0 | 0.22 | <0.01 | Loiret F2 |
| | | | | | | 3 | 0.11 | <0.01 | 961451 |
| | | | | | | 7 | 0.09 | <0.01 | |
| | | | | | | 14 | 0.03 | <0.01 | |
| | | | | | | 21 | 0.01 | <0.01 | |
| | | | | | | 28 | <0.01 | <0.01 | |
| France, 1995 (Golden Delicious | CS | 0.36 | 0.036 | 1000 | 2 | 0 | 0.29 | <0.01 | Maugio F4 |
| | | | | | | 3 | 0.16 | <0.01 | 961451 |
| | | | | | | 7 | 0.08 | <0.01 | |
| | | | | | | 14 | 0.07 | <0.01 | |
| | | | | | | 21 | 0.05 | <0.01 | |
| | | | | | | 28 | 0.02 | <0.01 | |
| France, 1995 (Golden Delicious | CS | 0.36 | 0.036 | 1000 | 2 | 0 | 0.32 | <0.01 | Meauzac F6 |
| | | | | | | 3 | 0.28 | <0.01 | 961451 |
| | | | | | | 7 | 0.17 | <0.01 | |
| | | | | | | 14 | 0.08 | <0.01 | |
| | | | | | | 21 | 0.15 | <0.01 | |
| | | | | | | 28 | 0.12 | <0.01 | |
| France, 1995 (Golden Delicious | CS | 0.36 | 0.036 | 1000 | 2 | 0 | 0.28 | <0.01 | Meauzac F7 |
| | | | | | | 3 | 0.23 | <0.01 | 961451 |
| | | | | | | 7 | 0.23 | <0.01 | |
| | | | | | | 14 | 0.10 | <0.01 | |
| | | | | | | 21 | 0.05 | <0.01 | |
| | | | | | | 28 | 0.03 | <0.01 | |
| France, 1995 (Red Chief) | CS | 0.36 | 0.036 | 1000 | 2 | 0 | 0.17 | <0.01 | Meauzac F8 |
| | | | | | | 3 | 0.12 | <0.01 | 961451 |
| | | | | | | 7 | 0.07 | <0.01 | |
| | | | | | | 14 | 0.11 | <0.01 | |
| | | | | | | 21 | 0.05 | <0.01 | |
| | | | | | | 28 | 0.03 | <0.01 | |
| France, 1995 (Granny Smith) | CS | 0.36 | 0.036 | 1000 | 2 | 0 | 0.20 | <0.01 | Meauzac F9 |
| | | | | | | 3 | 0.16 | <0.01 | 961451 |
| | | | | | | 7 | 0.07 | <0.01 | |
| | | | | | | 14 | 0.05 | <0.01 | |
| | | | | | | 21 | 0.03 | <0.01 | |
| | | | | | | 28 | 0.01 | <0.01 | |
| France, 1995 (Golden Delicious) | CS | 0.36 | 0.036 | 1000 | 2 | 0 | 0.20 | <0.01 | Herault F5 |
| | | | | | | 3 | 0.19 | <0.01 | 961451 |
| | | | | | | 7 | 0.10 | <0.01 | |
| | | | | | | 14 | 0.06 | <0.01 | |
| | | | | | | 21 | 0.05 | <0.01 | |
| | | | | | | 28 | 0.03 | <0.01 | |
| France, 1995 (Canada Gris) | EC | 0.36 | 0.036 | 1000 | 1 | 0 | 0.42 | <0.01 | F3 |
| | | | | | | 3 | 0.25 | 0.01 | 961450 |
| | | | | | | 7 | 0.11 | <0.01 | |
| | | | | | | 14 | 0.04 | <0.01 | |
| | | | | | | 21 | 0.04 | <0.01 | |
| | | | | | | 28 | 0.03 | <0.01 | |
| France, 1995 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 1 | 0 | 0.19 | <0.01 | Herault F4 |
| | | | | | | 3 | 0.04 | <0.01 | 961450 |
| | | | | | | 7 | 0.02 | <0.01 | |
| | | | | | | 14 | <0.01 | <0.01 | |
| | | | | | | 21 | <0.01 | <0.01 | |
| | | | | | | 28 | <0.01 | <0.01 | |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| France, 1995 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 1 | 0<br>3<br>7<br>14<br>21<br>28 | 0.34<br>0.10<br>0.07<br>0.03<br>0.11<br>0.08 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Meauzac F6<br>961450 |
| France, 1995 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 1 | 0<br>3<br>7<br>14<br>21<br>28 | 0.36<br>0.07<br>0.07<br>0.01<br>0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Meauzac F7<br>961450 |
| France, 1995 (Red Chief) | EC | 0.36 | 0.036 | 1000 | 1 | 0<br>3<br>7<br>14<br>21<br>28 | 0.2<br>0.04<br>0.03<br>0.01<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Meauzac F8<br>961450 |
| France, 1995 (Granny Smith) | EC | 0.36 | 0.036 | 1000 | 1 | 0<br>3<br>7<br>14<br>21<br>28 | 0.27<br>0.05<br>0.02<br>0.02<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Meauzac F9<br>961450 |
| France, 1995 (Jonagold) | EC | 0.36 | 0.036 | 1000 | 1 | 0<br>3<br>7<br>14<br>21<br>28 | 0.19<br>0.05<br>0.02<br><0.01<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Loiret F1<br>961450 |
| France, 1995 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 1 | 0<br>3<br>7<br>14<br>21 | 0.24<br>0.06<br>0.03<br>0.02<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Loiret F2<br>961450 |
| France, 1995 (Golden Delicious) | EC | 0.36 | 0.036 | 1000 | 1 | 0<br>3<br>7<br>14<br>21<br>28 | 0.18<br>0.08<br>0.04<br>0.02<br>0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Herault F5<br>961450 |
| French Southern Territories, 1994 (Red Chief) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.26<br>0.25<br>0.24<br>0.18<br>0.11 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Site II<br>91554 |
| French Southern Territories, 1994 (Golden Delicious) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21<br>39 | 0.30<br>0.11<br>0.06<br>0.04<br>0.02<br>0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Site I<br>91554 |
| France, 1994 (Golden Delicious) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21 | 0.28<br>0.20<br>0.01<br>0.04<br>0.02 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | St Hilaire F2<br>91554 |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| France, 1994 (Melrose) | CS | 0.36 | 0.036 | 1000 | 2 | 0<br>3<br>7<br>14<br>21<br>39 | 0.09<br>0.10<br>0.06<br>0.03<br>0.02<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | St Hilaire F1 91554 |
| PEAR | | | | | | | | | |
| Germany, 1994 (Alexander Lucas) | CS | 0.45 | | 1000 | 1 | 28 | <0.01 | | 407747 |
| Germany, 1994 (Alexander Lucas) | WP | 0.50 | | 1000 | 1 | 28 | <0.01 (2) | | 407712 |

Table 21. Residues in peaches from supervised trials in Italy. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| 1994 (Maria Bianca) | EC | 0.55 | 0.046 | 1200 | 2 | 0<br>7<br>14<br>21 | <0.01 0.21<br>0.02<br><0.01<br><0.01 | | 407267 |
| 1994 (Red Haven) | EC | 0.55 | 0.046 | 1200 | 2 | 0<br>7<br>14<br>21 | 0.08 0.01<br>0.03<br>0.01<br><0.01 | | 407259 |
| 1994 (Maria Bianca) | CS | 0.54 | 0.045 | 1200 | 2 | 0<br>7<br>14<br>21 | 0.05 0.11<br>0.11<br>0.08<br>0.06 | | 407224 |
| 1994 (Red Haven) | CS | 0.54 | 0.045 | 1200 | 2 | 0<br>7<br>14<br>21 | 0.22 0.27<br>0.21<br>0.15<br>0.10 | | 407216 |
| 1994 (Maria Bianca) | EC | 0.54 | 0.045 | 1200 | 2 | 0<br>7<br>14<br>21 | <0.01 0.22<br>0.02<br><0.01<br><0.01 | | 407208 |
| 1994 (Red Haven) | EC | 0.54 | 0.045 | 1200 | 2 | 0<br>7<br>14<br>21 | 0.01 0.07<br>0.03<br>0.01<br>0.01 | | 407194 |
| 1995 (K2) | CS | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28 | 0.85<br>1.6<br>1.7<br>0.84<br>0.46<br>0.09<br>0.10 | <0.01<br><0.01<br>0.01<br><0.01<br><0.01<br><0.01<br><0.01 | 9551-1 CHV 57C/952700 |

| Year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| 1995 (Fayette) | CS | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28 | 0.30<br>0.81<br>0.37<br>0.29<br>0.14<br>0.13<br>0.02 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | 9551-2<br>CHV<br>57C/952700 |
| 1995 (Duchessa D'Este) | CS | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28 | 0.59<br>1.3<br>0.87<br>1.4<br>0.32<br>0.05<br>0.22 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | 9551-3<br>CHV<br>57C/952700 |
| 1995 (Autumn Glo) | CS | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28<br>28 | 0.32<br>0.62<br>0.18<br>0.32<br>0.05<br>0.06<br>0.02<br>juice 0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br>juice <0.01 | 9551-4<br>CHV<br>57C/952700 |
| 1995 (Crest Haven) | CS | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28<br>28 | 0.03<br>0.65<br>0.39<br>0.27<br>0.08<br>0.08<br>0.03<br>juice <0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br>juice <0.01 | 9551-5<br>CHV<br>57C/952700 |
| 1995 (Hale) | CS | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28 | 0.50<br>1.6<br>0.74<br>0.49<br>0.26<br>0.16<br>0.08 | <0.01<br>0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | 9551-6<br>CHV<br>57C/952700 |
| 1995 (K2) | EC | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28 | 0.03<br>0.68<br>0.15<br>0.09<br>0.04<br>0.02<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | 9550-1<br>CHV<br>56C/952684 |
| 1995 (Fayette) | EC | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28 | 0.04<br>0.70<br>0.16<br>0.06<br>0.02<br>0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | 9550-2<br>CHV<br>56C/952684 |

| Year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| 1995 (Duchessa D'Este) | EC | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28 | <0.01 c 0.01<br>0.58<br>0.24<br>0.18 c 0.01<br>0.05<br>0.02 c 0.01<br>0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | 9550-3<br>CHV<br>56C/952684 |
| 1995 (Autumn Glo) | EC | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28<br>28 | 0.03<br>0.42<br>0.38<br>0.05<br>0.03<br>0.01<br><0.01<br>juice <0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br>juice <0.01 | 9550-4<br>CHV<br>56C/952684 |
| 1995 (Crest Haven) | EC | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28<br>28 | 0.32<br>0.87<br>0.33<br>0.20<br>0.10<br>0.04<br>0.02<br>juice <0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br>juice <0.01 | 9550-5<br>CHV<br>56C/952684 |
| 1995 (Hale) | EC | 0.54 | 0.045 | 1200 | 1<br>2 | 14<br>0<br>3<br>7<br>14<br>21<br>28 | 0.04<br>0.53<br>0.13<br>0.05<br>0.02<br>0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | 9550-6<br>CHV<br>56C/952684 |

c: sample from control plot

Table 22. Residues in wine grapes from supervised trials in Italy, France, Spain and Germany. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| France, 1994 (Chenin Blanc) | CS | 0.29 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>35 | 0.09<br>0.05<br>0.11<br>0.06<br>0.05<br>0.07 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | AP/2582/HR<br>F1<br>951174 |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| France, 1994 (Chenin Blanc) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.05<br>0.04<br>0.01<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Tours F1 951175 |
| France, 1994 (Grenache) | CS | 0.32 | 0.16 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>31 | 0.28<br>0.16<br>0.28<br>0.11<br>0.13<br>0.07 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | AP/2582/HR Site II 951174 |
| France, 1994 (Grenache) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.21<br>0.11<br>0.08<br>0.05<br>0.05 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | AP/2582/HR Site I 951174 |
| France, 1994 (Grenache) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.08<br>0.02<br><0.01<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Montpelier Site I 951175 |
| France, 1994 (Grenache) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21 | 0.16<br>0.03<br>0.02<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Nimes Site II 951175 |
| France, 1995 (Cabernet) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.25<br>0.22<br>0.24<br>0.17<br>0.10<br>0.09 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Montreuil-Bellay F2 960539 |
| France, 1995 (Cabernet) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.22<br>0.06<br>0.02<br>0.01<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Montreuil-Bellay F2 961449 |
| France, 1995 (Chenin) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.12<br>0.19<br>0.18<br>0.18<br>0.09<br>0.08 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Chancay F4 960539 |
| France, 1995 (Chenin) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.09<br>0.06<br>0.02<br><0.01<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Chancay F4 961449 |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| France, 1995 (Gris Meunier) | CS | 0.30 | 0.15 | 200 | 2 | 0 | 1.2 | 0.01 | Loiret F1 960539 |
| | | | | | | 3 | 1.1 | 0.01 | |
| | | | | | | 7 | 0.78 | 0.01 | |
| | | | | | | 14 | 0.57 | <0.01 | |
| | | | | | | 21 | 0.02 [1] | <0.01 [1] | |
| | | | | | | 28 | 0.41 | <0.01 | |
| France, 1995 (Gris Meunier) | EC | 0.30 | 0.15 | 200 | 2 | 0 | 0.72 | 0.01 | Loiret F1 961449 |
| | | | | | | 3 | 0.22 | 0.02 | |
| | | | | | | 7 | 0.06 | <0.01 | |
| | | | | | | 14 | 0.03 | <0.01 | |
| | | | | | | 21 | 0.47 [2] | <0.01 [2] | |
| | | | | | | 28 | 0.02 | <0.01 | |
| France, 1995 (Groleau) | CS | 0.30 | 0.15 | 200 | 2 | 0 | 0.23 | <0.01 | Chinon F3 960539 |
| | | | | | | 3 | 0.16 | <0.01 | |
| | | | | | | 7 | 0.17 | <0.01 | |
| | | | | | | 14 | 0.11 | <0.01 | |
| | | | | | | 21 | 0.09 | <0.01 | |
| | | | | | | 28 | 0.10 | <0.01 | |
| France, 1995 (Groleau) | EC | 0.30 | 0.15 | 200 | 2 | 0 | 0.18 | <0.01 | Loiret F3 961449 |
| | | | | | | 3 | 0.03 | <0.01 | |
| | | | | | | 7 | 0.01 | <0.01 | |
| | | | | | | 14 | <0.01 | <0.01 | |
| | | | | | | 21 | <0.01 | <0.01 | |
| | | | | | | 28 | <0.01 | <0.01 | |
| France, 1995 (Syrah) | CS | 0.30 | 0.15 | 200 | 2 | 0 | 0.36 | <0.01 | Tarn-et-Garonne F5 960539 |
| | | | | | | 3 | 0.41 | <0.01 | |
| | | | | | | 7 | 0.27 | <0.01 | |
| | | | | | | 14 | 0.16 | <0.01 | |
| | | | | | | 21 | 0.09 | <0.01 | |
| | | | | | | 28 | 0.09 | <0.01 | |
| France, 1995 (Syrah) | EC | 0.30 | 0.15 | 200 | 2 | 0 | 0.23 | <0.01 | Tarn-et Garonne F5 961449 |
| | | | | | | 3 | 0.13 | 0.01 | |
| | | | | | | 7 | 0.05 | <0.01 | |
| | | | | | | 14 | 0.01 | <0.01 | |
| | | | | | | 21 | <0.01 | <0.01 | |
| | | | | | | 28 | <0.01 | <0.01 | |
| France, 1995 (Tannat) | CS | 0.30 | 0.15 | 200 | 2 | 0 | 0.32 | <0.01 | Garonne F6 960539 |
| | | | | | | 3 | 0.28 | <0.01 | |
| | | | | | | 7 | 0.23 | <0.01 | |
| | | | | | | 14 | 0.15 | <0.01 | |
| | | | | | | 21 | 0.10 | <0.01 | |
| | | | | | | 28 | 0.10 | <0.01 | |
| France, 1995 (Tannat) | EC | 0.30 | 0.15 | 200 | 2 | 0 | 0.20 | <0.01 | Montech F6 961449 |
| | | | | | | 3 | 0.07 | <0.01 | |
| | | | | | | 7 | 0.03 | <0.01 | |
| | | | | | | 14 | <0.01 | <0.01 | |
| | | | | | | 21 | <0.01 | <0.01 | |
| | | | | | | 28 | <0.01 | <0.01 | |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| Germany, 1994<br>(Kerner) | CS | 0.12<br>+0.32 | 0.020 | 600<br>+1600 | 2 | 0<br>14<br>28<br>35<br>42<br>35<br>35 | 0.17<br>0.18<br>0.14<br>0.13<br>0.10<br>must <0.01<br>wine <0.01 | | 407771 |
| Germany, 1994<br>(Kerner) | WP | 0.12<br>+0.32 | 0.020 | 600<br>+1600 | 2 | 0<br>14<br>28<br>35<br>42<br>35<br>35 | 0.20<br>0.02<br><0.01<br><0.01<br><0.01<br>must <0.01<br>wine <0.01 | | 407828 |
| Germany, 1994<br>(Portugieser) | CS | 0.12<br>+0.32 | 0.020 | 600<br>+1600 | 2 | 0<br>14<br>28<br>35<br>42<br>35<br>35 | 0.14<br>0.11<br>0.04<br><0.01<br><0.01<br>must <0.01<br>wine <0.01 | | 407763 |
| Germany, 1994<br>(Portugieser) | WP | 0.12<br>+0.32 | 0.020 | 600<br>+1600 | 2 | 0<br>14<br>28<br>35<br>42<br>35<br>35 | 0.10<br><0.01<br><0.01<br>0.014<br>0.042<br>must <0.01<br>wine <0.01 | | 407801 |
| Germany, 1994<br>(Spätburgunder) | CS | 0.12<br>+0.32 | 0.020 | 600<br>+1600 | 2 | 0<br>14<br>28<br>35<br>43<br>35<br>35 | 0.14<br>0.10<br>0.06<br>0.120<br>0.102<br>must <0.01<br>wine <0.01 | | 407755 |
| Germany, 1994<br>(Spätburgunder) | WP | 0.12<br>+0.33 | 0.020 | 600<br>+1600 | 2 | 0<br>14<br>28<br>35<br>43<br>35<br>35 | 0.07<br><0.01<br><0.01<br><0.01<br><0.01<br>must <0.01<br>wine <0.01 | | 407798 |
| Italy, 1994<br>(Pampanuto)-white | EC | 0.23 | 0.046 | 500 | 2 | 0<br>7<br>14<br>21 | 0.07 <0.01<br><0.01<br><0.01<br><0.01 | | 407275 |
| Italy, 1994<br>(Pampanuto)-white | EC | 0.25 | 0.050 | 500 | 2 | 0<br>7<br>14<br>21 | 0.15 <0.01<br><0.01<br><0.01<br><0.01 | | 407291 |

| Country, year (variety) | Application | | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| Italy, 1994 (Pampanuto)-white | CS | 0.30 | 0.060 | 500 | 2 | 0<br>7<br>14<br>21 | 0.28 0.15<br>0.17<br>0.13<br>0.12 | | 407305 |
| Italy, 1994 (Pampanuto)-white | EC | 0.34 | 0.068 | 500 | 2 | 0<br>7<br>14<br>21 | 0.10 <0.01<br>0.01<br><0.01<br><0.01 <0.01 | | 407879 |
| Italy, 1994 (Sangiovese)-red | CS | 0.30 | 0.060 | 500 | 2 | 0<br>7<br>14<br>21 | 0.30 0.12<br>0.14<br>0.16<br>0.18 | | 407240 |
| Italy, 1994 (Sangiovese)-red | EC | 0.23 | 0.046 | 500 | 2 | 0<br>7<br>14<br>21 | 0.03 <0.01<br><0.01<br><0.01<br><0.01 | | 407283 |
| Italy, 1994 (Sangiovese)-red | EC | 0.34 | 0.068 | 500 | 2 | 0<br>7<br>14<br>21 | 0.06 <0.01<br>0.01<br><0.01<br><0.01 | | 407887 |
| Italy, 1994 (Sangiovese) | EC | 0.25 | 0.050 | 500 | 2 | 0<br>7<br>14<br>21 | 0.26 <0.01<br>0.01<br>0.01<br>0.01 | | 407232 |
| Spain, 1995 (Carignan) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.25<br>0.26<br>0.12<br>0.08<br>0.05<br>0.03 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Girona S8 960539 |
| Spain, 1995 (Carignan) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.29<br>0.04<br>0.01<br><0.01<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Girona S8 961449 |
| Spain, 1995 (Tempranillo) | CS | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.32<br>0.26<br>0.21<br>0.13<br>0.13<br>0.12 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Girona S7 960539 |
| Spain, 1995 (Tempranillo) | EC | 0.30 | 0.15 | 200 | 2 | 0<br>3<br>7<br>14<br>21<br>28 | 0.37<br>0.06<br>0.02<br>0.01<br><0.01<br><0.01 | <0.01<br><0.01<br><0.01<br><0.01<br><0.01<br><0.01 | Girona S7 961449 |

[1]21-day data not available because possible sample confusion with trial F1 961449
[2] suspected sample confusion with trial F1 960539

Table 23. Residues in onions from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels. All EC formulations.

| Location, year (variety) | Application | | | PHI days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Southport Lake) | 1.1 | 190 | 6 | 15 | bulb | <0.05 (2)<br>c <0.05 | <0.05 (2)<br>c 0.11 | MP-ON-3107 |
| CA, 1988 (Southport Lake) | 1.1 | 190 | ( 6 | 15 | bulb | <0.05 (2) | <0.05 (2) | MP-ON-3108 |
| WA, 1988 (Walla walla sweet) | 1.1 | 190 | 5 | 15 | bulb | <0.05 (2) | <0.05 (2) | MP-ON-3109 |
| TX, 1989 (Ben Shamen) | 1.0 | 75 | 6 | 15 | bulb | 0.11 0.13 | [1] | MP-ON-3110 |
| TX, 1989 (Ben Shamen) | 1.1 | 51 | ( 6 | 15 | bulb | 0.21 0.20<br>c 0.07 | [1] | MP-ON-3111 |
| MI, 1988 (Early Yellow Globe) | 1.2 | 210 | 6 | 15 | bulb | <0.05<br>c <0.05 | 0.08<br>c 0.08 | MP-ON-7087 |
| NY, 1988 (Early Yellow Globe) | 1.1 | 235 | 6 | 15 | bulb | 0.40 0.58 | <0.05 (2) | MP-ON-7088 |
| CA, 1989 | 1.1 | 370 | 6 | 15 | green | 0.38 0.37<br>c <0.05 | 0.33 0.19<br>c 0.20 | MP-ON-3112 |
| AZ, 1989 (Sweet Spanish) | 1.1 | 220 | 6 | 15 | green | 0.09 0.10<br>c 0.06 | [1] | MP-ON-3114 |
| CA, 1989 (K-99) | 1.1 | 94 | ( 6 | 15 | green | <0.05 (2) | <0.05 (2) | MP-ON-3115 |
| CA, 1989 (Evergreen) | 0.56 | 190 | ( 6 | 15 | green | <<u>0.05</u> (2)<br>c <0.05 | <u>0.24</u> 0.24<br>c 0.15 | MP-ON-3113 |
| ID, 1988 (Ebeneezer) | 1.1 | 280 | 6 | 15 | green | 0.16 0.08 | 0.05 <0.05 | MP-ON-7086 |

[1]measurement of paraoxon-methyl not possible because of co-eluting interference in GC
( aerial application. c: sample from control plot

Table 24. Residues in broccoli from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (Commander) | 4×1.7<br>+2×0.56 | 200 | 6 | 7 | <0.05 (2) | <0.05 (2) | MP-BR-3021 |
| TX, 1988 (Commander) | 1.7 | 200 | 6 | 21 | <0.05 (2) | <0.05 (2) | MP-BR-3021 |
| TX, 1988 (Commander) | 1.7 | 49 | ( 6 | 21 | <0.05 (2) | <0.05 (2) | MP-BR-3023 |

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (Commander) | 4×1.7 +2×0.56 | 49 | ( 6 | 7 | <0.05 (2) | <0.05 (2) | MP-BR-3023 |
| CA, 1989 | 4×1.7 +2×0.56 | 190 | 6 | 7 | <0.05 (2) | <0.05 (2) | MP-BR-3024 |
| CA, 1989 | 1.7 | 190 | 6 | 21 | <0.05 (2) | <0.05 (2) | MP-BR-3024 |
| CA, 1989 (501 Green Valiant) | 1.7 | 370 | 6 | 21 | 0.05 <0.05 | <0.05 (2) | MP-BR-3026 |
| CA, 1989 (501 Green Valiant) | 4×1.7 +2×0.56 | 370 | 6 | 7 | 0.06 0.10 | 0.12 0.14 | MP-BR-3026 |
| CA, 1989 (501 Green Valiant) | 2×1.7 +2×0.84 +3×1.7 | 190 | ( 7 | 21 | <0.05 (2) | <0.05 (2) | MP-BR-3028 |
| CA, 1989 (501 Green Valiant) | 2×0.56 +2×1.7 +3×0.56 | 190 | ( 7 | 7 | <0.05 (2) | <0.05 (2) | MP-BR-3028 |
| OR, 1988 (Gem) | 1.7 | 190 | 6 | 21 | <0.05 (2) | <0.05 (2) | MP-BR-3029 |
| OR, 1988 (Gem) | 4×1.7 +2×0.56 | 190 | 6 | 7 | <0.05 (2) | <0.05 (2) | MP-BR-3029 |

( aerial application.

Table 25. Residues in cabbage from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels. EC formulations.

| Location, year (variety) | Application | | | PHI days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Headstart) | 6×1.7 +1×0.56 | 375 | 7 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-3031 |
| CA, 1988 (Headstart) | 6×1.7 +1×0.56 | 375 | 7 | 10 | including wrapper leaves | <0.5 (2) [1] | 0.09 0.08 | MP-CB-3031 |
| CA, 1988 (Headstart) | 1.7 | 375 | 7 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-3031 |
| CA, 1988 (Headstart) | 1.7 | 375 | 7 | 21 | including wrapper leaves | <0.5 (2) [1] | 0.06 0.12 | MP-CB-3031 |
| CA, 1988 (Headstart) | 6×1.7 +1×0.56 | 200 | ( 7 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-3033 |

| Location, year (variety) | Application | | | PHI days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Headstart) | 6×1.7 +1×0.56 | 200 | ( 7 | 10 | including wrapper leaves | <0.5 (2) [1] | 0.24 0.19 | MP-CB-3033 |
| CA, 1988 (Headstart) | 1.7 | 200 | ( 7 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-3033 |
| CA, 1988 (Headstart) | 1.7 | 200 | ( 7 | 21 | including wrapper leaves | <0.5 (2) [1] | 0.08 0.08 | MP-CB-3033 |
| TX, 1988 (Bravo) | 5×1.7 +1×0.56 | 98 | 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-3034 |
| TX, 1988 (Bravo) | 5×1.7 +1×0.56 | 98 | 6 | 10 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-3034 |
| TX, 1988 (Bravo) | 1.7 | 98 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-3034 |
| TX, 1988 (Bravo) | 1.7 | 98 | 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-3034 |
| FL, 1989 (Bravo) | 1×1.1 +4×1.7 +1×0.56 | 520 | 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7018 |
| FL, 1989 (Bravo) | 1×1.1 +4×1.7 +1×0.56 | 520 | 6 | 10 | including wrapper leaves | <0.05 (2) | 0.21 0.22 | MP-CB-7018 |
| FL, 1989 (Bravo) | 1×1.1 +5×1.7 | 520 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7018 |
| FL, 1989 (Bravo) | 1×1.1 +5×1.7 | 520 | 6 | 21 | including wrapper leaves | <0.05 (2) | 0.23 0.22 | MP-CB-7018 |
| NJ, 1988 (Danish) | 5×1.7 +1×0.56 | 540 | 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7020 |
| NJ, 1988 (Danish) | 5×1.7 +1×0.56 | 540 | 6 | 10 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7020 |
| NJ, 1988 (Danish) | 1.7 | 540 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7020 |
| NJ, 1988 (Danish) | 1.7 | 540 | 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7020 |
| NY, 1988 (King Cole) | 5×1.7 +1×0.56 | 350 | 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7022 |
| NY, 1988 (King Cole) | 5×1.7 +1×0.56 | 350 | 6 | 10 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7022 |

| Location, year (variety) | Application | | | PHI days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| NY, 1988 (King Cole) | 1.7 | 350 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7022 |
| NY, 1988 (King Cole) | 1.7 | 350 | 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7022 |
| NY, 1988 (King Cole) | 5×1.7 +1×0.56 | 45 | ( 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7024 |
| NY, 1988 (King Cole) | 5×1.7 +1×0.56 | 45 | ( 6 | 10 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7024 |
| NY, 1988 (King Cole) | 1.7 | 45 | ( 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7024 |
| NY, 1988 (King Cole) | 1.7 | 45 | ( 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7024 |
| WI, 1988 (Gourmet) | 5×1.7 +1×0.56 | 190 | 6 | 10 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7025 |
| WI, 1988 (Gourmet) | 5×1.7 +1×0.56 | 190 | 6 | 10 | including wrapper leaves | <0.05 (2) | 0.07 0.06 | MP-CB-7025 |
| WI, 1988 (Gourmet) | 1.7 | 190 | 6 | 22 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7025 |
| WI, 1988 (Gourmet) | 1.7 | 190 | 6 | 22 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-CB-7025 |

( aerial application
[1] Overspray with Meta-systox left residues that caused interference with the analytical method for parathion-methyl.

Table 26. Residues in sweet corn from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 | 1.1 | 300 | 6 | 3 | ears | <0.05 (2) | <0.05 (2) | MP-CN-3047 |
| CA, 1988 (Jubilee) | 1.1 | 84 | ( 3 | 3 | ears | <0.05 (2) | <0.05 (2) | MP-CN-3049 |
| TX, 1988 (Funk's Sweet G-90) | 1.1 | 9 | ( 6 | 3 | ears | <0.05 (2) | <0.05 (2) | MP-CN-3050 |
| WA, 1988 (Jubilee) | 1.1 | 94 | ( 6 | 3 | ears | <0.05 (2) | <0.05 (2) | MP-CN-3185 |
| FL, 1988 (Merit) | 1.1 | 300 | 6 | 3 | ears | <0.05 (2) | <0.05 (2) | MP-CN-7056 |
| NY, 1988 (Jubilee) | 1.1 | 45 | ( 6 | 3 | ears | <0.05 0.09 | <0.05 (2) | MP-CN-7057 |
| WI, 1988 (Commander) | 1.1 | 38 | ( 6 | 3 | ears | <0.05 (2) | <0.05 (2) | MP-CN-7058 |

( aerial application. c: sample from control plot

Table 27. Residues in mustard greens from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1989 (Florida Broadleaf) | 5×1.7 +1×0.56 | 280 | 6 | 10 | greens | <0.05 (2) | <0.05 (2) | MP-MG-3101 |
| CA, 1989 (Florida Broadleaf) | 1.7 | 280 | 6 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-MG-3101 |
| CA, 1989 (Florida Broadleaf) | 5×1.7 +1×0.56 | 190 | ( 6 | 10 | greens | <0.05 (2) | <0.05 (2) | MP-MG-3103 |
| CA, 1989 (Florida Broadleaf) | 1.7 | 190 | ( 6 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-MG-3103 |
| TX, 1988 (Florida Broadleaf) | 5×1.7 +1×0.56 | 94 | 6 | 10 | greens | <0.05 (2) | <0.05 (2) | MP-MG-3104 |
| TX, 1988 (Florida Broadleaf) | 1.7 | 94 | 6 | 21 | greens | 0.09 <0.05 | <0.05 (2) | MP-MG-3104 |
| TX, 1988 (Florida Broadleaf) | 5×1.7 +1×0.56 | 47 | ( 6 | 10 | greens | 0.10 0.05 | <0.05 (2) | MP-MG-3106 |
| TX, 1988 (Florida Broadleaf) | 1.7 | 47 | ( 6 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-MG-3106 |
| FL, 1988 (Florida Broadleaf) | 2×1.7 +1×0.56 | 300 | 3 | 10 | greens | 0.15 0.51 | <0.05 0.09 | MP-MG-7080 |
| FL, 1988 (Florida Broadleaf) | 1.7 | 300 | 3 | 21 | greens | <0.05 0.06 | <0.05 (2) | MP-MG-7080 |
| LA, 1988 (Florida Broadleaf) | 2×1.7 +1×0.56 | 230 | 3 | 10 | greens | <0.05 (2) | <0.05 (2) | MP-MG-7082 |
| LA, 1988 (Florida Broadleaf) | 1.7 | 230 | 3 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-MG-7082 |
| OH, 1988 (Green Wave) | 2×1.7 +1×0.56 | 190 | 3 | 10 | greens | <0.05 (2) | <0.05 (2) | MP-MG-7084 |
| OH, 1988 (Green Wave) | 1.7 | 190 | 3 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-MG-7084 |

( aerial application.

Table 28. Residues in lettuce from supervised trials in the USA. All EC formulations.

| Lettuce, location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| Head , AZ, 1989 (Vanguard-75) | 1.1 | 240 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-3089 |
| Head , AZ, 1989 (Vanguard-75) | 1.1 | 240 | 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-3089 |

| Lettuce, location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| Head , CA, 1988 (Empire) | 1.1 | 190 | 6 | 21 | excluding wrapper leaves | 0.41 0.76 | <0.05 (2) | MP-LE-3091 |
| Head , CA, 1988 (Empire) | 1.1 | 190 | 6 | 21 | including wrapper leaves | 1.2 0.97 | <0.05 (2) | MP-LE-3091 |
| Head , CA, 1989 (Empire) | 1.1 | 190 | ( 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-3092 |
| Head , CA, 1989 (Empire) | 1.1 | 190 | ( 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-3092 |
| Head , FL, 1988 (Mesa 659) | 1.1 | 270 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-7068 |
| Head , FL, 1988 (Mesa 659) | 1.1 | 270 | 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-7068 |
| Head , MI, 1988 (Ithaca) | 1.2 | 210 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-7072 |
| Head , MI, 1988 (Ithaca) | 1.2 | 210 | 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-7072 |
| Head , NJ, 1988 (Montello) | 1.1 | 540 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-7070 |
| Head , NJ, 1988 (Montello) | 1.1 | 540 | 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-7070 |
| Head , TX, 1988 (Great Lakes 659) | 1.1 | 47 | ( 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-3094 |
| Head , TX, 1988 (Great Lakes 659) | 1.1 | 47 | ( 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-3094 |
| Head , TX, 1988 (Great Lakes 659) | 1.1 | 150 | 6 | 21 | excluding wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-3093 |
| Head , TX, 1988 (Great Lakes 659) | 1.1 | 150 | 6 | 21 | including wrapper leaves | <0.05 (2) | <0.05 (2) | MP-LE-3093 |
| Leaf , AZ, 1989 (Boston Dark Green) | 1.1 | 240 | 6 | 21 | leaves | <0.05 (2) | <0.05 (2) | MP-LE-3095 |
| Leaf , CA, 1988 (Waldmann's Green) | 1.1 | 190 | 6 | 21 | leaves | 1.3 1.6 | <0.05 (2) | MP-LE-3097 |
| Leaf , CA, 1989 (Waldmann's Green) | 1.1 | 190 | ( 6 | 21 | leaves | <0.05 (2) | <0.05 (2) | MP-LE-3098 |
| Leaf , FL, 1988 (Royal Red Leaf) | 1.1 | 280 | 6 | 21 | leaves | 0.07 0.11 | <0.05 (2) | MP-LE-7074 |
| Leaf , MI, 1988 (Black Seeded Simpson) | 1.1 | 210 | 6 | 21 | leaves | <0.05 (2) | <0.05 (2) | MP-LE-7078 |

| Lettuce, location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| Leaf , NJ, 1988 (Black Seeded Simpson) | 1.1 | 540 | 6 | 21 | leaves | 0.23 0.17 | <0.05 (2) | MP-LE-7076 |
| Leaf , TX, 1989 (Cos, Romaine) | 1.1 | 94 | 6 | 21 | leaves | <0.05 (2) | <0.05 (2) | MP-LE-3099 |
| Leaf , TX, 1989 (Black Seed Simpson) | 1.1 | 47 | ( 6 | 21 | leaves | <0.05 (2) | <0.05 (2) | MP-LE-3192 |

( aerial application.

Table 29. Residues in spinach from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Polka) | 5×1.1 +1×0.56 | 380 | 6 | 15 | 0.06 <0.05 | <0.05 (2) | MP-SP-3136 |
| CA, 1988 (Polka) | 1.1 | 380 | 6 | 21 | <0.05 (2) | <0.05 (2) | MP-SP-3136 |
| CA, 1988 (Walters) | 2×1.1 +1×0.56 | 200 | ( 3 | 15 | <0.05 (2) | <0.05 (2) | MP-SP-3138 |
| CA, 1988 (Walters) | 1.1 | 200 | ( 3 | 21 | <0.05 (2) | <0.05 (2) | MP-SP-3138 |
| CA, 1989 (St Helens) | 3×1.1 +1×0.56 | 260 | 4 | 15 | 0.05 0.05 | <0.05 (2) | MP-SP-3139 |
| CA, 1989 (St Helens) | 1.1 | 260 | 4 | 21 | 0.06 0.06 | <0.05 (2) | MP-SP-3139 |
| TX, 1989 (Coho) | 5×1.1 +1×0.56 | 97 | 6 | 15 | <0.05 (2) | <0.05 (2) | MP-SP-3141 |
| TX, 1989 (Coho) | 1.1 | 97 | 6 | 21 | <0.05 (2) | <0.05 (2) | MP-SP-3141 |
| TX, 1989 (Coho) | 5×1.1 +1×0.56 | 51 | ( 6 | 15 | <0.05 (2) | <0.05 (2) | MP-SP-3143 |
| TX, 1989 (Coho) | 1.1 | 51 | ( 6 | 21 | <0.05 (2) | <0.05 (2) | MP-SP-3143 |
| CO, 1988 (Melody) | 4×1.1 +1×0.56 | 470 | 5 | 15 | 0.30 <0.05 | 0.10 <0.05 | MP-SP-3187 |
| CO, 1988 (Melody) | 1.1 | 470 | 5 | 21 | <0.05 (2) | <0.05 (2) | MP-SP-3187 |
| NJ, 1988 (America) | 5×1.1 +1×0.56 | 540 | 6 | 15 | 0.07 0.06 | <0.05 (2) | MP-SP-7123 |
| NJ, 1988 (America) | 1.1 | 540 | 6 | 21 | 0.07 0.09 | <0.05 (2) | MP-SP-7123 |

( aerial application.

Table 30. Residues in lima beans and snap beans from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| LIMA BEANS | | | | | | | | |
| DE, 1988 (Fordhook 242) | 1.7 | 290 | 6 | 21 | pod | <0.05 (2) | <0.05 (2) | MP-LB-7008 |
| DE, 1988 (Fordhook 242) | 1.7 | 50 | ( 6 | 21 | pod | <0.05 (2) | <0.05 (2) | MP-LB-7010 |
| CA, 1988 (Baby White) | 1.7 | 94 | ( 6 | 21 | pod | <0.05 (2) | <0.05 (2) | MP-LB-3083 |
| CA, 1988 (Baby White) | 1.7 | 340 | 6 | 21 | pod | <0.05 (2) | <0.05 (2) | MP-LB-3081 |
| SNAP BEANS | | | | | | | | |
| CA, 1988 (Blue Lake) | 1.7 | 330 | 6 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-3084 |
| OR, 1988 (O.S.U. 91G) | 1.7 | 190 | 6 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-3087 |
| CA, 1988 (Blue Lake) | 1.7 | 330 | 6 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-3508 |
| CA, 1988 (Blue Lake) | 3.4 | 330 | 6 | 21 | cut pod | <0.05 | <0.05 | MP-LB-3508 |
| CA, 1988 (Blue Lake) | 3.4 | 330 | 6 | 21 | cannery waste | <0.05 | <0.05 | MP-LB-3508 |
| OR, 1988 (O.S.U. 91G) | 1.7 | 94 | 6 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-3509 |
| OR, 1988 (O.S.U. 91G) | 7.0 | 94 | 6 | 21 | cut pod | <0.05 | <0.05 | MP-LB-3509 |
| OR, 1988 (O.S.U. 91G) | 7.0 | 94 | 6 | 21 | cannery waste | 0.06 | <0.05 | MP-LB-3509 |
| FL, 1988 (Sprite) | 1.7 | 300 | 6 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-7011 |
| NY, 1988 (Improved Tendergreen) | 1.7 | 280 | 6 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-7013 |
| WI, 1988 (Venture VB 4004) | 1.7 | 240 | 4 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-7016 |
| CA, 1988 (Blue Lake 274) | 1.7 | 94 | ( 6 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-3086 |
| NY, 1988 (Improved Tendergreen) | 1.7 | 46 | ( 6 | 21 | whole pod | <0.05 (2) | <0.05 (2) | MP-LB-7015 |

( aerial application.

Table 31. Residues in soya beans from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| IL, 1988 (BSR-201) | 0.56 | 240 | 2 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-SY-7117 |
| MN, 1988 (Agri-Pro 1776) | 0.56 | 240 | 2 | 15 | dry seed | <0.05 (2) | 0.05 <0.05 | MP-SY-7118 |

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| LA, 1988 (Forrest) | 0.56 | 140 | 2 | 14 | dry seed | <0.05 (2) | <0.05 (2) | MP-SY-7119 |
| GA, 1988 (Coker 488) | 0.56 | 58 | 2 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-SY-7120 |
| IA, 1988 (Pioneer 9271) | 0.56 | 200 | 2 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-SY-7121 |
| NC, 1988 (Asgrow A5149) | 0.56 | 220 | 2 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-SY-7122 |
| IA, 1988 (Pioneer 9271) | 0.56 | 200 | 2 | 15 | dry seed | <0.05 | <0.05 | MP-SY-2101 |
| MO, 1988 (Williams 82) | 0.56 | 200 | 2 | 15 | dry seed | <0.05 | <0.05 | MP-SY-2102 |
| IA, 1990 (DK 265) | 0.56 | 190 | 6 | 15 | seed | <0.05 (2) | <0.05 (2) | MP-SY-3525-IA |
| MO, 1990 (Williams 82) | 0.56 | 190 | 6 | 15 | seed | <0.05 (2) | <0.05 (2) | MP-SY-3525-MO |

Table 32. Residues in field peas from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| WA, 1989 (Dark Skin 49) | 1.1 | 190 | 6 | 15 | dry seed | 0.12 <u>0.19</u> | <u><0.05</u> (2) | MP-PE-3116 |
| WA, 1989 (Dark Skin 49) | 5×1.1 +1×0.56 | 190 | 6 | 10 | dry seed | <u>0.24</u> 0.10 | <u><0.05</u> (2) | MP-PE-3116 |
| WA, 1989 (Dark Skin 49) | 1.1 | 94 | ( 6 | 15 | dry seed | <u><0.05</u> (2) | <u><0.05</u> (2) | MP-PE-3118 |
| WA, 1989 (Dark Skin 49) | 5×1.1 +1×0.56 | 94 | ( 6 | 10 | dry seed | <u>0.06</u> 0.05 | <u><0.05</u> (2) | MP-PE-3118 |
| DE, 1988 (Alaska) | 1.1 | 290 | 6 | 15 | dry seed | <u>0.18</u> 0.15 | <u><0.05</u> (2) | MP-PE-7089 |
| DE, 1988 (Alaska) | 5×1.1 +1×0.56 | 290 | 6 | 10 | dry seed | <u>0.07</u> 0.07 | <u><0.05</u> (2) | MP-PE-7089 |
| MN, 1988 (Alaska 146) | 1.1 | 190 | 4 | 15 | dry seed | <u><0.05</u> (2) | <u><0.05</u> (2) | MP-PE-7091 |
| MN, 1988 (Alaska 146) | 3×1.1 +1×0.56 | 190 | 4 | 10 | dry seed | <u><0.05</u> (2) | <u><0.05</u> (2) | MP-PE-7091 |
| ND, 1988 (Trapper) | 1.1 | 94 | 6 | 15 | dry seed | <u>0.06</u> 0.06 | <u><0.05</u> (2) | MP-PE-7093 |
| ND, 1988 (Trapper) | 5×1.1 +1×0.56 | 94 | 6 | 10 | dry seed | <0.05 <u>0.06</u> | <u><0.05</u> (2) | MP-PE-7093 |
| DE, 1988 (Alaska) | 1.1 | 47 | ( 6 | 10<br>15 | dry seed | <0.05 (2)<br><u><0.05</u> (2) | <0.05 (2)<br><u><0.05</u> (2) | MP-PE-7095 |

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| DE, 1988 (Alaska) | 5×1.1 +1×0.56 | 47 | ( 6 | 10 | dry seed | 0.13 0.16 | <0.05 (2) | MP-PE-7095 |
| WA, 1989 (Knight) | 1.1 | 94 | ( 4 | 15 | pod | <0.05 (2) | <0.05 (2) | MP-PE-3188 |
| WA, 1989 (Knight) | 3×1.1 +1×0.56 | 94 | ( 4 | 10 | pod | <0.05 (2) | <0.05 (2) | MP-PE-3188 |
| DE, 1988 (Wando) | 1.1 | 290 | 6 | 15 | pod | 0.60 0.68 | <0.05 (2) | MP-PE-7096 |
| DE, 1988 (Wando) | 5×1.1 +1×0.56 | 290 | 6 | 10 | pod | 0.21 0.19 | <0.05 (2) | MP-PE-7096 |
| MN, 1988 (Asgrow XPG 206) | 1.1 | 190 | 4 | 10 | pod | 0.08 <0.05 | <0.05 (2) | MP-PE-7098 |
| MN, 1988 (Asgrow XPG 206) | 3×1.1 +1×0.56 | 190 | 4 | 10 | pod | <0.05 (2) | <0.05 (2) | MP-PE-7098 |
| WI, 1988 (Ego) | 1.1 | 240 | 5 | 14 | pod | <0.05 (2) | <0.05 (2) | MP-PE-7100 |
| WI, 1988 (Ego) | 4×1.1 +1×0.56 | 240 | 5 | 9 | pod | <0.05 (2) | <0.05 (2) | MP-PE-7100 |
| WI, 1988 (9888F) | 1.1 | 38 | ( 5 | 15 | pod | <0.05 (2) | <0.05 (2) | MP-PE-7102 |
| WI, 1988 (9888F) | 4×0.56 +1×1.1 | 38 | ( 5 | 10 | pod | <0.05 (2) | <0.05 (2) | MP-PE-7102 |
| WA, 1989 (Knight) | 3×1.1 +1×0.56 | 190 | 4 | 10 | pods | <0.05 (2) | <0.05 (2) | MP-PE-3189 |

( aerial application.

Table 33. Residues in dried beans from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Baby White) | 1.7 | 340 | 6 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-DB-3072 |
| CA, 1988 (Red Kidney) | 1.7 | 94 | ( 6 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-DB-3074 |
| MI, 1988 (Seafarer Navy Bean) | 1.7 | 220 | 6 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-DB-7001 |
| NE, 1988 (Pinto) | 1.7 | 190 | 6 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-DB-7003 |
| NE, 1988 (Pinto) | 1.7 | 9 | ( 6 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-DB-7005 |

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| ND, 1988 (Topaz, Pinto) | 1.7 | 94 | 6 | 15 | dry seed | <0.05 (2) | <0.05 (2) | MP-DB-7006 |

( aerial application.

Table 34. Residues in carrots from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| MI, 1988 (Scarlet Nantes) | 1.2 | 210 | 6 | 15 | 0.38 0.35 | <0.05 (2) | MP-CT-7027 |
| CA, 1989 (Pacmor) | 1.1 | 190 | 6 | 15 | 0.79 0.57 | <0.05 (2) | MP-CT-3058 |
| CA, 1989 (Pacmor) | 1.1 | 190 | ( 6 | 15 | 0.09 0.22 | <0.05 (2) | MP-CT-3060 |
| TX, 1988 (Danver) | 1.1 | 150 | 6 | 15 | 0.47 0.49 | <0.05 (2) | MP-CT-3061 |
| TX, 1988 (Danver) | 1.1 | 47 | ( 6 | 15 | 0.07 0.26 | <0.05 (2) | MP-CT-3063 |
| WA, 1988 (Nantes Coreless) | 1.1 | 280 | 6 | 15 | 0.65 <0.05 | <0.05 (2) | MP-CT-3064 |

( aerial application.

Table 35. Parathion-methyl and paraoxon-methyl residues in potatoes and processed commodities from supervised trials in the USA (Cañez, 1990p) and Poland. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Location, year (variety) | Application | | | | | PHI, days | sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| ID, USA, 1988 (Russet Burbank) | EC | 1.7 | | 190 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | MP-PO-3122 |
| CA, USA, 1989 (Red La Sota) | EC | 1.7 | | 230 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | MP-PO-3125 |
| CA, USA, 1989 (Red La Sota) | EC | 1.7 | | 160 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | MP-PO-3501 |
| CA, USA, 1989 (Red La Sota) | EC | 3.4 | | 160 | 6 | 5 | tuber<br>chips<br>flakes<br>granules<br>wet peel<br>dried peel | <0.05<br><0.05<br><0.05<br><0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05<br><0.05 | MP-PO-3501 |
| ID, USA, 1988 (Russet Burbank) | EC | 1.7 | | 190 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | MP-PO-3502 |

| Location, year (variety) | Application | | | | | PHI, days | sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| ID, USA, 1988 (Russet Burbank) | EC | 1.7 | | 94 | ( 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | MP-PO-3124 |
| ID, USA, 1988 (Russet Burbank) | EC | 8.4 | | 190 | 6 | 5 | tuber<br>chips<br>flakes<br>granules<br>wet peel<br>dried peel | <0.05<br><0.05<br><0.05<br><0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05<br><0.05<br><0.05<br><0.05 | MP-PO-3502 |
| FL, USA, 1988 (Red Lasoda) | EC | 1.7 | | 350 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | MP-PO-7103 |
| WI, USA, 1988 (Russet Burbank) | EC | 1.7 | | 190 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | MP-PO-7107 |
| ME, USA, 1988 (Atlantic) | EC | 1.8 | | 430 | 6 | 5 | tuber | <0.05 (2) | <0.05 (2) | MP-PO-7105 |
| Poland, 1995 (Bzura and Ceza) | CS | 0.27 | 0.09 | 300 | 4 | 33 | tuber | <0.02 | | VR 589 [1] |
| Poland, 1995 (Aster) | CS | 0.45 | 0.15 | 330 | 1 | 48 | tuber | <0.02 | | VR 589 [1] |
| Poland, 1995 (Arkadia) | CS | 0.45 | 0.15 | 330 | 2 | 49 | tuber | <0.02 | | VR 589 [1] |
| Poland, 1992 (Sokól) | CS | 0.30 | 0.10 | 300 | 1 | 62 | tuber | <0.01 | | VR 589 [1] |

[1]Only summary information available
( aerial application.

Table 36. Parathion-methyl and paraoxon-methyl residues in sugar beet and its processed commodities (Cañez, 1990q) from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| ID, 1988 (WS 88) | 0.42 | 190 | 6 | 20 | root | <0.05 (2) | <0.05 (2) | MP-SB-3144 |
| CA, 1988 (SS-NB2) | 0.42 | 190 | 6 | 20 | root | <0.05 (2) | <0.05 (2) | MP-SB-3146 |
| CA, 1988 (SS-NB2) | 0.42 | 190 | ( 6 | 20 | root | <0.05 (2) | <0.05 (2) | MP-SB-3148 |
| ID, 1988 (WS 88) | 0.42 | 94 | ( 6 | 20 | root | <0.05 (2) | <0.05 (2) | MP-SB-3186 |
| CA, 1988 (SS-NB2) | 0.42 | 190 | 6 | 20 | root | <0.05 (2) | <0.05 (2) | MP-SB-3503 |
| ID, 1988 (WS 88) | 0.42 | 190 | 6 | 20 | root | <0.05 (2) | <0.05 (2) | MP-SB-3504 |
| MN, 1988 (Ultramono) | 0.42 | 190 | 6 | 20 | root | <0.05 (2) | <0.05 (2) | MP-SB-7124 |

| Location, year (variety) | Application | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| ND, 1988 (ACS ACH 176) | 0.42 | 94 | 6 | 20 | root | <0.05 (2) | <0.05 (2) | MP-SB-7126 |
| CA, 1988 (SS-NB2) | 2.1 | 190 | 6 | 20 | root<br>pulp,<br>dehydrated<br>molasses<br>refined sugar | <0.05<br><0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05<br><0.05 | MP-SB-3503 |
| ID, 1988 (WS 88) | 2.1 | 190 | 6 | 20 | root<br>pulp,<br>dehydrated<br>molasses<br>refined sugar | <0.05<br><0.05<br><0.05<br><0.05 | <0.05<br><0.05<br><0.05<br><0.05 | MP-SB-3504 |

( aerial application.

Table 37. Residues in Chinese turnips from supervised trials in the USA.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Purple Top White Globe) | 0.90 | 190 | 6 | 21 | greens | 1.8 1.3 | 0.06 0.05 | MP-TU-3149 |
| CA, 1988 (Purple Top White Globe) | 4×0.90 +2×0.56 | 190 | 6 | 7 | greens | 3.8 2.6 | 0.13 0.09 | MP-TU-3149 |
| CA, 1988 (Purple Top White Globe) | 0.90 | 190 | 6 | 15 | root | <0.05 (2) | <0.05 (2) | MP-TU-3149 |
| CA, 1988 (Purple Top White Globe) | 4×0.90 +2×0.56 | 190 | 6 | 7 | root | 0.11 <0.05 | <0.05 (2) | MP-TU-3149 |
| CA, 1988 (Purple Top White Globe) | 0.90 | 190 | ( 6 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-TU-3151 |
| CA, 1988 (Purple Top White Globe) | 4×0.90 +2×0.56 | 190 | ( 6 | 7 | greens | 0.22 <0.05 | <0.05 (2) | MP-TU-3151 |
| CA, 1988 (Purple Top White Globe) | 0.90 | 190 | ( 6 | 15 | root | <0.05 (2) | <0.05 (2) | MP-TU-3151 |
| CA, 1988 (Purple Top White Globe) | 4×0.90 +2×0.56 | 190 | ( 6 | 7 | root | <0.05 (2) | <0.05 (2) | MP-TU-3151 |
| TX, 1988 (Purple Top White Globe) | 0.90 | 150 | 6 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-TU-3152 |
| TX, 1988 (Purple Top White Globe) | 4×0.90 +2×0.56 | 150 | 6 | 7 | greens | 0.16 0.20 | <0.05 (2) | MP-TU-3152 |
| TX, 1988 (Purple Top White Globe) | 0.90 | 150 | 6 | 15 | root | <0.05 (2) | <0.05 (2) | MP-TU-3152 |
| TX, 1988 (Purple Top White Globe) | 4×0.90 +2×0.56 | 150 | 6 | 7 | root | <0.05 (2) | <0.05 (2) | MP-TU-3152 |
| TX, 1988 (Purple Top White Globe) | 0.90 | 47 | ( 6 | 21 | greens | 0.07 0.08 | <0.05 (2) | MP-TU-3154 |

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (Purple Top White Globe) | 4×0.90 +2×0.56 | 47 | ( 6 | 7 | greens | 0.91 0.94 | 0.14 0.13 | MP-TU-3154 |
| TX, 1988 (Purple Top White Globe) | 0.90 | 47 | ( 6 | 14 | root | <0.05 (2) | <0.05 (2) | MP-TU-3154 |
| TX, 1988 (Purple Top White Globe) | 4×0.90 +2×0.56 | 47 | ( 6 | 7 | root | <0.05 (2) | <0.05 (2) | MP-TU-3154 |
| WA, 1988 (Purple Top) | 0.90 | 280 | 6 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-TU-3155 |
| WA, 1988 (Purple Top) | 4×0.90 +2×0.56 | 280 | 6 | 7 | greens | 0.07 <0.05 | 0.05 <0.05 | MP-TU-3155 |
| WA, 1988 (Purple Top) | 0.90 | 280 | 6 | 15 | root | <0.05 (2) | <0.05 (2) | MP-TU-3155 |
| WA, 1988 (Purple Top) | 4×0.90 +2×0.56 | 280 | 6 | 7 | root | <0.05 (2) | <0.05 (2) | MP-TU-3155 |
| GA, 1988 (Purple Globe) | 0.90 | 290 | 6 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-TU-7130 |
| GA, 1988 (Purple Globe) | 4×0.90 +2×0.56 | 290 | 6 | 7 | greens | <0.05 (2) | <0.05 (2) | MP-TU-7130 |
| GA, 1988 (Purple Globe) | 0.90 | 290 | 6 | 15 | root | <0.05 (2) | <0.05 (2) | MP-TU-7130 |
| GA, 1988 (Purple Globe) | 4×0.90 +2×0.56 | 290 | 6 | 7 | root | <0.05 (2) | <0.05 (2) | MP-TU-7130 |
| NJ, 1988 (Purple Top White globe) | 0.90 | 280 | 6 | 21 | greens | <0.05 (2) | <0.05 (2) | MP-TU-7132 |
| NJ, 1988 (Purple Top White globe) | 4×0.90 +2×0.56 | 280 | 6 | 7 | greens | <0.05 (2) | <0.05 (2) | MP-TU-7132 |
| NJ, 1988 (Purple Top White globe) | 0.90 | 280 | 6 | 15 | root | <0.05 (2) | <0.05 (2) | MP-TU-7132 |
| NJ, 1988 (Purple Top White globe) | 4×0.90 +2×0.56 | 280 | 6 | 7 | root | <0.05 (2) | 0.07 <0.05 | MP-TU-7132 |

( aerial application.

Table 38. Residues in celery from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Florida 683) | 0.56 | 380 | 2 | 15<br>22 | excluding foliage | 0.34 0.40<br>0.17 0.29 | <0.05 (2)<br><0.05 (2) | MP-CY-3066 |
| CA, 1988 (Florida 683) | 1.1 | 380 | 2 | 15<br>22 | excluding foliage | 1.3 1.2<br>0.28 0.65 | <0.05 (2)<br><0.05 (2) | MP-CY-3066 |

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Florida 683) | 0.56 | 380 | 2 | 15<br>22 | including foliage | 1.7 2.5<br>1.0 1.1 | <0.05 (2)<br><0.05 (2) | MP-CY-3066 |
| CA, 1988 (Florida 683) | 1.1 | 380 | 2 | 15<br>22 | including foliage | 2.2 2.5<br>2.1 1.5 | <0.05 (2)<br><0.05 (2) | MP-CY-3066 |
| CA, 1988 (5270R) | 0.56 | 190 | ( 2 | 15<br>22 | excluding foliage | 0.06 0.06<br>0.20 0.05 | <0.05 (2)<br><0.05 (2) | MP-CY-3067 |
| CA, 1988 (5270R) | 1.1 | 190 | ( 2 | 15<br>22 | excluding foliage | 0.06 0.17<br>0.09 0.07 | <0.05 (2)<br><0.05 (2) | MP-CY-3067 |
| CA, 1988 (5270R) | 0.56 | 190 | ( 2 | 15<br>22 | including foliage | 1.2 1.3<br>0.09 0.29 | <0.05 (2)<br><0.05 (2) | MP-CY-3067 |
| CA, 1988 (5270R) | 1.1 | 190 | ( 2 | 15<br>22 | including foliage | 1.5 2.1<br>1.2 0.87 | 0.07 0.07<br><0.05 (2) | MP-CY-3067 |
| CA, 1988 (Florida 683) | 0.56 | 380 | 2 | 15<br>22 | excluding foliage | 0.81 0.86<br>0.69 0.69 | <0.05 (2)<br><0.05 (2) | MP-CY-3068 |
| CA, 1988 (Florida 683) | 1.1 | 380 | 2 | 15<br>22 | excluding foliage | 1.1 1.1<br>1.7 1.1 | <0.05 (2)<br><0.05 (2) | MP-CY-3068 |
| CA, 1988 (Florida 683) | 0.56 | 380 | 2 | 15<br>22 | including foliage | 1.8 2.0<br>1.6 1.7 | <0.05 (2)<br><0.05 (2) | MP-CY-3068 |
| CA, 1988 (Florida 683) | 1.1 | 380 | 2 | 15<br>22 | including foliage | 3.8 4.4<br>2.3 2.5 | <0.05 (2)<br><0.05 (2) | MP-CY-3068 |
| CA, 1989 (5275) | 0.56 | 260 | 2 | 15<br>22 | excluding foliage | 0.28 0.54<br>0.10 0.12 | <0.05 (2)<br><0.05 (2) | MP-CY-3070 |
| CA, 1989 (5275) | 1.1 | 260 | 2 | 15<br>22 | excluding foliage | 0.41 0.32<br>0.21 0.20 | <0.05 (2)<br><0.05 (2) | MP-CY-3070 |
| CA, 1989 (5275) | 0.56 | 260 | 2 | 15<br>22 | including foliage | 1.6 1.3<br>0.98 0.77 | <0.05 (2)<br><0.05 (2) | MP-CY-3070 |
| CA, 1989 (5275) | 1.1 | 260 | 2 | 15<br>22 | including foliage | 0.98 1.8<br>1.6 1.3 | <0.05 (2)<br><0.05 (2) | MP-CY-3070 |
| FL, 1989 (683) | 1.1 | 280 | 2 | 15<br>22 | including foliage | 4.0 3.2<br>1.9 2.8 | <0.05 (2)<br><0.05 (2) | MP-CY-7029 |
| MI, 1988 (Utah Tall 52-70R1MP) | 1.1 | 200 | 2 | 15<br>22 | including foliage | 3.4 4.7<br>2.4 4.3 | <0.05 (2)<br><0.05 (2) | MP-CY-7031 |
| NY, 1988 (Florida 683) | 1.1 | 230 | 2 | 15<br>22 | including foliage | 3.2 3.9<br>3.6 4.4 | <0.05 (2)<br><0.05 (2) | MP-CY-7033 |
| NY, 1988 (Florida 683) | 1.1 | 45 | ( 2 | 15<br>22 | including foliage | 0.76 0.87<br>0.83 0.84 | <0.05 (2)<br><0.05 (2) | MP-CY-7035 |

( aerial application.

Table 39. Residues in artichokes from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Green Globe) | 1.1 | 1870 | 4 | 7 | head | 1.8 0.41 | <0.05 (2) | MP-AR-3178 |
| CA, 1988 (Green Globe) | 1.1 | 470 | 4 | 7 | head | 1.3 0.98 | <0.05 (2) | MP-AR-3178 |
| CA, 1988 (Green Globe) | 1.1 | 1870 | 4 | 7 | head | 1.2 1.1 | <0.05 (2) | MP-AR-3176 |
| CA, 1988 (Green Globe) | 1.1 | 470 | 4 | 7 | head | 0.87 1.1 | <0.05 (2) | MP-AR-3176 |

Table 40. Residues in maize grain from supervised trials in the USA. Double-underlined residues are from treatments at twice the label rate and are valid for estimating maximum residue levels. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| CA, 1989 | 1.1 | 300 | 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-3169 |
| TX, 1988 (Dekalb 689) | 1.1 | 47 | ( 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-3170 |
| TX, 1988 (Dekalb 689) | 1.1 | 75 | 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-3171 |
| TX, 1988 (Dekalb 689) | 1.1 | 75 | 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-3512 |
| MO, 1989 (Funks G-4500) | 1.1 | 190 | 6 | 12 | <0.05 | <0.05 | MP-CN-3524 |
| GA, 1988 (Cargill Hybrid) | 1.1 | 56 | 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-7042 |
| IL, 1988 (F/S 2368A) | 1.1 | 160 | 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-7043 |
| MN, 1988 (Pioneer 3732) | 1.1 | 230 | 6 | 12 | 0.06 0.09 | <0.05 (2) | MP-CN-7044 |
| VA, 1988 (SX383) | 1.1 | 220 | 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-7045 |
| NE, 1988 (Funks G-4440) | 1.1 | 190 | 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-7046 |
| OH, 1988 (Pioneer 3324) | 1.1 | 100 | 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-7047 |
| OH, 1988 (Pioneer 3324) | 1.1 | 47 | ( 6 | 12 | <0.05 (2) | <0.05 (2) | MP-CN-7048 |

( aerial application.

Table 41. Residues in rice from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (L202) | 0.89 | 75 | ( 6 | 15<br>20 | grain | 2.2 2.3<br>c 0.34 | 0.16 0.17<br>c <0.05 | MP-RI-3131 |
| TX, 1988 (Lemont) | 0.89 | 47 | ( 6 | 15 | grain | 0.19 0.18 | 0.09 0.08 | MP-RI-3132 |
| CA, 1988 (M201) | 0.88 | 190 | 6 | 16 | grain | 2.0 1.9 | 0.10 0.12 | MP-RI-3514 |
| TX, 1988 (Gulfmont) | 0.88 | 170 | 6 | 15 | grain | 0.36 0.44 | 0.18 0.23 | MP-RI-3515 |
| AR, 1988 (V 7817) | 0.89 | 47 | ( 6 | 15 | grain | 0.27 0.25 | 0.17 0.15 | MP-RI-7109 |
| LA, 1988 (Lemont) | 0.89 | 94 | ( 6 | 15 | grain | 0.28 0.30 | 0.12 0.12 | MP-RI-7110 |
| CA, 1988 (L202) | 0.89 | 75 | ( 6 | 15 | straw | 7.3 7.5 | 0.11 0.15 | MP-RI-3131 |
| TX, 1988 (Lemont) | 0.89 | 47 | ( 6 | 15 | straw | <0.05 (2) | <0.05 (2) | MP-RI-3132 |
| AR, 1988 (V 7817) | 0.89 | 47 | ( 6 | 15 | straw | <0.05 0.06 | <0.05 (2) | MP-RI-7109 |
| LA, 1988 (Lemont) | 0.89 | 94 | ( 6 | 15 | straw | 0.20 0.13 | 0.05 <0.05 | MP-RI-7110 |

( aerial application. c: sample from control plot

Table 42. Residues in sorghum grain from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (TEY 75) | 1.1 | 150 | 6 | 21 | 1.0 0.16 | 0.07 <0.05 | MP-SG-3173 |
| TX, 1988 (Asgrow Topaz) | 1.1 | 47 | 6 | 21 | 0.36 0.20 | <0.05 (2) | MP-SG-3174 |
| TX, 1988 (Asgrow Topaz) | 1.1 | 9 | ( 6 | 21 | 0.09 0.08 | <0.05 (2) | MP-SG-3175 |
| MO, 1988 (Funk's G-623GBR) | 1.1 | 200 | 6 | 21 | 0.27 0.22 | <0.05 (2) | MP-SG-7111 |
| MO, 1988 (Funk's G-623GBR) | 1.1 | 22 | ( 6 | 21 | 0.09 0.19 | <0.05 (2) | MP-SG-7112 |
| NE, 1988 (NC + 271) | 1.1 | 190 | 6 | 21 | 0.40 0.36 | <0.05 (2) | MP-SG-7113 |

( aerial application. c: sample from control plot

Table 43. Residues in wheat grain from supervised trials in the USA (LeRoy, 1990d). Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels. All EC formulations.

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| MO, 1988 (Caldwell) | 1.4 | 260 | 6 | 13 | 0.78 | <0.05 | MP-WH-2103 |
| CA, 1989 (Anza, Hard red winter) | 1.4 | 190 | 6 | 14 | 4.5 5.1 | 0.45 0.49 | MP-WH-3157 |
| CA, 1989 (Anza, Hard red winter) | 2×1.4 +3×0.84 +1×0.28 | 190 | 6 | 0 | 4.4 3.5 | 0.17 0.17 | MP-WH-3157 |
| CA, 1989 (Anza, Hard red winter) | 4×1.4 +2×0.84 | 190 | 6 | 14 | 3.2 <u>3.7</u> | 0.36 <u>0.39</u> | MP-WH-3157 |
| WA, 1989 (Rojo) | 1.4 | 190 | 6 | 14 | 0.82 1.1 | <0.05 (2) | MP-WH-3159 |
| WA, 1989 (Rojo) | 2×1.4 +3×0.84 +1×0.28 | 190 | 6 | 0 | 1.1 1.1 | <0.05 (2) | MP-WH-3159 |
| WA, 1989 (Rojo) | 4×1.4 +2×0.84 | 190 | 6 | 14 | <u>1.1</u> 1.0 | <u>0.09</u> 0.08 | MP-WH-3159 |
| TX, 1989 (NK Pro 812) | 1.4 | 47 | 6 | 14 | 0.15 0.13 | <0.05 (2) | MP-WH-3161 |
| TX, 1989 (NK Pro 812) | 2×1.4 +3×0.84 +1×0.28 | 47 | 6 | 0 | 0.07 0.09 | <0.05 (2) | MP-WH-3161 |
| TX, 1989 (NK Pro 812) | 4×1.4 +2×0.84 | 47 | 6 | 14 | <0.05 <u>0.05</u> | <<u>0.05</u> (2) | MP-WH-3161 |
| WA, 1989 (Rojo) | 1.4 | 94 | ( 6 | 14 | 0.33 0.29 | <0.05 (2) | MP-WH-3181 |
| WA, 1989 (Rojo) | 2×1.4 +3×0.84 +1×0.28 | 94 | ( 6 | 0 | 0.28 0.24 | <0.05 (2) | MP-WH-3181 |
| WA, 1989 (Rojo) | 4×1.4 +2×0.84 | 94 | ( 6 | 14 | 0.27 <u>0.48</u> | <0.05 <u>0.05</u> | MP-WH-3181 |
| WA, 1989 (Rojo) | 1.4 | 190 | 6 | 14 | 0.88 0.74 | 0.05 0.05 | MP-WH-3520 |
| OH, 1988 (Becher) | 1.4 | 190 | 6 | 14 | 0.35 0.32 c <0.05 | <0.05 (2) c 0.06 | MP-WH-7134 |
| OH, 1988 (Becher) | 2×1.4 +3×0.84 +1×0.28 | 190 | 6 | 0 | 0.65 0.51 | 0.08 0.06 | MP-WH-7134 |
| OH, 1988 (Becher) | 4×1.4 +2×0.84 | 190 | 6 | 14 | <u>0.29</u> 0.27 | <<u>0.05</u> (2) | MP-WH-7134 |

| Location, year (variety) | Application | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | parathion-methyl | paraoxon-methyl | |
| VA, 1988 (Coker 797) | 1.4 | 220 | 6 | 14 | <0.05 (2) | <0.05 (2) | MP-WH-7136 |
| VA, 1988 (Coker 797) | 2×1.4 +3×0.84 +1×0.28 | 220 | 6 | 0 | 1.5 1.5 | <0.05 (2) | MP-WH-7136 |
| VA, 1988 (Coker 797) | 4×1.4 +2×0.84 | 220 | 6 | 14 | <0.05 (2) | <0.05 (2) | MP-WH-7136 |
| ND, 1988 (Marshall) | 1.4 | 94 | 6 | 14 | <0.05 (2) | <0.05 (2) | MP-WH-7138 |
| ND, 1988 (Marshall) | 2×1.4 +3×0.84 +1×0.28 | 94 | 6 | 0 | 0.49 0.25 | <0.05 (2) | MP-WH-7138 |
| ND, 1988 (Marshall) | 4×1.4 +2×0.84 | 94 | 6 | 14 | <0.05 (2) | <0.05 (2) | MP-WH-7138 |
| KS, 1989 (Arkan) | 1.4 | 210 | 6 | 14 | 1.2 1.6 | <0.05 (2) | MP-WH-7140 |
| KS, 1989 (Arkan) | 2×1.4 +3×0.84 +1×0.28 | 210 | 6 | 0 | 0.73 0.93 | 0.05 0.05 | MP-WH-7140 |
| KS, 1989 (Arkan) | 4×1.4 +2×0.84 | 210 | 6 | 14 | 1.1 0.83 | 0.05 <0.05 | MP-WH-7140 |
| KS, 1989 (Caldwell) | 1.4 | 47 | ( 6 | 14 | 0.22 0.21 | <0.05 (2) | MP-WH-7142 |
| KS, 1989 (Caldwell) | 2×1.4 +3×0.84 +1×0.28 | 47 | ( 6 | 0 | 1.4 0.98 | 0.34 0.28 | MP-WH-7142 |
| KS, 1989 (Caldwell) | 4×1.4 +2×0.84 | 47 | ( 6 | 14 | 0.21 0.08 | <0.05 (2) | MP-WH-7142 |

( aerial application. c: sample from control plot

Table 44. Residues in cotton seed from supervised trials in the USA. All EC formulations.

| Location, year (variety) | Application | | | Growth stage | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | | sample | parathion-methyl | paraoxon-methyl | |
| AR, 1998 (Paymaster 1220) | 3.4 | 28 | 10 | 90% open | 1 | seed | 1.5 1.5 | 0.02 0.02 | 9802.AR.CT.02(a) |
| AR, 1998 (Paymaster 1220) | 3.4 | 9 | ( 10 | 90% open | 1 | seed | 2.0 1.8 | 0.02 0.01 | 9802.AR.CT.02(b) |
| CA, 1998 (Acula Maxxa) | 3.4 | 9 | ( 10 | defoliated | 1 | seed | 1.7 1.5 | 0.01 0.01 | 9802.CA.CT.10 |
| CA, 1998 (Acula Maxxa) | 3.4 | 280 | 10 | defoliated | 1 | seed | 4.4 4.6 | 0.03 0.04 | 9802.CA.CT.10 |

| Location, year (variety) | Application | | | Growth stage | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1998 (Maxxa) | 3.4 | 280 | 10 | 70-80% open | 1 | seed | 7.4 6.9 | 0.08 0.07 | 9802.CA.CT.11 |
| CA, 1998 (Maxxa) | 3.4 | 190 | 10 | mature | 1 | seed | 8.9 8.1<br>c 0.01 | 0.21 0.17<br>c <0.01 | 9802.CA.CT.12 |
| GA, 1998 (Paymaster 1220) | 3.4 | 190 | 10 | open bolls | 1 | seed | 3.2 2.0<br>c 0.02 | 0.03 0.02<br>c <0.01 | 9802.GA.CT.01 |
| LA, 1998 (Stoneville 4740) | 3.4 | 9 | ( 10 | 90% open | 1 | seed | 7.0 7.4 | 0.03 0.04 | 9802.LA.CT.03 |
| LA, 1998 (Stoneville 4740) | 3.4 | 93 | 10 | 90% open | 1 | seed | 5.5 5.6 | 0.03 0.04 | 9802.LA.CT.03 |
| LA, 1998 (Stoneville 4740) | 3.4 | 94 | 10 | 90% open | 1 | seed | 5.4 4.6 | 0.03 0.03 | 9802.LA.CT.04 |
| TX, 1998 (Paymaster 1220) | 3.4 | 94 | 10 | harvestable | 1 | seed | 1.5 0.87 | 0.03 0.02 | 9802.TX.CT.05 |
| TX, 1998 (Paymaster 1220) | 3.4 | 92 | 10 | 90% open | 1 | seed | 2.1 2.5 | 0.02 0.02 | 9802.TX.CT.06(a) |
| TX, 1998 (Paymaster 1220) | 3.4 | 8 | ( 10 | 90% open | 1 | seed | 3.6 3.9 | 0.03 0.04 | 9802.TX.CT.06(b) |
| TX, 1998 (PM 2200 RR) | 3.4 | 94 | 10 | 90% open | 1 | seed | 0.64 0.64 | 0.02 0.02 | 9802.TX.CT.07 |
| TX, 1998 (Quikee) | 3.4 | 120 | 10 | 85% open | 1 | seed | 3.5 2.9<br>c 0.02 | 0.01 0.01<br>c <0.01 | 9802.TX.CT.08(a) |
| TX, 1998 (Quikee) | 3.4 | 9 | ( 10 | 85% open | 1 | seed | 1.7 1.9 | <0.01 (2) | 9802.TX.CT.08(b) |
| TX, 1998 (Paymaster 2200 | 3.4 | 120 | 10 | 95% open | 1 | seed | 5.4 6.8<br>c 0.01 | 0.02 0.02<br>c <0.01 | 9802.TX.CT.09(a) |
| TX, 1998 (Paymaster 2200 | 3.4 | 9 | ( 10 | 95% open | 1 | seed | 3.0 2.8 | 0.02<br><0.01 | 9802.TX.CT.09(b) |
| CA, 1988 (GC 510) | 3.4 | 190 | 10 | harvestable | 7 | seed [1] | <0.05 (2) | <0.05 (2) | MP-CS-3052 |
| CA, 1988 (GC 510) | 3.4 | 94 | ( 10 | harvestable | 7 | seed [1] | <0.05 (2) | <0.05 (2) | MP-CS-3054 |
| TX, 1988 (DPL41) | 3.4 | 94 | 10 | 80% open bolls | 7 | seed [1] | 0.91 1.4 | <0.05 (2) | MP-CS-3055 |
| TX, 1988 (DPL41) | 3.4 | 9 | ( 10 | 80% open bolls | 7 | seed [1] | 0.9 1.2 | <0.05 (2) | MP-CS-3057 |
| CA, 1988 (GC 510) | 8×3.4<br>+2×1.1 | 190 | 10 | harvestable | 0 | seed [1] | <0.05 (2) | <0.05 (2) | MP-CS-3052 |
| CA, 1988 (GC 510) | 8×3.4<br>+2×1.1 | 94 | ( 10 | harvestable | 0 | seed [1] | <0.05 (2) | <0.05 (2) | MP-CS-3054 |
| TX, 1988 (DPL41) | 8×3.4<br>+2×1.1 | 94 | 10 | 80% open bolls | 0 | seed [1] | 3.1 4.5 | 0.11 0.14 | MP-CS-3055 |

| Location, year (variety) | Application | | | Growth stage | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | | sample | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (DPL41) | 8×3.4<br>+2×1.1 | 9 | ( 10 | 80% open bolls | 0<br>0 | seed [1] | 2.0 2.2<br>c 0.08 | <0.05 (2)<br>c <0.05 | MP-CS-3057 |
| CA, 1989 (GC 510) | 3.4 | 99 | 6 | | 7 | seed | 9.5 22 | <0.05<br>0.07 | MP-CS-3522 |
| TX, 1988 (DPL 41) | 3.4 | 94 | 10 | | 7 | seed | 0.90 1.8<br>c 0.08<br>0.08 | 0.07 0.10<br>c <0.05<br>(2) | MP-CS-3523 |

[1] Sample described as 'seed cotton' in report, but is probably cotton seed.
( aerial application. c: sample from control plot

Table 45. Residues in oilseed from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels. All EC formulations.

| Location, year (variety) | Application | | | growth stage | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | | sample | parathion-methyl | paraoxon-methyl | |
| CANOLA | | | | | | | | | |
| ND, 1992 (Legend) | 2×0.28 + 2×0.56 | 47 | ( 4 | 110 cm tall | 28 | seed | <0.05 (2) | <0.05 (2) | 92146a |
| ND, 1992 (Legend) | 2×0.56 + 2×1.1 | 47 | ( 4 | 110 cm tall | 28 | seed | <0.05 (2) | <0.05 (2) | 92146a |
| MT, 1992 (Tobin) | 0.56 | 51 | ( 2 | flowering | 28 | seed | <0.05 (2) | <0.05 (2) | 92146b |
| MT, 1992 (Tobin) | 1.1 | 51 | ( 2 | flowering | 28 | seed | 0.063 0.056 | <0.05 (2) | 92146b |
| WA, 1992 (Series) | 0.56 | 51 | ( 2 | post-bloom | 28 | seed | <0.05 (2) | <0.05 (2) | 92146c |
| WA, 1992 (Series) | 1.1 | 51 | ( 2 | post-bloom | 28 | seed | <0.05 (2) | <0.05 (2) | 92146c |
| ID, 1992 (#104) | 0.56 | 41 | ( 2 | 5% bloom | 28 | seed | <0.05 (2) | <0.05 (2) | 92146d |
| ID, 1992 (#104) | 1.1 | 41 | ( 2 | post-bloom | 28 | seed | <0.05 (2) | <0.05 (2) | 92146d |
| SUNFLOWER SEED | | | | | | | | | |
| ND, 1988 (Sigco Hybrid 465A) | 1.1<br>1.1 | 190<br>47 | 3<br>3 | post-flower | 30<br>30 | seed (with shell) | <0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2) | MP-SS-7128 |
| ND, 1988 (Sigco Hybrid 465A) | 1.1 | 38 | ( 3 | post-flower | 30 | seed (with shell) | <0.05 (2) | <0.05 (2) | MP-SS-7129 |

( aerial application.

Table 46. Residues in alfalfa from supervised trials in the USA. In the 1998 trials, two applications per cutting were made at 4 ± 1 day intervals. Forage and hay samples were collected 15 days after the second application per cutting, and from 3 cuttings except in a Montana trial (2 cuttings) and a

California trial (4 cuttings). Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | % moisture[2] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water l/ha | no. per cut | | | | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Cuff-101) | EC | 1.4 | 370 | 2 | 83 | seed | | <0.05 (2) | <0.05 (2) | MP-AF-3001 |
| CA, 1988 (Cuff-101) | EC | 1.4 | 190 | ( 1 | 85 | seed | | <0.05 (2) | <0.05 (2) | MP-AF-3003 |
| WA, 1988 (La Rocca) | EC | 1.4 | 240 | 2 | 92 | seed | | <0.05 (2) | <0.05 (2) | MP-AF-3004 |
| WA, 1988 (La Rocca) | EC | 1.4 | 140 | ( 2 | 92 | seed | | <0.05 (2) | <0.05 (2) | MP-AF-3006 |
| MT, 1998 (Cenex 741) | EC | 1.1 | 28 | 2<br>2 | 15<br>15 | forage 1<br>forage 2 | 74.2<br>69.7 | 2.1 2.0<br>1.6 1.2 | 0.03 0.03<br>0.04 0.03 | 9801.MT.AF.10-a |
| MT, 1998 (Cenex 741) | EC | 1.1 | 9 | ( 2<br>( 2 | 15<br>15 | forage 1<br>forage 2 | 74.2<br>69.7 | 1.7 1.8<br>0.82 0.39 | 0.03 0.04<br>0.01 <0.01 | 9801.MT.AF.10-b |
| MT, 1998 (NK 919) | EC | 1.1 | 28 | 2<br>2 | 15<br>15 | forage 1<br>forage 2 | 72.7<br>53.2 | 0.97 1.1<br>0.46 0.36 | 0.02 0.02<br><0.01 (2) | 9801.MT.AF.09-a |
| MT, 1998 (NK 919) | EC | 1.1 | 9 | ( 2<br>( 2 | 15<br>15 | forage 1<br>forage 2 | 72.7<br>53.2 | 1.0 1.4<br>0.22 0.24 | 0.02 0.03<br><0.01 (2) | 9801.MT.AF.09-b |
| IA, 1998 (Winnbrand) | EC | 1.1 | 110 | 2<br>2<br>2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 81.0<br>77.2<br>81.7 | 0.96 1.1<br>0.82 0.84<br>5.9 3.6 | 0.01 0.01<br><0.01 (2)<br>0.06 0.05 | 9801.IA.AF.08 |
| MN, 1998 (Pioneer 5347LH) | EC | 1.1 | 110 | 2<br>2<br>2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 85.2<br>77.9<br>81.7 | 0.76 0.66<br>0.97 1.0<br>2.4 11 | <0.01 (2)<br>0.01 0.01<br>0.03 0.08 | 9801.MN.AF.07 |
| MN, 1998 (Pioneer) | EC | 1.1 | 46 | 2<br>2<br>2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 73.9<br>74.5<br>75.6 | 0.66 0.61<br>0.31 0.22<br>0.92 0.67 | 0.02 0.01<br><0.01 (2)<br>0.02 0.01 | 9801.MN.AF.06-a |
| MN, 1998 (Pioneer) | EC | 1.1 | 9 | ( 2<br>( 2<br>( 2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 73.9<br>74.5<br>75.6 | 8.6 6.0<br>2.7 0.42<br>2.3 1.3 | 0.11 0.08<br>0.02 <0.01<br>0.03 0.01 | 9801.MN.AF.06-b |
| OH, 1998 | EC | 1.1 | 82 | 2<br>2<br>2 | 14<br>14<br>14 | forage 1<br>forage 2<br>forage 3 | 75.4<br>76.5<br>78.6 | 0.61 0.70<br>0.74 0.65<br>1.3 1.5 | <0.01 0.01<br>0.01 <0.01<br>0.02 0.02 | 9801.OH.AF.04 |
| OH, 1998 (Mustang) | EC | 1.1 | 80 | 2<br>2<br>2 | 14<br>15<br>16 | forage 1<br>forage 2<br>forage 3 | 73.0<br>74.8<br>68.1 | 0.27 0.25<br>0.87 0.66<br>2.0 1.8 | <0.01 (2)<br>0.01 <0.01<br>0.02 0.02 | 9801.OH.AF.03-a |
| OH, 1998 (Mustang) | EC | 1.1 | 9 | ( 2<br>( 2<br>( 2 | 14<br>15<br>16 | forage 1<br>forage 2<br>forage 3 | 73.0<br>74.8<br>68.1 | 0.09 0.13<br>0.21 0.17<br>0.73 0.72 | <0.01 (2)<br><0.01 (2)<br>0.01 0.01 | 9801.OH.AF.03-b |
| PA, 1998 | EC | 1.1 | 270 | 2<br>2<br>2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 68.6<br>77.2<br>72.5 | 1.2 1.2<br>0.30 0.32<br>0.91 0.78 | 0.02 0.02<br>0.01 (2)<br>0.01 0.01 | 9801.PA.AF.01 |

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | % moisture[2] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water l/ha | no. per cut | | | | parathion-methyl | paraoxon-methyl | |
| VA, 1998 | EC | 1.1 | 250 | 2<br>2<br>2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 72.1<br>78.7<br>81.5 | 0.26 0.24<br>0.35 0.29<br>0.54 0.24 | <0.01 (2)<br><0.01 (2)<br><0.01 (2) | 9801.VA.AF.02 |
| WA, 1998 (Pioneer 5364) | EC | 1.1 | 28 | 2<br>2<br>2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 82.7<br>78.1<br>83.6 | 2.6 1.9<br>0.38 0.35<br>0.03 0.03 | 0.04 0.03<br><0.01 (2)<br><0.01 (2) | 9801.WA.AF.11 |
| WA, 1998 (Pioneer 5364) | EC | 1.1 | 9 | ( 2<br>( 2<br>( 2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 82.7<br>78.1<br>83.6 | 1.6 1.7<br>0.39 0.21<br>0.09 0.08 | 0.02 0.03<br><0.01 (2)<br><0.01 (2) | 9801.WA.AF.11 |
| WI, 1998 (Wrangler) | EC | 1.1 | 47 | 2<br>2<br>2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 79.7<br>77.5<br>76.5 | 1.1 0.67<br>0.49 0.57<br>11 5.9 | 0.02 0.02<br>0.01 0.02<br>0.13 0.09 | 9801.WI.AF.05-a |
| WI, 1998 (Wrangler) | EC | 1.1 | 9 | ( 2<br>( 2<br>( 2 | 15<br>15<br>15 | forage 1<br>forage 2<br>forage 3 | 79.7<br>77.5<br>76.5 | 2.2 1.8<br>0.34 0.55<br>8.5 6.9 | 0.03 0.03<br><0.01 0.01<br>0.11 0.10 | 9801.WI.AF.05-b |
| CA, 1998 (GT13R) | EC | 1.1 | 92 | 2<br>2<br>2<br>2 | 15<br>15<br>15<br>15 | forage 1<br>forage 2<br>forage 3<br>forage 4 | 78.2<br>70.9<br>64.6<br>83.6<br>64.6 | 0.57 0.91<br>0.23 0.32<br>0.89 1.3<br>6.8 6.0<br>c 0.02 | 0.01 0.03<br><0.01 (2)<br><0.01 0.01<br>0.11 0.10<br>c <0.01 | 9801.CA.AF.12 |
| MT, 1998 (Cenex 741) | EC | 1.1 | 28 | 2<br>4 | 15<br>15 | hay 1<br>hay 2 | 25.9<br>45.5 | 1.4 1.7<br>0.64 0.53 | 0.03 0.03<br>0.01 0.01 | 9801.MT.AF.10-a |
| MT, 1998 (Cenex 741) | EC | 1.1 | 9 | ( 2<br>( 2 | 15<br>15 | hay 1<br>hay 2 | 25.9<br>45.5 | 1.6 1.7<br>0.79 0.63 | 0.04 0.03<br>0.01 0.01 | 9801.MT.AF.10-b |
| MT, 1998 (NK 919) | EC | 1.1 | 28 | 2<br>2 | 15<br>15 | hay 1<br>hay 2 | 34.0<br>28.6 | 3.4 2.6<br>0.26 0.28 | 0.04 0.03<br><0.01 (2) | 9801.MT.AF.09-a |
| MT, 1998 (NK 919) | EC | 1.1 | 9 | ( 2<br>( 2 | 15<br>15 | hay 1<br>hay 2 | 34.0<br>28.6 | 2.1 2.3<br>0.38 0.39 | 0.03 0.03<br>0.01 (2) | 9801.MT.AF.09-b |
| IA, 1998 (Winnbrand) | EC | 1.1 | 110 | 2<br>2<br>2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 44.2<br>30.7<br>59.9 | 2.1 2.1<br>1.9 1.6<br>5.6 6.4 | 0.01 0.02<br>0.01 0.01<br>0.06 0.07 | 9801.IA.AF.08 |
| MN, 1998 (Pioneer 5347LH) | EC | 1.1 | 110 | 2<br>2<br>2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 66.3<br>38.5<br>59.3<br>59.3 | 1.3 1.3<br>1.3 1.3<br>4.4 23<br>c 0.02 | 0.01 0.01<br>0.01 <0.01<br>0.04 0.13<br>c <0.01 | 9801.MN.AF.07 |
| MN, 1998 (Pioneer) | EC | 1.1 | 46 | 2<br>2<br>2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 36.8<br>42.7<br>21.2 | 2.2 2.0<br>0.67 0.36<br>4.5 3.6 | 0.06 0.05<br><0.01 (2)<br>0.08 0.07 | 9801.MN.AF.06-a |
| MN, 1998 (Pioneer) | EC | 1.1 | 9 | ( 2<br>( 2<br>( 2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 36.8<br>42.7<br>21.2 | 17 16<br>1.4 1.1<br>8.4 8.8 | 0.17 0.18<br><0.01 (2)<br>0.07 0.08 | 9801.MN.AF.06-b |

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | % moisture[2] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water l/ha | no. per cut | | | | parathion-methyl | paraoxon-methyl | |
| OH, 1998 | EC | 1.1 | 82 | 2<br>2<br>2 | 14<br>14<br>14 | hay 1<br>hay 2<br>hay 3 | 24.9<br>6.9<br>24.3 | 1.1 1.3<br>1.4 1.5<br>3.5 3.0 | 0.01 0.01<br>0.01 0.01<br>0.04 0.03 | 9801.OH.AF.04 |
| OH, 1998 (Mustang) | EC | 1.1 | 80 | 2<br>2<br>2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 33.1<br>9.9<br>10.8 | 0.64 0.49<br>2.2 2.7<br>4.1 3.7 | <0.01 (2)<br>0.02 0.02<br>0.02 0.02 | 9801.OH.AF.03-a |
| OH, 1998 (Mustang) | EC | 1.1 | 9 | ( 2<br>( 2<br>( 2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 33.1<br>9.9<br>10.8 | 0.33 0.31<br>0.63 0.28<br>1.8 1.9 | <0.01 (2)<br><0.01 (2)<br>0.02 0.02 | 9801.OH.AF.03-b |
| PA, 1998 | EC | 1.1 | 270 | 2<br>2<br>2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 19.3<br>26.8<br>20.2 | 2.2 2.0<br>0.64 0.87<br>1.2 1.4 | 0.04 0.04<br>0.01 0.02<br>0.02 0.02 | 9801.PA.AF.01 |
| VA, 1998 | EC | 1.1 | 250 | 2<br>2<br>2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 11.9<br>29.0<br>22.2 | 0.36 0.35<br>0.64 0.48<br>0.55 1.0 | <0.01 (2)<br><0.01 (2)<br><0.01 (2) | 9801.VA.AF.02 |
| WA, 1998 (Pioneer 5364) | EC | 1.1 | 28 | 2<br>2<br>2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 50.1<br>8.7<br>16.9 | 3.6 4.2<br>0.28 0.38<br>0.08 0.37 | 0.06 0.06<br><0.01 (2)<br><0.01 (2) | 9801.WA.AF.11 |
| WA, 1998 (Pioneer 5364) | EC | 1.1 | 9 | ( 2<br>( 2<br>( 2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 50.1<br>8.7<br>16.9 | 5.7 3.0<br>0.38 0.19<br>0.46 0.36 | 0.08 0.05<br><0.01 (2)<br><0.01 (2) | 9801.WA.AF.11 |
| WI, 1998 (Wrangler) | EC | 1.1 | 47 | 2<br>2<br>2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 43.3<br>43.6<br>18.9 | 3.0 1.3<br>0.81 0.57<br>11 13 | 0.04 0.03<br>0.01 0.01<br>0.11 0.12 | 9801.WI.AF.05-a |
| WI, 1998 (Wrangler) | EC | 1.1 | 9 | ( 2<br>( 2<br>( 2 | 15<br>15<br>15 | hay 1<br>hay 2<br>hay 3 | 43.3<br>43.6<br>18.9 | 1.7 1.8<br>0.74 1.2<br>17 17 | 0.02 0.03<br>0.01 0.02<br>0.18 0.21 | 9801.WI.AF.05-b |
| CA, 1998 (GT13R) | EC | 1.1 | 92 | 2<br>2<br>2<br>2 | 15<br>15<br>15<br>15 | hay 1<br>hay 2<br>hay 3<br>hay 4 | 18.1<br>8.0<br>16.7<br>56.1<br>16.7<br>56.1 | 1.9 1.4<br>0.64 0.60<br>1.4 1.7<br>8.0 7.8<br>c 0.02<br>c 0.04 | 0.03 0.03<br>0.01 0.01<br>0.01 0.02<br>0.11 0.12<br>c <0.01<br>c <0.01 | 9801.CA.AF.12 |

[1] Forage 1, forage 2, hay 1, etc refer to the 1st, 2nd and later cuttings.
[2] Moisture levels were measured on the accompanying control sample.
( aerial application. c: sample from control plot

Table 47. Residues in clover from supervised trials in the USA.

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| CA, 1989 | EC | 1.1 | 190 | 2 | 0<br>7<br>15<br>20<br>25 | forage 2 | 30 19<br>0.19 0.38<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 0.12 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-3036 |
| CA, 1989 | EC | 1.4 | 190 | 2 | 0<br>7<br>15<br>20<br>25 | forage 2 | 45 39<br>0.49 0.58<br>0.06 0.06<br><0.05 (2)<br><0.05 (2) | 0.23 0.24<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-3036 |
| CA, 1989 | EC | 1.1 | 190 | ( 2 | 0<br>7<br>15<br>20<br>25 | forage 2 | 42 46<br>3.4 2.5<br>0.11 0.19<br><0.05 (2)<br><0.05 (2) | 0.31 0.33<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-3038 |
| CA, 1989 | EC | 1.4 | 190 | ( 2 | 0<br>7<br>15<br>20<br>25 | forage 2 | 71 67<br>4.8 5.1<br>0.26 0.30<br>0.07 <0.05<br><0.05 (2) | 0.56 0.49<br>0.06 0.06<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-3038 |
| ID, 1988 (Arlington Red) | EC | 1.1 | 94 | 2 | 0<br>7<br>15<br>21<br>25 | forage 2 | 28 60<br>0.55 0.94<br>0.13 0.07<br><0.05 (2)<br><0.05 (2) | 0.18 0.42<br>0.06 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-3041 |
| ID, 1988 (Arlington Red) | EC | 1.4 | 94 | 2 | 0<br>7<br>15<br>21<br>25 | forage 2 | 35 44<br>0.51 0.61<br>0.16 0.09<br>0.07 <0.05<br><0.05 (2) | 0.17 0.18<br>0.09 0.09<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-3041 |
| ID, 1988 (Arlington Red) | EC | 1.1 | 94 | ( 2 | 0<br>7<br>15<br>21<br>25 | forage 2 | 18 4.1<br>0.05 0.07<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 0.12 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-3180 |
| ID, 1988 (Arlington Red) | EC | 1.4 | 94 | ( 2 | 0<br>7 | forage 2 | 14 15<br>0.45 0.08 | 0.12 0.12<br><0.05 (2) | MP-CL-3180 |
| MN, 1988 (Mammonth) | EC | 1.1 | 190 | 2 | 0<br>7<br>15<br>20<br>25 | forage 2 | 54 49<br>9.9 9.6<br>6.1 6.5<br>2.4 3.0<br>0.86 1.4 | 0.36 0.34<br>0.09 0.07<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-7036 |
| MN, 1988 (Mammonth) | EC | 1.4 | 190 | 2 | 0<br>7<br>15<br>20<br>25 | forage 2 | 51 46<br>13 15<br>8.1 8.2<br>3.7 3.6<br>1.3 1.8 | 0.30 0.25<br>0.09 0.10<br>0.05 0.06<br><0.05 (2)<br><0.05 (2) | MP-CL-7036 |

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| NY, 1988 | EC | 1.1 | 260 | 2 | 0<br>7<br>15<br>20<br>26 | forage 2 | 3.6 3.5<br>0.62 0.53<br>0.23 0.18<br>0.08 0.12<br>0.14 0.13 | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-7038 |
| NY, 1988 | EC | 1.4 | 260 | 2 | 0<br>7<br>15<br>20<br>26 | forage 2 | 7.8 6.6<br>0.61 0.63<br>0.31 0.24<br>0.24 0.20<br>0.13 0.11 | 0.05 0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-7038 |
| WI, 1988 (Medium Red) | EC | 1.1 | 190 | 2 | 0<br>7<br>15<br>20<br>25 | forage 2 | 47 53<br>7.2 6.2<br>2.5 2.8<br>1.4 1.4<br>0.74 0.51 | 0.21 0.19<br>0.09 0.06<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CL-7040 |
| WI, 1988 (Medium Red) | EC | 1.4 | 190 | 2 | 0<br>7<br>15<br>20<br>25 | forage 2 | 60 69<br>9.1 8.9<br>2.5 2.2<br>1.6 1.3<br>0.32 0.33 | 0.26 0.33<br>0.11 0.11<br>0.05 <0.05<br><0.05 (2)<br><0.05 (2) | MP-CL-7040 |

[1] Forage 2 refers to 2nd cutting.
( aerial application.

Table 48. Residues in field pea forage, straw and vines from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| WA, 1989 (Dark Skin 49) | EC | 1.1 | 190 | 6 | 0<br>5<br>10<br>15 | forage, dried | 2.7 2.3<br>5.0 1.5<br>1.8 1.7<br>2.9 <u>3.6</u> | 0.11 0.08<br>0.25 0.11<br>0.19 0.20<br>0.23 <u>0.41</u> | MP-PE-3116 |
| WA, 1989 (Dark Skin 49) | EC | 5×1.1<br>+1×0.56 | 190 | 6 | 0<br>5<br>10<br>15 | forage, dried | 0.29 0.13<br>1.3 1.7<br><u>3.4</u> 1.8<br>1.6 1.3 | <0.05 (2)<br>0.11 0.15<br><u>0.28</u> 0.14<br>0.14 0.10 | MP-PE-3116 |
| WA, 1989 (Dark Skin 49) | EC | 1.1 | 94 | ( 6 | 0<br>5<br>10<br>15<br>20<br>25<br>10<br>15 | forage, dried | 16 4.2<br>0.56 0.73<br>0.49 0.22<br><u>0.66</u> 0.44<br>0.31 0.24<br>0.29 0.12<br>c<0.05<br>c <0.05 | 0.55 0.16<br>0.10 0.09<br>0.10 0.05<br><u>0.08</u> 0.06<br>0.07 0.06<br>0.06 <0.05<br>c 0.05<br>c 0.06 | MP-PE-3118 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| WA, 1989 (Dark Skin 49) | EC | 5×1.1<br>+1×0.56 | 94 | ( 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage, dried | 1.5 2.8<br>0.18 0.23<br>0.30 0.55<br>0.15 0.08<br>0.10 0.08<br>0.26 0.21 | 0.10 0.12<br>0.05 <0.05<br>0.08 0.14<br><0.05 (2)<br>0.09 0.06<br>0.07 0.05 | MP-PE-3118 |
| DE, 1988 (Alaska) | EC | 1.1 | 290 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage, dried | 46 41<br>11 15<br>4.8 4.8<br>6.6 7.6<br>6.3 6.0<br>5.5 7.6 | 0.22 0.17<br>0.09 0.13<br><0.05 (2)<br>0.10 0.09<br>0.11 0.10<br>0.09 0.11 | MP-PE-7089 |
| DE, 1988 (Alaska) | EC | 5×1.1<br>+1×0.56 | 290 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage, dried | 19 24<br>7.1 6.6<br>2.2 2.0<br>3.6 4.2<br>4.2 2.1<br>3.2 3.9 | 0.06 0.07<br>0.07 0.07<br><0.05 (2)<br>0.06 0.05<br>0.06 <0.05<br>0.05 0.07 | MP-PE-7089 |
| MN, 1988 (Alaska 146) | EC | 1.1 | 190 | 4 | 0<br>5<br>10<br>15<br>20<br>25 | forage, dried | 18 17<br>1.0 1.0<br>0.42 0.35<br>0.27 0.26<br>0.34 0.12<br>0.35 0.26 | 0.15 0.12<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7091 |
| MN, 1988 (Alaska 146) | EC | 3×1.1<br>+1×0.56 | 190 | 4 | 0<br>5<br>10<br>15<br>20<br>25 | forage, dried | 11 14<br>0.69 0.59<br>0.35 0.30<br>0.18 0.37<br>0.17 0.16<br>0.17 0.33 | 0.12 0.09<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7091 |
| ND, 1988 (Trapper) | EC | 1.1 | 94 | 6 | 0<br>5<br>10<br>15<br>20<br>26<br>20 | forage, dried | 23 22<br>14 8.1<br>10 8.5<br>9.5 7.7<br>5.5 4.6<br>0.53 0.82<br>c 0.05 | 0.39 0.33<br>0.30 0.24<br>0.29 0.15<br>0.21 0.16<br>0.10 0.11<br><0.05 (2)<br>c <0.05 | MP-PE-7093 |
| ND, 1988 (Trapper) | EC | 5×1.1<br>+1×0.56 | 94 | 6 | 0<br>5<br>10<br>15<br>20<br>25<br>20 | forage, dried | 12 8.8<br>5.8 5.7<br>4.3 4.7<br>3.2 5.2<br>2.6 3.2<br>0.47 0.30<br>c 0.05 | 0.24 0.21<br>0.14 0.17<br>0.10 0.11<br>0.09 0.09<br>0.06 0.06<br><0.05 (2)<br>c <0.05 | MP-PE-7093 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| DE, 1988 (Alaska) | EC | 1.1 | 47 | ( 6 | 0<br>5<br>10<br>15<br>20<br>25<br>0<br>10<br>15<br>20<br>25 | forage, dried | 5.4 <0.05<br><0.05 0.23<br>0.58 0.41<br>0.25 0.26<br>0.20 0.43<br>1.0 0.25<br>c 3.1 0.37<br>c 0.48<br>c 0.20 <0.05<br>c 0.20 <0.05<br>c 0.19 <0.05 | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c <0.05 (2)<br>c <0.05<br>c <0.05 (2)<br>c <0.05 (2)<br>c <0.05 (2) | MP-PE-7095 |
| DE, 1988 (Alaska) | EC | 5×1.1<br>+1×0.56 | 47 | ( 6 | 0<br>5<br>10<br>15<br>20<br>24<br>0<br>10<br>15<br>20<br>25 | forage, dried | 53 46<br>52 58<br>15 18<br>36 58<br>13 14<br>6.8 6.2<br>c 3.1 0.37<br>c 0.48<br>c 0.20 <0.05<br>c 0.20 <0.05<br>c 0.19 <0.05 | 0.17 0.18<br>0.30 0.35<br>0.18 0.18<br>0.43 0.52<br>0.08 0.12<br><0.05 (2)<br>c <0.05 (2)<br>c <0.05<br>c <0.05 (2)<br>c <0.05 (2)<br>c <0.05 (2) | MP-PE-7095 |
| WA, 1989 (Knight) | EC | 1.1 | 94 | ( 4 | 0<br>5<br>10<br>15<br>20<br>25 | forage, succu-lent | 0.35 0.78<br><0.05 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 0.10<br><0.05 0.06 | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 0.06<br><0.05 (2) | MP-PE-3188 |
| WA, 1989 (Knight) | EC | 3×1.1<br>+1×0.56 | 94 | ( 4 | 0<br>5<br>10<br>15<br>20<br>25 | forage, succu-lent | <0.05 0.53<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-3188 |
| WA, 1989 (Knight) | EC | 1.1 | 190 | 4 | 0<br>5<br>10<br>15 | forage, succu-lent | 5.0 7.8<br>0.23 0.26<br>0.12 <0.05<br>0.17 <0.05 | 0.11 0.15<br><0.05 (2)<br>0.07 <0.05<br>0.09 <0.05 | MP-PE-3189 |
| WA, 1989 (Knight) | EC | 3×1.1<br>+1×0.56 | 190 | 4 | 0<br>5<br>10<br>15 | forage, succu-lent | 1.5 2.1<br>0.11 <0.05<br>0.06 0.07<br>0.07 <0.05 | 0.05 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-3189 |
| DE, 1988 (Wando) | EC | 1.1 | 290 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage, succu-lent | 26 25<br>12 7.9<br>3.3 3.1<br>4.4 2.5<br>4.0 4.2<br>7.3 8.2 | 0.08 0.12<br>0.07 0.06<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7096 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| DE, 1988 (Wando) | EC | 5×1.1<br>+1×0.56 | 290 | 6 | 0<br>5<br>10<br>15<br>20<br>25 | forage, succu-lent | 10 13<br>5.4 3.8<br>1.4 1.5<br>2.9 2.4<br>4.4 3.4<br>3.0 4.9 | <0.05 0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7096 |
| MN, 1988 (Asgrow XPG 206) | EC | 1.1 | 190 | 4 | 0<br>5<br>10<br>15<br>20<br>25 | forage, succu-lent | 4.6 4.2<br>0.07 0.11<br>0.05 0.06<br><0.05 (2)<br>0.08 0.06<br><0.05 0.05 | 0.09 0.11<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7098 |
| MN, 1988 (Asgrow XPG 206) | EC | 3×1.1<br>+1×0.56 | 190 | 4 | 0<br>5<br>10<br>15<br>20<br>25 | forage, succu-lent | 2.4 2.1<br>0.07 0.20<br><0.05 0.06<br>0.10 0.15<br>0.08 0.11<br>0.07 0.08 | 0.08 0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7098 |
| WI, 1988 (Ego) | EC | 1.1 | 240 | 5 | 0<br>5<br>9<br>14<br>19<br>24 | forage, succu-lent | 21 20<br>0.14 0.24<br>0.12 0.08<br>0.13 0.13<br>0.06 0.08<br>0.09 0.07 | 0.02 0.18<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7100 |
| WI, 1988 (Ego) | EC | 4×1.1<br>+1×0.56 | 240 | 5 | 0<br>5<br>9<br>14<br>19<br>24 | forage, succu-lent | 5.0 6.0<br><0.05 0.06<br>0.06 <0.05<br>0.08 0.05<br><0.05 (2)<br><0.05 (2) | 0.07 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7100 |
| WI, 1988 (9888F) | EC | 4×0.56<br>+1×1.1 | 38 | ( 5 | 0<br>6<br>10<br>15<br>20<br>25 | forage, succu-lent | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7102 |
| WI, 1988 (9888F) | EC | 1.1 | 38 | ( 5 | 0<br>6<br>10<br>15<br>20<br>25 | forage, succu-lent | 0.13 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 0.05<br><0.05 (2) | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-PE-7102 |
| WA, 1989 (Dark Skin 49) | EC | 1.1 | 190 | 6 | 15 | straw | 5.0 3.6 | 0.42 0.46 | MP-PE-3116 |
| WA, 1989 (Dark Skin 49) | EC | 5×1.1<br>+1×0.56 | 190 | 6 | 10<br>10 | straw | 2.1 4.9<br>c <0.05 | 0.34 0.54<br>c 0.10 | MP-PE-3116 |
| WA, 1989 (Dark Skin 49) | EC | 1.1 | 94 | ( 6 | 15 | straw | 0.70 0.82 | 0.07 0.09 | MP-PE-3118 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| WA, 1989 (Dark Skin 49) | EC | 5×1.1 +1×0.56 | 94 | ( 6 | 10 | straw | 0.72 0.24 | 0.17 0.13 | MP-PE-3118 |
| DE, 1988 (Alaska) | EC | 1.1 | 290 | 6 | 15 | straw | 13 11 | 0.12 0.07 | MP-PE-7089 |
| DE, 1988 (Alaska) | EC | 5×1.1 +1×0.56 | 290 | 6 | 10 | straw | 2.1 2.6 | <0.05 (2) | MP-PE-7089 |
| MN, 1988 (Alaska 146) | EC | 1.1 | 190 | 4 | 15 | straw | 0.71 0.67 | <0.05 (2) | MP-PE-7091 |
| MN, 1988 (Alaska 146) | EC | 3×1.1 +1×0.56 | 190 | 4 | 10 | straw | 1.1 1.1 | <0.05 (2) | MP-PE-7091 |
| ND, 1988 (Trapper) | EC | 1.1 | 94 | 6 | 15<br>15 | straw | 3.1 3.5<br>c 0.25 | 0.46 0.42<br>c <0.05 | MP-PE-7093 |
| ND, 1988 (Trapper) | EC | 5×1.1 +1×0.56 | 94 | 6 | 10 | straw | 3.1 2.7 | 0.08 0.07 | MP-PE-7093 |
| DE, 1988 (Alaska) | EC | 1.1 | 47 | ( 6 | 10<br>15<br>10<br>15 | straw [1] | 1.7 0.80<br>0.43 0.48<br>c 0.90 0.38<br>c 0.33 | <0.05 (2)<br><0.05 (2)<br>c <0.05 (2)<br>c <0.05 | MP-PE-7095 |
| DE, 1988 (Alaska) | EC | 5×1.1 +1×0.56 | 47 | ( 6 | 10<br>10<br>15 | straw | 19 27<br>c 0.90 0.38<br>c 0.33 | 0.17 0.19<br>c <0.05 (2)<br>c <0.05 | MP-PE-7095 |
| WA, 1989 (Knight) | EC | 1.1 | 94 | ( 4 | 15 | vine | <0.05 (2) | <0.05 (2) | MP-PE-3188 |
| WA, 1989 (Knight) | EC | 3×1.1 +1×0.56 | 94 | ( 4 | 10 | vine | <0.05 (2) | <0.05 (2) | MP-PE-3188 |
| WA, 1989 (Knight) | EC | 3×1.1 +1×0.56 | 190 | 4 | 10 | vine | 0.21 0.15 | 0.07 0.05 | MP-PE-3189 |
| DE, 1988 (Wando) | EC | 1.1 | 290 | 6 | 15 | vine | 6.8 7.3 | <0.05 (2) | MP-PE-7096 |
| DE, 1988 (Wando) | EC | 5×1.1 +1×0.56 | 290 | 6 | 10 | vine | 1.1 1.6 | <0.05 (2) | MP-PE-7096 |
| MN, 1988 (Asgrow XPG 206) | EC | 1.1 | 190 | 4 | 10 | vine | 0.20 0.15 | <0.05 (2) | MP-PE-7098 |
| MN, 1988 (Asgrow XPG 206) | EC | 3×1.1 +1×0.56 | 190 | 4 | 10 | vine | 0.13 0.23 | <0.05 (2) | MP-PE-7098 |
| WI, 1988 (Ego) | EC | 1.1 | 240 | 5 | 14 | vine | 0.15 0.17 | <0.05 (2) | MP-PE-7100 |
| WI, 1988 (Ego) | EC | 4×1.1 +1×0.56 | 240 | 5 | 9 | vine | 0.08 <0.05 | <0.05 (2) | MP-PE-7100 |
| WI, 1988 (9888F) | EC | 1.1 | 38 | ( 5 | 15 | vine | <0.05 (2) | <0.05 (2) | MP-PE-7102 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| WI, 1988 (9888F) | EC | 4×0.56 +1×1.1 | 38 | ( 5 | 10 | vine | <0.05 (2) | <0.05 (2) | MP-PE-7102 |

[1] Contamination of control at levels comparable to those in the treated plot invalidates results
( aerial application. c: sample from control plot

Table 49. Residues in bean forage and fodder from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (Baby White) | EC | 1.7 | 340 | 6 | 0<br>7<br>15<br>21<br>28<br>21 | forage | 48 50<br>2.2 2.7<br>0.88 0.82<br>0.66 0.62<br>0.38 0.47<br>c 0.10 | 0.59 0.73<br>0.16 0.08<br>0.07 <0.05<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | MP-DB-3072 |
| CA, 1988 (Red Kidney) | EC | 1.7 | 94 | ( 6 | 0<br>7<br>15<br>21<br>28 | forage | 9.3 14<br>0.46 0.55<br>0.14 0.18<br>0.11 0.06<br>0.09 0.07 | 0.37 0.41<br>0.08 0.12<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-DB-3074 |
| MI, 1988 (Seafarer Navy Bean) | EC | 1.7 | 220 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 15 11<br>0.33 0.20<br>0.14 <0.05<br>0.07 0.10<br>0.08 0.07 | 0.34 0.27<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-DB-7001 |
| NE, 1988 (Pinto) | EC | 1.7 | 190 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 50 47<br>0.12 0.69<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 0.27 0.23<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-DB-7003 |
| NE, 1988 (Pinto) | EC | 1.7 | 9 | ( 6 | 0<br>7<br>15<br>21<br>28 | forage | 25 18<br>0.61 0.49<br>0.06 0.08<br><0.05 (2)<br><0.05 (2) | 0.13 0.16<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-DB-7005 |
| ND, 1988 (Topaz, Pinto) | EC | 1.7 | 94 | 6 | 0<br>7<br>15<br>21<br>28 | forage | 19 32<br>1.0 3.0<br>1.1 0.75<br>0.17 0.31<br>0.21 0.13 | 0.48 1.7<br>0.06 0.17<br>0.06 <0.05<br><0.05 (2)<br><0.05 (2) | MP-DB-7006 |
| CA, 1988 (Baby White) | EC | 1.7 | 340 | 6 | 15 | hay | 0.94 0.93 | 0.13 0.06 | MP-DB-3072 |
| CA, 1988 (Red Kidney) | EC | 1.7 | 94 | ( 6 | 15 | hay | 3.5 3.0<br>c 0.07 | 0.35 0.30<br>c <0.05 | MP-DB-3074 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| MI, 1988 (Seafarer Navy Bean) | EC | 1.7 | 220 | 6 | 15 | hay | 0.22 0.25 | <0.05 (2) | MP-DB-7001 |
| NE, 1988 (Pinto) | EC | 1.7 | 190 | 6 | 15 | hay | <0.05 (2) | 0.07 <0.05 | MP-DB-7003 |
| NE, 1988 (Pinto) | EC | 1.7 | 9 | ( 6 | 15 | hay | <0.05 0.06 | <0.05 (2) | MP-DB-7005 |
| ND, 1988 (Topaz, Pinto) | EC | 1.7 | 94 | 6 | 15 | hay | 1.2 1.4 | 0.05 0.12 | MP-DB-7006 |

( aerial application. c: sample from control plot

Table 50. Residues in maize and sweet corn forage and fodder from supervised trials in the USA.

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | Sample | parathion-methyl | paraoxon-methyl | |
| MAIZE | | | | | | | | | |
| CA, 1989 | EC | 1.1 | 300 | 6 | 12 | fodder | <0.05 (2) | <0.05 (2) | MP-CN-3169 |
| TX, 1988 (Dekalb 689) | EC | 1.1 | 47 | ( 6 | 12 | fodder | 0.96 0.79 | <0.05 (2) | MP-CN-3170 |
| TX, 1988 (Dekalb 689) | EC | 1.1 | 75 | 6 | 12 | fodder | 0.28 0.23 | <0.05 (2) | MP-CN-3171 |
| GA, 1988 (Cargill Hybrid) | EC | 1.1 | 56 | 6 | 12 | fodder | 1.2 1.1 | <0.05 (2) | MP-CN-7042 |
| IL, 1988 (F/S 2368A) | EC | 1.1 | 160 | 6 | 12 | fodder | 0.10 0.07 | <0.05 (2) | MP-CN-7043 |
| MN, 1988 (Pioneer 3732) | EC | 1.1 | 230 | 6 | 12<br>12 | fodder | 7.9 2.4<br>c 0.22 | 0.28 0.12<br>c <0.05 | MP-CN-7044 |
| VA, 1988 (SX383) | EC | 1.1 | 220 | 6 | 12 | fodder | <0.05 (2) | <0.05 (2) | MP-CN-7045 |
| NE, 1988 (Funks G-4440) | EC | 1.1 | 190 | 6 | 12 | fodder | 2.6 4.5 | 0.49 0.42 | MP-CN-7046 |
| OH, 1988 (Pioneer 3324) | EC | 1.1 | 100 | 6 | 12 | fodder | 2.3 17 | 0.08 0.47 | MP-CN-7047 |
| OH, 1988 (Pioneer 3324) | EC | 1.1 | 47 | ( 6 | 12 | fodder | 3.3 5.9 | <0.05 0.21 | MP-CN-7048 |
| CA, 1989 (Pioneer 3780) | EC | 1.1 | 300 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 0.06 0.07<br><0.05 (2)<br><0.05 (2)<br>0.10 0.55<br><0.05 (2) | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-3043 |
| TX, 1988 (Douglas King DK4161) | EC | 1.1 | 47 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 25 13<br>0.14 0.46<br>0.58 0.68<br>0.08 0.12<br><0.05 (2) | 0.43 0.22<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-3045 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | Sample | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (Douglas King DK4161) | EC | 1.1 | 9 | ( 6 | 0<br>6<br>12<br>18<br>24 | forage | 5.1 5.2<br>0.75 0.32<br><0.05 0.13<br>0.13 0.09<br>0.17 0.14 | 0.10 0.09<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-3183 |
| GA, 1988 (Cargill Hybrid) | EC | 1.1 | 56 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 94 91<br>2.5 0.16<br><0.05 0.05<br><0.05 0.65<br><0.05 (2) | 0.57 0.44<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-7049 |
| IL, 1988 (F/S 2368A) | EC | 1.1 | 160 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 21 19<br>0.27 0.22<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 0.49 0.37<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-7050 |
| MN, 1988 (Pioneer 3732) | EC | 1.1 | 230 | 6 | 0<br>6<br>12<br>18<br>24<br>24 | forage | 8.8 9.7<br>1.1 0.84<br>0.40 0.24<br>0.28 0.40<br>0.34 <0.05<br>c 0.21 | 2.6 3.5<br>0.07 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | MP-CN-7051 |
| VA, 1988 (SX383) | EC | 1.1 | 220 | 6 | 0<br>6<br>12<br>18<br>31 | forage | 0.62 0.54<br>0.12 0.11<br><0.05 (2)<br><0.05 0.07<br><0.05 (2) | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-7052 |
| NE, 1988 (Funks G-444D) | EC | 1.1 | 190 | 6 | 0<br>6<br>12<br>18<br>24<br>18 | forage | 9.9 15<br>1.4 0.98<br>55 37<br>0.65 0.19<br>0.22 0.43<br>c 0.07 | 0.09 0.06<br><0.05 (2)<br>0.32 0.11<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | MP-CN-7053 |
| OH, 1988 (Pioneer 3324) | EC | 1.1 | 100 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 8.9 9.2<br>0.25 0.27<br>0.16 0.18<br>0.17 0.18<br>0.20 0.18 | 0.17 0.22<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-7054 |
| OH, 1988 (Pioneer 3324) | EC | 1.1 | 47 | ( 6 | 0<br>6<br>12<br>18<br>24 | forage | 3.3 2.0<br>0.46 0.37<br>0.06 <0.05<br>0.14 0.11<br>0.12 0.12 | 0.08 0.06<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-7055 |
| CA, 1989 (Pioneer 3780) | EC | 1.1 | 300 | 6 | 12 | silage | 0.07 <0.05 | <0.05 (2) | MP-CN-3043 |
| TX, 1988 (Douglas King DK4161) | EC | 1.1 | 47 | 6 | 12 | silage | 0.13 0.54 | <0.05 (2) | MP-CN-3045 |
| TX, 1988 (Douglas King DK4161) | EC | 1.1 | 9 | ( 6 | 12 | silage | 0.14 <0.05 | <0.05 (2) | MP-CN-3183 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | Sample | parathion-methyl | paraoxon-methyl | |
| SWEET CORN | | | | | | | | | |
| CA, 1988 | EC | 1.1 | 300 | 6 | 3 | fodder | 0.15 0.11 | <0.05 (2) | MP-CN-3047 |
| CA, 1988 (Jubilee) | EC | 1.1 | 84 | ( 3 | 3 | fodder | 1.2 0.38 | 0.06 <0.05 | MP-CN-3049 |
| TX, 1988 (Funk's Sweet G-90) | EC | 1.1 | 9 | ( 6 | 3 | fodder | 1.2 0.84 | 0.11 0.06 | MP-CN-3050 |
| WA, 1988 (Jubilee) | EC | 1.1 | 94 | ( 6 | 3 | fodder | 0.11 <0.05 | <0.05 (2) | MP-CN-3185 |
| FL, 1988 (Merit) | EC | 1.1 | 300 | 6 | 3 | fodder | 2.1 1.2 | 0.08 0.07 | MP-CN-7056 |
| NY, 1988 (Jubilee) | EC | 1.1 | 45 | ( 6 | 3 | fodder | 0.44 0.55 | <0.05 (2) | MP-CN-7057 |
| WI, 1988 (Commander) | EC | 1.1 | 38 | ( 6 | 3 | fodder | 1.1 2.0 | 0.10 0.13 | MP-CN-7058 |
| CA, 1988 | EC | 1.1 | 300 | 6 | 0<br>6<br>12<br>18<br>24 | forage | 0.13 0.22<br>0.11 0.10<br>0.06 <0.05<br><0.05 0.09<br>0.09 0.10 | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 0.07<br><0.05 (2) | MP-CN-3047 |
| CA, 1988 (Jubilee) | EC | 1.1 | 84 | ( 3 | 0<br>6<br>12<br>18<br>24 | forage | 0.25 0.21<br>1.1 0.66<br>0.55 0.58<br>0.18 0.21<br>0.10 0.11 | <0.05 (2)<br>0.19 0.36<br>0.18 <0.05<br><0.05 0.09<br><0.05 (2) | MP-CN-3049 |
| TX, 1988 (Funk's Sweet G-90) | EC | 1.1 | 9 | ( 6 | 0<br>6<br>12<br>18<br>24 | forage | 9.4 3.2<br>1.4 1.1<br>0.32 0.19<br><0.05 (2)<br>0.08 0.12 | 0.25 0.06<br>0.09 0.08<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-3050 |
| WA, 1988 (Jubilee) | EC | 1.1 | 94 | ( 6 | 0<br>6<br>12<br>18<br>24 | forage | <0.05 (2)<br><0.05 0.13<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-3185 |
| FL, 1988 (Merit) | EC | 1.1 | 300 | 6 | 0<br>6<br>12<br>18<br>24<br>6 | forage | 18 17<br>1.0 0.96<br>0.26 0.39<br>0.32 0.25<br>0.08 0.10<br>c 0.05 | 0.20 0.27<br>0.13 0.18<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | MP-CN-7056 |
| NY, 1988 (Jubilee) | EC | 1.1 | 45 | ( 6 | 0<br>6<br>12<br>18<br>24 | forage | 3.6 2.3<br>0.05 0.16<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 0.09 0.07<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-7057 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | Sample | parathion-methyl | paraoxon-methyl | |
| WI, 1988 (Commander) | EC | 1.1 | 38 | ( 6 | 0<br>6<br>12<br>18<br>24 | forage | 18 18<br>0.56 0.56<br>0.23 0.29<br>0.17 0.14<br><0.05 (2) | 0.42 0.39<br>0.17 0.13<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-CN-7058 |

( aerial application. c: sample from control plot

Table 51. Residues in sorghum fodder and forage from supervised trials in the USA.

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (TEY 75) | EC | 1.1 | 150 | 6 | 21 | fodder | 0.83 0.61 | 0.05 <0.05 | MP-SG-3173 |
| TX, 1988 (Asgrow Topaz) | EC | 1.1 | 47 | 6 | 21 | fodder | 1.2 0.84 | 0.05 0.06 | MP-SG-3174 |
| TX, 1988 (Asgrow Topaz) | EC | 1.1 | 9 | ( 6 | 21 | fodder | 0.08 <0.05 | <0.05 (2) | MP-SG-3175 |
| MO, 1988 (Funk's G-623GBR) | EC | 1.1 | 200 | 6 | 21 | fodder | 0.16 0.33 | <0.05 (2) | MP-SG-7111 |
| MO, 1988 (Funk's G-623GBR) | EC | 1.1 | 22 | ( 6 | 22 | fodder | 0.25 0.21 | <0.05 (2) | MP-SG-7112 |
| NE, 1988 (NC + 271) | EC | 1.1 | 190 | 6 | 21 | fodder | 1.7 2.3 | 0.14 0.15 | MP-SG-7113 |
| TX, 1988 (TEY 75) | EC | 1.1 | 150 | 6 | 0<br>7<br>14<br>21<br>28<br>35 | forage | 10 7.2<br>1.3 0.56<br>0.29 0.58<br>0.62 1.3<br>0.15 0.12<br>0.16 0.10 | 0.31 0.21<br>0.06 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-SG-3133 |
| MO, 1988 (Funk's G-623GBR) | EC | 1.1 | 270 | 6 | 0<br>7<br>14<br>21<br>28<br>35<br>7 | forage | 10 10<br>0.66 0.38<br>0.09 0.07<br>0.13 0.11<br><0.05 (2)<br>0.07 0.11<br>c 0.09 | 0.25 0.28<br>0.05 <0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | MP-SG-7114 |
| MO, 1988 (Funk's G-623GBR) | EC | 1.1 | 22 | ( 6 | 0<br>7<br>14<br>21<br>28<br>35<br>0 | forage | 9.3 6.5<br>0.29 0.27<br>0.17 0.14<br>0.26 0.24<br>0.09 0.23<br>0.09 0.23<br>c 0.13 | 0.22 0.15<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | MP-SG-7115 |
| NE, 1988 (NC + 271) | EC | 1.1 | 190 | 6 | 0<br>7<br>14<br>21<br>28<br>35 | forage | 20 14<br>1.0 1.8<br>0.48 0.69<br>0.32 0.35<br>0.35 0.33<br>0.18 0.17 | 0.16 0.12<br><0.05 0.05<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-SG-7116 |

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | sample | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (TEY 75) | EC | 1.1 | 150 | 6 | 21 | hay | 0.29 0.53 | <0.05 (2) | MP-SG-3133 |
| MO, 1988 (Funk's G-623GBR) | EC | 1.1 | 270 | 6 | 21 | hay | 0.09 0.07 | <0.05 (2) | MP-SG-7114 |
| MO, 1988 (Funk's G-623GBR) | EC | 1.1 | 22 | ( 6 | 21 | hay | 0.30 0.10 | <0.05 (2) | MP-SG-7115 |
| NE, 1988 (NC + 271) | EC | 1.1 | 190 | 6 | 21 | hay | 0.64 0.64 | <0.05 (2) | MP-SG-7116 |
| TX, 1988 (TEY 75) | EC | 1.1 | 150 | 6 | 21 | silage | 0.13 0.10 | <0.05 (2) | MP-SG-3133 |
| MO, 1988 (Funk's G-623GBR) | EC | 1.1 | 270 | 6 | 21 | silage | 0.13 0.17 | <0.05 (2) | MP-SG-7114 |
| MO, 1988 (Funk's G-623GBR) | EC | 1.1 | 22 | ( 6 | 21 | silage | 0.27 0.11 | <0.05 (2) | MP-SG-7115 |
| NE, 1988 (NC + 271) | EC | 1.1 | 190 | 6 | 21 | silage | 0.26 0.24 | <0.05 (2) | MP-SG-7116 |

( aerial application. c: sample from control plot

Table 52. Residues in wheat forage and fodder from supervised trials in the USA (LeRoy, 1990d). Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Location, year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| WA, 1989 (Rojo) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 190 | 6 | 0 | forage | 1.5 2.5 | 0.30 0.36 | MP-WH-3165 |
| WA, 1989 (Rojo) | EC | 4×1.4<br>+2×0.84 | 190 | 6 | 0<br>7<br>14<br>21<br>28 | forage | 10 12<br>1.4 0.83<br>0.46 0.34<br>0.13 0.12<br>0.13 0.07 | 0.64 0.77<br>0.39 0.30<br>0.21 0.22<br>0.09 0.10<br>0.10 0.05 | MP-WH-3165 |
| WA, 1989 (Rojo) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 94 | ( 6 | 0 | forage | 3.5 4.8 | 0.76 0.70 | MP-WH-3182 |
| WA, 1989 (Rojo) | EC | 4×1.4<br>+2×0.84 | 94 | ( 6 | 0<br>7<br>14<br>21<br>28 | forage | 1.1 9.4<br>1.1 1.0<br>0.24 0.19<br>0.09 0.28<br>0.19 0.14 | 0.33 0.69<br>0.33 0.30<br>0.12 0.14<br>0.10 0.18<br>0.12 0.10 | MP-WH-3182 |
| OH, 1988 (Becker) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 190 | 6 | 0 | forage | 3.8 4.0 | 0.31 0.33 | MP-WH-7143 |

| Location, year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| OH, 1988 (Becker) | EC | 4×1.4<br>+2×0.84 | 190 | 6 | 0<br>7<br>14<br>21<br>28<br>7<br>14 | forage | 10 8.3<br>0.51 0.57<br>0.68 0.97<br>0.26 0.25<br>0.25 0.28<br>c 0.13<br>c 0.09 | 0.60 0.50<br>0.17 0.19<br>0.15 0.17<br>0.10 0.09<br>0.05 0.06<br>c <0.05<br>c <0.05 | MP-WH-7143 |
| ND, 1988 (Marshall) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 94 | 6 | 0 | forage | 3.4 3.2 | 0.33 0.22 | MP-WH-7145 |
| ND, 1988 (Marshall) | EC | 4×1.4<br>+2×0.84 | 94 | 6 | 0<br>7<br>14<br>21<br>28<br>0 | forage | 13 13<br>0.68 0.35<br>0.19 0.17<br>0.11 0.13<br>0.06 0.06<br>c 0.13 | 0.50 0.58<br>0.10 0.08<br>0.06 0.05<br><0.05 (2)<br><0.05 (2)<br>c <0.05 | MP-WH-7145 |
| KS, 1989 (Arkan) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 210 | 6 | 0 | forage | 2.3 6.0 | 0.18 0.30 | MP-WH-7147 |
| KS, 1989 (Arkan) | EC | 4×1.4<br>+2×0.84 | 210 | 6 | 0<br>7<br>14<br>21<br>28 | forage | 13 7.5<br>2.4 3.5<br>1.2 1.5<br>0.40 0.21<br>0.23 0.29 | 0.63 0.34<br>0.28 0.27<br>0.17 0.19<br><0.05 (2)<br><0.05 (2) | MP-WH-7147 |
| KS, 1989 (Caldwell) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 47 | ( 6 | 0 | forage | 7.5 7.1 | 0.32 0.31 | MP-WH-7149 |
| KS, 1989 (Caldwell) | EC | 4×1.4<br>+2×0.84 | 47 | ( 6 | 0<br>7<br>14<br>21<br>28 | forage | 5.4 6.5<br>0.35 0.23<br>0.12 0.07<br><0.05 0.07<br>0.07 0.05 | 0.27 0.29<br>0.07 0.06<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | MP-WH-7149 |
| WA, 1989 (Rojo) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 190 | 6 | 0 | hay | 3.4 3.8 | 3.4 2.4 | MP-WH-3165 |
| WA, 1989 (Rojo) | EC | 4×1.4<br>+2×0.84 | 190 | 6 | 14 | hay | 0.36 1.2 | 0.18 1.1 | MP-WH-3165 |
| WA, 1989 (Rojo) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 94 | ( 6 | 0 | hay | 3.1 2.3 | 0.35 0.24 | MP-WH-3182 |
| WA, 1989 (Rojo) | EC | 4×1.4<br>+2×0.84 | 94 | ( 6 | 14 | hay | 0.33 0.29 | 0.15 0.11 | MP-WH-3182 |
| OH, 1988 (Becker) | EC | 2×1.4<br>+3×0.84<br>+1×0.28 | 190 | 6 | 0 | hay | 3.8 3.7 | 0.54 0.58 | MP-WH-7143 |

| Location, year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| OH, 1988 (Becker) | EC | 4×1.4 +2×0.84 | 190 | 6 | 14 | hay | 0.32 1.0 | 0.13 0.23 | MP-WH-7143 |
| ND, 1988 (Marshall) | EC | 2×1.4 +3×0.84 +1×0.28 | 94 | 6 | 0 | hay | 4.7 3.9 | 0.19 0.21 | MP-WH-7145 |
| ND, 1988 (Marshall) | EC | 4×1.4 +2×0.84 | 94 | 6 | 14 | hay | 0.17 0.15 | 0.06 0.06 | MP-WH-7145 |
| KS, 1989 (Arkan) | EC | 2×1.4 +3×0.84 +1×0.28 | 210 | 6 | 0 | hay | 3.1 2.9 | 0.29 0.28 | MP-WH-7147 |
| KS, 1989 (Arkan) | EC | 4×1.4 +2×0.84 | 210 | 6 | 14 | hay | 0.98 0.83 | 0.12 0.09 | MP-WH-7147 |
| KS, 1989 (Caldwell) | EC | 2×1.4 +3×0.84 +1×0.28 | 47 | ( 6 | 0 | hay | 4.5 3.8 | 0.50 0.38 | MP-WH-7149 |
| KS, 1989 (Caldwell) | EC | 4×1.4 +2×0.84 | 47 | ( 6 | 14 | hay | 0.10 0.07 | <0.05 (2) | MP-WH-7149 |
| CA, 1989 (Anza, Hard red winter) | EC | 1.4 | 190 | 6 | 14 | straw | 4.5 3.4 | 0.59 0.28 | MP-WH-3157 |
| CA, 1989 (Anza, Hard red winter) | EC | 2×1.4 +3×0.84 +1×0.28 | 190 | 6 | 0 | straw | 5.0 4.0 | 0.22 0.16 | MP-WH-3157 |
| CA, 1989 (Anza, Hard red winter) | EC | 4×1.4 +2×0.84 | 190 | 6 | 14 | straw | 3.1 3.7 | 0.31 0.37 | MP-WH-3157 |
| WA, 1989 (Rojo) | EC | 1.4 | 190 | 6 | 14 | straw | 3.5 2.8 | 0.18 0.13 | MP-WH-3159 |
| WA, 1989 (Rojo) | EC | 2×1.4 +3×0.84 +1×0.28 | 190 | 6 | 0 | straw | 9.7 7.4 | 0.16 0.15 | MP-WH-3159 |
| WA, 1989 (Rojo) | EC | 4×1.4 +2×0.84 | 190 | 6 | 14 | straw | 2.6 2.2 | 0.16 0.12 | MP-WH-3159 |
| TX, 1989 (NK Pro 812) | EC | 1.4 | 47 | 6 | 14 | straw | 1.2 0.95 | 0.06 <0.05 | MP-WH-3161 |
| TX, 1989 (NK Pro 812) | EC | 2×1.4 +3×0.84 +1×0.28 | 47 | 6 | 0 | straw | 3.8 2.8 | 0.08 0.05 | MP-WH-3161 |
| TX, 1989 (NK Pro 812) | EC | 4×1.4 +2×0.84 | 47 | 6 | 14 | straw | 0.27 0.34 | <0.05 (2) | MP-WH-3161 |
| WA, 1989 (Rojo) | EC | 1.4 | 94 | ( 6 | 14 | straw | 1.8 1.2 | 0.15 0.18 | MP-WH-3181 |

| Location, year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| WA, 1989 (Rojo) | EC | 2×1.4 +3×0.84 +1×0.28 | 94 | ( 6 | 0 | straw | 1.9 2.5 | 0.15 0.17 | MP-WH-3181 |
| WA, 1989 (Rojo) | EC | 4×1.4 +2×0.84 | 94 | ( 6 | 14 | straw | 0.30 0.55 | 0.06 0.11 | MP-WH-3181 |
| OH, 1988 (Becher) | EC | 1.4 | 190 | 6 | 14 | straw | 1.0 0.88 | 0.11 0.09 | MP-WH-7134 |
| OH, 1988 (Becher) | EC | 2×1.4 +3×0.84 +1×0.28 | 190 | 6 | 0 | straw | 1.2 1.8 | <0.05 (2) | MP-WH-7134 |
| OH, 1988 (Becher) | EC | 4×1.4 +2×0.84 | 190 | 6 | 14 | straw | 0.74 0.79 | 0.09 0.11 | MP-WH-7134 |
| VA, 1988 (Coker 797) | EC | 1.4 | 220 | 6 | 14 | straw | <0.05 (2) | <0.05 (2) | MP-WH-7136 |
| VA, 1988 (Coker 797) | EC | 2×1.4 +3×0.84 +1×0.28 | 220 | 6 | 0 | straw | 12 21 | 0.14 0.24 | MP-WH-7136 |
| VA, 1988 (Coker 797) | EC | 4×1.4 +2×0.84 | 220 | 6 | 14 | straw | 0.13 <0.05 | <0.05 (2) | MP-WH-7136 |
| ND, 1988 (Marshall) | EC | 1.4 | 94 | 6 | 14 | straw | 0.16 0.10 | <0.05 (2) | MP-WH-7138 |
| ND, 1988 (Marshall) | EC | 2×1.4 +3×0.84 +1×0.28 | 94 | 6 | 0 | straw | 3.5 2.7 | 0.26 0.17 | MP-WH-7138 |
| ND, 1988 (Marshall) | EC | 4×1.4 +2×0.84 | 94 | 6 | 14 | straw | 0.28 0.09 | <0.05 (2) | MP-WH-7138 |
| KS, 1989 (Arkan) | EC | 1.4 | 210 | 6 | 14 | straw | 11 10 | 0.26 0.23 | MP-WH-7140 |
| KS, 1989 (Arkan) | EC | 2×1.4 +3×0.84 +1×0.28 | 210 | 6 | 0 | straw | 13 14 | 0.24 0.32 | MP-WH-7140 |
| KS, 1989 (Arkan) | EC | 4×1.4 +2×0.84 | 210 | 6 | 14 | straw | 4.0 5.7 | 0.11 0.17 | MP-WH-7140 |
| KS, 1989 (Caldwell) | EC | 1.4 | 47 | ( 6 | 14 | straw | 0.77 1.6 | <0.05 0.10 | MP-WH-7142 |
| KS, 1989 (Caldwell) | EC | 2×1.4 +3×0.84 +1×0.28 | 47 | ( 6 | 0 | straw | 0.49 0.74 | <0.05 0.05 | MP-WH-7142 |
| KS, 1989 (Caldwell) | EC | 4×1.4 +2×0.84 | 47 | ( 6 | 14 | straw | 0.65 0.85 | 0.05 0.08 | MP-WH-7142 |

( aerial application. c: sample from control plot

Table 53. Residues in pasture grasses from supervised trials in the USA. In the 1998 trials two applications per cutting were made at 7 ± 1 day intervals. Forage and hay samples were collected on the day of the second application at each cutting, and the hay placed in drying racks for 3-5 days. In the 1988 trials the hay was left in the field to dry for 2-5 days.

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | % moisture[2] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water l/ha | no./ cut | | | | parathion-methyl | paraoxon-methyl | |
| CA, 1998 (Bermuda Grass) | EC | 0.84 | 29 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 13.3<br>7.6 | 35 34<br>27 31 | 1.2 1.1<br>1.3 1.6 | 9803.CA.GR.12 |
| FL, 1998 (Coastal Bermuda Grass) | EC | 0.84 | 140 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 24.8<br>34.0 | 6.7 6.1<br>34 25 | 0.07 0.07<br>0.57 0.44 | 9803.FL.GR.04 |
| GA, 1998 (Coastal Bermuda Grass) | EC | 0.84 | 160 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 13.5<br>16.3<br>13.5<br>16.3 | 51 59<br>17 17<br>c 1.0<br>c 0.02 | 0.72 0.74<br>0.31 0.33<br>c 0.02<br>c <0.01 | 9803.GA.GR.03 |
| GA, 1998 (Coastal Bermuda Grass) | EC | 0.84 | 10 | ( 2<br>( 2 | 0<br>0 | hay 1<br>hay 2 | 13.5<br>16.3<br>13.5<br>16.3 | 53 62<br>6.2 7.8<br>c 1.0<br>c 0.02 | 1.3 1.5<br>0.20 0.25<br>c 0.02<br>c <0.01 | 9803.GA.GR.03 |
| IA, 1998 (Brome Grass) | EC | 0.84 | 120 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 48.1<br>26.4 | 12 13<br>80 82 | 0.52 0.61<br>1.6 1.5 | 9803.IA.GR.07 |
| MT, 1998 (Brome Grass, Meadow) | EC | 0.84 | 31 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 31.4<br>16.2<br>31.4<br>16.2 | 45 46<br>72 73<br>c 0.02<br>c 0.09 | 1.9 1.5<br>1.4 1.4<br>c 0.02<br>c <0.01 | 9803.MT.GR.10 |
| MT, 1998 (Brome Grass, Meadow) | EC | 0.84 | 9 | ( 2<br>( 2 | 0<br>0 | hay 1<br>hay 2 | 31.4<br>16.2<br>31.4<br>16.2 | 10 14<br>78 140<br>c 0.02<br>c 0.09 | 0.64 0.81<br>1.5 2.4<br>c 0.02<br>c <0.01 | 9803.MT.GR.10 |
| PA, 1998 (Fescue, Festorina) | EC | 0.84 | 300 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 29.0<br>30.6<br>29.0<br>30.6 | 62 61<br>195 187<br>c 0.02<br>c 0.01 | 3.5 3.3<br>4.4 4.4<br>c <0.01<br>c <0.01 | 9803.PA.GR.01 |
| TX, 1998 (Brome grass, MATUA) | EC | 0.84 | 130 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 46.5<br>31.2<br>31.2 | 4.2 3.8<br>74 63<br>c 0.01 | 0.03 0.03<br>0.94 0.86<br>c <0.01 | 9803.TX.GR.08 |
| WA, 1998 (Orchard grass) | EC | 0.84 | 28 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 21.0<br>9.9 | 19 18<br>30 29 | 0.86 0.76<br>0.33 0.36 | 9803.WA.GR.11 |
| WA, 1998 (Orchard grass) | EC | 0.84 | 9 | ( 2<br>( 2 | 0<br>0 | hay 1<br>hay 2 | 21.0<br>9.9 | 23 23<br>31 24 | 0.72 0.64<br>0.29 0.22 | 9803.WA.GR.11 |
| WI, 1998 (Brome grass) | EC | 0.84 | 47 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 27.0<br>38.2 | 21 19<br>54 54 | 0.84 0.67<br>0.75 0.77 | 9803.WI.GR.06 |
| WI, 1998 (Brome grass) | EC | 0.84 | 9 | ( 2<br>( 2 | 0<br>0 | hay 1<br>hay 2 | 27.0<br>38.2 | 50 54<br>62 52 | 1.7 1.6<br>1.5 1.6 | 9803.WI.GR.06 |

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | % moisture[2] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water l/ha | no./ cut | | | | parathion-methyl | paraoxon-methyl | |
| NJ, 1998 (Fescue, Kentucky 31) | EC | 0.84 | 300 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 27.9<br>19.9 | 28 24<br>110 108 | 1.1 1.0<br>5.4 4.8 | 9803.NJ.GR.02 |
| OH, 1998 (Fescue, Kentucky 31) | EC | 0.84 | 79 | 2<br>2 | 0<br>0 | hay 1<br>hay 2 | 16.2<br>17.4<br>17.4 | 17 17<br>63 58<br>c 0.02 | 0.67 0.71<br>3.2 3.3<br>c <0.01 | 9803.OH.GR.05 |
| OH, 1998 (Fescue, Kentucky 31) | EC | 0.84 | 9 | ( 2<br>( 2 | 0<br>0 | hay 1<br>hay 2 | 16.2<br>17.4<br>17.4 | 11 11<br>44 46<br>c 0.02 | 0.54 0.62<br>1.3 1.3<br>c <0.01 | 9803.OH.GR.05 |
| MT, 1998 (Brome Grass, Meadow) | EC | 0.84 | 29 | 2 | 0 | hay 1 | 50.2 | 35 | 2.6 | 9803.MT.GR.09 |
| MT, 1998 (Brome Grass, Meadow) | EC | 0.84 | 10 | ( 2 | 0 | hay 1 | 50.2 | 7.3 5.4 | 1.1 0.75 | 9803.MT.GR.09 |
| CA, 1988 (Bermuda grass) | EC | 0.89 | 190 | 3 | 15 | hay | | 0.63 1.4 | <0.05 0.06 | MP-BE-3009 |
| CA, 1988 (Bermuda grass) | EC | 0.89 | 190 | ( 3 | 15 | hay | | 0.49 0.54 | <0.05 (2) | MP-BE-3012 |
| GA, 1988 (Bermuda grass, coastal) | EC | 0.86 | 62 | 6 | 15 | hay | | 1.6 1.4 | <0.05 (2) | MP-BE-7059 |
| GA, 1988 (Bermuda grass, coastal) | EC | 0.87 | 96 | ( 6 | 15 | hay | | 0.96 0.49 | <0.05 (2) | MP-BE-7060 |
| SC, 1988 (Bermuda grass, coastal) | EC | 0.89 | 37 | 6 | 15 | hay | | 0.66 0.63 | <0.05 (2) | MP-BE-7061 |
| CA, 1988 (Bluegrass) | EC | 0.89 | 190 | 3 | 15 | hay | | 0.22 0.31 | 0.19 0.13 | MP-BL-3013 |
| CA, 1988 (Bluegrass) | EC | 0.89 | 190 | ( 3 | 15 | hay | | 0.58 0.64 | 0.09 0.06 | MP-BL-3016 |
| MO, 1988 (Bluegrass) | EC | 0.87 | 190 | 6 | 15 | hay | | 0.17 0.21 | 0.11 0.10 | MP-BL-7062 |
| MO, 1988 (Bluegrass) | EC | 0.89 | 33 | ( 6 | 15 | hay | | 0.12 0.09 | 0.10 0.09 | MP-BL-7063 |
| PA, 1988 (Bluegrass, Merit/Nassau/Baron) | EC | 0.89 | 250 | 6 | 15 | hay | | 1.0 0.97 | 0.11 0.12 | MP-BL-7064 |
| CA, 1988 (Fescue) | EC | 0.89 | 190 | 3 | 15 | hay | | 0.50 0.34 | 0.09 0.06 | MP-BO-3017 |
| CA, 1988 (Fescue) | EC | 0.89 | 190 | ( 3 | 15 | hay | | 0.25 0.23 | <0.05 (2) | MP-BO-3020 |
| MO, 1988 (Fescue, Jaguar) | EC | 0.87 | 190 | 6 | 15 | hay | | 0.19 0.11<br>c 0.07 | 0.07 0.05<br>c <0.05 | MP-BO-7065 |
| MO, 1988 (Fescue, Jaguar) | EC | 0.89 | 33 | ( 6 | 15 | hay | | <0.05<br>0.05<br>c 0.10 | <0.05 0.08<br>c <0.05 | MP-BO-7066 |
| PA, 1988 (Fescue, Tall and fine) | EC | 0.89 | 260 | 6 | 15 | hay | | 2.5 2.5 | 0.35 0.32 | MP-BO-7067 |

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | % moisture[2] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water l/ha | no./ cut | | | | parathion-methyl | paraoxon-methyl | |
| CA, 1998 (Bermuda Grass) | EC | 0.84 | 29 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 68.8<br>57.9 | 74 68<br>31 32 | 1.8 1.9<br>0.91 1.1 | 9803.CA.GR.12 |
| FL, 1998 (Coastal Bermuda Grass) | EC | 0.84 | 140 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 67.2<br>76.3 | 9.1 7.2<br>32 26 | 0.16 0.15<br>0.50 0.52 | 9803.FL.GR.04 |
| GA, 1998 (Coastal Bermuda Grass) | EC | 0.84 | 160 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 64.0<br>56.5<br>64.0<br>56.5 | 35 40<br>31 34<br>c 0.51<br>c 0.02 | 0.29 0.37<br>0.45 0.51<br>c <0.01<br>c <0.01 | 9803.GA.GR.03 |
| GA, 1998 (Coastal Bermuda Grass) | EC | 0.84 | 10 | ( 2<br>( 2 | 0<br>0 | forage 1<br>forage 2 | 64.0<br>56.5<br>64.0<br>56.5 | 12 14<br>13 13<br>c 0.51<br>c 0.02 | 0.28 0.36<br>0.36 0.37<br>c <0.01<br>c <0.01 | 9803.GA.GR.03 |
| IA, 1998 (Brome Grass) | EC | 0.84 | 120 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 66.7<br>60.5 | 24 23<br>69 98 | 0.37 0.49<br>1.1 1.7 | 9803.IA.GR.07 |
| MT, 1998 (Brome Grass, Meadow) | EC | 0.84 | 31 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 70.8<br>57.0<br>57.0 | 74 74<br>75 90<br>c 0.02 | 0.87 0.78<br>0.77 0.89<br>c <0.01 | 9803.MT.GR.10 |
| MT, 1998 (Brome Grass, Meadow) | EC | 0.84 | 9 | ( 2<br>( 2 | 0<br>0 | forage 1<br>forage 2 | 70.8<br>57.0<br>57.0 | 18 32<br>42 19<br>c 0.02 | 0.43 0.55<br>0.61 0.39<br>c <0.01 | 9803.MT.GR.10 |
| PA, 1998 (Fescue, Festorina) | EC | 0.84 | 300 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 74.2<br>68.0 | 56 53<br>125 123 | 0.82 1.0<br>0.63 0.63 | 9803.PA.GR.01 |
| TX, 1998 (Brome grass, MATUA) | EC | 0.84 | 130 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 58.3<br>69.8 | 46 54<br>95 107 | 0.42 0.44<br>0.57 0.55 | 9803.TX.GR.08 |
| WA, 1998 (Orchard grass) | EC | 0.84 | 28 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 72.0<br>76.8<br>76.8 | 28 19<br>38 32<br>c 0.01 | 0.87 0.63<br>0.12 0.08<br>c <0.01 | 9803.WA.GR.11 |
| WA, 1998 (Orchard grass) | EC | 0.84 | 9 | ( 2<br>( 2 | 0<br>0 | forage 1<br>forage 2 | 72.0<br>76.8<br>76.8 | 23 30<br>18 39<br>c 0.01 | 0.31 0.35<br>0.07 0.17<br>c <0.01 | 9803.WA.GR.11 |
| WI, 1998 (Brome grass) | EC | 0.84 | 9 | ( 2<br>( 2 | 0<br>0 | forage 1<br>forage 2 | 70.2<br>74.9 | 34 30<br>83 46 | 0.83 0.75<br>1.0 0.62 | 9803.WI.GR.06 |
| WI, 1998 (Brome grass) | EC | 0.84 | 47 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 70.2<br>74.9 | 17 14<br>44 36 | 0.61 0.46<br>0.55 0.37 | 9803.WI.GR.06 |
| NJ, 1998 (Fescue, Kentucky 31) | EC | 0.84 | 300 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 73.0<br>66.3 | 37 29<br>70 71 | 0.95 0.93<br>1.1 1.2 | 9803.NJ.GR.02 |
| OH, 1998 (Fescue, Kentucky 31) | EC | 0.84 | 9 | ( 2<br>( 2 | 0<br>0 | forage 1<br>forage 2 | 58.5<br>71.2 | 17 16<br>42 46 | 0.95 0.81<br>0.81 0.85 | 9803.OH.GR.05 |
| OH, 1998 (Fescue, Kentucky 31) | EC | 0.84 | 79 | 2<br>2 | 0<br>0 | forage 1<br>forage 2 | 58.5<br>71.2 | 24 29<br>75 62 | 0.66 0.83<br>2.1 2.3 | 9803.OH.GR.05 |

| Location, year (variety) | Application | | | | PHI, days | Sample[1] | % moisture[2] | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water l/ha | no./ cut | | | | parathion-methyl | paraoxon-methyl | |
| MT, 1998 (Brome Grass, Meadow) | EC | 0.84 | 29 | 2 | 0 | forage 1 | 58.2 | 66 56 | 0.29 0.25 | 9803.MT.GR.09 |
| MT, 1998 (Brome Grass, Meadow) | EC | 0.84 | 10 | ( 2 | 0 | forage 1 | 58.2 | 20 21 | 0.34 0.37 | 9803.MT.GR.09 |

[1] Forage 1, forage 2, hay 1, hay 2 refer to the 1st and 2nd cuttings.
[2] Moisture levels were measured on the accompanying control sample.
( aerial application. c: sample from control plot

Table 54. Parathion-methyl and paraoxon-methyl residues in beet fodder (Cañez, 1990q) and cotton gin trash from supervised trials in the USA. Double-underlined residues are from treatments according to GAP and are valid for estimating maximum residue levels.

| Crop, Location, year (variety) | Application | | | | Growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| BEET FODDER | | | | | | | | | |
| ID, 1988 (WS 88) | EC | 0.42 | 190 | 6 | | 60 | <0.05 (2) | <0.05 (2) | MP-SB-3144 |
| CA, 1988 (SS-NB2) | EC | 0.42 | 190 | 6 | | 60 | <0.05 (2) | <0.05 (2) | MP-SB-3146 |
| CA, 1988 (SS-NB2) | EC | 0.42 | 190 | ( 6 | | 60 | <0.05 (2) | <0.05 (2) | MP-SB-3148 |
| ID, 1988 (WS 88) | EC | 0.42 | 94 | ( 6 | | 60 | <0.05 (2) | <0.05 (2) | MP-SB-3186 |
| MN, 1988 (Ultramono) | EC | 0.42 | 190 | 6 | | 60 | <0.05 (2) | <0.05 (2) | MP-SB-7124 |
| ND, 1988 (ACS ACH 176) | EC | 0.42 | 94 | 6 | | 60 | <0.05 (2) | <0.05 (2) | MP-SB-7126 |
| COTTON GIN TRASH | | | | | | | | | |
| AR, 1998 (Paymaster 1220) | EC | 3.4 | 28 | 10 | 90% open | 1 | 32 43<br>c 0.08 | 0.46 0.66<br>c <0.01 | 9802.AR.CT.02(a) |
| AR, 1998 (Paymaster 1220) | EC | 3.4 | 9 | ( 10 | 90% open | 1 | 41 67 | 0.71 0.60 | 9802.AR.CT.02(b) |
| CA, 1998 (Maxxa) | EC | 3.4 | 280 | 10 | 70-80% open | 1 | 309 325<br>c 0.46 | 3.8 3.7<br>c 0.04 | 9802.CA.CT.11 |
| TX, 1998 (Paymaster 1220) | EC | 3.4 | 92 | 10 | 90% open | 1 | 200 131<br>c 0.21 | 2.0 1.6<br>c <0.01 | 9802.TX.CT.06(a) |
| TX, 1998 (Paymaster 1220) | EC | 3.4 | 8 | ( 10 | 90% open | 1 | 440 280<br>c 0.21 | 3.1 2.3<br>c <0.01 | 9802.TX.CT.06(b) |
| TX, 1998 (PM 2200 RR) | EC | 3.4 | 94 | 10 | 90% open | 1 | 27 33<br>c 0.07 | 0.93 1.1<br>c <0.01 | 9802.TX.CT.07 |
| TX, 1998 (Quikee) | EC | 3.4 | 9 | ( 10 | 85% open | 1 | 43 39<br>c 1.3 | 0.27 0.16<br>c <0.01 | 9802.TX.CT.08 |

| Crop, Location, year (variety) | Application | | | | Growth stage | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| TX, 1998 (Quikee) | EC | 3.4 | 120 | 10 | 85% open | 1 | 65 85<br>c 1.3 | 0.30 0.37<br>c <0.01 | 9802.TX.CT.08(a) |
| TX, 1998 (Paymaster 2200 | EC | 3.4 | 120 | 10 | 95% open | 1 | 114 136 | 0.63 0.60 | 9802.TX.CT.09(a) |
| TX, 1998 (Paymaster 2200 | EC | 3.4 | 9 | ( 10 | 95% open | 1 | 82 75 | 0.42 0.41 | 9802.TX.CT.09(b) |

( aerial application. c: sample from control plot

Table 55. Residues in hops from supervised trials in the USA.

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | Sample | parathion-methyl | paraoxon-methyl | |
| OR, 1990 (Nugget) | EC | 1.1<br>2.1<br>1.1<br>2.1 | 1400<br>1400<br>470<br>470 | 3<br>3<br>3<br>3 | 15<br>15<br>15<br>15 | fresh | 0.12<br>0.28<br>0.096<br>0.39 | 0.036<br>0.12<br><0.04<br>0.11 | 90:OR.017 |
| OR, 1990 (Nugget) | EC | 1.1<br>2.1<br>1.1<br>2.1 | 1400<br>1400<br>470<br>470 | 3<br>3<br>3<br>3 | 15<br>15<br>15<br>15 | dry | 0.43<br>0.78<br>0.40<br>0.87 | 0.16<br>0.29<br>0.17<br>0.32 | 90:OR.017 |
| ID, 1990 (Galena) | EC | 1.1<br>2.1<br>1.1<br>2.1 | 1400<br>1400<br>470<br>470 | 3<br>3<br>3<br>3 | 15<br>15<br>15<br>15 | fresh | 0.18<br>0.17<br>0.18<br>0.25 | 0.10<br>0.058<br>0.15<br>0.09 | 90:ID:005 |
| ID, 1990 (Galena) | EC | 1.1<br>2.1<br>1.1<br>2.1 | 1400<br>1400<br>470<br>470 | 3<br>3<br>3<br>3 | 15<br>15<br>15<br>15 | dry | 0.23<br>0.27<br>0.41<br>0.37 | 0.18<br>0.23<br>0.19<br>0.25 | 90:ID:005 |
| WA, 1990 (L1) | EC | 1.0<br>2.0<br>1.0<br>2.0 | 2100<br>2100<br>700<br>700 | 4<br>4<br>4<br>4 | 14<br>14<br>14<br>14 | fresh | 0.15<br>0.35<br>0.46<br>0.96 | 0.042<br>0.11<br>0.067<br>0.21 | 90:WA:015 |
| WA, 1990 (L1) | EC | 1.0<br>2.0<br>1.0<br>2.0 | 2100<br>2100<br>700<br>700 | 4<br>4<br>4<br>4 | 14<br>14<br>14<br>14 | dry | 0.36<br>1.2<br>0.99<br>2.7 | 0.13<br>0.41<br>0.35<br>0.90 | 90:WA:015 |

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### In processing

The Meeting received information on the fate of incurred residues of parathion-methyl and paraoxon-methyl during the processing of apples, peaches, grapes, olives, snap beans, soya beans, potatoes, sugar beet, wheat, maize, rice, cotton seed, sunflower seed and canola. Information on the fate during drying of hops is included in the supervised residue trials.

Apples. Apples from supervised trials in France (Table 20) were processed by Gilbert (1996h). 26-30 kg of apples were crushed and pressed (Figure 6). Residues were below the LOQ (0.01 mg/kg) in the juice, and about 4 times those in the apples in the dry pomace (Table 56).

Table 56. Residues in apples from supervised trials in France and their processed commodities (Gilbert, 1996h).

| Year (variety) | Application | | | | | PHI, days | sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| 1995 (Jonagold) | CS | 0.36 | 0.036 | 1000 | 2 | 14 | apples | 0.04 | <0.01 | Loiret F1 |
| | | | | | | | dry pomace | 0.17 | <0.01 | 961451 |
| | | | | | | | juice | <0.01 | <0.01 | |
| | | | | | | 21 | apples | 0.02 | <0.01 | 962321 |
| | | | | | | | dry pomace | 0.09 | <0.01 | |
| | | | | | | | juice | <0.01 | <0.01 | |
| 1995 (Golden Delicious) | CS | 0.36 | 0.036 | 1000 | 2 | 14 | apples | 0.03 | <0.01 | Loiret F2 |
| | | | | | | | dry pomace | 0.12 | <0.01 | 961451 |
| | | | | | | | juice | <0.01 | <0.01 | |
| | | | | | | 21 | apples | 0.01 | <0.01 | 962321 |
| | | | | | | | dry pomace | 0.08 | <0.01 | |
| | | | | | | | juice | <0.01 | <0.01 | |

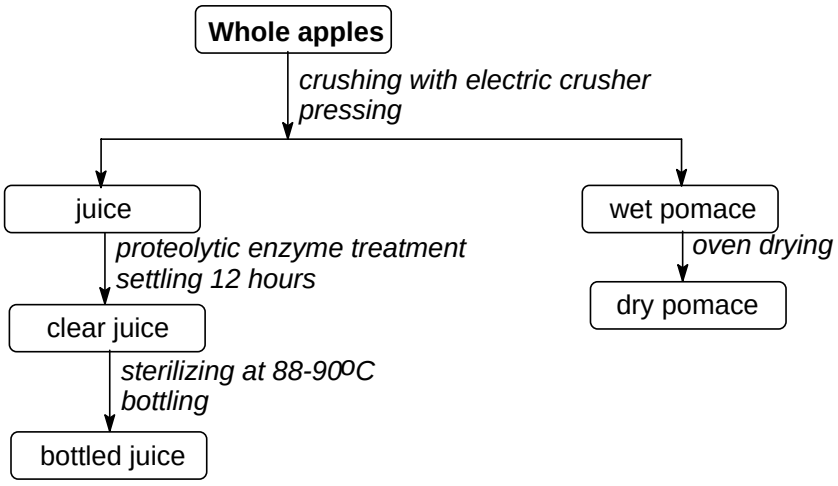



Figure 6. Apple processing (Gilbert, 1996h).

Peaches. Gilbert (1996g) described the processing of peaches to juice. A sample of 20 mature peaches, bruised fruit having been discarded, were cut with a knife and the stones removed. The peach pieces were crushed and juice separated from skin with an automatic sieve. The same procedure was followed by Gilbert and Roberts (1996). The residues in the juice are included in Table 21 (Trials 9551-4, 9551-5, 9550-4, 9550-5).

Grapes. In processing trials in Germany (407755, 407763, 407771, 407798, 407801 and 407828, Table 22) field samples of 41-52 kg were harvested 35 days after the final treatment.

Approximately 46 kg from trials 407771 and 407828 were processed to white wine. Grapes, complete with stems, were crushed, pressed and the sugar adjusted to a standard level. A volume of about 23 l of the must was transferred to a demijohn and yeast added. Fermentation proceeded for 34 days, and the young wine was treated with $SO_2$ and filtered after clarification for 10 days. Approximately 40 kg of grapes from trials 407755, 407763, 407798 and 407801 were processed to red wine. Grapes were destemmed and mashed, before heating to 83°C for about 3 minutes and then cooling to 25°C. The mash was pressed, the sugar level adjusted to a standard value, and processing completed as described above for white wine. The residues in the must and wine are shown in Table 22 with the supervised trials. In all cases parathion-methyl residues in the must and wine were below the LOQ (0.01 mg/kg), but residues in the grapes were also below LOQ in three of the trials. In two of the trials the parathion-methyl residues in grapes were 0.12 and 0.13 mg/kg, suggesting that parathion-methyl levels decrease by a factor of at least 12-13 in vinification (Blass and Waltz-Tylla, 1996).

Gilbert (1997b) described the processes (Figure 7) for wine, juice and raisin production from grapes treated with CS formulations of parathion-methyl in supervised trials (Table 57). Parathion-methyl levels in wine and juice were much lower than in the grapes while levels in raisins were 30-50% higher. After drying and destemming 1 kg of raisins was produced from 8 kg grapes.

Table 57. Parathion-methyl and paraoxon-methyl residues in grapes and their processed commodities in France (Gilbert, 1997b). The supervised trials are listed in Table 22.

| Year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| 1995 (Tannat) | CS | 0.30 | 200 | 2 | 14 | grapes | 0.15 | <0.01 | Garonne F6 960539 |
| | | | | | 14 | raisins | 0.22 | <0.01 | |
| | | | | | 14 | wine | 0.02 | <0.01 | |
| | | | | | 14 | juice | <0.01 | <0.01 | |
| | | | | | 21 | grapes | 0.10 | <0.01 | |
| | | | | | 21 | raisins | 0.13 | <0.01 | |
| | | | | | 21 | wine | 0.02 | <0.01 | |
| | | | | | 21 | juice | <0.01 | <0.01 | |
| | | | | | 21 | raisins | c 0.01 | | |
| 1995 (Syrah) | CS | 0.30 | 200 | 2 | 14 | grapes | 0.16 | <0.01 | Tarn-et-Garonne F5 960539 |
| | | | | | 14 | raisins | 0.21 | <0.01 | |
| | | | | | 14 | wine | 0.03 | <0.01 | |
| | | | | | 14 | juice | 0.01 | <0.01 | |
| | | | | | 21 | grapes | 0.09 | <0.01 | |
| | | | | | 21 | raisins | 0.14 | <0.01 | |
| | | | | | 21 | wine | 0.02 | <0.01 | |
| | | | | | 21 | juice | <0.01 | <0.01 | |
| | | | | | 21 | raisins | c 0.01 | | |

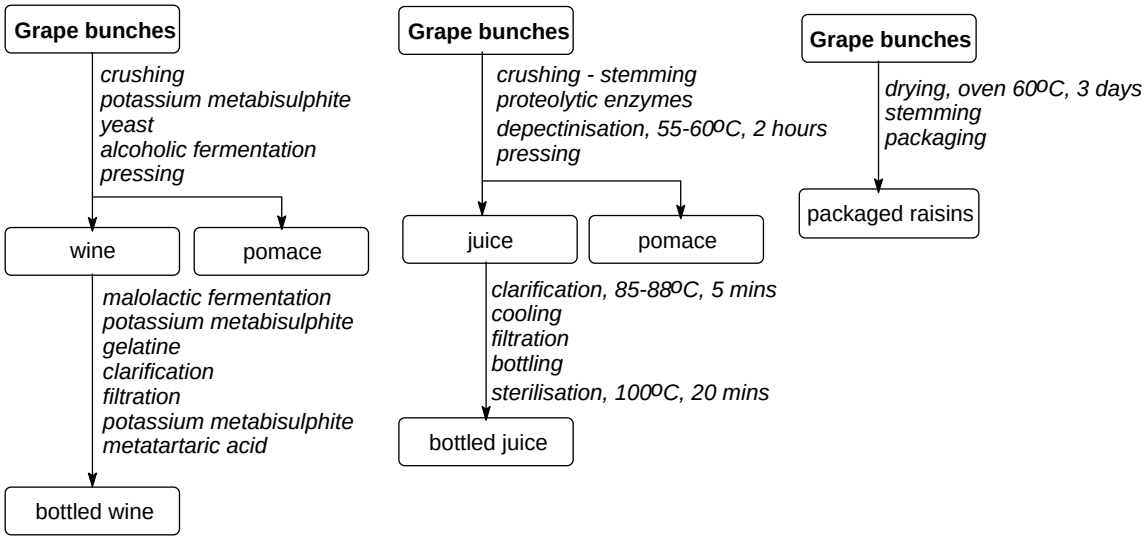



Figure 7. Processing of grapes to wine, juice and raisins (Gilbert, 1997b).

Olives. In a trial in Spain in 1994, olive trees were treated once with either a CS or EC parathion-methyl formulation (Fitexp, 1995). Olives were harvested at intervals after treatment and processed to oil. Details of the process and the analytical method were not available. Parathion-methyl residues in the oil were about 5-8 times those in the olives.

Table 58. Residues in olives and olive oil from trials in Spain in 1994 (Fitexp, 1995).

| Year (variety) | Application | | | PHI, days | parathion-methyl residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | no. | | olives | olive oil | |
| 1994 (Lechín) | CS | 0.495 | 1 | 0 | 4.7 | 30 | Fitexp, 1995 |
| | | | | 7 | 0.55 | 2.6 | |
| | | | | 14 | 0.44 | 1.1 | |
| | | | | 21 | 0.24 | 1.6 | |
| | | | | 28 | 0.19 | 0.35 | |
| 1994 (Lechín) | EC | 0.49 | 1 | 0 | 6.2 | 59 | Fitexp, 1995 |
| | | | | 7 | 3.4 | 18 | |
| | | | | 14 | 1.5 | 13 | |
| | | | | 21 | 1.2 | 5.4 | |
| | | | | 28 | 0.98 | 11 | |

Snap beans. LeRoy (1990c) described the processing of snap beans in trials MP-LB-3508 and MP-LB-3509 (Figure 8). Residues of parathion-methyl and paraoxon-methyl were below the LOQ of 0.05 mg/kg in the raw agricultural commodity and the cut pods even at exaggerated application rates. Residues and supervised trial conditions are shown in Table 30.

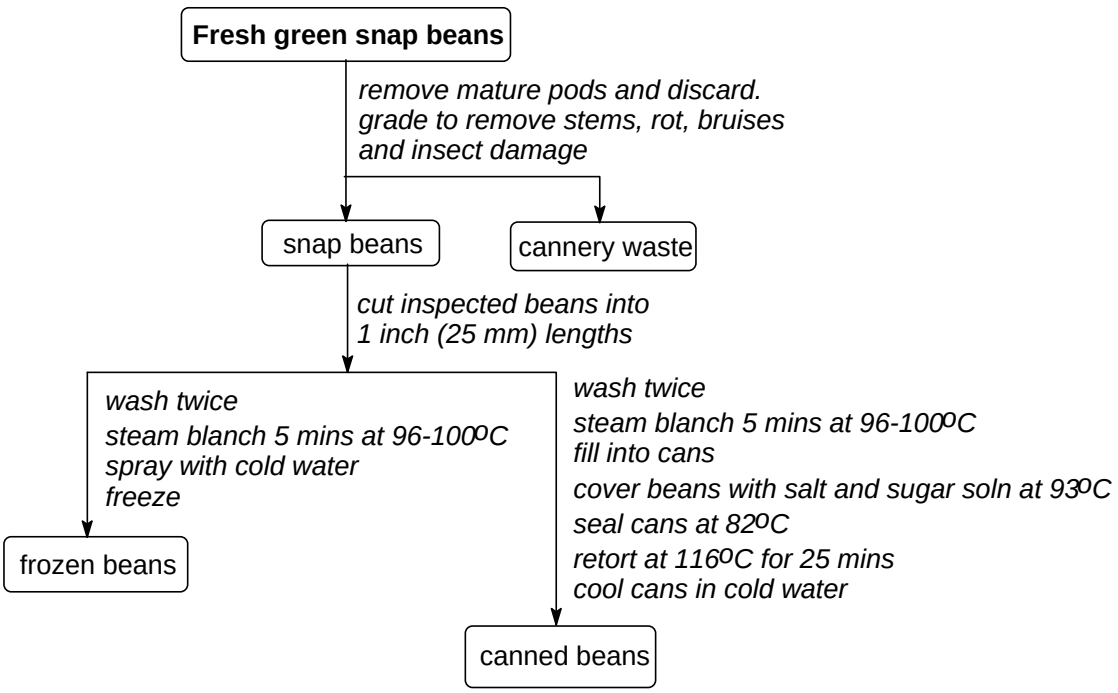



Figure 8. Processing of snap beans (LeRoy, 1990c).

Soya beans. Parathion-methyl was applied twice at 2.8 kg ai/ha (5 times the label rate) in two trials in the USA in 1988 and the beans harvested 15 days after the final treatment for processing (Figure 9). In one trial (MP-SY-2102) the residues were below the LOQ in all commodities. In trial MP-SY-2101 parathion-methyl levels were lower in the meal and higher in the oils (Table 59).

In similar trials in 1990 (LeRoy, 1992), parathion-methyl residues were below the LOQ in the seed in trial MP-SY-3525-IA but measurable in the oils. In trial MP-SY-3525-MO parathion-methyl residues were decreased in the meal and increased about 3-fold in the oils (Table 59). Processing of 28 and 32 kg soya bean seed followed the procedure in Figure 9.

Table 59. Parathion-methyl and paraoxon-methyl residues in soya beans and their processed commodities in the USA (Cañez, 1990o, LeRoy, 1992).

| Location, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| IA, 1988 (Pioneer 9271) | EC | 2.8 | | 200 | 2 | 15 | dry seed | 0.15 | <0.05 | MP-SY-2101 |
| | | | | | | | meal | <0.05 | <0.05 | |
| | | | | | | | hulls | 0.12 | <0.05 | |
| | | | | | | | crude oil | 0.71 | <0.1 | |
| | | | | | | | refined oil | 0.57 | <0.1 | |
| MO, 1988 (Williams 82) | EC | 2.8 | | 200 | 2 | 15 | dry seed | <0.05 | <0.05 | MP-SY-2102 |
| | | | | | | | meal | <0.05 | <0.05 | |
| | | | | | | | hulls | <0.05 | <0.05 | |
| | | | | | | | crude oil | <0.1 | <0.1 | |
| | | | | | | | refined oil | <0.1 | <0.1 | |

| Location, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| IA, 1990 (DK 265) | EC | 2.8 | | 190 | 6 | 15 | seed | <0.05 | <0.05 | MP-SY-3525-IA |
| | | | | | | | meal | <0.1 | <0.1 | |
| | | | | | | | hulls | <0.05 | <0.05 | |
| | | | | | | | crude oil | 0.06 | <0.05 | |
| | | | | | | | refined oil | 0.07 | <0.05 | |
| | | | | | | | soapstock | <0.05 | note [1] | |
| MO, 1990 (Williams 82) | EC | 2.8 | | 190 | 6 | 15 | seed | 0.17 | <0.05 | MP-SY-3525-MO |
| | | | | | | | meal | <0.1 | <0.1 | |
| | | | | | | | hulls | 0.19 | <0.05 | |
| | | | | | | | crude oil | 0.51 | <0.05 | |
| | | | | | | | refined oil | 0.49 | <0.05 | |
| | | | | | | | soapstock | 0.06 | note [1] | |

[1]paraoxon-methyl recoveries too low for quantification

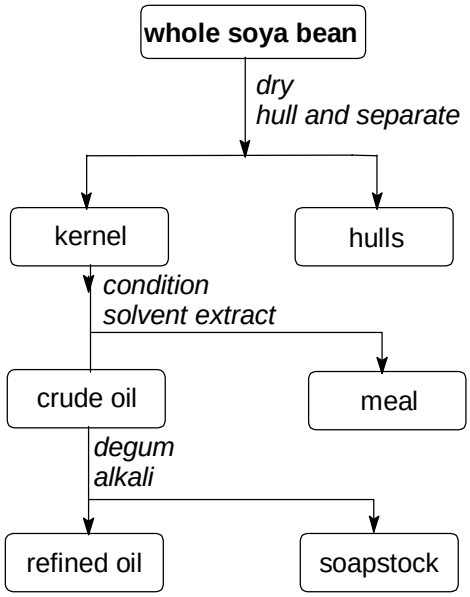



Figure 9. Soya bean processing (Cañez, 1990o).

Potatoes. Parathion-methyl was applied six times to potatoes at 3.4 and 8.4 kg ai/ha (2 and 5 times the label rate) in two trials in the USA in 1988 and 1989 and 68 kg tubers were harvested 5 days after the final treatment for processing (Cañez, 1990p). In both trials (MP-PO-3501 and MP-PO-3502) neither parathion-methyl nor paraoxon-methyl exceeded the LOQ (0.05 mg/kg) in the tubers or the processed commodities. The data are recorded in Table 35. In trial MP-PO-3502 the dry and wet peel samples originated from the flake process because they are normally fed to livestock. The dry and wet peel samples from the chip process were discarded because this is normal commercial practice.

Sugar beet. Parathion-methyl was applied six times to sugar beet at 2.1 kg ai/ha (5 times the label rate) in two trials in the USA in 1988 and the crops were harvested (140 kg roots) 20 days after the final treatment for processing (Cañez, 1990q). In both trials (MP-SB-3503 and MP-SB-3504) neither

parathion-methyl nor paraoxon-methyl exceeded the LOQ (0.05 mg/kg) in the sugar beet roots, molasses or refined sugar. The data are recorded in Table 37.

Wheat. Parathion-methyl was applied six times to wheat at 7.0 kg ai/ha (exaggerated rate) in two trials in the USA in 1988 and 1989 and the crops were harvested (18 and 27 kg grain) 13-14 days after the final treatment for milling (LeRoy, 1990d). Figure 10 is a schematic diagram of the milling process; Table 60 summarizes the results. Residues were concentrated in the bran, shorts and germ and decreased in the flour.

Table 60. Residues in wheat and its processed commodities from supervised trials in the USA (LeRoy, 1990d). EC formulations.

| Location, year (variety) | Application | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| MO, 1988 (Caldwell) | 7.0 | 260 | 6 | 13 | grain | 6.2 | 0.13 | MP-WH-2103 |
| | | | | | rough | 11 | 0.28 | |
| | | | | | bran | 15 | 0.19 | |
| | | | | | flour | 2.6 | 0.05 | |
| | | | | | flour, low grade | 1.5 | 0.09 | |
| | | | | | middlings | 4.4 | 0.08 | |
| | | | | | red dog | 5.5 | 0.10 | |
| | | | | | shorts and germ | 20 | 0.29 | |
| WA, 1989 (Rojo) | 7.0 | 190 | 6 | 14 | grain | 9.8 | 0.15 | MP-WH-3520 |
| | | | | | bran | 19 | 0.26 | |
| | | | | | flour, patent | 2.8 | 0.09 | |
| | | | | | middlings | 4.9 | 0.12 | |
| | | | | | red dog | 4.2 | 0.14 | |
| | | | | | shorts and germ | 9.0 | 0.24 | |

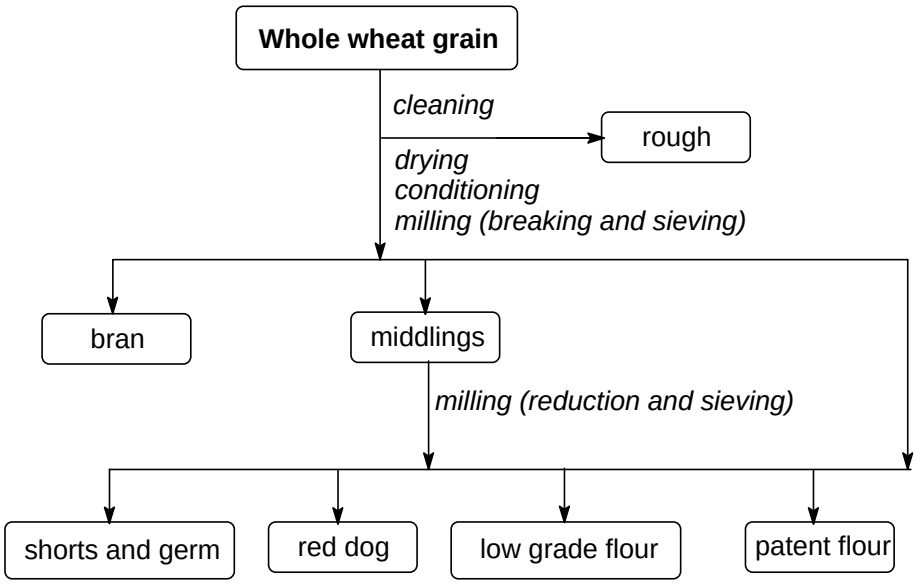



Figure 10. Wheat milling (LeRoy, 1990d).

Williams and Rice (1999b) measured the residues in aspirated grain fractions (AGF or grain dust) of field-treated wheat in Georgia, USA, in 1998. The crop was sprayed 6 times with parathion-methyl at 1.4 kg ai/ha using a tractor-mounted boom sprayer at about 7-day intervals and harvested 15 days after the final treatment. The harvested grain was put on a simulated commercial grain bucket elevator and drag conveyor system while an aspiration system collected and filtered the air from various points. The AGF were collected from the air on sieves. 139 kg of grain produced 212 g of AGF. Residue levels in the grain were 0.39 mg/kg parathion-methyl and 0.01 mg/kg paraoxon-methyl, which produced residues in the AGF of 0.33 mg/kg parathion-methyl and <0.01 mg/kg paraoxon-methyl.

Maize. Parathion-methyl was applied six times to maize at 5.6 kg ai/ha (5 times the label rate) in two trials in the USA in 1988 and 1989 and the crops harvested (about 160 kg grain) 12 days after the final treatment for wet and dry milling (LeRoy, 1990e). The milling process is shown in Figure 11 and the residues in Table 61. In the 1989 trial parathion-methyl appeared in all the dry milling fractions, but at higher levels in the starting grain. In wet milling, residues decreased in the flour and appeared in the oils at much the same level as in the grain.

Table 61. Residues in maize and its processed commodities from supervised trials and wet and dry milling in the USA (LeRoy, 1990e).

| Location, year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| TX, 1988 (Dekalb 689) | EC | 5.6 | 75 | 6 | 12 | whole grain | <0.05 (2) | <0.05 (2) | MP-CN-3512 |
| | | | | | | large grits | <0.05 | <0.05 | |
| | | | | | | medium grits | <0.05 | <0.05 | dry milling |
| | | | | | | small grits | <0.05 | <0.05 | |
| | | | | | | coarse meal | <0.05 | <0.05 | |
| | | | | | | meal | <0.05 | <0.05 | |
| | | | | | | flour | <0.05 | <0.05 | |
| | | | | | | crude oil | <0.05 | <0.05 | |
| | | | | | | refined oil | <0.05 | <0.05 | |
| TX, 1988 (Dekalb 689) | EC | 5.6 | 75 | 6 | 12 | whole grain | <0.05 (2) | <0.05 (2) | MP-CN-3512 |
| | | | | | | starch | <0.05 | <0.05 | |
| | | | | | | crude oil | <0.05 | <0.05 | wet milling |
| | | | | | | refined oil | <0.05 | <0.05 | |
| MO, 1989 (Funks G-4500) | EC | 5.6 | 190 | 6 | 12 | whole grain | 0.58 | <0.05 | MP-CN-3524 |
| | | | | | | large grits | 0.12 | <0.05 | |
| | | | | | | medium grits | 0.11 | <0.05 | dry milling |
| | | | | | | small grits | 0.43 | <0.05 | |
| | | | | | | coarse meal | 0.27 | <0.05 | |
| | | | | | | meal | 0.26 | <0.05 | |
| | | | | | | flour | 0.24 | <0.05 | |
| | | | | | | crude oil | 0.18 | <0.05 | |
| | | | | | | refined oil | 0.15 | <0.05 | |
| MO, 1989 (Funks G-4500) | EC | 5.6 | 190 | 6 | 12 | whole grain | 0.58 | <0.05 | MP-CN-3524 |
| | | | | | | starch | <0.05 | <0.05 | |
| | | | | | | crude oil | 0.77 | <0.05 | wet milling |
| | | | | | | refined oil | 0.60 | <0.05 | |

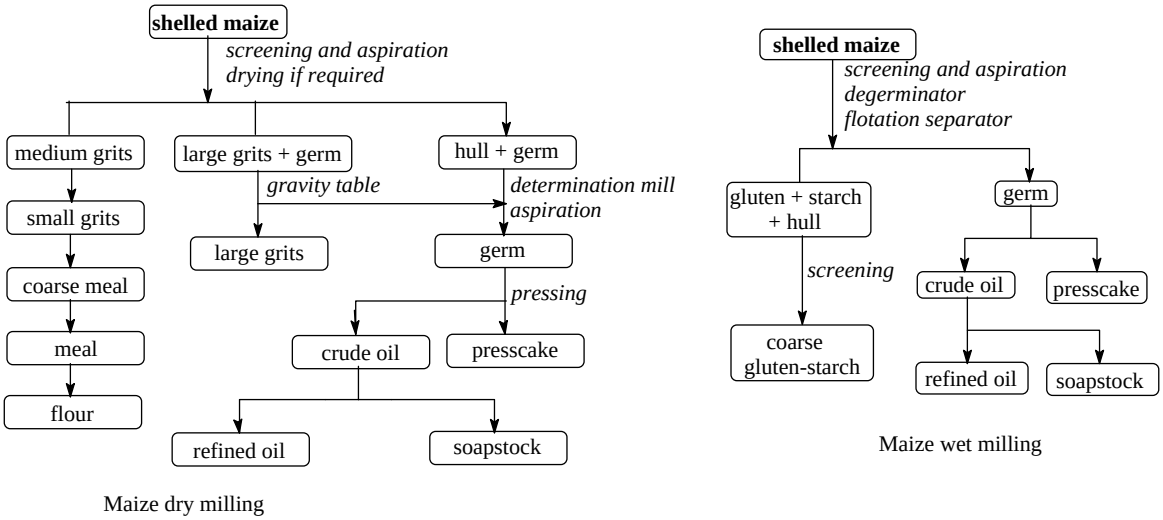



Figure 11. Dry and wet milling of maize (LeRoy, 1990e).

Rice. Parathion-methyl was applied six times to rice at 4.4 kg ai/ha (5 times the label rate) in two trials in the USA in 1988 and the crops harvested (about 9 and 45 kg grain) 15 and 16 days after the final treatment for milling (LeRoy, 1990f). Figure 12 shows the milling process and Table 62 summarizes the results. Residues at each stage of the process were higher in the outer layers of the hulls and bran, and decreased in the brown and still more in the polished rice.

Table 62. Residues in rice and its processed commodities from supervised trials and milling in the USA (LeRoy, 1990f).

| Location, year (variety) | Application | | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | kg ai/hl | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| CA, 1988 (M201) | EC | 4.4 | | 190 | 6 | 16 | grain<br>brown rice<br>hulls<br>bran<br>polished rice | 14<br>2.7<br>72<br>11<br>0.52 | 0.63<br><0.05<br>3.5<br>0.13<br><0.05 | MP-RI-3514 |
| TX, 1988 (Gulfmont) | EC | 4.4 | | 170 | 6 | 15 | grain<br>brown rice<br>hulls<br>bran<br>polished rice | 1.9<br>0.45<br>9.3<br>2.0<br>0.13 | 0.67<br><0.05<br>3.6<br>0.11<br><0.05 | MP-RI-3515 |

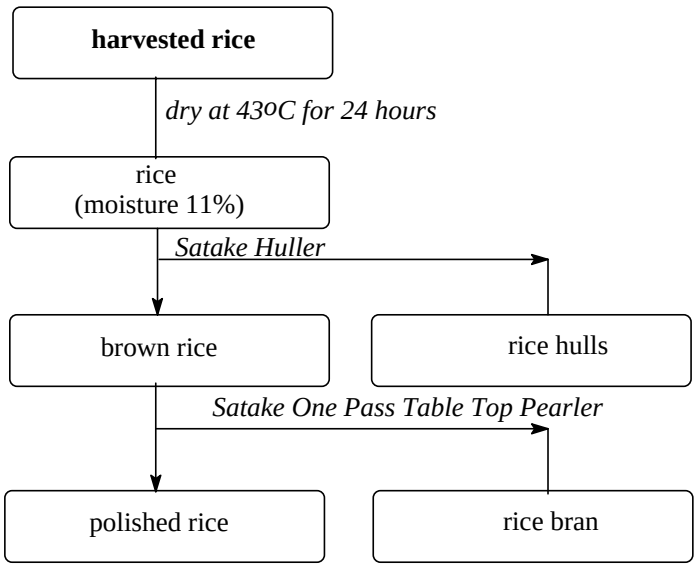



Figure 12. Processing of rice (LeRoy, 1990f).

Cotton seed. Parathion-methyl was applied six or 10 times to cotton at 6.7 and 17 kg ai/ha (exaggerated rates) in two trials in the USA in 1988 and 1989 and the crops harvested (about 11 and 7 kg grain) 7 days after the final treatment (LeRoy, 1990g). Figure 13 shows the milling process and Table 63 summarizes the results. Residues decreased in the meal in both trials, but were inconsistent in the oils.

Table 63. Residues in cotton seed and its processed commodities from supervised trials in the USA (LeRoy, 1990g).

| Location, year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| CA, 1989 (GC 510) | EC | 6.7 | 99 | 6 | 7 | cotton seed<br>meal<br>hulls<br>crude oil<br>refined oil<br>soapstock | 14<br>0.53<br>5.7<br>11<br>8.2<br>0.19 | 0.12<br><0.05<br><0.05<br><0.05<br>0.05<br>0.14 | MP-CS-3522 |
| TX, 1988 (DPL 41) | EC | 17 | 94 | 10 | 7 | cotton seed<br>meal<br>hulls<br>crude oil<br>refined oil<br>soapstock<br>cotton seed<br>hulls | 4.1<br>0.51<br>2.0<br>0.30<br>0.28<br>0.08<br>c 0.08 0.08<br>c0.07 | 0.21<br><0.05<br><0.05<br><0.05<br><0.05<br><0.05<br>c <0.05 (2)<br>c <0.05 | MP-CS-3523 |

c: sample from control plot

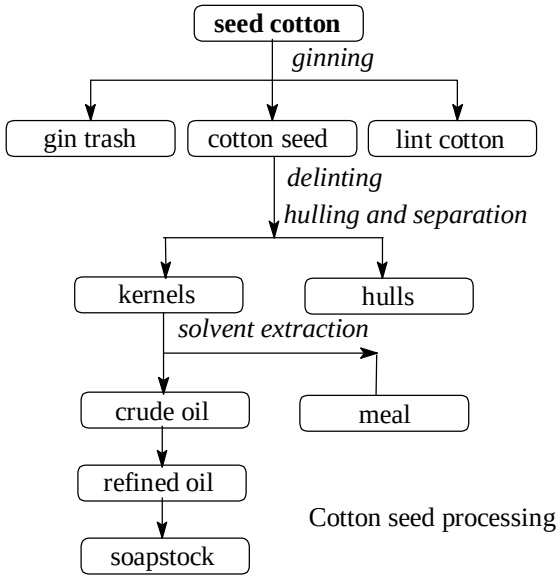



Figure 13. Processing of seed cotton (LeRoy, 1990g).

Sunflower seed. Parathion-methyl was applied three times by tractor mounted boom spray at about 6-day intervals to sunflowers at 5.6 kg ai/ha (5 times the label rate) in a trial in the USA in 1998 and the crop harvested (about 23 kg grain) 30 days after the final treatment (Williams and Rice 1999c). Figure 14 shows the process. Residues in the meal and refined oil were lower than in the seed (Table 64).

Table 64. Residues in sunflower seed and its processed commodities from a supervised trial in the USA (Williams and Rice, 1999c).

| Location, year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg (duplicate analyses) | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| TX, 1998 (NK-547) | EC | 5.6 | 120 | 3 | 30 | seed<br>meal<br>refined oil | 0.52 0.52<br>0.03 0.05<br>0.13 0.12 | <0.01 (2)<br><0.01 (2)<br><0.01 (2) | PM9805 |

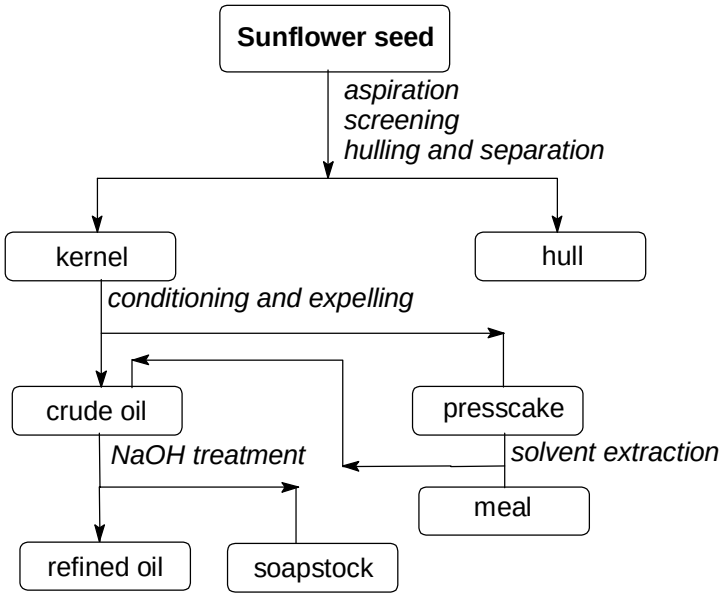



Figure 14. Sunflower seed processing (Williams and Rice, 1999c).

Oilseed rape (canola). Parathion-methyl was applied twice by tractor mounted boom spray to canola at the post-bloom stage at 5.6 kg ai/ha (5 times the label rate) in a trial in the USA in 1992 and the crop harvested (about 90 kg seed) 28 days after the final treatment (Belcher, 1993). The process was the same as for sunflower seed (Figure 14). Residues were higher in the oils and lower in the meal than in the seed (Table 65).

Table 65. Residues in canola seed and its processed fractions from a supervised trial in the USA (Belcher, 1993).

| Location, year (variety) | Application | | | | PHI, days | Sample | Residues, mg/kg (duplicate analyses) | | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | Form | kg ai/ha | water, l/ha | no. | | | parathion-methyl | paraoxon-methyl | |
| WA, 1992 (Series) | EC | 5.6 | 200 | 2 | 28 | seed<br>meal<br>crude oil<br>refined oil<br>processing waste | 0.39 0.41<br>0.085 0.095<br>0.95 1.0<br>0.79 0.81<br>0.39 0.42 | <0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2)<br><0.05 (2) | 92147 |

**Residues in the edible portion of food commodities**

Processing factors were calculated for all the processed commodities, both for parathion-methyl only and for combined parathion-methyl and paraoxon-methyl residues expressed as parathion-methyl. The calculated factors are shown in Table 66.

In the parathion-methyl-only calculation where the residue in the processed commodity was below the LOQ, the processing factor is reported as <(LOQ ÷ (residue in RAC)). In the total-residue calculation where the residue of paraoxon-methyl was below the LOQ it was assumed to be zero except when both parathion-methyl and paraoxon-methyl residues were below the LOQ, in which case the processing factor is reported as in the parathion-methyl-only calculation. For example:

| parathion-methyl | paraoxon-methyl | total residue | Processing factor |
|---|---|---|---|
| 4.91 | 0.12 | 5.04 | 5.04 ÷ R |
| 0.26 | <0.05 | 0.26 | 0.26 ÷ R |
| <0.05 | <0.05 | <0.05 | <(0.05 ÷ R) |

where R is the total residue in the RAC

Because parathion-methyl is usually the major component of the residue the processing factors for parathion-methyl only and for the total residue are usually much the same.

Table 66. Processing factors from the reported processing trials calculated for parathion-methyl residues and for combined residues of parathion-methyl and paraoxon-methyl expressed as parathion-methyl. Processing factors were calculated directly from unrounded results.

| COMMODITY | Residues, mg/kg | | | | Processing factors | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | | parathion-methyl + 1.065 × paraoxon-methyl | | |
| | Trial A | Trial B | Trial A | Trial B | A | B | mean | A | B | mean |
| APPLES, fruit | 0.04 | 0.02 | <0.01 | <0.01 | | | | | | |
| dry pomace | 0.17 | 0.09 | <0.01 | <0.01 | 4.3 | 4.5 | 4.4 | | | |
| juice | <0.01 | <0.01 | <0.01 | <0.01 | <0.25 | <0.5 | | | | |
| APPLES, fruit | 0.03 | 0.01 | <0.01 | <0.01 | | | | | | |
| dry pomace | 0.12 | 0.08 | <0.01 | <0.01 | 4.0 | 8.0 | 6.0 | | | |
| juice | <0.01 | <0.01 | <0.01 | <0.01 | <0.3 | <1 | | | | |
| PEACHES, fruit | 0.03 | 0.02 | | | | | | | | |
| juice | <0.01 | <0.01 | | | <0.33 | <0.5 | | | | |
| GRAPES, fruit | 0.12 | 0.13 | | | | | | | | |
| must | <0.01 | <0.01 | | | <0.083 | <0.077 | | | | |
| wine | <0.01 | <0.01 | | | <0.083 | <0.077 | | | | |
| GRAPES, day 14 | 0.16 | 0.15 | | | | | | | | |
| raisins, day 14 | 0.21 | 0.22 | | | 1.3 | 1.5 | 1.4 | | | |
| wine, day 14 | 0.03 | 0.02 | | | 0.19 | 0.13 | 0.16 | | | |
| juice, day 14 | 0.01 | <0.01 | | | 0.06 | <0.07 | 0.06 | | | |
| GRAPES, day 21 | 0.09 | 0.10 | | | | | | | | |
| raisins, day 21 | 0.14 | 0.13 | | | 1.6 | 1.3 | 1.4 | | | |
| wine, day 21 | 0.02 | 0.02 | | | 0.22 | 0.20 | 0.21 | | | |
| juice, day 21 | <0.01 | <0.01 | | | <0.11 | <0.10 | | | | |
| OLIVES, day 0 | 4.7 | 6.2 | | | | | | | | |
| olive oil, day 0 | 30 | 59 | | | 6.4 | 9.5 | 7.9 | | | |
| OLIVES, day 7 | 0.55 | 3.4 | | | | | | | | |
| olive oil, day 7 | 2.6 | 18 | | | 4.7 | 5.3 | 5.0 | | | |
| OLIVES, day 14 | 0.44 | 1.5 | | | | | | | | |
| olive oil, day 14 | 1.1 | 13 | | | 2.4 | 8.6 | 5.5 | | | |
| OLIVES, day 21 | 0.24 | 1.2 | | | | | | | | |
| olive oil, day 21 | 1.6 | 5.4 | | | 6.7 | 4.7 | 5.7 | | | |
| OLIVES, day 28 | 0.19 | 1.0 | | | | | | | | |
| olive oil, day 28 | 0.35 | 11.1 | | | 1.8 | 11.3 | 6.6 | | | |
| SOYA BEAN, dry seed | 0.15 | | | | | | | | | |
| meal | <0.05 | | | | <0.33 | | | | | |
| hulls | 0.12 | | | | 0.80 | | 0.80 | | | |
| crude oil | 0.71 | | | | 4.73 | | 4.7 | | | |
| refined oil | 0.57 | | | | 3.80 | | 3.8 | | | |
| SOYA BEAN, seed | 0.17 | | | | | | | | | |
| meal | <0.1 | | | | <0.6 | | | | | |
| hulls | 0.19 | | | | 1.1 | | 1.1 | | | |
| crude oil | 0.51 | | | | 3.0 | | 3.0 | | | |
| refined oil | 0.49 | | | | 2.9 | | 2.9 | | | |
| soapstock | 0.06 | | | | 0.35 | | 0.35 | | | |

| COMMODITY | Residues, mg/kg | | | | Processing factors | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | | parathion-methyl + 1.065 × paraoxon-methyl | | |
| | Trial A | Trial B | Trial A | Trial B | A | B | mean | A | B | mean |
| WHEAT, grain | 9.8 | 6.2 | 0.15 | 0.13 | | | | | | |
| bran | 19 | 15 | 0.26 | 0.19 | 2.0 | 2.4 | 2.2 | 1.9 | 2.4 | 2.2 |
| flour, patent | 2.8 | | 0.09 | | 0.28 | | 0.28 | 0.29 | | 0.29 |
| middlings | 4.9 | 4.4 | 0.12 | 0.08 | 0.50 | 0.71 | 0.61 | 0.51 | 0.70 | 0.61 |
| red dog | 4.2 | 5.5 | 0.14 | 0.10 | 0.43 | 0.87 | 0.65 | 0.44 | 0.87 | 0.65 |
| shorts and germ | 9.0 | 20 | 0.24 | 0.29 | 0.92 | 3.2 | 2.1 | 0.93 | 3.2 | 2.1 |
| rough | | 11 | | 0.28 | | 1.8 | 1.8 | | 1.8 | 1.8 |
| flour | | 2.6 | | 0.05 | | 0.42 | 0.42 | | 0.42 | 0.42 |
| flour, low grade | | 1.5 | | 0.09 | | 0.24 | 0.24 | | 0.25 | 0.25 |
| WHEAT, grain | 0.39 | | 0.01 | | | | | | | |
| aspirated grain fractions | 0.34 | | <0.01 | | 0.87 | | 0.87 | 0.85 | | 0.85 |
| MAIZE, whole grain | 0.58 | | <0.05 | | | | | | | |
| large grits | 0.12 | | <0.05 | | 0.21 | | 0.21 | | | |
| medium grits | 0.11 | | <0.05 | | 0.19 | | 0.19 | | | |
| small grits | 0.43 | | <0.05 | | 0.74 | | 0.74 | | | |
| coarse meal | 0.27 | | <0.05 | | 0.47 | | 0.47 | | | |
| meal | 0.26 | | <0.05 | | 0.45 | | 0.45 | | | |
| flour | 0.24 | | <0.05 | | 0.41 | | 0.41 | | | |
| crude oil | 0.18 | | <0.05 | | 0.31 | | 0.31 | | | |
| refined oil | 0.15 | | <0.05 | | 0.26 | | 0.26 | | | |
| MAIZE, whole grain | 0.58 | | <0.05 | | | | | | | |
| starch | <0.05 | | <0.05 | | <0.09 | | | | | |
| crude oil | 0.77 | | <0.05 | | 1.33 | | 1.33 | | | |
| refined oil | 0.6 | | <0.05 | | 1.03 | | 1.03 | | | |
| RICE, grain | 14 | 1.9 | 0.63 | 0.67 | | | | | | |
| brown rice | 2.7 | 0.45 | <0.05 | <0.05 | 0.19 | 0.24 | 0.21 | 0.18 | 0.17 | 0.18 |
| hulls | 72 | 9.3 | 3.5 | 3.6 | 5.1 | 5.0 | 5.0 | 5.1 | 5.1 | 5.1 |
| bran | 11 | 2.0 | 0.13 | 0.11 | 0.75 | 1.1 | 0.91 | 0.73 | 0.82 | 0.77 |
| polished rice | 0.52 | 0.13 | <0.05 | <0.05 | 0.037 | 0.070 | 0.053 | 0.035 | 0.051 | 0.043 |
| COTTON, seed | 14 | 4.1 | 0.12 | 0.21 | | | | | | |
| meal | 0.53 | 0.51 | <0.05 | <0.05 | 0.038 | 0.124 | 0.08 | 0.04 | 0.12 | 0.08 |
| hulls | 5.7 | 2.04 | <0.05 | <0.05 | 0.41 | 0.50 | 0.46 | 0.41 | 0.47 | 0.44 |
| crude oil | 11 | 0.3 | <0.05 | <0.05 | 0.81 | 0.073 | 0.44 | 0.81 | 0.07 | 0.44 |
| refined oil | 8.2 | 0.28 | 0.05 | <0.05 | 0.59 | 0.068 | 0.33 | 0.59 | 0.06 | 0.33 |
| soapstock | 0.19 | 0.08 | 0.14 | <0.05 | 0.014 | 0.020 | 0.017 | 0.02 | 0.02 | 0.021 |
| SUNFLOWER, seed | 0.52 | | <0.01 | | | | | | | |
| meal | 0.04 | | <0.01 | | 0.08 | | 0.08 | | | |
| refined oil | 0.13 | | <0.01 | | 0.24 | | 0.24 | | | |
| CANOLA, seed | 0.40 | | <0.05 | | | | | | | |
| meal | 0.09 | | <0.05 | | 0.22 | | 0.22 | | | |
| crude oil | 0.98 | | <0.05 | | 2.4 | | 2.4 | | | |
| refined oil | 0.80 | | <0.05 | | 2.0 | | 2.0 | | | |
| processing waste | 0.40 | | <0.05 | | 1.00 | | 1.00 | | | |
| HOPS, fresh | 0.46 | 0.15 | 0.067 | 0.042 | | | | | | |
| hops, dried | 0.99 | 0.36 | 0.35 | 0.13 | 2.2 | 2.4 | | 2.6 | 2.6 | |
| HOPS, fresh | 0.96 | 0.35 | 0.21 | 0.11 | | | | | | |
| hops, dried | 2.7 | 1.19 | 0.90 | 0.41 | 2.8 | 3.4 | | 3.1 | 3.5 | |
| HOPS, fresh | 0.096 | 0.12 | <0.04 | 0.036 | | | | | | |
| hops, dried | 0.40 | 0.43 | 0.17 | 0.16 | 4.2 | 3.6 | | 6.1 | 3.8 | |
| HOPS, fresh | 0.39 | 0.28 | 0.11 | 0.12 | | | | | | |
| hops, dried | 0.87 | 0.78 | 0.32 | 0.29 | 2.2 | 2.8 | | 2.4 | 2.7 | |
| HOPS, fresh | 0.18 | 0.18 | 0.15 | 0.1 | | | | | | |
| hops, dried | 0.41 | 0.23 | 0.19 | 0.18 | 2.3 | 1.3 | | 1.8 | 1.5 | |
| HOPS, fresh | 0.25 | 0.17 | 0.09 | 0.058 | | | | | | |
| hops, dried | 0.37 | 0.27 | 0.25 | 0.23 | 1.5 | 1.6 | | 1.8 | 2.2 | |

| COMMODITY | Residues, mg/kg | | | | Processing factors | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | parathion-methyl | | paraoxon-methyl | | parathion-methyl | | | parathion-methyl + 1.065 × paraoxon-methyl | | |
| | Trial A | Trial B | Trial A | Trial B | A | B | mean | A | B | mean |
| | | | | | hops mean | | 2.5 | | | 2.8 |

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Information was provided by the governments of Australia and The Netherlands.

Monitoring data from The Netherlands are shown in Table 67. Only 2 samples of lemons, 3 of oranges, 1 of tangerines, 4 of grapes, 1 of blue bilberries and 1 of broccoli contained residues above the MRLs.

Table 67. Residues found in monitoring in The Netherlands, 1994-1998. The LOQ was 0.02 mg/kg.

| Commodity | No. of samples | | Commodity | No. of samples | |
|---|---|---|---|---|---|
| | analysed | residues detected | | analysed | residues detected |
| apples | 1986 | 6 | lemon | 267 | 8 |
| apricots | 80 | 0 | lettuce | 4134 | 4 |
| bananas | 57 | 2 | mangoes | 191 | 1 |
| beans with pods | 617 | 2 | melons | 390 | 4 |
| blue bilberries | 104 | 2 | nectarines | 221 | 3 |
| broccoli | 154 | 0 | onion | 97 | 1 |
| celery | 233 | 2 | oranges | 1361 | 46 |
| Chinese cabbage | 297 | 2 | parsley | 368 | 3 |
| currants | 55 | 1 | peaches | 277 | 11 |
| endive | 1503 | 3 | peppers | 2132 | 2 |
| globe artichokes | 24 | 1 | radishes | 1010 | 1 |
| grapefruit | 388 | 15 | raspberries | 68 | 1 |
| grapes | 863 | 40 | strawberries | 3595 | 8 |
| iceberg lettuce | 471 | 3 | tangerines | 742 | 25 |
| kiwifruit | 223 | 3 | | | |

Parathion-methyl was monitored in the 1994 Australian Market Basket Survey in which 77 foods were analysed for pesticide residues (Marro, 1996). The estimated daily dietary intakes (expressed as µg/kg bw) for mean energy diets of parathion-methyl residues in food were adult males 0.0114, adult females 0.0169, boys aged 12 0.0123, girls aged 12 0.0105, toddlers 0.0279 and infants 0.0373.

Parathion-methyl was included in the targeted enforcement monitoring programmes in Australian States for 1992-1999. In Queensland no parathion-methyl residues (LOQ 0.02-0.1 mg/kg) were detected in 342 samples of fruits, vegetables and cereals in 1996-99. In Victoria parathion-methyl was detected (LOQ 0.01 mg/kg) in apples (6 of 23 samples) and pears (11 of 35 samples) in 1995-97, with the highest residue at 0.23 mg/kg. In New South Wales parathion-methyl was detected (LOQ 0.05 mg/kg) in 14 of 2621 samples of fruits and vegetables analysed from 1992 to 1997. No residue exceeded an MRL.

## NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was aware that the following national MRLs had been established.

| Country | MRL, mg/kg | Commodity |
|---|---|---|
| Australia | 1 | cotton seed, fruits, vegetables |
| | 0.05 | cotton seed oil crude |
| | 0.05* | edible offal mammalian, meat mammalian, milks |
| Netherlands | 0.2 | fruit, vegetables, tea |
| | 0.05 | other vegetable products |
| | 0.02* | other food commodities |
| Poland | 0.1 | cereal grains, fruits, vegetables |
| USA | 5 | alfalfa hay, birdsfoot trefoil hay |
| | 3 | almond hulls, sorghum fodder, sorghum forage |
| | 1.25 | alfalfa (fresh), birdsfoot trefoil forage |
| | 1 | apples. apricots, artichokes, avocados, barley, beans, beet greens, beets, blackberries, blueberries, boysenberries, Brassica vegetables, broccoli, Brussels sprouts, cabbage, carrots, cauliflower, celery, cherries, clover, collards, corn, corn forage, cranberries, cucumbers, currants, dates, dewberries, eggplants, endive, figs, garlic, gooseberries, grapes, grass forage, guavas, hops, kale, kohlrabi, leafy vegetables, lentils, lettuce, loganberries, mangoes, melons, mustard greens, nectarines, oats, okra, olives, onions, parsley, parsnips, parsnip greens, peaches, peanuts, pears, peas, pea forage, peppers, pineapples, plums, pumpkins, quinces, radishes, radish tops, raspberries, rice, rutabagas, rutabaga tops, soya bean hay, spinach, squash, strawberries, summer squash, Swiss chard, tomatoes, turnips, turnip greens, vetch, wheat, youngberries |
| | 0.75 | cottonseed |
| | 0.2 | mustard seed, rape seed, sunflower seed, guar beans |
| | 0.1 | almonds, sugar beets, sugar beet tops, filberts, pecans, potatoes, safflower seed, sorghum, soya beans, sweet potatoes, walnuts |

* indicates lower limit of determination (LOD) or MRL set at or about LOD.
[1] Australia residue definition: parathion-methyl.
[2] Netherlands residue definition: parent compound, expressed as parathion-methyl.

## APPRAISAL

Parathion-methyl was first evaluated by the Joint Meeting in 1965 and has been reviewed several times since. At its thirtieth session in 1998, the CCPR (ALINORM 99/24, Appendix VII) listed parathion-methyl for periodic review for residues by the 2000 JMPR. The Meeting received information on physical and chemical properties, metabolism and environmental fate, analytical methods, stability in frozen storage, registered uses, the results of supervised trials on fruits, vegetables, and field crops, and studies on processing.

### *Metabolism*

*Animals and birds*

When a lactating goat weighing 60 kg was given [*phenyl*-$^{14}$C]parathion-methyl orally at a dose of 35 mg per day (equivalent to 6.25 ppm in the diet) daily for 3 days, no residues of parathion-methyl or paraoxon-methyl were detected in tissues or milk. However, as only 35.5% of the administered radiolabel was recovered, a large proportion was unaccounted for.

When laying hens were given [*phenyl*-$^{14}$C]parathion-methyl orally three times at daily intervals at a dose of 0.5 mg/kg bw (equivalent to 6.25 ppm in the diet), the compound was detected in tissues but not in eggs, while paraoxon-methyl was detected in neither tissues nor eggs. The highest concentrations of parathion-methyl were found in skin and fat, suggesting a certain degree of solubility in fat. Only 54% of the radiolabel was accounted for.

The metabolites identified indicate that parathion-methyl is degraded by demethylation, oxidation, hydrolysis of the phosphate ester, reduction of the nitro group to an amine, and conjugation.

*Plants*

The Meeting received information on the fate of parathion-methyl in potatoes, cotton, and lettuce. Only 0.01-0.02% of the radiolabel in *potato* plants was found in the tubers 5 days after application of [*phenyl*-$^{14}$C]parathion-methyl as a foliar spray to the plants at a rate equivalent to 4.7 kg ai/ha. Parathion-methyl was identified in the tubers at a concentration of 0.001 mg/kg. In plants harvested 21 days after treatment, the tubers contained 0.13-0.14% of the radiolabel, and paraoxon-methyl was tentatively identified at a concentration of 0.002 mg/kg. Very low concentrations of nitrophenyl conjugates were also identified.

Parathion-methyl was found at a concentration of 0.008 mg/kg in *cotton seed* 10 days after foliar application of [*phenyl*-$^{14}$C]parathion-methyl to cotton plants at the equivalent of 0.38 kg ai/ha. Nitrophenol, *para*-nitrophenyl glucopyranoside, and *O*-demethyl-parathion-methyl were also components of the residue.

*Lettuce* plants harvested 14 and 21 days after foliar treatment with [*phenyl*-$^{14}$C]parathion-methyl at a rate equivalent to 1.23 kg ai/ha contained parathion-methyl residues at a concentration of 2.2 and 0.097 mg/kg, respectively. Neither paraoxon-methyl nor any other metabolite containing P=O was detected. The metabolites *para*-nitrophenol, *para*-nitrophenyl glucopyranoside, and *O*-demethyl-parathion-methyl were identified. In a further experiment on lettuce, the major metabolite was 4-nitrophenyl 6-*O*-malonyl-β-D-glucopyranoside.

The plant metabolites identified indicate that hydrolysis of parathion-methyl to nitrophenol is the major metabolic pathway, but *O*-demethylation and oxidation to paraoxon-methyl may also occur to a limited extent. *para*-Nitrophenol is readily conjugated.

The Meeting noted that no information was available on the fate of parathion-methyl in fruit crops but was informed that studies were planned to support the re-registration programme in the European Union.

***Environmental fate***

Parathion-methyl residues disappeared quickly (initial half-life, 3.9 days) during incubation in aerobic soil. The metabolites identified were *para*-nitrophenol and *O,O*-bis(4-nitrophenyl) *O*-methyl phosphorothioate. Under anaerobic conditions, parathion-methyl disappeared rapidly, with an initial half-life of 10.5 h. Nitrophenol was a major component of the residue during the first week, but the concentration declined rapidly thereafter. Parathion-methyl had medium to low mobility on four soils in a laboratory study and did not appear below the top 10 cm in two field studies of soil dissipation. No residues of paraoxon-methyl were detected in any sample in the field studies.

Direct photodegradation of parathion-methyl residues in water is likely to make a minor contribution to its overall disappearance from the environment. In aquatic field studies (rice paddies), parathion-methyl disappeared quickly, and was detectable in the water only on the day of application or the next day.

***Methods of analysis***

The analytical methods for parathion-methyl and paraoxon-methyl are based on gas-liquid chromatography with flame photometry after solvent extraction and simple clean-up by solvent

partition and reversed-phase column chromatography. Some variations are required for different substrates, particularly in the extraction step. A LOQ of 0.01 mg/kg is achieved for many substrates, but the validated LOQ for difficult substrates is 0.05 mg/kg.

The method used in many of the supervised trials in the USA included an initial 1-h reflux of the sample in acidic aqueous methanol before clean-up. The typical LOQ in trials conducted in the 1980s and early 1990s was 0.05 mg/kg, but 0.01 mg/kg was achieved in later trials.

***Stability of residues in stored analytical samples***

Residues of parathion-methyl in bluegrass hay, rape seed, celery, clover forage, dry bean seeds, dry pea seeds, dry pea straw, head cabbages, lettuce, maize fodder, maize forage, maize grain, mustard, onions, snap bean seeds and pods, soya bean seeds, succulent pea forage, succulent pea pods, sunflower seeds, turnip roots, turnip tops, wheat forage, wheat grain, and wheat straw, in processed crude rape seed oil and rape seed meal, and in soils were stable during frozen storage for the durations tested (mostly 2 years). Residues of paraoxon-methyl were also stable in frozen storage, with a few exceptions. The calculated times for a 30% decrease in the concentration of paraoxon-methyl residues were 5 months in rape seed, 12 months in crude rape seed oil, 7 months in rape seed meal, and 13 months in lettuce.

No information was available on the stability of parathion-methyl or paraoxon-methyl residues in frozen storage of fruits. Because parathion-methyl has been tested in many commodities with consistent results, it is probably stable in fruit matrices; however, the stability of paraoxon-methyl residues in fruits is not established. The Meeting was informed that studies of the stability in fruits in frozen storage are being planned to support the re-registration programme of the European Union. The concentrations of paraoxon-methyl decreased by 30% within about 250 days in a frozen sample of sandy loam and 22 days in one of loam soil.

***Definition of the residue***

Parathion-methyl and paraoxon-methyl are the most important components for the residue definition. The contributions of residues of the two components to the total residue in food and feed commodities in GAP trials at the recommended PHIs were examined. In 54 trials of food commodities and 155 of feed commodities, the concentrations of both components exceeded the LOQ. The total concentration of residues was closely related to that of parathion-methyl in both food and feed commodities, but the relationship was less close at lower concentrations.

The Meeting recommended that the residue definition for compliance with MRLs continue to be parathion-methyl and that for estimation of dietary intake should be the sum of parathion-methyl and paraoxon-methyl expressed as parathion-methyl (parathion-methyl + 1.065 ∞ paraoxon-methyl). The residue definition applies to plant commodities and it should be reconsidered when MRLs for animal commodities are recommended.

***Results of supervised trials***

Extensive data were provided from supervised trials on many crops: apple, pear, peach, grape, onion, broccoli, cabbage, sweet corn, mustard green, lettuce, spinach, lima bean, snap bean, soya bean, field pea, dried bean, carrot, potato, sugar beet, turnip, celery, artichoke, maize, rice, sorghum, wheat, cotton seed, rape seed, sunflower seed, alfalfa, clover, pasture grass, and hops. No relevant GAP was available to evaluate the data for pear, onion, broccoli, sweet corn, mustard green, lettuce, spinach, lima bean, snap bean, soya bean, carrot, turnip, celery, artichoke, sorghum, sunflower seed, clover, or hops.

The Meeting agreed to withdraw the recommended MRLs for globe artichoke (2 mg/kg), broccoli (0.2 mg/kg), carrot (1 mg/kg), celery (5 mg/kg), cherry (0.01* mg/kg), clover (10 mg/kg), common bean (0.05* mg/kg), garden pea (1 mg/kg) gooseberry (0.01* mg/kg), dry hops (1 mg/kg), lettuce head (0.05* mg/kg), lettuce leaf (0.5 mg/kg), lima bean (0.05* mg/kg), mustard greens (0.5 mg/kg), plums including prunes (0.01* mg/kg), red and black raspberries (0.01* mg/kg), spinach (0.5 mg/kg), turnip greens (2 mg/kg), and garden turnip (0.05* mg/kg). These MRLs were not supported by current GAP or by the results of supervised trials that matched current GAP.

The residue definition for dietary intake requires the addition of parathion-methyl and paraoxon-methyl residues expressed as parathion-methyl. In this calculation, concentrations of residues of paraoxon-methyl that were <LOQ were assumed to be 0, except when the concentrations of both parathion-methyl and paraoxon-methyl residues were <LOQ. For example:

| Parathion-methyl | Paraoxon-methyl | Total residue (parathion-methyl + 1.058 x paraoxon- methyl) |
|---|---|---|
| 3.20 | 0.34 | 3.56 |
| 0.42 | <0.05 | 0.42 |
| <0.05 | <0.05 | <0.05 |

The results of supervised trials on *apple* in Germany were not evaluated because there was no matching GAP. In France, parathion-methyl is registered for use on pome fruits at a spray concentration of 0.03 kg ai/hl with a PHI of 15 days. In 26 trials conducted in France in 1994 and 1995 at 0.036 kg ai/hl and a 14-day PHI, the concentrations of parathion-methyl residues, in rank order, were <0.01 (3 trials), 0.01 (3 trials), 0.02 (4 trials), 0.04 (2 trials), and 0.11 mg/kg after use of emulsifiable concentrate formulations, and 0.03 (2 trials), 0.04 (3 trials), 0.05, 0.06, 0.07, 0.10, 0.11, 0.12, 0.15, and 0.18 mg/kg after use of capsule suspension formulations. The residues of the two formulations appeared to be from different populations, higher concentrations generally arising from the capsule suspension formulation, and the Meeting agreed to use the data for the latter formulations for estimating the STMR value and MRL. The concentrations of the combined residues of parathion-methyl and paraoxon-methyl, in rank order (median in italics), in the 13 trials with capsule suspension formulations were 0.03 (2 trials), 0.04 (3 trials), 0.05, ***0.06***, 0.07, 0.10, 0.11, 0.14, 0.15, and 0.18 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR value of 0.06 mg/kg, and a HR value of 0.18 mg/kg for parathion-methyl in apples.

Parathion-methyl is registered in Italy for use on stone fruit at a spray concentration of 0.023-0.045 kg ai/hl and a PHI of 20 days. The concentrations of residues in *peach* in 18 trials in Italy in 1994 and 1995 that matched GAP were <0.01 (3 trials), 0.01 (4 trials), 0.02 (2 trials), and 0.04 mg/kg after use of emulsifiable concentrate formulations and 0.06 (2 trials), 0.08, ***0.09***, ***0.10***, 0.13, 0.16, and 0.22 mg/kg after use of capsule suspension formulations. The concentration of paraoxon-methyl residues did not exceed the LOQ (0.01 mg/kg). The residues of the two formulations appeared to be from different populations, higher concentrations generally arising from the capsule suspension formulation, and the Meeting agreed to use the data for the latter formulations for estimating the STMR value and MRL.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR value of 0.095 mg/kg, and a HR value of 0.22 mg/kg for parathion-methyl in peaches expressed for the whole fruit.

Parathion-methyl is registered in France for use on *grape* at an application rate of 0.3 kg ai/ha and a PHI of 21 days. In 18 trials conducted in France in accordance with the GAP, the concentrations of residues were <0.01 (8 trials) and 0.02 mg/kg from the use of emulsifiable concentrate formulations and 0.05 (2 trials), 0.09 (2 trials), ***0.10*** (3 trials), 0.13, and 0.41 mg/kg after use of capsule suspension formulations. In one trial, the concentration found at day 28 was used because it was higher than that

at day 21. In two further trials, the concentrations at day 21 were not available because of a sample mix-up, and the residues for day 28 (0.41 and 0.02 mg/kg) were used.

In Spain, parathion-methyl is registered for use on grapes at a spray concentration of 0.045-0.059 kg ai/hl and a PHI of 21 days. Four trials in Spain conducted in accordance with the French GAP resulted in a concentration of <0.01 mg/kg in the two trials of use of emulsifiable concentrate formulations and 0.05 and 0.13 mg/kg after use of capsule suspension formulations. Eight valid trials in Italy at the Spanish GAP (spray concentrations of 0.046-0.068 kg ai/hl; PHI, 21 days) resulted in concentrations of parathion-methyl residues of <0.01 (5 trials) and 0.01 mg/kg after use of emulsifiable concentrate formulations and 0.12 and 0.18 mg/kg after use of capsule suspension formulations. Paraoxon-methyl was not measured in the Italian trials and was not detected at the GAP PHI in the other trials. The residues of the two formulations appeared to be from different populations, higher concentrations generally arising from the capsule suspension formulation, and the Meeting agreed to use the data for the latter formulations for estimating the STMR value and MRL. The concentrations of parathion-methyl residues in grapes, in rank order, in the 13 trials with capsule suspension formulations were 0.05 (3 trials), 0.09 (2 trials), ***0.10*** (3 trials), 0.12, 0.13 (2 trials), 0.18, and 0.41 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR value of 0.10 mg/kg, and a HR value of 0.41 mg/kg for parathion-methyl in grapes.

Parathion-methyl is registered in the USA for use on *cabbage* at 0.56-1.7 kg ai/ha, with a PHI of 10 days for 0.56 kg ai/ha and 21 days for higher rates. A series of trials conducted in 1988 and 1989 with either six or seven applications at 1.7 kg ai/ha and a 21-day PHI or with a final application at the lower rate and a PHI of 10 days resulted in the concentrations of parathion-methyl residues in cabbages, including wrapper leaves, of <0.05 (12 trials) and <0.5 (4 trials) mg/kg.

In four trials in California with high LOQs, there was analytical interference due to overspray with another pesticide, demeton-*S*-methyl. However, the results of these trials cannot be ignored because paraoxon-methyl residues of 0.08-0.24 mg/kg were recorded. Concentrations of paraoxon-methyl of 0.22 and 0.23 mg/kg were found in cabbages in trials in Florida, where those of parathion-methyl residues were <0.05 mg/kg. This finding differs from those in other crops where parathion-methyl almost invariably constitutes most of the residue. The concentrations of paraoxon-methyl residues in the 16 trials, in rank order, were ***0.05*** (9 trials), 0.07, 0.09, 0.10, 0.13, 0.23, 0.24, and 0.26 mg/kg.

The Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.26 mg/kg for parathion-methyl in cabbages. The estimated maximum residue level replaces the current recommendation (0.2 mg/kg) for head cabbages.

Parathion-methyl is registered in the USA for use on *pea* for production of dried peas at 0.56-1.1 kg ai/ha, with a PHI of 10 days for 0.56 kg ai/ha and 15 days for higher rates. A series of 12 trials conducted in 1988 and 1989 with either four or six applications at 1.1 kg ai/ha and 15-day PHI or with a final application at the lower rate and a PHI of 10 days resulted in the following concentrations of parathion-methyl residues in dried peas: <0.05 (4 trials), ***0.06*** (3 trials), 0.07, 0.16, 0.18, 0.19, and 0.24 mg/kg. The concentration of paraoxon-methyl residues did not exceed the LOQ (0.05 mg/kg) in any of the trials.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR value of 0.06 mg/kg, and a HR value of 0.24 mg/kg for parathion-methyl in dried peas. The estimated maximum residue level replaces the current recommendation (0.2 mg/kg) for dried peas.

Parathion-methyl is registered in the USA for use on *bean* for production of dried bean at 0.56-1.7 kg ai/ha with a PHI of 15 days for 0.56 kg ai/ha and 21 days for higher rates. In six trials in

four states in 1988, with six applications at 1.7 kg ai/ha and harvesting at 15 days, no parathion-methyl or paraoxon-methyl (<0.05 mg/kg) was detected. The number of trials was limited, but in view of the absence of detectable residues and use of a shorter PHI in the trials than required by GAP, the Meeting agreed to recommend an MRL. Although no residues were detected, there was no evidence that residues were not present, and the STMR value should be equivalent to the LOQ.

The Meeting estimated a maximum residue level of 0.05* mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.05 mg/kg for parathion-methyl in dried beans. The estimated maximum residue level confirms the current recommendation for dried beans.

Parathion-methyl is registered in the USA for use on *potato* at 1.7 kg ai/ha with a PHI of 6 days. In eight trials in five states in 1988 and 1989, with six applications at 1.7 or 1.8 kg ai/ha and harvesting after 5 days, no parathion-methyl or paraoxon-methyl (<0.05 mg/kg) was detected in the tubers. Two processing trials with exaggerated application rates (3.4 and 8.4 kg ai/ha) also showed no residues. In the study of metabolism in potatoes, with an application rate equivalent to 4.7 kg ai/ha, residues of parathion-methyl (0.001 mg/kg) were found in tubers 5 days after treatment and of paraoxon-methyl (0.002 mg/kg) 21 days after treatment. The Meeting agreed that the finding of very low concentrations of residues (50 times less than the LOQ) even after an exaggerated application rate would allow establishment of STMR and HR values as ‘essentially zero’.

The Meeting estimated a maximum residue level of 0.05* mg/kg, an STMR value of 0, and a HR value of 0 mg/kg for parathion-methyl in potatoes. The estimated maximum residue level confirms the current recommendation for potatoes.

Parathion-methyl is registered in the USA for use on *sugar beet* at 0.28-0.43 kg ai/ha with a PHI of 20 days. In eight trials in four states in 1988, with six applications at 0.42 kg ai/ha and harvesting at 20 days, no parathion-methyl or paraoxon-methyl was detected (<0.05 mg/kg) in the roots. Two processing trials with an exaggerated application rate (2.1 kg ai/ha) also showed no residues.

The Meeting estimated a maximum residue level of 0.05* mg/kg, an STMR value of 0, and a HR value of 0 mg/kg for parathion-methyl in sugar beet. The estimated maximum residue level confirms the current recommendation for sugar beets.

Parathion-methyl is registered in the USA for use on *maize* at 0.28-0.56 kg ai/ha with a PHI of 12 days. In 12 trials in nine states in 1988 and 1989, with six applications at 1.1 kg ai/ha and harvesting at 12 days, no paraoxon-methyl was detected (<0.05 mg/kg) in the grain in any of the 12 trials; no parathion-methyl was detected in 11 trials, but a concentration of 0.09 mg/kg was found in one trial. The Meeting agreed to use the data, even though the application rate was exaggerated (twice the labelled amount) since concentrations <LOQ were found in all but one.

The Meeting estimated a maximum residue level of 0.1 mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.09 mg/kg for parathion-methyl in maize.

Parathion-methyl is registered in the USA for use on *rice* at 0.56-0.84 kg ai/ha with a PHI of 15 days. In six trials in four states in 1988, with six applications at 0.89 kg ai/ha and harvesting at 15 or 16 days, the concentrations of residues of parathion-methyl in rice grain were 0.19, 0.27, 0.30, 0.44, 2.0, and 2.3 mg/kg, and those of combined parathion-methyl and paraoxon-methyl residues were 0.29, 0.43, ***0.45***, ***0.68***, 2.1, and 2.5 mg/kg.

The Meeting considered that six trials were too few to allow estimation of an MRL for rice, a major commodity, and withdrew the current recommendations of 3 mg/kg for rice, 1 mg/kg for husked rice, and 10 mg/kg for rice straw and fodder.

Parathion-methyl is registered in the USA for use on *wheat* at 0.28-0.84 kg ai/ha with a PHI of 15 days. In nine trials in seven states in 1988 and 1989, with four applications at 1.4 kg ai/ha followed by two applications at 0.84 kg ai/ha and harvesting at 14 days, the concentrations of residues of parathion-methyl in wheat grain were <0.05 (2 trials), 0.05, 0.21, 0.29, 0.48, 1.1 (2 trials), and 3.7 mg/kg, and those of combined parathion-methyl and paraoxon-methyl residues were <0.05 (2 trials), 0.05, 0.21, ***0.29***, 0.53, 1.2 (2 trials), and 4.1 mg/kg. As the residue is derived mainly from the final application, the first four of the six applications at an exaggerated rate would not have affected the concentration of residue.

The Meeting estimated a maximum residue level of 5 mg/kg, an STMR value of 0.29 mg/kg, and a HR value of 4.1 mg/kg for parathion-methyl in wheat.

Parathion-methyl is registered in the USA for use on *cotton* at 0.15-3.4 kg ai/ha, with a PHI of 5 days for hand-picking and 0 days for mechanical picking. In a trial in 1989, the concentrations of parathion-methyl residues were 9.5 and 22 mg/kg 7 days after application of 3.4 kg ai/ha in a processing trial. In 18 trials in four states in 1998 with 10 applications at 3.4 kg ai/ha and harvesting 1 day after the final application, the concentrations of parathion-methyl in cotton seed were 0.64, 1.5 (2 trials), 1.7, 1.9, 2.0, 2.5, 3.0, 3.2, 3.5, 3.9, 4.6, 5.4, 5.6, 6.8, 7.4 (2 trials), and 8.9 mg/kg, and those of the combined parathion-methyl and paraoxon-methyl residues were 0.66, 1.5 (2 trials), 1.7, 1.9, 2.0, 2.5, 3.0, ***3.2***, ***3.5***, 3.9, 4.6, 5.4, 5.6, 6.8, 7.4, 7.5, and 9.1 mg/kg. The concentrations found in the 19 trials were 0.66, 1.5 (2 trials), 1.7, 1.9, 2.0, 2.5, 3.0, 3.2, *3.5*, 3.9, 4.6, 5.4, 5.6, 6.8, 7.4, 7.5, 9.1, and 22 mg/kg

The Meeting estimated a maximum residue level of 25 mg/kg, an STMR value of 3.5 mg/kg, and a HR value of 22 mg/kg for parathion-methyl in cotton seed.

Parathion-methyl is registered in the USA for use on *rape seed* at 0.56 kg ai/ha with a PHI of 28 days. In four trials on rape seed in four states in 1992 with two applications at 0.56 kg ai/ha or two applications at 0.28 kg ai/ha followed by two at 0.56 kg ai/ha and harvesting 28 days after the final application, no parathion-methyl or paraoxon-methyl was detected (<0.05 mg/kg) in rape seed. In a further four trials at twice the label rate and harvesting at the 28-day PHI, no paraoxon-methyl residues were detected in any trial; parathion-methyl was not detected in three trials, and a concentration of 0.06 mg/kg was found in the fourth. Although the four trials were conducted at twice the GAP rate, the Meeting considered that the data provided valid support as all the concentrations but one were <LOQ. The estimates of the MRL and the STMR and HR values were based on the results of the GAP trials with support from the trials at twice the label rate.

The Meeting estimated a maximum residue level of 0.05 mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.05 mg/kg for parathion-methyl in rape seed.

Parathion-methyl is registered in the USA for use on *alfalfa* at 0.28-1.1 kg ai/ha with a PHI of 15 days. In 18 trials in nine states in 1998 with two applications of 1.1 kg ai/ha per cutting (two to four cuttings in each trial, each cutting being regarded as a separate trial) and cutting 14 or 15 days after the second application, the concentrations of parathion-methyl residues in *alfalfa forage* were 0.03, 0.09, 0.13, 0.21, 0.24, 0.26, 0.27, 0.31, 0.32 (2 trials), 0.35, 0.38, 0.39, 0.46, 0.54, 0.55, 0.57, 0.66, 0.70, 0.73, 0.74, 0.76, 0.82, 0.84, 0.87, 0.91 (2 trials), 0.92, 1.0, 1.1 (3 trials), 1.2, 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, 2.0, 2.1, 2.2, 2.3, 2.6, 2.7, 5.9, 6.8, 8.5, 8.6, and 11 (2 trials) mg/kg of fresh weight or 0.18, 0.48, 0.51, 0.55, 0.83, 0.93, 1.0 (2 trials), 1.1, 1.2, 1.4, 1.6, 1.7, 1.8, 2.3, 2.4, 2.5 (2 trials), 2.7, 2.8, 2.9, 3.1, 3.3, 3.5, 3.7 (2 trials), 3.8 (2 trials), 4.0, 4.2, 4.5, 5.1 (2 trials), 5.3, 5.4, 5.8, 6.3, 7.0 (2 trials), 8.1, 9.4, 9.8, 11 (2 trials), 15, 32, 33, 36, 41, 47, and 60 mg/kg of dry weight. As moisture was measured in each accompanying control sample, the concentration of residues in each sample could be calculated on a dry weight basis.

The concentrations of combined parathion-methyl and paraoxon-methyl residues in alfalfa forage were 0.03, 0.09, 0.13, 0.21, 0.24, 0.26, 0.27, 0.31, 0.32 (2 trials), 0.35, 0.38, 0.39, 0.46, 0.54, 0.56, 0.59, 0.68, 0.71, 0.74, 0.75, 0.76, 0.83, 0.84, 0.88, 0.92, 0.94 (2 trials), 1.0, 1.1 (3 trials), 1.2, 1.3, 1.4, 1.5, 1.6, 1.7, 1.8, 2.0, 2.1, 2.2, 2.3, 2.6, 2.7, 6.0, 6.9, 8.6, 8.7, and 11 (2 trials) mg/kg of fresh weight or 0.18, 0.48, 0.51, 0.55, 0.83, 0.93, 1.0 (2 trials), 1.1, 1.2, 1.4, 1.6, 1.7, 1.8, 2.3, 2.5, 2.6 (2 trials), 2.7, 2.9 (2 trials), 3.2, 3.3, 3.5, ***3.7*** (2 trials), 3.9 (2 trials), 4.1, 4.3, 4.6, 5.1, 5.2, 5.4, 5.5, 5.8, 6.3, 7.1 (2 trials), 8.3, 9.6, 10, 11 (2 trials), 15, 33 (2 trials), 37, 42, 47, and 61 mg/kg of dry weight.

The Meeting estimated a maximum residue level of 70 mg/kg and an STMR value for parathion-methyl in alfalfa forage of 3.7 mg/kg (dry weight).

The concentrations of parathion-methyl residues in *alfalfa hay* were 0.28, 0.33, 0.36, 0.37, 0.38 (2 trials), 0.39, 0.46, 0.63, 0.64 (4 trials), 0.67, 0.79, 0.81, 0.87, 1.0, 1.2, 1.3 (3 trials), 1.4 (2 trials), 1.5, 1.7 (3 trials), 1.8, 1.9 (3 trials), 2.1, 2.2 (2 trials), 2.3, 2.7, 3.0, 3.4, 3.5, 4.1, 4.2, 4.5, 5.7, 6.4, 8.0, 8.8, 13, 17 (2 trials), and 23 mg/kg of fresh weight or 0.39, 0.41, 0.42 (2 trials), 0.45, 0.49, 0.55 (2 trials), 0.70 (2 trials), 0.90, 1.0, 1.2 (3 trials), 1.3, 1.4 (2 trials), 1.6, 1.7, 1.8, 2.0, 2.1 (3 trials), 2.3 (3 trials), 2.4, 2.7 (2 trials), 3.0, 3.2, 3.5 (2 trials), 3.8, 3.9, 4.6 (2 trials), 5.2, 5.3, 5.7, 8.4, 11 (2 trials), 16 (2 trials), 18, 21, 27, and 57 mg/kg of dry weight.

The concentrations of combined parathion-methyl and paraoxon-methyl residues in alfalfa hay were 0.28, 0.33, 0.36, 0.37, 0.38 (2 trials), 0.39, 0.46, 0.63, 0.64 (2 trials), 0.65 (2 trials), 0.67, 0.80, 0.82, 0.89, 1.0, 1.2, 1.3 (3 trials), 1.4 (2 trials), 1.5, 1.7 (3 trials), 1.8, 1.9 (3 trials), 2.1, 2.2, 2.3 (2 trials), 2.7, 3.0, 3.4, 3.5, 4.1, 4.3, 4.6, 5.8, 6.5, 8.1, 8.9, 13, 17 (2 trials), and 23 mg/kg of fresh weight or 0.39, 0.41, 0.42 (2 trials), 0.45, 0.49, 0.55 (2 trials), 0.70, 0.71, 0.90, 1.0, 1.2 (3 trials), 1.3, 1.5 (2 trials), 1.6, 1.7, 1.8, 2.1 (2 trials), 2.2 (2 trials), ***2.3*** (2 trials), 2.4 ( 2 trials), 2.8 (2 trials), 3.0, 3.2, 3.5, 3.6, 3.8, 3.9, 4.6, 4.7, 5.2, 5.4, 5.8, 8.5, 11, 12, 16 (2 trials), 18, 21, 27, and 57 mg/kg of dry weight.

The Meeting estimated a maximum residue level of 70 mg/kg and an STMR value of 2.3 mg/kg for parathion-methyl in alfalfa fodder (dry weight).

The concentrations of residues in pea hay or *pea fodder* (described in the trials as dried forage) and of *pea straw* were measured in the trials on field peas (see above) carried out according to GAP. The concentrations of parathion-methyl residues in pea hay or pea fodder (12 trials) were 0.35, 0.37, 0.55, 0.66, 1.0, 3.4, 3.6, 4.2, 5.2, 7.6, 9.5, and 58 mg/kg. The concentrations of parathion-methyl residues in pea straw (11 trials) were 0.71, 0.72, 0.82, 1.1, 2.6, 3.1, 3.5, 4.9, 5.0, 13, and 27 mg/kg. One of these trials was invalid because of an excessively contaminated control plot. Because dried forage and pea straw are included in the commodity pea hay or pea fodder (dry), the data on dried forage and pea straw were combined, and the higher residue in each trial was used to estimate the MRL and STMR value. The concentrations of parathion-methyl were then: 0.71, 0.72, 0.82, 1.0, 1.1, 4.2, 4.9, 5.0, 5.2, 9.5, 13, and 58 mg/kg, and those of the combined parathion-methyl and paraoxon-methyl residues become: 0.71, 0.90, 0.92, 1.0, 1.1, ***4.3***, ***5.3***, 5.4, 5.5, 9.7, 13, and 59 mg/kg.

Allowing for the standard 88% of dry matter in pea hay (FAO, 1997, p. 125), the Meeting estimated a maximum residue level of 70 mg/kg and an STMR value of 5.5 mg/kg (4.8/0.88) for parathion-methyl in pea hay or pea fodder (dry weight).

As noted above, parathion-methyl is registered in the USA for use on peas for production of dried peas. In 11 trials conducted in 1988 and 1989 with four or six applications at 1.1 kg ai/ha and a 15-day PHI or with a final application at 0.56 kg ai/ha and a PHI of 10 days, the concentrations of parathion-methyl residues in *pea vine* were <0.05 (4 trials), 0.08, 0.17, 0.20, 0.21, 0.23, 1.6, and 7.3 mg/kg of fresh weight. In the same trials, the concentrations of parathion-methyl residues on *succulent forage* were <0.05 (4 trials), 0.07, 0.08 (2 trials), 0.13, 0.15, 0.17, 4.9, and 8.2 mg/kg. As succulent forage is included in the commodity pea vines, the data for pea vines and succulent forage

were combined, and the higher residue value in each trial was used to estimate the MRL and STMR value. The concentrations of parathion-methyl data are then: <0.05 (4 trials), 0.08, 0.17 (2 trials), 0.20, 0.21, 0.23, 4.9, and 8.2 mg/kg, and those of the combined parathion-methyl and paraoxon-methyl residues become: <0.05 (4 trials), 0.08, ***0.17***, ***0.20***, 0.23, 0.27, 0.28, 4.9, and 8.2 mg/kg.

Allowing for the standard 25% of dry matter in pea vines (FAO, 1997, p. 125), the Meeting estimated a maximum residue level of 40 mg/kg and an STMR value of 0.74 mg/kg (0.185/0.25) for parathion-methyl in green pea vines (dry weight).

As noted above, parathion-methyl is registered in the USA for use on beans for production of dried beans. In six trials in four states in 1988 with six applications at 1.7 kg ai/ha and sampling of *bean forage* at a 21-day PHI, the concentrations of parathion-methyl residues were <0.05 (2 trials), 0.10, ***0.11***, 0.31, and 0.66 mg/kg. The residues are expressed on a fresh weight basis because the percent dry matter for bean forage is not provided in the *FAO Manual* (FAO, 1997).

The Meeting estimated a maximum residue level of 1 mg/kg and an STMR value of 0.11 mg/kg for parathion-methyl in green bean forage. The estimated maximum residue level confirms the current MRL recommendation for bean forage of 1 mg/kg.

As noted above, parathion-methyl is registered in the USA for use on wheat. In six trials in four states, *wheat hay* was cut 14 days after a final treatment with parathion-methyl at four applications of 1.4 kg ai/ha followed by two applications of 0.84 kg ai/ha, which was considered to represent GAP for the purposes of measuring residues. The resulting concentrations of residues in wheat hay were 0.10, 0.17, 0.33, 0.98, 1.0, and 1.2 mg/kg (fresh weight).

In nine trials on wheat in seven states in 1988 and 1999, parathion-methyl was applied four times at 1.4 kg ai/ha and then twice at 0.84 kg ai/ha, and *wheat straw* was harvested 14 days after the final treatment. The concentrations of parathion-methyl residues in wheat straw were 0.13, 0.28, 0.34, 0.55, 0.79, 0.85, 2.6, 3.7, and 5.7 mg/kg (fresh weight)

The data on wheat hay and straw were combined to support an MRL for wheat straw and fodder. The concentrations of the combined parathion-methyl and paraoxon-methyl residues in wheat straw and fodder become: 0.10, 0.13, 0.23, 0.28, 0.34, 0.49, 0.67, ***0.91***, 0.94, 1.1, 1.2, 2.4, 2.8, 4.1, and 5.9 mg/kg.

Allowing for the standard 88% of dry matter in wheat hay and straw (FAO, 1997, p. 127), the Meeting estimated a maximum residue level of 10 mg/kg and an STMR value of 10 and 1.03 mg/kg (0.91/0.88) for parathion-methyl in wheat straw and fodder (dry weight). The estimated maximum residue level confirms the current recommendation for wheat straw and fodder of 10 mg/kg.

Parathion-methyl is registered in the USA for use on forage grasses at 0.56-0.84 kg ai/ha with a PHI of 15 days for cutting or grazing. In 15 trials in six states in 1988 with six applications at 0.86-0.89 kg ai/ha and sampling of *pasture grass hay* after 15 days, the concentrations of parathion-methyl residues were 0.05, 0.12, 0.19, 0.21, 0.25, 0.31, 0.50, ***0.54***, 0.64, 0.66, 0.96, 1.0, 1.4, 1.6, and 2.5 mg/kg (fresh weight), and those of the combined parathion-methyl and paraoxon-methyl residues in pasture grass hay were 0.14, 0.23, 0.25, 0.26, 0.32, 0.45, 0.54, ***0.60***, 0.66, 0.70, 0.96, 1.1, 1.5, 1.6, and 2.9 mg/kg of fresh weight.

Allowing for the standard 88% of dry matter in hay of pasture grasses (FAO, 1997, p. 124), the Meeting estimated a maximum residue level of 5 mg/kg and an STMR value of 0.68 mg/kg (0.60/0.88) for parathion-methyl in grass hay or fodder (dry weight). The estimated maximum residue level confirms the current recommendation for hay or fodder of grasses of 5 mg/kg.

As noted above, parathion-methyl is registered in the USA for use on sugar beets at 0.28-0.43 kg ai/ha with a PHI of 60 days for *sugar beet tops* used as animal fodder. Neither parathion-methyl residues nor paraoxon-methyl residues were detected in beet fodder in six trials in four states in 1988, with six applications at 0.42 kg ai/ha and fodder harvesting at 60 days. Residues of parathion-methyl are unlikely to occur after such an interval, but there was no evidence that residues were not present.

The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR value of 0.05 mg/kg for parathion-methyl in beet tops and fodder (fresh weight). The estimated maximum residue level confirms the current recommendation for sugar beet leaves or tops of 0.05* mg/kg.

***Fate of residues during processing***

The Meeting received information on the fate of incurred residues of parathion-methyl and paraoxon-methyl during the processing of apple, peach, grapes, olive, soya bean, wheat, maize, rice, cotton seed, sunflower seed, rape seed, and hops, and processing factors were calculated for processed commodities derived from these raw agricultural commodities. The studies on apple, peach, grape, wheat, maize, cotton seed, and rape seed are summarized below because maximum residue levels are estimated for these raw agricultural commodities.

Processing factors were calculated for parathion-methyl residues only and for combined parathion-methyl and paraoxon-methyl residues. As parathion-methyl is the dominant component of the residue, the processing factor is similar with the two calculations. Nevertheless, since these factors are used in calculating the concentrations of residues in processed foods for the purpose of estimating dietary intake, that for the combined residue was used when available. When the concentration of residues in the processed commodity did not exceed the LOQ, the processing factor was calculated from the LOQ and was prefixed with a ‘less than’ symbol (<).

The factors for parathion-methyl in *apple* processed to dry pomace were 4.0, 4.3, 4.5, and 8.0 (mean, 5.2) and those for juice were <0.25, <0.5, <0.33, and <1. As no residues were detected in juice, the value <0.25 is the best estimate of the juice processing factor. Application of these factors to the STMR value and MRL for apples provides an STMR-P value of 0.31 mg/kg and a HR-P value of 1.04 mg/kg for dry apple pomace and an STMR-P value for juice of 0.015 mg/kg.

Parathion-methyl residues were not detected in *peach* juice in two trials in which the calculated processing factors were <0.33 and <0.5; the best estimate is <0.33. Application of the factor to the STMR value for peaches provides an STMR-P value for peach juice of 0.031 mg/kg.

The factors for the processing of *grape* to raisins were 1.3 (2 trials), 1.5, and 1.6 (mean, 1.4). Application of the factor to the STMR value and MRL for grapes provides an STMR-P value of 0.014 mg/kg and a HR-P value of 0.70 mg/kg for raisins. The Meeting estimated a maximum residue level for parathion-methyl in dried grapes of 1 mg/kg. The factors for processing of grapes to juice were 0.06, <0.07, <0.10, and <0.11, and the best estimate is 0.06. Application of the factor to the STMR value for grapes provides an STMR-P value for grape juice of 0.0006 mg/kg. The factors for processing of grapes to wine were <0.077, <0.083, 0.13, 0.19, 0.20, and 0.22 (mean, 0.15). Application of the factor to the STMR value for grapes provides an STMR-P value for wine of 0.0015 mg/kg.

The factors for processing of *wheat* to bran were 2.0 and 2.4 (mean, 2.2). Application of the factor to the STMR value for wheat provides an STMR-P value for wheat bran of 0.64 mg/kg. Application of the factor to the MRL for wheat gives a highest residue of parathion-methyl in wheat bran of 11.0 mg/kg. The Meeting estimated a maximum residue level for parathion-methyl in wheat bran of 10 mg/kg, which confirms the current recommendation for unprocessed wheat bran.

The factors for processing of wheat to flour were 0.29, 0.42, and 0.45 (mean, 0.39). Application of the factor to the STMR value for wheat provides an STMR-P value for flour of 0.11 mg/kg. Application of the factor to the MRL for wheat gives a highest residue of parathion-methyl in flour of 1.95 mg/kg. The Meeting estimated a maximum residue level for parathion-methyl in wheat flour of 2 mg/kg.

The processing factors for dry milling of *maize* were 0.21, 0.19, and 0.74 (mean, 0.38) for grits, 0.47 and 0.45 (mean, 0.46) for meal, 0.41 for flour, 0.31 for crude oil, and 0.26 for refined oil. The processing factors for wet milling of maize were <0.09 for starch, 1.33 for crude oil, and 1.03 for refined oil. Application of the factors to the STMR value and MRL for maize provides an STMR-P value of 0.023 mg/kg and a highest residue of 0.046 mg/kg in meal and STMR-P values of 0.021 mg/kg for maize flour, 0.019 mg/kg for grits, and 0.0045 mg/kg for starch. Application of the factor for flour (0.41) to the MRL for maize gives a highest residue of parathion-methyl in maize flour of 0.041 mg/kg. The Meeting estimated a maximum residue level for parathion-methyl in maize flour of 0.05 mg/kg.

The two processes resulted in different concentrations of residues in maize oil. The processing factors for oils were 0.31 and 0.26 with the dry process and 1.33 and 1.03 with the wet process. The Meeting agreed to use the values for the wet process, which, when applied to the STMR value for maize, provide STMR-P values of 0.067 mg/kg for crude oil and 0.051 mg/kg for refined oil. Application of the factors to the MRL for maize provides highest residues of 0.13 mg/kg in crude maize oil and 0.10 mg/kg in refined maize oil. The Meeting estimated maximum residue levels for parathion-methyl in crude maize oil and edible maize oil of 0.2 and 0.1 mg/kg, respectively.

The processing factors for *cotton seed* milling were 0.04 and 0.12 (mean, 0.08) for meal, 0.41 and 0.47 (mean, 0.44) for hulls, 0.81 and 0.07 (mean, 0.44) for crude oil, and 0.59 and 0.06 (mean, 0.33) for refined oil. Application of the factors to the STMR value and MRL for cotton seed provides an STMR-P value of 0.28 mg/kg and a HR-P value of 2.0 mg/kg in meal, an STMR-P value of 1.5 mg/kg and a HR-P value of 9.7 mg/kg in hulls, and STMR-P values of 1.54 mg/kg for crude oil and 1.16 mg/kg for refined oil. Application of the factors to the MRL for cotton seed provides highest residues of parathion-methyl of 11 mg/kg in crude cotton seed oil and 8.25 mg/kg in refined cotton seed oil. The Meeting estimated a maximum residue level of 10 mg/kg for parathion-methyl in both crude and edible cotton seed oil.

The processing factors for *rape seed* were 0.22 for meal, 2.4 for crude oil, and 2.0 for refined oil. Application of the factors to the STMR value and MRL for rape seed provides an STMR-P and a highest residue in rape seed meal of 0.011 mg/kg and an STMR-P value of 0.12 mg/kg for crude oil and 0.10 mg/kg for refined oil. Application of the factors for oil to the MRL for rape seed provides highest residues of parathion-methyl of 0.12 mg/kg in crude rape seed oil and 0.10 mg/kg in refined rape seed oil. The Meeting estimated a maximum residue level for parathion-methyl in crude and edible rape seed oil of 0.2 mg/kg.

***Residues in animal and poultry commodities***

The Meeting estimated the dietary burden of parathion-methyl residues in farm animals on the basis of the diets listed in Appendix IX of the *FAO Manual* (FAO, 1997). Calculation from MRLs (or HR values) provides concentrations in feed suitable for estimating MRLs for animal commodities, while calculation from STMR values for feed is suitable for estimating STMR values for animal commodities. The percent dry matter is considered to be 100% for MRLs and STMR values expressed in dry weight.

| Commodity | MRL or HR | Group | % dry matter | MRL/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |

| Commodity | MRL or HR | Group | % dry matter | MRL/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |
| Alfalfa fodder | 70 | AL | 100 | 70 | 70 | 60 | | 49.0 | 42.0 | |
| Alfalfa forage (green) | 70 | AL | 100 | 70 | | | | | | |
| Bean forage (green) | 1 | AL | 25 | 4.0 | | | | | | |
| Pea hay or pea fodder (dry) | 70 | AL | 100 | 70 | | | | | | |
| Pea vines (green) | 40 | AL | 100 | 40 | | | | | | |
| Hay or fodder (dry) of grasses | 5 | AS | 100 | 5.0 | | | | | | |
| Wheat straw and fodder, dry | 10 | AS | 100 | 10 | 10 | 10 | | 1.00 | 1.00 | |
| Sugar beet leaves or tops | 0.05 | AV | 23 | 0.22 | | | | | | |
| Maize meal | 0.046 | CF | 85 | 0.054 | | | | | | |
| Maize | 0.1 | GC | 88 | 0.11 | | | | | | |
| Wheat | 5 | GC | 89 | 5.62 | | 10 | 80 | | 0.56 | 4.49 |
| Apple pomace (dry) | 1.04 | AB | 100 | 1.04 | | | | | | |
| Cotton seed hulls | 9.7 | | 90 | 10.8 | 20 | 20 | | 2.16 | 2.16 | |
| Cotton seed meal | 2.00 | | 88 | 2.27 | | | 20 | | | 0.45 |
| Rape seed meal | 0.011 | | 88 | 0.025 | | | | | | |
| Total | | | | | | | | 52.2 | 45.7 | 4.95 |

| Commodity | STMR | Group | % dry matter | STMR/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |
| Alfalfa fodder | 2.3 | AL | 100 | 2.3 | | | | | | |
| Alfalfa forage (green) | 3.7 | AL | 100 | 3.7 | 45 | 10 | | 1.67 | 0.37 | |
| Bean forage (green) | 0.11 | AL | 25 | 0.44 | | | | | | |
| Pea hay or pea fodder (dry) | 5.5 | AL | 100 | 5.5 | 25 | 50 | | 1.38 | 2.75 | |
| Pea vines (green) | 0.74 | AL | 100 | 0.74 | | | | | | |
| Hay or fodder (dry) of grasses | 0.68 | AS | 100 | 0.68 | | 10 | | | 0.07 | |
| Wheat straw and fodder, dry | 1.03 | AS | 100 | 1.03 | 10 | 10 | | 0.10 | 0.10 | |
| Sugar beet leaves or tops | 0.05 | AV | 23 | 0.22 | | | | | | |
| Maize meal | 0.023 | CF | 85 | 0.027 | | | | | | |
| Maize | 0.05 | GC | 88 | 0.06 | | | | | | |
| Wheat | 0.29 | GC | 89 | 0.33 | | | 80 | | | 0.26 |
| Apple pomace (dry) | 0.31 | AB | 100 | 0.31 | | | | | | |
| Cotton seed hulls | 1.54 | | 90 | 1.71 | 20 | 20 | | 0.34 | 0.34 | |
| Cotton seed meal | 0.28 | | 88 | 0.32 | | | 20 | | | 0.06 |
| Rape seed meal | 0.011 | | 88 | 0.01 | | | | | | |
| Total | | | | | | | | 3.5 | 3.6 | 0.32 |

The dietary burdens of parathion-methyl in animal commodities (expressed as dry weight) used to estimate the MRL and STMR value are: 52 and 3.5 ppm for beef cattle, 46 and 3.6 ppm for dairy cows, and 4.95 and 0.32 ppm for poultry.

No suitable studies of farm animal feeding were available to allow conversion of the dietary burden of residues to MRLs and STMR values for animal and poultry commodities. The Meeting was informed that such studies will be initiated shortly.

## RECOMMENDATIONS

The Meeting estimated the maximum residue levels and STMRs shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs: parathion-methyl. For estimation of dietary intake: sum of parathion-methyl and paraoxon-methyl expressed as parathion-methyl.

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| AL 1020 | Alfalfa fodder | 70 | - | 2.3 | |

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| AL 1021 | Alfalfa forage (green) | 70 | - | 3.7 | |
| FP 0226 | Apple | 0.2 | - | 0.06 | 0.18 |
| JF 0226 | Apple juice | | | 0.015 | |
| AB 0226 | Apple pomace, dry | | | 0.31 | 1.04 |
| VS 0620 | Artichoke, Globe | W | 2 | | |
| AL 1030 | Bean forage (green) | 1 (fresh weight) | 1 | 0.11 | |
| VD 0071 | Beans (dry) | 0.05* | 0.05* | 0.05 | 0.05 |
| VB 0400 | Broccoli | W | 0.2 | | |
| VB 0041 | Cabbages, Head | 0.05 | 0.2 | 0.05 | 0.26 |
| VR 0577 | Carrot | W | 1 | | |
| VS 0624 | Celery | W | 5 | | |
| FS 0013 | Cherries | W | 0.01* | | |
| AL 1023 | Clover | W | 10 | | |
| VP 0526 | Common bean (pods and/or immature seeds) | W | 0.05* | | |
| SO 0691 | Cotton seed | 25 | - | 3.5 | 22 |
| | Cotton seed hulls | | | 1.54 | 9.7 |
| | Cotton seed meal | | | 0.28 | 2.0 |
| OC 0691 | Cotton seed oil, crude | 10 | - | 1.54 | |
| OR 0691 | Cotton seed oil, edible | 10 | - | 1.16 | |
| DF 0269 | Dried grapes (= Currants, Raisins and Sultanas) | 1 | | 0.14 | 0.70 |
| VP 0528 | Garden pea (young pods) | W | 1 | | |
| FB 0268 | Gooseberry | W | 0.01* | | |
| JF 0269 | Grape juice | | | 0.0006 | |
| FB 0269 | Grapes | 0.5 | - | 0.10 | 0.41 |
| AS 0162 | Hay or fodder (dry) of grasses | 5 | 5 | 0.68 | |
| DH 1100 | Hops, dry | W | 1 | | |
| VL 0482 | Lettuce, Head | W | 0.05* | | |
| VL 0483 | Lettuce, Leaf | W | 0.5 | | |
| VP 0534 | Lima bean (young pods and/or immature beans) | W | 0.05* | | |
| GC 0645 | Maize | 0.1 | - | 0.05 | 0.09 |
| CF 1255 | Maize flour | 0.05 | - | 0.021 | |
| | Maize grits | | | 0.019 | |
| | Maize meal | | | 0.023 | 0.046 |
| OC 0645 | Maize oil, crude | 0.2 | - | 0.067 | |
| OR 0645 | Maize oil, edible | 0.1 | - | 0.051 | |
| | Maize starch | | | 0.0045 | |
| VL 0485 | Mustard greens | W | 0.5 | | |
| AL 0072 | Pea hay or Pea fodder (dry) | 70 | - | 5.5 | |
| AL 0528 | Pea vines (green) | 40 | - | 0.74 | |
| FS 0247 | Peach | 0.3 | - | 0.095 | 0.22 |
| | Peach juice | | | 0.031 | |
| VD 0072 | Peas (dry) | 0.3 | 0.2 | 0.06 | 0.24 |
| FS 0014 | Plums (including Prunes) | W | 0.01* | | |
| VR 0589 | Potato | 0.05* | 0.05* | 0 | 0 |
| SO 0495 | Rape seed | 0.05 | - | 0.05 | 0.05 |
| | Rape seed meal | | | 0.011 | 0.011 |
| OC 0495 | Rape seed oil, crude | 0.2 | - | 0.12 | |
| OR 0495 | Rape seed oil, edible | 0.2 | - | 0.10 | |
| FB 0272 | Raspberries, Red, Black | W | 0.01* | | |
| GC 0649 | Rice | W | 3 | | |
| AS 0649 | Rice straw and fodder, dry | W | 10 | | |
| CM 0649 | Rice, husked | W | 1 | | |
| VL 0502 | Spinach | W | 0.5 | | |
| VR 0596 | Sugar beet | 0.05* | 0.05* | 0 | 0 |
| AV 0596 | Sugar beet leaves or tops | 0.05* (fresh weight) | 0.05* | 0.05 | |
| VL 0506 | Turnip greens | W | 2 | | |

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| VR 0506 | Turnip, Garden | W | 0.05* | | |
| GC 0654 | Wheat | 5 | 5 | 0.29 | 4.1 |
| CM 0654 | Wheat bran, unprocessed | 10 | 10 | 0.64 | |
| CF 1211 | Wheat flour | 2 | | 0.11 | |
| AS 0654 | Wheat straw and fodder, dry | 10 | 10 | 1.03 | |
| | Wine | | | 0.0015 | |

**Further work or information**

*Desirable*

1. Feeding studies in farm animals to permit estimation of maximum residue levels and STMR values for animal and poultry commodities.

2. Information on the stability of paraoxon-methyl in fruits in frozen storage.

3. Information on the metabolism of parathion-methyl in fruits.

## Dietary risk assessment

***Chronic intake***

The periodic review of parathion-methyl resulted in recommendations for new and revised MRLs and new STMR values for raw and processed commodities. Data on consumption were available for 17 food commodities and were used in calculating the dietary intake. The results are shown in Annex 3.

The international estimated daily intakes from the five GEMS/Food regional diets, based on estimated STMR values, represented 3-30% of the ADI. The Meeting concluded that the long-term intake of residues of parathion-methyl from uses that have been considered by the JMPR is unlikely to present a public health concern.

***Short-term intake***

The IESTI for parathion-methyl was calculated for the food commodities (and their processing fractions) for which maximum residue levels and STMR values were estimated and for which consumption data were available. The results are shown in Annex 4. The IESTI represented 0-30% of the acute RfD for the general population and 0-80% of the acute RfD for children.

The Meeting concluded that the acute intake of residues of parathion-methyl from uses that have been considered by the JMPR is unlikely to present a public health concern.

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MP-LB-7016, LeRoy 1990c
MP-LE-3089, Cañez 1990d
MP-LE-3091, Cañez 1990d
MP-LE-3092, Cañez 1990d
MP-LE-3093, Cañez 1990d
MP-LE-3094, Cañez 1990d
MP-LE-3095, Cañez 1990d
MP-LE-3097, Cañez 1990d
MP-LE-3098, Cañez 1990d
MP-LE-3099, Cañez 1990d
MP-LE-3192, Jones 1990b.
MP-LE-7068, Cañez 1990d
MP-LE-7070, Cañez 1990d
MP-LE-7072, Cañez 1990d
MP-LE-7074, Cañez 1990d
MP-LE-7076, Cañez 1990d
MP-LE-7078, Cañez 1990d
MP-MG-3101, Cañez 1989a
MP-MG-3103, Cañez 1989a
MP-MG-3104, Cañez 1989a
MP-MG-3106, Cañez 1989a
MP-MG-7080, Cañez 1989a
MP-MG-7082, Cañez 1989a
MP-MG-7084, Cañez 1989a
MP-ON-3107, Cañez 1990a
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MP-ON-3109, Cañez 1990a
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MP-PE-3189, LeRoy 1991
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MP-PE-7093, LeRoy 1991
MP-PE-7095, LeRoy 1991
MP-PE-7096, LeRoy 1991
MP-PE-7098, LeRoy 1991
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MP-PO-3502, Cañez 1990p
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MP-PO-7105, Cañez 1990p
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MP-RI-3514, LeRoy 1990f
MP-RI-3515, LeRoy 1990f
MP-RI-7109, Cañez 1990l
MP-RI-7110, Cañez 1990l
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MP-SB-3146, Cañez 1990q
MP-SB-3148, Cañez 1990q
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MP-SG-7113, Cañez 1990m
MP-SG-7114, Cañez 1990m
MP-SG-7115, Cañez 1990m
MP-SG-7116, Cañez 1990m
MP-SP-3136, Cañez 1989b
MP-SP-3138, Cañez 1989b
MP-SP-3139, Cañez 1989b
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MP-SP-3143, Cañez 1989b
MP-SP-3187, Cañez 1989b
MP-SP-7123, Cañez 1989b
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MP-SS-7129, Cañez 1989c
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MP-SY-7121, Cañez 1990f
MP-SY-7122, Cañez 1990f
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MP-TU-7132, Cañez 1990i
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MP-WH-3159, LeRoy 1990d
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MP-WH-3165, LeRoy 1990d
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MP-WH-3182, LeRoy 1990d
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MP-WH-7143, LeRoy 1990d
MP-WH-7145, LeRoy 1990d
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PAL-MP-BR, Cañez 1990b
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PAL-MP-CN-P, LeRoy 1990e.
PAL-MP-CS, Cañez 1990n
PAL-MP-CS-P, LeRoy 1990g
PAL-MP-CT, Cañez 1990h
PAL-MP-CY, Cañez 1990j
PAL-MP-DB, Cañez 1990g.
PAL-MP-LB, Cañez 1990e
PAL-MP-LB, LeRoy 1990c
PAL-MP-LE, Cañez 1990d
PAL-MP-LE, Jones 1990b.
PAL-MP-MG, Cañez 1989a
PAL-MP-ON, Cañez 1990a
PAL-MP-ON, Jones 1990a
PAL-MP-PE, LeRoy 1991
PAL-MP-PO, Cañez 1990p
PAL-MP-RI, Cañez 1990l
PAL-MP-RI-P, LeRoy 1990f
PAL-MP-SB, Cañez 1990q
PAL-MP-SG, Cañez 1990m
PAL-MP-SP, Cañez 1989b
PAL-MP-SS, Cañez 1989c
PAL-MP-SY, Cañez 1990f
PAL-MP-SY-P, Cañez 1990o
PAL-MP-TU, Cañez 1990i
PAL-MP-WH-P, LeRoy 1990d
PF-3769, Hellpointer 1992
PM9801, Williams and Rice 1999d.
PM9802, Williams and Rice 1999a
PM9803, Williams and Rice 1999e.
PM9804, Williams and Rice 1999b
PM9805, Williams and Rice 1999c
PSI 97.438, Boner 1998.
RA-2134/94, Blass 1995b
RA-2135/94, Blass 1995c
RA-2143/94, Blass 1995a
RA-2144/94, Blass 1996
RA-3144/94, Blass and Waltz-Tylla 1996
XBL97072, Boner 1998.

# PYRETHRINS (063)

## EXPLANATION

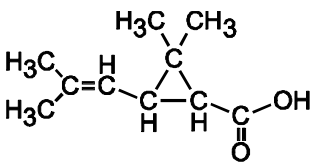
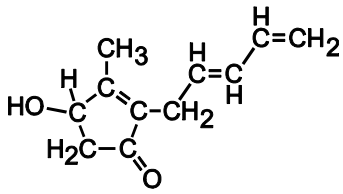
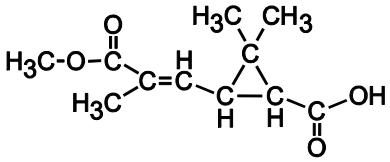
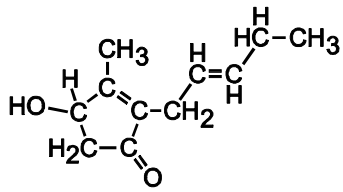
Residue aspects of the pyrethrins were first evaluated by the JMPR in 1966, with supplementary evaluations in 1969, 1972 and 1974. At its 26th (1994) Session, the CCPR noted that the compounds were originally scheduled for both toxicological and residue periodic review by the 1994 JMPR, but the toxicological evaluation was re-scheduled for 1999 and the residue review for 2000. The present review is within the CCPR Periodic Review Programme.

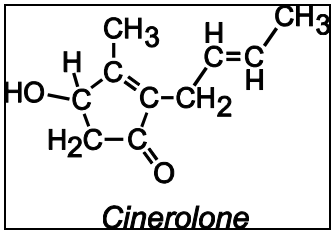
The manufacturer provided data on metabolism in animals and plants, environmental fate, stability of pesticide residues in storage, use pattern, residues in citrus, small fruits, brassica leafy vegetables, cucurbits, peppers, tomatoes, beans, peas, root and tuber vegetables, celery and mustard seeds after foliar treatment, and in beans, prunes and peanuts after warehouse treatment, and information on animal feeding studies, fate of residues in processing, residues in food in commerce, and national maximum residue limits. National maximum residue limits were reported by the government of Australia and information on GAP was provided by the governments of Germany and Poland. Residue data on celeriac and leeks were also provided by the German government.

## IDENTITY

The pyrethrins are a naturally-occurring group of six chemically-related esters, each of which is insecticidally active. Three (pyrethrins I) are esters of chrysanthemic acid, and three (pyrethrins II) of pyrethric acid. The alcohol moieties are pyrethrelone in pyrethrin 1 and 2, cinerolone in cinerin 1 and 2, and jasmolone in jasmolin 1 and 2. Table 1 gives the structures of the acid and alcohol moieties.

Table 1. Component moieties of pyrethrin esters.

| ACID | ALCOHOL |
|---|---|
|  *Chrysanthemic Acid* |  *Pyrethrolone* |
|  *Pyrethric Acid* |  *Jasmolone* |



*Cinerolone*

The six pyrethrin esters are designated collectively by the ISO common name "pyrethrins", with CIPAC No. 32. Pyrethrin 1 predominates. Information on the individual esters is provided below, where the IUPAC chemical names are according to Rothamsted nomenclature.

**pyrethrin 1**

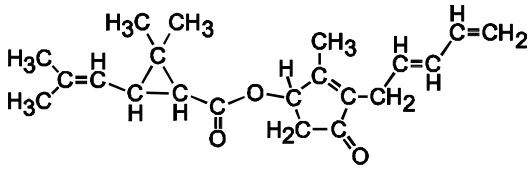
Chemical names:

IUPAC: (*Z*)-(*S*)-2-methyl-4-oxo-3-(penta-2,4-dienyl)cyclopent-2-enyl (1*R*)-*trans*-2,2-dimethyl-3-(2-methylprop-1-enyl)cyclopropanecarboxylate

CAS: [1*R*-[1α[*S**(*Z*)],3β]]-2-methyl-4-oxo-3-(2,4-pentadienyl)cyclopenten-1-yl 2,2-dimethyl-3-(2-methyl-1-propenyl)cyclopropanecarboxylate

CAS No.: 121-21-1

Structural formula:



*Pyrethrin 1*

Molecular formula: $C_{21}H_{28}O_3$ Molecular weight: 328.4

**cinerin 1**

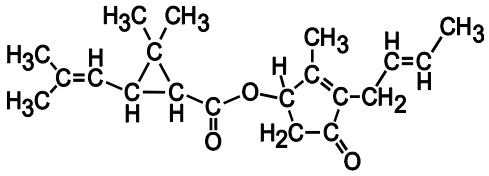
Chemical names:

IUPAC: (*Z*)-(*S*)-3-(but-2-enyl)-2-methyl-4-oxocyclopent-2-enyl (1*R*)-*trans*-2,2-dimethyl-3-(2-methylprop-1-enyl)cyclopropanecarboxylate

CAS: [1*R*-[1α[*S**(*Z*)],3β]]-3-(2-butenyl)-2-methyl-4-oxo-2-cyclopenten-1-yl 2,2-dimethyl-3-(2-methyl-1-propenyl)cyclopropanecarboxylate

CAS No.: 25402-06-6

Structural formula:



*Cinerin 1*

Molecular formula: $C_{20}H_{28}O_3$ Molecular weight: 316.4

**jasmolin 1**

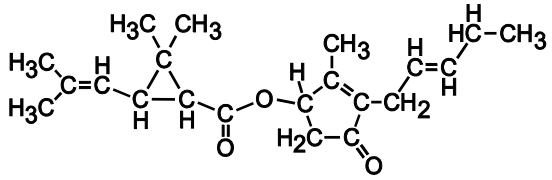
Chemical names:

IUPAC: (*Z*)-(*S*)-2-methyl-4-oxo-3-(pent-2-enyl)cyclopent-2-enyl (1*R*)-*trans*-2,2-dimethyl-3-(2-methylprop-1-enyl)cyclopropanecarboxylate

CAS: [1*R*-[1α[*S**(*Z*)],3β]]-2-methyl-4-oxo-3-(2-pentenyl)-2-cyclopenten-1-yl 2,2-dimethyl-3-(2-methyl-1-propenyl)cyclopropanecarboxylate

CAS No.: 4466-14-2

Structural formula:



*Jasmolin 1*

Molecular formula: $C_{21}H_{30}O_3$ Molecular weight: 330.4

**pyrethrin 2**

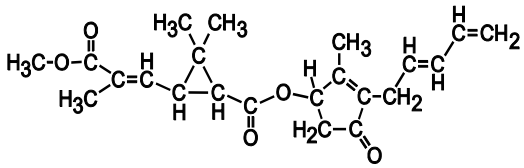
Chemical names:

(*Z*)-(*S*)-2-methyl-4-oxo-3-(penta-2,4-dienyl)cyclopent-2-enyl (*E*)-(1*R*)-*trans*-3-(2-methoxycarbonylprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

CAS: [1*R*-[1α[*S**(*Z*)],3β(*E*)]]-2-methyl-4-oxo-3-(2,4-pentadienyl)-2-cyclopenten-1-yl 3-(3-methoxy-2-methyl-3-oxo-1-propenyl)-2,2-dimethylcyclopropanecarboxylate

CAS No.: 121-29-9

Structural formula:



*Pyrethrin 2*

Molecular formula: $C_{22}H_{28}0_5$ Molecular weight: 372.4

**cinerin 2**

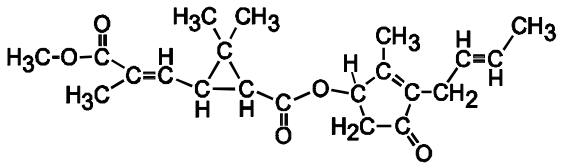
Chemical names:

IUPAC: (*Z*)-(*S*)-3-(but-2-enyl)-2-methyl-4-oxocyclopent-2-enyl (*E*)-(1*R*)-*trans* -3-(2-methoxycarbonylprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

CAS: [1*R*-[1α[*S**(*Z*)],3β(*E*)]]-3-(2-butenyl)-2-methyl-4-oxo-2-cyclopenten-1-yl 3-(3-methoxy-2-methyl-3-oxo-1-propenyl)-2,2-dimethylcyclopropanecarboxylate

CAS No.: 1172-63-0

Structural formula:



*Cinerin 2*

Molecular formula: $C_{21}H_{28}O_5$ Molecular weight: 360.4

**jasmolin 2**

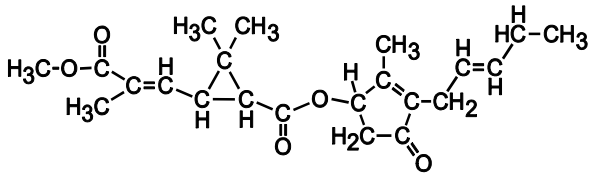
Chemical names:

IUPAC: (*Z*)-(*S*)-2-methyl-4-oxo-3-(pent-2-enyl)cyclopent-2-enyl (*E*)-(1*R*)-*trans*-3-(2-methoxycarbonylprop-1-enyl)-2,2-dimethylcyclopropanecarboxylate

CAS: [1*R*-[1α[*S**(*Z*)],3β(*E*)]]-2-methyl-4-oxo-3-(2-pentenyl)-2-cyclopenten-1-yl 3-(3-methoxy-2-methyl-3-oxo-1-propenyl)-2,2-dimethylcyclopropanecarboxylate

CAS No.: 121-20-0

Structural formula:



*Jasmolin 2*

Molecular formula: $C_{22}H_{30}O_5$ Molecular weight: 374.4

## Physical and chemical properties

Pure natural pyrethrins are relatively unstable at normal temperatures and storage conditions and are light-sensitive. The pure compounds are not an item of commerce. Except as noted below, data are provided for the technical active ingredient "pyrethrum extract 25%", the material that is stored, shipped, and used to formulate end-use product insecticides (Kenya, 1998). Where appropriate, physical and chemical properties are presented for the pure active ingredients pyrethrin 1 (PY 1) and pyrethrin 2 (PY 2).

Appearance: light amber viscous liquid.

Vapour pressure: PY 1: $2 \times 10^{-5}$ mm Hg
PY 2: $4 \times 10^{-7}$ mm Hg

| | |
|---|---|
| Melting point: | N/A |
| Boiling point: | 180°C (with decomposition) |
| Density: | 0.86 (specific gravity) |
| Partition coefficient: | PY 1: log $P_{OW}$ = 5.9<br>PY 2: log $P_{OW}$ = 4.3 |
| Solubility: | water: not soluble<br>organic solvents: readily soluble<br>lipids and fats: not determined |
| Hydrolysis (PY 1): | stable at pH 5 and pH 7 for 30 days<br>half-life at pH 9 = 17 days |
| Photolysis (PY 1): | in water at pH 7 PY1 forms an isomer in equilibrium with the parent compound when exposed to natural sunlight. The half-life of PY1 for this reaction is about 1 hour. Further degradation occurs, consistent with first-order kinetics. The overall half-life for parent and isomer is about 12 hours. |

## METABOLISM AND ENVIRONMENTAL FATE

Pyrethrin 1 is the main ester. [$^{14}$C]pyrethrin 1 (Figure 1) [$^{14}$C]PY labelled in the cyclopropane ring was used in all metabolism and environmental studies.

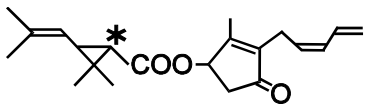



Figure 1. Site of radiolabel in [$^{14}$C]pyrethrin 1

### Animal metabolism

Rats. In a study to define the pharmacokinetic characteristics of [$^{14}$C]pyrethrin 1 metabolism (Selim, 1995e), rats were dosed with a single oral low dose (10 mg/kg bw), with a single oral high dose (100 mg/kg bw for the males and 50 mg/kg bw for the females) or with repeated oral low doses, in which unlabelled PY 1 at 10 mg/kg bw was given for 14 days before administration of a single dose of $^{14}$C-PY 1 at 10 mg/kg bw. A lower dose was administered to the females in the high dose group because previous studies showed that pyrethrum extract is more toxic to females than to males. Ten Charles River Sprague-Dawley CD® rats (5 males and 5 females) were used in each dose group. Rats were fasted for approximately 18 hours before the administration of the $^{14}$C-PY (between 13.6 and 18.4 μCi). Samples of urine and faeces were collected during the experiment. Seven days after the last dose, the rats were killed and samples of urine, faeces and tissues were collected for analysis. The results are shown in Tables 2-4.

Table 2. Excretion of radioactivity in rat urine, faeces and tissues after a single oral low dose.

| Time (hours) | Male (% of dose) | | Female (% of dose) | |
|---|---|---|---|---|
| | Urine | Faeces | Urine | Faeces |
| 0-4 | 1.2 | 0.00 | 3.77 | 0.90 |
| 4-8 | 6.6 | 0.03 | 3.61 | 0.00 |
| 8-12 | 5.8 | 0.61 | 8.43 | 0.04 |
| 12-24 | 15.0 | 27.0 | 17.2 | 13.63 |

| Time (hours) | Male (% of dose) | | Female (% of dose) | |
|---|---|---|---|---|
| | Urine | Faeces | Urine | Faeces |
| 24-36 | 7.2 | 6.7 | 10.62 | 11.4 |
| 36-48 | 3.6 | 24.0 | 6.28 | 10.6 |
| 48-72 | 1.5 | 3.48 | 3.52 | 11.3 |
| 72-96 | 0.51 | 0.70 | 0.58 | 1.65 |
| 96-120 | 0.24 | 0.25 | 0.30 | 0.29 |
| 120-144 | 0.27 | 0.12 | 0.22 | 0.11 |
| 144- 312 | 0.24 | 0.11 | 0.14 | 0.08 |
| **Total** | **42.1** | **63.1** | **54.7** | **50.0** |
| | **Total tissue (% of dose)** | | **Total tissue (% of dose)** | |
| 312 (death) | 0.46 | | 0.35 | |

Table 3. Excretion of radioactivity in rat urine, faeces and tissues after a single oral high dose.

| Time (hours) | Male (% of dose) | | Female (% of dose) | |
|---|---|---|---|---|
| | Urine | Faeces | Urine | Faeces |
| 0-4 | 1.05 | 0.00 | 1.82 | 4.36 |
| 4-8 | 3.19 | 0.00 | 3.23 | 0.00 |
| 8-12 | 4.53 | 12.00 | 11.8 | 4.29 |
| 12-24 | 14.2 | 29.4 | 16.7 | 13.88 |
| 24-36 | 4.94 | 14.4 | 8.73 | 12.32 |
| 36-48 | 2.00 | 9.51 | 3.87 | 11.45 |
| 48-72 | 1.28 | 4.33 | 1.98 | 2.68 |
| 72-96 | 0.44 | 0.68 | 0.50 | 0.70 |
| 96-120 | 0.25 | 0.36 | 0.35 | 0.26 |
| 120-144 | 0.17 | 0.21 | 0.26 | 0.14 |
| 144-312 | 0.12 | 0.15 | 0.12 | 0.08 |
| **Total** | **32.2** | **71.02** | **49.5** | **50.2** |
| | **Total tissue (% of dose)** | | **Total tissue (% of dose)** | |
| 312 (death) | 0.87 | | 0.57 | |

Table 4. Excretion of radioactivity in rat urine, faeces and tissues after repeated oral low doses.

| Time (hours) | Male (% of dose) | | Female (% of dose) | |
|---|---|---|---|---|
| | Urine | Faeces | Urine | Faeces |
| 0-4 | 2.26 | 0.00 | 2.20 | 0.00 |
| 4-8 | 10.1 | 0.00 | 14.4 | 0.00 |
| 8-12 | 7.45 | 0.02 | 13.1 | 0.93 |
| 12-24 | 15.8 | 23.0 | 16.2 | 23.01 |
| 24-36 | 5.79 | 16.6 | 5.78 | 13.3 |
| 36-48 | 3.38 | 8.10 | 2.89 | 10.3 |
| 48-72 | 1.41 | 6.21 | 1.33 | 2.89 |
| 72-96 | 0.37 | 0.89 | NA | 0.97 |
| 96-120 | 0.23 | 0.22 | 0.30 | 0.39 |
| 120-144 | 0.27 | 0.13 | 0.18 | 0.20 |
| 144-312 | 0.15 | 0.13 | 0.13 | 0.08 |
| **Total** | **47.2** | **55.3** | **57.1** | **52.2** |
| | **Total tissue (% of dose)** | | **Total tissue (% of dose)** | |
| 312 (death) | 0.57 | | 0.59 | |

NA: not analysed

In urine and faeces the excretion peaked after 12-24 hours, and the total excretion varied from 32.2% of the administered radioactivity (male urine after single high dose) to 71.0% (male faeces after single high dose). Tissues accounted for 0.35 (single low dose) to 0.87% (male single high dose). Radioactivity in tissues was higher in males than in females in the single dose groups, but did not differ in the repeated dose group.

In a second part of the study $^{14}$C-PY 1 was administrated orally to one group of five males and five females as a single oral dose of 10 mg/kg bw, and to another group of five males as a single oral dose of 100 mg/kg bw and five females as a single oral dose of 50 mg/kg bw. Urine and faeces were collected 24 and 48 hours after dosing. The urinary metabolites were qualitatively similar in males and females at all dose levels, and all of the metabolites present in the faeces were also present in the urine. The urine from males in the high-dose group was used to isolate, purify, and identify the major metabolites. The metabolites were isolated from composite urine by semi-preparative HPLC and collection of radioactive fractions. Two major and four minor metabolites were identified by chemical manipulation and mass spectrometry. The proposed metabolic pathways of PY 1 in rats are shown in Figure 2.

The distribution of the metabolites identified in Figure 2 was determined in composite urine and faeces samples collected in the first phase of the study (Selim, 1995e). The results are shown in Table 5.

The main metabolite in urine for all dosing regimens was metabolite C, chrysanthemic dicarboxylic acid (DCRA). In faeces the predominant compound was the parent, and the predominant metabolite for all dosing regimens was Metabolite E. Metabolites C and E represented over one-third of the total excreted radioactivity for all regimens in both male and female rats. Male and female rats metabolize PY 1 in a similar manner, regardless of the dose level.

Table 5. Distribution of pyrethrin 1 and metabolites in rat urine (U) and faeces (F), as a percentage of the administered dose.

| Dose group[2] | PY 1 | | Metabolite[1] | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | A | | B | | C (DCRA) | | D | | E | | F | |
| | U | F | U | F | U | F | U | F | U | F | U | F | U | F |
| SOL-M | 0.69 | 9.08 | 0.78 | ND | 1.33 | ND | 17.4 | 6.10 | 4.34 | ND | 9.54 | 15.1 | 2.01 | 4.90 |
| SOL-F | 6.88 | 5.21 | 3.25 | ND | 3.30 | ND | 16.4 | 4.99 | 5.32 | 4.10 | 5.06 | 13.7 | 1.74 | ND |
| SOH-M | 0.15 | 22.1 | 1.6 | ND | 1.56 | ND | 13.1 | 5.62 | 3.19 | 1.35 | 3.18 | 11.5 | 1.53 | 3.15 |
| SOH-F | 0.60 | 7.85 | 5.04 | ND | 5.25 | 2.52 | 14.3 | 4.07 | 3.60 | ND | 4.66 | 6.75 | 0.98 | ND |
| ROL-M | 0.78 | 6.49 | 1.24 | ND | 1.59 | ND | 17.7 | 8.03 | 4.05 | ND | 7.33 | 7.42 | 2.42 | 2.92 |
| ROL-F | ND | 4.80 | 5.92 | ND | 4.78 | 1.09 | 19.5 | 5.21 | 4.59 | 4.32 | 7.54 | 12.1 | ND | ND |

[1] See Figure 2
[2] M: male; F: female; SOL: single oral low at 10 mg/kg bw; SOH: single oral high at 50 mg/kg bw (F) or 100 mg/kg bw (M); ROL: repeat oral low, 14x 10 mg/kg bw
ND: not detected, <0.78% of administered dose
DCRA: chrysanthemic dicarboxylic acid

In a study to determine whether the metabolism of pyrethrin 1 in rats was affected by the dosing medium (Limoges, 1994) [$^{14}$C]pyrethrin 1 was administered to four pairs of male Charles River Sprague-Dawley CD®. Three pairs received single oral doses of 100 mg/kg bw in corn oil, food slurry, or dimethyl sulfoxide (DMSO). The fourth pair received 4 doses at 400 mg/kg bw over a 36-

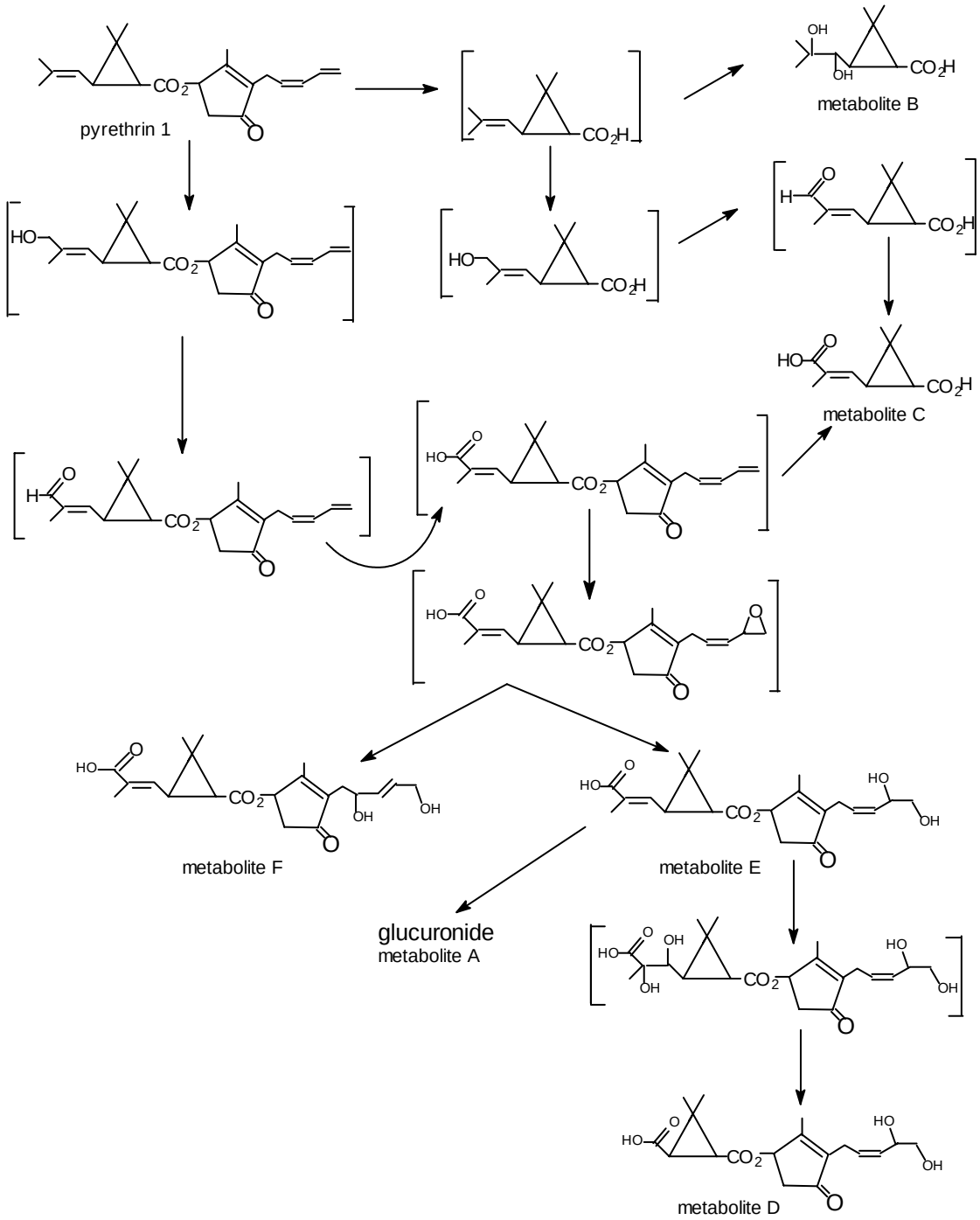
hour period in DMSO. Samples of urine and faeces collected 24, 48 and 72 hours after dosing were analysed. The results are shown in Table 6.

Table 6. Effect of dosing medium on metabolism of pyrethrin 1 in rats.

| Dosing medium | Faeces | Urine | | |
|---|---|---|---|---|
| | Total recovery of radioactivity, %[1] | Total recovery of radioactivity, %[1] | % of dosed radioactivity[2] | % of dosed radioactivity as DCRA[2] |
| Corn oil, 1 x 100 mg/kg bw | 55.0 | 29.3 | 28.4 | 13.5 |
| Corn oil, 1 x 100 mg/kg bw | 38.4 | 27.7 | 26.5 | 11.2 |
| DMSO-1 x 100 mg/kg bw | 56.6 | 33.8 | 29.8 | 12.3 |
| DMSO-4 x 400 mg/kg bw | 63.2 | 22.8 | 17.7 | 4.74 |

[1] Total of 24, 48 and 72-hour samples
[2] Individual urine samples were composited to represent >85% of the radioactivity excreted in the urine.

Figure 2. Proposed metabolic pathways of pyrethrin 1 in rats.



The vehicle used for dosing did not appreciably influence the percentage of radioactivity excreted in the urine or faeces or the percentage of the dose represented by DCRA in urine. On the other hand, administration of repeated high doses apparently decreased the proportion of the total radioactivity and of the metabolite excreted in urine.

Goats. In a trial to determine the metabolism and the radioactive residues in the milk and edible tissues of lactating goats following administration of [$^{14}$C]pyrethrin (Selim, 1995c) three goats were dosed by gavage with 7.6 and 8.3 mg/kg bw (corresponding to an expected dietary burden of 10 ppm in the feed) and 179 mg/kg bw (corresponding to an expected dietary burden of 300 ppm of $^{14}$C-PY1/kg). Two other goats were treated with a 1.8% oil- or water-base formulation applied directly to plucked or shaved skin, within an enclosure that was subsequently sealed to maximize exposure. The oral low-dose three and the dermally treated pair were dosed once a day for five days and the oral high-dose animals once a day for three days. Urine, faeces and milk were collected at 12-hour intervals from the start of the dosing period. Animals were slaughtered approximately five hours after the last dose and edible tissues were collected. Samples were extracted, analysed by HPLC and LSC (liquid scintillation counting) and the metabolites identified by GC-MS.

The high-dose group excreted up to 2.84 mg/l equivalents of PY1 in the milk after 24-36 hours (Table 7). In the low-dose group and the dermally-treated pair, the $^{14}$C in the milk reached 0.103 and 0.014 mg/l respectively, and the percentage of excreted radioactivity was higher in the urine than the faeces.

Table 7. Total radioactivity in the excreta and milk of goats treated with $^{14}$C-PY 1.

| Time (hours) | $^{14}$C, % of dose and mg/l as PY 1 | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | Oral high dose | Oral low dose | | | | Dermal water base | | | | Dermal oil base | | | |
| | Milk | Urine | Faeces | Milk | | Urine | Faeces | Milk | | Urine | Faeces | Milk | |
| | mg/l | % | % | % | mg/l | % | % | % | mg/l | % | % | % | mg/l |
| 0-12 | 1.58 | 4.90 | 0.21 | 0.12 | 0.105 | 0.25 | 0.00 | 0.01 | 0.003 | 0.57 | 0.00 | 0.02 | 0.010 |
| 12-24 | 1.07 | 5.05 | 3.41 | 0.05 | 0.044 | 0.26 | 0.10 | 0.02 | 0.005 | 0.59 | 0.03 | 0.02 | 0.010 |
| 24-36 | 2.84 | 7.77 | 3.45 | 0.11 | 0.103 | 0.38 | 0.18 | 0.01 | 0.006 | 1.02 | 0.36 | 0.02 | 0.013 |
| 36-48 | 1.39 | 3.81 | 4.48 | 0.05 | 0.043 | 0.35 | 0.22 | 0.01 | 0.005 | 0.78 | 0.16 | 0.02 | 0.012 |
| 48-60 | NA | 6.68 | 4.02 | 0.14 | 0.103 | 0.41 | 0.24 | 0.01 | 0.006 | 1.12 | 0.58 | 0.03 | 0.013 |
| 60-72 | NA | 3.55 | 4.57 | 0.06 | 0.043 | 0.37 | 0.21 | 0.02 | 0.007 | 0.83 | 0.49 | 0.02 | 0.012 |
| 72-84 | NA | 6.21 | 3.50 | 0.10 | 0.077 | 0.41 | 0.25 | 0.01 | 0.006 | 1.08 | 0.66 | 0.03 | 0.014 |
| 84-96 | NA | 4.11 | 3.57 | 0.06 | 0.048 | 0.37 | 0.25 | 0.01 | 0.006 | 0.94 | 0.58 | 0.02 | 0.013 |
| Death | NA | 3.25 | 2.36 | NA | NA | 0.24 | 0.14 | NA | NA | 0.58 | 0.55 | NA | NA |
| Total | | 45.61 | 29.57 | 0.69 | | 3.08 | 1.59 | 0.10 | | 7.55 | 3.41 | 0.18 | |

NA: not analysed

A higher concentration of $^{14}$C was found in the liver than the fat from the high oral dose, but a higher concentration in the fat than the liver from the dermal treatment (Table 8). Dermally-treated

goats retained 43.6 to 72.2% of the dose at the application site (treated skin + skin rinse).

Table 8. Distribution of total radioactive residues in tissues of goats treated with $^{14}$C-PY 1.

| | $^{14}$C, % of dose and mg/l as PY 1 | | | | | | |
|---|---|---|---|---|---|---|---|
| | Oral high dose | Oral low dose | | Dermal water base | | Dermal oil-based | |
| Sample | mg/l | % | mg/l | % | mg/l | % | mg/l |
| Fat | 3.65 | 0.39 | 0.42 | 0.06 | 0.037 | 0.19 | 0.076 |
| Liver | 7.71 | 0.47 | 0.41 | 0.02 | 0.006 | 0.08 | 0.022 |
| Kidney | 7.27 | 0.06 | 0.36 | <0.01 | 0.006 | 0.01 | 0.022 |
| Heart | NA | 0.03 | 0.11 | <0.01 | 0.002 | 0.01 | 0.017 |
| Leg muscle | 0.45 | 0.03 | 0.031 | <0.01 | 0.001 | 0.01 | 0.002 |
| Loin muscle | 0.48 | 0.03 | 0.025 | <0.01 | 0.002 | 0.01 | 0.005 |
| **Total edible tissues** | | **0.98** | | **0.08** | | **0.31** | |
| Treated skin | | | | 15.5 | | 13.0 | |
| Skin rinse | | | | 28.1 | | 59.2 | |

NA: not analysed

Samples of tissues and milk from goats after the oral and oil-based dermal treatments were analysed to identify and quantify the metabolites, using chromatographic and spectrometric methods (Table 9). In the orally-dosed goats pyrethrin 1 was the major compound, except in the kidneys, with the highest concentration in the fat, followed by the milk and liver. The metabolites were present mainly in the liver and kidneys. After dermal treatment the parent compound was found in low concentration in milk, liver and fat, and metabolites were found in liver, kidney and fat at levels below 0.005 mg/kg. In addition to the metabolites shown in Table 9, at least 17 others were observed at low levels, maximum 0.011 mg/kg.

The metabolic pathways proposed for pyrethrin 1 in goats are shown in Figure 3.

Table 9. Distribution of pyrethrin 1 and metabolites in edible products of goats dosed orally and dermally with [$^{14}$C]pyrethrin 1.

| Sample | $^{14}$C, mg/kg as PY 1 | | | | | | |
|---|---|---|---|---|---|---|---|
| | PY 1 | CRA | CRA-Glu | cC | C (DCRA) | E | G |
| | Oral high dose | | | | | | |
| Milk | 1.47 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Muscle, leg | 0.21 | 0.017 | <0.001 | 0.006 | <0.001 | <0.001 | 0.018 |
| Muscle, loin | 0.21 | 0.020 | <0.001 | 0.008 | <0.001 | <0.001 | 0.022 |
| Liver | 0.80 | 0.558 | 0.531 | 0.489 | 0.231 | 0.144 | 0.834 |
| Kidney | 0.164 | 0.369 | 3.306 | 0.078 | 0.169 | 0.829 | <0.001 |
| Fat | 2.26 | <0.001 | <0.001 | 0.073 | <0.001 | <0.001 | 0.305 |
| | Oral low dose | | | | | | |
| Milk | 0.056 | <0.001 | <0.001 | <0.001 | 0.001 | <0.001 | 0.003 |
| Muscle, leg | 0.013 | 0.003 | <0.001 | 0.001 | <0.001 | <0.001 | 0.002 |
| Muscle, loin | 0.009 | 0.002 | <0.001 | 0.001 | <0.001 | <0.001 | 0.001 |
| Liver | 0.093 | 0.025 | 0.003 | 0.033 | 0.010 | 0.009 | 0.085 |
| Kidney | 0.009 | 0.061 | 0.065 | 0.025 | 0.015 | 0.045 | 0.001 |
| Fat | 0.071 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | 0.034 |
| | Dermal oil-based | | | | | | |
| Milk | 0.010 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Muscle, leg | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Muscle, loin | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Liver | 0.002 | 0.003 | <0.001 | <0.001 | 0.001 | <0.001 | 0.003 |
| Kidney | <0.001 | <0.001 | 0.003 | <0.001 | 0.001 | 0.003 | <0.001 |

| Sample | $^{14}C$, mg/kg as PY 1 | | | | | | |
|---|---|---|---|---|---|---|---|
| | PY 1 | CRA | CRA-Glu | cC | C (DCRA) | E | G |
| Fat | 0.013 | <0.001 | <0.001 | 0.001 | <0.001 | <0.001 | 0.004 |

CRA: chrysanthemic acid; CRA-glu: glucuronic conjugate of chrysanthemic acid; DCRA: chrysanthemic dicarboxylic acid. Other metabolites are identified in Figure 3.

Hens. In a study to determine the route and rate of excretion, tissue distribution and nature of the radioactive residues, four groups of ten laying hens each were dosed with [$^{14}C$]pyrethrin 1 for 5 consecutive days (Selim, 1995d). Two groups were dosed orally by gavage at 7.66 or 475 ppm and two were treated dermally with a 1% oil- or water-based solution. The excreta and eggs were collected daily and analysed for total $^{14}C$. Tissues, eggs and excreta from the oral higher-dose group were used to identify the metabolites. The hens were killed four to six hours after the last dose and edible tissues collected for analysis.

In all treatment groups, $^{14}C$-PY1 in the eggs was found mainly in the white until the second day, and then was more concentrated in the yolk (Table 10).

Table 10. Concentrations of total $^{14}C$ in eggs of hens treated with $^{14}C$-PY1.

| Time (hours) | Oral high dose | | Oral low dose | | Oil based dermal | | Water based dermal | |
|---|---|---|---|---|---|---|---|---|
| | White | Yolk | White | Yolk | White | Yolk | White | Yolk |
| 0-24 | 0.112 | 0.026 | 0.01 | <0.001 | <0.001 | <0.001 | 0.001 | <0.001 |
| 24-48 | 0.932 | 0.484 | 0.006 | 0.002 | 0.002 | <0.001 | 0.003 | 0.003 |
| 48-72 | 0.774 | 1.55 | 0.009 | 0.017 | 0.003 | 0.010 | 0.004 | 0.018 |
| 72-96 | 0.575 | 2.723 | 0.009 | 0.031 | 0.004 | 0.023 | 0.005 | 0.032 |
| 96 (death) | 0.962 | 4.33 | 0.011 | 0.052 | 0.004 | 0.037 | 0.004 | 0.046 |

In tissues, the radioactivity in the high oral dose group in liver and fat was approximately 10 times that in muscle (Table 11). In the low oral dose and the dermal oil-based dose groups most of the radioactivity was found in excreta, and in the water-based dermal treatment group in the skin rinse. Pyrethrin 1 was poorly absorbed when applied to the skin of chickens, especially from the water-based formulation where >36% of the administered dose was found at the application site (treated skin + skin rinse).

Table 11. Total radioactivity in chickens treated with $^{14}C$-PY1.

| Sample | Oral high dose, μg/g as PY 1 | Oral low dose, % of administered dose | Dermal oil based, % of administered dose | Dermal water based, % of administered dose |
|---|---|---|---|---|
| Excreta | NA | 88.8 | 18.0 | 15.3 |
| Treated skin | NAp | NAp | 3.79 | 5.26 |
| Skin rinse | NAp | NAp | 8.29 | 31.3 |
| Liver | 15.2 | 0.342 | 0.081 | 0.063 |
| Fat | 10.2 | 0.040 | 0.193 | 0.147 |
| Breast muscle | 1.40 | 0.021 | 0.007 | 0.007 |
| Thigh muscle | 1.60 | 0.025 | 0.020 | 0.016 |
| Kidney | NA | 0.423 | 0.090 | 0.077 |
| Heart | NA | 0.066 | 0.022 | 0.030 |
| Untreated skin | NA | 0.035 | 0.049 | 0.062 |
| Gizzard | NA | 1.09 | 0.048 | 0.048 |
| Pre-formed eggs | NA | 0.076 | 0.049 | 0.047 |

NA: not analysed, NAp: not applicable

The six metabolites found in goats were also identified in the edible products of hens. The metabolic pathways proposed for goats and hens are shown on Figure 3. Table 12 shows the metabolite concentrations in edible products from the oral and oil-based dermal groups. Fat and egg yolk contained the highest concentrations of the parent compound. Liver contained the highest number of metabolites (all except G), with the highest concentrations of them in all dosed groups. Fat showed the highest concentration of the metabolite G after oral dosing. At least 19 other metabolites were observed at low levels, maximum 0.011 mg/kg.

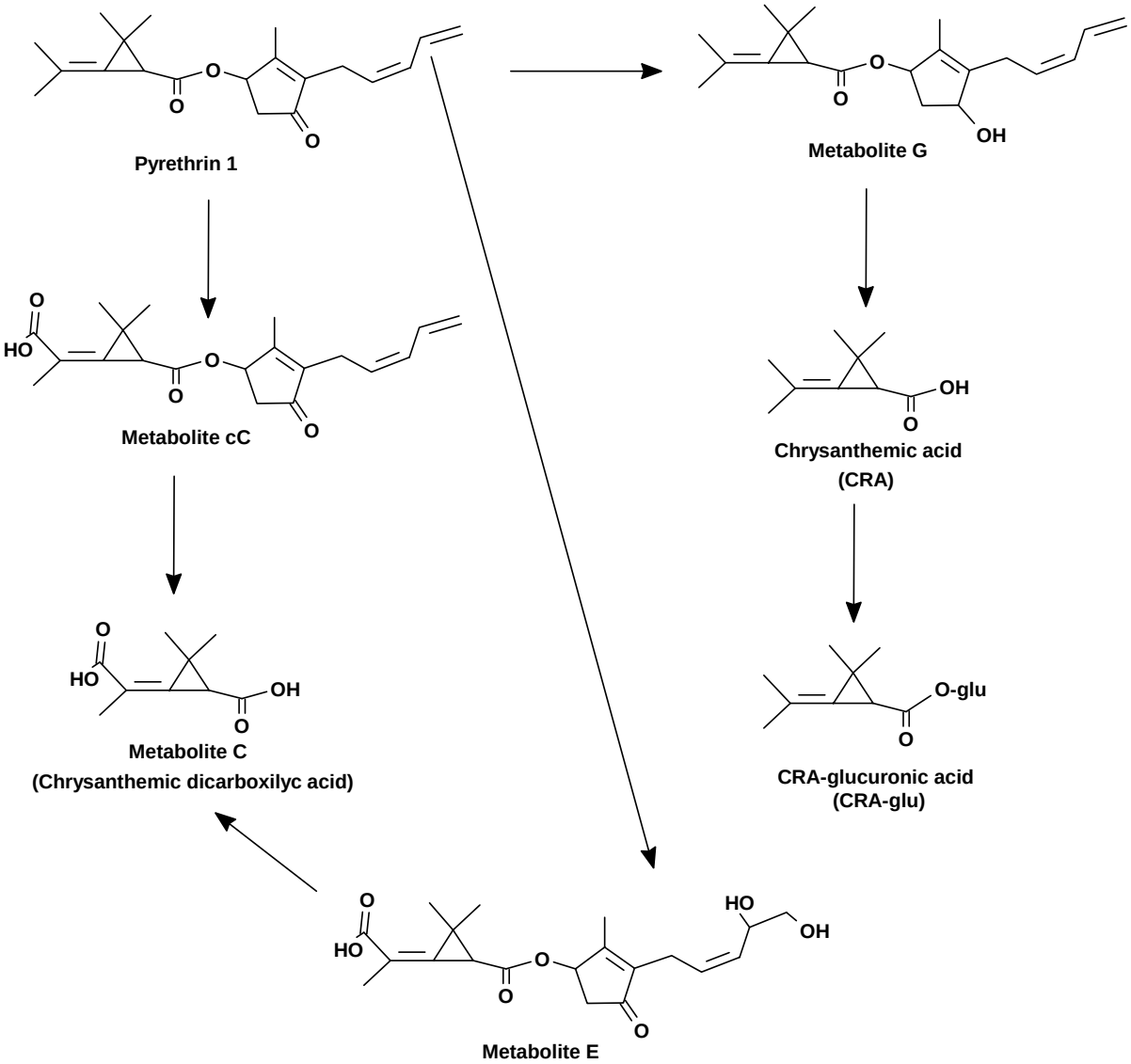



Figure 3. Proposed metabolic pathways of pyrethrin 1 in goats and hens.

Table 12. Distribution of Pyrethrin 1 and metabolites in edible products of hens dosed orally and dermally with [$^{14}$C]pyrethrin 1.

| Sample | $^{14}C$, mg/kg as PY1 | | | | | | |
|---|---|---|---|---|---|---|---|
| | PY 1 | CRA | CRA-Glu | cC | C (DCRA) | E | G |
| | Oral high dose | | | | | | |
| Egg yolk | 0.839 | 0.118 | <0.001 | 0.116 | <0.001 | <0.001 | <0.001 |
| Egg white | 0.132 | 0.269 | 0.026 | 0.122 | 0.019 | 0.029 | 0.014 |
| Breast muscle | <0.001 | 0.149 | 0.028 | 0.030 | 0.138 | 0.063 | 0.030 |
| Thigh muscle | 0.298 | 0.546 | <0.001 | 0.039 | 0.111 | 0.043 | <0.001 |
| Liver | 0.184 | 2.98 | 1.02 | 1.94 | 1.44 | 0.839 | <0.001 |
| Fat | 8.80 | 0.113 | <0.001 | 0.201 | <0.001 | <0.001 | 0.375 |
| | Oral low dose | | | | | | |
| Egg Yolk | 0.008 | 0.001 | <0.001 | 0.001 | <0.001 | <0.001 | <0.001 |
| Egg White | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Muscle | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Liver | 0.003 | 0.021 | 0.024 | 0.130 | 0.023 | 0.022 | <0.001 |
| Fat | 0.018 | 0.002 | <0.001 | 0.002 | <0.001 | <0.001 | 0.002 |
| | Dermal oil-base | | | | | | |
| Egg Yolk | 0.035 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Egg White | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Breast Muscle | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |
| Thigh Muscle | 0.006 | 0.002 | <0.001 | 0.001 | <0.001 | <0.001 | <0.001 |
| Liver | 0.004 | 0.029 | 0.001 | 0.011 | 0.003 | 0.004 | <0.001 |
| Fat | 0.156 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 | <0.001 |

CRA: chrysanthemic acid; CRA-glu: glucuronic conjugate of chrysanthemic acid; DCRA: chrysanthemic dicaroxylic acid

## Metabolism in plants

The fate of radiolabelled pyrethrin 1 ($^{14}C$-PY) following foliar application has been investigated in leaf lettuce (Selim, 1994a), potatoes (Selim, 1995a), and tomatoes (Selim, 1995b). The plants were dosed with a typical end-use formulation at 0.56 kg ai/ha, 10 times the maximum label rate. Five applications were made in all cases, at intervals of five days for tomatoes, seven days for lettuce, and fifteen days for potatoes. Tomato leaves and fruit, and potato leaves and tubers were collected five days after the last application. Lettuce leaves were collected separately on the day of the last application and ten days later. After extraction, aqueous, organic, and solid fractions were analysed by a combination of liquid scintillation counting (LSC) and HPLC, and metabolites were identified by GC-MS.

Pyrethrin 1 was degraded extensively, giving rise to at least 8 and as many as 19 extractable degradation products in the three crops investigated. Degradation followed similar pathways in all the crops. All identified products were chrysanthemic acid derivatives. Figure 4 shows the proposed degradation pathways.

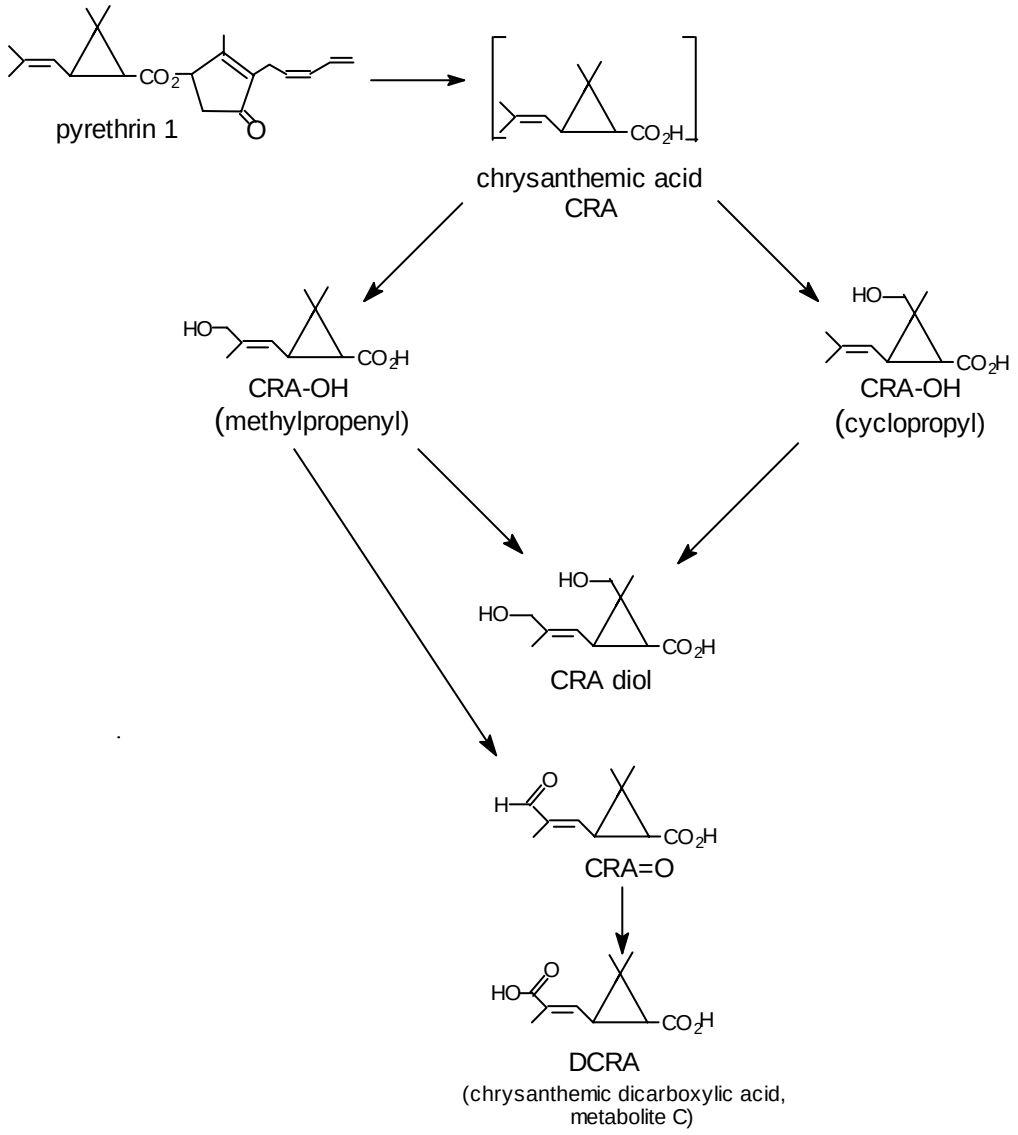



Figure 4. Proposed degradation pathways of pyrethrin 1 in plants

Table 13 shows the levels at which pyrethrin 1 and five major metabolites were found in the treated crops. The level of total radioactivity in tomato fruit was only 0.5% of that in the leaves, and in potato tubers only 0.1% of that in the leaves. The levels of Pyrethrin 1 in tomato fruit and potato tubers were 6% and 0.03% respectively of the levels in the leaves.

Table 13. Levels of pyrethrin 1 and identified metabolites in treated plants.

| Compounds | $^{14}$C, mg/kg, as PY 1 | | | | | |
|---|---|---|---|---|---|---|
| | Potato | | Tomato | | Lettuce | |
| | Leaf | Tuber | Leaf | Fruit | 0-day PHI | 10-day PHI |
| TRR | 550 | 0.564 | 365 | 1.86 | 35.6 | 10.2 |
| Pyrethrin 1 | 13.5 | 0.004 | 4.29 | 0.245 | 14.1 | 0.246 |

| Compounds | $^{14}C$, mg/kg, as PY 1 | | | | | |
|---|---|---|---|---|---|---|
| | Potato | | Tomato | | Lettuce | |
| | Leaf | Tuber | Leaf | Fruit | 0-day PHI | 10-day PHI |
| CRA-OH (methylpropenyl) | 4.93 | 0.018 | 7.83 | <0.004 | <0.004 | <0.004 |
| CRA-OH (cyclopropyl) | <0.004 | 0.025 | 22.5 | 0.419 | 1.50 | 2.10 |
| CRA=O | 10.3 | <0.004 | <0.004 | <0.004 | <0.004 | <0.004 |
| CRA diol | 2.28 | 0.19 | 8.35 | 0.260 | 1.00 | 0.623 |
| DCRA | <0.004 | <0.004 | <0.004 | <0.004 | 2.45 | 0.552 |

Degradation of pyrethrins on leaf surfaces exposed to sunlight occurs quickly. On potato and tomato leaves collected five days after application less than 3% of the total radioactive residue remained as pyrethrin 1. In lettuce collected immediately after the last spray had dried only 39.4% of the total radioactivity was present as the parent compound, and after 10 days the proportion was 2.4%. The rapid degradation on the plant surfaces indicates photolysis rather than metabolism by the plant. Photolysis occurs so rapidly and extensively that transport of pyrethrins into the plant where metabolism would occur is minimal.

## Environmental fate in soil

Photolysis. Two sandy loam soils treated with [$^{14}C$]pyrethrin 1 at a nominal concentration of 10 mg/kg were exposed to natural sunlight at 24°C for up to 24 hours with treated soil stored in the dark as a control (Testman, 1994). Exposed and control samples were collected after 0, 1, 2, 4 and 24 hours and pyrethrin levels determined by HPLC analysis of ethyl acetate extracts. Acidic volatiles were collected in a KOH trapping system. A mean recovery of radioactivity above 96% was demonstrated by liquid scintillation counting (LSC).

The half-life of pyrethrin 1 on irradiated soil was 12.9 hours, with the formation of numerous degradation products. No single product represented more than 10% of the applied radioactivity, so products other than carbon dioxide were not identified. On control soil pyrethrin 1 was degraded with a half-life of 82.9 hours.

Aerobic degradation. The degradation of [$^{14}C$]pyrethrin 1 was studied in a sandy loam soil treated at approximately 1.0 mg/kg and incubated for up to 181 days under aerobic conditions at 25 ± 1°C in the dark (Robinson, 1994). Acidic volatiles were collected in a KOH trapping system. A mean recovery of radioactivity above 91% was demonstrated by LSC. Soil extracts were analysed by HPLC. The results are shown in Table 14.

Table 14. Average distribution of radioactivity in extract fractions of soil treated with $^{14}C$-PY.

| Fraction | $^{14}C$, mean % of total applied, after interval (days) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 0.5 | 1 | 2 | 3 | 7 | 14 | 30 | 59 | 121 | 181 |
| KOH | NAp | 0.32 | 1.03 | 3.23 | 4.99 | 12.3 | 22.4 | 31.8 | 37.6 | 41.4 | 41.4 |
| Post-extraction solids (PES) | 2.40 | 11.4 | 12.3 | 17.2 | 23.5 | 36.9 | 40.0 | 40.0 | 38.6 | 35.1 | 34.0 |
| Aqueous | 0.07 | 0.28 | 0.42 | 0.70 | 1.12 | 2.52 | 2.44 | 1.91 | 2.01 | 1.31 | 1.70 |
| $CH_3CN/H_2O$ | 91.2 | 81.9 | 83.8 | 71.7 | 61.1 | 39.9 | 29.9 | 20.9 | 15.0 | 8.45 | 9.14 |
| $CH_3CN/CH_2Cl_2$ | 91.1 | 81.6 | 83.4 | 71.0 | 59.9 | 37.4 | 27.5 | 19.0 | 13.0 | 7.14 | 7.44 |
| Pyrethrin[1] | 85.4 | 65.3 | 65.4 | 48.2 | 15.6 | 11.9 | 4.02 | 2.08 | 0.52 | NA | NA |

| Fraction | $^{14}C$, mean % of total applied, after interval (days) | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 0.5 | 1 | 2 | 3 | 7 | 14 | 30 | 59 | 121 | 181 |
| Chrysanthemic acid[1] | 1.11 | 2.67 | 2.43 | 2.48 | 4.01 | 1.78 | 0.87 | 0.40 | 0.27 | NA | NA |

[1] In $CH_3CN/CH_2Cl_2$ fraction
NAp: not applicable; NA: not analysed

Pyrethrin 1 was degraded in soil with a half-life of about 2.2 days (calculated from 0 to 14 days). During incubation $^{14}C$ in the $CH_3CN/H_2O$ fraction steadily decreased, the bound $^{14}C$ (PES fraction) increased for 30 days and then decreased, and the amount of evolved $CO_2$ (KOH fraction) steadily increased. This pattern suggests that the extractable species, including pyrethrin 1, are converted to bound residues which are then degraded to $CO_2$.

Pyrethrin 1 and chrysanthemic acid were identified in $CH_3CN/CH_2Cl_2$ extracts of the treated soil (Table 14). Chrysanthemic acid reached a maximum concentration of about 4% of the initial pyrethrin 1 concentration on the 3rd day. Three other compounds at levels from 5 to 10% of the applied pyrethrin 1 and numerous additional products at levels below 5% of the initial pyrethrin 1 concentration were observed.

Fractionation of the post-extraction solids showed that about 10%, 12%, and 6% of the applied radioactivity was present in the humic acid, fulvic acid, and humin fractions respectively. Chrysanthemic acid was isolated from extracts of the fulvic acid fraction.

Terrestrial dissipation studies were conducted under worst-case conditions at three geographically distinct sites, Georgia, California, and Michigan, USA (Hattermann, 1992a,b). At each site a single application of a typical end-use formulation (pyrethrum + piperonyl butoxide) was made at a nominal rate of 0.52 kg ai/ha to bare soil. This rate is ten times the proposed maximum single-application rate, and represents the total amount of pyrethrins that could possibly be applied in a full season.

Before application, petri dishes 1.1 cm in depth were put in place with the top at the level of the soil surface, and were filled with soil from the site. At intervals up to one hour after application, plates were removed, covered, sealed, and chilled. Thereafter, soil cores were collected to a depth of 91 cm on a diagonal transect across the plots using a hydraulic probe with an acetate liner, beginning 24 hours after application and continuing for 97 to 179 days.

Soil samples were analysed by gas chromatography with electron capture detection after extraction and clean-up, allowing quantification of each of the three chrysanthemic esters. As the pyrethric esters represented 81.51% of the chrysanthemic, the concentrations of the latter were multiplied by 1.8151 to obtain the corresponding concentrations of total pyrethrins.

Half-lives, determined from pyrethrin residue levels at 5, 10, 20 and 40 min after application (Table 15), were one hour in Georgia and California and two hours in Michigan. At each site pyrethrin in the 0–15 cm soil layer was below the limit of detection, 0.10 mg/kg for total pyrethrins within one day of application. Pyrethrin was never detected at any site in soil collected at depths below 15 cm.

Table 15. Concentration of pyrethrins in petri dish soil samples following pyrethrum application.

| Nominal time after treatment, min | Pyrethrins found, μg/g | | | Total pyrethrins expected, μg/g | | |
|---|---|---|---|---|---|---|
| | California | Georgia | Michigan | California | Georgia | Michigan |
| 5 | 0.85 | 0.90 | 1.39 | 1.55 | 1.62 | 2.53 |
| 10 | 1.20 | 1.20 | 1.47 | 2.18 | 2.18 | 2.67 |

| | | | | | | |
|---|---|---|---|---|---|---|
| 20 | 0.94 | 0.82 | 1.38 | 1.71 | 1.49 | 2.50 |
| 40 | 0.69 | 0.64 | 1.15 | 1.21 | 1.15 | 2.08 |
| **Half life (hours)** | **0.96** | **0.96** | **1.92** | | | |

Volatility. The volatility of [$^{14}$C]pyrethrin 1 was studied in sandy loam soil at 50% and 75% of field moisture capacity and two air flow rates, 100 and 300 ml/min, with each of these four combinations in duplicate (Selim, 1994b). Pyrethrin 1, in a typical end-use formulation, was applied at a rate equivalent to 0.56 kg ai/ha, approximately 10 times the maximum label rate, and the system was incubated for 30 days. Air was passed over the soil and then through a series of traps (ethylene glycol and KOH) designed to capture organics and $CO_2$. The solutions from each trap were collected on days 1, 2, 3, 6, 9, 13, 16, 20, 23, 27 and 30. The trap contents and soil extracts were analysed by chromatographic and chemical methods. The recovery of total radioactivity was above 90% for each test system.

After 30 days most of the radioactivity remained with the soil, with approximately half of this extractable and half remaining bound. [$^{14}$C]pyrethrin 1 in soil extracts represented up to 9.1% of the applied radioactivity (Table 16). Four organosoluble degradation products were observed in the extracts.

The radioactivity trapped in the potassium hydroxide was determined to be mainly from $CO_2$ (Table 16), and that trapped in the ethylene glycol mainly from [$^{14}$C]chrysanthemic acid in all but one test system, together with two other organic products and low levels of pyrethrin 1.

Table 16. Radioactivity in soil extracts and trapping solutions.

| Sample | $^{14}$C, % of applied | | | |
|---|---|---|---|---|
| | 100 ml/min | | 300 ml/min | |
| | 50% moisture | 75% moisture | 50% moisture | 75% moisture |
| Soil extract | 42.6 | 43.4 | 39.7 | 36.8 |
| Pyrethrin 1 | 9.06 | 2.75 | 4.31 | 1.94 |
| Degradation products (4) | 4.84 | 13.7 | 13.6 | 16.80 |
| Soil after extraction | 35.4 | 33.1 | 36.1 | 37.8 |
| KOH trap | 5.24 | 8.28 | 5.57 | 8.30 |
| Ethylene glycol trap | 5.48 | 4.38 | 8.60 | 7.86 |
| Pyrethrin 1[1] | 0.038 | <0.05 | 0.14 | 0.19 |
| Degradation products (3)[1] | 5.58 | 4.52 | 8.70 | 8.23 |

[1] 10% of each ethylene glycol trap solution was composited and analysed for pyrethrin 1 and degradation products

Volatility rates were calculated from the radioactivity in the trapping solutions, assuming a value of 0.025% for pyrethrin 1 at 75% moisture and 100 ml/min. The volatility of all volatile species was 0.001 µg/cm$^2$/hour under all conditions. Volatility of pyrethrin 1 *per se* was 3.5 and 2.0 x $10^{-6}$ µg/cm$^2$/hour for 50 and 75% moisture respectively at 100 ml/min air flow, and 9.9 and 12 x $10^{-6}$ µg/cm$^2$/hour for 50 and 75% moisture respectively at 300 ml/min air flow.

Adsorption/desorption. The adsorption and desorption of [$^{14}$C]pyrethrin 1 were studied at concentrations of 0.05 mg/kg, 0.09 mg/kg, 0.50 mg/kg, and 0.81 mg/kg in sandy loam, silty clay loam, silt loam, and sand (Robinson and Reynolds, 1994). A 1:100 soil-to-solution ratio was used, and the equilibration time for both adsorption and desorption was three hours. In the adsorption experiment, two 50 ml glass centrifuge tubes containing ~0.3 g of soil were used for each soil type, with 2 positive controls containing treated 0.01 M $CaCl_2$ solution and 2 negative controls containing both soil and untreated 0.01 M $CaCl_2$ solution. The tubes were then shaken for 3 hours, centrifuged

for 10 min and duplicate aliquots of each supernatant removed for liquid scintillation counting (LSC). Upon completion of the adsorption procedure, 30 ml of fresh untreated 0.01 M $CaCl_2$ solution was added to the tubes, which were again shaken for 3 hours and centrifuged for 10 min. Duplicate samples of supernatant were removed for analysis by LSC. The mean recoveries of $^{14}C$ were more than 95% for all the soils.

HPLC analysis of the organic extracts of the adsorption and desorption supernatants and the desorption solids at the highest concentration showed pyrethrin as the major product. Low levels of chrysanthemic acid were also detected (<5.2% of radioactivity). The adsorption and desorption constants are shown in Table 17.

Table 17. Adsorption and desorption K values of pyrethrin 1.

| Soil | Adsorption | | Desorption | | Mobility class [1] |
|---|---|---|---|---|---|
| | $K_{ads}$ | $K_{OC}$ | $K_{des}$ | $K_{OC}$ | |
| Sandy loam | 268 | 12472 | 2332 | 108679 | Immobile |
| Silty clay loam | 310 | 16190 | 1151 | 60133 | Immobile |
| Silt loam | 430 | 74175 | 2600 | 448257 | Immobile |
| Sand | 198 | 37847 | 965 | 184767 | Immobile |

[1] Immobile: $K_{OC} > 5000$.

**Environmental fate in water and water/sediment systems**

Photolysis. The aqueous photolysis of [$^{14}C$]pyrethrin 1 was investigated at pH 7 at a nominal concentration of about 0.3 mg/kg, slightly below half the solubility (Selim, 1995g). Test solutions were exposed to natural sunlight for up to 72 hours at 25 ± 1°C. The mean recovery of radioactivity was above 97%. Within one hour of exposure, the level of pyrethrin 1 had decreased by about 50%, and approximately the same level of its *E*- isomer had formed (Table 18). Subsequently, the sum of the concentrations of the isomers decreased rapidly, following first-order kinetics. The overall photolytic half-life for the total of the two isomers was 11.8 hours. [$^{14}C$]pyrethrin 1 in the unexposed system was stable throughout the test period.

Table 18. Aqueous photolysis of [$^{14}C$]pyrethrin 1 in natural sunlight.

| Compound | $^{14}C$, % of initial, after exposure (hours) | | | | | | | Half-life, hours |
|---|---|---|---|---|---|---|---|---|
| | 0 | 1 | 2 | 4 | 8 | 20 | 30 | |
| Pyrethrin 1 | 96.0 | 46.6 | 38.7 | 27.3 | 19.7 | 8.4 | 7.0 | 9.2[1] |
| (*E*)- isomer | 3.6 | 44.2 | 53.2 | 48.9 | 35.4 | 15.8 | 11.9 | 11.8[2] |

[1] of PY1
[2] of PY1 + (*E*)- isomer

Hydrolysis in buffer. The hydrolysis of [$^{14}$C]pyrethrin 1 was investigated in aqueous buffers at pH 5, 7, and 9 at a nominal concentration of 0.4 mg/kg, approximately half the solubility (Selim, 1995f). The samples were wrapped in aluminium foil, incubated at 25°C, extracted after 0, 1, 3, 7, 14, 21 and 30 days and analysed by HPLC. The mean recovery of radioactivity at each pH was above 95%, determined by liquid scintillation counting. Pyrethrin 1 was stable at pH 5 and 7, showing about 5% degradation after 30 days. At pH 9 it was degraded with a half-life of 17 days, with approximately 35% of the parent compound remaining after 30 days.

Chrysanthemic acid was identified as the single main radioactive degradation product. Its concentration at pH 9 increased in proportion to the decrease of pyrethrin 1, accounting for 61% of the applied radioactivity after 30 days.

In order to isolate and identify unlabelled products arising from the alcohol portion of the pyrethrin 1 molecule (pyrethrolone), 2 l of an aqueous solution of [$^{14}$C]pyrethrin 1 at pH 9 was incubated for 30 days. A single unlabelled compound, a dimer of molecular weight 320, was isolated by semi-preparative HPLC and analysed by mass spectrometry. The proposed pathway for its formation is shown in Figure 5.

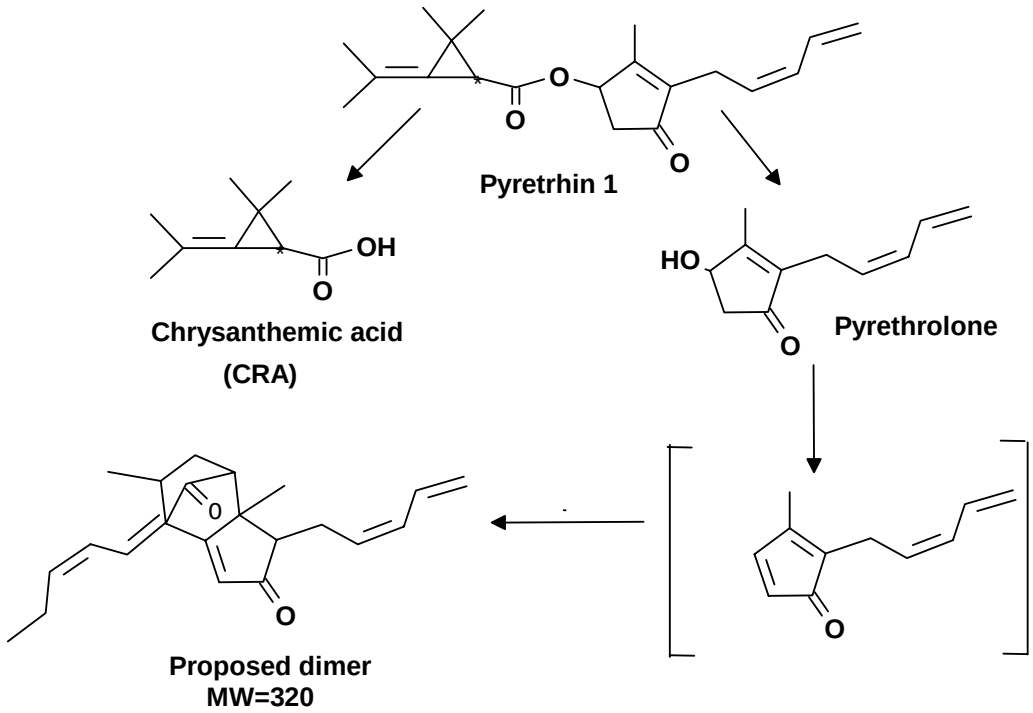



Figure 5. Proposed hydrolytic pathways of pyrethrin 1 at pH 9.

Degradation in water/sediment systems. The degradation of [$^{14}$C]pyrethrin 1 was studied at 25 ± 1°C in the dark in anaerobic and aerobic aquatic systems prepared from sandy loam hydrosoil mixed with pond water (Robinson and Wisocky, 1994a,b). The treatment rate was approximately 1 mg/kg, and incubation proceeded for 364 days under anaerobic and for 30 days under aerobic conditions. Volatiles were collected in a KOH trapping system. The mean recovery of radioactivity was above 95%. The hydrosoil and supernatant were extracted and analysed by HPLC.

Under anaerobic conditions pyrethrin 1 was degraded with a half-life of about 86 days. Table 18 shows the distribution of radioactivity at intervals after application. The radioactivity in the supernatant increased during incubation, and was mainly found in the organosoluble fraction during the first six months of the study. In soil the radioactivity in the organosoluble fraction decreased as the bound residues and $CO_2$ increased.

Under anaerobic conditions the main extractable compounds were pyrethrin 1, chrysanthemic acid, cyclopropane diacid, and jasmolin 1, whose structures are shown in Figure 6. Seventeen other degradation products, none of which represented more than 5% of the applied [$^{14}$C], were found at various intervals. The distribution of the main extractable compounds is shown in Table 20.

Table 19. Distribution of radioactivity in an anaerobic water/sediment system treated with [$^{14}$C]pyrethrin 1.

| Phase | Fraction | $^{14}$C, % of initial | | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | Day 0 | Day 14 | Day 31 | Day 90 | Day 180 | Day 270 | Day 364 |
| Supernatant | Total | 3.0 | 7.6 | 10.4 | 17.7 | 9.88 | 26.1 | 24.3 |
| | Organosoluble | 2.9 | 6.87 | 9.0 | 12.12 | 8.27 | 7.2 | 7.18 |
| Soil | Total [1] | 97.0 | 92.3 | 89.5 | 80.3 | 89.6 | 61.9 | 62.2 |
| | Organosoluble | 97.5 | 71.0 | 55.0 | 45.3 | 33.8 | 18.4 | 15.8 |
| | Bound | 2.35 | 19.9 | 30.9 | 40.7 | 50.3 | 40.4 | 46.2 |
| Volatiles | $CO_2$ | NAp | 0.13 | 0.07 | 1.98 | 0.57 | 12.0 | 13.5 |

[1] By difference, 100-(% in total supernatant + % as $CO_2$)
NAp: not applicable

Table 20. Distribution of pyrethrin 1 and main degradation products in organosoluble fractions of supernatant and sediment under anaerobic conditions.

| Time (days) | 14C, % of initial as | | | |
|---|---|---|---|---|
| | Pyrethrin 1 | Cyclopropane diacid | Chrysanthemic acid | Jasmolin 1 |
| 0 | 97.5 | <1 | <1 | <1 |
| 14 | 62.5 | <1 | 9.15 | <1 |
| 31 | 45.3 | <1 | 12.6 | <1 |
| 90 | 34.7 | <1 | 10.8 | 9.91 |
| 180 | 20.8 | <1 | 9.84 | 10.0 |
| 270 | 6.12 | 10.6 | 9.59 | 9.40 |
| 364 | 3.72 | 14.6 | 5.34 | 9.72 |

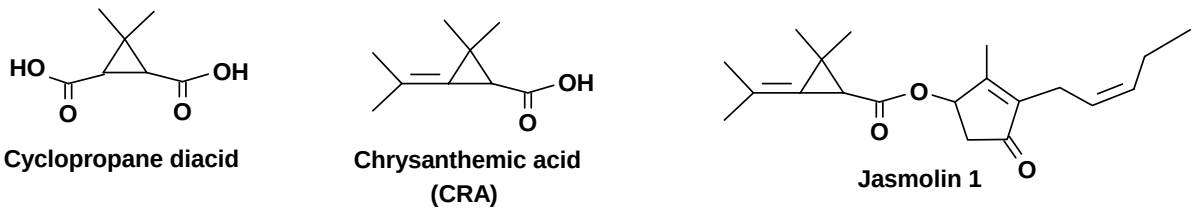



Figure 6. Structures of degradation products in an anaerobic water/sediment system.

Soil fractionation of the remaining bound residues yielded about 5.5%, 17%, and 14.9% of the applied radioactivity as humic acid, fulvic acid, and humin fractions respectively. Chrysanthemic acid was present in the extract of the fulvic acid fraction.

Under aerobic conditions, pyrethrin was degraded with a half-life of about 10.5 days. Table 21 shows the distribution of radioactivity at intervals after application. In comparison with the anaerobic system in the same period (Table 19), higher levels of radioactivity were found in the supernatant and as volatiles. In the soil, in contrast to the anaerobic system, most of radioactivity was bound.

Table 21. Distribution of radioactivity in an aerobic water/sediment system treated with [$^{14}$C]pyrethrin 1.

| Phase | Fraction | $^{14}$C, % of initial | | | | | |
|---|---|---|---|---|---|---|---|
| | | Day 0 | Day 3 | Day 7 | Day 14 | Day 21 | Day 30 |
| Supernatant | Total | 8.17 | 7.26 | 13.8 | 16.0 | 19.5 | 20.7 |
| | Organosoluble | 7.63 | 6.36 | 11.3 | 12.0 | 14.0 | 13.7 |
| Soil | Total [1] | 91.8 | 91.8 | 84.8 | 82.4 | 78.4 | 75.2 |
| | Organosoluble | 98.3 | 75.3 | 57.0 | 36.9 | 27.5 | 21.7 |
| | Bound | 1.21 | 15.76 | 29.8 | 42.6 | 46.9 | 51.2 |
| Volatiles | $CO_2$ | NAp | 0.98 | 1.39 | 1.58 | 2.07 | 4.12 |

[1]By difference, 100 -(% in total supernatant + % in $CO_2$)
NAp: Not applicable.

After 3 days incubation, pyrethrin and chrysanthemic acid accounted for 72.4 and 2.5% of the applied radioactivity respectively. After 30 days pyrethrin had decreased to 14.7% of the administered dose, and after 21 days chrysanthemic acid reached a maximum of 21.9%, decreasing to 18.6% after 30 days. Three additional degradation products were detected at various intervals, two only once, but none exceeded 5% of the initial concentration of pyrethrin 1. The third product was found only at the final three intervals and was below 3% of the initial concentration of pyrethrin 1 at day 30.

About 6.0%, 23.4%, and 10.3% of the applied radioactivity was present in the humic acid, fulvic acid, and humin fractions respectively. Chrysanthemic acid was detected in the fulvic acid fraction.

## METHODS OF RESIDUE ANALYSIS

### Analytical methods

Sample extracts are analysed for pyrethrins by gas chromatography with electron capture (EC) detection, allowing quantification of the pyrethrins I esters, pyrethrin 1, cinerin 1 and jasmolin 1, which are individually determined. No adjustment is made for the different responses to the compounds of the EC detector (Pyrethrin 1 produces the highest response), nor for the differing molecular weights. Pyrethrins II esters are thermolabile and degraded in the inlet of the gas chromatograph so total pyrethrin levels are estimated from the proportion of pyrethrins I and pyrethrins II in the formulation. In the formulation used for the trials on agricultural commodities the pyrethrins II content was 81.51% of pyrethrins I, so pyrethrins I concentrations were multiplied by 1.8151 to obtain the corresponding concentrations of total pyrethrins. In the formulation used for the animal feeding trials pyrethrins II were 92.0% of pyrethrins I, so pyrethrins I concentrations were multiplied by 1.92 to give the concentrations of total pyrethrins. A World Standard Pyrethrum Extract is available for calibration (Odinga, unknown year).

Raw and processed agricultural commodities from field trials were determined by EN-CAS Method No. ENC-14/93. Samples are extracted with an organic solvent, and cleaned up by silica gel adsorption. Some samples require a second adsorptive clean-up on alumina. The limit of

quantification (LOQ) for total pyrethrins was 0.036 mg/kg in all samples. Controls contained no appreciable interferants. Recoveries of pyrethrins I ranged from 61 to 139%, with a mean of 93%.

A second method was used to determine residues of pyrethrins in food items from the trials of warehouse treatments. Low-fat foods (navy beans and prunes) were extracted with an organic solvent and water, and high-fat foods (peanuts) with an organic solvent. After clean-up on a liquid-solid partition column, the eluate was analysed by gas chromatography with electron-capture detection. The limit of quantification for pyrethrins I was 0.1 mg/kg, and the limit of detection 0.05 mg/kg. The method was validated for each substrate by analysis of at least four fortified samples. Reported recoveries ranged from 65% to 120%.

A third method was used to analyse edible products of laying hens and dairy cattle, and is also applicable for enforcement of pyrethrin tolerances in animal-derived commodities. Samples are extracted with an organic solvent and cleaned up by silica gel adsorption. Liver, kidney, skin, muscle, eggs, and fat require a second adsorptive clean-up on alumina. Limits of quantification (LOQ) as total pyrethrins were 0.019 mg/kg for milk and eggs and 0.038 mg/kg for all animal tissues. Recoveries ranged from 67 to 112% at 1, 10 and 100 times the LOQ, with standard deviations of 1.5 to 10%.

### Stability of pesticide residues in stored analytical samples

The stability of pyrethrins in raw and processed commodities was determined by analyses of samples fortified at 1 mg/kg pyrethrins I stored up to 24 months under the same frozen conditions as the samples from the field and processing studies. Table 22 shows the percentage of the initial residue remaining at the last sampling time.

Table 22. Storage stability of pyrethrins in raw and processed agricultural commodities fortified with 1 mg/kg pyrethrins I.

| Commodity | Storage period (months) | % of initial residue remaining |
|---|---|---|
| Leaf lettuce | 24 | 79, 83 |
| Broccoli | 24 | 35, 40 |
| Succulent bean pods | 24 | 69, 54 |
| Succulent bean vine | 24 | 48, 62 |
| Succulent bean hay | 24 | 59, 52 |
| Beans | 12 | 80, 107 |
| Cannery waste | 3 | 93 |
| Cucumber | 24 | 94, 79 |
| Orange | 24 | 87, 93 |
| Orange oil | 24 | 90, 82 |
| Orange molasses | 24 | 84, 87 |
| Orange juice | 27 | 91, 90 |
| Orange dry pulp | 27 | 48, 46 |
| Grape | 24 | 66, 68 |
| Potato granule | 24 | 53, 85 |
| Potato chips | 24 | 77, 95 |
| Potato wet peel | 24 | 61, 77 |
| Tomato | 27 | 89, 90 |
| Tomato juice | 27 | 93, 96 |
| Tomato purée | 26 | 93, 96 |
| Tomato wet pomace | 25 | 70, 62 |
| Tomato dry pomace | 12 | 65, 69 |
| Peanuts | 12 | 65, 85 |
| Prunes | 12 | 67, 106 |
| Cattle milk, muscle and fat; hens eggs and skin | 12 | 75, 112 |

| Commodity | Storage period (months) | % of initial residue remaining |
|---|---|---|
| Cattle liver | 6 | 47 |
| Cattle kidney | 10 | 46 |

**Definition of the residue**

In a study of pyrethrin 1 metabolism by lettuce pyrethrin 1 was the main residue on the day of application, and chrysanthemic dicarboxylic acid was the only degradation product exceeding 10% of the pyrethrin residue, at 17%. Pyrethrin 1 is degraded extensively by photolysis on plants within 10 days after application, with no predominant product formed. The Meeting agreed that the definition of the residue for compliance with MRLs and for dietary intake estimation in plants should continue to be total pyrethrins, calculated as the sum of the six biologically-active pyrethrin components pyrethrin 1, pyrethrin 2, cinerin 1, cinerin 2, jasmolin 1 and jasmolin 2 determined after calibration with the World Standard Pyrethrum Extract. This standard can be obtained from the Pyrethrum Board of Kenya.

Pyrethrin 1 and pyrethrin 2 have log $P_{ow}$s of 5.9 and 4.3 respectively, which indicates fat-solubility. This is confirmed by the metabolism studies on animals, where the residue concentration of pyrethrin 1 in fat was significantly higher than in other tissues.

**USE PATTERN**

Table 23 summarizes GAP for the use pesticides that contain pyrethrins as active ingredients in the crops for which residue data were presented.

Table 23. Registered uses of pyrethrins from product labels.

| Crop | Country | Form, ai | Application [1] | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Spray conc., kg ai/hl | Rate, kg ai/ha | No. | |
| Beans | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| | | EC | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.00375 | NS | NS | 2 |
| Berries | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| | Germany | Liquid | Spray, broadcast | 0.04 | NS | 3 max. | 2 |
| | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Berries (except strawberries) | Germany | NS | Spray, broadcast | 0.0017 | NS | NS | 2 |
| Brassica (cole) leafy vegetables | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Brassica plants | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| Broad beans | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| Bulb vegetables | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Bush-beans, etc. | Germany | DP | Spray, broadcast | NS | 0.075 | NS | 1 |
| Bush-beans | Germany | DP | Dusting | NS | 0.075 | NS | NS |
| Cabbage | Italy | EC | Spray, broadcast | 0.0032 to 0.012 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.0375 | NS | NS | 2 |
| | Germany | DP | Spray, broadcast | NS | 0.075 | NS | 3 |
| Carrots | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| | | EC | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.00375 | NS | NS | 2 |
| Cauliflower | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| Citrus fruits | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Citrus | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| | | EC | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.00375 | NS | NS | 2 |
| Citrus fruits | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| Cattle | Costa Rica | NS | Direct App. | 0.01 | NA | NS | NA |

| Crop | Country | Form, ai | Application [1] | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Spray conc., kg ai/hl | Rate, kg ai/ha | No. | |
| Crops | New Zealand | EC | Spray, broadcast | 0.03 | NS | NS | 1 |
| Cucumbers | Germany | DP | Spray, broadcast | NS | 0.075 | NS | 2 |
| Cucurbit vegetables | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Dairies | Costa Rica | NS | Space Spray | 0.094 | NA | NS | NA |
| Eggplant | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| Field beans | Italy | EC | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.00375 | NS | NS | 2 |
| Fruit trees | Australia | NS | Spray, broadcast | 0.005 to 0.08 | NS | NS | 1 |
| Fruiting vegetables | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| Fruits (except strawberries) | Germany | NS | Spray, broadcast | 0.0036 | NS | 4 max. | 2 |
| Fruits | Australia | XX | Spray, broadcast | 0.0078 | NS | NS | 1 |
| | Netherlands | Liquid | Spray, broadcast | 0.004 | NS | NS | 2 |
| General crops | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| Goats | Costa Rica | NS | Direct App. | 0.01 | NA | NS | NA |
| Grapevine | Australia | NS | Spray, broadcast | 0.010 | NS | NS | 1 |
| | Italy | EC | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.00375 | NS | NS | 2 |
| Harvested fruits and vegetables | USA | NS | Space spray | NS | NS | NS | NA |
| Haricots (green beans) | Italy | EC | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.00375 | NS | NS | 2 |
| Harvested fruits | Costa Rica | NS | Direct Spray | 0.005 | NS | NS | NA |
| Horticultural crops | Italy | NS | Spray, broadcast | 0.0037 | NS | NS | 2 |
| Kohlrabi | Germany | DP | Spray, broadcast | NS | 0.075 | NS | 3 |
| Leaf vegetables | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Legume vegetables, leaves of | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Legume vegetables | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Legume vegetables, including leaves | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| Legumes | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| Lettuce | Germany | DP | Spray, broadcast | NS | 0.075 | NS | 3 |
| | | DP | Dusting | NS | 0.075 | NS | NS |
| | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| | | EC | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.00375 | NS | NS | 2 |
| Livestock quarters and dairies | USA | NS | Space Spray | 0.062 to 0.125 | NS | NS | NA |
| Livestock and poultry | Costa Rica | NS | Direct App. | 0.05 to 0.1 | NA | NS | NA |
| Livestock | Costa Rica | NS | Direct App. | 0.018 | NA | NS | NA |
| Livestock and poultry | USA | NS | Direct app. | 0.1 | NS | NS | NA |
| Milk rooms | Costa Rica | NS | Space Spray | 0.094 | NA | NS | NA |
| Milking parlours | Costa Rica | NS | Space Spray | 0.094 | NA | NS | NA |
| Peanut in bulk or bags | Costa Rica | NS | Surface treatment | 0.062 | NA | NS | NA |
| Potatoes | Italy | EC | Spray, broadcast | 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.00375 | NS | NS | 2 |
| | Germany | DP | Spray, broadcast | NS | 0.075 | NS | 0 |
| Poultry | Costa Rica | NS | Space spray | 0.094 | NA | NS | NA |
| | Costa Rica | NS | Direct App. | 0.094 | NA | NS | NA |
| Poultry houses | Costa Rica | NS | Space Spray | 0.094 | NA | NS | NA |

| Crop | Country | Form, ai | Application [1] | | | | PHI, days |
|---|---|---|---|---|---|---|---|
| | | | Method | Spray conc., kg ai/hl | Rate, kg ai/ha | No. | |
| Root and tuber vegetables, including leaves | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| Root vegetables | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| Root and tuber vegetables, leaves | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Root and tuber vegetables | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Small bush-fruits | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| Small fruits | USA | NS | Spray, broadcast | NS | 0.0045 to 0.056 | NS | 0.5 |
| | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Spinach | Germany | DP | Dusting | NS | 0.075 | NS | NS |
| Stalk vegetables | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| Stem vegetables | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| Stored fruits | Italy | NS | Spray, broadcast | 0.0032 | NS | NS | 2 |
| Stored fruits and vegetables | USA | NS | Space Spray | NS | NS | NS | NA |
| | | NS | Direct Spray | NS | NS | NS | NA |
| Strawberry | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| | Germany | NS | Spray, broadcast | 0.0017 | NS | NS | NS |
| | Italy | NS | Spray, broadcast | 0.004 | NS | NS | 2 |
| | | EC | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.0032 to 0.00375 | NS | NS | 2 |
| Subtropical fruits | Costa Rica | NS | Spray, broadcast | NS | 0.056 | NS | 0 |
| Tomato | Italy | NS | Spray, broadcast | 0.0032 to 0.004 | NS | NS | 2 |
| | | EC | Spray, broadcast | 0.004 | NS | NS | 2 |
| | | LC | Spray, broadcast | 0.00375 | NS | NS | 2 |
| | Germany | DP | Spray, broadcast | NS | 0.075 | NS | 2 |
| Tuber vegetables | Netherlands | Liquid | Spray, broadcast | 0.0048 | NS | NS | 2 |
| Vegetables (except capsicums and lettuces) | Australia | NS | Spray, broadcast | 0.005 | NS | NS | 1 |
| Vegetables | Australia | XX | Spray, broadcast | 0.0078 | NS | NS | 1 |
| | Netherlands | Liquid | Spray, broadcast | 0.004 | NS | NS | 2 |
| | Australia | NS | Spray, broadcast | 0.008 | NS | NS | 1 |
| Warehouse & storage-dried Foods | USA | NS | Space spray | 0..5 | NS | NS | NA |
| | | NS | Aerosol surface spray | 0.3 to 0.5 | NS | NS | NA |
| | | NS | Automatic sequential spray | 1.77 | NS | NS | NA |
| | | NS | Gas operated liquid dispenser | NS | NS | NS | NA |
| | | NS | Surface spray | 0.5 | NS | NS | NA |
| | | NS | Aerosol surface spray | 0.125 | NS | NS | NA |
| | | Dust | Dust crack and crevice | NS | NS | NS | NS |
| Warehouses | Costa Rica | NS | Space spray | 0.125 to 0.50 | NS | NS | NS |
| | | NS | Surface spray | 0.1 | NA | NS | NA |

NA: not applicable; NS: not specified; EC: emulsifiable concentrate; LC: liquid concentrate; DP: dustable powder; XX: microencapsulated timed release liquid concentrate

## RESIDUES RESULTING FROM SUPERVISED TRIALS

All trials were conducted in the USA. The formulation used was a mixture of piperonyl butoxide and pyrethrins, and was applied by broadcast ground spray according to maximum GAP (ten applications

of 0.056 kg ai/ha at intervals of 3 to 7 days), unless otherwise specified. Crops were sampled at a 0-day PHI from at least 12 separate areas of the plot. Orchard samples consisted of at least 16 fruit and were taken from all four quarters of the trees, from high and low areas and from exposed areas as well as those sheltered by foliage. In each trial, three sub-samples were analysed and the highest residue reported. Residues are reported as total pyrethrins, the sum of pyrethrin 1, pyrethrin 2, cinerin 1, cinerin 2, jasmolin 1 and jasmolin 2. As pyrethrins II are degraded during analysis, total pyrethrin levels are estimated from the proportion of pyrethrins I and pyrethrins II in the formulation by multiplying pyrethrins I concentrations by 1.8151 (pyrethrins II = 81.51% of pyrethrins I). Double-underlined values were considered for the estimation of maximum residue levels, supervised trials median residues (STMRs) and highest residues found in the supervised trials (HRs).

Citrus fruits. In seven US trials in 1992 (Report N°. 18001A014) residues were at or below the limit of quantification of 0.04 mg/kg (Table 24).

Table 24. Residues of total pyrethrins in citrus fruits treated with 10 applications at 0.056 kg ai/ha at a 0-day PHI.

| Location | Crop (Variety) | Application rate | | Residues, mg/kg |
|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | |
| AZ, Yuma | Lemons (Frost Newseller) | 1337 | 0.0042 | ≤0.04 |
| CA, Porterville | Lemons (Lisbon) | 2467 | 0.0023 | 0.04 |
| | Oranges (Washington Navel) | 2454 | 0.0023 | ≤0.04 |
| FL, Oviedo | Oranges (Carrizo) | 4072 | 0.0014 | ≤0.04 |
| TX, Raymondville | Oranges (Everhard Navel) | 2365 | 0.0024 | ≤0.04 |
| FL, Oviedo | Grapefruit (Flame) | 4072 | 0.0014 | ≤0.04 |
| TX, Raymondville | Grapefruit (Rio Red) | 2369 | 0.0024 | ≤0.04 |

Berries and other small fruits. In seven US trials in 1992, one on blackberries, two on blueberries, one on cranberries, one on grapes and two on strawberries (Report N°. 18003A020) residues in the fruit were 0.05 to 0.17 mg/kg (Table 25).

Table 25. Residues of pyrethrins in grapes and berries treated with 10 applications of 0.056 kg ai/ha, at a 0-day PHI.

| Location | Crop (Variety) | Application rate | | Residues, mg/kg |
|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | |
| OR, Salem | Blackberries (Evergreen) | 823 | 0.0068 | 0.10 |
| MI, Conklin | Blueberries (Blue Crop) | 1402 | 0.0040 | 0.08 |
| NC, Kenly | Blueberries (Woodard Rabbiteye) | 490 | 0.011 | 0.07 |
| MA, East Wareham | Cranberries (Early Black) | 302 | 0.018 | 0.05 |
| NY, Phelps | Grapes (Catawba) | 935 | 0.0060 | 0.17 |
| FL, Oviedo | Strawberries (Chandler) | 281 | 0.020 | 0.11 |

| | | | | |
|---|---|---|---|---|
| OR , Weston | Strawberries (Benton) | 220 | 0.025 | 0.12 |

Brassica leafy vegetables. In eight trials in 1992/93 (Report N°. 18014A004) residues varied from <0.04 to 0.39 mg/kg. The highest residues were in cabbage heads with wrapper leaves (Table 26).

Table 26. Residues of total pyrethrins in broccoli, cabbages and mustard greens treated with 0.056 kg ai/ha, at a 0-day PHI, 1992-3.

| Location | Crop (variety) | Application rate | | | Sample | Residues, mg/kg |
|---|---|---|---|---|---|---|
| | | No. | Water, l/ha | kg ai/hl | | |
| AK, Newport | Broccoli (Sultan F1 hybrid) | 10 | 187 | 0.030 | Heads | 0.06 |
| CA, Poplar | Broccoli (Early green sprouting) | 10 | 287 | 0.020 | Heads | 0.08 |
| OR, Salem | Broccoli (Pirate) | 12 | 196 | 0.029 | Heads | <0.04 |
| CA, Poplar | Cabbage (Copenhagen market) | 10 | 266 | 0.021 | Heads with wrapper leaves | 0.12 |
| FL, Oviedo | Cabbage (Tenacity) | 11 | 280 | 0.020 | Heads with wrapper leaves | 0.39 |
| | | | | | Heads without wrapper leaves | <0.04 |
| NY, Waterloo | Cabbage (Market prize) | 10 | 234 | 0.024 | Heads with wrapper leaves | 0.05 |
| | | | | | Heads without wrapper leaves | <0.04 |

Fruiting vegetables, cucurbits. In eight trials on cucurbits in 1992/93 (Report N°18013A007) residues in the fruit were at or below the limit of quantification, 0.04 mg/kg (Table 27).

Table 27. Residues of total pyrethrins in cantaloupes, cucumber and summer squash fruit using 10 applications of 0.056 kg ai/ha, at a 0-day PHI.

| Location | Crop (variety) | Application rate | | Residues, mg/kg |
|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | |
| AZ, Somerton | Cantaloupe (Topmark crowset) | 234 | 0.024 | <0.04 |
| CA, Porterville | Cantaloupe (Hales best jumbo) | 289 | 0.019 | 0.04 |
| MI, Mason | Cucumber (Dasher II) | 236 | 0.024 | <0.04 |
| NC, Lucama[1] | Cucumber (General Lee) | 219 | 0.026 | <0.04 |
| FL, Oviedo | Summer squash (Early summer crooknek) | 275 | 0.020 | <0.04 |
| GA, Montezuma | Summer squash (Ely yellow) | 187 | 0.030 | <0.04 |
| NJ, Baptistown | Summer squash (Black beauty) | 238 | 0.024 | <0.04 |
| TX, Uvalde | Summer squash (Aztec) | 154 | 0.037 | <0.04 |

[1] 11 applications

Peppers and tomatoes. In six trials in 1992/1993 (Report N°. 18015A005) residues were <0.04 mg/kg in the fruit (Table 28).

Table 28. Residues of total pyrethrins in peppers and tomatoes treated with 10 applications of 0.056 kg ai/ha, at a 0-day PHI.

| Location | Crop (variety) | Application rate | | Residues, mg/kg |
|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | |
| CA, Porterville | Pepper (Yolo wonder) | 295 | 0.019 | <0.04 |
| NC, Lucama | Pepper (CA wonder bell) | 208 | 0.027 | <0.04 |
| TX, Uvalde | Pepper (Jupiter) | 156 | 0.036 | <0.04 |
| FL, Oviedo | Tomato (Heartland) | 280 | 0.020 | <0.04 |
| MI, Conklin | Tomato (Peto 118) | 214 | 0.026 | <0.04 |
| NJ, Baptistown | Tomato (Better boy) | 252 | 0.022 | <0.04 |

Leafy vegetables. In nine trials in 1992/1993 (Reports N°. 18009A003 and 18009A001) residues in lettuce, radish leaves, sugar beets leaves and spinach were <0.04 to 1.8 mg/kg (Table 29).

Table 29. Residues of total pyrethrins in leafy vegetables treated with 10 applications of 0.056 kg ai/ha, at a 0-day PHI.

| Location | Crop (Variety) | Application rate | | Sample | Residues, mg/kg |
|---|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | | |
| CA, Poplar | Head lettuce (Iceberg Head) | 287 | 0.020 | Heads with wrapper leaves | 0.08 |
| | | | | Heads without wrapper leaves | <0.04 |
| FL, Oviedo | Head lettuce | 280 | 0.020 | Heads with wrapper leaves | 0.16 |
| | | | | Heads without wrapper leaves | <0.04 |
| AZ, Somerton | Leaf lettuce (Walomanns Green) | 241 | 0.023 | Leaves | 0.56 |
| FL, Oviedo | Leaf lettuce (BSS) | 280 | 0.020 | Leaves | 0.52 |
| GA, Montezuma | Mustard greens (Florida Broadleaf) | 191 | 0.029 | Green leaves | 0.90 |
| TX, Uvalde | Mustard greens (Giant Curled) | 153 | 0.037 | Green leaves | 0.64 |
| FL, Oviedo | Radish leaves (Early Scarlet) | 275 | 0.020 | Crowns with leaves attached | 1.8 |
| CO, Austin | Spinach (Polka) | 234 | 0.024 | Leaves | 0.75 |
| TX, Uvalde | Spinach (Fall Green) | 157 | 0.036 | Leaves | 1.0 |

Legume vegetables. In two trials on succulent beans and two on succulent peas in 1992/1993 (Report N°. 18007A012) residues in pods with seeds were <0.04 to 0.13 mg/kg (Table 30).

Table 30. Residues of total pyrethrins in succulent beans and peas treated at 0.056 kg ai/ha, at a 0-day PHI.

| Location | Crop (variety) | Application rate | | Sample | Residues, mg/kg |
|---|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | | |
| FL, Oviedo | Succulent bean (Green Crop) | 275 | 0.020 | Pod | 0.13 |
| WI, Delevan | Succulent bean (Atlantic) | 262 | 0.021 | Pod | <0.04 |
| CA, Poplar | Succulent pea (Wando Seed) | 225 | 0.025 | Pod | 0.09 |
| ND, Northwood | Succulent pea (Wando Seed) | 188 | 0.030 | Pod | <0.04 |

Pulses. In two trials on dry beans and two on dry peas in 1992/93 (Report N°. 18007A012) residues in the seed were <0.04 mg/kg. (Table 31).

Table 31. Residues of pyrethrins in the seeds of dry peas and beans treated with 10 applications of 0.056 kg ai/ha, at a 0-day PHI.

| Location | Crop Variety | Application rate | | Residues, mg/kg |
|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | |
| CO, Austin | Dry Beans (Bill Z) | 234 | 0.024 | <0.04 |
| ND, Northwood | Dry Bean (Upland Navy) | 187 | 0.030 | <0.04 |
| TX, Uvalde | Dry Pea (CA Blackeye Pea #5) | 157 | 0.036 | <0.04 |
| WA, Walla Walla | Dry Pea (Columbia) | 228 | 0.025 | <0.04 |

Root and tuber vegetables. In seven trials, one on carrots, three on potatoes, one on radishes and two on sugar beet (Report N°. 18009A001) the residues in the roots were all <0.04 mg/kg (Table 32).

Table 32. Residues of total pyrethrins in carrots, potatoes, radishes and sugar beets treated with 10 applications of 0.056 kg ai/ha, at a 0-day PHI.

| Location | Crop Variety | Application rate | | Sample | Residues, mg/kg |
|---|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | | |
| TX, Pearsall | Carrots (Imperator) | 154 | 0.036 | Roots with crowns removed, unwashed | <0.04 |
| CO, Austin | Potatoes | 234 | 0.024 | Tubers | <0.04 |
| ID, Middleton | Potatoes | 206 | 0.027 | Tubers | <0.04 |
| ME, Exeter | Potatoes | 195 | 0.029 | Tubers | <0.04 |
| FL, Oviedo | Radishes (Early Scarlet) | 275 | 0.020 | Roots with crowns removed, washed | <0.04 |
| MN, Fisher | Sugar beets (ACH-192) | 188 | 0.030 | Roots with crowns removed, unwashed | <0.04 |
| ND, Northwood | Sugar beets (ACH-192) | 186 | 0.030 | Roots with crowns removed, unwashed | <0.04 |

Celery. In two trials with 10 applications of 0.056 kg ai/ha (0.021 or 0.023 kg ai/hl) in 1993 in Michigan and California (Report N°. 18009A003) residues at a 0-day PHI were 0.16 and 0.70 mg/kg. When the leaves were removed, these values decreased to <0.04 and 0.07 mg/kg respectively.

Mustard seeds. In one trial in Georgia in 1993 with 10 applications of 0.056 kg ai/ha (0.030 kg ai/hl) residues were <0.04 mg/kg (Report N°. 18014A004).

Sugar beet leaves. In two trials in Minnesota and North Dakota with 10 applications of 0.056 kg ai/ha (0.020 kg ai/hl), residues in the crowns with leaves attached were 0.05 and 0.08 mg/kg.

Legume animal feed. In trials on beans and peas, the residues in the hay, vines and forage were determined. The bean hay samples were dried for 2 to 6 days in the open air, and the pea hay for up to 14 days in the field or glasshouse (Report N°. 18007A012) (Table 33).

Table 33. Residues of total pyrethrins in legume animal feed from peas and beans treated with 10 applications of 0.056 kg ai/ha, at a 0-day PHI.

| Location | Crop (variety) | Application rate | | Sample | Residues, mg/kg |
|---|---|---|---|---|---|
| | | Water, l/ha | kg ai/hl | | |
| FL, Oviedo | Succulent Bean (Green Crop) | 275 | 0.020 | Vine | 1.6 |
| | | | | Hay | 0.43 |
| WI, Delevan | Succulent Bean (Atlantic) | 262 | 0.021 | Vine | 0.22 |
| | | | | Hay | 0.09 |
| CO, Austin[1] | Dry Beans (Bill Z) | 234 | 0.024 | Vine | 0.38 |
| | | | | Hay | 0.48 |
| | | | | Forage | 0.32 |
| ND, Northwood | Dry Bean (Upland Navy) | 187 | 0.030 | Vine | 0.08 |
| | | | | Hay | 0.08 |
| | | | | Forage | 0.24 |
| CA, Poplar | Succulent Pea (Wando Seed) | 225 | 0.025 | Vine | 0.82 |
| | | | | Hay | 0.45 |
| ND, Northwood | Succulent Pea (Wando Seed) | 188 | 0.030 | Vine | 0.16 |
| | | | | Hay | 0.03 |
| TX, Uvalde | Dry Pea (CA Blackeye Pea #5) | 157 | 0.036 | Vine | 0.53 |
| | | | | Hay | 0.46 |
| | | | | Forage | 1.6 |
| WA, Walla Walla | Dry Pea (Columbia) | 228 | 0.025 | Vine | 0.62 |
| | | | | Hay | 0.07 |
| | | | | Forage | 0.62 |

[1] 13 applications

Warehouse treatments

Pyrethrins residues in food items were determined after simulated warehouse treatments at the label rate in two trials in March and April (Meinen, 1991a,b), but no information was reported on temperature or humidity conditions.

1. *Space Spray*: A pallet containing ten samples of each food item was placed in the centre of a 170 $m^3$ room. The room was treated twice a week for five weeks, fogging the entire room, at the normal label rate of 0.05 kg ai/1000 $m^3$ with a formulation containing 5% pyrethrins (w/w).

2. *Contact Spray*: A pallet containing ten samples of each food item was placed in the centre of a room 3.05 m by 4.0 m, and the room was sprayed twice a week for five weeks around the edges and round the pallet at the normal label rate of 0.003 kg ai/100 $m^2$ with a formulation containing 0.75% pyrethrins (w/w).

One sample of each food item was collected for analysis after each treatment: 0.9 kg of navy beans and 0.9 kg of Spanish peanuts in a cotton cloth bag and 0.34 kg of dried prunes in a commercial foil bag. Table 34 shows the residue levels. Each value is the result of a single analysis unless otherwise indicated.

Table 34. Pyrethrins residues, mg/kg, in food items after space and contact spray treatments in a simulated warehouse.

| Sample | No. of applications | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 |
| Beans S<br>C | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 |
| Prunes S<br>C | TR[1],<br>TR[1] | <0.05,<br><0.05 | <0.05,<br><0.05 | 0.11[1],<br>TR | <0.05,<br><0.05 | <0.05[1],<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05,<br><0.05 | <0.05 [1],<br><0.05 |
| Peanuts S<br>C | <0.05,<br><0.05 | <0.05,<br><0.05[1] | <0.05,<br><0.05 | TR,<br><0.05 | TR,<br><0.05 | TR,<br><0.05[1] | 0.16,<br><0.05[1] | 0.12,<br><0.05 | 0.23,<br><0.05 | 0.21,<br><0.05 |

S: space spray C: contact spray TR: Trace, >0.05 mg/kg, <0.10 mg/kg
[1] Mean of duplicate analyses

## Animal feeding studies

Dairy cattle. Nine lactating dairy cattle were divided into three groups and dosed orally daily at levels equivalent to 5 ppm, 15 ppm, and 50 ppm total pyrethrin in the diet representing 1, 3 and 10 times the predicted maximum dietary burden for 27 days (Winkler and Huff, 1995a) and were also treated dermally at a nominal level of 89 mg/day (maximum predicted level for combined direct and area spray). Milk was collected at intervals and the cattle were slaughtered 4 to 10 hours after the final dose and edible tissues collected. The level of pyrethrins II in the pyrethrum extract was 92% of pyrethrins I, so total pyrethrins were calculated by multiplying pyrethrins I concentrations by 1.92.

Levels of pyrethrins in the milk peaked at 7 to 11 days at all doses, after which they decreased and stabilized (Table 35).

Table 35. Mean total pyrethrins residue levels, mg/kg, in milk from cattle dosed orally and dermally.

| Dose, ppm in diet | Time (days) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 1 | 3 | 7 | 11 | 14 | 18 | 21 | 24 | 27 |
| 5 | <0.019 (3) | <0.019 (3) | 0.021, <0.019, 0.028 | <0.028, <0.024, <0.021 | 0.054, <0.019, 0.020 | 0.036, 0.019, 0.020 | 0.034, <0.019, 0.023 | 0.032, 0.019, 0.019 | 0.036, 0.027, 0.025 | 0.039, <0.019, 0.019 |
| 15 | <0.019 (3) | 0.028, 0.037, 0.063 | 0.041, 0.063, 0.099 | 0.044, 0.068, 0.153 | 0.036, 0.064, 0.132 | 0.040, 0.068, 0.310 | 0.055, 0.060, 0.134 | 0.039, 0.059, 0.127 | 0.044, 0.072, 0.116 | 0.055, 0.072, 0.133 |
| 50 | <0.019 (3) | 0.149, 0.036, 0.058 | 0.291, 0.158, 0.071 | 0.275, 0.078, 0.103 | 0.341, 0.142, 0.121 | 0.332, 0.126, 0.128 | 0.266, 0.144, 0.110 | 0.349, 0.121, 0.110 | 0.282, 0.123, 0.091 | 0.206, 0.098, 0.027 |

Table 36 shows the total pyrethrin levels in edible commodities after the last dose at 27 days. The highest residues were found in fat.

Table 36. Mean total pyrethrins in edible tissues from dairy cattle dosed orally and dermally for 27 days.

| Sample | Residue, mg/kg | | |
|---|---|---|---|
| | 5 ppm oral dose | 15 ppm oral dose | 50 ppm oral dose |
| Liver[1] | <0.038 (3) | <0.038 (2), 0.067 | 0.056 |
| Kidney[1] | <0.038 (3) | <0.038 (2), 0.05 | 0.168, 0.083, <0.038 |
| Muscle | <0.038 (3) | <0.038 (2), 0.122 | 0.170, 0.142, <0.038 |
| Fat | 0.064, 0.048, 0.075 | 0.232, 0.449, 0.788 | 1.03, 0.798, 2.71 |

[1] Storage stability trials indicated that pyrethrin I residues in liver decreased by 47% after 191 days of storage, and in kidney by a mean of 40% after 87 and 280 days. Samples were stored for >160 days.

Laying hens. Ten hens/treated group were dosed dermally by a space spray with 332 mg total pyrethrins/28 $m^3$ per day, (maximum label rate for area spray) and orally at levels equivalent to 3 ppm, 9 ppm, and 30 ppm in the diet with pyrethrin 1 for 25 to 27 consecutive days (Winkler and Huff, 1995b). Eggs were collected at intervals and all the hens were slaughtered 4 to 10 hours after the final dose and edible tissues collected.

Except for one sample from the middle dose group, residues of pyrethrins in eggs were detected only at the highest dose, after day 7 (Table 37). In the edible tissues residues at all doses were at or about the limit of quantification (0.04 mg/kg) in liver and muscle. Mean residues were up to 0.248 and 0.275 mg/kg in skin and fat respectively (Table 38).

Table 37. Mean total pyrethrins residue levels, mg/kg, in eggs from hens dosed orally at three levels and dermally by space spray at 332 mg/28 $m^3$/day. The 3 values in each cell are from 3 extractions of single composited samples.

| Dose, mg/kg | Time (days) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | 1 | 3 | 7 | 11 | 14 | 18 | 21 | 24 | 27 |
| 3 | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (3) |
| 9 | <0.019 (3) | <0.019 (3) | 0.020, <0.019, 0.033 | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (3) | <0.019 (2), 0.020 | <0.019 (3) | <0.019 (3) |
| 30 | <0.019 (3) | <0.019 (3) | <0.019 (3) | 0.020 (2), 0.026 | 0.022, 0.031, 0.023 | 0.027, 0.034, 0.028 | 0.025, 0.027, 0.034 | 0.030, 0.040, 0.035 | 0.036, 0.046, 0.025 | 0.031, 0.039, 0.024 |

Table 38. Mean total pyrethrin residue levels in the edible tissues of hens dosed orally at three levels and dermally by space spray at 332 mg total pyrethrins/28 $m^3$ per day. The 3 values in each cell are from 3 extractions of single composited samples.

| Sample | Residue, mg/kg | | |
|---|---|---|---|
| | 3 ppm oral dose | 9 ppm oral dose | 30 ppm oral dose |
| Liver | 0.038 (3) | <0.038 (3) | <0.038 (3) |
| Skin | 0.18 | 0.17 | 0.248 |
| Muscle | <0.038 (3) | 0.038 (3) | <0.038 (2), 0.046 |
| Fat | 0.064, 0.048, 0.075 | 0.172, 0.440, 0.076 | 0.278, 0.369, 0.177 |

Dermal transfer factors were derived from low-oral dose residue levels, on the assumption that none of the residues resulted from oral dosing. As an approximation, the residues in the low-dose group can be taken to represent maximum potential residue levels from dermal dosing.

## FATE OF RESIDUES IN PROCESSING

Oranges. Oranges were processed into juice, molasses, dry peel (dry pulp), and oil (Hattermann, 1994c, 1995a). Oranges were sorted and extraneous material such as leaves and stems removed. A sample of unwashed oranges was collected for analysis and stored at 23°C. The fruit was washed on a Pennwalt/Decco Tiltbelt fruit washer-drier, a standard commercial foam detergent cleaner was applied in a brush washer and the washed fruit rinsed again to remove the detergent.

Juice. Juice was extracted with a Commercial FMC 391B In-line Juice Extractor (equipped with continuous water-spray nozzles for maximum recovery of peel oil). The extractor produces juice, peel, and oil/water/peel-frit emulsion. Peel and oil/water/peel-frit emulsion undergo further processing in separate steps. The extracted juice drains down a strainer tube to a manifold under the extractor where it is collected and passed continuously from the extractor through a modified FMC Model 35 finisher. The finisher screens excess pulp from the juice as in commercial practice. Juice is collected in a 570 l stainless steel tank fitted with a motor and stirrer and volume-measuring device. At the end of the run, two cases of 24 cans of juice were canned and stored at 23°C.

Oil. The oil/water/peel-frit emulsion from the FMC extractor was passed through an Automatic Machinery Co. finisher, Model TRF, having a 0.05 cm screen and variable clearance. The solids were collected and combined with peel from the extractor for further processing. The oil emulsion was passed over a Syntron Model SF 152 shaker screen feeder equipped with a double deck vibrating screening trough, and the filtrate collected in a 190 l stainless steel tank. After a minimum of 5 hours under ambient conditions, the lower unemulsified water phase was drained off and the remaining concentrated oil emulsion stored at 0°C until processed (normally at least 16 hours).

After storage, any remaining water phase was siphoned off and the concentrated oil emulsion centrifuged in a laboratory De Laval Gyro Tester continuous centrifuge. The oil fraction was stored at -18°C for at least 16 hours to freeze out any remaining water. The thawed cold-pressed oil was filtered to remove suspended solids, anhydrous sodium sulfate was added to remove any remaining water and the mixture again filtered. The resulting cold-pressed oil was stored at ambient temperature in sealed nitrogen-purged glass bottles.

To produce dried pulp the peel, membrane and seed fraction from the FMC extractor and the solids from the oil/water/peel-frit emulsion finisher were combined and stored at ambient conditions for about 2-3 h until processed. In processing, the peel is transferred to the hopper in the pilot plant feed mill. As peel leaves the hopper, a liquid lime slurry is added continuously to add lime at a rate of 0.3% of the weight of peel. The peel is shredded to a more uniform particle size below about 12 mm. From the shredder, the peel is passed down a reaction conveyor and up an elevator to the press. The limed, chopped and reacted peel is passed through a continuous press with a back-pressure of 1 atmosphere. The press separates the peel into press cake and press liquor.

The press-cake was fed to a triple pass direct-fired dryer which was adjusted to produce dried citrus pulp of approximately 8-10% moisture with a minimum of charring. The temperature of the exhaust air from the dryer was controlled to be about 143°C, which is standard commercial practice.

The dried pulp was stored at -23°C, and the press liquor at 0°C until processed into molasses. To produce molasses the press liquor is heated to boiling under vacuum and concentrated in a Precision Scientific 3-litre laboratory concentrator to approximately 50° Brix. Small amounts of Dow Corning

antifoam B are added to inhibit foaming. The molasses was canned and stored at -23°C. The processing scheme is shown in Figure 7.

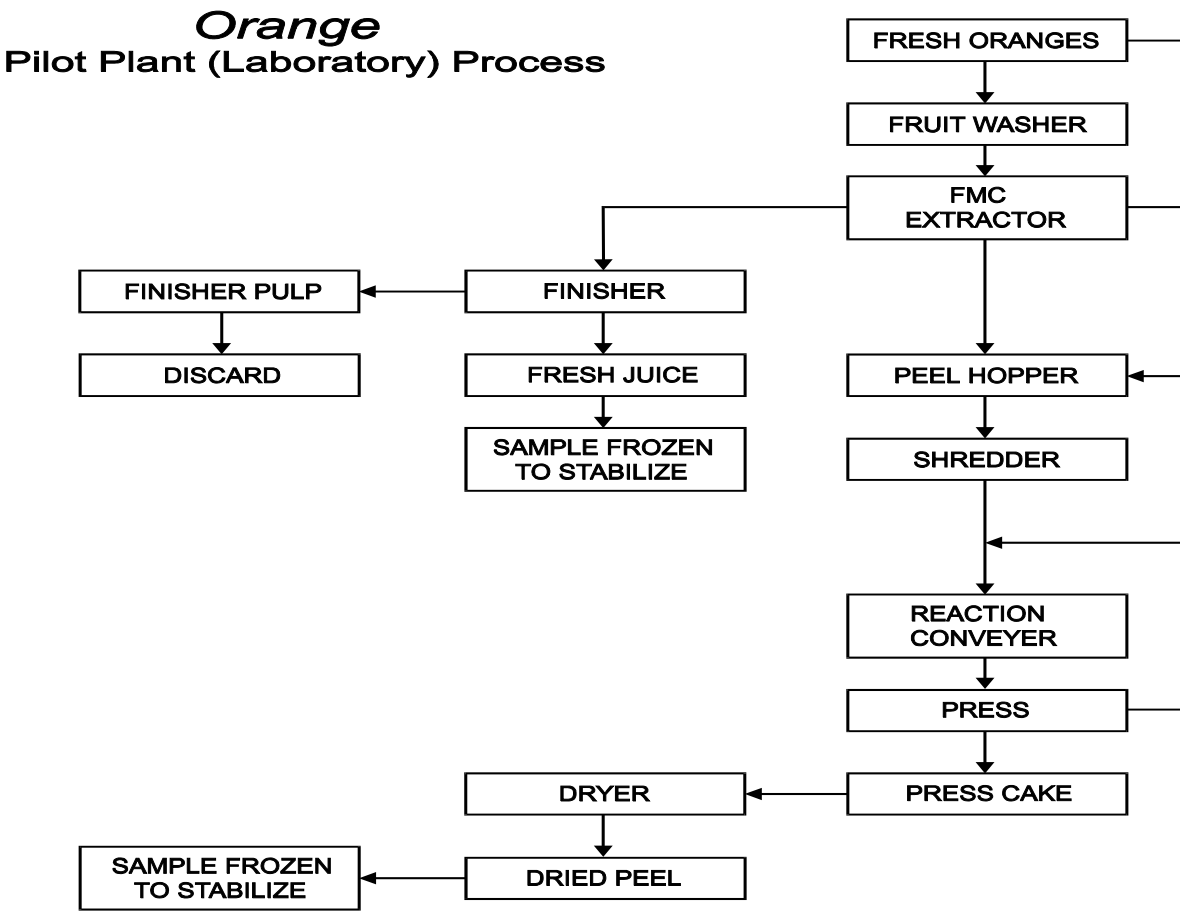



Figure 7. Flow chart for orange processing

Grapes. The laboratory-scale processing of grapes into juice, wet pomace, and dry pomace simulating commercial operations (Hattermann, 1994f) is shown in Figure 8. The main differences between this procedure and commercial practice is shown in Table 38.

Table 38. Laboratory and commercial processing of grapes.

| Step | Laboratory | Commercial |
|---|---|---|
| Washing | High pressure spray washer for 30 seconds. | Powerful sprays of water. |
| Pressing | Suntech fruit press. The method employs press racks and cloths and does not crush either the stems or seeds. The grapes are pressed at least twice for maximum recovery of fresh juice. The grape pulp after pressing (wet pomace), consisting of seeds, skins and stems is sampled and the remaining wet pomace is dried in a bin air drier and sampled as dry pomace. The juice recovered from the pressing operation is strained through a standard milk filter and sampled as fresh juice. | Rotary grape crusher where centrifugal force is applied to break up the grapes, separating the juice and pulp from the stems without crushing either the stems or seeds. The stems are discharged and the juice and pulp gravitate to a large receiver beneath the machine. The grape mass is then processed by either a hydraulic or continuous fruit juice pressing operation to remove the pulp from the juice. |

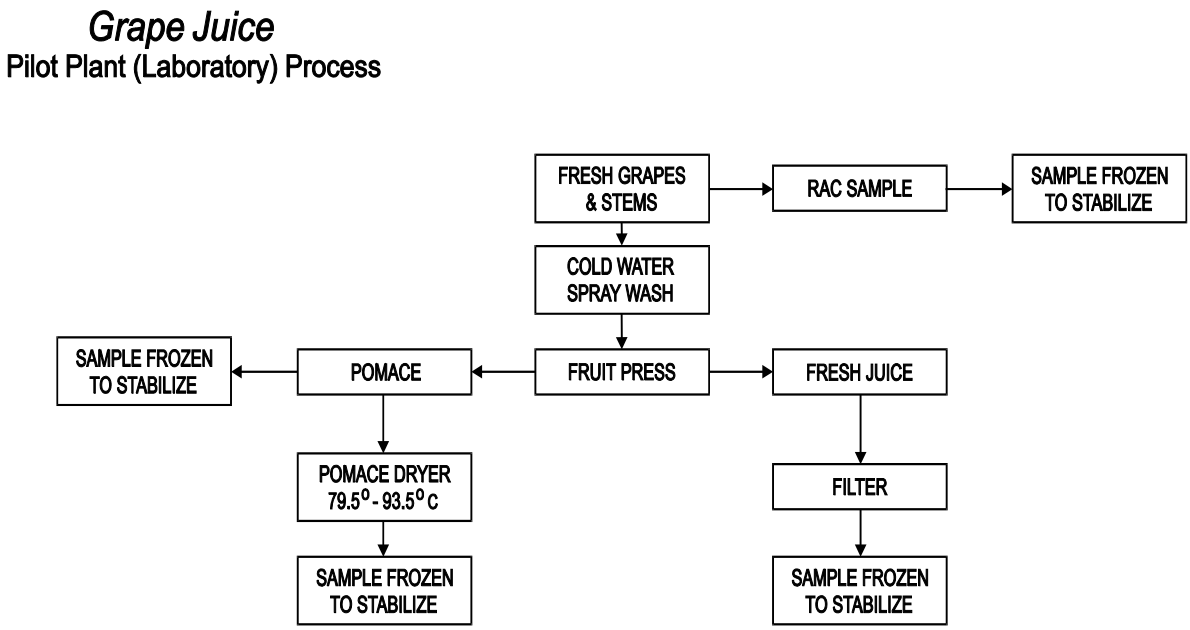



Figure 8. Flow chart for production of juice and pomace from grapes.

Grapes were processed into raisins by sun-drying in greenhouses for 24 days, and separated from raisin waste by screening. Figure 9 shows the procedure.

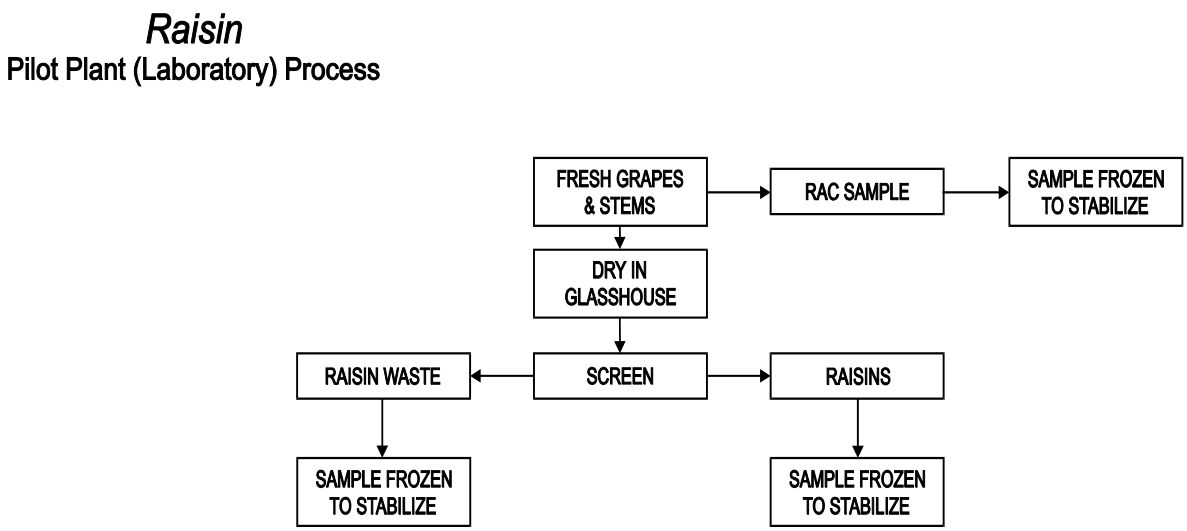



Figure 9. Flow chart for production of raisins from grapes**.**

Tomatoes. Typical canning variety tomatoes were processed in laboratory-scale simulated commercial operation into wet and dry pomace, purée and juice (Hattermann, 1996e,1999) (Figure 10). Table 39 shows the main differences between the laboratory and the commercial procedures.

Table 39. Laboratory and commercial procedures for tomatoes.

| Step | Laboratory | Commercial |
|---|---|---|
| Inspection | Sorted by hand and unsuitable fruit retained as cannery waste. All material is used. | On a moving inspection belt stones, leaves, and green, decomposed or unfit fruit are removed, and the tomatoes then sized and graded by machine to different sizes according to specified uses |
| Washing and rising | Soaked in kettle with 0.5% lye (NaOH) solution for 3 min at 54°C and batch rinsed with high pressure spray rinse for 30 sec. | Soaked in tank to remove drosophila eggs, larvae and other contaminants (lye solution at 0.5% sometimes used) for 3 min at 54°C, and rinsed through a series of spray nozzles |
| Sorting and trimming | Fruit inspected, and if necessary trimmed by hand. The sorting step was omitted as all available material was processed together. | Off-colour, defective fruit (rotten or mouldy areas, insect damage or sunscald) are removed and dumped, and the fruit sorted: large perfect fruit to the scalder; small and misshapen fruit to pulping line. |
| Coring | Omitted because canned whole tomatoes was not a required fraction. | The tomatoes for canning are forwarded to a coring operation |
| Peeling | Batch steam peeled in atmospheric steam cabinet at 80-100 psi for 30 sec per batch | Steam, lye, or infrared peeling |
| Final inspection | Omitted as all material was used. | Final inspection before canning to assess defects for grade classification |
| Crushing/chopping | Tomatoes hand fed into a pulper finisher machine and pulp/pomace separated from the juice. The juice was then frozen for concentration and the wet pomace sample packaged from the wet pomace recovered. The remaining wet pomace was dried for the dry pomace sample fraction. There is no commercial practice for the drying of wet pomace or tomato cannery waste. | The tomatoes are dropped into the chopper at the end of the trimming belt, and the juice is extracted by either a screw or paddle type extractor. |

| Step | Laboratory | Commercial |
|---|---|---|
| Juice concen-tration | Groen batch vacuum-pan concentrator. Purée was packed, sealed, heated for 20 min at 98-100°C and cooled under cold running tap water before packaging for purée sample fraction | Under reduced pressure and usually with double- or multiple-effect continuous evaporators, the juice is handled and the finished purée discharged at 10.6 ± 1% solids and paste at approximately 30-32% solids. The purée is packed in cans and immediately sealed and the cans cooled before casing |
| Paste | A sample of the finished paste was set aside for the manufacture of juice from concentrate. 1% salt was added and the temperature raised to 88-91°C, before packing and sealing. The sealed cans were heated for 20 min at 98-100°C and then cooled under running cold tap water before packaging. | The temperature of the finished paste is raised to approximately 90°C before it is packed in cans, immediately sealed and the cans cooled before casing |
| Juice | The finished paste is reconstituted with water, salt and ascorbic acid (vitamin C), heated to 88-91°C, packaged, sealed, and the temperature raised to 98-100°C for 20 min, and cooled under running cold tap water before packaging as the juice from concentrate sample fraction. | The tomato juice from concentrate must contain not less than 5.5% w/w of tomato solids. The paste is prepared as in the previous step and bulk packaged in 208 l drums for processing into products such as ketchup, juice from concentrate, or sauce. |

Succulent beans. Pods and plants were processed from whole plants from at least 12 locations in the plots, cutting approximately 2.5 cm off each end of some pods and discarding the middle section, into cannery waste (Hattermann, 1994i, 1995b). A composite sample of leaves, whole pods, and pod tips was collected for the 2.2 kg cannery waste sample consisting of 0.22 kg of leaves, 0.45 kg of whole pods, and 1.6 kg of pod tips.

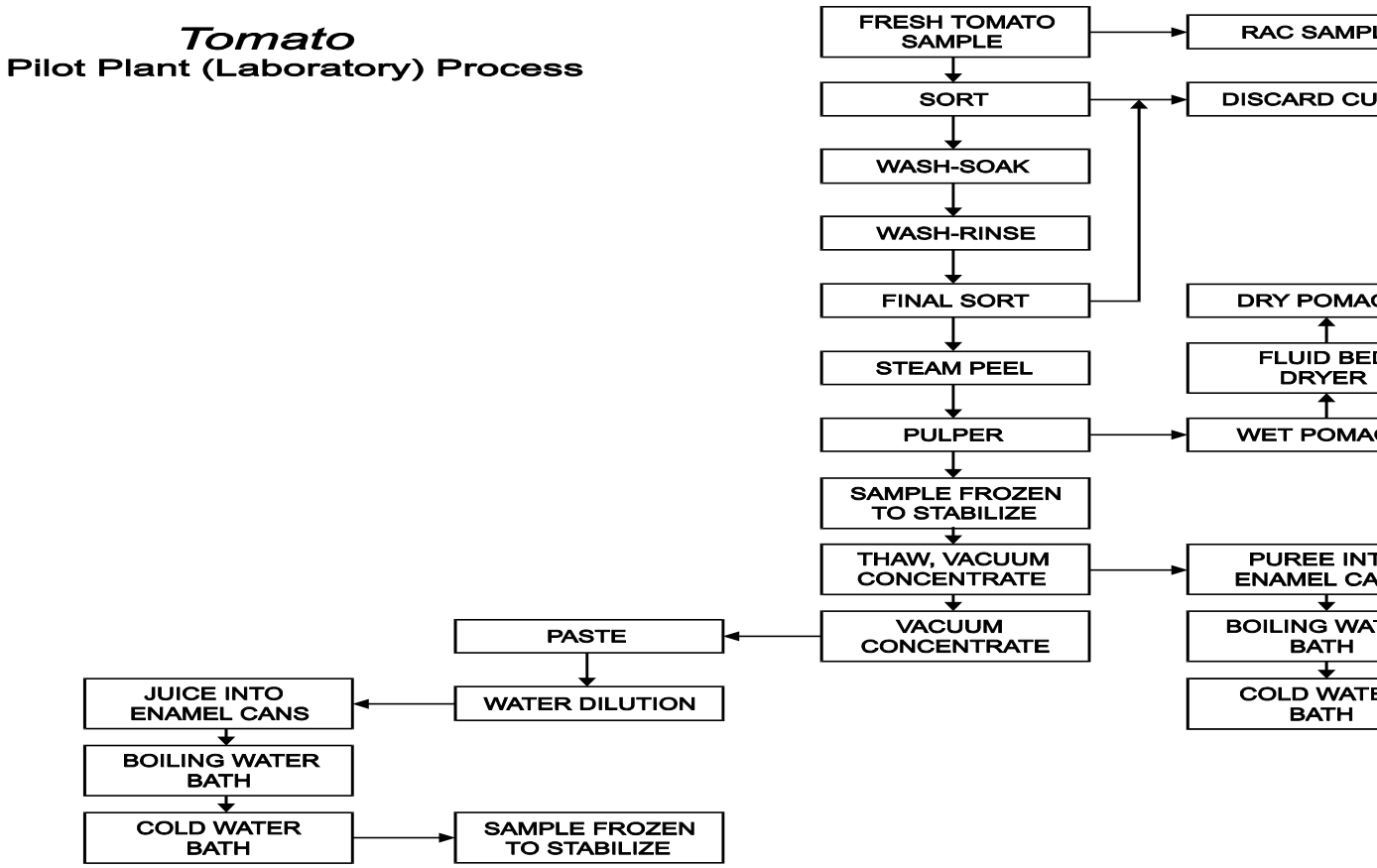


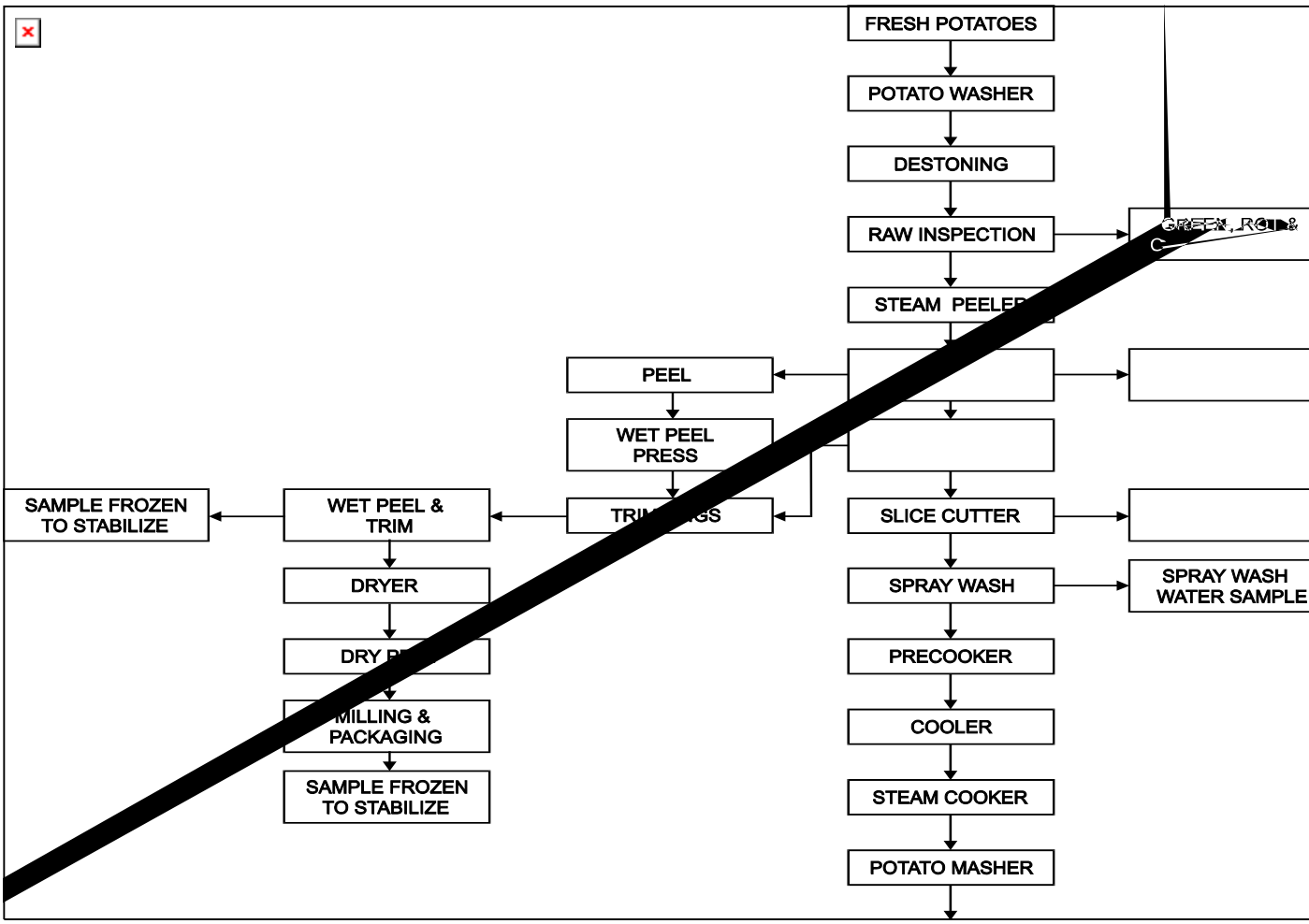

Figure 10. Flow chart for tomato processing

Potatoes. Potatoes were laboratory-scale processed, simulating commercial practices, into chips, wet peel from the granule-making process, and granules, equivalent to flakes (Hattermann, 1996b,c).

Figure 11 shows the laboratory procedure for producing potato granules. The main differences between this procedure and commercial operation are shown on Table 40.

Table 40. Laboratory and commercial production of potato granules.

| Step | Laboratory | Commercial |
|---|---|---|
| Washing | Tub washing for 5-10 min | Water flume and/or barrel washer and destoning machine |
| Peeling | Continuous batch 190 psi pressure steam peeler for ~10-20 seconds | Continuous batch pilot plant 80-85 psi pressure steam peeler for ~45-60 seconds |
| Cutting | ~1-1.3 cm slabs using a restaurant style food cutter | 1.3 cm slabs using a commercial model cutter |
| Starch removal | Batch spray washing the slices for ~30 seconds in cold water | Continuous cold water spray washer |
| Pre-cooking | At 70-77°C with target 71-74°C for 20-22 min using a batch 150 l steam-jacketed kettle, cooled below 32°C under cold running tap water | At 71-74°C continuous auger style pre-cooker for 20 min, then cold running tap water continuous auger style cooler for 20 min to cool slices to below 32°C |
| Cooking | At 94-100 °C for 40-42 min in an atmospheric-pressure flowing steam batch-style steam cooker. | Continuous auger steam cooker at 96-100°C for 35-45 min. |
| Ricing | Mashed in a restaurant-style meat grinder without the grinding attachment. | Auger containing a ricing/mashing grid |
| Add-back process | Pre-weighed additives are added together with the mashed potatoes and mixed for ~60 seconds. Packaged in approximately 1 kg plastic containers and frozen for later dehydration | Continuous primary mixer where additives are added. Dry potato granules (0.13 mm) are added to the wet mash at the rate of 1 kg of dry granules to 0.5 kg of wet mash |
| Drying | Fluidized bed | 38-43°C conditioning belt for 30 min. Flash-dried at 260-304°C for ~30 seconds to 13-17% moisture. Fluidized-bed dried to 8-10% moisture. |
| Granule | Screened through 30- and 60-mesh screens. The over-60 mesh add-back material is retained. The product below 60 mesh is packaged into the potato granule sample fraction. | Sifted through a 32-mesh screen, the <32 mesh product is adjusted to 7-7.5% moisture at 38°C on an ambient fluid bed cooler, sifted through a 105-mesh screen; the product over 105 mesh is used for add-back "seed" supply and the below 105-mesh product is packed. |


FRESH POTATOES
POTATO WASHER
DESTONING
RAW INSPECTION
STEAM PEELER
PEEL
WET PEEL PRESS
SAMPLE FROZEN TO STABILIZE
WET PEEL & TRIM
DRYER
MILLING & PACKAGING
SAMPLE FROZEN TO STABILIZE
SLICE CUTTER
SPRAY WASH
SPRAY WASH WATER SAMPLE
PRECOOKER
COOLER
STEAM COOKER
POTATO MASHER

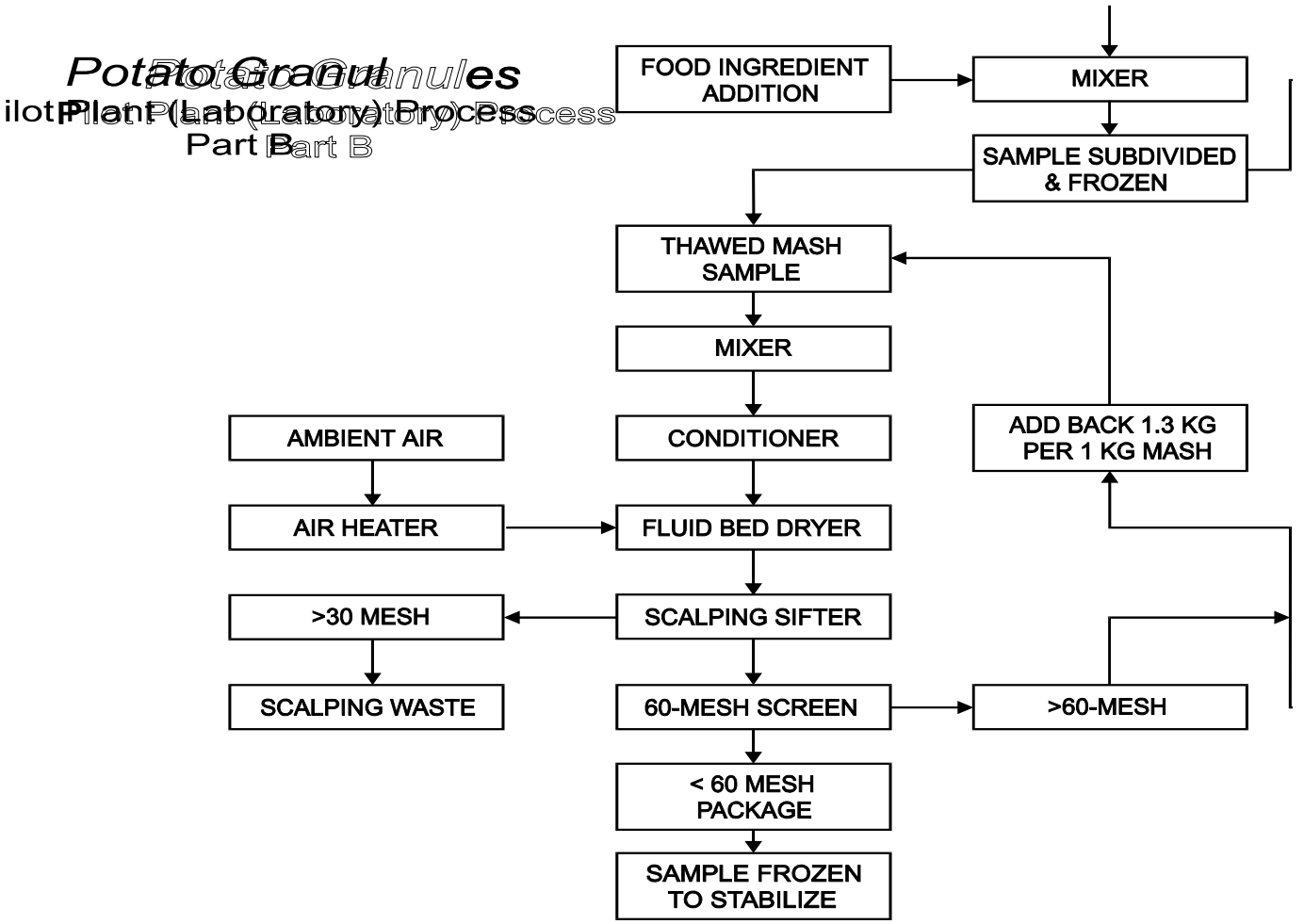



Figure 11. Flow chart for potato processing

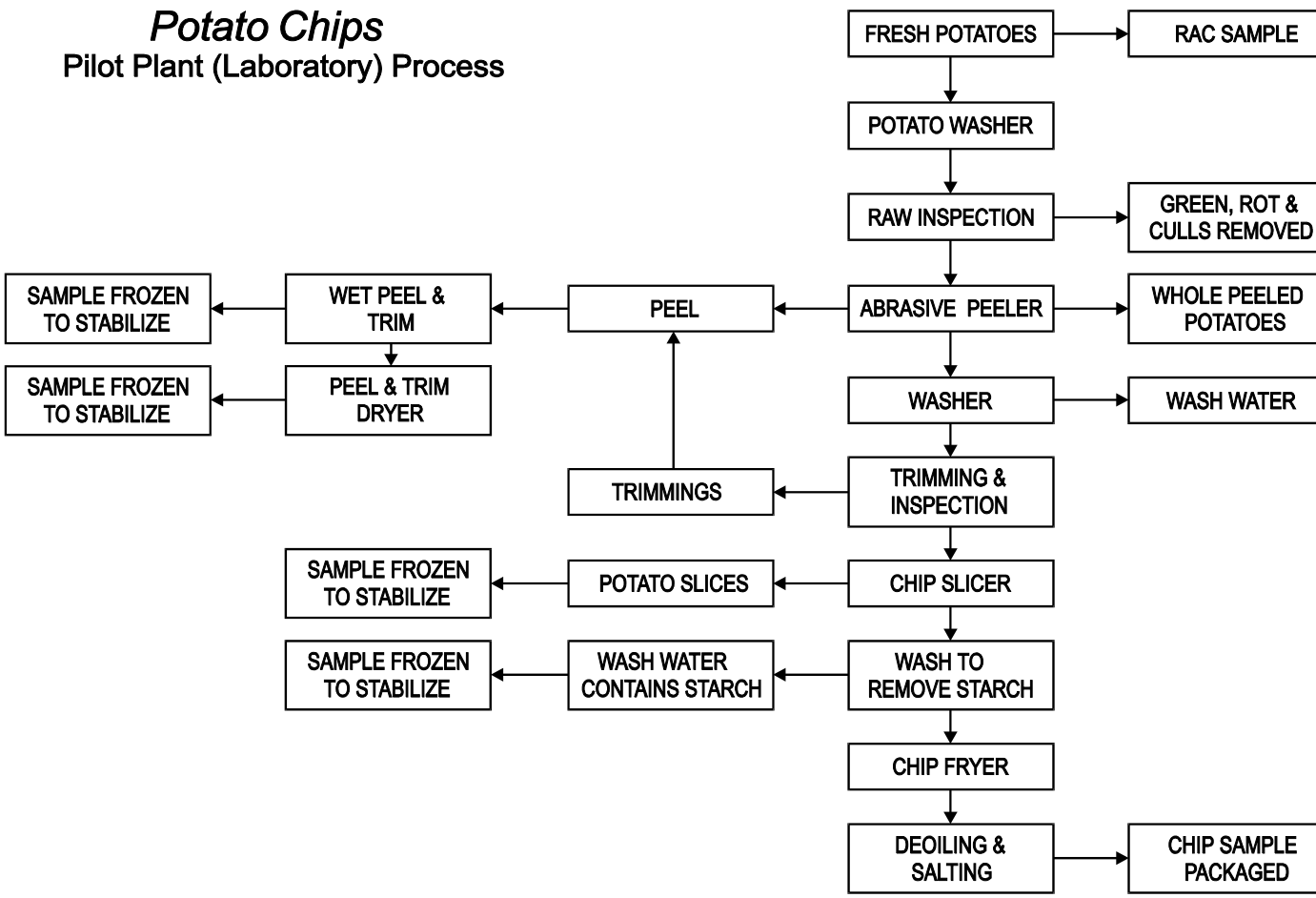



Figure 12. Flow chart for potato chip production

Figure 12 shows the laboratory procedure to process potato chips. The main differences between this procedure and the commercial operation are shown on Table 41.

Table 41. Laboratory and commercial procedures for processing potato chips.

| Step | Laboratory | Commercial |
|---|---|---|
| Washing | Tub washing for 5-10 min | Water flume and/or barrel washer and destoning machine |
| Peeling | Peeled for 25-35 seconds in batches using an abrasive peeler | Continuous abrasive peeler |
| Cutting | Restaurant-style food cutter/slicer | Urschel model CC cutter |
| Frying | Electrically heated restaurant-style deep fat fryer at ~163-191°C for 60-90 seconds | Continuous deep fat fryer and chain-conveyed through hot oil at 185°C for ~60 seconds |
| Draining and packing | Draining free oil in a restaurant-style draining tray and salting the chips by hand | Chain conveyor to allow oil to drain, salting conveyor, inspection, and to packaging |

Sugar beet. Sugar beets were processed by simulated commercial operations in a laboratory into dehydrated pulp, molasses, and refined sugar (Hattermann, 1994k,l). Although there are variations in the details, all processors used essentially the same basic method.

The main differences between the laboratory and commercial operations are shown in Table 42. Figure 13 shows a flow chart of the laboratory processing.

Table 42. Laboratory and commercial procedures for processing sugar beet.

| Step | Laboratory | Commercial |
|---|---|---|
| Sample | Beets stored at -15 ± 8°C until processing. | Beets are fresh or may be frozen in outside piles before processing |
| Washing | Stainless steel tub-washed using warm water | Water flume and a washing section where trash and field dirt are removed |
| Cutting | Into 1-3 mm thickness using a LanElec vegetable slicer | Into cossettes (strips) by large rotary cutting wheels fitted with cutting knives with corrugated cutting edges. The thickness of the cossettes produced depends partly on the type of diffuser used. The thickness could be as much as 4 mm |
| Diffusing | Batches of cossettes in stainless steel mesh baskets are moved by hand from cell to cell in one direction while diffusion liquid is transferred from cell to cell in the opposite direction. Temperature is maintained at 70 °C. | Continuous screw, continuous chain, or a series of individual cells with means to transfer pulp and water from cell to cell |
| Pressing | Pressed by Suntech fruit press to remove free juice, which is then returned to the diffuser | Pressed to remove free juice which is then returned to the diffuser for thin juice recovery |
| Drying | Laboratory bin air dryer to <10% moisture | Heated rotary dryers to <10% moisture |
| First | In batches in a 75 l steam-jacketed stainless steel kettle. A milk of lime slurry containing | Raw juice of 10-15% Brix is purified by the addition of lime and carbon dioxide gas, either continuously or |

| Step | Laboratory | Commercial |
|---|---|---|
| carbonation | about 11% CaO is added slowly while gassing with bottled $CO_2$. The pH is ~10 to aid in keeping the alkalinity as close to 0.100% CaO/100 ml as possible. Alkalinity and CaO content are checked by titration. | batchwise. The first carbonation is done at 80-85°C. Lime addition may vary depending on beet quality from 1.4-2.0%. |
| Clarification | Settle in 114 l stainless steel kettle. Supernatant liquor is decanted and the sludge filtered through Buchner funnels | Multi-tray clarifiers and rotary vacuum filters |
| Second carbonation | In batches in a 75 l steam jacketed stainless steel kettle at 90-95°C. Gassing is regulated by use of phenolthalein, and by laboratory titration. | Continuously in a large tank. The juice is gassed at 90-95°C to an optimum alkalinity, about 0.015 gm CaO/100 ml, to minimize lime salts |
| Filtering | Buchner funnels | Various types of industrial filters such as pressure leaf filters |
| Concentration | Groen steam-jacketed vacuum pan. Steam temperature is automatically regulated at approximately 93°C. Vacuum is maintained at approximately 450 mm Hg | Multiple-effect evaporators of various designs |
| Increase Brix | Boiled to sugar in a small laboratory vacuum pan of similar design to full scale pans. Vacuum is maintained at 100 mm Hg | Addition of lower grade sugar such as intermediate and raw sugars from a three-boiling scheme. The enriched thick juice is called standard liquor and white sugar is made from it. The liquor is boiled to sugar in large vacuum pans of various designs under conditions that cause the syrup to be supersaturated |
| Molasses | Massecuite (remaining materials) from the vacuum pan is centrifuged for the recovery of white sugar. It is washed and the initial spin-off syrup is molasses. | Large centrifuges to separate the white sugar from mother liquor. The sugar in the centrifuge basket is washed with hot clean water. The spin-off syrup is subjected to further processing for additional sugar recovery. The syrup after further sugar recovery is molasses |
| Sugar | The wet sugar is dried with a Kitchen Aid mixer. It is stirred in the mixing bowl as warm air is blown against the bowl and into the stirred sugar to promote drying. | Wet sugar from the centrifuge is dried in large rotary dryers, through which hot air is blown. As the sugar dries, the mixing in the heater and coolers prevents agglomeration and this results in white granulated sugar |

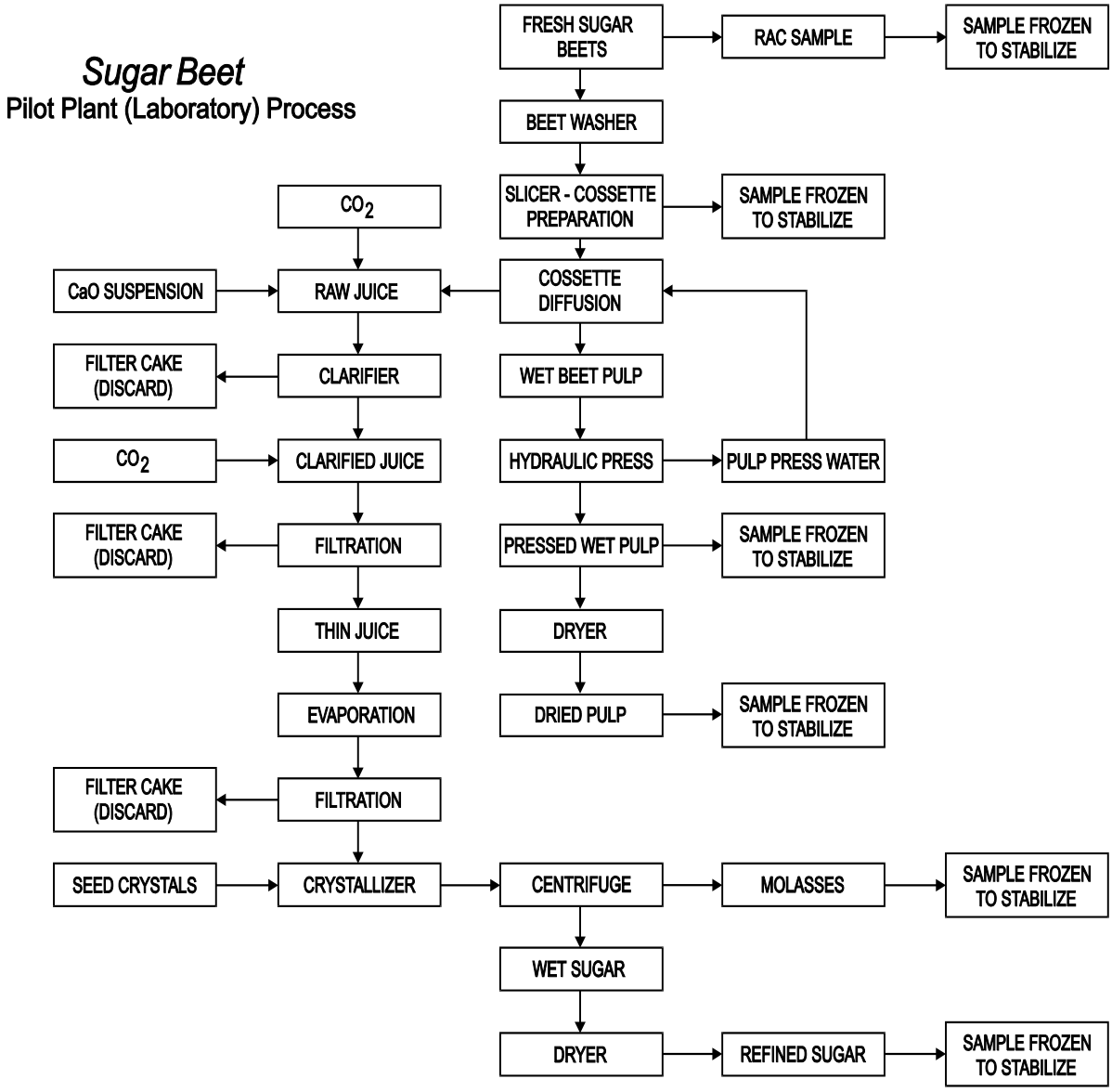



Figure 13. Flow chart for sugar beet processing

All commodities were treated 10-11 times with 0.28 kg ai/ha, a fivefold rate. Samples were collected at a 0-day PHI. The results before and after processing are shown in Table 43.

Table 43. Mean residues in raw commodities (RAC) and processed products of plants treated with pyrethrin, and calculated processing factors.

| Commodity | Sample | Total pyrethrins, mg/kg | Processing factor |
|---|---|---|---|
| Orange | Fruit (RAC) | 0.061 | |

| Commodity | Sample | Total pyrethrins, mg/kg | Processing factor |
|---|---|---|---|
| | Dry pulp | 0.458 | 7.51 |
| | Oil | 1.23 | 20.3 |
| | Molasses | 0.042 | 0.69 |
| | Juice | <0.04 | <0.66 |
| Grape | Fruit (RAC) | 0.084 | |
| | Raisin | <0.04 | <0.48 |
| | Raisin waste | <0.04 | <0.48 |
| | Wet pomace | 0.111 | 1.32 |
| | Dry pomace | 0.423 | 5.03 |
| | Juice | <0.04 | <0.48 |
| Tomato | Fruit (RAC) | 0.077 | |
| | Wet pomace | 0.674 | 8.75 |
| | Dry pomace | 2.24 | 29.0 |
| | Purée | <0.04 | <0.52 |
| | Juice | <0.04 | <0.52 |
| Succulent bean | Pods (RAC) | 0.342 | |
| | Cannery waste | 1.195 | 3.49 |
| Potato | Tuber (RAC) | <0.04 | |
| | Granules | <0.04 | NC |
| | Chips | <0.04 | NC |
| | Wet peel | <0.04 | NC |
| Sugar beet | Root (RAC) | <0.04 | |
| | Dry pulp | <0.04 | NC |
| | Sugar | <0.04 | NC |
| | Molasses | <0.04 | NC |

NC: could not be calculated

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

In 1998, the US Food and Drug Administration (FDA), which is charged with enforcing tolerances in imported and domestically produced foods shipped in interstate commerce, analysed 458 domestic and 287 imported samples of food products for residues of pyrethrins (US FDA, 1999). Pyrethrins were not detected in any of the products.

## NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs were reported.

| Country | Crop | MRL, mg/kg, total pyrethrins |
|---|---|---|
| Australia | Cereal grains | 3 |
| | Dried fruits, dried vegetables, fruits, oilseed, tree nuts, vegetables | 1 |
| Austria | Cereals, fish meat | 3 |
| | Fruit, vegetables | 1 |
| | Other | 0.05 |
| Belgium | Grains | 3 |
| | Fruits, vegetables | 1 |
| | Others | 0*(0.05) |
| Canada | Raw cereals | 3 |

| Country | Crop | MRL, mg/kg, total pyrethrins |
|---|---|---|
| | Almonds, apples, beans, blackberries, blueberries, boysenberries, cherries, cocoa beans, copra, crab apples, currants, dewberries, figs, gooseberries, grapes, guavas, huckleberries, loganberries, mangoes, muskmelons, oranges, peaches, peanuts, pears, peas, pineapple, plums, raspberries, tomatoes, walnuts | 1 |
| China | Grains | 0.5 |
| Czech Republic | Cereals, dried fish, maize, rice | 3 |
| | Wheat | 2 |
| | Dried fruits, dried vegetables, fruits (apple, cherry, pear), nuts, oil seeds, | 1 |
| | Flour, pepper, potato, tomato, vegetables | 0.1 |
| Denmark | Cereals | 3 |
| | Berries and small fruits, cabbage, citrus fruits, fruits and vegetables, fungi, legumes, misc. fruits, onions, pome fruits, roots and tubers, stem vegetables, stone fruits, wood nuts | 1 |
| Finland | Cereal grains | 2 |
| | Others | 1 |
| France | Cereals | 3 |
| | Fruit and vegetables | 1 |
| Germany | Cereals, oilseeds | 3 |
| | Fruits, vegetables | 1 |
| | Other foods of plant origin | 0.5 |
| Hungary | Barley grain, corn, oat grain, rice (brown), rice (polished), rye grain, triticale, wheat grain | 3 |
| | Dried fruit, dried vegetables, fruit, oil-containing seeds, peanut, vegetables | 1 |
| Iceland | Mango | 5 |
| | Cereals/cereal products, fish/fish products | 3 |
| | Brassica vegetables, bulbs, cane fruit, citrus fruit, cucurbits-edible skin, cucurbits-inedible skin, flowering brassica, fruiting vegetables, fungi, grapes, table/wine, head brassica, herbs, kohlrabi, leafy brassica, legume vegetables, lettuce and similar, miscellaneous, oilseeds, other small fruit & berries, pome fruit, potatoes, pulses, root & tuber vegetables, spinach, stem vegetables, stone fruit, strawberry, sweet corn, tree nuts, watercress, wild berries & wild fruit, witloof chicory | 1 |
| Indonesia | Cereals, fish (dry) | 3 |
| | Fruits (dry), vegetables (dry) | 1 |
| | Eggs | 0.2 |
| | Milk | 0.1 |
| | Meat | 0.01 |
| Ireland | Barley, buckwheat, grain sorghum, maize, millet, oats, paddy rice, rye, triticale and other cereals, wheat | 3 |
| | All other products | 1 |
| Italy | Cereals in bulk, legumes in bulk | 3 |

| Country | Crop | MRL, mg/kg, total pyrethrins |
|---|---|---|
| | Forage legumes, fruit, garden vegetables, potatoes, sugar beet, sunflower seeds, tobacco | 1 |
| Kenya | Cereal grains, dried fish | 3 |
| | Dried fruits, dried vegetables, oilseeds, treenuts | 1 |
| Korea | African millet, barley, buckwheat, corn (maize), foxtail millet, oats, rice, rye, wheat | 3 |
| | Almonds, apples, apricots, asparagus, avocados, bananas, broad beans, carrots, celery, cherries, chestnuts, Chinese cabbage, Chinese quince, cotton seed, crown daisies, cucumbers, eggplant, garlic, ginger, gingko nuts, grapefruit, grapes, green onions, green peppers, iceberg lettuce, Japanese apricots, kale, kidney beans, kiwi fruit, Korean plums, leek, lemons, lettuce, mandarin oranges, mangoes, melons, mung beans, onions, oranges, other citrus fruit, other fruits, other legumes, other mushrooms, other nuts, other seedcrop plants, papaya, peaches, peanuts, pears, peas, pecans, persimmons, pineapple, radish greens, radishes (roots), red beans, sesame seeds, shitake mushrooms, soybeans, spinach, strawberries, sunflower seeds, taro, tomatoes, walnuts, watermelon, Western cabbage | 1 |
| Malaysia | Cereal grains, fish and seafood (dried) | 3 |
| | Cocoa beans, copra, fruits (dried and fresh), leafy vegetables (dried and fresh), non-leafy vegetables (dried and fresh), root vegetables (dried and fresh) | 1 |
| | Meat (dried) | 0.1 |
| Netherlands | Buckwheat cereal, smoked fish | 3 |
| | Dried fruits, dried vegetables, fruit, oilseed, vegetables | 1 |
| | Tropical seed | 0.5 |
| | Other | 0*(0.05) |
| New Delhi | Fruits and vegetables | 1 |
| | Foodgrains, milled foodgrains | nil |
| New Zealand | Fruit, vegetables | 1 |
| Norway | Fruit, vegetables, potato | 1 |
| Romania | Dry fish, grain, grain seeds | 3 |
| | Dry vegetables and fruits, fresh vegetables and fruits, nuts, oilseeds | 1 |
| | Other products vegetal origin | 0.05 |
| Singapore | Cereal grains, dried fish | 3 |
| | Dried fruits, dried vegetables, fruits, nuts, oilseeds, vegetables | 1 |
| Slovak Republic | Cereals, fish, dried | 3 |
| | Fruit, fruit (dried), nuts, oil seeds, peanuts (kernels), vegetables, vegetables (dried) | 1 |
| South Africa | Cereal grains | 2 |
| | Cotton seed, fruit (dried), groundnuts, nuts (dried), other oil seeds, sunflower seeds, vegetables (dried) | 1 |
| Spain | Grains | 3 |
| | Berries and small fruit, bulb vegetables, citrus fruit, fresh aromatic herbs and leaf vegetables, fruit with or without shell, fruits and peponides, fungi, green legumes (fresh), legumes, other fruits, potatoes, root and tuber vegetables, seed fruit, stone fruit, vegetables of the genus brassica, young stalks | 1 |
| | Dried products, hay and forage crops, hops, oilseeds, other edible seeds, other infusions, other products for consumption (tobacco, sugar beet, sugarcane, other), spices, tea and other infusions | 0.50 |
| Switzerland | Cereal, infusion plants, tea | 3 |
| | Dried fruits, dried vegetables, fruit, oil seeds, vegetables | 1 |
| | Non specified food stuffs | 0.5 |

| Country | Crop | MRL, mg/kg, total pyrethrins |
|---|---|---|
| | Cereal products | 0.3 |
| | Fancy mushrooms | 0.1 |
| | Milk | 0.02 |
| Sweden | Cereal, seeds, hulled grain, | 3 |
| | All kinds of cabbages and lettuces, citrus fruits, cucumber vegetables with edible peel, cucumber vegetables without edible peel, legumes, fresh, misc. Fruits, *e.g.* Banana, kiwi, mango, mushrooms, nuts, onions, all kinds, pome fruits, potatoes, root vegetables, small fruits and berries, both cultivated and wild, solanaceae vegetables, spices, stem vegetables, stone fruits, sucker maize, table grapes | 1 |
| Taiwan | Mushrooms | 1 |
| UK | Cereals (wheat, rye, barley, oats, triticale, maize, rice, other cereals | 3 |
| USA | Barley (post-harvest), buckwheat (post-harvest), bird seed mixture (post-harvest), corn (including popcorn) (post-harvest), rice (post-harvest), rye (post-harvest), wheat (post-harvest) | 3 |
| | Cotton seed (post-harvest), crab apples (post-harvest), currants (post-harvest), dewberries (post-harvest), figs (post-harvest), flaxseed (post-harvest), gooseberries (post-harvest), grain sorghum (post-harvest), grapes (post-harvest), guavas (post-harvest), loganberries (post-harvest), mangoes (post-harvest), muskmelons (post-harvest), oats (post-harvest), oranges (post-harvest), peaches (post-harvest), peanuts (with shell removed) (post-harvest), pears (post-harvest), peas (post-harvest), pineapples (post-harvest), plums (fresh prunes) (post-harvest), raspberries (post-harvest), ,tomatoes (post-harvest), walnuts (post-harvest) | 1 |
| | Milk fat | 0.5a |
| | Poultry fat, meat, and meat by-products | 0.2 |
| | Eggs, fat, meat and meat by-products of cattle, goats, hogs, horses and sheep | 0.1 |
| | Potatoes (post-harvest), sweet potatoes (post-harvest) | 0.05 |
| Yugoslavia | Cereals | 3 |
| | Fruit, processed cereals, vegetables | 1 |
| | Fat, meat, meat products (fat basis), | 0.05 |

[a] Reflecting negligible residues in milk.

## APPRAISAL

Pyrethrins are a naturally occurring insecticide containing six biologically active, chemically related esters. The esters of chrysanthemic acid (pyrethrins I) are pyrethrin 1, cinerin 1, and jasmolin 1, and the esters of pyrethric acid (pyrethrins II) are pyrethrin 2, cinerin 2, and jasmolin 2. Pyrethrin 1 is the predominant compound. Pyrethrins are used not only on crops but also used as a direct spray on farm animals.

Pyrethrins were last evaluated for residues in food by the JMPR in 1974. At its twenty-sixth session, the CCPR noted that these compounds were originally scheduled for toxicological and

residue evaluation by the 1994 Joint Meeting but the evaluation of residues had been postponed to 2000.

For this periodic review, the manufacturer provided relevant supporting studies, information on GAP, and data on residues in citrus, small fruits, leafy vegetables, cucurbits, peppers, tomatoes, beans, peas, root and tuber vegetables, celery and mustard seeds after foliar treatment, and beans, prunes, and peanuts after treatment in a warehouse. National maximum residue limits, information on GAP, and data on residues in celeriac and leeks were provided by the governments of Australia, Germany, and Poland.

***Metabolism***

*Animals*

*Rats* were given [$^{14}$C]pyrethrin 1 either at a single dose of 10 mg/kg bw or a single dose of 100 mg/kg bw for males and 50 mg/kg bw for females, or unlabelled pyrethrin 1 at a dose of 10 mg/kg bw for 14 days before a single radiolabelled dose. Excreta were collected periodically, and the animals were killed 7 days after the last dose. The radiolabel in urine after all treatments represented 32-47% of the administered dose in males and 49-57% in females. In faeces, the amount of radiolabel represented 55-71% of the dose in males and 50-52% in females. In both males and females, the concentration of excreted radiolabel peaked after 12-24 h, but animals given the repeated low dose excreted the radiolabel more rapidly than those given single doses. The concentrations of radiolabel in tissues represented a greater proportion of the administered dose in males than in females given the single doses: 0.46 and 0.35% for males and females at the low dose and 0.87 and 0.57% at the high dose, respectively, while the values were similar after the repeated doses: 0.57 and 0.59% for males and females, respectively.

Pyrethrin 1 is metabolized in rats by cleavage of the ester bond to form the corresponding acid and alcohol and by oxidation at a number of sites. The parent compound and five metabolites were identified in excreta. The major metabolite in urine after all dosing regimens was chrysanthemic dicarboxylic acid, and the compounds excreted predominantly in faeces were pyrethrin 1 and metabolite E, a dihydrodiol product of pyrethrin 1 (with oxidation on the vinyl group of the alcohol portion of the molecule), via formation of a monocarboxylic acid intermediate. Chrysanthemic dicarboxylic acid and metabolite E represented over one-third of the radiolabel excreted with all three regimens in both male and female rats. Males and females metabolized pyrethrin 1 similarly, regardless of the dose.

In another study, the percent of radiolabel excreted in urine and faeces and the percent of the dose represented by chrysanthemic dicarboxylic acid in urine did not differ appreciably when rats received the compound in corn oil, food slurry, or dimethyl sulfoxide. Repeated administration of high doses of pyrethrin apparently decreased the percent total radiolabel excreted and the percent excreted as chrysanthemic dicarboxylic acid.

*Lactating goats* received [$^{14}$C]pyrethrin 1 by gavage at a dose of 7.6, 8.3 (dietary burden, 10 ppm), or 179 mg/kg bw (dietary burden, 300 ppm) or dermally as a 1.8% oil- or water-based formulation The goats given the low oral dose or the dermal dose received [$^{14}$C]pyrethrin 1 once a day for 5 days, and those given the high dose received it once a day for 3 days. *Laying hens* were dosed orally for 5 days at 7.7 or 475 ppm or treated dermally with a 1% oil- or water-based solution.

Most of radiolabel in goats and hens treated orally was found in the excreta (75 and 89% of the administered radiolabel, respectively). Goats treated dermally retained 44-72% of the dose on the application site, while hens retained 12-37% of the dose. Milk from the goat given the high oral dose

contained up to 2.8 mg/kg equivalents of pyrethrin 1 after 24-36 h, while that of goats given the low dose contained 0.10 mg/kg. In the milk of animals treated dermally, the radiolabel represented 0.003-0.007 mg/kg with the water-based solution and 0.010-0.014 mg/kg with the oil-based solution. In goat tissues, the concentrations of radiolabel in liver (7.7 mg/kg pyrethrin equivalents), kidney (7.3 mg/kg), and fat (3.6 mg/kg) were highest with the high oral dose. Muscle of these animals contained 0.45-0.48 mg/kg of pyrethrin equivalents. In goats at the low dose, the concentrations of radiolabel in fat, liver, and kidney were 0.36-0.42 mg/kg, and those in muscle were 0.02-0.03 mg/kg. Fat contained the highest concentration in the dermally treated goats (0.08 and 0.04 mg/kg).

Up to the second day, the radiolabel was found mainly in the white of eggs of treated hens, with 0.93 mg/kg of pyrethrin equivalents at the high oral dose and 0.002-0.006 mg/kg with the low oral dose and dermal treatment. After 48 h, the radiolabel was concentrated in the yolk, representing 1.6-4.3 mg/kg at termination of the study in the group given the high oral dose and 0.010-0.05 mg/kg in those given the low oral and dermal treatments. The concentration of radiolabel in tissues of birds given the high oral dose ranged from 1.4 mg/kg in muscle to 15 mg/kg in liver. In those given the low oral dose, most of the radiolabel was found in gizzard (1.1% of the administered dose), kidney (0.42%), and liver (0.34%). In the tissues of dermally treated hens, most of the radiolabel was found in treated skin (3.8 and 5.3%) and fat (0.19 and 0.15% of the administered dose). In both goats and hens, pyrethrin 1 can undergo hydrolysis to form *trans*-chrysanthemic acid, which is readily conjugated *in vivo* to the corresponding □-glucuronic conjugate and to other conjugates of the free acid. Pyrethrin 1 is converted by oxidation to a corresponding monocarboxylic acid derivative, which, like the parent, can be oxidized to a dihydrodiol (metabolite E). Both of these metabolites can be hydrolytically converted to chrysanthemic dicarboxylic acid. The parent molecule can also undergo reduction at the □,□-unsaturated ketone position to form metabolite G.

Goats and hens given the low oral or dermal dose had low concentrations (<0.2 mg/kg) of pyrethrin 1 and its metabolites in all edible products. Those at the high dose had the highest concentrations of parent compound in fat (2.3 mg/kg in goats and 8.8 mg/kg in hens), milk (1.5 mg/kg), and eggs (0.97 mg/kg). In goats, no metabolites were detected in milk (<0.01 mg/kg), while liver and kidney had the highest concentrations of individual metabolites (0.078-3.3 mg/kg). Chrysanthemic acid was the major metabolite in eggs (0.39 mg/kg) and liver (3.0 mg/kg) of hens given the high dose.

The metabolism of pyrethrins in animals and birds thus involves hydrolysis of the ester bond and oxidation at various sites. The main metabolite in rat excreta is chrysanthemic dicarboxylic acid. The parent compound, chrysanthemic acid, monocarboxylic acid, and dicarboxylic acid were also present in milk and eggs. In goats, chrysanthemic acid represented up to 7% of the residue in muscle, up to 15% in liver, and three times the concentration of the parent compound in kidney. In hens, the concentration of chrysanthemic acid was as much as 10 times that of the parent compound in liver.

*Plants*

The fate of pyrethrins after five foliar applications of [$^{14}$C]pyrethrin 1 on *leaf lettuce*, *potatoes*, and *tomatoes* at 0.56 kg ai/ha (10 times the GAP rate) was investigated. The plants were placed in boxes lined with polyethylene sheeting and exposed to sunlight in a greenhouse with a translucent plastic roof and sides composed of bird- and rodent-proof wire. Tomato leaves and fruit and potato leaves and tubers were collected 5 days after treatment, and lettuce leaves 0 and 10 days after treatment.

Pyrethrin 1 degraded extensively, yielding at least eight and as many as 19 extractable metabolites, showing similar metabolic pathways in each crop. The identified metabolites were chrysanthemic acid derivatives produced by cleavage of the ester bond.

Only minimal uptake or translocation of pyrethrin 1 and its degradation products occurs, probably because of the relatively high lipophilicity (log $P_{OW}$ ~ 6) of the parent compound, which results in little tendency to cross the cuticle of plant surfaces and enter the largely aqueous regions in which metabolism by enzymes can occur. The concentrations of total radiolabel in potato and tomato leaves (550 and 365 mg/kg pyrethrin equivalents, respectively) were 1000 and 200 times higher than those in the tuber and fruit, respectively. In lettuce, the concentrations of radiolabel (36 mg/kg) and pyrethrin 1 (14 mg/kg) at day 0 had decreased substantially 10 days after the last application, to 10 and 0.25 mg/kg, respectively. The concentrations of pyrethrin 1 and its metabolites in tomato fruit and potato tubers (<0.004-0.42 mg/kg) were 0.03-8% of the corresponding values in leaves. The lower concentrations of both total residues and parent pyrethrin 1 in potato tubers than in tomato fruit reflect the limited direct application to tomatoes, which are sheltered by the leaf canopy, whereas in potatoes the radiolabel can reach the tubers only by translocation. The concentrations of all metabolites found in lettuce decreased between day 0 and day 10, except that of cyclopropyl-substituted hydroxychrysanthemic acid, which increased from 1.5 mg/kg pyrethrin equivalents to 2.1 mg/kg within the same period.

***Environmental fate***

*Degradation in soil*

The half-life of [$^{14}$C]pyrethrin 1 applied at a rate of 10 mg/kg to sandy loam soil exposed to natural sunlight at 24 $^{o}$C for up to 24 h was 12.9 h, and no degradate contained >10% of the applied radiolabel. Only $CO_2$ was identified.

The degradation of [$^{14}$C]pyrethrin 1 was studied after application at a rate of 1.0 mg/kg to a sandy loam soil under aerobic conditions at 25 ± 1 $^{o}$C in the dark for up to 181 days. Pyrethrin 1 and extractable species metabolized to bound residues and thereafter to $CO_2$, with a half-life of about 2.2 days. Chrysanthemic acid was identified in organic extracts of the treated soil, its concentration reaching a maximum of about 4% of the initial concentration of pyrethrin 1 after 3 days. Three other degradates were observed at concentrations of 5-10% of the amount of pyrethrin 1 applied. About 6-10% of the applied radiolabel was present as humic acid, fulvic acid, and humin fractions.

The terrestrial dissipation of pyrethrins applied as an end-use formulation to bare soil was studied in California, Georgia, and Michigan (USA) at a nominal rate of 0.52 kg ai/ha (total amount of pyrethrins applied throughout a season). The half-lives were 1-2 h, and within 1 day of application pyrethrin had dissipated in the 0-15-cm soil horizon to below the limit of detection, 0.10 mg/kg for total pyrethrins. Pyrethrin 1 was not detected at depths below 15 cm.

The volatility of [$^{14}$C]pyrethrin 1 applied at a rate of 0.56 kg ai/ha (10 times the label rate) to sandy loam soil was studied at 50 and 75% of the field moisture capacity and flow rates of 100 and 300 ml/min. After 30 days, most the radiolabel remained in the soil, 37-43% having been extracted and 33-38% bound. [$^{14}$C]Pyrethrin 1 in soil extracts represented 1.9% (75% moisture, 300 ml/min) to 9.1% of the applied dose (50% moisture, 100 ml/min). Four degradates that were soluble in organic solvents were observed in the extracts. The radiolabel trapped in ethylene glycol was mainly associated with chrysanthemic acid, in all but one test system, with two other organic degradates (representing 4-9% of the applied radiolabel) and with pyrethrin 1 (<0.05-0.19% of the applied dose). The volatility rates were not affected by air flow or by the amount of moisture in the soil. The

volatility from soil of pyrethrin *per se* was considerably lower (2-12 $\cdot 10^{-6}$ µg/cm$^2$ per h) than that of all volatile components (0.001 mg/cm$^2$ per h).

The adsorption and desorption of [$^{14}$C]pyrethrin 1 were studied at concentrations of 0.05, 0.09, 0.50, and 0.81 mg/kg in sandy loam, silty clay loam, silt loam, and sand soils. A 1:100 ratio of soil to solution and a 3-h equilibration time were used for both adsorption and desorption experiments. The adsorption constants varied from 268 to 430 and the desorption constants from 965 to 2600. The $K_{OC}$ value ranged from 12 472 to 448 257, indicating that pyrethrin 1 is immobile.

## 1. Fate in water and sediment systems

The aqueous photolysis of a test solution of [$^{14}$C]pyrethrin 1 containing 0.3 mg/kg at pH 7 that was exposed to natural sunlight for up to 72 h at 25 ± 1 °C was investigated. Within 1 h of exposure, the concentration of pyrethrin 1 had decreased to 47%, and that of the *E* isomer reached 44% of the applied dose. After 30 days, these values were 7 and 12%, respectively. The overall photolytic half-life for the two isomers was 11.8 h.

The hydrolysis of [$^{14}$C]pyrethrin 1 at 0.4 mg/kg in buffered aqueous solutions at pH 5, 7, and 9 was investigated for 30 days in the dark at 25 °C. Pyrethrin 1 was stable at pH 5 and 7 (5% degradation); at pH 9, the half-life was 17 days. At this pH, approximately 35% of the radiolabel was found as pyrethrin 1 after 30 days, and 61% as chrysanthemic acid, the main radiolabelled degradate. A single non-radiolabelled degradate, a dimer of a relative molecular mass of 320, was isolated. The proposed pathway for formation of the dimer involves hydrolysis of pyrethrin 1 to pyretholone, rapid elimination of water from pyretholone to form the corresponding cyclopentadienone, and Diels-Alder condensation of the cyclopentadienone to form the observed dimer.

The anaerobic and aerobic aquatic degradation of [$^{14}$C]pyrethrin 1 was studied at 25 ± 1 °C in a dark system prepared from sandy loam hydrosoil. The treatment rate was approximately 1 mg/kg, and incubation proceeded for 364 days under anaerobic and for 30 days under aerobic conditions. Pyrethrin 1 degraded with a half-life of 86 and 10.5 days under anaerobic and aerobic conditions, respectively. The concentration of radiolabel increased during incubation in the supernatant but decreased in soil, as the bound residues and the amount lost as $CO_2$ increased. After 30 days, the concentration of radiolabel in soil under anaerobic conditions was higher in the organosoluble fraction (55%) than in bound residues (31%). Under aerobic conditions, the situation was reversed, 51% of the applied radiolabel being bound and 22% in the organosoluble fraction in the same period. After 364 days under anaerobic conditions, 24% of the applied radiolabel was present in the supernatant and 62% in soil, mostly bound or as $CO_2$.

The principal extractable species under anaerobic conditions were pyrethrin 1 and three degradates, chrysanthemic acid, cyclopropane diacid, and jasmolin 1. Chrysanthemic acid appeared after 14 days (representing 9.2% of the applied dose); the concentration remained stable until day 270 and decreased to 5.3% by 364 days. Cyclopropane diacid was detected at day 270, representing 11% of the applied dose, increasing to 15% at the end of the study. Jasmolin 1 was first detected at day 90 at concentrations that remained stable until the end of the study (9-10 % of the applied dose).

Under aerobic conditions, the principal extractable species were pyrethrin 1 and chrysanthemic acid, corresponding to 72 and 2.5% of the applied dose, respectively, 3 days after application. The concentration of pyrethrin reached a minimum after 30 days (15% of the dose), and that of chrysanthemic acid reached a maximum after 21 days (22% of the dose). In both anaerobic and aerobic systems, radiolabel was present in humic acid, fulvic acid, and humin fractions, corresponding to approximately 6-23% of the applied dose. Chrysanthemic acid was found in the fulvic acid fraction.

Thus, pyrethrin 1 is an immobile compound with low volatility. In water in the dark, it degrades with a half-life of 10.5 days under aerobic conditions and 86 days under anaerobic conditions, while the photolytic half-life is ~ 12 h. Degradation occurs more rapidly under basic conditions, with a half-life of 17 days at pH 9. In soil, pyrethrin 1 degrades in the dark with a half-life of 3.2 days, while the photolysis half-life is 13 h. The main metabolite detected in water and soil was chrysanthemic acid.

### *Methods of analysis*

Methods for determining residues of pyrethrins in vegetable and animal products were presented. In all methods, the extracts are analysed by gas chromatography with electron capture detection. Pyrethrins I are quantified by summing the responses of the three esters, pyrethrin 1, cinerin 1, and jasmolin 1, which are determined individually in the chromatographic method. No adjustment is made for the specific responses of the esters in the electron capture detector (pyrethrin 1 has a stronger response) or for relative molecular mass. As pyrethrin II esters degrade during analysis, the concentration of total pyrethrins is estimated from the proportions of esters of pyrethrins I and pyrethrins II in the formulation. In food trials, where the concentration of pyrethrins II represents 81.5% of that of pyrethrins I, the concentrations of pyrethrins I were multiplied by 1.81 to obtain the corresponding concentrations of total pyrethrins. In animals, where the concentration of pyrethrins II represents 92.0% of that of pyrethrins I, the concentrations of the latter were multiplied by 1.92 to obtain the corresponding concentrations of total pyrethrins. A world standard pyrethrum extract is available for calibration of the method from the Pyrethrum Board of Kenya.

Samples of raw and processed agricultural commodities are extracted in an organic solvent and cleaned up by silica gel adsorption or silica gel-alumina adsorption. The LOQ for total pyrethrins was 0.04 mg/kg in all matrices. The recovery of pyrethrins I ranged from 61 to 139%, with a mean of 93%.

In a second method, used for food items treated in warehouses, samples are extracted with either an organic solvent and water for low-fat foods (navy beans and prunes) or an organic solvent for high-fat foods (peanuts), followed by clean-up with liquid-solid partition. The LOQ for pyrethrins I was 0.1 mg/kg, and the limit of detection was 0.05 mg/kg. The method was validated for each matrix by analysis of at least four fortifications. The reported recoveries ranged from 65 to 120%.

A third method was used to analyse edible products from laying hens and dairy cattle and is also applicable for enforcement of tolerances for pyrethrins in animal-derived commodities. In this method, samples are extracted with an organic solvent, and the extract is cleaned up by silica gel adsorption or silica gel-alumina adsorption (for liver, kidney, skin, muscle, eggs, and fat). The LOQs were 0.02 mg/kg for milk and eggs and 0.04 mg/kg for all animal tissues, as total pyrethrins. The recoveries ranged from 67 to 112% at 1, 10, and 100 times the LOQ, with standard deviations of 1.5 to 10%.

### *Stability of residues in stored analytical samples*

The stability of pyrethrin residues in frozen samples was examined in representative commodities for which trials at 1 mg/kg were submitted. In samples of broccoli, bean pods, vines, and hay, dry orange pulp, dry and wet tomato pomace, liver, and kidney, only 35-70% of the concentration of pyrethrins remained after 12-27 months of storage. In all the other commodities, pyrethrin was stable, >80% remaining after storage.

***Definition of the residue***

On the day of application, pyrethrin 1 is the major compound in lettuce, chrysanthemic dicarboxylic acid being the only degradation product present at a level >10% of that of the pyrethrin residue (17%). Pyrethrin 1 is degraded extensively by photolysis within 10 days after application to plants, and no predominant metabolite is formed. The toxicological evaluation of pyrethrins (Annex 6, reference 86) was based on studies conducted with pyrethrum extract. The ADI and acute RfD derived take into account the toxicity of metabolites of the six related esters.

The Meeting agreed that the residue definition for compliance with the MRLs and for estimating dietary intake is total pyrethrins, calculated as the sum of the six biologically active pyrethrin esters: pyrethrin 1, pyrethrin 2, cinerin 1, cinerin 2, jasmolin 1, and jasmolin 2, after calibration with the World Standard Pyrethrum Extract.

Pyrethrins are fat-soluble, with log $P_{OW}$ values of 5.9 for pyrethrin 1 and 4.3 for pyrethrin 2.

***Results of supervised trials***

All of the available trials were conducted in the USA during 1992-96 according to the maximum GAP of 10 foliar applications of 0.056 kg ai/ha with no PHI, unless otherwise specified. As esters of pyrethrins II degrade during analysis, total pyrethrin concentrations are estimated from the proportions of esters of pyrethrins I and pyrethrins II in the formulation (pyrethrins II representing 81.5% of that of pyrethrins I) and multiplying the concentrations of pyrethrins I by 1.81.

Seven trials were conducted with *citrus* fruit: two on *lemon*, three on *orange*, and two on *grapefruit*. The concentrations of residues (median in italics) were *<0.04* (6 trials) and 0.04 mg/kg. The Meeting agreed that, although seven trials are normally considered to be too few to allow recommendation of an MRL for a major commodity such as citrus, the concentrations of residues found, which were below or at the LOQ, reflect the amounts of pyrethrin residues remaining after foliar application according to GAP. The Meeting recommended an MRL of 0.05 mg/kg, an STMR value of 0.04 mg/kg, and a HR value of 0.04 mg/kg for pyrethrins in citrus.

One trial was conducted on *blackberry*, two on *blueberry*, and one on *cranberry*, in which the concentrations of residues in fruit were 0.10, 0.08, 0.07, and 0.05 mg/kg, respectively. Two trials were conducted on *strawberries*, giving residue concentrations of 0.11 and 0.12 mg/kg, and one trial was carried out on grapes, with a concentration of 0.17 mg/kg. As insufficient data were available from trials performed according to GAP, the Meeting could not recommend an MRL for pyrethrins in berries, strawberries, or grapes.

Three trials were conducted on *broccoli*, giving residue values of <0.04, 0.06, and 0.08

mg/kg. In three trials on *cabbage*, the residue concentrations were 0.05, 0.12, and 0.39 mg/kg. Cabbage heads with wrapper leaves removed had a residue concentration <0.04 mg/kg. As insufficient data were available from trials performed according to GAP, the Meeting could not recommend an MRL for pyrethrins in broccoli and cabbage.

Eight trials were conducted on cucurbits: two on *cantaloupe*, two on *cucumber*, and four on *summer squash*. The concentrations of residues in fruit were <0.04 (7 trials) and 0.04 mg/kg. The Meeting agreed to recommend an MRL of 0.05 mg/kg, an STMR value of 0.04 mg/kg, and a HR value of 0.04 mg/kg for pyrethrins in fruiting cucurbits.

Three trials were conducted on *pepper* and three on *tomato*, giving residue concentrations <0.04 mg/kg in the fruit. The Meeting agreed that residues in fruiting cucurbits can be used to support the data on peppers and tomatoes and recommended an MRL of 0.05 mg/kg, an STMR value of 0.04 mg/kg, and a HR value of 0.04 mg/kg for pyrethrins in tomatoes and peppers.

Nine trials were conducted on leafy vegetables. The concentrations of residues were 0.08 and 0.16 mg/kg in *head lettuce*, 0.52 and 0.56 mg/kg in *leaf lettuce*, 1.8 mg/kg in *radish* leaves, 0.75 and 1.0 mg/kg in *spinach*, and 0.64 and 0.90 mg/kg in *mustard greens*. As the values in the different commodities are not within the same range, they could not be combined. As insufficient data from trials performed according to GAP were submitted, the Meeting could not recommend an MRL for pyrethrins in lettuce, radish leaves, spinach, and sugar beet leaves.

In two trials conducted on succulent *bean*, the residue concentrations in seeds with pods were <0.04 and 0.13 mg/kg. In two trials on succulent *pea*, the concentrations in seeds with pods were <0.04 and 0.09 mg/kg. As insufficient data from trials performed according to GAP were submitted, the Meeting could not recommend an MRL for pyrethrins in succulent beans and peas.

Seven trials were conducted on root and tuber vegetables: one on *carrot*, three on *potato*, two on *radish*, and four on *sugar beet*. The concentration of residues in the roots of all commodities was <0.04 mg/kg. A study of metabolism in potatoes treated with 10 times the maximum label rate showed that the concentration of pyrethrin 1 in tubers was 0.004 mg/kg 5 days after application. The Meeting agreed that it is unlikely that residues would be present in roots after a 0-day PHI and recommended an MRL of 0.05* mg/kg, an STMR value of 0, and a HR value of 0.04 mg/kg for pyrethrins in root and tuber vegetables.

Two trials were conducted on *celery*, giving residue concentrations of 0.16 and 0.70 mg/kg. When the leaves were removed, these values fell to <0.04 and 0.07 mg/kg, respectively. As insufficient data were available from trials performed according to GAP, the Meeting could not recommend an MRL for pyrethrins in celery.

One trial was conducted on *mustard seed*, in which the concentration of residues was <0.04 mg/kg. As insufficient data were available from trials performed according to GAP, the Meeting could not recommend an MRL for pyrethrins in mustard seeds.

In four trials conducted on beans, the concentrations of residues in *bean vine*, in rank order, were 0.08, 0.22, 0.38, and 1.6 mg/kg. In *bean hay* samples dried for 2-6 days in the open air, the concentrations were 0.08, 0.09, 0.43, and 0.48 mg/kg. The concentrations in forage were 0.24 and 0.32 mg/kg. Residues were measured in *pea vines* in four studies, the concentrations of residues being 0.16, ***0.53***, ***0.62***, and 0.82 mg/kg, and those in *pea hay* dried for up to 14 days in the field or in a greenhouse were 0.03, 0.07, 0.45, and 0.46 mg/kg. The concentrations in forage were 0.62 and 1.6 mg/kg.

The Meeting agreed that residues in bean vines are within the same population as residues in pea vines and can be used to support a recommendation for pea vines. The concentrations were 0.08, 0.16, 0.22, ***0.38***, ***0.53***, 0.62, 0.82, and 1.6 mg/kg of fresh vine. When the median (0.53 mg/kg) and the maximum (1.6 mg/kg) values were corrected for moisture content (75%, FAO Manual, p. 125), they were 2.15 and 6.4 mg/kg, respectively, of dry matter. The Meeting recommended an MRL of 10 mg/kg and an STMR value of 2.15 mg/kg for pyrethrins in dried pea vines.

The Meeting agreed that residues in bean and pea hay represent a single residue population and can be combined. The concentrations were 0.03, 0.07, 0.08, ***0.09***, ***0.43***, 0.45, 0.46, and 0.48 mg/kg of fresh weight. When the median (0.26 mg/kg) and the maximum (0.48 mg/kg) values were corrected for the moisture content of pea hay (12%, FAO, 1997, p. 125), the values were 0.295 and 0.545 mg/kg, respectively, of dry weight. The Meeting recommended an MRL of 1 mg/kg and an STMR value of 0.295 mg/kg for pyrethrins in pea hay or fodder.

Two trials were conducted on *sugar beet leaf*, giving residue concentrations of 0.05 and 0.08 mg/kg. As insufficient data from trials performed according to GAP were submitted, the Meeting could not recommend an MRL for pyrethrins in sugar beet leaves.

Twenty trials were conducted with bagged *navy bean* treated in a warehouse with pyrethrins at up to 10 applications of the label rate by a space spray (0.05 kg ai/1000 $m^3$) and by contact spray (0.003 kg ai/100 $m^2$). The concentration of residues in samples collected after each treatment was <0.05 mg/kg (limit of detection). The LOQ in the trials was 0.10 mg/kg.

In two trials conducted on *dried bean* and two on *dried pea* treated by foliar application, the concentration in seeds was <0.04 mg/kg. The Meeting recommended an MRL of 0.1 mg/kg, an STMR value of 0.05, and a HR value of 0.05 mg/kg for pyrethrins in pulses based on post-harvest use.

Twenty trials were conducted on harvested *peanut* treated in a warehouse with 10 applications at the label rate by a space spray (0.05 kg ai/1000 $m^3$) and by contact spray (0.003 kg ai/100 $m^2$). The concentrations in samples collected after each treatment with a space spray were <0.05 (3 trials) (limit of detection), <0.10 (3 trials) (LOQ), 0.12, 0.16, 0.21, and 0.23 mg/kg. Under contact spray conditions, the concentration was <0.05 (10 trials) mg/kg. The concentrations after post-harvest use were ***<0.05*** (13 trials), <0.10 (3 trials), 0.12, 0.16, 0.21, and 0.23 mg/kg. The Meeting recommended an MRL of 0.5 mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.23 mg/kg for pyrethrins in peanuts after post-harvest treatment.

Trials were conducted with bagged, harvested *prunes* treated in a warehouse with 10 applications at the label rate by a space spray (0.003 kg ai/1000 $m^3$) and by contact spray (0.003 kg ai/100 $m^2$). The concentrations in samples collected after each treatment under space spray conditions were <0.05 (8 trials) (limit of detection), <0.10 (LOQ), and 0.11 mg/kg. Under contact spray conditions, the concentrations were <0.05 (8 trials) and <0.10 (2 trials) mg/kg. The concentrations after post-harvest use were ***<0.05*** (16 trials), <0.10 (3 trials), and 0.11 mg/kg. The Meeting noted that the residues in prunes can be extensive and recommended an MRL of 0.2 mg/kg, an STMR value of 0.05 mg/kg, and a HR value of 0.11 mg/kg for pyrethrins in dried fruits after post-harvest treatment.

***Fate of residues during processing***

*Oranges* treated 10 times at 0.28 kg ai/ha (five times the label rate) were processed in a laboratory into juice, molasses, dry peel (dry pulp), and oil, simulating commercial operations. The concentrations of residues were 0.06 mg/kg in fruit, decreased in molasses with a processing factor of 0.69, and not detected in juice (processing factor, <0.66). The residue was concentrated in dry pulp, with a processing factor of 7.5, and in oil, with a factor of 20.3. On the basis of an STMR value of 0.04 mg/kg and the processing factors derived, the Meeting estimated an STMR-P value of 0.026 for citrus juice, 0.0276 for citrus molasses, 0.300 for dry citrus pulp, and 0.812 for citrus oil.

*Grapes* treated 10 times at 0.28 kg ai/ha (five times the label rate) were processed in a laboratory into juice, simulating commercial operations, into wet and dry pomace, and into raisins. The concentration of residues in fruit (0.08 mg/kg) increased after processing to wet and dry pomace, with processing factors of 1.32 and 5.03, respectively. Residues were not detected in raisins, raisin waste, or juice (processing factor, <0.48).

*Tomatoes* treated 10 times at 0.28 kg ai/ha (five times the label rate) were processed in a laboratory, simulating commercial operations, into wet pomace, dry pomace, purée, and juice. The concentration of residues in fruit (0.08 mg/kg) increased after processing to wet and dry pomace, with processing factors of 8.8 and 29.0 respectively. No residues were detected in purée or juice (processing factor, <0.48). On the basis of an STMR value of 0.04 mg/kg and the processing factors derived, the Meeting estimated STMR-P values of 0.352 for wet pomace, 0.808 for dry tomato pomace, and 0.018 for tomato juice and tomato purée.

Succulent *beans* treated 10 times at 0.28 kg ai/ha (five times the label rate) were processed into cannery waste as a composite sample of leaves, whole pods, and pod tips. The concentrations of residues were 0.34 mg/kg in pods and 1.2 mg/kg in cannery waste (processing factor, 3.5).

*Potatoes* treated 10 times at 0.28 kg ai/ha (five times the label rate) were processed in a laboratory by separate procedures simulating commercial practice, into chips, wet peel from the granule-making process, and granules, equivalent to flakes. No residues were detected in raw or processed commodities.

*Sugar beets* treated 10 times at 0.28 kg ai/ha (five times the label rate) were processed in a laboratory, simulating commercial operations, into dehydrated pulp, molasses, and refined sugar. No residues were detected in raw or processed commodities.

***Residues in animal commodities***

*Lactating dairy cows* were given pyrethrin orally and dermally daily for up to 27 days, at an oral dose of 5, 15, or 50 mg/kg and a dermal dose of 89 mg/day. The concentration of pyrethrins in milk peaked between 7-11 days at 0.03, 0.09, and 0.20 mg/kg at the three doses, respectively. At the low and medium dose, these concentrations remained approximately the same until the end of the study; at the high dose, the concentration decreased to 0.11 mg/kg at day 27. Residues were detected only in fat of animals at the low dose, but concentrations of 0.05-1.5 mg/kg were found in liver, kidney, muscle, and fat of animals at the higher doses.

The dietary burden was calculated from the MRL and STMR values for pea vines estimated by the Meeting (10 and 2.15 mg/kg, respectively) and the percent of the diet of dairy cows (50%) as described in the *FAO Manual* (FAO, 1997, pp. 121-127). The dietary burden based on the MRL is 5 ppm, and that based on the STMR value is 1.1 ppm.

The Meeting agreed that dermal exposure can contribute to residues in animal commodities,

as the study of metabolism in goats treated dermally with a 1.8% oily solution showed detectable residues in milk, liver, and fat (0.010, 0.002, and 0.013 mg/kg, respectively). The Meeting also agreed that the concentrations of residues found in studies in which animals were exposed orally and dermally are overestimates, as it is unlikely that animals would be exposed by both routes on a daily basis. The Meeting considered that an estimate of a maximum residue level for pyrethrins in cattle commodities was precluded.

*Hens* were dosed both dermally and orally with pyrethrin 1 for 25-27 days. The oral doses were 3, 9, and 30 mg/kg in the diet, and the dermal dose was 332 mg/28 $m^3$ [~ 12 mg/$m^3$] per day, expressed as total pyrethrins, representing the maximum label rate for spraying of premises. Except for one sample from a bird at the intermediate dose, which contained a concentration of 0.02 mg/kg on day 3, residues of pyrethrins in eggs were only just detectable on day 7 after the highest dose, at concentrations of 0.02-0.04 mg/kg. The concentrations of residues in edible tissues of birds at all doses were at or around the LOQ (0.038 mg/kg) in liver and muscle. In skin, they were 0.18, 0.17, and 0.25 mg/kg, and those in fat were 0.06, 0.23, and 0.27 mg/kg at the three doses, respectively.

No recommendations are available for commodities used for poultry feed that would allow calculation of the dietary burden for poultry. The Meeting agreed that it is unlikely that hens would be exposed to pyrethrins both orally and dermally on a daily basis and that the concentrations in poultry commodities derived from such studies will be overestimates. The Meeting considered that an estimate of a maximum residue level for pyrethrins in poultry commodities was precluded.

### *Residues in food in commerce or at consumption*

A total of 745 domestic and imported samples of food products in the USA were analysed in 1998 for residues of pyrethrins. They were not detected in any of the products.

### *National maximum residue limits*

MRLs were provided from 33 countries in Africa, Asia, Europe, and North America. The values ranged from 0.05* to 5 mg/kg. The residue definition is the sum of the six esters, as total pyrethrins.

## RECOMMENDATIONS

The Meeting estimated the maximum residue levels and STMRs shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and estimation of dietary intake: total pyrethrins, calculated as the sum of pyrethrins 1 and 2, cinerins 1 and 2, and jasmolins 1 and 2, determined after calibration with the World Standard Pyrethrum Extract.

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| GC 0080 | Cereal grains | W | 3Po | | |
| FC 0001 | Citrus fruits | 0.05 | - | 0.04 | 0.04 |
| JF 0001 | Citrus juice | | | 0.026 | |

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| DM 0001 | Citrus molasses | | | 0.0276 | 0.0276 |
| AB 0001 | Citrus pulp, dry | | | 0.342 | 0.342 |
| | Citrus oil | | | 0.812 | 0.812 |
| MD 0180 | Dried fish | W | 3Po | | |
| DF 0167 | Dried fruits | 0.2Po | 1Po | 0.05 | 0.11 |
| DV 0168 | Dried vegetables | W | 1Po | | |
| VC 0045 | Fruiting vegetables, cucurbits | 0.05* | | 0.04 | 0.04 |
| SO 0088 | Oilseed | W | 1Po | | |
| AL 0072 | Pea hay or Pea fodder (dry) | 1 | - | 0.295 | |
| AL 0528 | Pea vines (green) | 10 (dry wt.) | - | 2.15 (dry wt.) | |
| SO 0697 | Peanut | 0.5 Po | - | 0.05 | 0.23 |
| VO 0051 | Peppers | 0.05* | - | 0.04 | 0.04 |
| VD 0070 | Pulses | 0.1 | - | 0.05 | 0.05 |
| VR 0075 | Root and tuber vegetables | 0.05* | - | 0 | 0.04 |
| VO 0448 | Tomato | 0.05* | - | 0.04 | 0.04 |
| | Tomato pomace, dry | | | 0.808 | 0.808 |
| | Tomato pomace, wet | | | 0.352 | 0.352 |
| JF 0448 | Tomato juice | | | 0.018 | |
| | Tomato purée | | | 0.018 | |
| TN 0085 | Tree nuts | W | 1Po | | |

## Further work or information

*Desirable*

Feeding studies in ruminants.

## Dietary risk assessment

### *Chronic intake*

The ADI for pyrethrins is 0-0.04 mg/kg bw. The international estimated daily intake was calculated for commodities consumed by humans for which STMR values were estimated by the Meeting. The results are shown in Annex 3. The international estimated daily intakes from the five GEMS/Food regional diets, based on estimated STMR values represented 0% of the ADI. The Meeting concluded that the intake of residues of pyrethrins resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

### *Short-term intake*

The acute RfD for pyrethrins is 0.2 mg/kg bw. The IESTI was calculated for the commodities for which STMR and HR values were estimated and for which data on consumption were available. The results are shown in Annex 4. The IESTI represented 0-3% of the acute RfD for the general population and 0-8% of that for children.

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# PYRIPROXYFEN (200)

## EXPLANATION

Pyriproxyfen was first evaluated in 1999 and MRLs were recommended for citrus fruits, cotton seed and its processed commodities, and animal commodities. That Meeting requested information on the fate of pyriproxyfen during the processing of oranges and at a late stage of the Meeting results of an orange processing trial were submitted. As there was no time for evaluation, it was held over until the present Meeting.

## METHODS OF RESIDUE ANALYSIS

### Analytical methods

Green (1998) described various modifications of methods RM-33P-1 and RM-33M-2 for the determination of residues of pyriproxyfen and 4'-OH-Pyr (4-(4-hydroxyphenoxy)phenyl (RS)-2-(2-pyridyloxy)propyl ether) in oranges, peel and pulp. These are variations of methods described by the 1999 JMPR which take account of the type of sample. Pyriproxyfen is determined by GLC with a nitrogen-phosphorus detector, and 4'-OH-Pyr by HPLC with a fluorescence detector. Recoveries from oranges of pyriproxyfen at fortifications of 0.02 and 0.10 mg/kg were mean 91%, range 66-111%, n=27, and of 4'-OH-Pyr at fortifications of 0.02-0.50 mg/kg were mean 88%, range 73-103%, n=16, some of the latter involved fortification with the conjugate 4'-Glu-Pyr. Recoveries of both compounds were satisfactory from fortified dried pulp, oil, oranges, fresh peel and peeled fruit.

### Stability of residues in stored analytical samples

The storage stability of pyriproxyfen and 4'-OH-Pyr residues in oranges was also tested (Green, 1998). 20 g samples of orange fortified with pyriproxyfen at 0.10 mg/kg were stored in re-sealable plastic bags in a freezer at approximately -20°C and analysed at intervals up to 122 days. 30 g samples of orange fortified with 4'-OH-Pyr at 0.10 mg/kg were stored in the same manner and analysed at intervals up to 90 days. The residues were stable under these conditions (Table 1).

Table 1. Frozen storage stability of pyriproxyfen and 4'-OH-Pyr in orange samples fortified at 0.10 mg/kg (Green, 1998) and stored at approximately -20°C. The reported remaining residue is not adjusted for procedural recoveries.

| Pyriproxyfen | | | 4'-OH-Pyr[1] | |
|---|---|---|---|---|
| Storage period, days | Residue | Procedural recovery, % | Storage period, days | Remaining residue |
| 0 | 0.095 0.095 | 95 95 | 0 | 0.086 0.082 0.084 |
| 36 | 0.100 0.103 | 84 | 64 | 0.099 0.107 |
| 58 | 0.091 0.079 | 89 | 90 | 0.089 0.089 |
| 91 | 0.081 0.083 | 97 | | |
| 122 | 0.078 0.098 | 94 | | |

[1]Procedural recoveries of 4'-OH-Pyr were affected by a contaminated control sample

## RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials on oranges in the USA.

Where residues were not detected, data are recorded in the tables as below the limit of quantification (LOQ), e.g. <0.01 mg/kg. Residue data, application rates and spray concentrations have generally been rounded to 2 significant figures or, for residues near the LOQ, to 1 significant figure. Although trials included control plots, no data are given because residues in control samples did not exceed the LOQ. The results are not corrected for analytical recoveries.

Oranges at sites in California, Florida and Texas were treated at intervals of 20-22 days with pyriproxyfen using airblast sprayers. Samples consisted of 24 mature fruit except in the processing trial where 200 kg fruit were harvested. Freezer storage periods (from sample collection to extraction for analysis) for orange samples ranged from 3 to 95 days.

Table 2. Residues of pyriproxyfen in oranges from supervised trials in the USA. Some analyses are reported at 0.01 mg/kg, but recoveries were not tested below 0.02 mg/kg. All EC formulations.

| Location, year (variety) | Application | | | | PHI, days | Residues, mg/kg | | Ref. |
|---|---|---|---|---|---|---|---|---|
| | kg ai/ha | kg ai/hl | Water, l/ha | No. | | pyriproxyfen | 4'-OH-Pyr | |
| FL, 1995 (Navel) | 0.12 | 0.0066 | 1900 | 3 | 1<br>7<br>21 | 0.18 0.13<br>0.12 0.13<br>0.10 0.11 | | V-11121-A |
| FL, 1995 (Navel) | 0.12 | 0.0066 | 1900 | 3 | 1<br>7<br>21 | 0.14 0.15<br>0.13 0.19<br>0.13 0.13 | | V-11121-B |
| CA, 1996 (Washington Navel) | 0.12 | 0.0066 | 1900 | 3 | 1<br>7<br>21 | 0.14 0.15<br>0.10 0.18<br>0.14 0.16 | | V-11121-C |
| CA, 1996 (Washington Navel) | 0.062 | 0.003 | 1900 | 3 | 1<br>7<br>21 | 0.10 0.09<br>0.11 0.09<br>0.09 0.10 | | V-11121-C |
| CA, 1996 (Lane Navel) | 0.12 | 0.0066 | 1800 | 3 | 1 | 0.09 0.05 | <0.05 (2) | V-11121-D |
| CA, 1996 (Olinda Navel) | 0.12 | 0.0066 | 1900 | 3 | 1 | 0.11 0.15 | <0.05 (2) | V-11121-E |
| FL, 1996 (Mid Sweet) | 0.12 | 0.0067 | 1900 | 3 | 1 | 0.21 0.20 | 0.02 0.02 | V-11121-F |
| TX, 1997 (Everhard Navel) | 0.12 | 0.0066 | 1900 | 3 | 1 | 0.13 0.14 | <0.01 (2) | V-11121-G |
| FL, 1997 (Navel) | 0.13 | 0.0065 | 1900 | 3 | 1 | 0.15 0.13 | <0.01 (2) | V-11121-H |
| FL, 1997 (Navel) | 0.12 | 0.0066 | 1900 | 3 | 1 | 0.12 0.14 | <0.01 (2) | V-11121-I |
| FL, 1997 (Navel) | 0.12 | 0.0068 | 1800 | 3 | 1 | 0.21 0.22 | <0.01 (2) | V-11121-J |
| FL, 1997 (Valencia) | 0.12 | 0.0069 | 1800 | 3 | 1 | 0.16 0.16 | 0.01 0.01 | V-11121-K |

| | Application | | | | | Residues, mg/kg | | |
|---|---|---|---|---|---|---|---|---|
| FL, 1998 (Rohde Red Valencia) | 0.12 | 0.0068 | 1800 | 3 | 1 | 0.21 0.23 | <0.01 (2) | V-11121-L |
| FL, 1998 (Rohde Red Valencia) | 0.25 | 0.0136 | 1800 | 3 | 1 | 0.40 0.36 | 0.02 0.01 | V-11121-L |
| FL, 1998 (Rohde Red Valencia) | 0.25 | 0.0136 | 1800 | 3 | 1 | 0.35<br>oil 26.1<br>pulp dry 2.22<br>juice <0.01 | 0.02<br>oil 1.2<br>pulp dry 0.11<br>juice <0.01 | V-11121-M |
| FL, 1998 (Valencia) | 0.12 | 0.0067 | 1800 | 3 | 1 | 0.21 0.23<br>pe 0.76 0.62<br>pu <0.01 (2) | 0.01 0.01<br>pe 0.03 0.02<br>pu <0.01 (2) | V-11121-N |
| FL, 1998 (Valencia) | 0.25 | 0.0134 | 1800 | 3 | 1 | 0.40 0.41<br>pe 2.1 2.1<br>pu 0.01 0.01 | 0.02 0.02<br>pe 0.05 0.06<br>pu <0.01 (2) | V-11121-N |

pe: fresh orange peel
pu: pulp (peeled orange)
pulp dry: solids from orange processing, i.e. shredded peel after oil extraction + solids after juice extraction

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### In processing

Green (1998) described the laboratory procedure designed to simulate commercial processing. Approximately 200 kg oranges were taken and the damaged fruit discarded. The remaining oranges were batch-tub washed for 5 minutes, again inspected for damage and field debris, then scarified in a Hobart Abrasive Peeler in batches of 5-10 kg. The resulting oil-water emulsion from the peel was screened to recover the oil fraction, and the oil separated from the emulsion by centrifuging. The remaining emulsion was frozen, thawed and separated by centrifuging. The oils were combined and a sample taken for analysis.

The scarified oranges were placed in a Hollymatic Juice Extractor, and the juice passed through a 0.6 mm screen to remove membranes, seeds and peel fragments before determination of the residues.

The shredded peel and remaining solids from the juice extraction were combined to produce wet pulp, which was treated with lime (CaO) and pressed to provide the liquor for molasses. The pressed pulp was dried to less than 10% moisture in a forced air drier and milled in a hammermill. A sample of dried pulp was taken for analysis.

The results of the trials on oranges and their processed commodities are given in Table 2.

### Residues in the edible portion of food commodities

Residues of pyriproxyfen and 4'-OH-Pyr were measured in the peel and peeled oranges as well as in the whole fruit in trial V-11121-N (Table 2). Where 0.25 kg ai/ha was applied the residues of pyriproxyfen were 0.41 mg/kg in the fruit and 0.01 mg/kg in the edible portion, giving a factor of 0.024 for the “process” of peeling oranges.

From the processing study V-11121-M the calculated processing factors for pyriproxyfen are 75 for the production of oil, 6.3 for dry pulp, and <0.03 for juice. The corresponding factors for 4'-OH-Pyr are 60, 5.5 and <0.5 respectively.

## APPRAISAL

Pyriproxyfen was first evaluated in 1999, and MRLs were recommended for citrus fruits, cotton seed and its processed commodities, and animal commodities. Information on the fate of pyriproxyfen during the processing of oranges, listed by the 1999 JMPR as desirable, has been provided.

### *Fate of residues during processing*

Data from supervised trials on *oranges* in USA in 1995 and 1996 were provided. In one trial, involving one treatment at the normal rate and one at an exaggerated rate, residues of pyriproxyfen and the metabolite 4′-hydroxypyriproxyfen were measured on peel and peeled orange and on whole orange. In another trial, pyriproxyfen was applied at an exaggerated rate, and the harvested oranges (200 kg) were processed by a procedure simulating commercial processing for juice and oil production.

In the trial at the exaggerated rate, the concentrations of residues were 0.41 mg/kg in peeled fruit and 0.01 mg/kg in the edible portion, resulting in a processing factor of 0.024 for orange to peeled orange. When treatment was at the normal rate, the concentrations of residues were 0.22 mg/kg in whole oranges and not detected (<0.01 mg/kg) in peeled orange. No residues of 4′-hydroxypyriproxyfen were detected (<0.01mg/kg) in the edible portion with either treatment. The 1999 JMPR reported that pyriproxyfen residues were not detected (<0.01 mg/kg) in the edible portion in 24 tests during trials on citrus.

The calculated processing factors in the trial of simulated commercial processing were 75 for oranges to oil, 6.3 for oranges to dry pulp (used as animal feed), and <0.03 for oranges to juice. Application of the factors to the median concentration (0.12 mg/kg) in whole oranges in the 11 trials that complied with GAP (Annex 6, reference 86) resulted in STMR-P values of 9.0 mg/kg for orange oil, 0.76 mg/kg for dried citrus pulp, and 0.0036 mg/kg for orange juice. Application of the factor for dried orange pulp (6.3) to the recommended maximum residue level for oranges (1 mg/kg) results in a HR-P value for dried citrus pulp of 6.3 mg/kg.

### *Residues in animal commodities*

The Meeting estimated the dietary burden of pyriproxyfen residues in farm animals on the basis of the diets listed in Appendix IX of the *FAO Manual* (FAO, 1997). Calculation from MRLs (or HR values) provides concentrations in feed suitable for estimating MRLs for animal commodities, while calculation from STMR values for feed is suitable for estimating STMR values for animal commodities. The percent dry matter is considered to be 100% for MRLs and STMR values expressed in dry weight. The information on cotton was evaluated in 1999.

| Commodity | MRL or HR | Group | % dry | MRL/ | % of diet | | Concentration of | |
|---|---|---|---|---|---|---|---|---|
| | | | | | Beef | Dairy | Beef | Dairy |
| Cotton gin trash | 5 | | 100 | 5.00 | 20 | 20 | 1.00 | 1.00 |
| Cotton seed (with lint) | 0.05 | SO | 88 | 0.057 | 25 | 25 | 0.01 | 0.01 |
| Cotton seed meal | 0.005 | SO | 89 | 0.006 | | | | |
| Citrus pulp, dry | 6.3 | AB | 91 | 6.9 | 20 | 20 | 1.38 | 1.38 |
| Total | | | | | | | 2.40 | 2.40 |

| Commodity | STMR | Group | % dry | MRL/ | % of diet | | Concentration of residue, mg/kg | |
|---|---|---|---|---|---|---|---|---|
| | | | | | Beef | Dairy | Beef | Dairy |

| Commodity | STMR | Group | % dry | MRL/ | % of diet | | Concentration of residue, mg/kg | |
|---|---|---|---|---|---|---|---|---|
| | | | | | Beef | Dairy | Beef | Dairy |
| Cotton gin trash | 0.91 | | 100 | 0.91 | 20 | 20 | 0.18 | 0.18 |
| Cotton seed (with | 0.01 | SO | 88 | 0.011 | 25 | 25 | 0.00 | 0.00 |
| Cotton seed meal | 0.001 | SO | 89 | 0.001 | | | | |
| Citrus pulp, dry | 0.76 | AB | 91 | 0.80 | 20 | 20 | 0.17 | 0.17 |
| Total | | | | | | | 0.35 | 0.35 |

The dietary burdens of pyriproxyfen for estimation of MRLs and STMR values (residue concentrations in animal feeds expressed as dry weight) are 2.4 and 0.35 ppm for beef cattle and 2.4 and 0.35 ppm for dairy cows.

The dietary burdens of cattle estimated by the 1999 JMPR were 1.0 ppm for estimation of the MRL and 0.18 ppm for estimation of the STMR value. As the value of 1.0 ppm was derived from the 3 ppm feeding level in the animal transfer studies, the revised dietary burden (2.4 ppm) does not change the recommended maximum residue level. Similarly, the revised dietary burden for the STMR value (0.35 ppm) does not change the estimated STMR values for animal commodities

**RECOMMENDATIONS**

The Meeting estimated the STMR levels shown below.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: pyriproxyfen.

The residue is fat-soluble.

| Commodity | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|
| CCN | Name | | |
| AB 0001 | Citrus pulp, dry | 0.76 | 6.3 |
| JF 0004 | Orange juice | 0.0036 | |
| | Orange oil | 9.0 | |

## Dietary risk assessment

***Chronic intake***

The 1999 JMPR concluded that the intake of pyriproxyfen from the five GEMS/Food regional diets represents essentially 0% of the ADI and that the intake of pyriproxyfen resulting from uses that have been considered by the JMPR is unlikely to present a public health concern. The additional information on citrus processing does not change that conclusion.

***Short-term intake***

The 1999 JMPR concluded that an acute RfD for pyriproxyfen is unnecessary. The Meeting therefore concluded that the short-term dietary intake of pyriproxyfen residues is unlikely to present a risk to consumers.

## REFERENCES

Green, C.A. 1998. Magnitude of the residue of pyriproxyfen in/on oranges and orange processing fractions. Project V-95-11121. Valent U.S.A. Corporation. Unpublished.

# THIABENDAZOLE (065)

## EXPLANATION

Thiabendazole was evaluated by the JMPR several times in the period 1970-1981 and was reviewed in 1997 under the CCPR Periodic Review Programme.

The 1997 JMPR recommended MRLs for banana (post-harvest), mushroom, potato, witloof chicory (sprouts), cattle milk, meat and edible offal, poultry meat and eggs, and recommended withdrawal of the MRLs for apple, cereal grains, citrus fruits, bulb onion, pear, strawberry, sugar beet, sugar beet leaves and tops, molasses and pulp (dry), tomato, meat and edible offal of cattle, goats, horses, pigs and sheep, and milks.

At the 23rd Session (1999) of the CCPR (ALINORM 99/24A, para. 65) the Committee recommended withdrawal of the CXLs for cereal grains, bulb onion, sugar beet, sugar beet leaves and tops, molasses and pulp (dry), and tomato but retained the CXLs for apple, citrus fruits, pear and strawberry as new data would be available to the 2000 JMPR. The CCPR recommended that the definition of the residue and details of analytical methods should be clarified by the JMPR/JECFA before the proposed draft MRLs for some animal products were advanced further.

After 1997 the interests in thiabendazole were transferred to a different manufacturer who reported data on analytical methods and uses of thiabendazole in several countries, and processing studies on apples, citrus fruits, and ware potatoes to the present Meeting. The results of supervised trials were reported for mandarins, oranges, apples, pears, ware potatoes, avocados, mangoes, melons, papayas and strawberries. The government of Germany provided information on use patterns and the government of The Netherlands on methods of residue analysis, use patterns, monitoring data and national MRLs.

## METHODS OF RESIDUE ANALYSIS

### Analytical methods

Campbell (2000) validated analytical methods M-027 and M-025.1, for the analysis of animal tissues and eggs respectively, for thiabendazole and its main metabolites 5-hydroxy-thiabendazole and benzimidazole.

Samples were extracted with 6 M HCl (24 h at 90-95ºC) and cleaned up by acid and base partition with ethyl acetate, followed by cation exchange solid-phase extraction (SPE). Extracts were analysed by cation exchange HPLC in compound- and tissue-specific mobile phases with fluorescence detection. The conditions are shown in Table 1.

Table 1. Conditions for HPLC analysis.

| Analyte | HPLC mobile phase | Fluorescence detection | |
|---|---|---|---|
| | | Excitation (nm) | Emission (nm) |
| Thiabendazole | 25:75 A/W pH=3.0[1] or 3.4[2] | 305[1], 265[2] | 380 |
| Benzimidazole | 25:75 A/W pH=3.4 | 265 | 380 |
| 5-OH-thiabendazole | 10:90 A/W pH=3.0 | 325 | 575 |

A/W = acetonitrile/water (0.05 M $KH_2PO_4$)
[1] Dairy and poultry meat, cow liver and kidneys
[2] Eggs, poultry skin with attached fat

The LOD of method M-027 was 0.03 mg/kg for each analyte in dairy and poultry meat, poultry skin with attached fat, cow liver and cow kidney. The LOD of method M-025.1 was 0.03 mg/kg for each analyte in eggs. The combined LOD (sum of the three LODs for each analyte) was 0.09 mg/kg. Mean recoveries were >70% and repeatability ($RSD_r$) was <20% for samples fortified with 0.03 mg/kg thiabendazole, 5-hydroxy-thiabendazole and benzimidazole before extraction (Table 2). Problems were encountered with thiabendazole in cow liver ($RSD_r$>20%) and benzimidazole in cow kidney (recovery <70%). Control samples of eggs, liver and kidney contained low apparent residues of benzimidazole (0.010-0.018 mg/kg) and control samples of liver of 5-OH-thiabendazole (0.010 mg/kg); recoveries were corrected for these interferences.

Table 2. Mean recoveries and $RSD_r$ for samples fortified with 0.03 mg/kg.

| Commodity | Thiabendazole | Benzimidazole | 5-OH-thiabendazole |
|---|---|---|---|
| Eggs | 99.3 ± 10.1% (n=8) | 76.1 ± 8.5% (n=8) | 85.1 ± 8.8% (n=8) |
| Poultry skin with attached fat | 81.2 ± 10.3% (n=8) | 85.1 ± 9.2% (n=8) | 89.1 ± 7.5% (n=8) |
| Poultry meat | 71.4 ± 3.5% (n=4) | 73.4 ± 2.0% (n=4) | 93.1 ± 5.6% (n=4) |
| Dairy meat | 69.7 ± 5.2% (n=4) | 93.3 ± 3.6% (n=4) | 71.6 ± 7.2% (n=4) |
| Cow liver | 90.1 ± 22.9% (n=6) | 94.2 ± 19.5% (n=7) | 69.6 ± 16.9% (n=7) |
| Cow kidney | 75.5 ± 4.6% (n=8) | 65.8 ± 13.4% (n=8) | 105.3 ± 5.1% (n=8) |

$RSD_r$ = repeatability expressed as relative standard deviation

The Ministry of Health, Welfare and Sport of The Netherlands (1996a,b) submitted two enforcement methods for the determination of thiabendazole in non-fatty foods (less than 5% fat). The first (1996a) was a multi-residue method using gas chromatography. The residues in fruits, vegetables and potatoes were extracted with a mixture of acetone, dichloromethane and petroleum ether (1:1:1). The residues in nuts, cereals, pulses, oil and tropical seeds, dried fruits, garlic, herbs and spices were extracted with a mixture of acetone and dichloromethane (1:1). There was no clean-up. Residues were determined by GLC with ITD (ion trap detection) on either DB-1, DB-5 or DB-1701 columns and detection at m/z = 129, 174 and 201-203. The limit of quantification was 0.05 mg/kg. Recoveries and repeatabilities ($RSD_r$) were 57% ± 22% (n=10) at a fortification level of 0.23 mg/kg and 83% ± 11% (n=10) at 1.16 mg/kg in lettuce.

In the second method (1996b), for the determination of benomyl, carbendazim and thiabendazole in fruits and vegetables, samples were extracted with a mixture of acetone, dichloromethane and petroleum ether (1:1:1), and the extract cleaned up on-line by solid-phase extraction (SPE) on diol-bonded silica cartridges. Determination was by HPLC (Shodex DE-163) with UV (280 nm) and fluorescence detection (excitation 235 nm; emission 280 nm) in tandem. The limit of quantification was 0.05 mg/kg. Recoveries and repeatabilities ($RSD_r$) were 98.5 ± 3% (n=10; orange) and 103.5 ± 4.5% (n=10; lettuce) at a fortification level of 0.1 mg/kg, and 103.8 ± 1.3% (n=10; orange) and 107.9% ± 2.5% (n=10; lettuce) at 1.0 mg/kg.

## USE PATTERN

The major registered or approved uses of thiabendazole on food crops are shown in Table 3 (post-harvest and pre-planting uses), Table 4 (pre-harvest uses) and Table 5 (wound treatment). The information was provided by the manufacturer except where otherwise indicated (* or **). Most of it was reported in summarized form: a hatch (#) indicates that original labels were available and bold that the information on the label differs from that in the summary. In such cases the information on the label is recorded.

Table 3. Registered post-harvest and pre-planting uses of thiabendazole.

| Crop | I/F | Country | Form | Application | | | | | Waiting time, days |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Method | g ai/t | l/t | g ai/l | No. | |
| Apple; pear | I | Australia # | DP 900 | Dip (30 s) | | | 1.0 | 1 | |
| | I | Belgium # | SC 450 | Dip or drench | | | 1.0 | 1 | |
| | I | Brazil # | WP 600 | Dip or drench (1-3 min) | | | 0.9 | 1 | |
| | I | France | SC 450 | Spray or dip | | 2 | 0.45-0.6 | 1 | |
| | I | France | SL 220 | Spray | | | 0.44 | 1 | |
| | I | Italy # | SC 500 | Dip or drench | | | 0.5-1.15 | 1 | 30 |
| | I | Italy # | SL 220 | Spray | | | 0.33-0.44 | 1 | 30 |
| | I | Kenya # | SC 450 | 30 s dip or drench | | | 0.55-1.1 | 1 | |
| | I | Mexico # | SC 450 | **Pre-harvest** | | | 0.45-0.9 | 1 | |
| | I | Mexico # | WP 600 | | | | **0.12-0.48** | 1 | |
| | I | Portugal # | SC 450 | 20-30 s dip or high volume spray | | | 0.56-0.99 | 1 | |
| | I | Portugal | SC 500 | Dip or high volume spray | | | 0.56-0.99 | 1 | |
| | I | S. Africa # | SC 450; SC 500 | 30 s drench or spray (3 min) | | | 1.05 | 1 | |
| | I | Spain # | SC 225[c] | 25-30 s dip or drench | | | 0.9-1.1 | | |
| | I | Spain # | SC 330[a] | 25-30 s dip or drench | | | 1.0-1.3 | | |
| | I | Spain | SC 450; SC 500; SL 220 | Dip or drench | | | 1.1 | 1 | |
| | I | Spain # | WP 600 | 20-30 s dip or drench | | | 0.9-1.2 | | |
| | I | USA # | **SC 492** | Dip, drench or spray (3 min) | | | 0.6 | 1 | |
| | I | USA | TC 985 | Dip or drench | | | 0.5 | | |
| | I | Venezuela | SC 450 | Drench | | | 0.45-1.1 | 1 | 8-13? |
| Avocado | I | Kenya # | SC 450 | Dip or drench | | | 1.1-3.4 | 1 | |
| | I | S. Africa # | SC 450; SC 500 | Dip (20 s) | | | 1.35 | 1 | |
| | I | S. Africa # | SC 450; SC 500 | Wax in water emulsion | | | 0.35 + wax | 1 | |
| | I | Venezuela | SC 450 | Drench | | | 0.9-2.25 | 1 | 8-13? |
| Banana | I | Australia # | DP 900 | Dip (2-4 min) | | | **0.15-0.42** | 1 | |
| | I | Brazil # | **SC 485** | Drench or spray | | | 0.20-0.45 | 1 | |
| | I | Brazil # | WP 600 | Drench | | | **0.24-0.48** | 1 | |
| | I | China | SC 450 | Dip or drench | | | 0.22-0.34 | 1 | |
| | I | France | SL 220 | Dip, drench or spray | | | 0.44 | 1 | |
| | I | France | SC 450 | | | | 0.45 | 1 | |
| | I | Israel | SC 450 | Dip or spray | | | 0.3 | 1 | |
| | I | Kenya # | SC 450 | Dip or drench | | | 0.20-0.40 | 1 | |
| | I | Mexico # | SC 450 | Drench or spray | | | 0.22-0.45 | 1 | |
| | I | Mexico # | WP 600 | | | | **0.24-0.48** | 1 | |
| | I | Portugal # | SC 450 | Dip (2-4 min) or high volume spray | | | 0.225 | 1 | |
| | I | Portugal | SC 500 | Dip or high volume spray | | | 0.225 | 1 | |
| | I | S. Africa # | SC 450; SC 500 | | | | 0.22 | 1 | |
| | I | Spain | SC 450; SC 500 | Dip or drench | | | 0.45 | 1 | |
| | I | Spain # | WP 600 | 25-30 s dip or drench | | | 0.21-0.45 | 1 | |
| | I | USA | TC 985 | | | | 0.2 | | |
| Banana; plantain | I | Belize # | SL 220 | Dip, drench or spray | | | 0.2-0.4 | 1 | |
| | I | Columbia # | SL 220 | Dip, drench or spray | | | 0.2-0.4 | 1 | |
| | I | Columbia # | SC 450 | Drench or spray | | | 0.22-0.45 | 1 | |
| | I | Costa Rica # | SL 220; SC 450; SC 500 | Dip, drench or spray | | | **0.2-0.4** | 1 | |

| Crop | I/F | Country | Form | Application | | | | | Waiting time, days |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Method | g ai/t | l/t | g ai/l | No. | |
| | I | Dominican Republic # | SL 220 | Dip, drench or spray | | | 0.2-0.4 | 1 | |
| | I | Guatemala # | SL 220; SC 450; SC 500 | Dip, drench or spray | | | **0.2-0.4** | 1 | |
| | I | Honduras # | SL 220; SC 450 | Dip, drench or spray | | | **0.2-0.4** | 1 | |
| | I | Nicaragua # | SC 450 | Dip, drench or spray | | | **0.2-0.4** | 1 | |
| | I | Panama # | **SL 220;** SC 450 | Dip, drench or spray | | | **0.2-0.4** | 1 | |
| | I | Venezuela | SC 450 | Drench | | | 0.23-0.45 | 1 | 8-13? |
| Barley | F | Poland ** | FS 10[j] | Seed dressing | 0.4 | 4 | 0.1 | | |
| | F | Poland ** | FS 25[l] | Seed dressing | 1.2 | 2 | 0.625 | | |
| | F | Germany | FS 25[m] | Seed dressing before sowing | 50 | | | 1 | |
| Cabbage (white, red) | F | Germany | SC 443 | Atomizing spraying | 0.332 | 0.5 | | 2 | 10 |
| Citrus fruit | I | Australia # | DP 900 | | | | 1.0 | 1 | |
| | I | Brazil # | **SC 485** | Drench, spray | | | **0.5-5.0** | 1 | |
| | I | Brazil # | WP 600 | Dip or drench | | | **0.5-4.8** | 1 | |
| | I | China | SC 450 | Dip or drench | | | 0.45-1.35 | 1 | |
| | I | Colombia # | SC 450 | Spray or drench | | | 0.45-0.90 | 1 | |
| | I | Costa Rica # | SC 450 | Spray or drench | | | 0.45-0.90 | 1 | |
| | I | Costa Rica # | SC 500 | Spray or drench | | | 2.0-5.0 | 1 | |
| | I | France | SC 450 | | | | 2.0 | 1 | |
| | I | Guatemala # | SC 450 | Spray or drench | | | 0.45-0.90 | 1 | |
| | I | Honduras # | SC 450 | Spray or drench | | | 0.45-0.90 | 1 | |
| | I | Israel | SC 450 | | | | 1.1 | 1 | |
| | I | Kenya # | SC 450 | Dip or drench | | | 0.90 | 1 | |
| | I | Mexico # | SC 450 | Drench or spray | | | **0.45-4.5** | 1 | |
| | I | Mexico # | WP 600 | Drench or spray | | | 0.9-1.1 | 1 | |
| | I | Nicaragua # | SC 450 | Spray or drench | | | 0.45-0.90 | 1 | |
| | I | Panama # | SC 450 | Spray or drench | | | 0.45-0.90 | 1 | |
| | I | Portugal # | SC 450 | Dip (20 s) or high volume spray | | | **5.0** | 1 | |
| | I | Portugal | SC500 | Drench or high volume spray | | | 2.0 | 1 | |
| | I | S. Africa # | SC 450 SC 500 | Spray or brush | | | 1.0-2.0 | 1 | |
| | I | S. Africa # | SC 450; SC 500 | Wax emulsion | | | 0.38-1.0 + wax | 1 | |
| | I | Spain | EC 5[b] | Spray (2nd application) | 5 | 1 | 5.0 | | |
| | I | Spain # | SC 125[d] | 25-30 s drench | | | 0.5 | 1 | |
| | I | Spain # | SC 225[c] | 25-30 dip or drench | | | 0.9-1.1 | | |
| | I | Spain # | SC 330[l] | 25-30 s dip or drench | | | 1.0-1.3 | | |
| | I | Spain | SC 330[e] | 1. Drench<br>2. Spray | 3.3-5.0 (2nd) | 1 (2nd) | 1.0-1.3 (1st); 3.3-5.0 (2nd) | 2 | |
| | I | Spain | SC 450 | Drench | | | 1.8 | 1 | |
| | I | Spain | SC 500 | 1. Drench<br>2. Spray + wax | 7 (2nd) + wax | | 1.8 (1st) | 2 | 7 (1st); 0 (2nd) |
| | I | Spain | SC 600 | 1. Drench<br>2. Spray + wax | 5 (2nd) + wax | | 1.2-1.8 (1st) | 2 | |
| | I | Spain | SL 220 | Drench | | | 2.2 | 1 | |
| | I | Spain # | WP 600 | 25-30 s dip or drench | | | 1.2-1.8 | | |
| | I | USA | Wax 1 | Spray in wax | 0.8-2.0 | | | | |
| | I | USA | SC 30 | Spray | | | 1.0 | | |
| | I | USA | TC 985 | Spray in wax | | | 2.7 | | |
| | I | USA | TC 985 | Spray | | | 2.0 | | |
| | I | USA | TC 985 | Drench | | | 1.0-2.0 | | |
| | I | USA | TC 985 | Spray in wax | 2.9-3.2 | | | | |

| Crop | I/F | Country | Form | Application | | | | | Waiting time, days |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Method | g ai/t | l/t | g ai/l | No. | |
| | I | Venezuela | SC 450 | - | | | 0.45-2.0 | 1 | |
| Grass | I | UK | WS 240[f] | Pre-planting seed treatment | 360 | | | 1 | |
| Horse beans | F | Germany | WS 240[f] | Pre-planting seed treatment | 360 | 5-10 | | 1 | |
| Mango | I | Guatemala # | SC 500 | Dip or drench | | | 1.0-2.5 | 1 | |
| | I | Kenya # | SC 450 | Dip or drench | | | 1.1-3.4 | 1 | |
| | I | USA | TC 985 | Dip or drench | | | 2.0 | | |
| | I | Venezuela | SC 450 | Drench | | | 0.9-1.8 | 1 | 8-13? |
| Papaya | I | USA | TC 985 | Dip or drench | | | 1.0-2.0 | | |
| Peas | F | Germany | WS 240[f] | Pre-planting seed treatment | 360 | 5-10 | | 1 | |
| Potato (w) | I | Austria # | SC 450 | Spray | 45 | 4 | 11.25 | 1 | |
| (w) | I | Austria # | SC 450 | Dip (3 min) | | | 2.7 | 1 | |
| (w) | I | Austria | SC 450 | Split application | 27(1st) 54(2nd) | | | 2 | |
| (s) | I | Belgium # | SC 450 | Seed treatment | 30-45 | 2 | 15-22 | 1 | |
| (s) | I | Belgium | SC 500 | Seed treatment | 30-45 | 2 | | 1 | |
| (w, s) | I | Belgium | SC 250[g] | | 30 | | | 1 | |
| (w) | I | Brazil # | WP 600 | Drench | 45 | 2 | 22.5 | 1 | |
| (s) | I | Brazil # | WP 600 | Pre-planting dip | | | 1.8 | 1 | |
| (w) | I | Colombia | SC 450 | Spray or dip | 45 | | 1.8 | 1 | |
| (w) | I | Costa Rica | SC 450 | Spray or dip | 45 | | 1.8 | 1 | |
| (w) | I | Costa Rica | SC 500 | Spray or dip | 45 | | 1.8 | 1 | |
| (w, s) | I | France | SC 450 | Drench, spray | 60 | 2 | 29.9 | 1 | |
| (w) | I | France | SL 220 | Spray | | | 30.8 | 1 | |
| (s) | I | France | SC 225[c] | Pre-planting seed treatment | 45 | | | | |
| (w, s) | I, F | Germany | SC 450; SC 443 | Low volume spray; | 27 | 0.06 | | 1 | 28 |
| (s) | F | Germany | SC 450; SC 443 | Spray; pre-planting seed treatment | 27-54 | 0.12 | | | |
| (w) | I | Guatemala | SC 450 | Spray or dip | 45 | | 1.8 | 1 | |
| (w) | I | Honduras | SC 450 | Spray or dip | 45 | | 1.8 | 1 | |
| (s, w) | I | Ireland | SL 220 | Spray; spinning disk equipment; ultra low volume; 2 weeks after lifting | 44 | 0.2-2 | | | 21 |
| (s, w) | I | Ireland | SC 450 | Spray | 40.5 | | | 1 | 21 |
| (s, w) | I | Ireland | SC 450 | Spinning disk | | 0.09 | 450 | 1 | 21 |
| (s, w) | I | Ireland | SC 450 | Ultra low volume | | 1-2 | 20.25-40.5 | 1 | 21 |
| (s) | I | Ireland | DS 100[h] | Seed treatment; spinning disk; 2 weeks after lifting | 40 | | 20-40 | | 42+ |
| (s, w) | I | Italy # | SC 500 | Spray | **40** | 1-2 | | | 30 |
| (s) | I | Italy # | SC 500 | 3-5 min dip; pre-planting seed treatment | | | **1.8** | 1 | |
| (w, s) | I | Luxembourg | SC 250[g] | | 30 | | | 1 | |
| (w, s) | I | Nether-lands* | SC 450 | Spray shortly after harvest | **30** | | | 1 | 60 |
| (s) | I | Nether-lands* | **EC 250[g]** | Spray shortly after harvest | **30** | | | 1 | 0 |
| (w) | I | New Zealand | SC 450 | Spray | 42 | | | 1 | |
| (w) | I | Nicaragua | SC 450 | Spray or dip | 45 | | 1.8 | 1 | |
| (s) | I | Norway | SC 500 | Spray | 50 | | | 1 | |
| (w) | I | Panama | SC 450 | Spray or dip | 45 | | 1.8 | 1 | |
| (w) | I | S. Africa # | SC 450; SC 500 | **Spray** | | **2-4** | **1.6-3.15** | **1** | |

| Crop | I/F | Country | Form | Application | | | | | Waiting time, days |
|---|---|---|---|---|---|---|---|---|---|
| | | | | Method | g ai/t | l/t | g ai/l | No. | |
| (s) | I | S. Africa # | SC 450; SC 500 | **Spray** | | **2-4** | **10.5-21.0** | **1** | |
| (s) | | S. Africa # | SC 450; SC 500 | **Pre-planting dip (5 min)** | | | **4** | **1** | |
| (s) | | S. Africa # | SC 450; SC 500 | **Pre-planting; ultra low volume spray** | **67** | | | | |
| (w, s) | I | S. Africa # | SC 450; SC 500 | **(2-4 min ) dip** | | | **2.0** | **1** | |
| (w, s) | I | S. Africa # | SC 450; SC 500 | **Ultra low volume spray** | **45** | | | **1** | |
| (w) | I | UK # | SL 220 | Spinning disk; within 2 weeks after lifting | **44** | **0.2** | | 1 | 21 |
| (w) | I | UK # | SL 220 | Ultra low volume spray; within 2 weeks after lifting | **44** | **1-2** | | 1 | 21 |
| (w, s) | I | UK | SC 450; SC 500 | Spinning disk | 40.5 | 0.09 | 450 | 1 | 21 |
| (w, s) | I | UK | SC 450; SC 500 | Ultra low volume spray | 40.5 | 2 | 20.25 | 1 | 21 |
| (w, s) | I | UK # | DS 100[h] | Spinning disk; within 2 weeks after lifting | **40** | **0.4** | | 1 | 42 |
| (w, s) | I | UK # | DS 100[h] | Ultra low volume spray; within 2 weeks after lifting | **40** | **1-2** | | 1 | 42 |
| (s) | I | UK | SC 300[i] | Pre-planting seed treatment | 30 | | | | |
| (w) | I | USA # | SC 492 | 20 s dip or spray | 6.7 (spray) | | 1.6 (dip) | 1-2 | |
| (w) | I | Venezuela | SC 450 | Drench | | | 1.35-2.25 | 1 | 8-13? |
| Rape | F | Poland ** | WS 35[k] | Seed dressing | 49 | | | | |
| Rye | F | Poland ** | FS 25[l] | Seed dressing | 1.25 | 2 | 0.625 | | |
| Sweet potato | I | USA # | **SC 492** | 1-2 min dip | | | **4.1** | 1 | |
| Triticale | F | Poland ** | FS 25[l] | Seed dressing | 1.25 | 2 | 0.625 | | |
| Wheat | F | Poland ** | FS 25[l] | Seed dressing | 1.25-1.88 | 2-3 | 0.625 | | |
| Witloof, chicory (endive roots) | I | Belgium # | SC 450 | Spray | 40 | 20 | 2.0 | 1 | |
| | I | Belgium # | SC 450 | Drench (1-2 min) | | | 1.0 | 1 | |
| | I | France | SC 450 | Dip, drench | | 2 | 0.84-1.03 | 1 | |
| | I | France | SC 450 | Mist spray | 40 | 8 | 4.5 | 1 | |

I = indoor treatment; F = field treatment; Potato (w) = ware potato; Potato (s) = seed potato
* data submitted by the Plant Protection Service of The Netherlands
** data submitted by the Institute of Plant Protection of Poland
[a] formulation also contains 100 g/l dicloran
[b] formulation also contains 2 g/l imazalil and 180 g/l wax
[c] formulation also contains 75 g/l imazalil
[d] formulation also contains 400 g/l fosetyl-Al
[e] formulation also contains 200 g/l guazatine
[f] formulation also contains 450 g/kg metalaxyl
[g] formulation also contains 125 g/l imazalil
[h] formulation also contains 300 g/l tecnazene
[i] formulation also contains 100 g/l imazalil
[j] formulation also contains 30 g/l flutriafol, 400 g/l ethirimol
[k] formulation also contains 400 g/kg furatiocarb and 25 g/kg metalaxyl
[l] formulation also contains 25 g/l flutriafol
[m] formulation also contains 15 g/l imazalil and 37.5 g/l flutriafol

Table 4. Registered pre-harvest uses of thiabendazole.

| Crop | I/F | Country | Application | | | | | | | PHI, days |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Form | Method | Rate, kg ai/ha | Vol., l/ha | Spray, g ai/l | No. | Interval, days | |
| Apple; pear | F | Mexico # **(2 labels)** | WP 600 | Foliar spray; at flowering | **0.3-0.6; 0.4-1.4** | | | 2 | 10-15 | 0 |
| Avocado | F | Mexico # | WP 600 | Foliar spray; at flowering | **0.3-0.45** | | 1.1-3.2 | 2 | 14-21 | 15 |
| Banana; plantain | F | Guatemala # | SC 500 | Foliar spray | 0.036 | | | | | |
| Citrus | F | Venezuela | SC 450 | Foliar spray | | 200-800 | 0.675 | 1 | | 5 |
| | F | Venezuela | SC 450 | Aerial | | 50-100 | 0.675 | 1 | | 5 |
| Mango | F | Mexico # | WP 600 | Foliar spray; at flowering | **0.3-0.45** | | | 2 | 14-21 | 15 |
| Melon | F | Guatemala# | SC 500 | foliar spray | | | 0.6-1.35 | 1 | | 3 |
| | F | Venezuela | SC 450 | foliar spray | | 200-800 | 0.675 | 1 | | 5 |
| | F | Venezuela | SC 450 | Aerial | | 50-100 | 0.675 | | | 5 |
| Mushroom | I | Australia # | DP 900 | At casing[1] | | | 230 g ai/t | 1 | | |
| | I | Kenya # | SC 450 | At casing | | | 225 g ai/t | | | |
| | I | Kenya # | SC 450 | during cropping, between flushes | 1.4-14 | | | | | |
| | I | S. Africa # | SC 450; SC 500 | After casing, drench between breaks | 6.3 | | 1.15-1.5 | | | |
| | I | UK | WP 600 | Pre-casing drench or weekly spray during flushes | | | | 1-4 | 7 | 5 |
| | I | USA # | **SC 492** | Spray at casing, fuzzing, pinning, between breaks | **12.5 (1st); 6.2 (2nd-4th)** | | | 1-4 | | 1 |
| Strawberry | F | Kenya # | SC 450 | foliar spray | | | 0.45-0.90 | 3 | | 14 |
| | F | Venezuela | SC 450 | foliar spray | | 200-800 | 0.675 | 1 | | 5 |
| | F | Venezuela | SC 450 | Aerial | | 50-100 | 0.675 | | | 5 |

I = indoor treatment; F = field application;
[1]Incorporated in water used to wet the peat mass

Table 5. Registered uses of thiabendazole as wound paste. All field uses in Germany. All applications by painting on cleanly cut out wounds.

| Crop | Form, g ai/kg |
|---|---|
| Blackberries | PA 10.1[1] |
| | PA 10 |
| Blueberries | PA 10.1[1] |
| | PA 10 |
| Currants (black, white, red) | PA 10.1[1] |
| | PA 10 |
| Gooseberries | PA 10.1[1] |
| | PA 10 |
| Hazelnut | PA 10.1[1] |
| | PA 10 |
| Pome fruit | PA 10.1[1] |
| | PA 10.1[1] |
| Raspberries | PA 10.1[1] |
| | PA 10.1[1] |
| Stone fruit | PA 10.1[1] |
| | PA 10.1[1] |
| Walnut | PA 10.1[1] |
| | PA 10 |

[1] Formulation also contains 20 g/kg imazalil and 10 g/kg azaconazole

## RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of supervised trials on citrus fruits (oranges and mandarins), pome fruits (apples and pears), strawberries, tropical fruits with inedible peel (avocados, mangoes and papayas), melons and ware potatoes are shown in Tables 6 to 12.

Two or more residues shown for a single location, type of sample, and PHI were in replicate samples from the same pre- or post-harvest treatment. Where reports listed replicate analytical results their means are shown in the Tables. The residues are not corrected for recovery or control values except as indicated. Underlined residues reflect current GAP.

Table 6. Residues from post-harvest applications to mandarins in Spain.

Table 7. Residues from post-harvest applications to oranges in Spain.

Table 8. Residues from post-harvest applications and wound treatments to apples and pears in Spain, France and Germany.

Table 9. Residues from pre-harvest applications to strawberries in Spain.

Table 10. Residues from post-harvest applications to tropical fruits with inedible peel in South Africa, Costa Rica, Brazil and Belize.

Table 11. Residues from pre-harvest applications to melons in Spain.

Table 12. Residues from post-harvest application to ware potatoes in The Netherlands.

Citrus fruits. Details of post-harvest trials according to GLP on citrus fruits in Spain (1998) were reported.

Mandarins and oranges were treated with a post-harvest SC formulation using either (a) a single drench application (2.0 g ai/l) on ripe fruit, (b) a drench on green fruit plus a spray application on degreened fruit two days later (1.8 + 6.0 g ai/l) or (c) a spray on green fruit plus a spray on degreened fruit two days later (3 + 4 g ai/l). Using commercial equipment 35 boxes of citrus fruits (700 kg) were drenched; 12 boxes of which were sprayed automatically simulating conditions in packing houses. The fruits were then waxed with an emulsion of 1 l/t "Teycer", and stored at 2-5ºC in a normal atmosphere (75-85% humidity, 800-2000 ppm $CO_2$). Single and duplicate samples (24 mandarins/12 oranges, 1.7-3.5 kg) were collected randomly and stored at or below -18ºC, then separated by hand into pulp and peel. The residue in the whole fruit was calculated from the weights of pulp and peel (51-72% w/w of pulp in mandarins; 51-61% of pulp in oranges). Thiabendazole was determined by a modification of method M-049 (extraction with 0.1 M HCl and HPLC with fluorescence detection). The LOD was 0.01 mg/kg in the pulp, 0.02 mg/kg in the peel and 0.02 mg/kg in the whole fruit. In control samples of pulp thiabendazole was below the LOD, but the peel contained ≤0.08 mg/kg in mandarins and ≤0.13 mg/kg in oranges. The recoveries were 91-108% (n=30) from the pulp and 72-104% (n=26) from peel at fortification levels at the LOD and at 0.05, 0.1, 1.0 and 2.0 mg/kg thiabendazole.

Table 6. Residues of thiabendazole in or on mandarins from post-harvest applications in supervised trials in Spain (1998).

| Location | Variety | Application | | | | | | Sample | DAT | Residues, mg/kg (c=control) | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Form | g ai/t | l/t | g ai/l | Interval, days | Type | | | | |
| Carcer[1,2] (Valencia) | Clemen-villa | SC500 | | | 2.0 | | 30 sec drench + wax | peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit | 0<br>0<br>0<br>7<br>7<br>7<br>15<br>15<br>15 | 1.7, c=0.03<br>0.01<br>0.65<br>1.6<br><0.01<br>0.64<br>1.3, c=0.02<br>0.01<br>0.51 | Kissling, 1999a (2276/98) |
| Carcer[1,2] (Valencia) | Clemen-tina de Nules | SC 500 | | | 2.0 | | 30 sec drench + wax | peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit | 0<br>0<br>0<br>7<br>7<br>7<br>15<br>15<br>15 | 1.2<br>0.01<br>0.50<br>1.2<br><0.01<br>0.46<br>0.87, c=0.02<br><0.01<br>0.37 | Kissling, 1999b (2277/98) |
| Picassent[1,2] | Clemen-villa | SC 500 | | | 2.0 | | 150 sec drench + wax | peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit | 0<br>0<br>0<br>7<br>7<br>7<br>15<br>15<br>15 | 3.0, c=0.05<br>0.02<br>1.1, c=0.02<br>3.5<br>0.01<br>1.4<br>2.7, c=0.08<br>0.04<br>1.0, c=0.04 | Kissling, 1999c (2278/98) |
| Picassent[1,2] | Clemen-tina de Nules | SC 500 | | | 2.0 | | 150 sec drench + wax | peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit | 0<br>0<br>0<br>7<br>7<br>7<br>15<br>15<br>15 | 3.9, c=0.06<br>0.01<br>1.6, c=0.02<br>3.4<br><0.01<br>1.4<br>3.9, c=0.04<br><0.01<br>1.6 | Kissling, 1999d (2279/98) |
| Carcer[1,2], Paterna[3] | Clemen-tina de Nules | SC 500 | - 6.0 | - 1.0 | 1.8 + 6.0 | 2 | 30 sec drench + spray + wax | peel<br>pulp<br>whole fruit<br>controls | 0<br>0<br>0<br>0 | 3.3, 4.6<br>2.8, 4.4<br><0.01, <0.01<br>0.01, 0.03<br>1.5, 2.2,<br>1.4, 1.7<br>c=0.03 (peel)<br>c=0.05 (peel),<br>c=0.03 (fruit) | Kissling, 1999e (2284/98), Kissling, 1999f (2285/98) |
| Picassent[1,2], Paterna[3] | Clemen-tina de Nules | SC 500 | - 6.0 | - 1.0 | 1.8 + 6.0 | 2 | 150 sec drench + spray + wax | peel<br>pulp<br>whole fruit<br>controls | 0<br>0<br>0<br>0 | 3.2, 4.4<br>2.3, 4.3<br>0.02, 0.01<br>0.09, 0.04<br>1.2, 2.2,<br>0.77, 1.4,<br>c=0.04 (peel)<br>c=0.06 (peel)<br>c=0.02 (fruit)<br>c=0.03 (fruit) | Kissling, 1999g (2286/98) Kissling, 1999h (2287/98) |

| Location | Variety | Application | | | | | | Sample | DAT | Residues, mg/kg (c=control) | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Form | g ai/t | l/t | g ai/l | Interval, days | Type | | | | |
| Paterna[1,2,3] (Valencia) | Clementina de Nules | SC 500 | 3.0 + 4.0 | 1.0 + 1.0 | 3.0 + 4.0 | 2 | spray + spray + wax | peel<br>pulp<br>whole fruit<br>controls | 0<br>0<br>0<br>0 | 1.6, 1.3<br>1.1, 1.0<br>0.07, 0.07<br>0.03, 0.02<br>0.49, 0.42,<br>0.35, 0.32,<br>c=0.02 (peel)<br>c=0.02 (peel) | Kissling, 1999i (2292/98)<br>Kissling, 1999j (2293/98) |
| Paterna[1,2,3] (Valencia) | Clementina de Nules | SC 500 | 3.0 + 4.0 | 1.0 + 1.0 | 3.0 + 4.0 | 2 | spray + spray + wax | peel<br>pulp<br>whole fruit | 0<br>0<br>0 | 0.91, 1.6<br>1.6, 1.1<br><0.01, <0.01<br><0.01, <0.01<br>0.40, 0.78<br>0.77, 0.51 | Kissling, 1999k (2294/98)<br>Kissling, 199l (2295/98) |

DAT: days after treatment
[1] field location
[2] first treatment location
[3] second treatment location

Table 7. Residues of thiabendazole in or on oranges from post-harvest applications in supervised trials in Spain (1998).

| Location | Variety | Application | | | | | | Sample | DAT | Residues, mg/kg (c=control) | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Form | g ai/t | l/t | g ai/l | Interval, days | Type | | | | |
| Carcer[1,2] (Valencia) | Orange; Navelina | SC 500 | | | 2.0 | | 30 sec drench + wax | peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit | 0<br>0<br>0<br>7<br>7<br>7<br>15<br>15<br>15 | 0.94; c=0.03<br><0.01<br>0.40; c=0.02<br>0.69<br><0.01<br>0.29<br>0.79; c=0.03<br><0.01<br>0.33; c=0.02 | Kissling, 1999m (2280/98) |
| Carcer[1,2] (Valencia) | Orange; Salustiana | SC 500 | | | 2.0 | | 30 sec drench + wax | peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit | 0<br>0<br>0<br>7<br>7<br>7<br>15<br>15<br>15 | 1.3; c=0.02<br>0.01<br>0.53<br>1.2<br>0.01<br>0.50<br>0.98;<br><0.01<br>0.41 | Kissling, 1999n (2281/98) |
| Picassent[1,2] | Orange; Navelina | SC 500 | | | 2.0 | | 150 sec drench + wax | peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit | 0<br>0<br>0<br>7<br>7<br>7<br>15<br>15<br>15 | 3.9; c=0.13<br><0.01<br>1.6; c=0.06<br>3.3<br><0.01<br>1.4<br>3.8; c=0.10<br><0.01<br>1.5; c=0.04 | Kissling, 1999o (2282/98) |

| Location | Variety | Application | | | | | | Sample | DAT | Residues, mg/kg (c=control) | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | Form | g ai/t | l/t | g ai/l | Interval, days | Type | | | | |
| Picassent[1,2] | Orange; Salustiana | SC 500 | | | 2.0 | | 150 sec drench + wax | peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit<br>peel<br>pulp<br>whole fruit | 0<br>0<br>0<br>7<br>7<br>7<br>15<br>15<br>15 | 3.0; c=0.02<br>0.01<br>1.2<br>2.7<br>0.01<br>1.1<br>2.8; c=0.03<br><0.01<br>1.2 | Kissling, 1999p (2283/98) |
| Carcer[1,2]; Paterna[3] | Orange; Navelina | SC 500 | - 6.0 | - 1.0 | 1.8 + 6.0 | | 30 sec drench + spray + wax | peel<br>pulp<br>whole fruit | 0<br>0<br>0 | 3.2, 3.9<br><0.01, <0.01<br>1.4, 1.9;<br>c=0.02 (peel) | Kissling, 1999q (2289/98) |
| Picassent[1,2]; Paterna[3] | Orange; Navelina | SC 500 | - 6.0 | - 1.0 | 1.8 + 6.0 | | 150 sec drench + spray + wax | peel<br>pulp<br>whole fruit | 0<br>0<br>0 | 2.0, 2.2<br><0.01, <0.01<br>0.89, 1.1;<br>c=0.13 (peel);<br>c=0.06 (fruit) | Kissling, 1999r (2288/98) |
| Picassent[1,2]; Paterna[3] | Orange; Navelina | SC 500 | - 6.0 | - 1.0 | 1.8 + 6.0 | | 150 sec drench + spray + wax | peel<br>pulp<br>whole fruit<br>controls | 0<br>0<br>0 | 3.66, 2.68<br>2.77, 3.32<br>0.01, 0.01<br><0.01, 0.01<br>1.6, 1.1;<br>1.2, 1.3;<br>c=0.03 (peel)<br>c=0.05 (peel);<br>c=0.03 (fruit) | Kissling, 1999s (2290/98)<br><br>Kissling, 1999t (2291/98) |
| Paterna[1,2,3] (Valencia) | Orange; Navelina | SC 500 | 3.0 + 4.0 | 1.0 + 1.0 | 3.0 + 4.0 | | spray + spray + wax | peel<br>pulp<br>whole fruit | 0<br>0<br>0 | 0.99, 0.85<br>1.4, 1.1<br><0.01, <0.01<br><0.01, <0.01<br>0.41, 0.37<br>0.52, 0.43;<br>c=0.02 (peel) | Kissling, 1999u (2296/98)<br>Kissling, 1999v (2297/98) |
| Paterna[1,2,3] (Valencia) | Orange; Navelina | SC 500 | 3.0 + 4.0 | 1.0 + 1.0 | 3.0 + 4.0 | | spray + spray + wax | peel<br>pulp<br>whole fruit | 0<br>0<br>0 | 1.6, 1.6<br>1.6, 2.0<br><0.01, <0.01<br><0.01, <0.01<br>0.65, 0.70<br>0.68, 0.83 | Kissling, 1999w (2298/98)<br>Kissling, 1999x (2299/98) |

DAT: days after treatment
[1] field location
[2] first treatment location
[3] second treatment location

Pome fruits. Details of post-harvest trials on pome fruits in France (1998) and Spain (1991) were reported, together with some information on wound treatment trials on apples in Germany (1998).

The GLP trials in France were on post-harvest apples with SC formulations using either a dip simulating local practice (1.1 g ai/l) or a drench with commercial equipment (1.1 g ai/l). Two boxes of 15-20 kg apples were used in the former and three paloxes (stackable wooden containers) each of 300 kg apples in the latter. The apples were stored at 0-2ºC in a normal atmosphere (93-100% relative humidity). Single samples of 1.7-2.7 kg were collected randomly from both sets of samples and stored at or below -18ºC. Thiabendazole was determined by modified method M-049 (HPLC with fluorescence detection). In control samples thiabendazole was <0.02 mg/kg. Recoveries were 99%-105% and 92%-96% at fortification levels of 0.02 and 2.0 mg/kg thiabendazole respectively.

In the trials in Spain (not GLP) apples and pears were drenched post-harvest with SC formulations containing thiabendazole alone or thiabendazole plus ethoxyquin, an anti-scald agent. Samples were stored at -0.8 to +1.0ºC in a normal atmosphere of 90% humidity. In each trial three replicates each of 2-3 kg fruit were analysed in two laboratories. The first presented results near the LOD 0-32 days after treatment, and the second results 100 times higher 32-160 days after treatment. Details of the analyses were not provided, but the first laboratory used GLC with an FPD instead of the usual HPLC. The residues shown in Table 8 are from the second laboratory only. Thiabendazole was not detected (<0.01 mg/kg) in the control samples.

Table 8. Residues of thiabendazole in apples and pears from post-harvest and wound treatment applications in supervised trials in Spain, France and Germany. Whole fruit analysed.

| Country; year; treatment location | Fruit and variety | Application | | | | | DAT | Residues, mg/kg | Ref. |
|---|---|---|---|---|---|---|---|---|---|
| | | Form | g ai/t | l/t | g ai/l | Type | | | |
| Spain; 1991; Sudanell | Apple Golden Delicious | Tecto 20S | | | 1.1 | drench | 32<br>61<br>147 | 1.6, 1.7, 2.2<br>2.0, 1.8, 2.7<br>1.8, 2.2, 1.3 | Tecnidex 1992; |
| Spain; 1991; Sudanell | Apple Golden Delicious | Tecto 20S | | | 1.1[a] | drench | 32<br>61<br>147 | 2.5, 1.8, 2.0<br>2.5, 1.5, 1.7<br>1.6, 1.6, 2.6 | Tecnidex 1992 |
| Spain; 1991; Sudanell | Pear Blanquilla | Tecto 20S | | | 1.1 | drench | 75<br>160 | 2.0, 1.8, 1.7<br>1.6, 1.9, 2.5 | Tecnidex 1992 |
| Spain; 1991; Sudanell | Pear Blanquilla | Tecto 20S | | | 1.1[b] | drench | 75<br>160 | 1.8, 1.6, 1.7<br>1.8, 2.3, 2.3 | Tecnidex 1992 |
| France (North); 1998; Ste-Maure-de Touraine | Apple; Granny Smith | SC 500 | | | 1.1 | 60 sec dip | 0<br>15<br>30 | 2.0<br>1.5<br>1.7 | Kissling, 1999y (2243/98) |
| France (North); 1998; Ste-Maure-de Touraine | Apple; Rouges Americaines | SC 500 | | | 1.1 | 60 sec dip | 0<br>15<br>30 | 1.7<br>1.5<br>1.5 | Kissling, 1999z (2244/98) |
| France (North); 1998; Ste-Maure-de Touraine | Apple; Golden Delicious | SC 500 | | | 1.1 | 60 sec dip | 0<br>15<br>30 | 1.5<br>1.4<br>1.7 | Kissling, 1999aa (2245/98) |
| France (North); 1998; Ste-Maure-de Touraine | Apple; Braeburn | SC 500 | | | 1.1 | 60 sec dip | 0<br>15<br>30 | 1.4<br>1.5<br>1.5 | Kissling, 1999ab (2246/98) |
| France (North); 1998; Vernantes | Apple Braeburn | SC 500 | 1200 | 1100 | 1.1[c] | 45 sec drench | 0<br>14<br>28 | 1.6<br>1.4<br>1.3 | Kissling, 1999ac (2247/98) |
| France (North); 1998; Vernantes | Apple Granny Smith | SC 500 | 1200 | 1100 | 1.1[c] | 45 sec drench | 0<br>14<br>28 | 1.6<br>1.7<br>1.5 | Kissling, 1999ad (2248/98) |
| France (North); 1998; Lignieres-de-Touraine | Apple Granny Smith | SC 500 | 1200 | 1100 | 1.1 | 120 sec drench | 0<br>14<br>29 | 1.7<br>1.6<br>1.6 | Kissling, 1999ae (2249/98) |
| France (North); 1998; Lignieres-de-Touraine | Apple Golden Delicious | SC 500 | 1200 | 1100 | 1.1 | 120 sec drench | 0<br>14<br>29 | 1.8<br>1.7<br>1.9 | Kissling, 1999af (2250/98) |
| Germany 1998 | Apple Jonathan | PA | 25 g formulation per 450 $cm^2$; about 0.25 g/tree | | | Wound paste before flowering | 125 | <0.05 | BBA 3688 |

DAT: days after treatment
[a] also contains 0.72 g/l ethoxyquin
[b] also contains 2.16 g/l ethoxyquin
[c] also contains 0.6% (v/v) diphenylamine (DPA, antioxidant)

Strawberries. Details of residue trials in 1993 on pre-harvest strawberries in Spain were reported, but only summaries for trials in 1989 and 1991. Residues were reported in the 1989 trials as the means of quadruplicate analyses, and in the 1991 trials as two results, each the mean of triplicates. The two

results were assumed to be from duplicate samples from the same plot. The two 1989 trials were evaluated by the 1997 JMPR.

The 1993 trials (not GLP) were in the field or in macro-tunnels with SC formulations sprayed from a motorized backpack at rates of 1.8 kg ai/ha and 90 g ai/hl. Samples of 1-1.5 kg each were taken. Details of storage conditions and analytical methods were not included. Residues of thiabendazole in control samples were 0.01-0.06 mg/kg.

Table 9. Residues of thiabendazole in or on strawberries from pre-harvest open field and macro-tunnel spray applications in supervised trials in Spain. Whole fruit analysed.

| Year; Location | Variety | Application | | | | | PHI days | Residues, mg/kg | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|
| | | Form | No | kg ai/ha | kg ai/hl | Type | | | |
| 1989; Moguer; Huelva | Chandler | SC 450 | 1 | 1.2 | 0.07 | under plastic | 0<br>3<br>7<br>14 | 1.7 (n=4)<br>1.6 (n=4)<br>1.4 (n=4)<br>1.1 (n=4) | Spain, 1991 (HU 1-89) |
| 1989; Moguer; Huelva | Chandler | SC 450 | 1 | 1.2 | 0.07 | open field | 0<br>3<br>7<br>14 | 0.78 (n=4)<br>0.33 (n=4)<br>0.43 (n=4)<br>0.10 (n=4) | Spain, 1991 (HU 2-89) |
| 1991; Lucena del Puerto; Huelva | Chandler | SC 450 | 1 | 1.8 | 0.09 | open field | 0<br>3<br>7 | 2.5 (n=3), 1.8 (n=3)<br>1.3 (n=3), 1.3 (n=3)<br>0.87 (n=3), 0.50 (n=3) | Spain, 1992 (065-91-9011R)<br>Spain, 1992 (065-91-9010R) |
| 1993; Bonares; Huelva | Oso Grande | SC 450 | 1 | 1.8 | 0.09 | open field | 0<br>3 | 4.6, 3.2 (c=0.04, 0.03)<br>1.6, 1.6 | Spain, 1993a (065-93-9007); Spain, 1993b (065-93-9008) |
| 1993; Moguer; Huelva | Chandler | SC 450 | 1 | 1.8 | 0.09 | open field | 0<br>3 | 1.6, 2.7 (c=0.02 , 0.02)<br>2.3, 1.8 | Spain, 1993c (065-93-9011); Spain, 1993d (065-93-9012) |
| 1993; Rociana del condado; Huelva | Oso Grande | SC 450 | 1 | 1.8 | 0.09 | open field | 0<br>3 | 5.2, 5.0 (c=0.04, 0.06)<br>1.9, 2.7 | Spain, 1993e (065-93-9013); Spain, 1993f (065-93-9014) |
| 1993; Lucena del Puerto; Huelva | Chandler | SC 450 | 1 | 1.8 | 0.09 | macro-tunnel | 0<br>3 | 3.4, 3.9 (c=0.03, 0.01)<br>2.2, 2.6 | Spain, 1993g, (065-93-9009; Spain, 1993h; 065-93-9010) |

ND: not detected; LOD not stated

Tropical fruits with inedible peel. Detailed reports were available for post-harvest residue trials on avocados in South Africa (1977) and Costa Rica (1996), mangoes in Brazil (1994) and Belize (1996), and papayas in Belize (1996). The trials on avocados and papayas in Costa Rica and on mangoes in Belize were only reported in part (23 pages of 476; appendices missing).

Only limited information on analysis in the South African trials on avocados in 1977 (GLP) was reported. Each fruit was halved, the stone discarded and the skin removed from one half. Composite samples with and without peel were homogenized and analysed in duplicate by GLC with an FID (limit of detection 0.02 mg/kg thiabendazole). Mean recoveries at a fortification level of 2.0 mg/kg were 94% (n=2). Control samples contained 0.05 mg/kg in the pulp and 0.23 mg/kg in the whole fruit.

The 1996 trials on avocados and papayas in Costa Rica and on mangoes in Belize (all GLP) were with SC formulations as a post-harvest dip or spray application to run-off. For dipping, the fruit in a mesh bag was dipped for 30 sec in a freshly-prepared solution. Spraying with a hand-held boom attached to a $CO_2$-backpack sprayer was directed from above and below the fruit on a suspended wire mesh tray. Duplicate samples each of 24 fruits were treated and stored frozen at temperatures from -6

to -22ºC for 16-34 days. Avocado and mango stones were removed before homogenization, partitioning into ethyl acetate, and clean-up and determination by cation-exchange HPLC with fluorescence detection (Merck method M-067). The recoveries at fortification levels of 0.05, 0.5 and 10.0 mg/kg were 85 ± 13% (n=8) for avocados, 95 ± 14% (n=7) for mangoes and 91 ± 10% (n=11) for papayas. In control samples thiabendazole could not be determined (LOD=0.05 mg/kg)

In the trials on mangoes and avocados residues in the whole fruit could not be calculated because the stones were not weighed. The manufacturer concluded from published sources that the weight of a mango stone is about 20 to 23% of the weight of the whole fruit (Morton, 1987), and from residue trials using another pesticide that an avocado stone weights about 15% of the whole fruit (Bull and Adams, 1987).

Table 10. Residues of thiabendazole in or on tropical fruits with inedible peel (avocados, mangoes, papayas) from single post-harvest applications in supervised trials in Belize, Brazil, Costa Rica and South Africa.

| Country; Year; Location[2] | Fruit; variety | Application | | | | | Sample | DAT | Residues, mg/kg | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Form | g ai/t | l/t | g ai/l | Type | | | | |
| S. Africa | avocado | - | | | 3.00 | | stoneless fruit<br>pulp<br>stoneless fruit<br>pulp<br>stoneless fruit<br>pulp | 0<br>0<br>3<br>3<br>7<br>7 | 3.8 (n=2)<br>0.48 (n=2)<br>2.0 (n=2)<br>0.34 (n=2)<br>1.6 (n=2)<br>0.25 (n=2) | 1977 (0311/8833/ P295) |
| S. Africa | avocado | - | | | 6.00 | | stoneless fruit<br>pulp;<br>stoneless fruit<br>pulp<br>stoneless fruit<br>pulp | 0<br>0<br>3<br>3<br>7<br>7 | 4.7 (n=2)<br>0.64 (n=2)<br>3.0 (n=2)<br>0.33 (n=2)<br>1.8 (n=2)<br>0.20 (n=2) | 1977 (0311/8833/ P295) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 450 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 6.2 | Rice 1997a (43327) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 450 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 7.1 | Rice 1997a (43327) |
| Costa Rica; 1996 San Jose[2] | avocado; Haas | SC 450 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 6.0 | Rice 1997a (43327) |
| Costa Rica; 1996 San Jose[2] | avocado; Haas | SC 450 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 6.9 | Rice 1997a (43327) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 450 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 4.8 | Rice 1997a (43327) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 450 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 5.6 | Rice 1997a (43327) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 483 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 8.1 | Rice 1997a (43327) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 483 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 6.7 | Rice 1997a (43327) |
| Costa Rica; 1996 San Jose[2] | avocado; Haas | SC 483 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 7.0 | Rice 1997a (43327) |
| Costa Rica; 1996 San Jose[2] | avocado; Haas | SC 483 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 7.8 | Rice 1997a (43327) |

| Country; Year; Location[2] | Fruit; variety | Application | | | | | Sample | DAT | Residues, mg/kg | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Form | g ai/t | l/t | g ai/l | Type | | | | |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 483 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 5.0 | Rice 1997a (43327) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 483 | | | 3.25 | 30 sec dip | stoneless fruit | 0 | 8.0 | Rice 1997a (43327) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 450 | | | 3.25 | spray to run off | stoneless fruit | 0 | 8.1; 8.9; 6.5; 7.0 | Rice 1997a (43327) |
| Costa Rica; 1996 San Jose[2] | avocado; Haas | SC 450 | | | 3.25 | spray to run off | stoneless fruit | 0 | 11; 10 (n=4) | Rice 1997a (43327) |
| Costa Rica; 1996 Dulce Nombre[2] | avocado; Haas | SC 483 | | | 3.25 | spray to run off | stoneless fruit | 0 | 8.5; 8.9; 11; 8.5 | Rice 1997a (43327) |
| Costa Rica; 1996 San Jose[2] | avocado; Haas | SC 483 | | | 3.25 | spray to run off | stoneless fruit | 0 | 14 (n=2); 8.8 | Rice 1997a (43327) |
| Brazil; 1994; Patos (CE)[1] Ceasa de Cariacica (ES)[2] | mango; Espada | WP 60 | | | 1.98 | 3 min dip | peel<br>pulp | 0<br>0 | 0.47<br><0.03 | Garozi 1996a |
| Brazil; 1994; Patos (CE)[1] Ceasa de Cariacica (ES)[2] | mango; Espada | WP 60 | | | 3.96 | 3 min dip | peel<br>pulp | 0<br>0 | 0.74<br>0.12 | Garozi 1996a |
| Brazil; 1994; Patos (CE)[1] Ceasa de Cariacica (ES)[2] | mango; Rosa | WP 60 | | | 1.98 | 3 min dip | peel<br>pulp | 0<br>0 | 0.56<br>0.03 | Garozi 1996b |
| Brazil; 1994; Patos (CE)[1] Ceasa de Cariacica (ES)[2] | mango; Rosa | WP 60 | | | 3.96 | 3 min dip | peel<br>pulp | 0<br>0 | 1.02<br>0.09 | Garozi 1996b |
| Belize; 1996; Dangria[2] | mango | SC 450 | | | 2.5 | 30 sec dip | stoneless fruit | 0 | 2.6, 1.5<br>1.7, 1.5 | Rice 1997b (43326) |
| Belize; 1996; Belize city[2] | mango | SC 450 | | | 2.5 | 30 sec dip | stoneless fruit | 0 | 2.6, 2.3 (n=3) | Rice 1997b (43326) |
| Belize; 1996; Dangria[2] | mango | SC 483 | | | 2.5 | 30 sec dip | stoneless fruit | 0 | 1.9, 1.8<br>1.3, 1.5 | Rice 1997b (43326) |
| Belize; 1996; Belize city[2] | mango | SC 483 | | | 2.5 | 30 sec dip | stoneless fruit | 0 | 2.1, 2.0 | Rice 1997b (43326) |
| Belize; 1996; Dangria[2] | mango | SC 450 | | | 2.5 | spray to run off | stoneless fruit | 0 | 3.2, 4.3<br>3.0, 3.6 | Rice 1997b (43326) |
| Belize; 1996; Belize city[2] | mango | SC 450 | | | 2.5 | spray to run off | stoneless fruit | 0 | 3.8, 4.6 | Rice 1997b (43326) |
| Belize; 1996; Dangria[2] | mango | SC 483 | | | 2.5 | spray to run off | stoneless fruit | 0 | 3.1, 2.7 (n=3)<br>3.0, 2.5 | Rice 1997b (43326) |
| Belize; 1996; Belize city[2] | mango | SC 483 | | | 2.5 | spray to run off | stoneless fruit | 0 | 3.9, 3.8 | Rice 1997b (43326) |
| Costa Rica; 1996 San Jose[2] | papaya; Havaiana | SC 450 | | | 2.0 | 30 sec dip | whole fruit | 0 | 2.0, 3.5<br>2.8, 3.8 | Rice 1997c (43325) |
| Costa Rica; 1996 Dulce Nombre[2] | papaya; Havaiana | SC 450 | | | 2.0 | 30 sec dip | whole fruit | 0 | 3.5, 3.2 | Rice 1997c (43325) |
| Costa Rica; 1996 San Jose[2] | papaya; Havaiana | SC 483 | | | 2.0 | 30 sec dip | whole fruit | 0 | 2.6 (n=3), 1.9<br>3.0, 3.2 | Rice 1997c (43325) |

| Country; Year; Location[2] | Fruit; variety | Application | | | | | Sample | DAT | Residues, mg/kg | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|---|
| | | Form | g ai/t | l/t | g ai/l | Type | | | | |
| Costa Rica; 1996 Dulce Nombre[2] | papaya; Havaiana | SC 483 | | | 2.0 | 30 sec dip | whole fruit | 0 | 2.8, 3.8 (n=4) | Rice 1997c (43325) |
| Costa Rica; 1996 San Jose[2] | papaya; Havaiana | SC 450 | | | 2.0 | spray to run off | whole fruit | 0 | 5.1, 5.0 4.9, 4.3 | Rice 1997c (43325) |
| Costa Rica; 1996 Dulce Nombre[2] | papaya; Havaiana | SC 450 | | | 2.0 | spray to run off | whole fruit | 0 | 5.1, 4.0 | Rice 1997c (43325) |
| Costa Rica; 1996 San Jose[2] | papaya; Havaiana | SC 483 | | | 2.0 | spray to run off | whole fruit | 0 | 2.6, 3.3 4.2, 3.9 | Rice 1997c (43325) |
| Costa Rica; 1996 Dulce Nombre[2] | papaya; Havaiana | SC 483 | | | 2.0 | spray to run off | whole fruit | 0 | 3.1, 3.8 | Rice 1997c (43325) |

[1] field location
[2] treatment location

Melons. Detailed reports were available for pre-harvest residue trials (not GLP) on melons in Spain (1997). The trials were carried out with SC formulations in a plastic greenhouse using a hydropneumatic backpack sprayer (3 x 0.9 kg ai/ha) with two plots and one control plot per trial. A single sample of five melons was taken from each plot for analysis. Details of storage conditions were not given. Samples were analysed by fluorescent HPLC, but analytical details were in Spanish. Thiabendazole was not detected in control samples (<0.005 mg/kg).

Table 11. Residues of thiabendazole in or on melons from pre-harvest applications in plastic greenhouses in supervised trials in Spain (1997). Whole fruit analysed.

| Location | Variety | Application | | | | | PHI, days | Residues, mg/kg | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|---|
| | | Form | No. | Interval, days | kg ai/ha | kg ai/hl | | | |
| Yegua Verde (Almeria) | Yupi | SC 480 | 3 | 14 | 0.9 | 0.2 | 0<br>7 | 0.74; 0.82<br>0.29; 0.33 | Sanchez 1997 (11R) |
| La Mojonera (Almeria) | Melina | SC 480 | 3 | 14 | 0.9 | 0.2 | 0<br>7 | 0.35; 0.39<br>0.57; 0.53 | Sanchez 1997 (12R) |
| La Mojonera (Almeria) | Aitana | SC 480 | 3 | 14 | 0.9 | 0.2 | 0<br>7 | 0.19; 0.17<br>0.081; 0.052 | Sanchez 1997 (13R) |
| La Mojonera (Almeria) | Yupi | SC 480 | 3 | 14 | 0.9 | 0.2 | 0<br>7 | 0.15; 0.12<br>0.30; 0.31 | Sanchez 1997 (14R) |
| La Mojonera (Almeria) | Melina | SC 480 | 3 | 14 | 0.9 | 0.2 | 0<br>3<br>7<br>14 | 0.34; 0.36<br>0.26; 0.28<br>0.23; 0.21<br>0.33; 0.42 | Sanchez 1997 (15R) |
| La Mojonera (Almeria) | Melina | SC 480 | 3 | 14 | 0.9 | 0.2 | 0<br>3<br>7<br>14 | 0.21; 0.24<br>0.18; 0.13<br>0.41; 0.40<br>0.44; 0.43 | Sanchez 1997 (16R) |
| La Puebla de Vicar (Almeria) | Amarillo canario | SC 480 | 3 | 14 | 0.9 | 0.2 | 0<br>3<br>7<br>14 | 0.67; 0.69<br>0.14; 0.19<br>0.13; 0.15<br>0.13; 0.11 | Sanchez 1997 (17R) |
| La Mojonera (Almeria) | Melina | SC 480 | 3 | 14 | 0.9 | 0.2 | 0<br>3<br>7<br>14 | 0.39; 0.46<br>0.49; 0.53<br>0.21; 0.20<br>0.036; 0.036 | Sanchez 1997 (18R) |

Ware potatoes. Detailed reports were available for post-harvest residue trials on ware potatoes in The Netherlands (1998). The GLP trials were carried out with single post-harvest spray applications of SC

formulations with commercial Mafex equipment (spinning disk; 4-5 min/t) at a nominal rate of 30 g ai/t and 60 g ai/t. Samples were stored under commercial conditions at 14-15ºC for one week, followed by storage at 9.5ºC for 2-3 weeks, then at 3-5ºC for five months (relative humidity 90%). Single samples of 22-49 potatoes (2.2-5.5 kg) were randomly collected and stored frozen at temperatures below -18ºC for 1-7 months. Homogenized samples were analysed for thiabendazole by modified method M-049 (extraction with 0.1 M HCl and HPLC with fluorescence detection). Low levels of thiabendazole (<0.02-0.05 mg/kg) were found in control samples. Analytical recoveries were 98-105% (n=8), 98% (n=1) and 102-107% (n=4) from fortifications at the LOD, at 0.2 mg/kg and at 2.0 mg/kg thiabendazole respectively.

Table 12. Residues of thiabendazole in or on ware potatoes from single post-harvest spinning-disc spray applications in supervised trials in Bant, The Netherlands (1998). Whole tubers with peel analysed.

| Variety | Application | | | | | DAT[1] | Residues, mg/kg | Ref. (Report no.) |
|---|---|---|---|---|---|---|---|---|
| | Form | g ai/t | l/t | g ai/l | Spin rate | | | |
| Agria | SC 500 | 37.6 | | | spray c 4 min/t | 0<br>20<br>57<br>122<br>178 | 3.3 (c<0.02)<br>2.6<br>3.6 (c<0.02)<br>4.0<br>5.6 (c<0.02) | Kissling 1999ag (2235/98) |
| Santé | SC 500 | 36.0 | | | spray 4 min/t | 0<br>20<br>57<br>122<br>178 | 2.4 (c=0.02)<br>1.7<br>2.3 (c<0.02)<br>1.9<br>1.9 (c<0.02) | Kissling 1999ah (2236/98) |
| Modesta | SC 500 | 33.4 | | | spray 5 min/t | 0<br>20<br>57<br>122<br>178 | 4.2 (c=0.03)<br>2.8<br>5.1 (c<0.02)<br>4.2<br>5.4 (c=0.02) | Kissling 1999ai (2237/98) |
| Bintje | SC 500 | 37.1 | | | spray 5 min/t | 0<br>20<br>57<br>122<br>178 | 4.6 (c<0.02)<br>5.5<br>8.0 (c<0.02)<br>6.9<br>6.7 (c=0.05) | Kissling; 1999aj (2238/98) |
| Agria | SC 500 | 64.4 | | | spray 5.5 min/t | 0<br>19<br>56<br>121<br>177 | 4.1 (c<0.02)<br>5.8<br>5.1 (c<0.02)<br>5.9<br>7.9 (c<0.02) | Kissling; 1999ak (2239/98) |
| Santé | SC 500 | 66.9 | | | spray 4 min/t | 0<br>19<br>56<br>121<br>177 | 2.5 (c=0.02)<br>2.4<br>1.9 (c<0.02)<br>2.1<br>3.2 (c<0.02) | Kissling; 1999al (2240/98) |
| Modesta | SC 500 | 64.3 | | | spray 5 min/t | 0<br>19<br>56<br>121<br>177 | 11 (c=0.03)<br>5.2<br>5.2 (<0.02)<br>6.5<br>11 (c=0.02) | Kissling; 1999am (2241/98) |
| Bintje | SC 500 | 78 | | | spray 5 min/t | 0<br>19<br>56<br>121<br>177 | 5.9 (c<0.02)<br>5.6<br>6.4 (c<0.02)<br>9.3<br>8.2 (c=0.05) | Kissling; 1999am (2242/98) |

[1] days after treatment

## FATE OF RESIDUES IN STORAGE AND PROCESSING

### In processing

Oranges. In two processing trials in Spain (1998) oranges were treated post-harvest with an SC 500 formulation by either (a) a single 30 sec drench application (2.0 g ai/l) on ripe fruit or (b) a 30 sec drench on green fruit plus a spray on degreened fruit two days later (1.8 + 6.0 g ai/l). After the application(s) the oranges were waxed with an emulsion containing 1 l/t "Teycer" and stored at 2-5ºC until sample collection. Samples were collected randomly by hand (200 kg) and stored at ambient temperatures until processing 3 days later. 100 kg oranges were then washed manually for 5 min in vessels containing cold tap water, and separate samples were processed in duplicate into juice and marmalade by simulated industrial processes.

To prepare juice and pomace 25-30 kg washed oranges were cut in half and squeezed in a citrus fruit squeezer. The raw juice was pasteurised for 20-25 min (2 min at 89ºC) in a plate pasteuriser. The pulp and peel were combined and cut in small pieces to produce wet pomace. The wet pomace was dried for 6-7 hours at 63-65ºC in a dry box until the moisture content was 10%, yielding dry pomace.

Marmalade was prepared from 0.5 kg washed fruit with peel cut in a slicing machine. The cut oranges were mixed with glucose syrup, sugar and citric acid, the mixture boiled for 3 min and mixed with a pectin/sugar solution at 75ºC. The marmalade was cooled for two hours and sampled.

Processed samples were stored below -18ºC until analysis. Thiabendazole was determined according to modified method M-049 (extraction with 0.1 M HCl and HPLC with fluorescence detection). Residues in whole fruit were calculated from the weights of pulp and peel (51-61% of pulp). The LOD was 0.01 mg/kg in pulp, juice and marmalade, 0.02 mg/kg in peel, and 0.10 mg/kg in wet and dry pomace. The calculated LOD for whole fruit was 0.02 mg/kg thiabendazole. In pulp control samples thiabendazole was below the LOD, but peel and pomace control samples contained low levels of residue (≤0.31 mg/kg). Analytical recoveries were 72%-111% (n=19) from fortifications at the LOD and at 0.02, 0.05, 0.1, 0.2, 1.0, 2.0 and 10 mg/kg thiabendazole.

Apples. In a processing trial in France (1998) apples were treated post-harvest with a 120 sec drench application of an SC 500 formulation using commercial equipment (1.1 g ai/l). Apples (200 kg) were collected randomly by hand and stored cold at 0-2ºC in a normal atmosphere (93-100% relative humidity) until processing after 14-15 days. The apples were washed manually for 5 min in vessels containing cold tap water, and separate samples were processed in duplicate into juice and sauce by simulated industrial processes.

To prepare juice and pomace 40 kg washed apples were sliced in a slicer and pressed in a juice press (pressure 1-2 MPa), separating the mash into raw juice and wet pomace. The insoluble components of the raw juice were removed in a separator and the juice was pasteurized in a plate pasteuriser (2 min; 85ºC ). Wet pomace was dried for 8-9 hours at 63-65ºC in a dry box until the moisture content was 10% to produce dry pomace.

To prepare apple sauce 1.5 kg washed apples were cut in halves and boiled in water (0.5 l water + 1.5 kg fruit) for 15 min at 98-100ºC. The fruit were passed through a sieve and sugar (16.5% of the dry matter), citric acid and ascorbic acid were added (pH 3.0-4.5). The apple sauce was transferred to a 1.5 l tin. After closing, the tin was pasteurized for 20 min at 95ºC in an autoclave.

Processed samples were stored below -18ºC until analysis. Thiabendazole was determined according to modified method M-049 (HPLC with fluorescence detection). The LOD was 0.01 mg/kg in juice, 0.02 mg/kg in fruit, wet pomace and purée, and 0.2 mg/kg in dry pomace. Thiabendazole in control samples was below the LOD. Analytical recoveries were 92-105% (n=14) in fruit, juice, wet

pomace and dry pomace from fortifications at the LOD and at 0.02-0.2-1.0-2.0-6.0 mg/kg thiabendazole. For purée the analytical recoveries were 119% at 0.02 mg/kg and 97% at 2.0 mg/kg.

Ware potatoes. In a processing trial in The Netherlands (1998) ware potatoes were treated with a post-harvest spray application of an SC 500 formulation with commercial Mafex equipment (spinning disc; 5 min/t; 78 g ai/t). Samples were stored under commercial conditions at 14-15ºC for one week, then at 9.5ºC for one week, and finally at 3-5ºC (relative humidity 90%) until processing after 22 or 62 days. The potatoes were washed and divided into four parts for four processes: boiling, microwave boiling, deep frying and crisp production. Each quarter was divided into two or four sub-samples, each of which was individually processed.

*Washing.* 60 kg unpeeled potatoes were washed manually for 5 min in vessels containing cold tap water.

*Boiling*. 2 kg washed unpeeled tubers were completely covered with water and boiled at 100ºC (15-20 min until beginning of boiling + 28-30 min boiling).

*Microwave boiling.* 2 kg washed unpeeled tubers plus 200 ml water were microwave-cooked for 11 min at 800 W and allowed to cool for 2 min.

*Deep frying*. 5 kg washed unpeeled tubers were peeled with an industrial peeler and cut into potato sticks with a slicing machine. The sticks were blanched for 3 min at 50-82ºC and deep fried for 2 min at 180ºC in pure vegetable oil, then deep-frozen below -18ºC. The deep-frozen chips were finally fried at 180ºC in a deep fat fryer with pure vegetable oil to a golden brown colour (15-19 min).

*Crisp production.* 5 kg washed unpeeled tubers were peeled with an industrial peeler and sliced into 1 mm discs with a slicing machine. The discs were fried at 180ºC in a deep fat fryer with pure vegetable oil (12-14 min).

Processed samples were stored frozen at temperatures below -18ºC . Homogenized samples were analysed for thiabendazole by modified method M-049. Residues in the whole potato (washed and unwashed) were calculated from the residues in the pulp and peel and the weights of the peel and pulp. The LOD was 0.01 mg/kg in peeled potatoes (fresh, boiled or deep fried), crisps and cooking liquid, and 0.02 mg/kg in peel and whole potatoes (fresh or boiled). Thiabendazole residues in control samples were below the LOD. Analytical recoveries were 92-108% (n=29) from fortifications at the LOD and at 0.02-0.1-1.0-2.0-4.0-5.0-10.0 mg/kg thiabendazole.

Table 13. Residues of thiabendazole in processed fractions of oranges, apples and ware potatoes treated post-harvest (1998).

| Crop; variety; Country; Location | Sample | DAT | Residues, mg/kg (c=control) | Processing factor | Ref. (Report no.) |
|---|---|---|---|---|---|
| Orange; Navelina Spain; Carcer[1,2] (Valencia) | whole fruit | 10 | 0.37 (c=0.06) | - | Kissling, 1999m (2280/98) treatment a |
| | peel (unwashed) | | 0.87 | 2.4 | |
| | pulp (unwashed) | | <0.01 | - | |
| | whole fruit, washed | | 0.13 | 0.35 | |
| | peel (washed) | | 0.32 | | |
| | pulp (washed) | | <0.01 | | |
| | pasteurized juice | | 0.04; 0.05 | 0.11; 0.14 | |
| | pomace, wet | | 0.39 (c=0.02) | 1.05 | |
| | pomace, dry | | 1.8 (c=0.31) | 4.84 | |
| | marmalade | | 0.06; 0.11 | 0.16; 0.30 | |

| Crop; variety; Country; Location | Sample | DAT | Residues, mg/kg (c=control) | Processing factor | Ref. (Report no.) |
|---|---|---|---|---|---|
| Orange; Navelina Spain; Carcer[1,2]; Paterna[3] | whole fruit | 3 | 1.3 | - | Kissling, 1999r (2288/98) |
| | peel (unwashed) | | 3.4 | 2.6 | |
| | pulp (unwashed) | | <0.01 | - | |
| | whole fruit, washed | | 1.1 | 0.85 | |
| | peel (washed) | | 2.5 | | treatment b |
| | pulp (washed) | | <0.01 | | |
| | pasteurized juice | | 0.05; 0.04 | 0.04; 0.03 | |
| | pomace, wet | | 1.8 | 1.36 | |
| | pomace, dry | | 8.6 | 6.48 | |
| | marmalade | | 0.42; 0.52 | 0.32; 0.39 | |
| Apple; Golden Delicious; France (North); Lignieres-de-Touraine[2] | whole fruit | 14 | 1.93 juice; 1.90 purée | - | Kissling, 1999af (2250/98) |
| | washed fruit | | 1.50; 1.12 | 0.78; 0.58 | |
| | pasteurized juice | | 0.80; 0.83; 0.95; 1.04 | 0.41; 0.43; 0.49; 0.54 | |
| | pomace, wet | | 1.56; 1.93 | 0.81; 1.00 | |
| | pomace, dry | | 6.53; 7.67; 8.41; 9.45 | 3.38; 3.97; 4.36; 4.90 | |
| | apple purée | | 0.71; 0.74; 0.89; 0.76 | 0.37; 0.39; 0.47; 0.40 | |
| ware potato; Bintje; Netherlands Bant[2] | whole potato | 22 | 11 | - | Kissling; 1999am (2242/98) |
| | potato, peeled | | 0.80 | 0.07 | |
| | potato, peel | | 55 | 5.0 | |
| | washed whole potato | | 2.8; 3.4 | 0.25; 0.31 | |
| | washed potato, peeled | | 0.33; 0.29 | 0.03; 0.03 | |
| | washed potato, peel | | 14.7; 21.1 | 1.31; 1.88 | |
| | whole potato, boiled | | 1.3; 1.2 | 0.12; 0.10 | |
| | boiled potato, peeled | | 0.73; 0.67 | 0.07; 0.06 | |
| | cooking liquid (boiling) | | 4.6; 4.5 | | |
| | whole potato, mw boiled | | 2.9; 3.9; 2.8; 3.3 | 0.26; 0.35; 0.25; 0.29 | |
| | mw boiled potato, peeled | | 1.8; 1.6; 1.8; 2.1 | 0.16; 0.14; 0.17; 0.18 | |
| | cooking liquid (mw) | | 6.2; 7.0 | | |
| | potato, deep fried | | 0.06; 0.17; 0.12; 0.19 | 0.005; 0.015; 0.011; 0.017 | |
| | potato crisps | | 0.06; 0.06; 0.09; 0.06 | 0.005; 0.005; 0.008; 0.005 | |
| ware potato; Bintje; Netherlands Bant[2] | whole potato | 62 | 12 | - | Kissling; 1999am (2242/98) |
| | potato, peeled | | 0.98 | 0.08 | |
| | potato, peel | | 72 | 6 | |
| | washed whole potato | | 2.8; 3.4 | 0.23; 0.28 | |
| | washed potato, peeled | | 0.20; 0.35 | 0.02; 0.03 | |
| | washed potato, peel | | 16; 17 | 1.27; 1.38 | |
| | whole potato, boiled | | 0.56; 0.93 | 0.05; 0.08 | |
| | boiled potato, peeled | | 0.83; 0.92 | 0.07; 0.07 | |
| | cooking liquid (boiling) | | 2.8; 3.8 | | |
| | whole potato, mw boiled | | 2.3; 2.8; 4.9; 3.3 | 0.19; 0.23; 0.40; 0.27 | |
| | mw boiled potato, peeled | | 1.2; 1.7; 2.2; 1.5 | 0.10; 0.14; 0.18; 0.12 | |
| | cooking liquid (mw) | | 5.4; 7.4 | | |
| | potato, deep fried | | 0.04; 0.08; 0.10; 0.12 | 0.003; 0.007; 0.008; 0.010 | |
| | potato crisps | | 0.02; 0.06; 0.08; 0.02 | 0.002; 0.005; 0.007; 0.002 | |

DAT = days after treatment
mw boiled = microwave boiled
[1] field location
[2] first treatment location
[3] second treatment location

## RESIDUES IN THE EDIBLE PORTION OF FOOD COMMODITIES

The available information is reported above in ‘Fate of residues in processing’.

## RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Fruit, vegetables and potatoes were analysed in The Netherlands in 1994-1996, 1997 and 1998 for residues of thiabendazole (Anon., 2000). Details of sample collection and analytical methods were not given. Samples in which residues were found are tabulated in Table 14. In 1994-1996 residues were not found in pomelos (n=6), apricots (n=80), peaches (n=252), nectarines (n=221), blackberries (n=244), blue hilberries (n=104), pineapples (n=75), beetroot (n=83), carrots (n=407), small onions (n=97), peppers (n=1525), aubergines (n=148), courgettes (n=206), sweet corn (n=27), brassica vegetables (n=968), leafy vegetables except iceberg lettuce (n=6182), legume vegetables (n=786), fennel (n=52), leeks (441) or rhubarb (n=62).

Table 14. Results of monitoring in The Netherlands of food in commerce (1994-1996, 1997 and 1998).

| Group | Commodity | Samples analysed | | | Samples with residues[1] | | | Mean residue, mg/kg | | | Maximum residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | '94-'96 | '97 | '98 | '94-'96 | '97 | '98 | '94-'96 | '97 | '98 | '94-'96 | '97 | '98 |
| Citrus fruit | Grapefruit | 301 | 87 | 73 | 129 | 28 | 4 | 0.39 | 0.40 | <0.05 | <6.0 | <6.0 | <6.0 |
| | Lemon | 243 | 51 | 24 | 50 | 2 | 3 | 0.22 | <0.05 | 0.07 | <6.0 | <6.0 | <6.0 |
| | Lime | 13 | 10 | 8 | 0 | 1 | 1 | | <0.05 | <0.05 | | <6.0 | <6.0 |
| | Orange | 902 | 335 | 124 | 328 | 108 | 29 | 0.55 | 0.55 | 0.31 | 7.4 | ? | <6.0 |
| | Tangerine | 560 | 112 | 70 | 190 | 21 | 16 | 0.07 | 0.28 | 0.30 | 6.5 | <6.0 | <6.0 |
| Pome fruit | Apple | 1495 | 398 | 93 | 46 | 4 | 1 | <0.05 | <0.05 | <0.05 | <5.0 | <5.0 | <5.0 |
| | Pear | 366 | | 32 | 6 | | 2 | <0.05 | | 0.12 | <5.0 | | <5.0 |
| Stone fruit | Plum | 437 | | | 7 | | | <0.05 | | | 0.13 | | |
| Berries | Grapes | 667 | | | 7 | | | <0.05 | | | 0.15 | | |
| | Strawberry | 2378 | 779 | | 6 | 2 | | <0.05 | <0.05 | | <5.0 | <5.0 | |
| | Raspberry | 247 | | | 12 | | | <0.05 | | | 0.15 | | |
| | Currants (red, white, black) | 450 | | | 2 | | | <0.05 | | | 0.06 | | |
| Miscella-neous fruit | Avocado | 125 | | 25 | 1 | | 1 | <0.05 | | 0.06 | 0.13 | | 1.8 |
| | Banana | 57 | 38 | 14 | 9 | 10 | 3 | <0.05 | 0.19 | 0.16 | <3.0 | ? | <3.0 |
| | Fig | 48 | | | 2 | | | <0.05 | | | 0.07 | | |
| | Kiwifruit | 223 | | | 7 | | | <0.05 | | | 0.49 | | |
| | Mango | 191 | 63 | 43 | 8 | 2 | 2 | 0.12 | 0.10 | 0.11 | 10 | 2.9 | ? |
| | Passion fruit | 40 | | | 2 | | | <0.05 | | | 0.44 | | |
| Other fruit and fruit products | - | 385 | 152 | 51 | 9 | 3 | 1 | 0.09 | 0.07 | 0.18 | 5.2 | ? | 6.7 |
| Root and tuber vegetables | Radish | 1010 | | | 1 | | | <0.05 | | | 0.06 | | |
| Fruiting vegetables | Tomato | 1108 | 1242 | | 14 | 6 | | <0.05 | <0.05 | | <2.0 | <2.0 | |
| | Cucumber | 951 | 249 | | | 1 | | | <0.05 | | | 0.12 | |
| | Melon | 390 | 104 | 51 | 11 | 1 | 2 | 0.08 | <0.05 | <0.05 | 3.3 | 0.25 | ? |
| Brassica vegetables | Broccoli | 154 | | 42 | 0 | | 2 | | | <0.05 | | | <5.0 |
| Leafy vegetables | Iceberg lettuce | 471 | | | 1 | | | <0.05 | | | 0.09 | | |
| | Endive | 1137 | | 234 | 0 | | 1 | | | <0.05 | | | 1.9 |
| | Other herbs | | | 27 | | | 1 | | | <0.05 | | | 0.11 |
| Stem vegetables | Celery | 233 | 187 | | 2 | 2 | | <0.05 | <0.05 | | 0.60 | 0.74 | |
| Potatoes | Ware potato | 325 | 1437 | | 14 | 1 | | <0.05 | <0.05 | | <5.0 | <5.0 | |
| Other arable products | - | 699 | 97 | | | 2 | | | <0.05 | | | 1.6 | |

[1] Limit of determination 0.05 mg/kg

## NATIONAL MAXIMUM RESIDUE LIMITS

National MRLs reported to the Meeting are shown below.

For plant products the residue is defined as thiabendazole. For animal products the residue is defined as the sum of thiabendazole and 5-hydroxy-thiabendazole, expressed as thiabendazole.

| Country | Commodity | MRL, mg/kg, expressed as thiabendazole | Residue definition |
|---|---|---|---|
| The Netherlands | Citrus fruit | 6 | thiabendazole |
| | Nuts | 0.1* | thiabendazole |
| | Pome fruit | 5 | thiabendazole |
| | Strawberry | 5 | thiabendazole |
| | Banana | 3 | thiabendazole |
| | Onions | 0.1 | thiabendazole |
| | Tomato | 2 | thiabendazole |
| | Broccoli | 5 | thiabendazole |
| | Ware potato | 5 | thiabendazole |
| | Tea | 0.1* | thiabendazole |
| | Hops | 0.1* | thiabendazole |
| | Wheat | 0.2 | thiabendazole |
| | Rice | 0.2 | thiabendazole |
| | Other vegetable products | 0.05 | thiabendazole |
| | Muscle, fat, liver, kidney (cow) | 0.1 | thiabendazole plus 5-OH-thiabendazole |
| | Milk | 0.1 | thiabendazole plus 5-OH-thiabendazole |
| | Other animal products | 0.1* | thiabendazole plus 5-OH-thiabendazole |
| Poland | Citrus fruit | 10 | thiabendazole |
| | Banana | 3.0 | thiabendazole |
| | Cereal grains | 0.05 | thiabendazole |
| | Potato | 0.05 | thiabendazole |
| | Eggs | 0.1 | thiabendazole? |

## APPRAISAL

Thiabendazole was evaluated by the Joint Meeting in 1997 within the periodic review programme of the CCPR, when it recommended the withdrawal of MRLs for a number of commodities. At its twenty-third session, the CCPR (ALINORM 99/24A, para 65) decided to retain the Codex MRLs for apples, citrus fruits, pears, and strawberries, as new data would become available for review by the Joint Meeting in 2000. Information was made available to the present Meeting on analytical methods for animal products, GAP, and the results of supervised trials on mandarins, oranges, apples, pears, strawberries, avocados, mangoes, papayas, melons, and potatoes.

Thiabendazole is registered in many countries for use as a fungicide before and after harvesting and as a drug in veterinary and human medicine. Its main use in plant protection is after harvesting.

### *Methods of analysis*

Two methods were validated at a LOQ of 0.03 mg/kg for thiabendazole, benzimidazole, and 5-hydroxy-thiabendazole in meat (dairy and poultry), poultry skin with attached fat, eggs, cattle liver,

and cattle kidney. After fortification at 0.03 mg/kg, the mean recovery was 70-99% for thiabendazole, 66-94% for benzimidazole, and 72-105% for 5-hydroxythiabendazole.

***Definition of the residue***

The 1997 JMPR defined the residue in plant products for compliance with MRLs and for estimation of dietary intake as thiabendazole. For animal products, the residue is defined as the sum of thiabendazole and 5-hydroxythiabendazole for compliance with MRLs, and as the sum of thiabendazole, 5-hydroxythiabendazole, and its sulfate conjugate for estimation of dietary intake.

***Results of supervised trials and stability of residues in stored samples***

Trials of one or two post-harvest applications were conducted in Spain in 1998 on *orange* and *mandarin*. In eight trials (four on mandarins, four on oranges), the application rate of 2.0 g ai/l corresponded to the Spanish use pattern for single drench applications (0.5-2.2 g ai/l). In five trials (two on mandarins, three on oranges), the rate of 1.8 g ai/l (drench) plus 6.0 g ai/t (spray) were conducted according to the Spanish use pattern for double drench and spray applications (1.0-1.8 g ai/l drench plus 3.3-7 g ai/t spray). Four trials (two on mandarins, two on oranges) of spraying at 3.0 g ai/t plus 4.0 g ai/t were conducted for a new registration and did not correspond to an existing GAP; these data could therefore not be used to estimate maximum residue levels.

The concentrations of residues in mandarins (whole fruit) remained stable or decreased slightly during storage up to 15 days. The concentrations were higher after longer single drench treatment (30 s versus 150 s) and with more treatments (single versus double). With the double treatments, there was no difference between short and long drenching times. The concentrations of residues of thiabendazole in trials that complied with the GAP (median in italics) were 0.50, 0.65, ***1.4***, ***1.6***, and 2.2 (2 trials) mg/kg in the whole fruit and *0.01* (3 trials), *0.03*, 0.04, and 0.09 mg/kg in the pulp.

The concentrations of residues in oranges (whole fruit) remained stable or decreased slightly during storage up to 15 days. The concentrations were higher after longer single drench treatment (30 s versus 150 s) and with more treatments (single versus double). With the double treatments, there was no difference between short and long drenching times. The concentrations of residues of thiabendazole in trials that complied with the GAP were 0.40, 0.53, 1.1, ***1.2***, 1.6 (2 trials), and 1.9 mg/kg in the whole fruit and <*0.01* (4 trials) and 0.01 (3 trials) mg/kg in the pulp.

The Meeting observed that the results for mandarin and orange are comparable. Since this is to be expected from post-harvest treatment, the Meeting decided to combine the data on these two citrus fruits. The concentrations of residues of thiabendazole in trials on mandarin and orange that complied with GAP were 0.40, 0.50, 0.53, 0,65, 1.1, 1.2, ***1.4***, 1.6 (3 trials), 1.9, and 2.2 (2 trials) mg/kg in the whole fruit and <0.01 (4 trials), ***0.01*** (6 trials), 0.03, 0.04, and 0.09 mg/kg in the pulp.

The Meeting estimated a maximum residue level of 3 mg/kg for thiabendazole in unwashed whole citrus fruit arising from double post-harvest application (drench plus spray) to replace the previous recommendation of 10 mg/kg for citrus fruits. The Meeting estimated an STMR value of 0.01 mg/kg and a HR value of 0.09 mg/kg for thiabendazole in the edible part of citrus fruit (pulp).

Post-harvest residue trials were conducted in northern France in 1998 and in Spain in 1991 on *apple* and *pear* treated by a single post-harvest dip or drenching at 1.1 g ai/l. Eight trials carried out in France were evaluated against the Belgian use pattern for apples and pears of 1.0 g ai/l. The concentrations of residues were maintained during storage up to 30 days, and those after dipping or drenching for 45-120 s were similar. Four trials in Spain, two on apples and two on pears, were conducted according to the national use pattern (0.9-1.3 g ai/l) but could not be evaluated because of conflicting analytical results and insufficient detail in the description of the analytical methods used.

The concentrations of thiabendazole residues in the trials in France were 1.5, 1.6, ***1.7*** (4 trials), 1.9, and 2.0 mg/kg**.**

One trial of the use of a paste formulation of thiabendazole for the treatment of wounds was conducted in Germany in 1998 on apple trees before flowering. The trial was according to the national GAP. It confirmed that residues would not be expected in pome fruit after wound treatment (<0.05 mg/kg).

Although all the information available came from trials on apples, the Meeting considered that similar residues would be found in pears treated after harvesting. The Meeting therefore estimated a maximum residue level of 3 mg/kg for thiabendazole in pome fruit (apple and pear) arising from single post-harvest applications. Since no new data were provided on pre-harvest uses on apples and pears, the Meeting confirmed the 1997 decision to recommend the withdrawal of the CXLs of 10 mg/kg for apples and 10 mg/kg for pears for these uses. The Meeting estimated an STMR value of 1.7 mg/kg and a HR value of 2.0 mg/kg for thiabendazole in pome fruit (apple and pear) arising from single post-harvest applications.

Seven trials were conducted in Spain in 1989, 1991, and 1993 on *strawberry* sprayed once before harvesting. The concentrations of residues decreased with longer intervals before harvesting (up to 14 days). The trials in 1991 and 1993 (one in macro-tunnels and four in the open field) at 90 g ai/hl and the trials in 1989 (one under plastic and one in the open field) at 70 g ai/hl were conducted according to the Spanish use pattern (Annex 6, reference 80, p. 795: 0.30-0.90 kg ai/ha, 45-90 g ai/hl; PHI, 3 days). The concentrations of residues were similar in trials conducted indoors and in the open field. The concentrations of thiabendazole in trials that complied with the Spanish GAP were 0.43, 1.3, ***1.6*** (2 trials), 2.3, 2.6, and 2.7 mg/kg.

On the basis of the seven Spanish trials, the Meeting estimated a maximum residue level of 5 mg/kg, an STMR value of 1.6 mg/kg, and a HR value of 2.7 mg/kg for strawberries.

Eighteen trials of post-harvest application were conducted on *avocado* in South Africa in 1977 and Costa Rica in 1996. The concentrations of residues decreased during storage. The trials in South Africa (one at 3.0 g ai/l and one at 6.0 g ai/l) were not conducted according to the national use pattern (0.35-1.36 g ai/l), but the use pattern in Kenya (1.1-3.4 g ai/l) can be used to evaluate the trial conducted at 3.0 g ai/l, which showed a residue concentration on the day of treatment of 3.8 mg/kg in stoneless fruit. The 16 trials in Costa Rica, conducted at 3.25 g ai/l of suspension concentrate formulations applied by dip or spray, were also evaluated against the Kenyan use pattern. Spray application resulted in higher concentrations than a 3-min dip. The concentrations of thiabendazole in trials that complied with the Kenyan GAP were 3.8, 4.8, 5.0, 5.6, 6.0, 6.2, 6.7, 6.9, ***7.0***, 7.1, 7.8, 8.0, 8.1, 8.9, 11 (2 trials), and 14 mg/kg in stoneless fruit. In the two South African trials, residues were measured in both stoneless fruit and flesh 0, 3, and 7 days after treatment, providing ratios of residue in flesh versus stoneless fruit of 0.13 ± 0.02.

According to the Codex classification, the portion of the commodity to which the MRL applies and which is analysed is the whole commodity after removal of the stone but calculated on the basis of the whole fruit. The manufacturer provided a study which indicated that the avocado stone contributes about 15% to the weight of the whole fruit. Therefore, the concentrations of residues were multiplied by a factor of 1/1.15 = 0.87, resulting in concentrations of 3.3, 4.2, 4.4, 4.9, 5.2, 5.4, 5.8, 6.0, ***6.1***, 6.2, 6.8, 7.0 (2 trials), 7.7, 9.6 (2 trials), and 12 mg/kg in the whole fruit.

In order to obtain the values for the edible portion of avocado, the concentrations in stoneless fruit were multiplied by the ratio of 0.13 for concentrations in flesh versus stoneless fruit obtained in the South African trials. This yielded concentrations in avocado flesh of 0.5, 0.6, 0.7 (2 trials), 0.8 (2 trials), ***0.9*** (4 trials), 1.0 (2 trials), 1.1, 1.2, 1.4 (2 trials), and 1.8 mg/kg.

The Meeting estimated a maximum residue level of 15 mg/kg for thiabendazole in avocado (whole fruit) arising from a single post-harvest application. The Meeting estimated an STMR value of 0.9 mg/kg and a HR value of 1.8 mg/kg for thiabendazole in the edible portion of avocado.

Twelve trials on *mango* treated by dip or spray after harvesting were conducted in Brazil in 1994 and Belize in 1996. For the trials in Brazil (two at 1.98 g ai/l and two at 3.96 g ai/l by dipping in a wettable powder formulation), no national use pattern was available, but that in Venezuela (drenching; 0.9-1.8 g ai/l) can be used to evaluate the two trials at 1.98 g ai/l. The concentrations of residues on the day of treatment were <0.03 and 0.03 mg/kg for pulp; those in whole fruit were not reported. For the eight trials in Belize (at 2.5 g ai/l by dipping or spraying with suspension concentrate formulations), no national use pattern was available, but that of Guatemala (1.0-2.5 g ai/l) can be used. The concentrations of residues in stoneless fruit (with peel) were 1.9, 2.1, and *2.6* mg/kg after dipping and ***3.1***, 3.9, 4.3, and 4.6 mg/kg after spraying. The Meeting noted that, contrary to what might be expected, spray application resulted in marginally higher concentrations of residues. It nevertheless decided to combine the data sets.

According to the Codex classification, the portion of the commodity to which the MRL applies and which is analysed is the whole commodity after removal of the stone but calculated on the basis of the whole fruit. The manufacturer provided a study which indicated that the mango stone contributes 20-23% to the weight of the whole fruit. Therefore, the concentrations of residues were multiplied by a factor of 1/1.20 = 0.83, yielding values of 1.6, 1.7, ***2.2*** (2 trials), ***2.6***, 3.2, 3.6, and 3.8 mg/kg in the whole fruit.

The Meeting estimated a maximum residue level of 5 mg/kg for thiabendazole in whole mangoes arising from a single post-harvest application. The Meeting estimated an STMR value of 2.85 mg/kg and a HR value of 4.6 mg/kg for thiabendazole in stoneless mangoes, since insufficient information on the residue in the edible portion was available.

Eight trials were conducted in Costa Rica in 1996 on *papaya* treated after harvesting at 2.0 g ai/l by dipping or spraying with suspension concentrate formulations. They were evaluated in comparison with the GAP in the USA (1.0-2.0 g ai/l). The concentrations of thiabendazole were 3.2, 3.5, ***3.8*** (3 trials), 4.2, and 5.1 (2 trials) mg/kg.

The Meeting estimated a maximum residue level of 10 mg/kg for thiabendazole in whole papayas arising from a single post-harvest application. The Meeting estimated an STMR value of 3.8 mg/kg and a HR value of 5.1 mg/kg in whole papayas, as information was not available on the residue in the edible portion.

Trials were conducted in Spain in 1997 on *melon* in plastic greenhouses sprayed three times before harvesting. Eight trials at three applications of 0.9 kg ai/ha (200 g ai/hl) were conducted according to the Spanish use pattern (Annex 6, reference 80, p. 795: 0.45-0.90 kg ai/ha; 68-90 g ai/hl; PHI, 3 days). In four of the trials, residues were measured only 0 and 7 days after treatment. Data from day 0 in these trials and from day 3 in the other four trials were used, except when the values obtained after the longer PHI were higher. The concentrations in whole fruit were 0.19 (2 trials), 0.31, ***0.42***, ***0.44***, 0.53, 0.57, and 0.82 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg for thiabendazole in whole melons arising from pre-harvest application. The Meeting estimated an STMR value of 0.43 mg/kg and a HR value of 0.82 mg/kg for thiabendazole in whole melon, as information on the residue in the edible portion was not available.

Eight trials of a single post-harvest application of suspension concentrate formulations by spinning-disc spray were conducted on *potato* in The Netherlands in 1998. Four trials at 30 g ai/t were conducted according to the Dutch use pattern for this formulation (30 g ai/t; PHI, 60 days). For the remaining four trials (at 60 g ai/t), no Dutch use pattern was available, and the French use pattern (60

g ai/t; spray; no PHI specified) was used. The concentrations of residue in unpeeled tubers remained unchanged during storage up to 178 days. As the values in the trials at 30 and 60 g ai/t were not significantly different, the residues were treated as one group. The highest concentrations of residues in whole potato tubers were 2.4, 3.2, 5.4, ***5.6***, ***7.9***, 8.0, 9.3, and 11 mg/kg.

In trials of post-harvest treatment of potatoes from the United Kingdom and the USA evaluated by the Meeting in 1997, the concentrations of residues of thiabendazole on unwashed potatoes were 1.9, 2.0, 2.2, 2.4, ***2.6***, ***4.2***, 5.4, 5.5, 7.3, and 11 mg/kg.

Since the data sets evaluated in 1997 and by the present Meeting represent the same population, the Meeting decided to combine the two. The concentrations were 1.9, 2.0, 2.2, 2.4 (2 trials), 2.6, 3.2, 4.2, 5.4 (2 trials), 5.5, 5.6, 7.3, 7.9, 8.0, 9.3, and 11 (2 trials) mg/kg. The Meeting confirmed the maximum residue level of 15 mg/kg for thiabendazole in whole potatoes proposed by the 1997 JMPR. The present Meeting established an STMR value of 5.4 mg/kg to replace the previous STMR value of 3.4 mg/kg and a HR value of 11 mg/kg.

***Fate of residues during processing***

Information was provided to the Meeting on the fate of thiabendazole during the processing of oranges, apples, and potatoes.

Washing removed 19-65% of the residue from *oranges*, and the remainder was concentrated in wet and dry pomace. The calculated mean processing factors in two trials of industrial processing of oranges were 0.6 for washing, 0.08 for pasteurized juice, 0.3 for marmalade, *1.2* for wet pomace, and *5.7* for dry pomace.

The processing factors derived by the 1997 JMPR related to washed fruit, except those for marmalade made in a preserving pan (0.32) and in a microwave oven (0.37), which agree with that calculated by the present Meeting.

The median residue concentrations in processed commodities (STMR-P) calculated from the processing factors and the STMR value for unwashed whole citrus fruit (1.4 mg/kg) in supervised trials were 0.08 x 1.4 = 0.11 mg/kg for pasteurized orange juice; 0.3 x 1.4 = 0.42 mg/kg for marmalade; 1.2 x 1.4 = 1.7 mg/kg for wet orange pomace; and 5.7 x 1.4 = 8.0 mg/kg for dry orange pomace.

Washing removed 20-42% of the residue from *apples*, and the residue was concentrated in dry pomace. The calculated industrial processing factors for *apples* were *0.68* for washing, 0.47 for pasteurized juice, 0.41 for apple sauce, 0.92 for wet pomace, and 4.2 for dry pomace.

The median residue concentrations in processed commodities (STMR-P) calculated from the processing factors and the STMR value for whole apples (1.7 mg/kg) were 0.47 x 1.7 = 0.8 mg/kg for pasteurized apple juice; 0.41 x 1.7 = 0.7 mg/kg for apple sauce (purée); 0.92 x 1.7 = 1.6 mg/kg for wet apple pomace; and 4.2 x 1.7 = 7.1 mg/kg for dry apple pomace.

The Joint Meeting in 1997 discussed processing studies on potatoes extensively and noted that residues are transferred from the peel to the potatoes during peeling, as the average concentration was 1.54 mg/kg in potatoes peeled before washing and 0.08 mg/kg in those peeled after washing. Potatoes are always washed before peeling during industrial processing and either before or after peeling or both in the kitchen. In 1997, the Meeting therefore concluded that it was more appropriate to estimate the effect of peeling washed potatoes. By multiplying the STMR value derived in 1997 by the processing factors for both washing and peeling, the 1997 Meeting estimated an STMR-P value for washed peeled potatoes of 0.02 mg/kg and an STMR-P value of 0.44 mg/kg for washed potatoes.

The effects of frying, microwave and oven cooking, washing, boiling, baking, and crisping of potatoes treated with thiabendazole were also evaluated in 1997. That Meeting concluded that baking and frying did not change the residue content substantially. Furthermore, noting that baked potatoes may be consumed with or without the peel and fried potatoes may be prepared in a variety of ways, the 1997 Meeting recommended the use of STMR-P values for washed potatoes ( 0.44 mg/kg) and for washed peeled potatoes (0.02 mg/kg) for estimating dietary intake.

The present Meeting received the results of a new trial of the effects of washing, boiling, and frying of potatoes and the preparation of potato crisps. The calculated mean processing factors for unwashed potatoes were 0.08 for peeling, 0.27 for washing, 0.03 for peeling and washing, 0.09 for boiling, 0.07 for peeling after boiling, 0.29 for microwave boiling, 0.15 for peeling after microwave boiling, 0.01 for deep frying, and 0.00 for crisp processing. The mean of the processing factors for washing derived in 1997 and by the present Meeting was 0.2.

The Meeting agreed with the decision of the 1997 Meeting to use only the STMR-P values for washed and washed and peeled potatoes for estimating dietary intake. The median residue concentrations in processed commodities (STMR-P) calculated from the processing factors and the STMR value for unwashed whole potatoes (5.4 mg/kg) were 1.08 mg/kg (0.2 x 5.4) for washed potatoes and 0.16 mg/kg (0.03 x 5.4) for washed peeled potatoes. These STMR-P values replace the previous recommendations. Furthermore, an STMR-P value for wet potato peel of 30 mg/kg (5.5 x 5.4) is recommended for estimating animal dietary intake.

***Residues in animal and poultry commodities***

The Meeting estimated the dietary burden of thiabendazole residues in farm animals on the basis of the diets listed in Appendix IX of the *FAO Manual* (FAO, 1997). Calculation from MRLs (or HR values) provides concentrations in feed suitable for estimating MRLs for animal commodities, while calculation from STMR values for feed is suitable for estimating STMR values for animal commodities.

| Commodity | HR, mg/kg | Group | % dry matter | HR/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |
| Wet apple pomace | 1.8 (0.92 x 2.0) | AB | 40 | 4.5 | - | - | - | - | - | - |
| Dry citrus pulp | 12.5 (5.7 x 2.2) | AB | 91 | 13.7 | 20 | 20 | - | 2.74 | 2.74 | - |
| Wet potato peel | 60 (5.5 x 11) | SM | 15 | 400 | 75 | 40 | - | 300 | 160 | - |
| Total | | | | | | | | 303 | 163 | - |

| Commodity | STMR, mg/kg | Group | % dry matter | STMR/ dry matter | % of diet | | | Concentration of residue, mg/kg | | |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | Beef cattle | Dairy cows | Poultry | Beef cattle | Dairy cows | Poultry |
| Wet apple pomace | 1.6 (0.92 x 1.7) | AB | 40 | 4.0 | - | - | - | - | - | - |
| Dry citrus pulp | 8.0 (5.7 x 1.4) | AB | 91 | 8.8 | 20 | 20 | - | 1.76 | 1.76 | - |
| Wet potato peel | 30 (5.5 x 5.4) | SM? | 15 | 200 | 75 | 40 | - | 150 | 80 | - |
| Total | | | | | | | | 152 | 82 | - |

The dietary burdens of thiabendazole used for estimating MRLs and STMR values (residue concentrations in animal feeds expressed as dry weight) are 303 and 152 ppm for beef cattle and 163 and 82 ppm for dairy cows. Poultry are not exposed to thiabendazole residues. In the 28-day study evaluated by the 1997 JMPR in which dairy cows were fed diets containing 25, 75, or 250 ppm thiabendazole, the mean concentrations of thiabendazole and 5-hydroxythiabendazole residues on day 29 were 0.02 mg/kg in fat, 0.60 mg/kg in kidney, 0.21 mg/kg in liver, and 0.02 mg/kg in muscle at

250 ppm; and 0.03 mg/kg in fat, 0.27 mg/kg in kidney, 0.13 mg/kg in liver, and 0.02 mg/kg in muscle at 75 ppm. The highest concentration of thiabendazole plus 5-hydroxythiabendazole in milk reached on day 28 was 0.15 mg/kg at 250 ppm and 0.12 mg/kg at 75 ppm. The results with the lower dose indicated that a plateau had already been reached by day 28.

The residue definition for dietary intake of animal products is the sum of thiabendazole, 5-hydroxy-thiabendazole, and its sulfate conjugate. The sulfate conjugate is included since it is the main residue in milk. In the analytical method used for milk (Annex 6, reference 80, p. 791), the sulfate conjugate is presumed to be hydrolysed to 5-hydroxythiabendazole. The Meeting therefore estimated STMR values for cattle kidney, liver, meat, and milk on the basis of the feeding study evaluated by the 1997 Meeting.

The Meeting thus recommended withdrawal of the existing proposed MRLs and STMR values for cattle milk, meat, and edible offal. It estimated maximum residue levels of 1 mg/kg in cattle kidney, 0.3 mg/kg in cattle liver, 0.1 mg/kg in cattle meat, and 0.2 mg/kg in cows' milk, and STMR values of 0.5 mg/kg in cattle kidney, 0.2 mg/kg in cattle liver, 0.02 mg/kg in cattle meat, and 0.12 mg/kg in cows' milk.

At its thirty-second session, the CCPR requested (ALINORM 01/24, paragraph 104) the Joint Meeting to review the MRL for edible offal of cattle. It noted that the residue definition includes the sum of thiabendazole and 5-hydroxythiabendazole and considered that the MRL of 0.1 mg/kg might be too low.

The 1997 JMPR reviewed a study of transfer in ruminants within the periodic review of thiabendazole and recommended MRLs for cattle meat, milk, and offal. A MRL of 0.1 mg/kg was recommended for offal. At the recommended feeding level at that time of 25 ppm, the maximum concentrations of thiabendazole residues were 0.020 mg/kg in kidney and 0.049 mg/kg in liver. The LOQ of the analytical methods was reported to be 0.1 mg/kg for each analyte. Thus, the appropriate MRL would necessarily be 0.1 mg/kg plus 0.1 mg/kg or 0.2 mg/kg.

The manufacturer has now indicated that the LOQ of two methods for thiabendazole, benzimidazole, and 5-hydroxythiabendazole has been validated at 0.03 mg/kg. The combined LOQ would be 0.06 mg/kg, that is, 0.03 plus 0.03 mg/kg, on the basis of the Codex residue definition. The average recoveries of thiabendazole, benzimidazole, and 5-hydroxythiabendazole from cattle liver fortified at 0.03 mg/kg with each analyte were 90, 69, and 94%, respectively, and those from cattle kidney similarly fortified were 75, 105, and 65%, respectively ($n$ = 6-8). The standard deviation for liver was excessive (17-23%).

The Meeting concluded that the proposed MRL of 0.1 mg/kg for the combined residue of thiabendazole and 5-hydroxythiabendazole, expressed as thiabendazole, in offal is appropriate, given the recent validations of the analytical method at 0.03 mg/kg for each analyte. However, in the light of the new data evaluated by the present Meeting (see above), the proposal is withdrawn and replaced by a recommendation for a maximum residue level of 1 mg/kg in cattle kidney and 0.3 mg/kg in cattle liver.

## RECOMMENDATIONS

The Meeting estimated the maximum residue levels and STMRs shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and estimation of dietary intake for plant commodities: thiabendazole. For compliance with MRLs for animal commodities: sum of thiabendazole and 5-hydroxythiabendazole. For estimation of dietary intake for animal commodities: sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate.

| Commodity | | MRL, mg/kg | | STMR, mg/kg | HR, mg/kg |
|---|---|---|---|---|---|
| CCN | Name | New | Previous | | |
| FP 0226 | Apple | W[1] | W[2] | | |
| JF 0226 | Apple juice | | | 0.8 | |
| AB 0226 | Apple pomace, dry | | | 7.1 | |
| | Apple pomace, wet | | | 1.6 | |
| | Apple purée | | | 0.7 | |
| FI 0326 | Avocado | 15 Po | - | 0.9 | 1.8 |
| MM 0812 | Cattle meat | 0.1 | 0.05 | 0.02 | 0.02 |
| ML 0812 | Cattle milk | 0.2 | 0.05 | 0.12 | 0.15 |
| MO 0812 | Cattle, Edible offal of | W | 0.1 | | |
| M0 1280 | Cattle, kidney | 1 | | 0.5 | 0.6 |
| MO 1281 | Cattle, liver | 0.3 | | 0.2 | 0.21 |
| FC 0001 | Citrus fruits | 3 Po | W[2] | 0.01 | 0.09 |
| FI 0345 | Mango | 5 Po | - | 2.85 | 4.6 |
| VC 0046 | Melons, except Watermelon | 1 | - | 0.43 | 0.82 |
| JF 0004 | Orange juice | | | 0.11 | |
| | Orange pomace, dry | | | 8 | |
| | Orange pomace, wet | | | 1.7 | |
| FI 0350 | Papaya | 10 Po | - | 3.8 | 5.1 |
| FP 0230 | Pear | W[1] | W[2] | | |
| FP 0009 | Pome fruits | 3 Po | - | 1.7 | 2.0 |
| VR 0589 | Potato | 15 Po | 15 | 5.4 | |
| | Potato peel, wet | | | 30 | |
| | Potato, washed | | | 1.08 | |
| | Potato, washed and peeled | | | 0.16 | 11 |
| FB 0275 | Strawberry | 5 | W | 1.6 | 2.7 |

[1] Now included in recommendation for Pome fruits
[2] Recommendation of the 1997 JMPR

## Dietary risk assessment

### *Chronic intake*

STMR values have been estimated for 13 commodities, and the 1997 JMPR estimated STMR values for five additional commodities: bananas, mushrooms, poultry meat and eggs, and witloof chicory (sprouts).

The international estimated daily intakes from the five GEMS/Food regional diets, based on estimated STMR values, represented 1-9% of the ADI of 0-0.1 mg/kg bw. The Meeting concluded that long-term intake of residues of thiabendazole resulting from uses that have been considered by the JMPR is unlikely to present a public health concern.

### *Short-term intake*

The toxicological profile of thiabendazole includes effects of concern that would indicate a need for an acute RfD. The Meeting recommended that this task be referred to JECFA, which conducted the most recent toxicological assessment of this chemical.

The IESTI for thiabendazole was calculated as described in Section 3 for the commodities for which maximum residue levels and STMR values were estimated and for which data on consumption were available. The results are shown in Annex 3. The IESTI varied from 0 to 0.287 mg/kg bw for the

general population and from 0 to 0.939 mg/kg bw for children. As no acute RfD has been established yet, the risk assessment for thiabendazole was not finalized.

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# ANNEX 1

## ACUTE DIETARY INTAKES, ACUTE REFERENCE DOSES, RECOMMENDED MAXIMUM RESIDUE LIMITS, AND SUPERVISED TRIALS MEDIAN RESIDUE VALUES RECORDED BY THE 2000 MEETING

The table below lists maximum ADIs, acute RfDs, recommended MRLs, and STMR and HR levels. The application of the HR levels is explained in the report of the 1999 Meeting (Annex 6, reference 86, Section 2.4). The PTDI and ERLs for DDT are given in a separate table. The rationale of the PTDI is explained in the report of the 1994 Meeting (Annex 6, reference 71, Section 2.3). Pesticides for which the estimated dietary intake might, on the basis of the available information, exceed their ADIs are marked with footnotes as explained in detail in the report of the 1999 Meeting (Section 2.2). Footnotes are also applied to specific commodities in which the acute RfD of a pesticide might be exceeded if the food commodity when consumed. These distinctions apply only to new compounds and to those re-evaluated within the CCPR periodic review programme.

STMR levels were introduced in 1996 in response to recommendations of a Joint FAO/WHO Consultation on Guidelines for Predicting the Dietary Intake of Pesticide Residues held in York, United Kingdom, in 1995. The report of the 1996 Meeting (Annex 6, reference 77) explains the reasons for their introduction and gives details of the procedures used in their calculation.

In general, the MRLs recommended for compounds that have been reviewed previously are additional to, or amend, those recorded in the reports of earlier Meetings. If a recommended MRL is an amendment, the previous value is also recorded. All recommendations for compounds re-evaluated within the CCPR periodic review programme are listed, however (even if identical to existing Codex or draft MRLs), because such re-evaluations replace the original evaluation rather than supplement it.

Temporary ADIs are indicated by the letter T, and the year in which re-evaluation is scheduled is given in parentheses below the ADI. All recommended MRLs for compounds with temporary ADIs are necessarily temporary, but other recommendations are designated as temporary (TMRLs) until the required information has been provided and evaluated, irrespective of the status of the ADI.

The table includes the Codex reference number of each compound and the Codex classification number (CCN) of each commodity, to facilitate reference to the Codex maximum limits for Pesticide Residues (*Codex Alimentarius*, Vol. 2B) and other documents and working documents of the Codex Alimentarius Commission.

The abbreviations and symbols used in the table and not defined elsewhere are as follows:

| | |
|---|---|
| * | at or about the limit of quantification |
| N | new compound |
| Po | accommodates post-harvest treatment of the commodity |
| PoP | accommodates post-harvest treatment of the primary food commodity (classes D and E in the Codex classification) |
| R | reviewed within CCPR periodic review programme |
| T | temporary |
| V | accommodates veterinary uses |
| W | The previous recommendation is withdrawn, or withdrawal of the recommended MRL or existing Codex or draft MRL is recommended. |

| Pesticide | ADI (mg/kg bw) | Commodity CCN | Commodity Name | Recommended MRL (mg/kg) New | Recommended MRL (mg/kg) Previous | STMR (mg/kg) | HR (mg/kg) |
|---|---|---|---|---|---|---|---|
| Abamectin (177) | 0.002 | MO 1281 | Cattle liver | 0.1 | 0.1 | | |
| | | MF 0812 | Cattle fat | 0.1 | 0.1 | | |
| | | MO 1280 | Cattle kidney | 0.05 | 0.05 | | |
| | | MM 0812 | Cattle meat | 0.01* | 0.01* | | |
| | | ML 0812 | Cattle milk | 0.005 | 0.005 | | |
| | | MO 0814 | Goat edible offal | 0.1 | 0.1 | | |
| | | MM 0814 | Goat meat | 0.01* | 0.01* | | |
| | | ML 0814 | Goat milk | 0.005 | 0.005 | | |
| | | *Residue* | | | | | |
| | | For compliance with MRL for plant commodities: sum of avermectin $B_{1a}$, avermectin $B_{1b}$, 8,9-*Z*-avermectin $B_{1a}$, and 8,9-*Z*-avermectin $B_{1b}$ | | | | | |
| | | For compliance with MRL for animal commodities: sum of avermectin $B_{1a}$ and 8,9-*Z*-avermectin $B_{1a}$ | | | | | |
| | | For estimation of dietary intake: sum of avermectin $B_{1a}$, avermectin $B_{1b}$, 8,9-*Z*-avermectin $B_{1a}$, and 8,9-Z-avermectin $B_{1b}$ | | | | | |
| | | *Note*: The numerical values for the recommended MRLs remain unchanged, but the residue definition is revised. | | | | | |
| Captan (007) (R) | 0.1 | TN 0660 | Almonds | 0.3 | – | 0.05 | 0.2 |
| | | FP 0226 | Apple | W | 20 | – | – |
| | | AB 0226 | Apple pomace, dry | W | 2 | 4.95 | 7.8 |
| | | FB 0020 | Blueberries | 20 | 20 | 6.9 | 18 |
| | | FS 0013 | Cherries | 25 | 40 | 11 | 21 |
| | | VC 0424 | Cucumber | 3 | – | 0.22 | 1.5 |
| | | DF 0269 | Dried grapes (currants, raisins and sultanas) | 50 | 50 | 5.6 | 33 |
| | | FB 0269 | Grapes | 25 | 25 | 3.7 | 22 |
| | | VC 0046 | Melons, except watermelon | 10 | – | 0.04 | 0.13 |
| | | FS 0245 | Nectarine | 3 | 5 | 1.0 | 1.8 |
| | | FS 0247 | Peach | 20 | 15 | 4.7 | 16 |
| | | FP 0230 | Pear | W | 10 | – | – |
| | | FS 0014 | Plums (including prunes) | 10 | 5 | 1.4 | 7.9 |
| | | FP0009 | Pome fruits | 15 Po | – | 5.3 | 11 |
| | | VR 0589 | Potato | 0.05 | – | 0.05 | 0.05 |
| | | DF 0014 | Prunes | | | 0.15 | 0.84 |
| | | FB 0272 | Raspberries, red, black | 20 | – | 8.3 | 18 |
| | | FB 0275 | Strawberry | 15 | 30 | 4.15 | 12 |
| | | VO 0448 | Tomato | 5 | 2 | 0.64 | 2.3 |
| | | VJ 0448 | Tomato juice | | | 0.06 | 0.23 |
| | | | Tomato puree | | | 0.06 | 0.23 |
| | | *Residue* | | | | | |
| | | For compliance with MRL and estimation of dietary intake: captan | | | | | |
| | | Acute RfD: Unnecessary | | | | | |
| | | Periodic review for residues only | | | | | |
| Chlormequat (015) (R) | 0.05 | GC 0640 | Barley | 2 | 0.5 | 0.15 | 1.8 |
| | | | Barley beer | | | 0.0023 | |
| | | | Barley malt | | | 0.1 | |
| | | | Barley pearl | | | 0.009 | |
| | | AS 0640 | Barley straw and fodder, dry | W | 20 | | |
| | | PE 0112 | Eggs | 0.1 | – | 0.04 | 0.064 |
| | | MM 0814 | Goat meat | 0.2 | – | 0.04 | 0.11 |

| Pesticide | ADI (mg/kg bw) | Commodity CCN | Commodity Name | Recommended MRL (mg/kg) New | Recommended MRL (mg/kg) Previous | STMR (mg/kg) | HR (mg/kg) |
|---|---|---|---|---|---|---|---|
| Chlormequat (contd) | | MO 0098 | Kidney of cattle, goats, pigs, and sheep | 0.5 | – | 0.084 | 0.35 |
| | | MO 0099 | Liver of cattle, goats, pigs, and sheep | 0.1 | – | 0.042 | 0.88 |
| | | AS 0645 | Maize fodder | 7[1] | – | 2.3[1] | |
| | | AF 0645 | Maize forage | 15[1] | – | 6.5[1] | |
| | | MM 0097 | Meat of cattle, pigs, and sheep | 0.2 | – | 0.04 | 0.11 |
| | | ML 0107 | Milk of cattle, goats, and sheep | 0.5 | – | 0.018 | 0.35 |
| | | GC 0647 | Oats | 10 | 10 | 1.2 | 7.1 |
| | | | Oat flakes | | | 0.25 | |
| | | AF 0647 | Oat forage, green | 100[1] | 20 | 12.7[1] | |
| | | AS 0647 | Oat straw and fodder, dry | W | 20 | | |
| | | FP 0230 | Pear[2] | 10 | 10 | 4.2 | 6.3 |
| | | PM 0110 | Poultry meat | 0.04* | – | 0 | 0 |
| | | PO 0111 | Poultry, edible offal of | 0.1 | – | 0.0096 | 0.053 |
| | | SO 0495 | Rape-seed | | | 2.05 | |
| | | OC 0495 | Rape-seed oil, crude | | | 0.037 | |
| | | GC 0650 | Rye | 3 | 3 | 0.26 | 2 |
| | | CM 0650 | Rye bran, unprocessed | 10 | 10 | 0.83 | |
| | | CF 1250 | Rye flour | 3 | – | 0.26 | |
| | | AF 0650 | Rye forage (green) | 100[1] | 20 | 12.7[1] | |
| | | AS 0650 | Rye straw and fodder, dry | W | 20 | | |
| | | CF 1251 | Rye wholemeal | 4 | 3 | 0.34 | |
| | | | Rye wholemeal bread | | | 0.25 | |
| | | AS 0081 | Straw and fodder (dry) of cereal grains | 30[1] | – | 4.2[1] | |
| | GC 0653 | Triticale | | 3 | – | 0.26 | 2 |
| | GC 0654 | Wheat | | 3 | 2 | 0.26 | 2 |
| | CM 0654 | Wheat bran, unprocessed | | 10 | 5 | 0.94 | |
| | | CF 1211 | Wheat flour | 2 | 0.5 | 0.11 | |
| | | AS 0654 | Wheat straw and fodder, dry | W | 20 | | |
| | | CF 1212 | Wheat wholemeal | 5 | 2 | 0.31 | |
| | | | Wheat wholemeal bread | | | 0.18 | |
| | | *Residue* | | | | | |
| | | For compliance with MRL and for estimation of dietary intake: Chlormequat cation (usually used as the chloride) | | | | | |
| | | [1] Expressed on dry weight basis | | | | | |
| | | [2] The information provided to the JMPR precludes an estimate that the dietary intake would be below the acute RfD. | | | | | |
| | | Acute RfD: 0.05 mg/kg bw | | | | | |
| Chlorpropham (N) | 0.03 | Acute RfD: 0.03 mg/kg bw | | | | | |
| Chlorpyrifos (017) (R) | 0.01 | AL 1020 | Alfalfa fodder | 5 dry wt. | – | 0.81 dry wt. | |
| | | AL 1021 | Alfalfa forage (green) | 20 dry wt. | – | 1.2 dry wt. | |
| | | TN 0660 | Almonds | 0.05 | – | 0.05 | 0.05 |
| | | | Almond, hulls | | – | 2.3 | 3.2 |
| | | FP 0226 | Apple | W[1] | 1 | | |
| | | JF 0226 | Apple juice | | | 0.027 | |
| | | AB 0226 | Apple pomace, dry | | – | 1.2 | 6.2 |
| | | | Apple pomace, wet | | | 0.34 | 1.9 |
| | | FI 0327 | Banana | 2 | – | 0.01 | 0.05 |
| | | VB 0400 | Broccoli | 2 | – | 0.02 | 1.4 |

| Pesticide | ADI (mg/kg bw) | Commodity CCN | Name | Recommended MRL (mg/kg) New | Previous | STMR (mg/kg) | HR (mg/kg) |
|---|---|---|---|---|---|---|---|
| | | VB 0041 | Cabbages, head | 1 | 0.05* | 0.15 | 0.94 |
| | | VR 0577 | Carrot | 0.1 | 0.5 | 0.025 | 0.05 |
| Chlorpyrifos (contd) | | MO 1280 | Cattle, kidney | 0.01 | | 0.01 | 0.01 |
| | | MO 1281 | Cattle, liver | 0.01 | | 0.01 | 0.01 |
| | | MM 0812 | Cattle meat | 1 (fat) | 2 (fat)V | 0.02 | 0.02 |
| | | VB 0404 | Cauliflower | 0.05 | 0.05* | 0.01 | 0.02 |
| | | VS 0624 | Celery | W | 0.05* | | |
| | | VL 0467 | Chinese cabbage (type pe-tsai) | 1 | 1 | 0.18 | 0.60 |
| | | FC 0001 | Citrus fruits | 2 | 2 | 0.08 | 0.4 |
| | | JF 0001 | Citrus juice | | – | 0.007 | |
| | | | Citrus oil | | – | 2.6 | 13 |
| | | AB 0001 | Citrus pulp, dry | | – | 0.72 | 3.6 |
| | | SB 0716 | Coffee | 0.05 | | 0.010 | 0.014 |
| | | VP 0526 | Common bean (pods and/or immature seeds) | 0.01 | 0.2 | 0.01 | 0.01 |
| | | SO 0691 | Cottonseed | W | 0.05* | | |
| | | OC 0691 | Cottonseed oil, crude | W | 0.05* | | |
| | | DF 0269 | Dried grapes (currants, raisins, and sultanas) | 0.1 | 2 | 0.017 | 0.07 |
| | | VO 0440 | Eggplant | W | 0.2 | | |
| | | PE 0112 | Eggs | 0.01* | 0.05* | 0.001 | 0.01 |
| | | FB 0269 | Grapes | 0.5 | 1 | 0.085 | 0.32 |
| | | JF 0269 | Grape juice | | – | 0.005 | |
| | | | Grapes, wine | | – | 0.007 | |
| | | VL 0480 | KaleW | 1 | | | |
| | | FI 0341 | Kiwifruit | W | 2 | | |
| | | VL 0482 | Lettuce, head | W | 0.1 | | |
| | | GC 0645 | Maize | 0.05 | – | 0.015 | |
| | | AF 0645 | Maize forage | 20 dry wt. | – | 8.2 dry wt. | |
| | | AS 0645 | Maize fodder | 10 dry wt. | – | 2.8 dry wt. | |
| | | | Maize, milled by-products | | – | 0.02 | 0.09 |
| | | OR 0645 | Maize oil, edible | 0.2 | – | 0.03 | |
| | | CF 0645 | Maize meal | | – | 0.01 | |
| | | ML 0106 | Milks | W | 0.01* | | |
| | | ML 0107 | Milk of cattle, goats, and sheep | 0.02 | | 0.005 | |
| | | VO 0450 | Mushrooms | W | 0.05* | | |
| | | VA 0385 | Onion, bulb | 0.2 | 0.05* | 0.04 | 0.08 |
| | | VP 0063 | Peas (pods and succulent immature seeds) | 0.01 | | 0.01 | 0.01 |
| | | AL 0528 | Pea vines, green | 1 dry wt. | | 0.10 dry wt. | |
| | | FS 0247 | Peach | 0.5 | – | 0.042 | 0.33 |
| | | FP 0230 | PearW[1] | 0.5 | | | |
| | | TN 0672 | Pecan | 0.05* | – | 0.05 | 0.05 |
| | | VO 0051 | Peppers | W | 0.5 | | |
| | | VO 0445 | Peppers, sweet | 2 | | 0.38 | 1.4 |
| | | FS 0014 | Plums (including prunes) | 0.5 | – | 0.04 | 0.20 |
| | | MO 0818 | Pigs, edible offal of | 0.01* | | 0.00 | 0.01 |
| | | MM 0818 | Pig meat | 0.02 (fat) | | 0.001 | 0.01 |
| | | FP 0009 | Pome fruits | 1 | – | 0.17 | 0.94 |
| | | VR 0589 | Potato | W | 0.05* | | |
| | | PM 0110 | Poultry meat | 0.01 (fat) | 0.1 (fat) | 0.001 | 0.01 |
| | | PO 0111 | Poultry, edible offal of | 0.01* | | 0.00 | 0.01 |
| | | FB 0272 | Raspberries, red, black | W | 0.2 | – | – |
| | | GC 0649 | RiceW | 0.1 | | | |

| Pesticide | ADI (mg/kg bw) | Commodity CCN | Name | Recommended MRL (mg/kg) New | Previous | STMR (mg/kg) | HR (mg/kg) |
|---|---|---|---|---|---|---|---|
| | | MO 0822 | Sheep, edible offal of | 0.01 | | 0.01 | 0.01 |
| | | MM 0822 | Sheep meat | 1 (fat) | 0.2 (fat)V | 0.02 | 0.02 |
| Chlorpyrifos (contd) | | GC 0651 | Sorghum | 0.5 | – | 0.04 | |
| | | AS 0651 | Sorghum straw and fodder, dry | 2 dry wt. | – | 0.29 dry wt. | |
| | | | Sorghum, flour | | – | 0.008 | |
| | | FB 0275 | Strawberry | 0.3 | – | 0.09 | 0.15 |
| | | VR 0596 | Sugar beet | 0.05 | 0.05* | 0.015 | 0.03 |
| | | AV 0596 | Sugar beet leaves or tops | 40 dry wt. | – | 3.0 dry wt. | |
| | | VO 0447 | Sweet corn | 0.01* | | 0.01 | 0.01 |
| | | VO 0448 | Tomato | 0.5 | 0.5 | 0.13 | 0.33 |
| | | JF 0448 | Tomato, juice | | – | 0.026 | |
| | | | Tomato, paste | | – | 0.026 | |
| | | PM 0848 | Turkey meat | W | 0.2 fat (V) | | |
| | | TN 0678 | Walnuts | 0.05* | – | 0.05 | 0.05 |
| | | GC 0654 | Wheat | 0.5 | – | 0.015 | |
| | | AS 0654 | Wheat straw and fodder, dry | 5 dry wt. | – | 0.54 dry wt. | |
| | | | Wheat, milled by–products | | – | 0.03 | 0.75 |
| | | CF 1211 | Wheat flour | 0.1 | – | 0.002 | |
| | | CM 0654 | Wheat bran, unprocessed | | | 0.03 | |
| | | | Wheat shorts | | | 0.03 | |

*Residue*
For compliance with MRL and estimation of dietary intake:chlorpyrifos
The residue is fat-soluble.
Acute RfD: 0.1 mg/kg bw
Periodic review for residues only
[1]Replaced by recommendation for pome fruit

| | | |
|---|---|---|
| Deltamethrin (135) (R) | 0. 01 | Acute RfD: 0.05 mg/kg bw<br>ADI unchanged<br>Periodic review for toxicology only |
| Dinocap (087 )[1] | 0.008 | Acute RfD:0.008 mg/kg bw (for women of childbearing age);<br>0.03 mg/kg bw (for children and for the general population other than women of childbearing age)<br>[1] The information provided to the JMPR precludes an estimate that the acute dietary intake from the consumption of grape by women of childbearing age would be below the acute RfD |
| Dodine (084) (R) | 0.1 | Acute RfD: 0.2 mg/kg bw<br>Previous ADI: 0.01 mg/kg bw<br>Periodic review for toxicology only |
| Fenitrothion (037) (R) | 0.005 | Acute RfD: 0.04 mg/kg<br>ADI unchanged<br>Periodic review for toxicology only |
| Fenthion (039 )[1] | 0.007 | *Residue*<br>For compliance with MRL and estimation of dietary intake: sum of fenthion, its oxygen analogue and their sulfoxides and sulfones, expressed as fenthion<br>The residue is fat-soluble.<br>Acute RfD: 0.01 mg/kg bw<br>[1] The 1995 recommendation to withdraw the MRL for meat (of mammals other than marine mammals) and milks is confirmed. |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Fipronil | 0.0002 | Acute RfD: 0.003 mg/kg bw (for fipronil and fipronil-desulfinyl, alone or in combination)<br>ADI: group ADI for fipronil and fipronil-desulfinyl | | | | | |
| Pesticide | ADI (mg/kg bw) | Commodity | | Recommended MRL (mg/kg) | | STMR (mg/kg) | HR (mg/kg) |
| | | CCN | Name | New | Previous | | |
| Imazalil (110) (R) | 0.03 | Acute RfD: Unnecessary<br>ADI unchanged<br>Periodic review for toxicology only | | | | | |
| Malathion (049) | 0.3 | CF 1211 | Wheat flour | 0.2 | 2 PoP | 0.0092 | |
| | | CF 1212 | Wheat wholemeal | W | 2 PoP | | |
| | | CM 0654 | Wheat bran, unprocessed | W | 20 PoP | | |
| | | *Residue*<br>For compliance with MRL and estimation of dietary intake): malathion<br>Acute RfD: may be necessary but not yet established | | | | | |
| Mevinphos (053 ) | 0.0008 | VB 0400 | Broccoli | W | W | | |
| | | FC 0001 | Citrus fruits | W | W | | |
| | | VC 0424 | Cucumber | W | W | | |
| | | FB 0269 | Grapes | W | W | | |
| | | VC 0046 | Melons, except watermelon | W | W | | |
| | | VP 0063 | Peas (pods and succulent immature seeds) | W | W | | |
| | | VL 0502 | Spinach | W | W | | |
| | | FB 0275 | Strawberry | W | W | | |
| | | VO 0506 | Tomato | W | W | | |
| | | *Residue*<br>For compliance with MRL and for estimation of dietary intake: sum of (*E*)- and (*Z*)-mevinphos<br>Acute RfD: 0.003 mg/kg bw | | | | | |
| Parathion (58) (R) | 0.004 | FP 0226 | Apple[1] | 0.2 | 0.05* | 0.025 | 0.16 |
| | | JF 0226 | Apple juice | | | 0.0018 | |
| | | AB 0226 | Apple pomace, dry | | | 0.078 | 0.62 |
| | | FS 0240 | Apricot | W | 1 | | |
| | | GC 0640 | Barley[2] | 7 | | 1.95 | 5.1 |
| | | AS 0640 | Barley straw and fodder, dry | 30 | | 7.75 | |
| | | SO 0691 | Cottonseed | 3 | 1 | 0.35 | 2.1 |
| | | VA 0384 | Leek | W | 0.05 | | |
| | | FC 0204 | Lemon | W | 0.5 | | |
| | | GC 0645 | Maize | 0.1 | 0.1 | 0.05 | 0.09 |
| | | CF1255 | Maize flour | 0.1 | | 0.034 | |
| | | AS 0645 | Maize fodder | 30 | | 2.13 | |
| | | AF 0645 | Maize forage | 10 | | 2.28 | |
| | | | Maize grits | | | 0.05 | |
| | | CF 0645 | Maize meal | | | 0.037 | 0.074 |
| | | | Maize starch | | | 0.014 | |
| | | OC 0645 | Maize oil, crude | 0.3 | | 0.12 | |
| | | OR 0645 | Maize oil, edible | 0.3 | | 0.12 | |
| | | FC 0206 | Mandarin | W | 0.5 | | |
| | | OC 0305 | Olive oil, virgin | W | 2 | | |
| | | FT 0305 | Olives | W | 0.5 | | |
| | | FC 0004 | Oranges, sweet, sour | W | 0.5 | | |
| | | FS 0247 | Peach | W | 1 | | |
| | | VR 0589 | Potato | W | 0.05* | | |
| | | GC 0651 | Sorghum | 5 | 5 | 1.06 | 4.2 |

| Pesticide | ADI (mg/kg bw) | Commodity CCN | Name | Recommended MRL (mg/kg) New | Previous | STMR (mg/kg) | HR (mg/kg) |
|---|---|---|---|---|---|---|---|
| | | | Sorghum bran | | | 2.0 | |
| | | | Sorghum flour | | | 0.42 | |
| Parathion (contd) | | | Sorghum grits | | | 0.49 | |
| | | | Sorghum starch | | | 0.016 | |
| | | AF 0651 | Sorghum forage, green | 10 | | 3.1 | |
| | | AS 0651 | Sorghum straw and fodder, dry | 15 | | 2.8 | |
| | | VD 0541 | Soya bean, dry | 0.05* | 0.05* | 0.05 | 0.05 |
| | | AL 0541 | Soya bean fodder | 2 | | 0.63 | |
| | | SO 0702 | Sunflower seed | 0.05* | 0.05* | 0.05 | 0.05 |
| | | | Sunflower seed meal | | | 0.0025 | 0.0025 |
| | | OR 0702 | Sunflower seed oil, edible | 0.05* | | 0.021 | |
| | | VO 0447 | Sweet corn | 0.05* | | 0.05 | 0.05 |
| | | GC 0654 | Wheat | 1 | | 0.125 | 0.96 |
| | | CM 0654 | Wheat bran, unprocessed | | | 0.58 | |
| | | CF 1211 | Wheat flour | | | 0.044 | |
| | | | Wheat shorts (animal feed) | | | 0.10 | 0.80 |
| | | AS 0654 | Wheat straw and fodder, dry | 20 | | 3.7 | |
| | | ***Residue*** | | | | | |
| | | For compliance with MRLs: parathion | | | | | |
| | | For estimation of dietary intake: Sum of parathion and paraoxon expressed as parathion. | | | | | |
| | | Acute RfD: 0.01 mg/kg bw | | | | | |
| | | Periodic review for residues only | | | | | |
| | | [1]The information provided to the JMPR precludes an estimate that the acute dietary intake of children would be below the acute reference dose | | | | | |
| | | [2] The information provided to the JMPR precludes an estimate that the acute dietary intake of the general population would be below the acute reference dose | | | | | |
| Parathion-methyl (59) (R) | 0.003 | AL 1020 | Alfalfa fodder | 70 | | 2.3 | |
| | | AL 1021 | Alfalfa forage (green) | 70 | | 3.7 | |
| | | FP 0226 | Apple | 0.2 | | 0.06 | 0.18 |
| | | JF 0226 | Apple juice | | | 0.015 | |
| | | AB 0226 | Apple pomace, dry | | | 0.31 | 1.04 |
| | | VS 0620 | Artichoke, Globe | W | 2 | | |
| | | AL 1030 | Bean forage, green, fresh weight | 1 | 1 | 0.11 | |
| | | VD 0071 | Bean (dry) | 0.05* | 0.05* | 0.05 | 0.05 |
| | | VB 0400 | Broccoli | W | 0.2 | | |
| | | VB 0041 | Cabbages, head | 0.05 | 0.2 | 0.05 | 0.26 |
| | | VR 0577 | Carrot | W | 1 | | |
| | | VS 0624 | Celery | W | 5 | | |
| | | FS 0013 | Cherries | W | 0.01* | | |
| | | AL 1023 | Clover | W | 10 | | |
| | | VP 0526 | Common bean (pods and/or immature seeds) | W | 0.05* | | |
| | | SO 0691 | Cottonseed | 25 | | 3.5 | 22 |
| | | | Cottonseed hulls | | | 1.54 | 9.7 |
| | | | Cottonseed meal | | | 0.28 | 2.00 |
| | | OC 0691 | Cottonseed oil, crude | 10 | | 1.54 | |
| | | OR 0691 | Cottonseed oil, edible | 10 | | 1.16 | |
| | | DF 0269 | Dried grapes (currants, raisins and sultanas) | 1 | | 0.14 | 0.70 |
| | | VP 0528 | Garden pea (young pods) | W | 1 | | |
| | | FB 0268 | Gooseberry | W | 0.01* | | |
| | | JF 0269 | Grape juice | | | 0.0006 | |
| | | FB 0269 | Grapes | 0.5 | | 0.10 | 0.41 |

| Pesticide | ADI (mg/kg bw) | Commodity CCN | Name | Recommended MRL (mg/kg) New | Previous | STMR (mg/kg) | HR (mg/kg) |
|---|---|---|---|---|---|---|---|
| | | AS 0162 | Hay or fodder (dry) of grasses | 5 | 5 | 0.68 | |
| | | DH 1100 | Hops, dry | W | 1 | | |
| Parathion-methyl (contd) | | VL 0482 | Lettuce, head | W | 0.05* | | |
| | | VL 0483 | Lettuce, leaf | W | 0.5 | | |
| | | VP 0534 | Lima bean (young pods and/or immature beans) | W | 0.05* | | |
| | | GC 0645 | Maize | 0.1 | | 0.05 | 0.09 |
| | | CF 1255 | Maize flour | 0.05 | | 0.021 | |
| | | | Maize grits | | | 0.019 | |
| | | | Maize meal | | | 0.023 | 0.046 |
| | | OC 0645 | Maize oil, crude | 0.2 | | 0.067 | |
| | | OR 0645 | Maize oil, edible | 0.1 | | 0.051 | |
| | | | Maize starch | | | 0.0045 | |
| | | VL 0485 | Mustard greens | W | 0.5 | | |
| | | AL 0072 | Pea hay or pea fodder, dry | 70 | | 5.5 | |
| | | AL 0528 | Pea vines, green | 40 | | 0.74 | |
| | | FS 0247 | Peach | 0.3 | | 0.095 | 0.22 |
| | | | Peach juice | | | 0.031 | |
| | | VD 0072 | Peas, dry | 0.3 | 0.2 | 0.06 | 0.24 |
| | | FS 0014 | Plums (including prunes) | W | 0.01* | | |
| | | VR 0589 | Potato | 0.05* | 0.05* | 0 | 0 |
| | | SO 0495 | Rape seed | 0.05 | | 0.05 | 0.05 |
| | | | Rape seed meal | | | 0.011 | 0.011 |
| | | OC 0495 | Rape-seed oil, crude | 0.2 | | 0.12 | |
| | | OR 0495 | Rape-seed oil, edible | 0.2 | | 0.10 | |
| | | FB 0272 | Raspberries, red, black | W | 0.01* | | |
| | | GC 0649 | Rice | W | 3 | | |
| | | AS 0649 | Rice straw and fodder, dry | W | 10 | | |
| | | CM 0649 | Rice, husked | W | 1 | | |
| | | VL 0502 | Spinach | W | 0.5 | | |
| | | VR 0596 | Sugar beet | 0.05* | 0.05* | 0 | 0 |
| | | AV 0596 | Sugar beet leaves or tops (fresh weight) | 0.05* | 0.05* | 0.05 | |
| | | VL 0506 | Turnip greens | W | 2 | | |
| | | VR 0506 | Turnip, garden | W | 0.05* | | |
| | | GC 0654 | Wheat | 5 | 5 | 0.29 | 4.1 |
| | | CM 0654 | Wheat bran, unprocessed | 10 | 10 | 0.64 | |
| | | CF 1211 | Wheat flour | 2 | | 0.11 | |
| | | AS 0654 | Wheat straw and fodder, dry | 10 | 10 | 1.03 | |
| | | | Wine | | | 0.0015 | |

***Residue***
For compliance with MRL: parathion-methyl.
For estimation of dietary intake: Sum of parathion-methyl and paraoxon- methyl expressed as parathion-methyl
Acute RfD: 0.03 mg/kg bw
Periodic review for residues only

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| Pyrethrins (063) (R) | 0.04 | GC 0080 | Cereal grains | W | 3Po | | |
| | | FC 0001 | Citrus fruit | 0.05 | | 0.04 | 0.04 |
| | | JF 0001 | Citrus juice | | | 0.026 | |
| | | DM 0001 | Citrus molasses | | | 0.0276 | 0.0276 |
| | | AB 0001 | Citrus pulp, dry | | | 0.300 | 0.300 |
| | | | Citrus oil | | | 0.812 | 0.812 |
| | | MD 0180 | Dried fish | W | 3Po | | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | DF 0167 | Dried fruit | 0.2Po | 1Po | 0.05 | 0.11 |
| | | DV 0168 | Dried vegetables | W | 1Po | | |
| Pesticide | ADI (mg/kg bw) | Commodity | | Recommended MRL (mg/kg) | | STMR (mg/kg) | HR (mg/kg) |
| | | CCN | Name | New | Previous | | |
| Pyrethrins (contd) | | VC 0045 | Fruiting vegetables, curcubits | 0.05* | | 0.04 | 0.04 |
| | | SO 0088 | Oilseed | W | 1Po | | |
| | | AL 0072 | Pea hay, dry | 1 | | 0.295 | |
| | | AL 0528 | Pea vine, dry | 10 | | 2.15 | |
| | | SO 0697 | Peanut | 0.5 Po | | 0.05 | 0.23 |
| | | VO 0051 | Peppers | 0.05* | | 0.04 | 0.04 |
| | | VD 0070 | Pulses | 0.1 Po | | 0.05 | 0.05 |
| | | VR 0075 | Root and tuber vegetables | 0.05* | | 0 | 0.04 |
| | | VO 0448 | Tomato | 0.05* | | 0.04 | 0.04 |
| | | | Tomato pomace, dry | | | 0.808 | 0.808 |
| | | | Tomato pomace, wet | | | 0.352 | 0.352 |
| | | JF 0448 | Tomato, juice | | | 0.018 | |
| | | | Tomato, puree | | | 0.018 | |
| | | TN 0085 | Tree nuts | W | 1Po | | |
| | | *Residue* | | | | | |
| | | For compliance with MRL and estimation of dietary intake: total pyrethrins, calculated as the sum of pyrethrins 1 and 2, cinerins 1 and 2, and jasmolins 1 and 2, determined after calibration with the World Standard pyrethrum extract | | | | | |
| | | Acute RfD: 0.2 mg/kg bw | | | | | |
| | | Periodic review for residues only | | | | | |
| Pyriproxifen (200) | 0.1 | | Orange oil | | | 9.0 | |
| | | AB 0001 | Citrus pulp, dry | | | 0.76 | 6.3 |
| | | JF 0004 | Orange juice | | | 0.0036 | |
| | | *Residue* | | | | | |
| | | For compliance with MRL and for estimation of dietary intake): pyriproxyfen | | | | | |
| | | The residue is fat-soluble. | | | | | |
| | | Acute RfD: Unnecesssary | | | | | |
| Thiabendazole (065) | 0.1 | FP 0226 | Apple | W[1] | W | | |
| | | | Apple juice | | | 0.8 | |
| | | | Apple pomace, dry | | | 7.1 | |
| | | | Apple pomace, wet | | | 1.6 | |
| | | | Apple puree | | | 0.7 | |
| | | FI 0326 | Avocado | 15 Po | – | 0.9 | 1.8 |
| | | FC 0001 | Citrus fruits | 3 Po | W | 0.01 | 0.09 |
| | | ML 0812 | Cattle milk | 0.2 | 0.05 | 0.12 | 0.15 |
| | | MM 0812 | Cattle meat | 0.1 | 0.05 | 0.02 | 0.02 |
| | | MO 0812 | Cattle, edible offal of | W | 0.1 | | |
| | | MO 1280 | Cattle, kidney | 1 | | 0.5 | 0.6 |
| | | MO 1281 | Cattle, liver | 0.3 | | 0.2 | 0.21 |
| | | FI 0345 | Mango | 5 Po | – | 2.85 | 4.6 |
| | | VC 0046 | Melon, except watermelon | 1 | – | 0.43 | 0.82 |
| | | FI 0350 | Papaya | 10 Po | – | 3.8 | 5.1 |
| | | FP 0230 | Pear | W[1] | W | | |
| | | FP 0009 | Pome fruits | 3 Po | – | 1.7 | 2.0 |
| | | VR 0589 | Potato | 15 Po | 15 | 5.4 | 11 |
| | | | Potato, washed | | | 1.08 | |
| | | | Potato, washed and peeled | | | 0.16 | |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| | | | Potato peel, wet | | | 30 | |
| | | FB 0275 | Strawberry | 5 | W | 1.6 | 2.7 |
| | | | Orange pomace, dry | | | 8 | |
| | | | Orange pomace, wet | | | 1.7 | |
| Pesticide | ADI (mg/kg bw) | Commodity | | Recommended MRL (mg/kg) | | STMR (mg/kg) | HR (mg/kg) |
| | | CCN | Name | New | Previous | | |
| Thiabendazole (contd) | | JF 0004 | Orange juice | | 0.11 | | |
| | | *Residue* | | | | | |
| | | For compliance with MRL and estimation of dietary intake for plant commodities: thiabendazole | | | | | |
| | | For compliance with MRL for animal commodities: sum of thiabendazole and 5-hydroxythiabendazole | | | | | |
| | | For estimation of dietary intake for animal commodities: sum of thiabendazole, 5-hydroxythiabendazole and its sulfate conjugate | | | | | |
| | | ADI established at the forty-eighth meeting of the Joint FAO/WHO Expert Committee on Food Additives (WHO Technical Report Series No. 879, 1998). | | | | | |
| | | Acute RfD: May be necessary but not yet established | | | | | |
| | | [1]Replaced by recommendation for pome fruit | | | | | |
| Thiodicarb (154) (R) | 0.03 | Acute RfD: 0.04 mg/kg bw | | | | | |
| | | Periodic review for toxicology only | | | | | |
| | | ADI unchanged | | | | | |

## Provisional tolerable daily intake and extraneous residue limits for DDT

| PTDI (mg/kg bw) | Commodity | | ERL (mg/kg) | |
|---|---|---|---|---|
| | CCN | Name | New[a] | Previous |
| 0.01 | MM 0095 | Meat (from mammals other than marine mammals) | 1–5 (fat) | 5 (fat)[b] |
| | PM 0110 | Poultry meat | 0.1–0.3 (fat) | – |
| | Acute RfD: Unnecessary | | | |

[a] The Meeting estimated the total concentrations of DDT corresponding to violation rates of 0.1%, 0.2%, and 0.5% for mammalian and poultry meat according to the procedure described in Section 4.7 of this Report. The Meeting concluded that the selection of an acceptable violation rate and the weight given to the information provided by individual countries are risk management issues, not scientific ones. The CCPR should decide which violation rate is acceptable and whether each contributing country or each analysed sample should be given the same weight. Therefore, the table shows a range of values for ERLs**.** For dietary intake calculations, the worst case assumption, the highest ERLs of 5 mg/kg for mammalian meat and 0.3 mg/kg for poultry meat, were used. This resulted in intake well below the PTDI.

[b] Recommendation of the 1996 JMPR

## ANNEX 2

## INDEX OF REPORTS AND EVALUATIONS OF PESTICIDES BY THE JMPR

Numbers in parentheses after the names of pesticides are Codex classification numbers. The abbreviations used are:

T, evaluation of toxicology
R, evaluation of residue and analytical aspects
E, evaluation of effects on the environment

| | |
|---|---|
| Abamectin (177) | 1992 (T,R), 1994 (T,R), 1995 (T), 1997 (T,R), 2000 (R) |
| Acephate (095) | 1976 (T,R), 1979 (R), 1981 (R), 1982 (T), 1984 (T,R), 1987 (T), 1988 (T), 1990 (T,R), 1991 (corr. to 1990 R evaluation), 1994 (R), 1996 (R) |
| Acrylonitrile | 1965 (T,R) |
| Aldicarb (117) | 1979 (T,R), 1982 (T,R), 1985 (R), 1988 (R), 1990 (R), 1991 (corr. to 1990 evaluation), 1992 (T), 1993 (R), 1994 (R), 1996 (R) |
| Aldrin (001) | 1965 (T), 1966 (T,R), 1967 (R), 1974 (R), 1975 (R), 1977 (T), 1990 (R), 1992 (R) |
| Allethrin | 1965 (T,R) |
| Aminocarb (134) | 1978 (T,R), 1979 (T,R) |
| Aminomethylphosphonic acid (AMPA, 198) | 1997 (T,R) |
| Amitraz (122) | 1980 (T,R), 1983 (R), 1984 (T,R), 1985 (R), 1986 (R), 1989 (R), 1990 (T,R), 1991 (R & corr. to 1990 R evaluation), 1998 (T) |
| Amitrole (079) | 1974 (T,R), 1977 (T), 1993 (T,R), 1997 (T), 1998 (R) |
| Anilazine (163) | 1989 (T,R), 1992 (R) |
| Azinphos-ethyl (068) | 1973 (T,R), 1983 (R) |
| Azinphos-methyl (002) | 1965 (T), 1968 (T,R), 1972 (R), 1973 (T), 1974 (R), 1991 (T,R), 1992 (corr. to 1991 report), 1993 (R), 1995 (R) |
| Azocyclotin (129) | 1979 (R), 1981 (T), 1982 (R),1983 (R), 1985 (R), 1989 (T,R), 1991 (R), 1994 (T) |
| Benalaxyl (155) | 1986 (R), 1987 (T), 1988 (R), 1992 (R), 1993 (R) |
| Bendiocarb (137) | 1982 (T,R), 1984 (T,R), 1989 (R), 1990 (R) |
| Benomyl (069) | 1973 (T,R), 1975 (T,R), 1978 (T,R), 1983 (T,R), 1988 (R), 1990 (R), 1994 (R), 1995 (T,E), 1998 (R) |
| Bentazone (172) | 1991 (T,R), 1992 (corr. to 1991 report, Annex I), 1994 (R), 1995 (R), 1998 (T,R), 1999 (corr. to 1998 report ) |
| BHC (technical-grade) | 1965 (T), 1968 (T,R), 1973 (T,R) (see also Lindane) |
| Bifenthrin (178) | 1992 (T,R), 1995 (R), 1996 (R), 1997 (R) |
| Binapacryl (003) | 1969 (T,R), 1974 (R), 1982 (T), 1984 (R), 1985 (T,R) |
| Bioresmethrin (093) | 1975 (R), 1976 (T,R), 1991 (T,R) |
| Biphenyl | See Diphenyl |
| Bitertanol (144) | 1983 (T), 1984 (R), 1986 (R), 1987 (T), 1988 (R), 1989 (R), 1991 (R), 1998 (T), 1999 (R) |

| | |
|---|---|
| Bromide ion (047) | 1968 (R), 1969 (T,R), 1971 (R), 1979 (R), 1981 (R), 1983 (R), 1988 (T,R), 1989 (R), 1992 (R) |
| Bromomethane (052) | 1965 (T,R), 1966 (T,R), 1967 (R), 1968 (T,R), 1971 (R), 1979 (R), 1985 (R), 1992 (R) |
| Bromophos (004) | 1972 (T,R), 1975 (R), 1977 (T,R), 1982 (R), 1984 (R), 1985 (R) |
| Bromophos-ethyl (005) | 1972 (T,R), 1975 (T,R), 1977 (R) |
| Bromopropylate (070) | 1973 (T,R), 1993 (T,R) |
| Butocarboxim (139) | 1983 (R), 1984 (T), 1985 (T), 1986 (R) |
| Buprofezin (173) | 1991 (T,R), 1995 (R), 1996 (corr. to 1995 report.), 1999 (R) |
| *sec*-Butylamine (089) | 1975 (T,R), 1977 (R), 1978 (T,R), 1979 (R), 1980 (R), 1981 (T), 1984 (T,R: withdrawal of temporary ADI, but no evaluation) |
| Cadusafos (174) | 1991 (T,R), 1992 (R), 1992 (R) |
| Campheclor (071) | 1968 (T,R), 1973 (T,R) |
| Captafol (006) | 1969 (T,R), 1973 (T,R), 1974 (R), 1976 (R), 1977 (T,R), 1982 (T), 1985 (T,R), 1986 (corr. to 1985 report), 1990 (R), 1999 (acute Rf D) |
| Captan (007) | 1965 (T), 1969 (T,R), 1973 (T), 1974 (R), 1977 (T,R), 1978 (T,R), 1980 (R), 1982 (T), 1984 (T,R), 1986 (R), 1987 (R and corr. to 1986 R evaluation), 1990 (T,R), 1991 (corr. to 1990 R evaluation), 1994 (R), 1995 (T), 1997 (R), 2000 (R) |
| Carbaryl (008) | 1965 (T), 1966 (T,R), 1967 (T,R), 1968 (R), 1969 (T,R), 1970 (R), 1973 (T,R), 1975 (R), 1976 (R), 1977 (R), 1979 (R), 1984 (R), 1996 (T) |
| Carbendazim (072) | 1973 (T,R), 1976 (R), 1977 (T), 1978 (R), 1983 (T,R), 1985 (T,R), 1987 (R), 1988 (R), 1990 (R), 1994 (R), 1995 (T,E), 1998 (T,R) |
| Carbofuran (096) | 1976 (T,R), 1979 (T,R), 1980 (T), 1982 (T), 1991 (R), 1993 (R), 1996 (T), 1997 (R), 1999 (corr. to 1997 report) |
| Carbon disulfide (009) | 1965 (T,R), 1967 (R), 1968 (R), 1971 (R), 1985 (R) |
| Carbon tetrachloride (010) | 1965 (T,R), 1967 (R), 1968 (T,R), 1971 (R), 1979 (R), 1985 (R) |
| Carbophenothion (011) | 1972 (T,R), 1976 (T,R), 1977 (T,R), 1979 (T,R), 1980 (T,R), 1983 (R) |
| Carbosulfan (145) | 1984 (T,R), 1986 (T), 1991 (R), 1992 (corr. to 1991 report), 1993 (R), 1997 (R), 1999 (R) |
| Cartap (097) | 1976 (T,R), 1978 (T,R), 1995 (T,R) |
| Chinomethionat (080) | 1968 (T,R) (as oxythioquinox), 1974 (T,R), 1977 (T,R), 1981 (T,R), 1983 (R), 1984 (T,R), 1987 (T) |
| Chlorbenside | 1965 (T) |
| Chlordane (012) | 1965 (T), 1967 (T,R), 1969 (R), 1970 (T,R), 1972 (R), 1974 (R), 1977 (T,R), 1982 (T), 1984 (T,R), 1986 (T) |
| Chlordimeform (013) | 1971 (T,R), 1975 (T,R), 1977 (T), 1978 (T,R), 1979(T), 1980(T), 1985(T), 1986 (R), 1987 (T) |
| Chlorfenson | 1965 (T) |
| Chlorfenvinphos (014) | 1971 (T,R), 1984 (R), 1994 (T), 1996 (R) |

| | |
|---|---|
| Chlormequat (015) | 1970 (T,R), 1972 (T,R), 1976 (R), 1985 (R), 1994 (T,R), 1997 (T), 1999 (acute Rf D), 2000 (R) |
| Chlorobenzilate (016) | 1965 (T), 1968 (T,R), 1972 (R), 1975 (R), 1977 (R), 1980 (T) |
| Chloropicrin | 1965 (T,R) |
| Chloropropylate | 1968 (T,R), 1972 (R) |
| Chlorothalonil (081) | 1974 (T,R), 1977 (T,R), 1978 (R), 1979 (T,R), 1981 (T,R), 1983 (T,R), 1984 (corr. to 1983 report and T evaluation), 1985 (T,R), 1987 (T), 1988 (R), 1990 (T,R), 1991 (corr. to 1990 evaluation), 1992 (T), 1993 (R), 1997 (R) |
| Chlorpropham | 1965 (T), 2000 (T) |
| Chlorpyrifos (017) | 1972 (T,R), 1974 (R), 1975 (R), 1977 (T,R), 1981 (R), 1982 (T,R), 1983 (R), 1989 (R), 1995 (R), 1999 (T), 2000 (R) |
| Chlorpyrifos-methyl (090) | 1975 (T,R), 1976 (R, Annex I only), 1979 (R), 1990 (R), 1991 (T,R), 1992 (T and corr. to 1991 report), 1993 (R), 1994 (R) |
| Chlorthion | 1965 (T) |
| Clethodim (187) | 1994 (T,R), 1997 (R), 1999 (R) |
| Clofentezine (156) | 1986 (T,R), 1987 (R), 1989 (R), 1990 (R), 1992 (R) |
| Coumaphos (018) | 1968 (T,R), 1972 (R), 1975 (R), 1978 (R), 1980 (T,R), 1983 (R), 1987 (T), 1990 (T,R) |
| Crufomate (019) | 1968 (T,R), 1972 (R) |
| Cyanophenfos (091) | 1975 (T,R), 1978 (T: ADI extended, but no evaluation), 1980, (T), 1982 (R), 1983 (T) |
| Cycloxydim (179) | 1992 (T,R), 1993 (R) |
| Cyfluthrin (157) | 1986 (R), 1987 (T and corr. to 1986 report), 1989 (R), 1990 (R), 1992 (R) |
| Cyhalothrin (146) | 1984 (T,R), 1986 (R), 1988 (R) |
| Cyhexatin (tricyclohexyltin hydroxide) (067) | 1970 (T,R), 1973 (T,R), 1974 (R), 1975 (R), 1977 (T), 1978 (T,R), 1980 (T), 1981 (T), 1982 (R), 1983 (R), 1985 (R), 1988 (T), 1989 (T), 1991 (T,R), 1992 (R), 1994 (T) |
| Cypermethrin (118) | 1979 (T,R), 1981 (T,R), 1982 (R), 1983 (R), 1984 (R), 1985 (R), 1986 (R), 1987 (corr. to 1986 evaluation), 1988 (R), 1990 (R) |
| Cyromazine (169) | 1990 (T,R), 1991 (corr. to 1990 R evaluation), 1992 (R) |
| 2,4-D (020) | 1970 (T,R), 1971 (T,R), 1974 (T,R), 1975 (T,R), 1980 (R), 1985, (R), 1986 (R), 1987 (corr. to 1986 report, Annex I), 1996 (T), 1997 (E), 1998 (R) |
| Daminozide (104) | 1977 (T,R), 1983 (T), 1989 (T,R), 1991 (T) |
| DDT (021) | 1965 (T), 1966 (T,R), 1967 (T,R),1968 (T,R), 1969 (T,R), 1978 (R), 1979 (T), 1980 (T), 1983 (T), 1984 (T), 1993 (R), 1994 (R), 1996 (R) |
| Deltamethrin (135) | 1980 (T,R), 1981 (T,R), 1982 (T,R), 1984 (R), 1985 (R), 1986 (R), 1987 (R), 1988 (R), 1990 (R), 1992 (R), 2000 (T) |
| Demeton (092) | 1965 (T), 1967 (R), 1975 (R), 1982 (T) |
| Demeton-*S*-methyl (073) | 1973 (T,R), 1979 (R), 1982 (T), 1984 (T,R), 1989 (T,R), 1992 (R), 1998 (R) |

| | |
|---|---|
| Demeton-*S*-methylsulphon (164) | 1973 (T,R), 1982 (T), 1984 (T,R), 1989 (T,R), 1992 (R) |
| Dialifos (098) | 1976 (T,R), 1982 (T), 1985 (R) |
| Diazinon (022) | 1965 (T), 1966 (T), 1967 (R), 1968 (T,R), 1970 (T,R), 1975 (R), 1979 (R), 1993 (T,R), 1994 (R), 1996 (R), 1999 (R) |
| 1,2-Dibromoethane (023) | 1965 (T,R), 1966 (T,R), 1967 (R), 1968 (R), 1971 (R), 1979 (R), 1985 (R) |
| Dichlorfluanid (082) | 1969 (T,R), 1974 (T,R), 1977 (T,R), 1979 (T,R), 1981 (R),1982 (R), 1983 (T,R), 1985 (R) |
| 1,2-Dichloroethane (024) | 1965 (T,R), 1967 (R), 1971 (R), 1979 (R), 1985 (R) |
| Dichlorvos (025) | 1965 (T,R), 1966 (T,R), 1967 (T,R), 1969 (R), 1970 (T,R), 1974 (R), 1977 (T), 1993 (T,R) |
| Dicloran (083) | 1974 (T,R), 1977 (T,R), 1998 (T,R) |
| Dicofol (026) | 1968 (T,R), 1970 (R), 1974 (R), 1992 (T,R), 1994 (R) |
| Dieldrin (001) | 1965 (T), 1966 (T,R), 1967 (T,R), 1968 (R), 1969 (R), 1970, (T,R), 1974 (R), 1975 (R), 1977 (T), 1990 (R), 1992 (R) |
| Diflubenzuron (130) | 1981 (T,R), 1983 (R), 1984 (T,R), 1985 (T,R), 1988 (R) |
| Dimethipin (151) | 1985 (T,R), 1987 (T,R), 1988 (T,R), 1999 (T) |
| Dimethoate (027) | 1965 (T), 1966 (T), 1967 (T,R), 1970 (R), 1973 (R in evaluation of formothion), 1977 (R), 1978 (R), 1983 (R) 1984 (T,R) 1986 (R), 1987 (T,R), 1988 (R), 1990 (R), 1991 (corr. to 1990 evaluation), 1994 (R), 1996 (T), 1998 (R) |
| Dimethrin | 1965 (T) |
| Dinocap (087) | 1969 (T,R), 1974 (T,R), 1989 (T,R), 1992 (R), 1998 (R), 1999 (R), 2000 (T) |
| Dioxathion (028) | 1968 (T,R), 1972 (R) |
| Diphenyl (029) | 1966 (T,R), 1967 (T) |
| Diphenylamine (030) | 1969 (T,R), 1976 (T,R), 1979 (R), 1982 (T), 1984 (T,R), 1998 (T) |
| Diquat (031) | 1970 (T,R), 1972 (T,R), 1976 (R), 1977 (T,R), 1978 (R), 1994 (R) |
| Disulfoton (074) | 1973 (T,R), 1975 (T,R), 1979 (R), 1981 (R), 1984 (R), 1991 (T,R), 1992 (corr. to 1991 report, Annex I), 1994 (R), 1996 (T), 1998 (R) |
| Dithianon (180) | 1992 (T,R), 1995 (R), 1996 (corr. to 1995 report) |
| Dithiocarbamates (105) | 1965 (T), 1967 (T,R), 1970 (T,R), 1983 (R propineb, thiram), 1984 (R propineb), 1985 (R), 1987 (T thiram), 1988 (R thiram), 1990 (R), 1991 (corr. to 1990 evaluation), 1992 (T thiram), 1993 (T,R), 1995 (R), 1996 (T,R ferbam, ziram;, R thiram) |
| 4,6-Dinitro-*ortho*-cresol (DNOC) | 1965 (T) |
| Dodine (084) | 1974 (T,R), 1976 (T,R), 1977 (R), 2000 (T) |
| Edifenphos (099) | 1976 (T,R), 1979 (T,R), 1981 (T,R) |

| | |
|---|---|
| Endosulfan (032) | 1965 (T), 1967 (T,R), 1968 (T,R), 1971 (R), 1974 (R), 1975 (R), 1982 (T), 1985 (T,R), 1989 (T,R), 1993 (R), 1998 (T) |
| Endrin (033) | 1965 (T), 1970 (T,R), 1974 (R), 1975 (R), 1990 (R), 1992 (R) |
| Ethephon (106) | 1977 (T,R), 1978 (T,R), 1983 (R), 1985 (R), 1993 (T), 1994 (R), 1995 (T), 1997 (T) |
| Ethiofencarb (107) | 1977 (T,R), 1978 (R), 1981 (R), 1982 (T,R), 1983 (R) |
| Ethion (034) | 1968 (T,R), 1969 (R), 1970 (R), 1972 (T,R), 1975 (R), 1982 (T), 1983 (R), 1985 (T), 1986 (T), 1989 (T), 1990 (T), 1994 (R) |
| Ethopropophos (149) | 1983 (T), 1984 (R), 1987 (T), 1999 (R) |
| Ethoxyquin (035) | 1969 (T,R), 1998 (T) |
| Ethylene dibromide | See 1,2-Dibromoethane |
| Ethylene dichloride | See 1,2-Dichloroethane |
| Ethylene oxide | 1965 (T,R), 1968 (T,R), 1971 (R) |
| Ethylenethiourea (ETU) (108) | 1974 (R), 1977 (T,R), 1986 (T,R), 1987 (R), 1988 (T,R), 1990 (R), 1993 (T,R) |
| Etofenprox (184) | 1993 (T,R) |
| Etrimfos (123) | 1980 (T,R), 1982 (T,R[1]), 1986 (T,R), 1987 (R), 1988 (R), 1989 (R), 1990 (R) |
| Fenamiphos (085) | 1974 (T,R), 1977 (R), 1978 (R), 1980 (R), 1985 (T), 1987 (T), 1997 (T), 1999 (R) |
| Fenarimol (192) | 1995 (T,R,E), 1996 (R and corr. to 1995 report) |
| Fenbuconazole (197) | 1997 (T,R) |
| Fenbutatin oxide (109) | 1977 (T,R), 1979 (R), 1992 (T), 1993 (R) |
| Fenchlorfos (036) | 1968 (T,R), 1972 (R), 1983 (R) |
| Fenitrothion (037) | 1969 (T,R), 1974 (T,R), 1976 (R), 1977 (T,R), 1979 (R), 1982, (T) 1983 (R), 1984 (T,R), 1986 (T,R), 1987 (R and corr. to 1986 R evaluation), 1988 (T), 1989 (R), 2000 (T) |
| Fenpropathrin (185) | 1993 (T,R) |
| Fenpropimorph (188) | 1994 (T), 1995 (R), 1999 (R) |
| Fenpyroximate (193) | 1995 (T,R), 1996 (corr. to 1995 report.), 1999 (R) |
| Fensulfothion (038) | 1972 (T,R), 1982 (T), 1983 (R) |
| Fenthion (039) | 1971 (T,R), 1975 (T,R), 1977 (R), 1978 (T,R), 1979 (T), 1980 (T), 1983 (R), 1989 (R), 1995 (T,R,E), 1996 (corr. to 1995 report), 1997 (T), 2000 (R) |
| Fentin compounds (040) | 1965 (T), 1970 (T,R), 1972 (R), 1986 (R), 1991 (T,R), 1993 (R), 1994 (R) |
| Fenvalerate (119) | 1979 (T,R), 1981 (T,R), 1982 (T), 1984 (T,R), 1985 (R), 1986 (T,R), 1987 (R and corr. to 1986 report), 1988 (R), 1990 (R), 1991 (corr. to 1990 R evaluation) |
| Ferbam | See Dithiocarbamates, 1965 (T), 1967 (T,R), 1996 (T,R) |
| Fipronil | 1997 (T), 2000 (T) |
| Fipronil-desulfinyl | 1997 (T) |
| Flucythrinate (152) | 1985 (T,R), 1987 (R), 1988 (R), 1989 (R), 1990 (R), 1993 (R) |
| Flumethrin (195) | 1996 (T,R) |
| Flusilazole (165) | 1989 (T,R), 1990 (R), 1991 (R), 1993 (R), 1995 (T) |

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|---|---|
| Folpet (041) | 1969 (T,R), 1973 (T), 1974 (R), 1982 (T), 1984 (T,R), 1986 (T), 1987 (R), 1990 (T,R), 1991 (corr. to 1990 R evaluation), 1993 (T,R), 1994 (R), 1995 (T), 1997 (R), 1998 (R), 1999 (R) |
| Formothion (042) | 1969 (T,R), 1972 (R), 1973 (T,R), 1978 (R), 1998 (R) |
| Glufosinate-ammonium (175) | 1991 (T,R), 1992 (corr. to 1991 report, Annex I), 1994 (R), 1998 (R), 1999 (T,R)GLYPHOSATE (158) 1986 (T,R), 1987 (R and corr. to 1986 report), 1988 (R), 1994 (R), 1997 (T,R) |
| Guazatine (114) | 1978 (T.R), 1980 (R), 1997 (T,R) |
| Haloxyfop (194) | 1995 (T,R), 1996 (R and corr. to 1995 report) |
| Heptachlor (043) | 1965 (T), 1966 (T,R), 1967 (R), 1968 (R), 1969 (R), 1970 (T,R), 1974 (R), 1975 (R), 1977 (R), 1987 (R), 1991 (T,R), 1992 (corr. to 1991 report, Annex I), 1993 (R), 1994 (R) |
| Hexachlorobenzene (044) | 1969 (T,R), 1973 (T,R), 1974 (T,R), 1978(T), 1985 (R) |
| Hexaconazole (170) | 1990 (T,R), 1991 (R and corr. to 1990 R evaluation), 1993 (R) |
| Hexythiazox (176) | 1991 (T,R), 1994 (R), 1998 (R) |
| Hydrogen cyanide (045) | 1965 (T,R) |
| Hydrogen phosphide (046) | 1965 (T,R), 1966 (T,R), 1967 (R), 1969 (R), 1971 (R) |
| Imazalil (110) | 1977 (T,R), 1980 (T,R), 1984 (T,R), 1985 (T,R), 1986 (T), 1988 (R), 1989 (R), 1991 (T), 1994 (R), 2000 (T) |
| Iprodione (111) | 1977 (T,R), 1980 (R), 1992 (T), 1994 (R), 1995 (T) |
| Isofenphos (131) | 1981 (T,R), 1982 (T,R), 1984 (R), 1985 (R), 1986 (T,R), 1988 (R), 1992 (R) |
| Kresoxim-methyl (199) | 1998 (T,R) |
| Lead arsenate | 1965 (T), 1968 (T,R) |
| Leptophos (088) | 1974 (T,R), 1975 (T,R), 1978 (T,R) |
| Lindane (048) | 1965 (T), 1966 (T,R), 1967 (R), 1968 (R), 1969 (R), 1970 (T,R, published as Annex VI to 1971 evaluations), 1973 (T,R), 1974 (R), 1975 (R), 1977 (T,R), 1978 (R), 1979 (R), 1989 (T,R), 1997 (T) |
| Malathion (049) | 1965 (T), 1966 (T,R), 1967 (corr. to 1966 R evaluation), 1968 (R), 1969 (R), 1970 (R), 1973 (R), 1975 (R), 1977 (R), 1984 (R), 1997 (T), 1999 (R), 2000 (R) |
| Maleic hydrazide (102) | 1976 (T,R), 1977 (T,R), 1980 (T), 1984 (T,R), 1996 (T), 1998 (R) |
| Mancozeb (050) | 1967 (T,R), 1970 (T,R), 1974 (R), 1977 (R), 1980 (T,R), 1993 (T,R) |
| Maneb | See Dithiocarbamates, 1965 (T), 1967 (T,R), 1987 (T), 1993 (T,R) |
| Mecarbam (124) | 1980 (T,R), 1983 (T,R), 1985 (T,R), 1986 (T,R), 1987 (R) |

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|---|---|
| Metalaxyl (138) | 1982 (T,R), 1984 (R), 1985 (R), 1986 (R), 1987 (R), 1989 (R), 1990 (R), 1992 (R), 1995 (R) |
| Methacrifos (125) | 1980 (T,R), 1982 (T), 1986 (T), 1988 (T), 1990 (T,R), 1992 (R) |
| Methamidifos (100) | 1976 (T,R), 1979 (R), 1981 (R), 1982 (T,R[2]), 1984 (R), 1985 (T), 1989 (R), 1990 (T,R), 1994 (R), 1996 (R), 1997 (R) |
| Methidathion (051) | 1972 (T,R), 1975 (T,R), 1979 (R), 1992 (T,R), 1994 (R), 1997 (T) |
| Methiocarb (132) | 1981 (T,R), 1983 (T,R), 1984 (T), 1985 (T), 1986 (R), 1987 (T,R), 1988 (R), 1998 (T), 1999 (R) |
| Methomyl (094) | 1975 (R), 1976 (R), 1977 (R), 1978 (R), 1986 (T,R), 1987 (R), 1988 (R), 1989 (T,R), 1990 (R), 1991 (R) |
| Methoprene (147) | 1984 (T,R), 1986 (R), 1987 (T and corr. to 1986 report), 1988 (R), 1989 (R) |
| Methoxychlor | 1965 (T), 1977 (T) |
| Methyl bromide (052) | See Bromomethane |
| Metiram (186) | 1993 (T), 1995 (R) |
| Mevinphos (053) | 1965 (T), 1972 (T,R), 1996 (T), 1997 (E,R), 2000 (R) |
| MGK 264 | 1967 (T,R) |
| Monocrotophos (054) | 1972 (T,R), 1975 (T,R), 1991 (T,R), 1993 (T), 1994 (R) |
| Myclobutanil (181) | 1992 (T,R), 1997 (R), 1998 (R) |
| Nabam | See Dithiocarbamates, 1965 (T), 1976 (T,R) |
| Nitrofen (140) | 1983 (T,R) |
| Omethoate (055) | 1971 (T,R), 1975 (T,R), 1978 (T,R), 1979 (T), 1981 (T,R), 1984 (R), 1985 (T), 1986 (R), 1987 (R), 1988 (R), 1990 (R), 1998 (R) |
| Organomercury compounds | 1965 (T), 1966 (T,R), 1967 (T,R) |
| Oxamyl (126) | 1980 (T,R), 1983 (R), 1984 (T), 1985 (T,R), 1986 (R) |
| Oxydemeton-methyl (166) | 1965 (T, as demeton-*S*-methyl sulfoxide), 1967 (T), 1968 (R), 1973 (T,R), 1982 (T), 1984 (T,R), 1989 (T,R), 1992 (R), 1998 (R), 1999 (corr. to 1992 report) |
| Oxythioquinox | See Chinomethionat |
| Paclobutrazol (161) | 1988 (T,R), 1989 (R) |
| Paraquat (057) | 1970 (T,R), 1972 (T,R), 1976 (T,R), 1978 (R), 1981 (R), 1982 (T), 1985 (T), 1986 (T) |
| Parathion (058) | 1965 (T), 1967 (T,R), 1969 (R), 1970 (R), 1984 (R), 1991 (R), 1995 (T,R), 1997 (R), 2000 (R) |
| Parathion-methyl (059) | 1965 (T), 1968 (T,R), 1972 (R), 1975 (T,R), 1978 (T,R), 1979 (T), 1980 (T), 1982 (T), 1984 (T,R), 1991 (R), 1992 (R), 1994 (R), 1995 (T), 2000 (R) |
| Penconazole (182) | 1992 (T,R), 1995 (R) |
| Permethrin (120) | 1979 (T,R), 1980 (R), 1981 (T,R), 1982 (R), 1983 (R), 1984 (R), 1985 (R), 1986 (T,R), 1987 (T), 1988 (R), 1989 (R), 1991 (R), 1992 (corr. to 1991 report), 1999 (T) |

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|---|---|
| 2-Phenylphenol (056) | 1969 (T,R), 1975 (R), 1983 (T), 1985 (T,R), 1989 (T), 1990 (T,R), 1999 (T,R) |
| Phenothrin (127) | 1979 (R), 1980 (T,R), 1982 (T), 1984 (T), 1987 (R), 1988 (T,R) |
| Phenthoate (128) | 1980 (T,R), 1981 (R), 1984 (T) |
| Phorate (112) | 1977 (T,R), 1982 (T), 1983 (T), 1984 (R), 1985 (T), 1990 (R), 1991 (R), 1992 (R), 1993 (T), 1994 (T), 1996 (T) |
| Phosalone (060) | 1972 (T,R), 1975 (R), 1976 (R), 1993 (T), 1994 (R), 1997 (T), 1999 (R) |
| Phosmet (103) | 1976 (R), 1977 (corr. to 1976 R evaluation), 1978 (T,R), 1979 (T,R), 1981 (R), 1984 (R), 1985 (R), 1986 (R), 1987 (R and corr. to 1986 R evaluation), 1988 (R), 1994 (T), 1997 (R), 1998 (T) |
| Phosphine | See Hydrogen phosphide |
| Phosphamidon (061) | 1965 (T), 1966 (T), 1968 (T,R), 1969 (R), 1972 (R), 1974 (R), 1982 (T), 1985 (T), 1986 (T) |
| Phoxim (141) | 1982 (T), 1983 (R), 1984 (T,R), 1986 (R), 1987 (R), 1988 (R) |
| Piperonyl butoxide (062) | 1965 (T,R), 1966 (T,R), 1967 (R), 1969 (R), 1972 (T,R), 1992 (T,R), 1995 (T) |
| Pirimicarb (101) | 1976 (T,R), 1978 (T,R), 1979 (R), 1981 (T,R), 1982 (T), 1985 (R) |
| Pirimiphos-methyl (086) | 1974 (T,R), 1976 (T,R), 1977 (R), 1979 (R), 1983 (R), 1985 (R), 1992 (T), 1994 (R) |
| Prochloraz (142) | 1983 (T,R), 1985 (R), 1987 (R), 1988 (R), 1989 (R), 1990 (R), 1991 (corr. to 1990 report, Annex I, and R evaluation), 1992 (R) |
| Procymidone(136) | 1981 (R), 1982 (T), 1989 (T,R), 1990 (R), 1991 (corr. to 1990 Annex I), 1993 (R), 1998 (R) |
| Profenofos (171) | 1990 (T,R), 1992 (R), 1994 (R), 1995 (R) |
| Propamocarb (148) | 1984 (T,R), 1986 (T,R), 1987 (R) |
| Propargite (113) | 1977 (T,R), 1978 (R), 1979 (R), 1980 (T,R), 1982 (T,R), 1999 (T) |
| Propham (183) | 1965 (T), 1992 (T,R) |
| Propiconazole (160) | 1987 (T,R), 1991 (R), 1994 (R) |
| Propineb | 1977 (T,R), 1980 (T), 1983 (T), 1984 (R), 1985 (T,R), 1993 (T,R) |
| Propoxur (075) | 1973 (T,R), 1977 (R), 1981 (R), 1983 (R), 1989 (T), 1991 (R), 1996 (R) |
| Propylenethiourea (PTU, 150) | 1993 (T,R), 1994 (R), 1999 (T) |
| Pyrazophos (153) | 1985 (T,R), 1987 (R), 1992 (T,R), 1993 (R) |
| Pyrethrins (063) | 1965 (T), 1966 (T,R), 1967 (R), 1968 (R), 1969 (R), 1970 (T), 1972 (T,R), 1974 (R), 1999 (T), 2000 (R) |
| Pyriproxyfen | 1999 (R,T), 2000 (R) |
| Quintozene (064) | 1969 (T,R) 1973 (T,R), 1974 (R), 1975 (T,R), 1976 (Annex I, corr. to 1975 R evaluation), 1977 (T,R), 1995 (T,R), 1998 (R) |
| 2,4,5-T (121) | 1970 (T,R), 1979 (T,R), 1981 (T) |

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|---|---|
| Tebuconazole (189) | 1994 (T,R), 1996 (corr. to Annex II of 1995 report), 1997 (R) |
| Tebufenozide (196) | 1996 (T,R), 1997 (R), 1999 (R) |
| Tecnazine (115) | 1974 (T,R), 1978 (T,R), 1981 (R), 1983 (T), 1987 (R), 1989 (R), 1994 (T,R) |
| Teflubenzuron (190) | 1994 (T), 1996 (R) |
| Terbufos (167) | 1989 (T,R), 1990 (T,R) |
| Thianedazole (065) | 1970 (T,R), 1971 (R), 1972 (R), 1975 (R), 1977 (T,R), 1979 (R), 1981 (R), 1997 (R), 2000 (R) |
| Thiodicarb (154) | 1985 (T,R), 1986 (T), 1987 (R), 1988 (R), 2000 (T) |
| Thiometon (076) | 1969 (T,R), 1973 (T,R), 1976 (R), 1979 (T,R), 1988 (R) |
| Thiphanate-methyl (077) | 1973 (T,R), 1975 (T,R), 1977 (T), 1978 (R), 1988 (R), 1990 (R), 1994 (R), 1995 (T,E), 1998 (T,R) |
| Thiram (105) | See Dithiocarbamates, 1965 (T), 1967 (T,R), 1970 (T,R), 1974 (T), 1977 (T), 1983 (R), 1984 (R), 1985 (T,R), 1987 (T), 1988 (R), 1989 (R), 1992 (T), 1996 (R) |
| Tolclofos-methyl (191) | 1994 (T,R) 1996 (corr. to Annex II of 1995 report) |
| Tolylfluanid (162) | 1988 (T,R), 1990 (R), 1991 (corr. to 1990 report) |
| Toxaphene | See Camphechlor |
| Triadimefon (133) | 1979 (R), 1981 (T,R), 1983 (T,R), 1984 (R), 1985 (T,R), 1986 (R), 1987 (R and corr. to 1986 R evaluation), 1988 (R), 1989 (R), 1992 (R), 1995 (R) |
| Triadimenol (168) | 1989 (T,R), 1992 (R), 1995 (R) |
| Triazolylalanine | 1989 (T,R) |
| Triazophos (143) | 1982 (T), 1983 (R), 1984 (corr. to 1983 report, Annex I), 1986 (T,R), 1990 (R), 1991 (T and corr. to 1990 R evaluation), 1992 (R), 1993 (T,R) |
| Trichlorfon (066) | 1971 (T,R), 1975 (T,R), 1978 (T,R), 1987 (R) |
| Trichloronat | 1971 (T,R) |
| Trichloroethylene | 1968 (R) |
| Tricyclohexyltin hydroxide | See Cyhexatin |
| Triforine (116) | 1977 (T), 1978 (T,R), 1997 (T) |
| Triphenyltin compounds | See Fentin compounds |
| Vamidothion (078) | 1973 (T,R), 1982 (T), 1985 (T,R), 1987 (R), 1988 (T), 1990 (R), 1992 (R) |
| Vinclozolin (159) | 1986 (T,R), 1987 (R and corr. to 1986 report and R evaluation), 1988 (T,R), 1989 (R), 1990 (R), 1992 (R), 1995 (T) |
| Zineb (105) | See Dithiocarbamates, 1965 (T), 1967 (T,R), 1993 (T) |
| Ziram (105) | See Dithiocarbamates, 1965 (T), 1967 (T,R), 1996 (T,R) |

# ANNEX II

## PREVIOUS FAO AND WHO DOCUMENTS


1. FAO/WHO. 1962 Principles governing consumer safety in relation to pesticide residues. Report of a meeting of a WHO Expert Committee on Pesticide Residues held jointly with the FAO Panel of Experts on the Use of Pesticides in Agriculture. FAO Plant Production and Protection Division Report, No. PL/1961/11; WHO Technical Report Series, No. 240.

2. FAO/WHO. 1964 Evaluations of the toxicity of pesticide residues in food. Report of a Joint Meeting of the FAO Committee on Pesticides in Agriculture and the WHO Expert Committee on Pesticide Residues. FAO Meeting Report, No. PL/1963/13; WHO/Food Add./23.

3. FAO/WHO. 1965a Evaluations of the toxicity of pesticide residues in food. Report of the Second Joint Meeting of the FAO Committee on Pesticides in Agriculture and the WHO Expert Committee on Pesticide Residues. FAO Meeting Report, No. PL/1965/10; WHO/Food Add./26.65.

4. FAO/WHO. 1965b Evaluations of the toxicity of pesticide residues in food. FAO Meeting Report, No. PL/1965/10/1; WHO/Food Add./27.65.

5. FAO/WHO. 1965c Evaluation of the hazards to consumers resulting from the use of fumigants in the protection of food. FAO Meeting Report, No. PL/1965/10/2; WHO/Food Add./28.65.

6. FAO/WHO. 1967a Pesticide residues in food. Joint report of the FAO Working Party on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 73; WHO Technical Report Series, No. 370.

7. FAO/WHO. 1967b Evaluation of some pesticide residues in food. FAO/PL:CP/15; WHO/Food Add./67.32.

8. FAO/WHO. 1968a Pesticide residues. Report of the 1967 Joint Meeting of the FAO Working Party and the WHO Expert Committee. FAO Meeting Report, No. PL:1967/M/11; WHO Technical Report Series, No. 391.

9. FAO/WHO. 1968b 1967 Evaluations of some pesticide residues in food. FAO/PL:1967/M/11/1; WHO/Food Add./68.30.

10. FAO/WHO. 1969a Pesticide residues in food. Report of the 1968 Joint Meeting of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 78; WHO Technical Report Series, No. 417.

11. FAO/WHO. 1969b 1968 Evaluation of some pesticide residues in food. FAO/PL:1968/M/9/1; WHO/Food Add./69.35.

12. FAO/WHO. Pesticide residues in food. Report of the 1969 Joint Meeting

1970a of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 84; WHO Technical Report Series, No. 458.

13. FAO/WHO. 1969 Evaluation of some pesticide residues in food.
1970b FAO/PL:1969/M/17/1; WHO/Food Add./70.38

14. FAO/WHO. Pesticide residues in food. Report of the 1970 Joint Meeting
1971a of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 87; WHO Technical Report Series, No. 474.

15. FAO/WHO. 1970 Evaluation of some pesticide residues in food.
1971b AGP:1970/M/12/1; WHO/Food Add./71.42.

16. FAO/WHO. Pesticide residues in food. Report of the 1971 Joint Meeting
1972a of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 88; WHO Technical Report Series, No. 502.

17. FAO/WHO. 1971 Evaluation of some pesticide residues in food.
1972b AGP:1971/M/9/1; WHO Pesticide Residues Series, No. 1.

18. FAO/WHO. Pesticide residues in food. Report of the 1972 Joint Meeting
1973a of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 90; WHO Technical Report Series, No. 525.

19. FAO/WHO. 1972 Evaluation of some pesticide residues in food.
1973b AGP:1972/M/9/1; WHO Pesticide Residues Series, No. 2.

20. FAO/WHO. Pesticide residues in food. Report of the 1973 Joint Meeting
1974a of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 92; WHO Technical Report Series, No. 545.

21. FAO/WHO. 1973 Evaluation of some pesticide residues in food.
1974b FAO/AGP/1973/M/9/1; WHO Pesticide Residues Series, No.3.

22. FAO/WHO. Pesticide residues in food. Report of the 1974 Joint Meeting
1975a of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Agricultural Studies, No. 97; WHO Technical Report Series, No. 574.

23. FAO/WHO. 1974 Evaluation of some pesticide residues in food.
1975b FAO/AGP/1974/M/9/11; WHO Pesticide Residues Series, No.4.

24. FAO/WHO. Pesticide residues in food. Report of the 1975 Joint Meeting
1976a of the FAO Working Party of experts on Pesticide Residues and the WHO Expert Committee on Pesticide Residues. FAO Plant Production and Protection Series, No.1; WHO Technical Report Series, No. 592.

25. FAO/WHO. 1975 Evaluation of some pesticide residues in food.
1976b AGP:1975/M/13; WHO Pesticide Residues Series, No. 5.

26. FAO/WHO. Pesticide residues in food. Report of the 1976 Joint Meeting
1977a of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues.
FAO Food and Nutrition Series, No. 9; FAO Plant Production and Protection Series, No. 8; WHO Technical Report Series, No. 612.

27. FAO/WHO. 1976 Evaluation of some pesticide residues in food.
1977b AGP:1976/M/14.

28. FAO/WHO. Pesticide residues in food - 1977. Report of the Joint Meeting
1978a of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues.
FAO Plant Production and Protection Paper 10 Rev.

29. FAO/WHO. Pesticide residues in food: 1977 evaluations.
1978b FAO Plant Production and Protection Paper 10 Sup.

30. FAO/WHO. Pesticide residues in food - 1978. Report of the Joint Meeting
1979a of the FAO Panel of Experts on Pesticide Residues and the Environment and the WHO Expert Group on Pesticide Residues.
FAO Plant Production and Protection Paper 15.

31. FAO/WHO. Pesticide residues in food: 1978 evaluations.
1979b FAO Plant Production and Protection Paper 15 Sup.

32. FAO/WHO. Pesticide residues in food - 1979. Report of the Joint Meeting
1980a of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 20.

33. FAO/WHO. Pesticide residues in food: 1979 evaluations.
1980b FAO Plant Production and Protection Paper 20 Sup.

34. FAO/WHO. Pesticide residues in food - 1980. Report of the Joint Meeting
1981a of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 26.

35. FAO/WHO. Pesticide residues in food: 1980 evaluations.
1981b FAO Plant Production and Protection Paper 26 Sup.

36. FAO/WHO. Pesticide residues in food - 1981. Report of the Joint Meeting
1982a of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 37.

37. FAO/WHO. Pesticide residues in food: 1981 evaluations.
1982b FAO Plant Production and Protection Paper 42.

38. FAO/WHO. Pesticide residues in food - 1982. Report of the Joint Meeting
1983a of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 46.

39. FAO/WHO. Pesticide residues in food: 1982 evaluations.
1983b FAO Plant Production and Protection Paper 49.

40. FAO/WHO. Pesticide residues in food - 1983. Report of the Joint Meeting
1984 of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 56.

41. FAO/WHO. Pesticide residues in food: 1983 evaluations.
1985a FAO Plant Production and Protection Paper 61.

42. FAO/WHO. Pesticide residues in food - 1984. Report of the Joint Meeting
1985b of the FAO Panel of Experts on Pesticide Residues in Food and the Environment and the WHO Expert Group on Pesticide Residues. FAO Plant Production and Protection Paper 62.

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| Source | Count | Commodity | Year start | Year end | Comp | Input data | | | Intervals | | | | | | | | | | | | | check | | | | Estimated Violation rates | | | | | | Estimated percentiles | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | lower | 0.00 | 0.10 | 1.05 | 2.55 | 5.05 | 10.00 | | | | | | | | | | | median | average | rsd | | | | | | | | | |
| | | | | | | # samples\upper | 0.10 | 1.05 | 2.55 | 5.05 | 10.00 | ##### | | | | | | Mean | m | max | | | | | | 0.5 | 1 | 2 | 3 | 5 | | 0.5% | 0.2% | 0.1% |
| FAO 142 Table 1 | Australia | Beef (fat) | 1989 | 1994 | DDT | 39854 | 39790 | 61 | 1 | 1 | 0 | 1 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | 0.71% | 0.30% | 0.12% | 0.06% | 0.03% | | 0.67 | 1.35 | 2.22 |
| FAO 142 Table 1 | Australia | Beef (fat) | 1989 | 1994 | DDE | 39854 | 38432 | 1394 | 24 | 3 | 1 | 0 | | | | | | | | ok | | | 0.00 | 0.02 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | Beef (fat) | 1989 | 1994 | TDE | 39854 | 39799 | 53 | 2 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | Buffalo(fat) | 1989 | 1994 | DDT | 432 | 432 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | Buffalo(fat) | 1989 | 1994 | DDE | 432 | 429 | 3 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | Buffalo(fat) | 1989 | 1994 | TDE | 432 | 439 | 1 | 0 | 0 | 0 | 0 | | | | | | | | not ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | Deer(fat) | 1989 | 1994 | DDT | 110 | 110 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | 0.16% | 0.06% | 0.02% | 0.01% | 0.00% | | 0.21 | 0.42 | 0.69 |
| FAO 142 Table 1 | Australia | Deer(fat) | 1989 | 1994 | DDE | 110 | 109 | 0 | 1 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.01 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | Deer(fat) | 1989 | 1994 | TDE | 110 | 110 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | Game goat (fat) | 1989 | 1994 | DDT | 87 | 87 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| FAO 142 Table 1 | Australia | Game goat (fat) | 1989 | 1994 | DDE | 87 | 87 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | Game goat (fat) | 1989 | 1994 | TDE | 87 | 87 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | goat (fat) | 1989 | 1994 | DDT | 927 | 925 | 2 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | goat (fat) | 1989 | 1994 | DDE | 927 | 916 | 7 | 0 | 0 | 0 | 0 | | | | | | | | not ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | goat (fat) | 1989 | 1994 | TDE | 927 | 924 | 3 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | horse (fat) | 1989 | 1994 | DDT | 1939 | 1930 | 9 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | 0.81% | 0.34% | 0.13% | 0.07% | 0.03% | | 0.74 | 1.50 | 2.45 |
| FAO 142 Table 1 | Australia | horse (fat) | 1989 | 1994 | DDE | 1939 | 1868 | 70 | 1 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.02 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | horse (fat) | 1989 | 1994 | TDE | 1939 | 1936 | 2 | 1 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | kangeroo (fat) | 1989 | 1994 | DDT | 482 | 482 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | 0.16% | 0.06% | 0.02% | 0.01% | 0.00% | | 0.21 | 0.43 | 0.71 |
| FAO 142 Table 1 | Australia | kangeroo (fat) | 1989 | 1994 | DDE | 482 | 476 | 6 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.01 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | kangeroo (fat) | 1989 | 1994 | TDE | 482 | 482 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | sheep (fat) | 1989 | 1994 | DDT | 29270 | 29228 | 41 | 0 | 0 | 0 | 0 | | | | | | | | not ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | sheep (fat) | 1989 | 1994 | DDE | 29270 | 26940 | 2314 | 13 | 0 | 0 | 0 | | | | | | | | not ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | sheep (fat) | 1989 | 1994 | TDE | 29270 | 29241 | 28 | 1 | 0 | 0 | 0 | | | | | | | | ok | | | | | | | | | | | | | | |
| FAO 142 Table 1 | Australia | porcine (fat) | 1989 | 1994 | DDT | 15900 | 15823 | 74 | 2 | 0 | 1 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | 0.37% | 0.14% | 0.05% | 0.03% | 0.01% | | 0.39 | 0.79 | 1.30 |
| FAO 142 Table 1 | Australia | porcine (fat) | 1989 | 1994 | DDE | 15900 | 15684 | 210 | 5 | 1 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.01 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | porcine (fat) | 1989 | 1994 | TDE | 15900 | 15858 | 40 | 2 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | rabbit(fat) | 1989 | 1994 | DDT | 570 | 570 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | 0.02% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.09 | 0.15 |
| FAO 142 Table 1 | Australia | rabbit(fat) | 1989 | 1994 | DDE | 570 | 570 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| FAO 142 Table 1 | Australia | rabbit(fat) | 1989 | 1994 | TDE | 570 | 569 | 1 | 0 | 0 | 0 | 0 | | | | | | | | ok | | | 0.00 | 0.00 | 3.04 | | | | | | | | | |
| | | | | | | lower | 0.00 | 0.00 | 0.00 | 0.01 | 0.02 | 0.02 | 0.05 | 0.11 | 0.21 | 0.51 | 1.05 | 2.05 | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.00 | 0.00 | 0.01 | 0.02 | 0.02 | 0.05 | 0.11 | 0.21 | 0.51 | 1.05 | 2.05 | 5.00 | | max | | | | | | | | | | | | | | |
| FAO 142 Table2 | Germany | meat (fat) | 1993 | 1993 | sum | 777 | 128 | 0 | 87 | 54 | 102 | 230 | 119 | 39 | 17 | 1 | 0 | 0 | # | ok | | | 0.01 | 0.11 | 3.04 | 3.37% | 1.67% | 0.77% | 0.47% | 0.24% | | 2.84 | 5.74 | 9.40 |
| FAO 142 Table2 | Germany | Meat (fresh) | 1993 | 1993 | sum | 1080 | 618 | 192 | 221 | 36 | 7 | 3 | 2 | 0 | 1 | 0 | 0 | 0 | # | ok | | | 0.00 | 0.01 | 3.04 | 0.12% | 0.04% | 0.01% | 0.01% | 0.00% | | 0.17 | 0.34 | 0.56 |
| FAO 142 Table2 | Germany | sheep (fat) | 1993 | 1993 | sum | 87 | 6 | 0 | 2 | 4 | 14 | 18 | 11 | 24 | 6 | 1 | 1 | 0 | # | ok | | | 0.01 | 0.17 | 3.04 | 5.31% | 2.79% | 1.35% | 0.85% | 0.46% | | 4.67 | 9.43 | 15.44 |
| FAO 142 Table2 | Germany | sheep (fresh) | 1993 | 1993 | sum | 30 | 8 | 3 | 9 | 2 | 5 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | # | ok | | | 0.00 | 0.04 | 3.04 | 1.26% | 0.56% | 0.23% | 0.13% | 0.06% | | 1.09 | 2.21 | 3.62 |
| FAO 142 Table2 | Germany | rabbit(fat) | 1993 | 1993 | sum | 15 | 0 | 0 | 0 | 2 | 2 | 3 | 3 | 1 | 1 | 3 | 0 | 0 | # | ok | | | 0.02 | 0.31 | 3.04 | 8.52% | 4.76% | 2.46% | 1.61% | 0.91% | | 8.24 | 16.64 | 27.25 |
| FAO 142 Table2 | Germany | rabbit(fresh) | 1993 | 1993 | sum | 140 | 39 | 35 | 44 | 9 | 8 | 4 | 1 | 0 | 0 | 0 | 0 | 0 | # | ok | | | 0.00 | 0.02 | 3.04 | 0.63% | 0.26% | 0.10% | 0.05% | 0.02% | | 0.61 | 1.22 | 2.00 |
| ~~FAO 142 Table2~~ | ~~Germany~~ | ~~bacon/poultry~~ | ~~1993~~ | ~~1993~~ | ~~sum~~ | ~~671~~ | ~~220~~ | ~~0~~ | ~~37~~ | ~~74~~ | ~~147~~ | ~~131~~ | ~~50~~ | ~~8~~ | ~~2~~ | ~~0~~ | ~~0~~ | ~~2~~ | ~~#~~ | ~~ok~~ | | | | | | | | | | | | | | |
| FAO 142 Table2 | Germany | Meat salted | 1993 | 1993 | sum | 29 | 17 | 0 | 0 | 6 | 3 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | # | ok | | | 0.00 | 0.02 | 3.04 | 0.43% | 0.17% | 0.06% | 0.03% | 0.01% | | 0.44 | 0.90 | 1.47 |
| | | | | | | lower | 0.00 | 0.02 | 0.50 | 1.00 | 2.00 | 5.00 | | | | | | | | | | | median | average | rsd | | | | | | | | | |
| | | | | | | # samples\upper | 0.02 | 0.50 | 1.00 | 2.00 | 5.00 | ##### | | | | | | Mean | | median | | | | | | 0.5 | 1 | 2 | 3 | 5 | | | | |
| FAO 142 Table 3 | New Zea | Lambs | 1990 | 1991 | sum | 244 | 121 | 119 | 1 | 3 | 0 | 0 | | | | | | 0.1300 | # | # | ok | | 0.02 | 0.08 | 2.04 | 2.45% | 0.84% | 0.25% | 0.11% | 0.04% | | 1.36 | 2.23 | 3.16 |
| FAO 142 Table 3 | New Zea | Lambs | 1991 | 1992 | sum | 159 | 64 | 84 | 4 | 7 | 0 | 0 | | | | | | 0.2400 | # | # | ok | | 0.03 | 0.13 | 2.04 | 4.91% | 1.90% | 0.63% | 0.30% | 0.11% | | 2.27 | 3.74 | 5.29 |
| FAO 142 Table 3 | New Zea | Lambs | 1992 | 1993 | sum | 261 | 123 | 127 | 7 | 2 | 2 | 0 | | | | | | 0.1900 | # | # | ok | | 0.02 | 0.09 | 2.04 | 3.16% | 1.13% | 0.34% | 0.16% | 0.06% | | 1.63 | 2.67 | 3.79 |
| FAO 142 Table 3 | New Zea | Lambs | 1993 | 1994 | sum | 301 | 123 | 161 | 13 | 4 | 0 | 0 | | | | | | 0.1900 | # | # | ok | | 0.03 | 0.12 | 2.04 | 4.39% | 1.66% | 0.54% | 0.26% | 0.09% | | 2.08 | 3.42 | 4.85 |
| FAO 142 Table 3 | New Zea | Sheep | 1990 | 1991 | sum | 203 | 118 | 77 | 5 | 1 | 2 | 0 | | | | | | 0.2100 | # | # | ok | | 0.02 | 0.06 | 2.04 | 1.78% | 0.58% | 0.16% | 0.07% | 0.02% | | 1.09 | 1.79 | 2.54 |
| FAO 142 Table 3 | New Zea | Sheep | 1991 | 1992 | sum | 158 | 71 | 78 | 5 | 4 | 0 | 0 | | | | | | 0.2000 | # | # | ok | | 0.03 | 0.10 | 2.04 | 3.73% | 1.37% | 0.43% | 0.20% | 0.07% | | 1.84 | 3.02 | 4.28 |
| FAO 142 Table 3 | New Zea | Sheep | 1992 | 1993 | sum | 84 | 47 | 34 | 2 | 1 | 0 | 0 | | | | | | 0.2000 | # | # | ok | | 0.02 | 0.06 | 2.04 | 1.93% | 0.64% | 0.18% | 0.08% | 0.03% | | 1.15 | 1.89 | 2.68 |
| FAO 142 Table 3 | New Zea | Sheep | 1993 | 1994 | sum | 103 | 35 | 61 | 3 | 2 | 2 | 0 | | | | | | 0.2400 | # | # | ok | | 0.04 | 0.16 | 2.04 | 6.41% | 2.60% | 0.90% | 0.45% | 0.17% | | 2.83 | 4.65 | 6.58 |
| FAO 142 Table 3 | New Zea | cow | 1990 | 1991 | sum | 202 | 132 | 69 | 1 | 0 | 0 | 0 | | | | | | 0.0850 | # | # | ok | | 0.01 | 0.04 | 2.04 | 0.90% | 0.26% | 0.07% | 0.03% | 0.01% | | 0.70 | 1.16 | 1.64 |
| FAO 142 Table 3 | New Zea | cow | 1991 | 1992 | sum | 261 | 136 | 117 | 5 | 3 | 0 | 0 | | | | | | 0.1400 | # | # | ok | | 0.02 | 0.07 | 2.04 | 2.31% | 0.78% | 0.23% | 0.10% | 0.03% | | 1.30 | 2.14 | 3.03 |
| FAO 142 Table 3 | New Zea | cow | 1992 | 1993 | sum | 132 | 85 | 47 | 1 | 0 | 0 | 0 | | | | | | 0.0800 | # | # | not ok | | | | | | | | | | | | | |
| FAO 142 Table 3 | New Zea | cow | 1993 | 1994 | sum | 164 | 87 | 71 | 4 | 2 | 0 | 0 | | | | | | 0.1600 | # | # | ok | | 0.02 | 0.07 | 2.04 | 2.26% | 0.77% | 0.22% | 0.10% | 0.03% | | 1.28 | 2.11 | 2.99 |
| FAO 142 Table 3 | New Zea | Suckling calves | 1990 | 1991 | sum | 306 | 105 | 175 | 18 | 8 | 0 | 0 | | | | | | 0.2300 | # | # | ok | | 0.04 | 0.16 | 2.04 | 6.47% | 2.63% | 0.91% | 0.46% | 0.18% | | 2.85 | 4.68 | 6.63 |
| FAO 142 Table 3 | New Zea | Suckling calves | 1991 | 1992 | sum | 309 | 63 | 206 | 26 | 7 | 7 | 0 | | | | | | 0.3100 | # | # | ok | | 0.08 | 0.29 | 2.04 | 12.62% | 5.86% | 2.34% | 1.27% | 0.54% | | 5.25 | 8.62 | 12.21 |
| FAO 142 Table 3 | New Zea | Suckling calves | 1992 | 1993 | sum | 310 | 117 | 188 | 5 | 0 | 0 | 0 | | | | | | 0.1300 | # | # | ok | | 0.03 | 0.11 | 2.04 | 4.22% | 1.59% | 0.51% | 0.24% | 0.09% | | 2.02 | 3.32 | 4.70 |

| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FAO 142 Table 3 | New Zea | Suckling calves | 1993 | 1994 | sum | 301 | 84 | 199 | 9 | 6 | 2 | 1 | | | | | | 0.2900 | # | # | ok | | 0.05 | 0.19 | 2.04 | | 7.75% | 3.26% | 1.17% | 0.60% | 0.24% | | 3.33 | 5.47 | 7.75 |
| FAO 142 Table 3 | New Zea | pigs | 1990 | 1991 | sum | 232 | 144 | 85 | 3 | 0 | 0 | 0 | | | | | | 0.0700 | # | # | ok | | 0.01 | 0.05 | 2.04 | | 1.17% | 0.36% | 0.09% | 0.04% | 0.01% | | 0.83 | 1.36 | 1.93 |
| FAO 142 Table 3 | New Zea | pigs | 1991 | 1992 | sum | 305 | 125 | 175 | 1 | 2 | 2 | 0 | | | | | | 0.1200 | # | # | ok | | 0.03 | 0.10 | 2.04 | | 3.75% | 1.38% | 0.43% | 0.20% | 0.07% | | 1.84 | 3.03 | 4.29 |
| FAO 142 Table 3 | New Zea | pigs | 1992 | 1993 | sum | 288 | 118 | 161 | 4 | 3 | 1 | 1 | | | | | | 0.1700 | # | # | ok | | 0.03 | 0.11 | 2.04 | | 4.03% | 1.50% | 0.48% | 0.23% | 0.08% | | 1.95 | 3.21 | 4.54 |
| FAO 142 Table 3 | New Zea | pigs | 1993 | 1994 | sum | 100 | 31 | 66 | 2 | 1 | 0 | 0 | | | | | | 0.1200 | # | # | ok | | 0.04 | 0.15 | 2.04 | | 5.97% | 2.39% | 0.82% | 0.41% | 0.15% | | 2.67 | 4.38 | 6.20 |
| FAO 142 Table 3 | New Zea | deer | 1990 | 1991 | sum | 10 | 4 | 4 | 1 | 0 | 0 | 0 | | | | | | | | | not ok | | | | | | | | | | | | | | |
| FAO 142 Table 3 | New Zea | deer | 1991 | 1992 | sum | 17 | 13 | 3 | 1 | 0 | 0 | 0 | | | | | | | | | ok | | 0.01 | 0.03 | 2.04 | | 0.57% | 0.16% | 0.04% | 0.01% | 0.00% | | 0.54 | 0.89 | 1.26 |
| FAO 142 Table 3 | New Zea | deer | 1992 | 1993 | sum | 15 | 9 | 6 | 0 | 0 | 0 | 0 | | | | | | 0.1700 | # | # | ok | | 0.01 | 0.05 | 2.04 | | 1.23% | 0.38% | 0.10% | 0.04% | 0.01% | | 0.86 | 1.41 | 2.00 |
| FAO 142 Table 3 | New Zea | deer | 1993 | 1994 | sum | 185 | 83 | 91 | 9 | 2 | 0 | 0 | | | | | | 0.1700 | # | # | ok | | 0.03 | 0.10 | 2.04 | | 3.72% | 1.37% | 0.43% | 0.20% | 0.07% | | 1.83 | 3.01 | 4.27 |
| FAO 142 Table 3 | New Zea | goat | 1990 | 1991 | sum | 7 | 5 | 2 | 0 | 0 | 0 | 0 | | | | | | 0.1700 | # | # | ok | | 0.01 | 0.03 | 2.04 | | 0.55% | 0.15% | 0.04% | 0.01% | 0.00% | | 0.53 | 0.87 | 1.23 |
| FAO 142 Table 3 | New Zea | goat | 1991 | 1992 | sum | 17 | 13 | 4 | 0 | 0 | 0 | 0 | | | | | | 0.1700 | # | # | ok | | 0.01 | 0.02 | 2.04 | | 0.35% | 0.09% | 0.02% | 0.01% | 0.00% | | 0.41 | 0.68 | 0.96 |
| FAO 142 Table 3 | New Zea | goat | 1992 | 1993 | sum | 13 | 9 | 4 | 0 | 0 | 0 | 0 | | | | | | 0.1700 | # | # | ok | | 0.01 | 0.03 | 2.04 | | 0.65% | 0.18% | 0.04% | 0.02% | 0.01% | | 0.58 | 0.96 | 1.36 |
| FAO 142 Table 3 | New Zea | goat | 1993 | 1994 | sum | 30 | 29 | 1 | 0 | 0 | 0 | 0 | | | | | | 0.1700 | # | # | ok | | 0.00 | 0.00 | 2.04 | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.07 | 0.11 | 0.16 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| FAO 142 Table 4 | New Zea | Lambs (known | 1993 | 1994 | sum | 403 | 7 | 183 | 82 | 60 | 58 | 13 | | | | | | 1.2000 | # | | ok | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | lower | 0 | 0 | 1 | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0 | 1 | 5 | | | | | | | | | | | | | | | | | | | | | | | | | | |
| FAO 142 Table 5 | Norway | bovine fat | 1990 | 1994 | sum | 537 | 536 | 1 | 0 | | | | | | | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.03 |
| FAO 142 Table 5 | Norway | pig fat | 1990 | 1994 | sum | 537 | 536 | 1 | 0 | | | | | | | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.03 |
| FAO 142 Table 5 | Norway | sheep fat | 1990 | 1994 | sum | 149 | 149 | 0 | 0 | | | | | | | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| FAO 142 Table 5 | Norway | reindeer fat | 1990 | 1994 | sum | 31 | 31 | 0 | 0 | | | | | | | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| FAO 142 Table 5 | Norway | moose fat | 1990 | 1994 | sum | 169 | 169 | 0 | 0 | | | | | | | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| | | | | | | lower | 0.00 | 0.01 | 0.06 | 0.11 | 0.51 | | | | | | | | | | | | median | average | rsd | | | | | | | | | | |
| | | | | | | # samples\upper | 0.01 | 0.06 | 0.11 | 0.51 | 1.00 | | | | | | | Mean | | median | | | | | | | 0.5 | 1 | 2 | 3 | 5 | | | | |
| FAO 142 Table 6 | Thailand | cattle meat | 1993 | 1993 | sum | 30 | 2 | 23 | 2 | 3 | 0 | | | | | | | | | | ok | | 0.03 | 0.04 | 0.96 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.16 | 0.20 | 0.23 |
| FAO 142 Table 6 | Thailand | cattle meat | 1994 | 1994 | sum | 123 | 2 | 94 | 16 | 11 | 0 | | | | | | | | | | ok | | 0.04 | 0.04 | 0.96 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.19 | 0.24 | 0.27 |
| FAO 142 Table 6 | Thailand | pig meat | 1993 | 1993 | sum | 65 | 1 | 48 | 10 | 6 | 0 | | | | | | | | | | ok | | 0.04 | 0.05 | 0.96 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.20 | 0.25 | 0.28 |
| FAO 142 Table 6 | Thailand | pig meat | 1994 | 1994 | sum | 157 | 1 | 129 | 19 | 8 | 0 | | | | | | | | | | ok | | 0.03 | 0.04 | 0.96 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.18 | 0.22 | 0.25 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| | | | | | | lower | 0.00 | 0.01 | 0.11 | 0.21 | 0.31 | 0.51 | 1.01 | 2.51 | 5.01 | | | targets | | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.01 | 0.11 | 0.21 | 0.31 | 0.51 | 1.01 | 2.51 | 5.01 | ##### | | | gemiddel | | med | | | | | | | 0.5 | 1 | 2 | 3 | 5 | | | | |
| FAO 142 Table 7 | USA | horse | 1991 | 1991 | sum | 106 | 105 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.04 | 0.06 |
| FAO 142 Table 7 | USA | bull | 1991 | 1991 | sum | 25 | 25 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.01 |
| FAO 142 Table 7 | USA | steer | 1991 | 1991 | sum | 1967 | 1922 | 27 | 10 | 2 | 3 | 2 | 1 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.02% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.09 | 0.15 |
| FAO 142 Table 7 | USA | beef cow | 1991 | 1991 | sum | 272 | 264 | 3 | 1 | 3 | 1 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.03% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.07 | 0.13 | 0.22 |
| FAO 142 Table 7 | USA | Heifer | 1991 | 1991 | sum | 1327 | 1301 | 18 | 5 | 1 | 0 | 1 | 1 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.07 | 0.12 |
| FAO 142 Table 7 | USA | dairy cow | 1991 | 1991 | sum | 217 | 211 | 4 | 0 | 1 | 1 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.02% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.06 | 0.11 | 0.18 |
| FAO 142 Table 7 | USA | formula-fed calf | 1991 | 1991 | sum | 319 | 319 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| FAO 142 Table 7 | USA | Non-formula ca | 1991 | 1991 | sum | 243 | 238 | 3 | 1 | 0 | 0 | 1 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.09 | 0.14 |
| FAO 142 Table 7 | USA | heavy calf | 1991 | 1991 | sum | 280 | 272 | 3 | 3 | 1 | 1 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.03% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.06 | 0.12 | 0.20 |
| FAO 142 Table 7 | USA | mature sheep | 1991 | 1991 | sum | 27 | 27 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.01 |
| FAO 142 Table 7 | USA | lamb | 1991 | 1991 | sum | 320 | 314 | 2 | 1 | 3 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.08 | 0.14 |
| FAO 142 Table 7 | USA | goat | 1991 | 1991 | sum | 94 | 92 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 1 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.03 | 0.05 | 0.08 |
| FAO 142 Table 7 | USA | Market hog | 1991 | 1991 | sum | 329 | 326 | 1 | 0 | 1 | 0 | 1 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.04 | 0.07 |
| FAO 142 Table 7 | USA | boar or stag | 1991 | 1991 | sum | 61 | 60 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.03 | 0.06 | 0.10 |
| FAO 142 Table 7 | USA | sow | 1991 | 1991 | sum | 253 | 247 | 3 | 1 | 0 | 0 | 0 | 1 | 1 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.03 | 0.06 | 0.10 |
| FAO 142 Table 7 | USA | rabbit | 1991 | 1991 | sum | 81 | 78 | 1 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.04% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.08 | 0.17 | 0.27 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| FAO 142 Table 8 | USA | horse | 1992 | 1992 | sum | 98 | 93 | 2 | 0 | 1 | 0 | 2 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.08% | 0.03% | 0.01% | 0.00% | 0.00% | | 0.13 | 0.26 | 0.43 |
| FAO 142 Table 8 | USA | bull | 1992 | 1992 | sum | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.04 | 0.07 |
| FAO 142 Table 8 | USA | steer | 1992 | 1992 | sum | 196 | 184 | 4 | 5 | 1 | 1 | 1 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.01 | 3.04 | | 0.10% | 0.04% | 0.01% | 0.01% | 0.00% | | 0.15 | 0.31 | 0.50 |
| FAO 142 Table 8 | USA | beef cow | 1992 | 1992 | sum | 56 | 51 | 2 | 2 | 0 | 1 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.01 | 3.04 | | 0.19% | 0.07% | 0.02% | 0.01% | 0.01% | | 0.24 | 0.48 | 0.78 |
| FAO 142 Table 8 | USA | Heifer | 1992 | 1992 | sum | 131 | 127 | 3 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.02% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.06 | 0.12 | 0.19 |
| FAO 142 Table 8 | USA | dairy cow | 1992 | 1992 | sum | 240 | 200 | 23 | 9 | 5 | 0 | 3 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.02 | 3.04 | | 0.55% | 0.22% | 0.08% | 0.05% | 0.02% | | 0.54 | 1.09 | 1.78 |
| FAO 142 Table 8 | USA | formula-fed calf | 1992 | 1992 | sum | 334 | 331 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.06 |
| FAO 142 Table 8 | USA | Non-formula ca | 1992 | 1992 | sum | 293 | 280 | 3 | 7 | 2 | 0 | 0 | 1 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.05% | 0.02% | 0.01% | 0.00% | 0.00% | | 0.09 | 0.19 | 0.31 |
| FAO 142 Table 8 | USA | heavy calf | 1992 | 1992 | sum | 295 | 246 | 29 | 15 | 2 | 1 | 1 | 0 | 1 | 0 | | | | | | ok | | 0.00 | 0.02 | 3.04 | | 0.49% | 0.20% | 0.07% | 0.04% | 0.02% | | 0.49 | 0.99 | 1.63 |
| FAO 142 Table 8 | USA | mature sheep | 1992 | 1992 | sum | 25 | 22 | 2 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.01 | 3.04 | | 0.26% | 0.10% | 0.03% | 0.02% | 0.01% | | 0.30 | 0.60 | 0.98 |
| FAO 142 Table 8 | USA | lamb | 1992 | 1992 | sum | 317 | 278 | 13 | 10 | 4 | 7 | 4 | 1 | 0 | 0 | | | | | | ok | | 0.00 | 0.01 | 3.04 | | 0.37% | 0.15% | 0.05% | 0.03% | 0.01% | | 0.39 | 0.79 | 1.30 |
| FAO 142 Table 8 | USA | goat | 1992 | 1992 | sum | 104 | 99 | 2 | 1 | 0 | 0 | 1 | 1 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.04% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.09 | 0.17 | 0.28 |
| FAO 142 Table 8 | USA | Market hog | 1992 | 1992 | sum | 3285 | 3192 | 42 | 20 | 12 | 11 | 4 | 2 | 0 | 2 | | | | | | ok | | 0.00 | 0.00 | 3.04 | | 0.02% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.06 | 0.12 | 0.19 |
| FAO 142 Table 8 | USA | boar or stag | 1992 | 1992 | sum | 60 | 54 | 1 | 2 | 3 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.01 | 3.04 | | 0.28% | 0.11% | 0.04% | 0.02% | 0.01% | | 0.31 | 0.63 | 1.04 |
| FAO 142 Table 8 | USA | sow | 1992 | 1992 | sum | 259 | 243 | 8 | 3 | 1 | 1 | 2 | 0 | 1 | 0 | | | | | | ok | | 0.00 | 0.01 | 3.04 | | 0.08% | 0.03% | 0.01% | 0.00% | 0.00% | | 0.13 | 0.27 | 0.44 |

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|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| FAO 142 Table 8 | USA | rabbit | 1992 | 1992 | sum | 71 | 70 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.00 | 3.04 | | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.03 | 0.05 | 0.08 |
| FAO 142 Table 9 | USA | horse | 1993 | 1993 | sum | 425 | 383 | 17 | 14 | 2 | 6 | 3 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.01 | 3.04 | | | 0.27% | 0.10% | 0.04% | 0.02% | 0.01% | | 0.31 | 0.63 | 1.03 |
| FAO 142 Table 9 | USA | bull | 1993 | 1993 | sum | 555 | 521 | 13 | 12 | 7 | 0 | 0 | 2 | 0 | 0 | | | | | ok | | 0.00 | 0.01 | 3.04 | | | 0.10% | 0.04% | 0.01% | 0.01% | 0.00% | | 0.15 | 0.31 | 0.51 |
| FAO 142 Table 9 | USA | steer | 1993 | 1993 | sum | 523 | 494 | 13 | 10 | 0 | 3 | 3 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.01 | 3.04 | | | 0.10% | 0.03% | 0.01% | 0.01% | 0.00% | | 0.15 | 0.30 | 0.49 |
| FAO 142 Table 9 | USA | beef cow | 1993 | 1993 | sum | 651 | 605 | 23 | 13 | 4 | 5 | 1 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.01 | 3.04 | | | 0.14% | 0.05% | 0.02% | 0.01% | 0.00% | | 0.19 | 0.38 | 0.63 |
| FAO 142 Table 9 | USA | Heifer | 1993 | 1993 | sum | 546 | 524 | 11 | 8 | 1 | 1 | 1 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.00 | 3.04 | | | 0.05% | 0.02% | 0.01% | 0.00% | 0.00% | | 0.10 | 0.20 | 0.33 |
| FAO 142 Table 9 | USA | dairy cow | 1993 | 1993 | sum | 272 | 220 | 15 | 17 | 11 | 7 | 2 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.03 | 3.04 | | | 1.00% | 0.44% | 0.17% | 0.10% | 0.05% | | 0.90 | 1.81 | 2.96 |
| FAO 142 Table 9 | USA | formula-fed calf | 1993 | 1993 | sum | 529 | 525 | 3 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.00 | 3.04 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.04 | 0.06 |
| FAO 142 Table 9 | USA | Non-formula ca | 1993 | 1993 | sum | 458 | 437 | 8 | 4 | 2 | 5 | 1 | 1 | 0 | 0 | | | | | ok | | 0.00 | 0.00 | 3.04 | | | 0.07% | 0.02% | 0.01% | 0.00% | 0.00% | | 0.12 | 0.24 | 0.39 |
| FAO 142 Table 9 | USA | heavy calf | 1993 | 1993 | sum | 498 | 413 | 52 | 18 | 7 | 3 | 2 | 3 | 0 | 0 | | | | | ok | | 0.00 | 0.02 | 3.04 | | | 0.56% | 0.23% | 0.09% | 0.05% | 0.02% | | 0.55 | 1.11 | 1.82 |
| FAO 142 Table 9 | USA | mature sheep | 1993 | 1993 | sum | 470 | 425 | 22 | 8 | 8 | 3 | 2 | 2 | 0 | 0 | | | | | ok | | 0.00 | 0.01 | 3.04 | | | 0.22% | 0.08% | 0.03% | 0.01% | 0.01% | | 0.27 | 0.54 | 0.88 |
| FAO 142 Table 9 | USA | lamb | 1993 | 1993 | sum | 574 | 493 | 39 | 29 | 7 | 4 | 2 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.02 | 3.04 | | | 0.47% | 0.19% | 0.07% | 0.04% | 0.02% | | 0.47 | 0.96 | 1.57 |
| FAO 142 Table 9 | USA | goat | 1993 | 1993 | sum | 533 | 501 | 17 | 10 | 2 | 2 | 1 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.01 | 3.04 | | | 0.10% | 0.04% | 0.01% | 0.01% | 0.00% | | 0.15 | 0.31 | 0.51 |
| FAO 142 Table 9 | USA | Market hog | 1993 | 1993 | sum | 499 | 488 | 3 | 4 | 3 | 1 | 0 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.00 | 3.04 | | | 0.02% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.06 | 0.12 | 0.19 |
| FAO 142 Table 9 | USA | boar or stag | 1993 | 1993 | sum | 452 | 436 | 7 | 2 | 3 | 1 | 1 | 1 | 0 | 1 | | | | | ok | | 0.00 | 0.00 | 3.04 | | | 0.04% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.08 | 0.16 | 0.26 |
| FAO 142 Table 9 | USA | sow | 1993 | 1993 | sum | 537 | 511 | 12 | 6 | 4 | 3 | 1 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.00 | 3.04 | | | 0.08% | 0.03% | 0.01% | 0.00% | 0.00% | | 0.13 | 0.25 | 0.41 |
| FAO 142 Table 9 | USA | rabbit | 1993 | 1993 | sum | 84 | 81 | 2 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.00 | 3.04 | | | 0.04% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.08 | 0.16 | 0.26 |
| | | | | | | lower | 0.00 | 0.04 | 0.11 | 0.21 | 0.31 | 0.51 | 1.01 | 2.51 | 5.01 | | | targets | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.04 | 0.11 | 0.21 | 0.31 | 0.51 | 1.01 | 2.51 | 5.01 | ##### | | | gemiddel | med | | | | | | | | 0.5 | 1 | 2 | 3 | 5 | | | | |
| FAO 142 Table 10 | USA | cattle | 1994 | 1994 | sum | 3955 | 3657 | 151 | 66 | 39 | 31 | 7 | 2 | 1 | 1 | | | | | | | 0.00 | 0.02 | 3.04 | | | 0.57% | 0.24% | 0.09% | 0.05% | 0.02% | | 0.56 | 1.13 | 1.85 |
| FAO 142 Table 10 | USA | pigs | 1994 | 1994 | sum | 1457 | 1346 | 57 | 27 | 14 | 8 | 3 | 1 | 0 | 1 | | | | | | | 0.00 | 0.02 | 3.04 | | | 0.57% | 0.23% | 0.09% | 0.05% | 0.02% | | 0.56 | 1.13 | 1.84 |
| FAO 142 Table 10 | USA | horses | 1994 | 1994 | sum | 217 | 213 | 1 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | 0.00 | 0.00 | 3.04 | | | 0.05% | 0.02% | 0.01% | 0.00% | 0.00% | | 0.10 | 0.20 | 0.32 |
| FAO 142 Table 10 | USA | sheep/goats | 1994 | 1994 | sum | 900 | 692 | 91 | 55 | 27 | 15 | 18 | 2 | 0 | 0 | | | | | | | 0.01 | 0.10 | 3.04 | | | 3.18% | 1.57% | 0.71% | 0.43% | 0.22% | | 2.68 | 5.40 | 8.85 |
| | | | | | | lower | 0.00 | 0.01 | 0.01 | 0.1 | | | | | | | | targets | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.01 | 0.01 | 0.10 | 5 | | | | | | | | gemiddel | med | | | | | | | | 0.5 | 1 | 2 | 3 | 5 | | | | |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~pig kidney~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~23~~ | ~~23~~ | ~~0~~ | ~~0~~ | ~~0~~ | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~lamb kidney~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~25~~ | ~~23~~ | ~~2~~ | ~~0~~ | ~~0~~ | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~ox kidney~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~31~~ | ~~31~~ | ~~0~~ | ~~0~~ | ~~0~~ | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~lamb liver~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~24~~ | ~~24~~ | ~~0~~ | ~~0~~ | ~~0~~ | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~ox liver~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~24~~ | ~~24~~ | ~~0~~ | ~~0~~ | ~~0~~ | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | |
| FAO 142 Table 14 | UK | rabbit | 1994 | 1994 | sum | 62 | 52 | 1 | 9 | 0 | | | | | | | | | | ok | | 0.00 | 0.01 | 3.04 | | | 0.26% | 0.10% | 0.03% | 0.02% | 0.01% | | 0.30 | 0.60 | 0.98 |
| | | | | | | lower | 0.00 | 0.01 | 0.10 | 1 | | | | | | | | targets | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.01 | 0.10 | 1.00 | 5 | | | | | | | | gemiddel | med | | | | | | | | 0.5 | 1 | 2 | 3 | 5 | | | | |
| FAO 142 Table 14 | UK | lamb | 1994 | 1994 | sum | 74 | 38 | 23 | 13 | 0 | | | | | | | | | | ok | | 0.01 | 0.14 | 3.04 | | | 4.22% | 2.15% | 1.01% | 0.63% | 0.33% | | 3.61 | 7.29 | 11.94 |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~cattle kidney fat~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~116~~ | ~~108~~ | ~~8~~ | ~~0~~ | ~~0~~ | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~pig kidney fat~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~114~~ | ~~108~~ | ~~6~~ | ~~0~~ | ~~0~~ | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~sheep kidney fa~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~114~~ | ~~96~~ | ~~10~~ | ~~0~~ | ~~0~~ | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | | **All representative data** | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | | critical level (mg/kg) | | | 0.5 | 1 | 2 | 3 | 5 | percentile | 0.50% | 0.20% | 0.10% |
| | | | | | | | | | | | | | | | | | | | | | | | average | | | 1.2% | 0.5% | 0.2% | 0.1% | 0.0% | average | 0.77 | 1.39 | 2.12 |
| | | | | | | | | | | | | | | | | | | | | | | | weighted average | | | 0.7% | 0.3% | 0.1% | 0.1% | 0.0% | nted average | 0.59 | 1.16 | 1.86 |
| | | | | | | | | | | | | | | | | | | | | | | | **New Zealand data** | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | | critical level (mg/kg) | | | 0.5 | 1 | 2 | 3 | 5 | percentile | 0.50% | 0.20% | 0.10% |
| | | | | | | | | | | | | | | | | | | | | | | | average | | | 3.4% | 1.3% | 0.4% | 0.2% | 0.1% | average | 1.66 | 2.72 | 3.86 |
| | | | | | | | | | | | | | | | | | | | | | | | weighted average | | | 4.3% | 1.7% | 0.6% | 0.3% | 0.1% | nted average | 2.06 | 3.38 | 4.79 |

| Source | Count | Commodity | Year start | Year end | Comp | Input data | | | Intervals | | | | | | | | | | | | check | | | | | | Estimated Violation rates | | | | | | Estimated percentiles | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | | lower | 0.00 | 0.10 | 1.05 | 2.55 | 5.05 | 10.00 | | | | | | | | | | | median | average | rsd | | | | | | | | | | |
| | | | | | | # samples\upper | 0.10 | 1.05 | 2.55 | 5.05 | 10.00 | ##### | | | | | | | | | | | | 0.5 | | 1 | 2 | 3 | 5 | | 0.5% | 0.2% | 0.1% | | |
| FAO 142 Table 1 | Australia | Emu (fat) | 1989 | 1994 | DDT | 9 | 9 | 0 | 0 | 0 | 0 | 0 | | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 2.14% | 0.52% | 0.10% | 0.03% | 0.01% | | 1.02 | 1.50 | 1.98 |
| FAO 142 Table 1 | Australia | Emu (fat) | 1989 | 1994 | DDE | 9 | 7 | 2 | 0 | 0 | 0 | 0 | | | | | | | | | ok | | 0.04 | 0.08 | 2.07 | | | | | | | | | | |
| FAO 142 Table 1 | Australia | Emu (fat) | 1989 | 1994 | TDE | 9 | 9 | 0 | 0 | 0 | 0 | 0 | | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | | | | | | | | |
| FAO 142 Table 1 | Australia | Poultry (fat) | 1989 | 1994 | DDT | 2167 | 2161 | 4 | 2 | 0 | 0 | 0 | | | | | | | | | ok | | 0.00 | 0.01 | 2.07 | | 0.69% | 0.13% | 0.02% | 0.01% | 0.00% | | 0.58 | 0.85 | 1.12 |
| FAO 142 Table 1 | Australia | Poultry (fat) | 1989 | 1994 | DDE | 2167 | 2007 | 151 | 9 | 0 | 0 | 0 | | | | | | | | | ok | | 0.02 | 0.04 | 2.07 | | | | | | | | | | |
| FAO 142 Table 1 | Australia | Poultry (fat) | 1989 | 1994 | TDE | 2167 | 2165 | 1 | 1 | 0 | 0 | 0 | | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | | | | | | | | |
| | | | | | | lower | 0.00 | 0.00 | 0.00 | 0.01 | 0.02 | 0.02 | 0.05 | 0.11 | 0.21 | 0.51 | 1.05 | 2.05 | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.00 | 0.00 | 0.01 | 0.02 | 0.02 | 0.05 | 0.11 | 0.21 | 0.51 | 1.05 | 2.05 | 5.00 | # | | | | | | | | | | | | | | | | |
| FAO 142 Table2 | Germany | poultry (fat) | 1993 | 1993 | sum | 74 | 17 | 0 | 3 | 13 | 19 | 15 | 4 | 3 | 0 | 0 | 0 | 0 | # | ok | | | 0.01 | 0.02 | 2.07 | | 0.05% | 0.01% | 0.00% | 0.00% | 0.00% | | 0.20 | 0.30 | 0.40 |
| FAO 142 Table2 | Germany | Poultry (fresh) | 1993 | 1993 | sum | 63 | 42 | 10 | 11 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | # | ok | | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.03 |
| ~~FAO 142 Table2~~ | ~~Germany~~ | ~~poultry liver~~ | ~~1993~~ | ~~1993~~ | ~~sum~~ | ~~14~~ | ~~11~~ | ~~0~~ | ~~3~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~#~~ | ~~ok~~ | | | | | | | | | | | | | | | | |
| ~~FAO 142 Table2~~ | ~~Germany~~ | ~~bacon/poultry~~ | ~~1993~~ | ~~1993~~ | ~~sum~~ | ~~671~~ | ~~220~~ | ~~0~~ | ~~37~~ | ~~74~~ | ~~147~~ | ~~131~~ | ~~50~~ | ~~0~~ | ~~2~~ | ~~0~~ | ~~0~~ | ~~2~~ | ~~#~~ | ~~ok~~ | | | | | | | | | | | | | | | | |
| | | | | | | | 1E-06 | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | 0.1 | 0.2 | 0.5 | 1 | 2 | 5 | 1E+05 | | | | | | | | | | | | | | | | | | | | | | |
| ZBES-2802-00 525 | Germany | chicken meat | 1994 | 1997 | sum | 215 | 207 | 3 | 3 | 1 | 1 | 0 | 0 | | | | | | | | ok | | 0.01 | 0.03 | 2.07 | | 0.16% | 0.02% | 0.00% | 0.00% | 0.00% | | 0.31 | 0.45 | 0.60 |
| | | | | | | lower | 0 | 0 | 1 | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0 | 1 | 5 | | | | | | | | | | | | | | | | | | | | | | | | | | |
| FAO 142 Table 5 | Norway | hens | 1990 | 1994 | sum | 145 | 145 | 0 | 0 | | | | | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| | | | | | | lower | 0.00 | 0.01 | 0.06 | 0.11 | 0.51 | | | | | | | | | | | | median | average | rsd | | | | | | | | | | |
| | | | | | | # samples\upper | 0.01 | 0.06 | 0.11 | 0.51 | 1.00 | | | | | | | | | | | | 0.5 | | 1 | | 2 | 3 | 5 | | | | | | |
| FAO 142 Table 6 | Thailand | chicken meat | 1993 | 1993 | sum | 9514 | 47 | 3618 | 3481 | 2342 | 26 | | | | | | | | | | ok | | 0.07 | 0.08 | 0.77 | | 0.16% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.39 | 0.47 | 0.55 |
| FAO 142 Table 6 | Thailand | chicken meat | 1994 | 1994 | sum | 14650 | 457 | 9475 | 3669 | 1027 | 22 | | | | | | | | | | ok | | 0.04 | 0.05 | 0.77 | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.23 | 0.28 | 0.32 |
| FAO 142 Table 6 | Thailand | duck meat | 1993 | 1993 | sum | 2291 | 27 | 1778 | 395 | 90 | 1 | | | | | | | | | | ok | | 0.03 | 0.04 | 0.77 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.20 | 0.24 | 0.28 |
| FAO 142 Table 6 | Thailand | duck meat | 1994 | 1994 | sum | 1810 | 19 | 1575 | 199 | 17 | 0 | | | | | | | | | | ok | | 0.03 | 0.04 | 0.77 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.17 | 0.20 | 0.24 |
| | | | | | | | 0.00 | 0.01 | 0.05 | 0.09 | 0.13 | 0.17 | 0.21 | 0.51 | 1.01 | 2.00 | | | | | | | | | | | | | | | | | | | |
| | | | | | | | 0.01 | 0.05 | 0.09 | 0.13 | 0.17 | 0.21 | 0.51 | 1.01 | 2.00 | #### | | | | | | | | | | | | | | | | | | | |
| F:PR-DATA INDEX 200 | Thailand | chicken meat | 1997 | 1997 | sum | 3882 | 180 | 3113 | 477 | 71 | 13 | 11 | 13 | 0 | 2 | 2 | | | | | ok | | 0.02 | 0.03 | 0.77 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.14 | 0.18 | 0.21 |
| F:PR-DATA INDEX 200 | Thailand | chicken meat | 1998 | 1998 | sum | 2910 | 923 | 1801 | 156 | 16 | 4 | 5 | 4 | 0 | 1 | 0 | | | | | ok | | 0.01 | 0.02 | 0.77 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.08 | 0.10 | 0.12 |
| F:PR-DATA INDEX 200 | Thailand | chicken meat | 1999 | 1999 | sum | 2537 | 1194 | 1208 | 93 | 24 | 6 | 3 | 6 | 2 | 1 | 0 | | | | | ok | | 0.01 | 0.01 | 0.77 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.07 | 0.08 | 0.09 |
| F:PR-DATA INDEX 200 | Thailand | duck meat | 1997 | 1997 | sum | 399 | 103 | 288 | 5 | 0 | 0 | 2 | 1 | 0 | 0 | 0 | | | | | ok | | 0.01 | 0.02 | 0.77 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.08 | 0.10 | 0.12 |
| F:PR-DATA INDEX 200 | Thailand | duck meat | 1998 | 1998 | sum | 464 | 347 | 115 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | ok | | 0.01 | 0.01 | 0.77 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.05 | 0.05 |
| F:PR-DATA INDEX 200 | Thailand | duck meat | 1999 | 1999 | sum | 96 | 86 | 10 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | ok | | 0.00 | 0.01 | 0.77 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.03 |
| | | | | | | lower | 0.00 | 0.01 | 0.11 | 0.21 | 0.31 | 0.51 | 1.01 | 2.51 | 5.01 | | | | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.01 | 0.11 | 0.21 | 0.31 | 0.51 | 1.01 | 2.51 | 5.01 | ##### | | | | | | | | | | | | 0.5 | 1 | 2 | 3 | 5 | | | | |
| FAO 142 Table 7 | USA | young chicken | 1991 | 1991 | sum | 3197 | 3195 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.01 | 0.01 |
| FAO 142 Table 7 | USA | mature chicken | 1991 | 1991 | sum | 338 | 337 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.01 | 0.02 |
| FAO 142 Table 7 | USA | young turkey | 1991 | 1991 | sum | 3267 | 3243 | 23 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.02 |
| FAO 142 Table 7 | USA | mature turkey | 1991 | 1991 | sum | 205 | 202 | 2 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.04 |
| FAO 142 Table 7 | USA | duck | 1991 | 1991 | sum | 108 | 108 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| FAO 142 Table 7 | USA | goose | 1991 | 1991 | sum | 62 | 58 | 4 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.06 | 0.08 |
| FAO 142 Table 8 | USA | young chicken | 1992 | 1992 | sum | 415 | 415 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| FAO 142 Table 8 | USA | mature chicken | 1992 | 1992 | sum | 321 | 317 | 3 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.03 |
| FAO 142 Table 8 | USA | young turkey | 1992 | 1992 | sum | 319 | 306 | 13 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.03 | 0.05 | 0.06 |
| FAO 142 Table 8 | USA | mature turkey | 1992 | 1992 | sum | 203 | 190 | 12 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.06 | 0.08 |
| FAO 142 Table 8 | USA | duck | 1992 | 1992 | sum | 109 | 109 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| FAO 142 Table 8 | USA | goose | 1992 | 1992 | sum | 38 | 37 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.04 | 0.05 |
| FAO 142 Table 9 | USA | young chicken | 1993 | 1993 | sum | 498 | 495 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.02 |
| FAO 142 Table 9 | USA | mature chicken | 1993 | 1993 | sum | 457 | 447 | 9 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.04 |
| FAO 142 Table 9 | USA | young turkey | 1993 | 1993 | sum | 519 | 508 | 10 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.04 |
| FAO 142 Table 9 | USA | mature turkey | 1993 | 1993 | sum | 228 | 224 | 4 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.04 |
| FAO 142 Table 9 | USA | duck | 1993 | 1993 | sum | 322 | 321 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.02 |
| FAO 142 Table 9 | USA | goose | 1993 | 1993 | sum | 3 | 3 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| 205-7760:12/30/98 | USA | young chicken | 1995 | 1995 | sum | 485 | 482 | 2 | 0 | 0 | 0 | 1 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.03 |

| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 205-7760:12/30/98 | USA | mature chicken | 1995 | 1995 | sum | 532 | 524 | 6 | 1 | 1 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.04 |
| 205-7760:12/30/98 | USA | young turkey | 1995 | 1995 | sum | 523 | 505 | 17 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.03 | 0.04 | 0.06 |
| 205-7760:12/30/98 | USA | mature turkey | 1995 | 1995 | sum | 261 | 244 | 15 | 0 | 0 | 1 | 0 | 1 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.06 | 0.08 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 205-7760:12/30/98 | USA | young chicken | 1996 | 1996 | sum | 178 | 176 | 2 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.02 | 0.03 |
| 205-7760:12/30/98 | USA | mature chicken | 1996 | 1996 | sum | 187 | 183 | 3 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.05 |
| 205-7760:12/30/98 | USA | young turkey | 1996 | 1996 | sum | 391 | 381 | 9 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.04 | 0.05 |
| 205-7760:12/30/98 | USA | mature turkey | 1996 | 1996 | sum | 68 | 64 | 4 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.06 | 0.07 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 205-7760:12/30/98 | USA | young chicken | 1997 | 1997 | sum | 274 | 273 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.01 | 0.02 |
| 205-7760:12/30/98 | USA | mature chicken | 1997 | 1997 | sum | 102 | 101 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.03 |
| 205-7760:12/30/98 | USA | young turkey | 1997 | 1997 | sum | 148 | 148 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| 205-7760:12/30/98 | USA | mature turkey | 1997 | 1997 | sum | 107 | 107 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 205-7760:12/30/98 | USA | young chicken | 1998 | 1998 | sum | 269 | 268 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.01 | 0.02 |
| 205-7760:12/30/98 | USA | mature chicken | 1998 | 1998 | sum | 95 | 94 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.04 |
| 205-7760:12/30/98 | USA | young turkey | 1998 | 1998 | sum | 245 | 244 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.01 | 0.02 | 0.02 |
| 205-7760:12/30/98 | USA | mature turkey | 1998 | 1998 | sum | 150 | 145 | 5 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.03 | 0.04 | 0.05 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | lower | 0.00 | 0.04 | 0.11 | 0.21 | 0.31 | 0.51 | 1.01 | 2.51 | 5.01 | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.04 | 0.11 | 0.21 | 0.31 | 0.51 | 1.01 | 2.51 | 5.01 | ##### | | | | | | | | | | | | | 0.5 | 1 | 2 | 3 | 5 | | | | |
| FAO 142 Table 10 | USA | poultry | 1994 | 1994 | sum | 1990 | 1973 | 13 | 3 | 0 | 1 | 0 | 0 | 0 | 0 | | | | | | | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.05 | 0.08 | 0.10 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | lower | 0.00 | 0.008 | 0.01 | 0.1 | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.008 | 0.01 | 0.10 | 5 | | | | | | | | | | | | 0.5 | | 1 | | 2 | 3 | 5 | | | | | | | | |
| ~~FAO 142 Table 14~~ | ~~UK~~ | ~~chicken liver~~ | ~~1994~~ | ~~1994~~ | ~~sum~~ | ~~31~~ | ~~30~~ | ~~1~~ | ~~0~~ | ~~0~~ | | | | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | | |
| FAO 142 Table 14 | UK | pheasant | 1994 | 1994 | sum | 68 | 63 | 5 | 0 | 0 | | | | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.03 | 0.04 | 0.06 |
| FAO 142 Table 14 | UK | wood pidgeon | 1994 | 1994 | sum | 30 | 29 | 0 | 1 | 0 | | | | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.04 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | lower | 0.00 | 0.010 | 0.02 | 0.05 | 1.00 | 2.00 | 5.00 | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | # samples\upper | 0.010 | 0.02 | 0.05 | 1.00 | 2.00 | 5.00 | 10.00 | | | | | | | | | | | 0.5 | | | 1 | 2 | 3 | 5 | | | | | | |
| Additional info | UK | chicken meat | 1990 | 1990 | sum | 118 | 115 | 2 | 1 | 0 | 0 | 0 | 0 | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.02 | 0.03 | 0.04 |
| Additional info | UK | chicken meat | 1992 | 1992 | sum | 72 | 64 | 0 | 7 | 1 | 0 | 0 | 0 | | | | | | | | ok | | 0.00 | 0.01 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.07 | 0.10 | 0.13 |
| Additional info | UK | chicken lean br | 1995 | 1995 | sum | 35 | 35 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| Additional info | UK | chicken > 10 % | 1995 | 1995 | sum | 37 | 37 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| ~~Additional info~~ | ~~UK~~ | ~~chicken liver~~ | ~~1991~~ | ~~1991~~ | ~~sum~~ | ~~18~~ | ~~18~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | ~~0~~ | | | | | | | | ~~ok~~ | | | | | | | | | | | | | | | | |
| Additional info | UK | poultry | 1996 | 1996 | sum | 24 | 24 | 0 | 0 | 0 | 0 | 0 | 0 | | | | | | | | ok | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.00 | 0.00 | 0.00 |
| Additional info | UK | composite mea | 1996 | 1996 | sum | 24 | 22 | 2 | 0 | 0 | 0 | 0 | 0 | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| | | | | | | | 0.0000 | 0.0100 | 0.1000 | 0.2000 | 0.5000 | 1.0000 | 2.0000 | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | 0.0100 | 0.1000 | 0.2000 | 0.5000 | 1.0000 | 2.0000 | ##### | | | | | | | | | | | | | | | | | | | | | | | | |
| Fax about Nat. Vet. | Poland | chicken (fat) | 1996 | 1996 | sum | 408 | 54 | 334 | 15 | 2 | 3 | 0 | 0 | | | | | | | | ok | | 0.03 | 0.06 | 2.07 | | | 1.08% | 0.23% | 0.04% | 0.01% | 0.00% | | 0.72 | 1.06 | 1.39 |
| Fax about Nat. Vet. | Poland | chicken (fat) | 1997 | 1997 | sum | 406 | 124 | 261 | 8 | 4 | 5 | 4 | 0 | | | | | | | | ok | | 0.02 | 0.04 | 2.07 | | | 0.49% | 0.09% | 0.01% | 0.00% | 0.00% | | 0.50 | 0.73 | 0.96 |
| Fax about Nat. Vet. | Poland | chicken (fat) | 1998 | 1998 | sum | 218 | 57 | 153 | 5 | 3 | 0 | 0 | 0 | | | | | | | | ok | | 0.02 | 0.04 | 2.07 | | | 0.52% | 0.10% | 0.01% | 0.00% | 0.00% | | 0.51 | 0.75 | 0.99 |
| Fax about Nat. Vet. | Poland | chicken (fat) | 1999 | 1999 | sum | 182 | 59 | 121 | 1 | 1 | 0 | 0 | 0 | | | | | | | | ok | | 0.01 | 0.03 | 2.07 | | | 0.32% | 0.05% | 0.01% | 0.00% | 0.00% | | 0.41 | 0.60 | 0.79 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | 0.0000 | 2.5000 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | | | | | | | 2.5000 | ##### | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Email R. Ashkenaz | Israel | poultry(fat) | 1990 | 1990 | sum | 27 | 27 | 0 | | | | | | | | | | | | ok | | | 0.00 | 0.01 | 2.07 | | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.10 | 0.15 | 0.20 |
| Email R. Ashkenaz | Israel | poultry(fat) | 1991 | 1991 | sum | 58 | 58 | 0 | | | | | | | | | | | | ok | | | 0.00 | 0.01 | 2.07 | | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.12 | 0.18 | 0.24 |
| Email R. Ashkenaz | Israel | poultry(fat) | 1992 | 1992 | sum | 48 | 48 | 0 | | | | | | | | | | | | ok | | | 0.00 | 0.01 | 2.07 | | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.12 | 0.17 | 0.23 |
| Email R. Ashkenaz | Israel | poultry(fat) | 1993 | 1993 | sum | 71 | 71 | 0 | | | | | | | | | | | | ok | | | 0.00 | 0.01 | 2.07 | | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.13 | 0.19 | 0.25 |
| Email R. Ashkenaz | Israel | poultry(fat) | 1994 | 1994 | sum | 86 | 86 | 0 | | | | | | | | | | | | ok | | | 0.00 | 0.01 | 2.07 | | | 0.01% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.12 | 0.18 | 0.24 |
| Email R. Ashkenaz | Israel | poultry(fat) | 1998 | 1998 | sum | 187 | 187 | 0 | | | | | | | | | | | | ok | | | 0.00 | 0.00 | 2.07 | | | 0.00% | 0.00% | 0.00% | 0.00% | 0.00% | | 0.04 | 0.06 | 0.08 |
| | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
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| | | | | | | | | | | | | | | | | | | | | | | | | critical level (mg/kg) | | | | 0.5 | 1 | 2 | 3 | 5 | percentile | 0.50% | 0.20% | 0.10% |
| | | | | | | | | | | | | | | | | | | | | | | | | | | average | | 0.08% | 0.02% | 0.00% | 0.00% | 0.00% | average | 0.10 | 0.15 | 0.19 |
| | | | | | | | | | | | | | | | | | | | | | | | | | weighted average | | | 0.07% | 0.01% | 0.00% | 0.00% | 0.00% | hted average | 0.19 | 0.24 | 0.29 |