THIODICARB (154)

EXPLANATION

Thiodicarb was reviewed for residues in 1985, and supervised field trial data for various crops were considered in 1987 and 1988. The 22nd Session of the CCPR decided to combine the MRLs for methomyl and thiodicarb into a single list (ALINORM 91/24 A, para. 126, p.21). The toxicology of thiodicarb was reviewed by the 2000 JMPR. The present review is a re-evaluation within the CCPR Periodic Review Programme.

The manufacturer submitted data on product chemistry, metabolism, environmental fate, analytical methods, storage stability, animal transfer and survey samples. The governments of Australia and Germany submitted label information.

IDENTITY

Chemical name

IUPAC: 3,7,9,13-tetramethyl-5,11-dioxa-2,8,14-trithia-4,7,9,12-tetra-azapentadeca-3,12-diene-6,10-dione

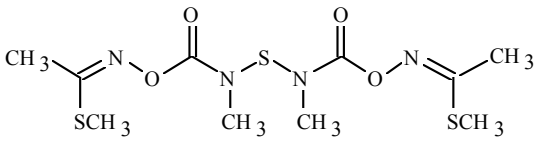
C.A. dimethyl *N*,*N*′-[thiobis[(methylimino)carbonyloxy]]bis(ethanimidothioate)

Chemical group: carbamates

BSI common name: thiodicarb

Chemical Abstracts registry No. (CAS No.): 59669-26-0

Structural formula



Thiodicarb

Empirical formula: $C_{10}H_{18}N_4O_4S_3$

Molecular weight: 354.5

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

		Reference
Appearance	White powder containing small aggregates	Robles and Bascou, 2000
Melting point:	172.6°C	Robles and Bascou, 2000
Decomposition point	184.7°C	Robles and Bascou, 2000
Octanol/water partition coefficient (shake-flask method)	log P_{OW} 1.62 at 25 °C	Cookinham, 1999a
Hydrolysis:		
PH 5 (buffered, 25°C)	DT_{50} = 78.4 days	Feung and Weisbach, 1991b
PH 7 (buffered, 25°C)	DT_{50} = 31.6 days	
PH 9 (buffered, 25°C)	DT_{50} = 0.48 days	
Photolysis:	DT_{50} = 7.6 days, (k=1.05 x 10^{-6} sec^{-1})	
	The UV/Vis spectrum showed that the molar absorption coefficient (ε) was below 10 $l.mol^{-1}.cm^{-1}$ when λ was ≥ 290nm. Quantum yield (Φ_{300}) study not conducted.	Feung and Blanton, 1987
Dissociation constant:	No dissociation observed	Cookinham, 1999b
Vapour pressure:	2.7 X 10^{-3} Pa at 25°C 2.7 X 10^{-3} Pa at 30°C 6.9 X 10^{-4} Pa at 35°C	Schweitzer, 1993
Henry's law constant	K = 4.31.10^{-2} $Pa.m^3.mol^{-1}$	Bascou, 1999
Solubility:		
Water		
deionised water (25°C)	22.19 µg/ml	
water buffered (pH 3, 25°C)	26.88 µg/ml	Cookinham, 1999c
water buffered (pH 5, 25°C)	29.83 µg/ml	
Water buffered (pH 7,25°C)	24.47 µg/ml	
Organic solvents		
dichloromethane	21 g/ml at 23°C	
hexane	0.000463 g/ml at 23°C	Lipscomb, 1987
methanol	0.32 g/ml at 23°C	
1,4 dioxane	0.65 g/ml at 25°C	
Relative density:	1.47 g/ml^{-1} at 20°C	Robles and Bascou, 2000

Technical material

Property	Results	Reference
Appearance	Off-white powder containing small aggregates	Robles and Bascou, 2000
Melting point:	167.1°C	Robles and Bascou, 2000
Decomposition point	171.6°C	
Purity		
Minimum purity	94.1%	Robles and Bascou, 2000
Main impurities	methomyl	
Relative density:	1.48 g/ml^{-1} at 20°C	Robles and Bascou, 2000
Surface tension	71.97 mN.m^{-1} at 20°C	Robles and Bascou, 2000
Stability:		
Thermal	Stable for 30 days at 25°C and 54°C	South and Pitts, 1993
Flammability	Not highly inflammable or autoflammable under the test conditions	Francois, 1999
Oxidising properties	The oxidising property test performed in air was positive, but no combustion was observed in an inert atmosphere.	Francois, 1999
Explosivity	No shock, friction or thermal sensibility to explosion	Francois, 1999
Estimated photochemical oxidative degradation	Thiodicarb was slightly degraded (<5%) after exposure to artificial sunlight (xenon lamp).	South and Pitts, 1993

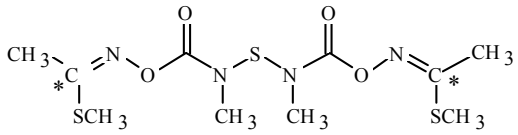
FORMULATIONS

The following list includes the major formulations developed for the use of thiodicarb on crops.

Formulation type	ai content	principal brand names
Suspension concentrate (SC)	300, 320, 375 g/l	Larvin®, Securex®, Souverain®
Flowable concentrated for seed treatment (FS)	350, 400, 500 g/l	Semevin®, Futur®
Water dispersible granule (WG)	800 g/kg	Larvin®, Relark®
Wettable powder (WP)	250, 750 g/kg	Larvin®
Granular bait (GB) and bait -ready to use- (RB)	20, 40 g/kg	Genesis®,Judge®, Skipper®
Ultra low volume liquid (UL)	350 g/l	Larvin®

METABOLISM AND ENVIRONMENTAL FATE

In all studies with [14C]thiodicarb the C=N carbons were labelled as shown below.



[^{14}C]thiodicarb.

* Denotes the ^{14}C carbons.

Animal metabolism

The metabolism of thiodicarb in laboratory rats and monkeys, and in domestic goats and chickens was reported.

Rats. Groups of 10 Sprague Dawley rats, 5 of each sex, were given single oral doses of 2 or 16 mg/kg bw thiodicarb (^{14}C, 87.5% purity, specific activity 23 mCi/mmol) by gavage (Hiles, 1987; Andrawes and Bailey, 1979; Huhtanen and Dorough, 1976).

Thiodicarb was rapidly and extensively absorbed. The time of maximum concentration (t_{max}) of ^{14}C in the blood was 1 to 4 hours after treatment at both doses and a high percentage of the applied dose was eliminated in the urine and respired gases. The results are shown in Table 1.

Table 1. Elimination of radioactivity in the respiratory gases, urine, and faeces of rats during 7 days after oral treatment at 2 and 16 mg/kg bw, average of 5 animals (Hiles, 1987).

Sample	Average cumulative % of applied dose			
	2 mg/kg bw		16 mg/kg bw	
	Male	Female	Male	Female
Urine	34	21	34	31
Faeces	5.3	9.2	4.4	4.2
Respired gases	38	50.5	42	43.1
Cage residue	0.35	0.88	0.46	3.6
Carcase	8.7	7.5	8.0	6.9
Total	86.3	89.0	89.7	88.6

Acetonitrile and carbon dioxide accounted for more than 99% of the radioactivity in the respired gases. Conversion of test material to CO_2 was rapid, with 58 to 74% eliminated in the first 6 hours and 89 to 95% within 24 hours. Elimination of acetonitrile was slower, 9 to 16% during the first 6 hours and 67 to 75% after 24 hours.

Residues in the carcase and tissues were 7-9% of the applied dose after seven days, and were highest in the red blood cells.

Acetonitrile was identified as a significant metabolite in urine. Minor amounts (1% or less of the urinary radioactivity) were identified in the involatile organosoluble fraction as methomyl, methomyl oxime, methomyl sulfoxide, and methomyl oxime sulfoxide. Water-soluble metabolites accounted for more than 86% of the radioactivity in the urine.

Monkeys. The absorption, distribution, metabolism and excretion of [^{14}C]thiodicarb (radiochemical purity >97%, specific activity 17.72 mCi/mM) were studied in four male cynomolgus monkeys aged 1 to 2.5 years placed in individual metabolic chambers immediately after being given single oral doses at a nominal rate of 5 mg/kg (Hawkins *et al.,* 1993). Urine was collected 6 and 24 hours after dosing and every 24 hours for 7 days (168 hours). Faeces, cage debris, and cage washings were collected

daily. Expired gases were trapped and analysed after 6, 24 and 48 hours. After one week the monkeys were killed and lungs, brain, gastrointestinal tract, liver, fat, muscle and skin sampled for the characterization and quantification of radiolabelled compounds. Approximately 60% of the radioactivity was excreted or expired during the first 24 hours. Over the 7-day period, an average of 31% of the administered dose was excreted in the urine (including cage wash), and only 4.6% in the faeces (Table 2).

4.7% of the dose was measured in the tissues. In the 48 hours after dosing when volatiles were collected an average of 28% of the administered dose was eliminated as $^{14}CO_2$, and 8.6% as [^{14}C]acetonitrile. The overall recovery of ^{14}C was 74-81%. Presumably the missing material could be attributed to volatiles exhaled after 48 hours.

Table 2. Distribution of radioactivity in monkeys given single oral doses of thiodicarb (5 mg/kg) expressed as a percentage of the administered dose (Hawkins *et al.*, 1993).

Sample, time after dosing (h)		Animal number				Mean ± SD
		Q168	R100	R69	R189	
Expired air, acetonitrile	0-6	2.0	1.9	1.7	2.1	1.9 ± 0.18
	6-24	3.8	3.0	3.4	6.0	4.0 ± 1.6
	24-48	2.9	1.7	1.5	4.2	2.6 ± 1.2
	Total	8.8	6.6	6.6	12	8.6 ± 2.7
Expired air, $^{14}CO_2$	0-6	14	23	16	17	17 ± 4.0
	6-24	12	5.9	6.9	10	8.6 ± 2.7
	24-48	2.2	1.1	2.5	1.2	1.8 ± 0.71
	Total	27	30	25	29	28 ± 2.1
Total expired air		36	37	32	41	36 ± 3.8
Urine	0-6	15	29	22	19	21 ± 5.8
	6-24	5.1	3.7	4.7	6.2	5.0 ± 1.0
	24-48	1.4	1.2	1.6	2.2	1.61 ± 0.44
	48-72	0.60	0.51	0.33	0.41	0.46 ± 0.12
	72-96	0.30	0.28	0.27	0.19	0.26 ± 0.05
	96-120	0.24	0.19	0.15	0.15	0.18 ± 0.04
	120-144	0.17	0.14	0.13	0.15	0.15 ± 0.02
	144-168	0.14	0.11	0.10	0.12	0.12 ± 0.02
	Total urine	23	35	29	28	29 ± 4.9
Cage washes		4.1	0.88	3.1	2.0	2.5 ± 1.4
Total faeces		4.8	4.5	5.4	3.8	4.6 ± 0.63
TOTAL		67.9	77.3	69.5	74.8	72

Metabolic degradation was extensive. At least 18 metabolites were found in the urine, none of which individually accounted for more than 5% of the applied dose; the metabolites identified using co-chromatography in two different systems were acetonitrile (1.2-2.9% of the dose), acetic acid (0.4-0.9%), and acetamide (0.8-1.0%).

No radioactive components in the urine corresponded to thiodicarb or its major degradation product methomyl. Chromatography also confirmed the absence of *syn*- and *anti*-forms of methomyl oxime (MHTA), methomyl sulfoxide, and methomyl oxime sulfoxide. Following treatment with *E.coli* ß-glucuronidase, radioactivity associated with the major polar component decreased from 11.9% of the applied dose to 4.2%, and that associated with eight components increased, including those corresponding to acetic acid (0.5-2.1% of the dose) and acetonitrile (3.2-3.8%).

Concentrations in the tissues were highest in the liver (0.8-1.5 µg thiodicarb equivalents/g) and fat (0.4-0.6 µg equivalents/g), and 0.1-0.3 µg equivalents/g in other tissues. Major polar metabolites were detected in extracts of whole blood (0.09-0.16 µg equivalents/g) and liver (0.5-1.1

µg equivalents/g). In liver, a component corresponding to acetic acid (0.12-0.1 µg equivalents/g) was confirmed by both reverse-phase and ion-exchange HPLC, but was not found in blood. No radioactive components in whole blood or liver corresponded to thiodicarb, the *syn*- or *anti*-forms of methomyl, methomyl oxime, methomyl oxime sulfoxide, acetonitrile, or acetamide.

Hens. The metabolic fate of [^{14}C]thiodicarb (21.35 mCi/mM, 1.37 x 10^5 dpm/ µg) was studied in laying hens. Groups consisting of 3 sub-groups of 3 hens (9 total per group) were dosed orally for 21 days at 15, 29, or 102 ppm in the diet (specific activity 1.37 x 10^5 dpm/µg, 9.15 x 10^4 dpm/µg, and 2.74 x 10^4 dpm/µg respectively) (Andrawes and Bailey, 1980) Faeces and eggs were analysed during the treatment period and for seven days afterwards. Tissue samples were taken for analysis 6 hours, 3 days and 7 days after the last dose (1 hen from each sub-group was killed at each interval).

The total radioactive residue (TRR) was measured by combustion and liquid scintillation counting (LSC). The level of detection was 0.01 mg/kg. Eggs and tissues were extracted with acetone-water and separated into volatile, organosoluble and water-soluble residue fractions. The volatile fraction was analysed by distillation and gas-liquid chromatography (GLC). Gel permeation was used to separate metabolites in the organosoluble and water-soluble fractions. The metabolites were identified and quantified by two-dimensional TLC, radio-autography and LSC.

Radioactivity reached a plateau within one day in the faeces, two days in egg whites and ten days in the yolks of the high-dose group. Levels of radioactivity declined during the withdrawal period. The results are shown in Tables 3 and 4.

Table 3. Distribution of radioactivity in the faeces and eggs of laying hens dosed with [^{14}C]thiodicarb for 21 days (Andrawes and Bailey, 1980).

Day	mg/kg [^{14}C]thiodicarb equivalents								
	15 ppm			29 ppm			102 ppm		
	Faeces	Yolk	White	Faeces	Yolk	White	Faeces	Yolk	White
1	1.6	--	--	3.2	--	--	14.	--	--
2	1.3	--	--	3.7	--	--	15	1.9	2.0
3	1.4	--	--	4.4	--	--	15	--	--
4	--	--	--	--	--	--	--	4.5	2.0
6	--	--	--	--	--	--	--	10.2	1.5
7	1.2	--	--	5.0	--	--	19	--	--
8	--	--	--	--	--	--	--	13	1.7
10	--	--	--	--	--	--	--	14	1.5
12	--	1.4	0.19	--	4.0	0.58	--	15	2.0
14	1.5	1.3	0.23	6.1	4.6	0.77	30	15.	2.0
21	1.4	1.5	0.21	4.5	4.6	0.59	13	13	1.6
Withdrawal									
1	0.36	--	--	2.0	--	--	10.	14	1.3
2	0.16	--	--	0.53	--	--	1.5	--	--
3	0.08	--	--	0.25	--	--	1.1	10.	0.21
5	--	--	--	--	--	--	--	6.5	0.11
7	0.06	--	--	0.18	--	--	0.37	2.2	0.05

Table 4. Distribution of radioactivity in the tissues of hens dosed with [^{14}C]thiodicarb in the feed for 21 days following a 6 hours, 3 days, or 7 days withdrawal period (Andrawes and Bailery, 1980).

Sample	mg/kg [^{14}C]thiodicarb equivalents at slaughter								
	15 ppm			29 ppm			102 ppm		
	6 h	3 days	7 days	6 h	3 days	7 days	6 h	3 days	7 days
Breast muscle	0.46	0.19	0.14	1.0	0.53	0.39	3.4	1.9	1.3
Thigh muscle	0.53	0.21	0.25	1.2	0.79	0.63	4.2	2.7	1.9
Leg muscle	0.53	0.30	0.24	1.2	0.76	0.59	4.2	2.9	1.6
Subcutaneous fat	0.97	0.94	0.52	2.5	1.89	1.8	5.8	8.0	5.7
Abdominal fat	1.3	0.77	0.58	2.9	1.89	1.6	6.6	7.5	6.1
Skin	0.57	0.38	0.26	1.6	1.05	0.71	4.3	4.0	1.6
Heart	0.71	0.25	0.21	1.6	0.82	0.61	3.7	3.0	1.5
Kidney	1.4	0.68	0.27	3.4	1.74	0.89	8.5	4.7	1.7
Gizzard	0.66	0.26	0.16	1.3	0.70	0.51	3.0	2.7	1.1
Liver	1.5	0.41	0.27	3.5	1.08	0.63	10.	4.6	1.6
Plasma	0.35	0.08	0.04	1.2	0.21	0.11	3.9	0.86	0.32
RBCs	2.3	1.4	0.67	7.5	4.45	2.7	20.	18.	8.4

The absence of thiodicarb and its potential metabolites methomyl, methomyl oxime, methomyl oxime sulfoxide and methomyl methylol (hydroxymethyl-methomyl, see Figure 2) was confirmed in all samples. Low levels of acetonitrile (volatile) and acetamide (water-soluble) were detected in eggs (Table 5). In addition, some radioactivity was associated with lipids and other natural products through incorporation of $^{14}CO_2$ (e.g. cholesterol and lecithin).

Table 5. Characterization of radioactivity in the eggs of hens dosed with [^{14}C]thiodicarb in the diet (Andrawes and Bailey, 1980).

Feeding level (ppm)	Metabolite/fraction	^{14}C (mg/kg as compound where applicable)			
		Day 14		Day 21	
		Yolk	White	Yolk	White
15	Acetonitrile	0.012	0.037	0.024	0.024
	Acetamide	NF	0.007	NF	0.006
	Lipids*	1.2	NA	1.2	NA
	Unextracted*	0.20	0.049	0.22	0.044
29	Acetonitrile	0.083	0.14	0.076	0.081
	Acetamide	NF	0.020	NF	0.022
	Lipids*	3.8	NA	3.5	NA
	Unextracted*	0.58	0.13	0.57	0.15
102	Acetonitrile	0.16	0.27	0.15	0.21
	Acetamide	NF	0.055	NF	0.029
	Lipids*	10. (68% of TRR)	NA	10. (77% of TRR)	NA
	Unextracted*	1.9	0.70	1.6	0.38

NF: not found
NA: not applicable
* ^{14}C as thiodicarb

Extractable radioactivity in the tissues accounted for about 50% of the total 6 hours after the last dose. Low levels of acetonitrile and acetamide were found in the liver and muscle but not in abdominal fat (Table 6).

Table 6. Characterization of radioactivity in the liver, muscle and fat of hens dosed with [^{14}C]thiodicarb (Andrawes and Bailey, 1980).

Metabolite or fraction	^{14}C (mg/kg as thiodicarb) at intervals after 21 days dosing								
	102 ppm dose			29 ppm dose			15 ppm dose		
	6 h	3 days	7 days	6 h	3 days	7 days	6 h	3 days	7 days
Liver									
Acetonitrile	0.44	0.076	0.018	0.134	0.017	0.005	0.047	0.007	0.003
Acetamide[1]	0.051	NC	NC	NA	NA	NA	NA	NA	NA
Unknown lipids	2.5	0.84	0.53	NA	NA	NA	NA	NA	NA
Unknown polar[2]	0.84	0.18	0.043	NA	NA	NA	NA	NA	NA
Involatile	NA	NA	NA	1.2	0.33	0.22	0.43	0.14	0.054
Unextractable	4.7	2.0	0.20	1.5	0.68	0.41	0.84	0.2	0.11
Recovery (% of TRR)	85%								
Breast muscle									
Acetonitrile	0.27	0.011	0.001	0.1	0.017	0.005	0.030	0.002	Trace
Acetamide[1]	0.009	NC	NC	NA	NA	NA	NA	NA	NA
Unknown lipids	0.44	0.45	0.22	NA	NA	NA	NA	NA	NA
Unknown polars[2]	0.062	0.052	0.034	NA	NA	NA	NA	NA	NA
Involatile	NA	NA	NA	1.257	0.33	0.220	0.082	0.047	0.035
Unextractable	1.3	1.4	1.00	0.52	0.40	0.23	0.21	0	0.11
Recovery (% of TRR)	61%								
Thigh muscle									
Acetonitrile	0.22	0.009	0.005	0.067	0.003	Trace	0.035	0.002	Trace
Acetamide[1]	0.006	NC	NC	NA	NA	NA	NA	NA	NA
Unknown lipids	2.5	0.84	0.53	NA	NA	NA	NA	NA	NA
Unknown polars[2]	0.84	0.048	0.042	NA	NA	NA	NA	NA	NA
Involatile	NA	NA	NA	0.36	0.22	0.21	0.15	0.084	0.099
Unextractable	1.2	1.1	0.95	0.64	0.43	0.35	0.24	0.16	0.16
Recovery (% of TRR)	113%								
Abdominal fat									
Acetonitrile	ND	ND	ND	NA	NA	NA	NA	NA	NA
Acetamide[1]	ND	ND	ND	NA	NA	NA	NA	NA	NA
Unknown lipids	5.6	12.	9.4	NA	NA	NA	NA	NA	NA
Unknown polars[2]	ND	ND	ND	NA	NA	NA	NA	NA	NA
Unextractable	0.16	0.26	0.31	NA	NA	NA	NA	NA	NA
Recovery (% of TRR)	87%								

[1] Only tissue samples at 6 h contained sufficient total water-soluble radioactivity to allow separation and confirmation of absolute acetamide levels
[2] Fraction containing any acetamide present in 3 and 7 day samples
NC: level not confirmed
NA: not analysed
ND: not detected

Goats. The metabolic fate of [^{14}C]thiodicarb (specific activity 17.72 mCi/mmol) was studied in two lactating goats after 7 days of dosing at levels equivalent to 200 and 300 ppm in the diet (Hanlon and Norris, 1991; Hanlon, 1994).

The goats were placed in indirect respiration chambers for the collection of volatiles for approximately 10 hours on the 6th day. Faeces, urine and milk were collected twice daily during the treatment period. Blood, gut content and tissue samples were taken for analysis within 18 hours of the last dose. The nominal level of detection of ^{14}C in combustion samples was 0.01 mg/kg as thiodicarb and for LSC measurement 0.002 μg.

Urine samples were analysed directly by HPLC and TLC. Milk samples were extracted with acetonitrile and hexane, stomach contents and faeces with methanol, and selected tissues by water and then by methanol. Extracted solids were treated with protease to liberate additional residues, and other

fractions were treated with various enzymes and/or base as appropriate. Extracts were analysed by a combination of chromatographic techniques including HPLC and TLC.

Trapped volatile compounds were acetonitrile and carbon dioxide. Volatile production during 10 hours was extrapolated to cover the entire dosing period, and the total ^{14}C residues associated with respiration were estimated to be 21 and 23% of the total dose for goats 1 and 2.

Table 7. Distribution of radioactivity in goats dosed with [^{14}C]thiodicarb for 7 days (Hanlon and Norris, 1991; Hanlon, 1994).

	^{14}C, % of total dose					
	Tissues[1]	Milk	Urine	Faeces	Volatiles[2]	Total
Goat 1	8.6	6.4	8.9	7.7	21.	52
Goat 2	14.	3.1	5.8	3.5	23.	50
Average	11.	4.7	7.3	5.6	22.	51

[1] including blood and gut contents
[2] Extrapolated from 10-h period.

Total radioactivity appeared to reach a plateau within three days in the faeces, urine and milk. The levels in the milk and tissues are shown in Table 8.

Table 8. Distribution of residues of [^{14}C]thiodicarb in goats dosed for 7 days (Hanlon and Norris, 1991; Hanlon, 1994).

Sample	Cumulative % of applied dose			^{14}C as thiodicarb (mg/kg)		
	Goat 1	Goat 2	Mean	Goat 1	Goat 2	Mean
Milk	6.4	3.1	4.7	15*	20*	17.*
Liver	0.93	1.1	1.0	25.	23.	24.
Kidney	0.08	0.11	0.10	13	14	13.
Muscle	3.3	3.9	3.6	4.3	4.2	4.3
Fat	0.66	0.26	0.46	1.4	0.45	0.91
Blood	1.6	2.0	1.8	10.	11	11

* Maximum level over the 7 days dosing

Gut contents contained acetonitrile (15 and 24% of the TRR), acetamide (5.8 and 7.1%), thiodicarb (6.3 and 5.5%) and methomyl (3.2 and 7.5%). Faeces from early and late periods were found to contain thiodicarb and methomyl as the main radioactive residues. In urine only acetonitrile and acetamide were identified.

Goat 2 became ill in the latter part of the study with resultant decline in urine, faeces and milk outputs. Because of this the tissues from goat 2 were used mainly for method development, and definitive work was done on tissues from goat 1. Between 70 and 91% of the radioactive residues were extractable from liver, kidney and muscle with water. No thiodicarb, methomyl, or methomyl metabolites were found in any of these tissues. Acetonitrile and acetamide were detected in liver, kidney and muscle; one of the radioactive components was an unknown water-soluble polar compound which was converted to acetic acid by alkaline hydrolysis (Table 9).

Table 9. Residues in the edible tissues of goat 1 (dosed at 200 ppm dietary equivalent) as a percentage of the total administered ^{14}C and mg/kg thiodicarb equivalents (Hanlon and Norris, 1991; Hanlon, 1994).

Metabolite	Liver		Kidney		Muscle	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Acetonitrile	32.	8.2	23	2.9	72	3.1
Acetamide	5.9	1.5	10.	1.3	14.	0.62
Acetic acid*	57.	14.	43.	5.4	11.	0.49
Total	95.	24.	76.	9.6	97.	4.2

*From alkaline hydrolysis of an unknown polar compound

Most of the TRR was extracted from fat and milk samples with acetonitrile and hexane. The acetonitrile extracts were analysed by HPLC and the ^{14}C residues in the hexane were characterized by saponification. Post-extraction solids were also examined further in both cases (Tables 10 and 11).

Table 10. Residues in the fat of two goats as a percentage of total ^{14}C-residues and mg/kg thiodicarb equivalents.

Metabolite	Goat 1		Goat 2	
	% of TRR	mg/kg	% of TRR	mg/kg
Acetonitrile	9.9	0.14	11.1	0.05
Acetamide*	6.0	0.08	25	0.11
Non-saponifiable fatty acids	1.3	0.02	1.0	0.01
Saponifiable fatty acids	46	0.63	15	0.07
Other saponifiable lipids	27	0.37	26	0.12
Total	90	1.2	79	0.36

*Acetamide may be due in part to hydrolysis of acetonitrile during concentration of extracts

Table 11. Residues in milk as % of total ^{14}C residues and mg/kg thiodicarb equivalents.

Metabolite	Goat 1		Goat 2	
	% of TRR	mg/kg	% of TRR	mg/kg
Acetonitrile	29	4.1	18	1.8
Acetamide	0.0	0.0	4.0	0.41
Lactose	11	1.6	23.	2.3
Acetic acid	0.0	0.0	4.0	0.41
Non-saponifiable fatty acids	0.6	0.09	0.3	0.03
Saponifiable fatty acids	18	2.6	8.2	0.84
Other saponifiable lipids	14	2.6	9.7	1.0
Losses*	8.4	1.2	7.8	0.80
Solids (bound)	1.0	0.14	2.5	0.25
Total	82	12	77	7.9

*Losses during transfers, etc., not volatility

In the supplementary report (Hanlon, 1994), saponifiable fatty acids in hexane extracts of the milk and fat were identified as [^{14}C]palmitic and [^{14}C]myristic acids and a water-soluble saponifiable lipid in milk yielded [^{14}C]glycerol. In addition, material retained on the column from HPLC of an aqueous fraction of liver and kidney was further characterized as amino acids or proteins, which release acetonitrile and acetic acid upon strong basic hydrolysis. The combined results of the original and supplementary reports show identification or characterization of acceptable percentages of the TRR in liver (>90%), kidney (>75%), muscle (>95%), fat (>63%) and milk (>69%).

An older study on lactating cows (Feung *et al.,* 1980) corroborates the results of the goat study.

In ruminants and poultry the transfer and distribution of radioactivity was the result of thiodicarb metabolism to acetonitrile and carbon dioxide followed by incorporation of ^{14}C fragments into natural pathways. No thiodicarb, methomyl, or intact methomyl metabolites were detected in any edible tissues, eggs, or milk, even after feeding at dietary concentrations as high as 100 ppm in poultry or 290 ppm in goats.

The results of animal metabolism studies are consistent with thiodicarb being extensively metabolized and almost no residual material was retained in any species. Thiodicarb is rapidly degraded to *syn*- and *anti*-methomyl, and subsequently to CO_2 and acetonitrile, which are primarily eliminated by respiration and in the urine. A portion of the CO_2 fraction can be incorporated to natural products. Acetonitrile which is not eliminated can be further converted to CO_2, acetic acid and acetamide, all of which can be subsequently incorporated into natural products. There is no evidence of direct transfer of carbamate residues to edible substrates in lactating goats, even when animals are administered exaggerated levels (200-300 ppm). The proposed metabolic pathways for thiodicarb in mammals and poultry are shown in Figure 1.

Plant metabolism

The metabolism in plants was studied using [^{14}C]thiodicarb in root crops (potato and carrot), a fruiting vegetable (tomato), cereal grains (wheat, maize and sweet corn) and oilseed crops (cotton, soya beans and peanuts).

Potatoes. The absorption, translocation and metabolism of thiodicarb in foliage and tubers following the application of radiolabelled material to the upper leaf surfaces was reported by Feung and Chancey (1979c). [^{14}C]Thiodicarb (specific activity about 41,000 dpm/µg) was spread on the top surfaces of leaves of 6 greenhouse-reared plants approximately 60 cm high at a rate approximating 1.12 kg ai/ha. The plants were watered daily and harvested when the tubers were of good size. The time from application to harvest was not specified. Harvested plants were separated into foliage and tubers and stored in a freezer until analysis.

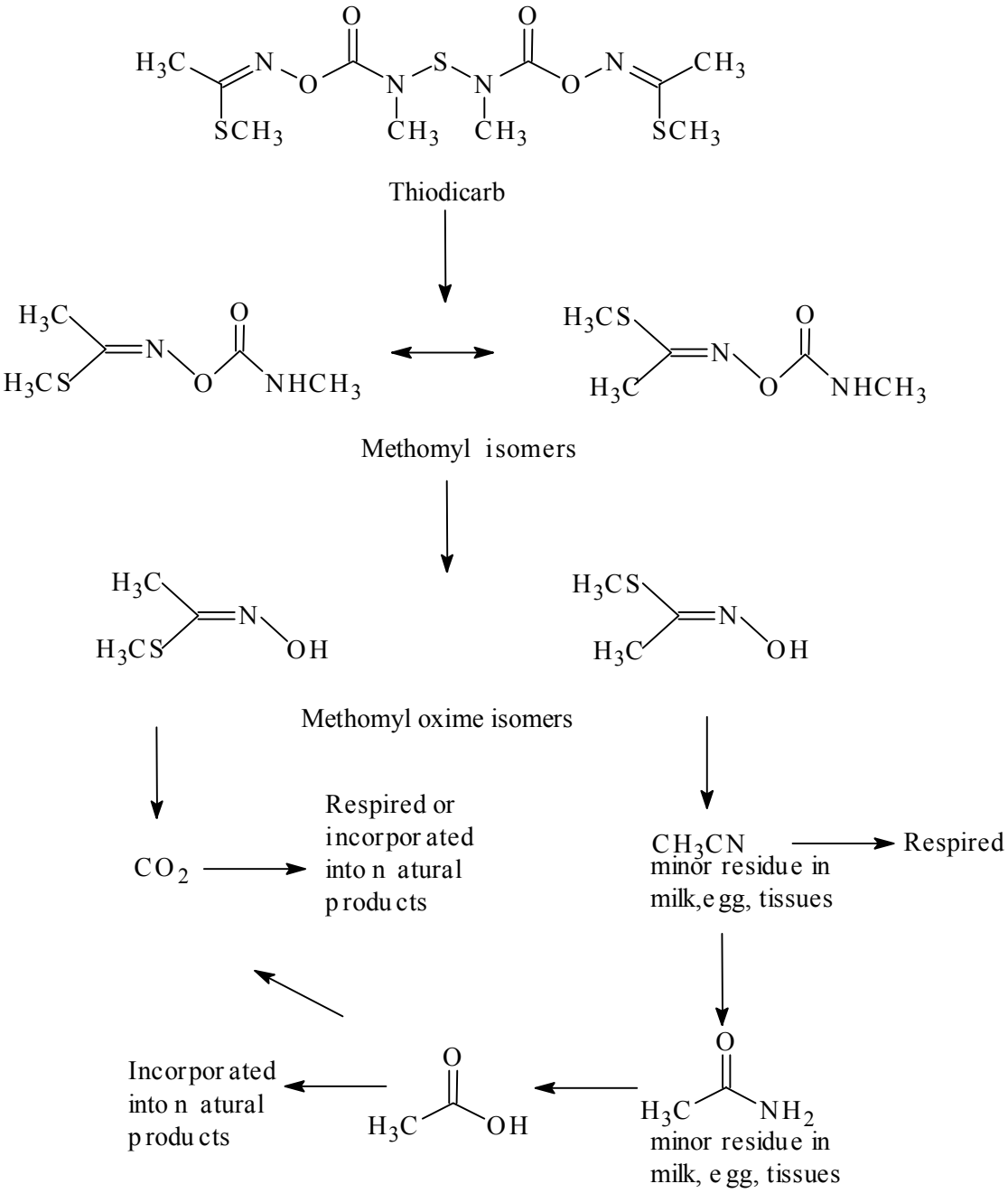
The radioactive residue in the foliage accounted for 59% of the applied dose, but in tubers for only 0.12%. The remaining radioactivity (approximately 40%) is presumed to represent the amount of volatile $^{14}CO_2$ and $^{14}CH_3CN$ lost from the leaf surface following thiodicarb degradation. Isolated metabolites were determined by two-dimensional thin-layer chromatography.

he very low level of ^{14}C found in the tubers was indicative of the very low potential for translocation of thiodicarb from leaves to tubers. The extractable radioactivity (approximately 0.024 mg/kg as thiodicarb, or 50% of the total radioactive residue in the tubers) was almost entirely water-soluble. A single polar metabolite was seen in the organosoluble fraction (representing less than 5% of the radioactivity in the tuber, <<0.01 mg/kg) that did not compare with any known standard on TLC.

Isolation of metabolites from the tuber was not possible owing to the extremely low level of radioactivity in the acetonitrile extract following enzymatic treatment of the water-soluble fraction. The low level of radioactivity arose from translocation from the leaves and/or incorporation of $^{14}CO_2$ released from the parent compound into naturally occurring plant components.

pproximately 53% of the radioactivity in foliage was organosoluble. Nine components were observed on TLC, four of which were identified as thiodicarb (major), methomyl and methomyl methylol (minor), and methomyl oxime (trace).

Figure 1. Metabolic fate of thiodicarb in animals.



Note: methomyl oxime was not observed in tissues, milk or eggs.

The remaining 5 components accounted for only 0.54% of the radioactivity in the fraction and did not match any of the standards on TLC. A small percentage of the foliage radioactivity (4%) was found in the water-soluble fraction. After enzymolysis, three aglycones, methomyl, methomyl oxime and methomyl methylol, were seen on TLC.

Carrots. Feung and Chancey (1978c) spread approximately 10 μCi of [^{14}C]thiodicarb on the top surface of the leaves of six-week old greenhouse plants with a microsyringe. The plants were manually watered daily and harvested 28 days later, separated into aerial portions and roots, and then stored in a freezer until analysis.

Appreciable activity was found in the aerial portions, but only traces in the root extracts. High recoveries (90.47% of the applied) indicated little volatilisation on the leaves. The low activity in the roots (0.06% of that applied) was attributed to poor translocation of thiodicarb or its metabolites from the leaves to the roots. It did not allow any isolation or determination of metabolites.

In the foliage, four of nine organosoluble metabolites were tentatively identified by two-dimensional co-chromatography on TLC as unchanged thiodicarb (79% of the applied radioactivity), methomyl (8.2%), *N*-hydroxymethyl methomyl (0.18%) and methomyl oxime (0.09%). The remaining metabolites did not match the authentic standards.

TLC of the water-soluble fraction before enzymolysis showed a single radioactive spot remaining at the origin. After enzymolysis, eight aglycones were resolvable. Three were identified by two-dimensional TLC as methomyl (0.29% of the applied radioactivity), *N*-hydroxymethyl-methomyl (0.02%) and methomyl oxime (0.01%). The remaining metabolites did not behave like any of the authentic standards on TLC in any of the solvents used. Methomyl and methomyl oxime were verified by mass spectrometry.

Tomatoes. The disposition and metabolism of [^{14}C]thiodicarb was investigated in foliage and fruit by Feung and Chancey (1979b).

A solution of [^{14}C]thiodicarb (about 6000 mg/kg, specific activity approximately 41,000 dpm/μg) was spread on the top surfaces of tomato leaves at the time of flower cluster initiation at a rate approximating 1.12 kg ai/ha. The plants were maintained in a greenhouse until mature, when the fruits were harvested, weighed and stored in the freezer until analysis. The foliage was also collected and frozen until analysis.

Frozen tissues from harvested plants were ground in a blender with acetonitrile/acetone/water. The homogenate was centrifuged and filtered and the plant residue washed several times with mixed solvent and air-dried at room temperature. The radioactivity of each fraction was determined by liquid scintillation counting. The filtrate was diluted with chloroform and partitioned into organosoluble and water-soluble fractions. The organosoluble fraction was concentrated in a rotary evaporator. The aqueous fraction was concentrated and a portion subjected to enzyme hydrolysis, extracted with acetonitrile/chloroform and separated into aglycone and aqueous fractions. The aqueous fraction was then refluxed with 1N HCl and partitioned into organic and aqueous fractions.

Approximately 50% of the radioactivity was lost from the leaf surface as volatiles, presumably CO_2 and CH_3CN. Appreciable radioactivity was detected in leaves (approximately 49% of the applied dose). Radioactivity measurements indicated very low activity in tomato fruit (0.45% of the applied dose), evidence of low absorption and translocation. Although metabolites in the fruit were fractionated and isolated characterization was not possible owing to the very low radioactivity. The low level of radioactivity in the fruit was thought to be the result of incorporation of $^{14}CO_2$, released from degraded metabolites, into naturally occurring plant components.

In total, organic and aqueous extracts of the foliage accounted for about 45% of the applied ^{14}C, with unextractable residues amounting to a further 4%. Identification of metabolites was by comparison against authentic standards using two-dimensional TLC. The results are shown in Table 12.

Table 12. Compounds identified in tomato leaves (Feung and Chancey, 1979b).

Compound	% of total recovered radioactivity in leaves fraction[1]
Organosoluble	
– thiodicarb	78.
– methomyl	5.9
– methomyl oxime	0.18
– others	<3
Water-soluble, conjugates of	
– methomyl	<1
– methomyl oxime	0.16
– methomyl methylol	<1
Unextractable	0.8

[1] Represents 49% of applied radioactivity

Four organosoluble compounds were identified (96% of organic fraction, 84% of the total radioactivity in the leaves): thiodicarb (major), methomyl (minor), methomyl oxime (very minor) and methomyl methylol (trace). The remaining 5 organosoluble metabolites accounted for approximately 4% of the fraction and did not match any of the standards on TLC. Three of the water-soluble components (12% of the fraction, 0.5% of the total radioactivity in leaves) were identified as conjugates of methomyl, methomyl oxime and methomyl methylol. The remaining unidentified water-soluble components accounted for 88% of the fraction but only 0.8% of the total radioactivity in the leaves.

Feung and Jeffs (1986) investigated the metabolism of [^{14}C]thiodicarb in tomato foliage and fruit following application of radiolabelled material to both tomatoes and foliage. Three experiments were conducted, each with two plants treated 6 times at 6-day intervals at a rate approximating 1.12 kg ai/ha/treatment.

In experiment 1, a solution of [^{14}C]thiodicarb was spread on the top surfaces of tomato leaves, equivalent to a season rate of 6.72 kg ai/ha. The plants were maintained in a greenhouse for 6 days after the last treatment, when the fruits and foliage were harvested, weighed, chopped and stored in the freezer until analysis.

In experiment 2, only the fruits were treated, harvested 6 days after the last treatment and washed with 70% methanol to remove surface compounds. The washed fruit were weighed, chopped and stored in the freezer until analysis. The methanol rinse was concentrated and analysed by TLC.

In experiment 3, both fruits and foliage were treated and harvested 6 days after the last treatment. The tomatoes were processed, stored and analysed in the same way as in experiment 2. The foliage was stored with the fruit in the freezer.

Samples of fruit and foliage from all experiments were blended with methanol and the homogenate filtered; this was repeated and the residue cake washed twice with methanol and dried. Radioactivity was measured in the fractions. Residues were extracted from fruit and foliage as in previous studies, separating organosoluble and water-soluble components.

Approximately 57-63% of the applied radioactivity was lost as volatiles in the three experiments (presumably as CO_2 and CH_3CN). The results are shown in Table 13. The foliar applications resulted in very little residue in the fruit and fruit applications resulted in very little residue in the foliage.

Table 13. Distribution of the applied radioactivity in tomato fruit and foliage (Feung and Jeffs, 1986).

Sample	^{14}C, % of applied					
	Experiment 1, foliage treated		Experiment 2, fruit treated		Experiment 3, both fruit and foliage treated	
	Extractable	Unextractable	Extractable	Unextractable	Extractable	Unextractable
Residues rinsed from fruit surface	NA	NA	29.7 25.1	NA	2.91 3.30	NA
Residues in foliage	33.3 34.8	8.61 8.12	0.35 0.33	0.10 0.45	26.6 23.1	6.28 7.17
Residues in fruit	0.35 0.12	0.33 0.55	8.47 6.81	1.96 1.83	1.06 1.63	0.66 0.57
Total involatile	42.6 43.6		40.6 34.5		37.5 35.7	

NA: not applicable

Residues rinsed from the surface of fruits in experiments 2 and 3 were identified by two-dimensional TLC of the concentrated rinse solutions; ten radioactive compounds were observed. Three were identified as thiodicarb (73 to 83% of the rinsed fraction), methomyl (3 to 5%) and methomyl methylol (<1 to 4%). The remaining 6 metabolites, representing 13 to 17% of the rinsed fraction, did not match authentic standards on TLC. No acetamide was found in the extracts rinsed from the fruit surface (limit of detection estimated to be 0.03 to 0.05 mg/kg).

Compounds in the bulk samples of fruit and foliage were identified by two-dimensional TLC and mass spectrometry. In the fruit extracts from experiment 2 (which contained the highest total radioactive residue in fruit), methomyl (1% of the applied dose) and methomyl oxime (0.43% of the applied dose) were identified in the organosoluble fraction. In the foliage from experiments 1 and 3, 11 radioactive compounds were found in the organosoluble fraction; three were identified as thiodicarb (major), methomyl (minor to major) and methomyl methylol (minor to trace). The remaining metabolites accounted for less than 16% of the radioactivity in the leaves and did not match any of the standards on TLC. In both fruit and foliage, the water-soluble metabolites were determined to be natural products resulting from the incorporation of radioactive thiodicarb fragments (e.g. CO_2). No acetamide was found in either fruit or foliage.

Sweet corn and wheat. Approximately 5 µCi of [^{14}C]thiodicarb in 30 µl of acetonitrile/acetone/water (0.5:1.5:1.0) was injected into the stems of 3-week-old greenhouse-grown sweet corn and wheat plants which were maintained in a greenhouse for 7 days, then harvested, weighed and stored in the freezer until analysis (Feung and Chancey, 1977b). Samples were extracted and analysed as previously described.

Approximately 26% and 53% of the radioactivity was lost from sweet corn and wheat respectively, presumably as volatiles such as CO_2 and CH_3CN. In sweet corn, organosoluble plus water-soluble fractions accounted for about 70% of the applied ^{14}C, with unextractable residues amounting to a further 4%. In wheat the extracts accounted for about 34%, and the unextractable residues for about 14%, of the applied dose.

Compounds were identified by comparison with authentic standards using two-dimensional TLC and mass spectrometry. Four organosoluble compounds were identified in both sweet corn and wheat (accounting for >95% of the fraction in each case): thiodicarb and methomyl (major in both), and methomyl oxime and methomyl sulfoxide (very minor in both). The remaining 5 organosoluble metabolites did not match any of the standards on TLC. Three of the water-soluble components were identified as conjugates of methomyl, methomyl oxime and methomyl sulfoxide (all <1% in both plants), with most of organo-insoluble radioactivity unchanged by enzymatic hydrolysis.

Feung and Blanton (1986a) investigated the metabolism of [^{14}C]thiodicarb in sweet corn following application of radiolabelled material to surfaces of the foliage, particularly to verify that acetamide is not a metabolite of thiodicarb in sweet corn. [^{14}C]thiodicarb (224 mg, 18 mCi) was painted onto the foliage of 8 plants. In experiment 1 the application was made to leaves and ears, including silks, and in experiment 2 to the leaves only. A total of 4 treatments were made at 7-day intervals, representing a seasonal use rate of 4.48 kg ai/ha.

The plants were maintained in a greenhouse for 7 days after the last treatment, when the ears (kernels and cobs) and the foliage, including ear sheath and silk, were harvested, weighed, chopped and stored in the freezer until analysis. Samples were blended twice with methanol and the homogenate filtered, and the residue cake washed twice with methanol and dried. Radioactivity was measured in the fractions. Residues were extracted by a procedure which separated organosoluble fractions, precipitates (presumed natural products), pigments (foliage only) and unextractable components.

Approximately 64-67% of the applied radioactivity was presumably lost as CO_2 and CH_3CN during the seven days after the last application. There was very little residue penetration through the foliage; only minute quantities of radioactivity were found in kernels and cobs (Table 14).

Table 14. Distribution of applied radioactivity in sweet corn foliage, kernels and cobs (Feung and Blanton, 1986)**.**

Sample	^{14}C, % of applied					
	Experiment 1 (leaves + ear)			Experiment 2 (leaves)		
	Organo-soluble	Natural products	Unextracted	Organo-soluble	Natural products	Unextracted
Foliage	27	3.4	3.5	27	3.2	0.73
Kernels	0.012	0.007	0.052	0.017	0.019	0.093
Cobs	0.018	0.004	0.012	0.014	0.005	0.013

Identification of residues was made by comparison with authentic standards using two-dimensional TLC. No acetamide was found in any sample.

The highest residues were observed in foliage, where 31 to 34% of the applied radioactivity was recovered. Approximately 85 to 89% of this fraction (27-28% of the applied) was organosoluble; 9 radiolabelled components were observed. The main compounds were identified as thiodicarb (68 to 76% of the foliage fraction, about 20% of the applied dose) and methomyl (19 to 21% of the fraction, about 6% of the applied dose). Methomyl oxime and methomyl methylol were identified at about 0.3% of the applied dose in experiment 1 and at trace levels in experiment 2. The additional unidentified organosolubles accounted for less than 10% of the radioactivity in the fraction (about 3 to 4% of the applied dose). Less than 5% of the applied radioactivity was water-soluble, postulated to be mainly natural constituents derived from the incorporation of labelled metabolites. Only about 1% of the radioactivity was recovered as free metabolites after enzymatic hydrolysis.

In the kernels, most of the radioactivity (72 to 74%) could not be extracted. Only 26 to 28% of the radioactive residue was organosoluble, with 13 to 17% as organosoluble metabolites and the rest precipitated or unextractable. Unextractable and precipitated residues were postulated to be natural kernel constituents resulting from the absorption of radiolabelled carbon dioxide or incorporation of other metabolites. The low residue in kernels precluded identification of any individual metabolites; 4 compounds were observed by TLC, none of which corresponded to any reference standard. Results in cobs were similar, with only about half in the organosoluble fraction but no component corresponding to known authentic standards.

Cotton. Two studies were reported on the metabolism of thiodicarb in cotton plants following stem injection and leaf application (Feung and Chancey, 1977a) .

In one [^{14}C]thiodicarb (50 µg, 4 x 10^6 dpm, 2 µCi, specific activity 8.1 X 10^4 dpm/µg) was injected into the stem of 4 to 5 week cotton plants which were maintained in a greenhouse and harvested 7, 14, 21 and 28 days after treatment. Samples were frozen until analysis (at least 8 hours). In the other [^{14}C]thiodicarb (160 µg, 13 X 10^6 dpm, 6 µCi) was applied by stem injection to some plants and by topical application to the top surfaces of leaves of other 4-week plants. Both groups of plants were maintained in enclosed glass containers and volatiles were collected in a series of traps at intervals of 1, 4 and 7 days after application. Samples were analysed as previously described and extracted by a procedure similar to that used for tomato leaves.

Carbon dioxide and acetonitrile accounted for most of the radioactivity determined as volatile components in the second experiment. The percentage of the total applied radioactivity released as $^{14}CO_2$ and $^{14}CH_3CN$ was greater from leaf treatment than stem injection, with a ratio of CO_2:CH_3CN of 2:1 for injected and 1:20 for leaf-treated plants.

In the first experiment apparent volatile compounds, presumed to be mostly carbon dioxide and acetonitrile, accounted for 70% of the applied radioactivity in the 28-day samples. The remaining extractable residues (28%) showed that thiodicarb was metabolized in cotton to at least 7 water-soluble and 6 organosoluble components (7 and 21% of the radioactivity respectively). It was observed that after short treatment times, the organosoluble metabolites were predominant, whereas longer times resulted in a decrease in organosoluble concentrations with a corresponding increase in water-soluble compound levels. Only a small proportion of the radioactivity (0.7 to 2.6%) remained in the plant tissues after extraction (Table 15).

Table 15. Recovery of the applied radioactivity from injected cotton plants measured at selected intervals (Feung and Chancey, 1977a)

Fraction	^{14}C, % of applied			
	7 DAT	14 DAT	21 DAT	28 DAT
Organosoluble	53.1	8.0	8.4	6.7
Water-soluble	11.6	25.2	21.7	21.2
Unextractable	0.7	2.6	1.7	1.5
Presumed volatiles	34.6	64.8	67.9	69.7

DAT: days after treatment
Presumed volatiles: radioactivity not accounted for by combined extractable and unextractable fractions

Compounds were identified or characterized by co-chromatography against authentic standards in one and two-dimensional TLC with mass spectrometry and nuclear magnetic resonance as appropriate.

Of the 6 organosoluble compounds (53 to 7% of the applied ^{14}C, declining over the duration of the 4-week growth period) three were identified, accounting for 50 to 98% of the fraction: thiodicarb (initially major), methomyl (finally major) and methomyl oxime (trace). The three unidentified compounds (2 to 50% of the fraction) accounted for between 1 and 4% of the total applied dosage.

Of the 7 water-soluble components (12 to 25% of the applied ^{14}C), 2 were identified as conjugates of methomyl and methomyl oxime (probably glycoside esters). The remaining 5 components, which accounted for over 80% of the radioactivity in the aglycone fraction, were thought to be naturally occurring products formed by incorporation of $^{14}CO_2$ from the absence of N-S and N-C bonds by NMR as well as their different behaviour from standards on TLC in all solvent systems used.

The absorption, translocation and metabolism of [^{14}C]thiodicarb (purity >98.5%) in or on cotton after a leaf surface application was also investigated by Feung and Chancey (1978a).

A solution of [^{14}C]thiodicarb (about 6000 ppm, specific activity 12,000 dpm/µg) was spread on the top surfaces of cotton leaves at the flower bud stage at a rate approximating 1.12 kg ai/ha. The plants were maintained in a greenhouse until the bolls were mature, when they were harvested and the seeds de-linted. The senescent leaves were also collected and all samples were frozen until analysis. Small branches were also taken from treated plants 14 days after application to investigate absorption and translocation. Frozen seed and leaves from the harvested plants were ground in a blender with acetonitrile/water. Previously described extraction and fractionation procedures were followed to separate organosoluble and water-soluble compounds.

In order to assess the extent of absorption and translocation, the branches removed after 14 days were pressed and dried and then exposed to X-ray film for 21 days. Absorption and translocation of radioactivity were both poor; most of the radioactivity still remained on the leaf surface. Radioactivity measurements in harvested seed and lint samples indicated very low activity in lint (0.05% of the applied dose) and seeds (0.09%), further evidence of low absorption and translocation.

Although seed samples were fractionated, the characterization of metabolites was not possible owing to the very low radioactivity. The low level of radioactivity found in seeds and lint was thought to be the result of incorporation of $^{14}CO_2$ released from the parent compound into naturally occurring plant components.

The identification of compounds in and on senescent leaves was by two-dimensional TLC, and infrared and mass spectrometry.

Table 16. Compounds isolated from senescent leaves of cotton (Feung and Chancey, 1978a).

Compounds	% of total radioactivity in leaves
Organosoluble	
– thiodicarb	21.85
– methomyl	12.42
– methomyl oxime	0.14
– methomyl methylol	0.51
– others	1.25
Water-soluble, conjugates of	
– methomyl	3.1
– methomyl oxime	0.84
– methomyl methylol	1.71
– others	4
Unextractable	20

Eleven organosoluble and nine water-soluble components were isolated. Four of the organosoluble components were identified, accounting for 95% of this fraction, 35% of the total radioactivity in the leaves: thiodicarb (major), methomyl (major), methomyl oxime (minor) and methomyl methylol (minor). The remaining organosoluble metabolites (about 5% of the fraction) did not match authentic standards on TLC. Three of the water-soluble components (about 60% of the fraction, 6% of the total radioactivity in the leaves) were identified as conjugates of methomyl, methomyl oxime and methomyl methylol. The remaining metabolites (40% of this fraction) did not correspond to any of the standards on TLC. Unextractable residues accounted for approximately 20% of the total radioactivity in the leaves.

Soya beans. The disposition and metabolism of thiodicarb in or on soya beans after a leaf surface application was investigated by Feung and Chancey (1979a).

A solution of [^{14}C]thiodicarb (purity >98.5%, about 6000 ppm, specific activity 12,000 dpm/µg) was spread on the top surfaces of soya bean leaves at the flower bud stage at a rate approximating 1.12 kg ai/ha, purity >98.5%. The plants were maintained in a greenhouse until the

pods were mature, when they were harvested and the seeds separated from the hulls. The senescent leaves were also collected and all samples frozen until analysis.

Radioactivity was very low in the seed (0.18% of the applied dose) and hulls (0.19%), evidence of low absorption and translocation. The radioactivity was too low for the characterization of metabolites. The radioactivity in the seeds and lint was thought to be the result of incorporation of $^{14}CO_2$, released from degraded metabolites, into naturally occurring plant components.

Residues were extracted from the leaves as from tomato leaves, separating organosoluble and water-soluble components. Identification was by two-dimensional TLC and mass spectrometry.

Table 17. Compounds isolated from soya bean leaves (Feung and Chancey, 1979a).

Compounds	% of applied radioactivity
Organosoluble	
– thiodicarb	85
– methomyl	5.7
– methomyl oxime	Trace
– others	<1
Water-soluble, conjugates of	
– methomyl	0.10
– methomyl oxime	0.03
– methomyl methylol	0.13
Unextractable	<5

Three organosoluble compounds (99% of the fraction, 91% of the total radioactivity in the leaves) were identified: thiodicarb (major), methomyl (minor) and methomyl oxime (trace). The remaining radioactivity (1% of the fraction) did not match any of the standards on TLC. Of seven water-soluble components, three (65% of the fraction, 0.3% of the total radioactivity in the leaves) were identified as conjugates of methomyl, methomyl oxime and methomyl methylol. Two other minor components were found to correspond to unidentified water-soluble compounds from cotton, indicating consistency of metabolism. The unextractable residues accounted for <5% of the total radioactivity in the leaves.

Peanuts. Feung and Blanton (1986b) investigated the potential for the formation of acetamide as a metabolite of thiodicarb in peanut foliage, roots, nuts and shells. The [^{14}C]thiodicarb (purity >98%, 18.0 mCi) was applied topically to the foliage four times at the rate of 1.1 kg ai/ha at 7-day intervals. Samples harvested 21 days after the last treatment were individually analysed.

Appreciable radioactivity was found in foliage (21.8% of the applied dose), while only 0.197%, 0.499% and 0.207% were detected in the roots, nuts and shells respectively. Approximately 77% of the applied radioactivity was unaccounted for, possibly owing to volatilization.

In foliage, almost 60% of the radioactive residue was organosoluble. It was analysed by two-dimensional TLC. In this fraction, thiodicarb (7.28% of the applied dose) and methomyl (1.70%) were respectively the major component and the major metabolite. The remaining 5 minor metabolites (all <1.5% of the applied dose) did not match any of the authentic standards on TLC. No acetamide was found as a metabolite of thiodicarb.

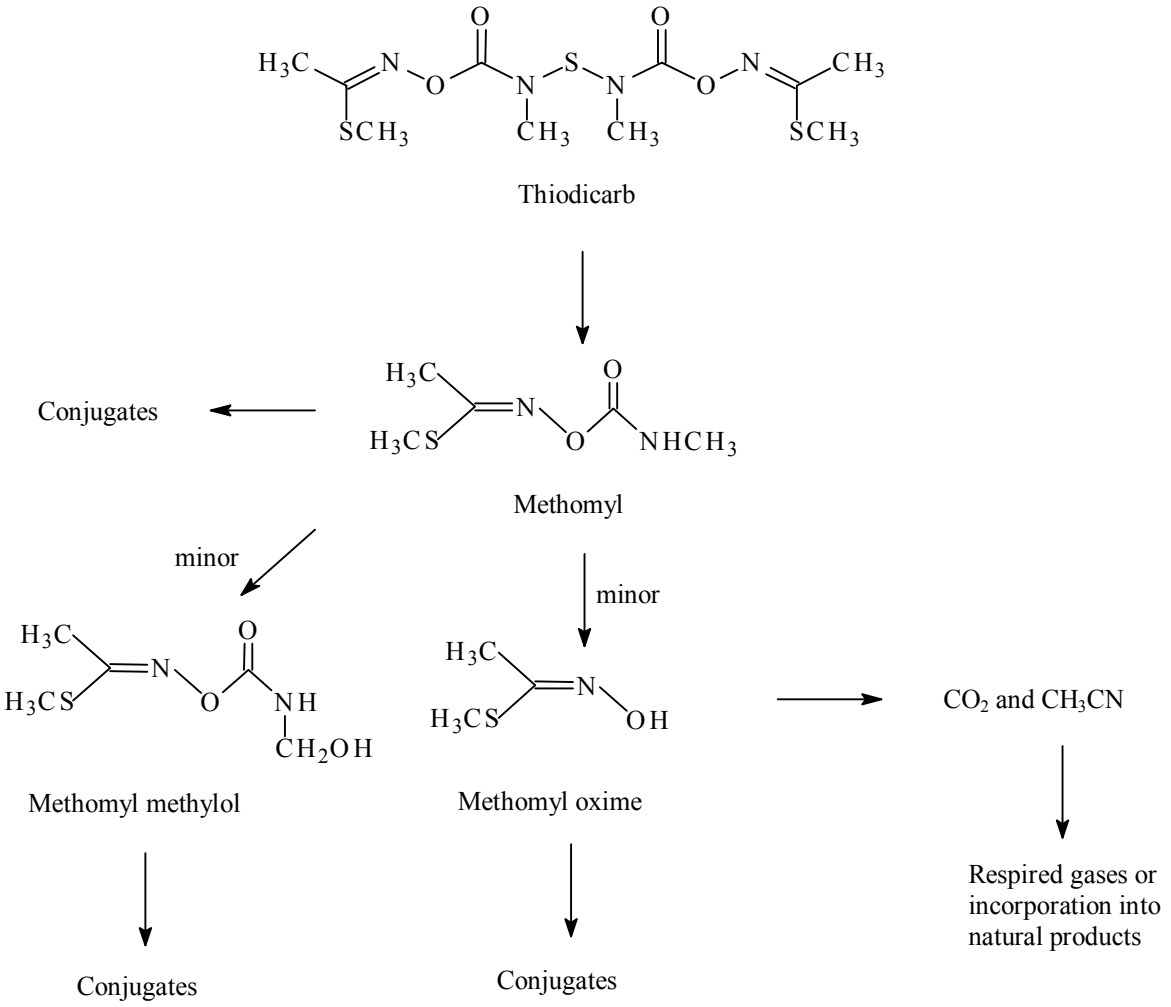
In roots, nuts and shells, most of the ^{14}C residues (53-68%) could not be extracted from the tissue. The organosoluble extract of roots represented 28% of the total radioactivity, while in nuts and shells 23 and 18% of the total radioactivity was organosoluble.

The organosoluble fractions isolated from nuts and shells were further analysed by two-dimensional co-chromatography. At least three radioactive spots were detected, none of which matched any of the authentic standards on TLC. In roots, five spots were detected; again, none

matched authentic standards. These organosoluble metabolites were believed to be natural constituents of roots, nuts and shells. Acetamide was not detected as a metabolite of thiodicarb in any of the samples.

The metabolism of thiodicarb has been found to be qualitatively similar among plant species. It is cleaved at an N-S bond to form methomyl, which is hydrolysed to methomyl oxime, thence further metabolized to CO_2 and acetonitrile. A small amount of methomyl can also be hydroxylated to methomyl methylol. Additional polar metabolites can be envisaged to result from conjugation or incorporation of ^{14}C fragments into natural products. The proposed metabolic pathways are shown in Figure 2.

Figure 2. Proposed metabolic pathways of thiodicarb in plants.



The volatile components $^{14}CO_2$ and [^{14}C]acetonitrile together generally accounted for the loss of 50% or more of the applied dose. In general thiodicarb was the major component recovered from plant foliage, with methomyl or conjugates thereof being major metabolites. Minor free metabolites such as methomyl oxime and methomyl methylol accounted for less than 2% of the extracted radioactivity, with apparent conjugates of these metabolites also present in varying amounts in mature plant foliage. Significant translocation of residues from foliage to seeds, fruits, grain or tubers does not occur**.** However, residues of methomyl were found in supervised field trials on maize in the corn + cob from the foliar application of methomyl.

Environmental fate in soil

Photolysis. Doble *et al.* (2000) incubated thiodicarb under aerobic conditions at 20 ± 2°C with a clay loam soil at a rate equivalent to 1 kg ai/hectare for a period of 21 days. Throughout the study, the moisture content of the soil was maintained at approximately 45% of the maximum water-holding capacity.

The photolysis units, fitted with quartz glass lids and connected to a series of trapping solutions to collect any volatile products evolved, were irradiated with xenon lamps. At each sampling duplicate irradiated and control samples were extracted and the components characterized and quantified by HPLC. Selected extracts were also analysed by TLC to provide confirmation of the compounds present. The associated trapping solutions were also taken for analysis. The traps were changed once during the study.

The overall recovery of radioactivity was 103% for irradiated samples and 99% for control samples. Volatile radioactivity reached a maximum of almost 50% of the applied dose in control and 45% in irradiated samples. It was determined to be associated with carbon dioxide.

Unextractable residues increased steadily and reached a maximum of about 30% of the applied radioactivity at 21 days in both irradiated and control soil. HPLC and TLC of extracts showed that thiodicarb was quickly degraded to the major product methomyl, which reached a maximum of 82% of the applied dose in the control soil and 92% in the irradiated soil at day 2. It then decreased to about 20% by the end of the study.

Methomyl oxime was detected only once in one of the duplicate samples at day 1 in the control soil at 0.5% of the applied radioactivity.

The DT_{50} and DT_{90} of thiodicarb, calculated using a kinetic modelling program, were 0.4 and 1.5 days in control soil and 0.9 and 3 days in irradiated soil.

Aerobic degradation. In two different studies sandy loam soil (pH 5.4, 0.49% organic matter, 70% sand, 17% silt, 13% clay, 9.24% water-holding capacity at 0.33 bar) in metabolism flasks was treated with 10 mg/kg of [^{14}C]thiodicarb (specific activity 20.05 mCi/mmole and radiochemical purity 99%) and maintained under aerobic conditions at 75% moisture content at 1/3 bar in an environmental chamber in the dark at 25 ± 1°C for up to 60 days (Feung and Weisbach, 1991a). Viability in the soil was verified at the beginning and end of the experiment. Volatile organic compounds were trapped in methanol, and CO_2 in 2-ethoxyethanol/ethanolamine, 2:1. Duplicate samples were taken at 0, 0.5, 1, 3, 7, 14, 21, 30 and 60 days, and extracted with methanol and acidified methanol; the extracts were concentrated and analysed by TLC.

Radioactivity in the soil decreased gradually to 42% of the applied dose at day 60 while volatiles increased to 53% over the same period. The volatile radioactive product retained in the methanol traps was identified as acetonitrile by HPLC and GC-MS, and that in the ethoxyethanol-ethanolamine traps was identified as carbon dioxide by re-trapping in NaOH and precipitating with barium chloride.

Thiodicarb steadily decreased as time progressed. It was rapidly degraded to methomyl which in turn was degraded, although more slowly than thiodicarb, to methomyl oxime which was further degraded to acetonitrile and carbon dioxide in a ratio of about 1:28. Methomyl oxime never exceeded 3.2% of the applied dose at any time and was generally less than 1%. A plot of thiodicarb against time followed apparent first-order kinetics. The half-life was calculated to be 1.5 days and the DT_{90} 5.1 days.

Methomyl increased rapidly to 80% of the applied radiocarbon at day 7 and then gradually decreased when most of the thiodicarb had been degraded. The linear portion of the degradation curve

(days 7, 14, 21, 30 and 60) was used to calculate the degradation rate. Methomyl had a half-life of 27 days and a DT_{90} of 90 days.

Three UK soils, a sandy loam (77% sand, 14% silt, 10% clay, 1.8% organic carbon, pH 6.0), a high-pH clay loam (20% sand, 52% silt, 28% clay, 4.6% organic carbon, pH 7.6) and a clay loam (24% sand, 53% silt, 23% clay, 1.9% organic carbon, pH 6.9), were treated with [^{14}C]thiodicarb at a rate equivalent to 1 kg ai/ha and incubated in the dark at a temperature of 20°C, the clay loam also at 10°C, for 56 days (Burr, 2000). After treatment the soil flasks were connected to a series of trapping solutions to collect volatile products, and flasks were removed for analysis at intervals.

Throughout the study, the moisture content of the soil was maintained at approximately 45% of the maximum water-holding capacity.

The soils were extracted and the components identified and quantified by HPLC. Selected extracts were analysed by LC-MS to confirm structural identity. At each sampling the radioactivity in the traps was quantified and the traps were replenished between samplings.

Volatile radioactivity was shown to be due to CO_2, produced rapidly in all the soils. After 56 days incubation the levels reached 61% in the high-pH clay loam, 66% in the sandy loam, 59% in the clay loam incubated at 20°C and 40% in the clay loam incubated at 10°C.

The levels of extractable ^{14}C decreased to <5% after 56 days in all the soils incubated at 20°C and <20% in the soil incubated at 10°C. The unextractable ^{14}C reached a maximum of about 34-35% after 28 days in the three soils incubated at 20°C, with some evidence of a decrease at 56 days, and a maximum of about 30% in the clay loam incubated at 10°C.

The levels of thiodicarb in the extracts decreased rapidly to form one major product, methomyl, which reached a maximum of 80% of the applied radioactivity in the high-pH clay loam, 63% in the sandy loam and 78% in the clay loam incubated at both 20°C and 10°C. The quantity of methomyl in the extracts decreased with time and no further major degradation products of methomyl were detected. There were low levels of minor products, including methomyl oxime, but none of these exceeded 4% of the applied radioactivity at any time.

The DT_{50} and DT_{90} of thiodicarb, calculated with a kinetic modelling program, are shown in Table 18.

Table 18. DT_{50} and DT_{90} of thiodicarb in aerobic conditions (Feung and Weisbach, 1991b).

Soil	DT_{50} (days)	DT_{90} (days)
High-pH clay loam at 20°C	0.01	0.23
Sandy Loam at 20°C	1.2	4.9
Clay Loam at 20°C	0.67	2.2
Clay Loam at 10°C	2.0	6.7

Anaerobic degradation. A clay loam soil was flooded with deionized water and purged with nitrogen for 42-43 days before treatment to establish anaerobic conditions. A solution of [^{14}C]thiodicarb (specific activity 3.96 MBq mg^{-1}; radio purity 98.0%) in a minimal volume of methanol was applied to the water surface at a nominal application rate equivalent to 1 kg ai/ ha, and the soil was incubated at 20°C in the dark (Clarke, 2000). During incubation the system was continually purged with nitrogen, which was then passed through two traps containing methanol, a further trap containing water and a final trap containing 2M aqueous potassium hydroxide to trap liberated volatile materials. Duplicate samples were removed from the system for analysis after 0, 16, 28, 74, 123 and 240 min incubation. At each sampling point the water phase was separated from the soil and the two phases analysed separately.

The radioactivity in the water phase was quantified by LSC of representative aliquots. The nature of the radioactive material present in the water phase was determined by HPLC and structural

confirmation of the species was obtained, where possible, by LC-MS-MS of representative water samples. Because of the rapid degradation of thiodicarb the amount of [^{14}C]thiodicarb present at time 0 was taken as the radio purity value. The results actually found in the duplicates at this time are shown in Table 19 as having incubation periods of 5 and 8 min owing to the inevitable delay between treatment and chromatographic analysis.

All soil samples were extracted with methanol by shaking at room temperature. The radioactivity present in the extracts was quantified by LSC of representative aliquots. The methanol extracts of each soil sample were combined and those containing >5% of the applied radioactivity were analysed by HPLC. The soil was then air-dried, ground to a fine powder and the residual radioactivity quantified by combustion of representative subsamples.

After 240 minutes incubation 78% of the applied radioactivity remained in the water phase. A further 12% was extracted from the soil, 3.5% remained unextracted and 1.4% was present as volatile material.

The mean total recovery of ^{14}C from each soil sample was 94%. All recoveries were within the range 90-98% except one of the 123 min samples, from which the recovery was 88%.

Under anaerobic conditions in water, thiodicarb was degraded extremely quickly through the transient intermediate *S*-methly *N*-[*N*-methyl-*N*-(methylaminothio)carbamoyloxy]thioacetamidate to form acetonitrile, methomyl and methomyl oxime (Table 19).

Table 19. Thiodicarb degradation in water under anaerobic condition (Clarke, 2000).

Incubation time (min)	^{14}C, % of applied							
	0	5	8	16	28	74	123	240
thiodicarb	98	61	32	0.9	0.7	nd	nd	nd
methomyl	nd	2.2	2.8	2.5	2.3	2.3	2.0	1.3
methomyl oxime	nd	nd	nd	nd	nd	0.3	0.2	0.8
acetonitrile	1.5	24.	35	69	69	78	73	75
S-methyl *N*-[*N*-methyl-*N*-(methylaminothio)carbamoyloxy]thioacetamidate	0.5	9.4	28.2	17.4	17.1	7.4	4.8	0.9
Minor products (total)*	nd	nd	nd	nd	nd	0.3	0.3	1.7

nd : not detected
* up to three were observed, each <1.2% of the applied radioactivity

HPLC analysis of soil extracts containing >5% of the applied radioactivity showed that no thiodicarb was present. The radioactivity detected was attributed to acetonitrile and methomyl oxime, which were present at 10% and 0.3% of the applied radioactivity respectively after 240 minutes.

The DT_{50} and DT_{90} values for thiodicarb degradation in the water phase were calculated as 6.0 and 12.6 minutes respectively, using a modelling program. Nearly all the applied radioactivity (about 90%) was present in the water phase during the first 28 minutes and <1% of this was present as thiodicarb at this time point, so the DT_{50} and DT_{90} values for thiodicarb degradation in the entire system are equivalent to those in the water phase.

Soil adsorption/desorption. Cranor (1991) determined the adsorption isotherms and Freundlich constants of four soils equilibrated with aqueous solutions of [^{14}C]thiodicarb (specific activity 23.0 mCi/mmole). The characteristics of the soils are shown in Table 20.

Table 20. Characteristics of soils used in adsorption/desorption study (Cranor, 1991).

Characteristic	Soil #21	Soil #36	Soil #79	Soil #92
Classification	Silt loam	Clay	Sandy loam	Sand
% Organic matter	2.4	2.4	0.8	0.5
% Sand	14	8	54	92
% Silt	68	34	36	4

Characteristic	Soil #21	Soil #36	Soil #79	Soil #92
Classification	Silt loam	Clay	Sandy loam	Sand
% Clay	18	58	10	4
CEC (meq/100 g)	10	26	4.7	0.3
pH	7.1	6.7	6.5	7.4
% Field moisture capacity at 1/3 bar	28	37	9.5	1.9
Bulk density (g/cm^3)	1.2	1.2	1.5	1.6

One-g portions of each sterilized soil were equilibrated with 5 ml aliquots of each [^{14}C]thiodicarb solution in 0.01 M $CaCl_2$ with nominal concentrations of 2.0, 1.0, 0.8 and 0.5 µg/ml. Equilibration was in darkness on a mechanical shaker in an environmental chamber at 25 ± 1^{o}C for 24 h. Each of the suspensions was then centrifuged and the supernatant removed by pipetting. The volume of the supernatant was measured and triplicate aliquots radioassayed.

For desorption, appropriate volumes of 0.01 M $CaCl_2$ solution were added to each sample tube according to the volume removed after the adsorption phase. The soil suspensions were shaken in darkness for 24 h as before, then centrifuged and the supernatants removed.

Table 21. Freundlich adsorption/desorption constants for thiodicarb in four soils (Cranor, 1991).

Soil	% organic carbon	Adsorption		Desorption	
		K_d	K_{oc}	K_d	K_{oc}
Silt loam	1.2	4.5	373	5.3	444
Clay	1.2	14	1167	6.2	518
Sandy loam	0.4	1.3	335	3.4	855
Sand	0.25	0.16	64	0.20	79

K_{oc} values above 5,000 denote immobility in soil, 2,000-5,000 slight mobility, 500-2,000 low mobility, 150-500 medium mobility and 50-150 high mobility. The results indicated that thiodicarb had low mobility in clay, medium mobility in silt loam and sandy loam, and high mobility in sand.

Residues in rotational crops. In a study with a confined sandy loam soil (Jordan and Wyatt, 1994) two plots were established in a Lexan-covered, open-air structure in Lucama, Wilson County, North Carolina, USA. [^{14}C]Thiodicarb (specific activity 20,362 dpm/µg, radiochemical purity 99.7%) was applied at the rate of 6.7 kg ai/ha. The confined soil was then planted with rotational crops, mustard greens, radishes and wheat, at 31, 125 and 364 days after treatment (DAT). Before planting, the soil was tilled 8-10 cm to prepare a suitable seedbed. Soil samples were taken for analysis immediately after treatment and at the times of planting and harvesting the rotational crops. Mature samples of all crops and the immature forage samples of wheat plants were taken for analysis.

Immediately after treatment, the soil residue averaged 2.13 mg/kg and decreased steadily to 0.12 mg/kg at 364 days. Most of the applied radioactivity was at the depth of 0-15 cm. 0-15 cm soil cores were extracted with methanol followed by acidified acetone, and the extracts analysed by TLC and HPLC. The remaining solid was extracted with 0.5 N NaOH, and the unextractable solid (humin) combusted. The NaOH extracts were adjusted to pH 1 and centrifuged. The supernatant (fulvic acid) and the precipitate (humic acid) were radioassayed.

The identification and characterization of ^{14}C residues in the soil are shown in Table 22. Thiodicarb was rapidly degraded in soil as only 5.4% of the radiocarbon in the 31 DAT soil was due to unchanged parent compound. Methomyl increased rapidly and accounted for 47% of the TRR in the 31 DAT soil. These results are consistent with previous studies of degradation in soil.

Table 22. Characterization of ^{14}C residues in soil (Jordan and Wyatt, 1994).

Fraction		0 DAT		31 DAT	
		% of TRR	mg/kg as thiodicarb	% of TRR	mg/kg as thiodicarb
Organic extracts	Thiodicarb	82	1.5	5.4	0.03
	Methomyl	0	0	47	0.20
	Unknown	0	0	0.2	<0.01
	Subtotal	82	1.5	53	0.23
Solid	Fulvic acid	--	--	13	0.05
	Humic acid	--	--	4.1	0.02
	Humin	--	--	18	0.07
	Subtotal	9.9	0.18	35	0.14
Total		92	1.7	88	0.37

The residue levels in the rotational crops are shown in Table 23.

Table 23. Total radioactive residues rotational crops (Jordan and Wyatt, 1994).

Time	^{14}C (mg/kg thiodicarb equivalents)					
	Wheat forage	Mustard greens	Radish tops	Radish roots	Wheat grain	Wheat straw
Day 0	--	--	--	--	--	--
31 DAT	2.1	1.3	1.2	1.6	0.48	2.4
125 DAT	0.28	0.28	0.24	0.11	0.21	0.81
364 DAT	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

The identification and characterization of the residues from 31 and 125 DAT crop samples are shown in Tables 24 to 27. Methomyl was the major component identified in aqueous extracts before hydrolysis (up to 0.14 mg/kg, the level in radish tops from the 31-day plant-back interval), and acetic acid the main identified compound after either acid or base hydrolysis. Glucose was also identified in aqueous extracts of radish tops (up to 0.14 mg/kg) after hydrolysis with β-glucosidase. Unidentified polar residues (Unknowns 1 and 2) constituted up to 36% of the TRR in aqueous extracts. Analysis of the aqueous extracts after extensive hydrolysis suggested that these were likely to consist of natural products and conjugates. Insoluble bound final residues constituted up to 10% of the TRR.

In general, most of the radiocarbon residue consisted of natural products: fatty acids (1 to 11% of the TRR), water-soluble polysaccharides, proteins and lipids (5 to 20% of the TRR), starch, protein, pectin, lignin, hemicellulose and cellulose fractionated from cell walls (17 to 63% of the TRR). As in degradation in plants and soil, it was proposed that thiodicarb is quickly metabolized to

methomyl, thence via an assumed methomyl oxime intermediate to acetic acid and carbon dioxide, which are then incorporated into natural products.

Table 24. Characterization of ^{14}C residues in 31 DAT wheat (Jordan and Wyatt, 1994).

Fraction	Wheat forage		Wheat straw		Wheat grain	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Aqueous extract						
Unknown 1	21.9	0.45	17.2	0.42	5.4	0.03
Unknown 2	5.1	0.10				
Methomyl	3.6	0.07				
Aqueous extract- acid hydrolysed [1]						
Unknown 1	13.6	0.28	4.4	0.11		
Acetic acid	8.5	0.17	9.4	0.23	5.4	0.03
Bound to column	8.6	0.18	3.4	0.08		
Aqueous extract- base hydrolysed [1]						
Unknown 1	8.2	0.17	7.4	0.18		
Unknown 2	10.7	0.22				
Acetic acid	9.4	0.19	7.3	0.18		
Methomyl oxime	2.3	0.05				
Bound to column			2.5	0.06		
Hexane extract						
Fatty acids					1.6	<0.01
Fatty acid region #1	1.0	0.02	2.3	0.06		
Fatty acid region #2	0.7	0.02	2.5	0.06		
Fatty acid region #3	2.1	0.05				
Fatty acid region #4	1.3	0.03				
Fatty acid region #5	1.4	0.03				
Fatty acid region #6	4.3	0.09				
Bound residues - cell wall fractionation						
Phosphate - polysaccharide fraction						
Region #1 (TLC origin)	3.0	0.06	1.9	0.05	9.5	0.05
Region #2	0.1	<0.01	3.8	0.09		
Methomyl			2.4	0.06		
Bound to column			7.5	0.18		
MeOH/CH_2Cl_2 extract - protein fraction						
Region #1 (TLC origin)	0.6	0.01	2.5	0.06	8.6	0.04
Acetone/phosphate extract						
Lipid fraction					0.6	<0.01
Region #1 (TLC origin)	1.2	0.02	2.3	0.06		
Starch	0.7	0.01	2.7	0.07	9.1	0.04
Proteins	14.1	0.29	4.6	0.11	23.6	0.11
Pectins	10.6	0.22	2.8	0.07	9.5	0.05
Lignin	4.3	0.09	11.0	0.27	9.7	0.05
Hemicellulose	0.4	<0.01	12.2	0.30	6.7	0.03
Cellulose	1.0	0.02	2.2	0.05	0.8	<0.01
Unextractable solids	1.9	0.04	9.2	0.22	4.9	0.02
Total recoveries	79	1.6	87	2.1	90	0.42

[1] Not included in total.

Table 25. Characterization of ^{14}C residues in 31 DAT mustard greens and radishes (Jordan and Wyatt, 1994).

Fraction	Mustard greens		Radish tops		Radish roots	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	M/kg
Aqueous extract						
Unknown 1	17.	0.23	16.	0.19	11.	0.18
Unknown 2	6.3	0.08				
Methomyl	9.4	0.12	12.	0.14	5.5	0.09
Aqueous extract - acid hydrolysed [1]						
Unknown 1	11	0.14	17.	0.20	9.1	0.15

Fraction	Mustard greens		Radish tops		Radish roots	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	M/kg
Unknown 2	6.5	0.08				
Acetic acid	16.	0.21	10.9	0.13	7.6	0.12
Aqueous extract - base hydrolysed [1]						
Unknown 1	7.6	0.10	6.5	0.08		
Unknown 2	10.	0.13	4.3	0.05		
Acetic acid	8.6	0.11	9.5	0.11		
Methomyl oxime	6.8	0.09	7.8	0.09		
Aqueous extract - β-glucosidase treated [1]						
Glucose	--	--	12.1	0.14		
Hexane extract – fatty acids						
Fatty acid region #1	1.9	0.03	2.1	0.02	1.2	0.02
Fatty acid region #2	1.0	0.01	1.6	0.02	4.9	0.08
Fatty acid region #3	1.0	0.01	2.4	0.03	3.5	0.05
Fatty acid region #4	1.2	0.02	5.2	0.06		
Fatty acid region #5	0.8	0.01				
Bound residues – cell wall fractionation						
Phosphate - polysaccharide fraction						
Region #1 (TLC origin)	3.9	0.05	6.8	0.08	3.9	0.06
Region #2	0.5	<0.01	0.5	<0.01		
$MeOH/CH_2Cl_2$ extract - protein fraction	0.7	<0.01	1.0	0.01	2.0	0.03
Acetone/phosphate extract – lipid fraction						
Region #1 (TLC origin)	1.6	0.02	3.0	0.03	0.7	0.01
Starch	0.8	0.01	1.4	0.02	1.8	0.03
Proteins	7.7	0.10	10.8	0.13	6.2	0.10
Pectins	6.1	0.08	9.3	0.11	19.0	0.30
Lignin	1.2	0.02	1.9	0.02	6.1	0.10
Hemicellulose	0.4	<0.01	0.5	<0.01	3.4	0.05
Cellulose	0.1	<0.01	0.5	<0.01	2.3	0.04
Unextractable solids	0.4	<0.01	1.5	0.02	4.6	0.07
Total recoveries	62.	0.79	77	0.88	76.	1.2

[1] Not included in total.

Table 26. Characterization of ^{14}C residues in 125 DAT wheat (Jordan and Wyatt, 1994).

Fraction	Wheat forage		Wheat straw		Wheat grain	
	% of TRR	ppm	% of TRR	ppm	% of TRR	ppm
Aqueous extract					4.5	<0.01
Unknown 1	22	0.06	4.6	0.04		
Unknown 2	14	0.04	7.7	0.06		
Unknown 3			4.0	0.03		
Methomyl	7.0	0.02				
Aqueous extract - acid hydrolysed [1]						
Unknown 1	18	0.05	4.0	0.03		
Acetic acid	25	0.07	12.3	0.10		
Hexane extract						
Fatty acids					1.0	<0.01
Fatty acid region #1	2.3	<0.01	0.5	<0.01		
Fatty acid region #2	1.8	<0.01	1.6	0.02		
Fatty acid region #3	0.8	<0.01	0.8	<0.01		
Fatty acid region #4	1.9	<0.01	0.2	<0.01		
Bound residues - cell wall fractionation						
Phosphate- polysaccharide fraction						
Region #1 (TLC origin)	7.8	0.02	11.4	0.09	10.3	0.02
$MeOH/CH_2Cl_2$ extract						
Protein fraction	1.5	<0.01				
Region #1 (TLC origin)			3.8	0.03	8.5	0.02
Acetone/phosphate extract						

Fraction	Wheat forage		Wheat straw		Wheat grain	
	% of TRR	ppm	% of TRR	ppm	% of TRR	ppm
Lipid fraction	1.6	<0.01			1.0	<0.01
Region #1 (TLC origin)			2.7	0.02		
Starch	1.4	<0.01	6.4	0.05	9.8	0.02
Proteins	7.9	0.02	5.4	0.04	24.	0.05
Pectins	10.	0.03	4.2	0.03	13.	0.03
Lignin	4.4	0.01	7.4	0.06	8.9	0.02
Hemicellulose	6.6	0.02	12.2	0.10	6.5	0.01
Cellulose	1.5	<0.01	1.9	0.02	0.9	<0.01
Unextractable solids	2.0	<0.01	9.8	0.08	9.9	0.02
Total recoveries	94.	0.22	85	0.67	98.	0.19

[1] Not included in the total.

Table 27. Characterization of ^{14}C residues in 125 DAT mustard greens and radishes (Jordan and Wyatt, 1994).

Fraction	Mustard greens		Radish tops		Radish roots	
	% of TRR	mg/kg	% of TRR	mg/kg	% of TRR	mg/kg
Aqueous extract						
Unknown 1	22.	0.06	34.	0.08	30.	0.03
Unknown 2	10.2	0.03				
Methomyl	14	0.04	14.	0.03	14	0.02
Thiodicarb[1]	16.	0.04	7.2	0.02		
Aqueous extract- acid hydrolysed [2]						
Unknown 1	7.8	0.02	25.	0.06		
Unknown 2	9.4	0.03				
Acetic acid	45.	0.12	30.	0.07		
Hexane extract						
Fatty acids					9.2	0.01
Fatty acid region #1	1.3	<0.01	5.1	0.01		
Fatty acid region #2	1.6	<0.01	1.4	<0.01		
Fatty acid region #3	2.5	<0.01	1.7	<0.01		
Fatty acid region #4	1.1	<0.01	2.5	<0.01		
Bound residues- cell wall fractionation						
Phosphate- polysaccharide fraction					3.5	<0.01
Region #1 (TLC origin)	6.8	0.02	9.0	0.02		
MeOH/CH_2Cl_2 extract						
Protein fraction	2.6	<0.01	5.7	0.01	2.9	<0.01
Acetone/phosphate extract						
Lipid fraction	0.8	<0.01	2.2	<0.01	1.1	<0.01
Starch	1.5	<0.01	2.0	<0.01	1.5	<0.01
Proteins	4.4	0.01	17.9	0.04	9.2	0.01
Pectins	17.	0.05	18.3	0.04	25.	0.03
Lignin	2.7	<0.01	5.2	0.01	5.2	<0.01
Hemicellulose	1.4	<0.01	2.4	<0.01	1.6	<0.01
Cellulose	0.3	<0.01	0.5	<0.01	1.2	<0.01
Unextractable solids	1.3	<0.01	2.6	<0.01	1.0	<0.01
Total recoveries	107.	0.25	130.	0.26	105.	0.10

[1] The thiodicarb may have been an artefact: it appears in the 125-day mustard greens and radish tops at levels up to 0.04 mg/kg but it was not present in the 31-day plants and only at very low levels in the soil. It may have been the result of contamination or analysis error.
[2] Not included in the total.

The results show that thiodicarb was rapidly metabolized to compounds such as methomyl, acetic acid (and glucose conjugates of related compounds). It was further metabolized to characterized natural product fractions, which account for a large percentage of the residues. At the high rate of 6.72 kg ai/ha in a worst-case system (confined soil), residues in rotational crops were significant at plant-

back intervals up to 125 days and negligible (TRR <0.01 mg/kg) in crops planted 1 year after application. Thiodicarb was not considered to be a measurable residue in any crop at any plant-back interval, whereas methomyl was observed in wheat forage, mustard greens and radishes (roots and tops) at the 31- and 125-day plant-back intervals, but only at low levels (31 days, 0.07 to 0.14 mg/kg; 125 days, 0.02 to 0.04 mg/kg).

Environmental fate in water and water/sediment systems

Hydrolysis. The rate of hydrolysis of radiolabelled thiodicarb (purity>99%, specific activity 20.05 mCi/mmol) at a concentration of 12 mg/l was determined at pH 5, 7 and 9 in sterile aqueous buffer solutions (acetate 0.01 M, phosphate 0.01 M and borate 0.1 M respectively) in the dark at 25 ± 1°C (Feung and Weisbach, 1991b).

Samples were taken at 0, 1, 3, 7, 14, 21 and 30 days at all pH levels, as well as 0.17 and 0.33 days at pH 9, for LSC and TLC. Products were identified by 2-dimensional TLC, HPLC and MS. Recovery of radioactivity during the incubation period ranged from 93-105% of the applied dose.

Five radioactive components were detected by TLC (Table 28). Thiodicarb, methomyl and methomyl oxime were identified by 2D-TLC co-chromatography, HPLC and MS. Unknown 0 at the TLC origin was scraped and separated into two components by HPLC. These both fragmented into methomyl and methomyl oxime partial structures on MS, but they could not be identified.

Table 28. Degradation products of thiodicarb hydrolysis (Feung and Weisbach, 1991b).

Products	^{14}C, % of applied dose at intervals (days)								
	0	0.17	0.33	1	3	7	14	21	30
pH 5									
Unknown 0*	1.0	-	-	0.38	0.31	0.68	1.6	2.5	4.4
Unknown 1	0.19	-	-	0.31	0.87	0.94	1.3	1.4	2.1
Thiodicarb	96	-	-	97	94	92	87	84	72
Methomyl	2.9	-	-	1.9	4.3	6.5	9.4	12	20
Methomyl oxime	0.00	-	-	0.22	0.29	0.29	0.37	0.46	0.92
pH 7									
Unknown 0*	0.58	-	-	0.59	1.5	2.2	4.9	6.3	10.
Unknown 1	0.20	-	-	0.22	0.41	0.42	0.42	0.73	0.77
Thiodicarb	97	-	-	96	92	85	72	65	49
Methomyl	1.8	-	-	3.0	5.8	11	21	26	36
Methomyl oxime	0.00	-	-	0.20	0.08	0.94	1.4	1.8	3.1
pH 9									
Unknown 0*	0.46	3.8	7.2	10	11	12	10	7.3	4.4
Unknown 1	0.0	2.5	6.1	7.2	5.3	1.6	0.01	0.00	0.00
Thiodicarb	96	66	39	8.7	1.1	0.56	0.37	0.00	0.00
Methomyl	2.9	25	43	66	66	54	40	29	19
Methomyl oxime	0.36	2.1	4.0	7.5	16	32	49	64	77

* 2 components

Thiodicarb is hydrolyzed to methomyl. Methomyl is more stable than thiodicarb, but is itself hydrolysed to methomyl oxime, particularly at pH 9.

Thiodicarb hydrolysis followed apparent first-order kinetics at a rate dependent on the pH. The calculated half-life of thiodicarb was 78 days at pH 5, 32 days at pH 7 and 0.48 day at pH 9. In pH 9 buffer, methomyl reached a maximum at 1 h, then steadily decreased with a half-life of 15 days.

Photolysis. Feung and Blanton (1987) exposed thiodicarb to outdoor natural sunlight in Research Triangle Park, North Carolina, USA, with a dark control. Weather conditions including sunlight intensity, cloud coverage and air temperature during the irradiation period were reported.

[^{14}C]thiodicarb (specific activity 23 mCi/mmol, radiochemical purity 99.3%) was dissolved in an aqueous 0.05 M phosphate buffer at pH 6 at a concentration of 10 mg/l (the water solubility of thiodicarb was 35 mg/l at 25°C). The temperature was maintained at 25 ± 1°C. Two organic traps each containing 90 ml acetone/dry ice and two CO_2 traps each containing 100 ml of 2-ethoxyethanol/ethanolamine (2:1) at ambient temperature were used to trap volatile components. Sampling intervals were 0, 1, 3, 7, 14, 21 and 23 days. Analyses were carried out by direct LSC and TLC.

Volatile radioactivity was detected as the radioactivity in the irradiated solution decreased, while in the dark control the radioactivity remained essentially unchanged (Table 29).

Table 29. ^{14}C balance of thiodicarb during natural sunlight photolysis (Feung and Blanton, 1987).

Time (days)	Treatment	% of initial radioactivity					
		Buffer solution	CO_2 traps		Acetonitrile traps	Rinse	Total recoveries
			1	2			
0	dark	100	-	-	-	-	100
	irradiated	100	-	-	-	-	100
1	dark	98	-	-	-	-	98
	irradiated	94	0.19	0.06	1.0	-	94
3	dark	98	-	-	-	-	98
	irradiated	92	0.46	0.12	2.9	-	95
7	dark	98	-	-	-	-	98
	irradiated	88	0.65	0.14	6.2	-	95
14	dark	98	-	-	-	-	98
	irradiated	77	2.9	0.32	12	-	92
21	dark	97	-	-	-	-	97
	irradiated	72	3.9	0.37	15	-	91
23	dark	98	-	-	-	-	98
	irradiated	72	4.4	0.43	15	0.17	92

Photolysis followed apparent first-order kinetics. After 23 days of exposure, only 12% of the initial [^{14}C]thiodicarb was present as such in the aqueous solution, while 67% remained in the dark control. The half-life of thiodicarb was calculated to be 7.6 days under natural sunlight and 37 days in the dark.

Methomyl was the major degradation product (Table 30). Methomyl methylol and methomyl oxime were also identified (≤2% of the applied radioactivity). Identification was based on two-dimensional TLC co-chromatography with authentic reference standards. The volatile compounds were identified as CO_2 when trapped in 2-ethoxyethanol/ethanolamine and as acetonitrile when trapped in acetone-dry ice. There were 9 other degradation products, each less than 6% (most 0.1-1%) of the initial dose.

Table 30. Photodecomposition products of thiodicarb in water at pH 6 under natural sunlight (North Carolina, USA).

Products	^{14}C, % of initial dose at intervals (days)						
	0	1	3	7	14	21	23
Irradiated							
Unknown 0	-	-	-	0.08	0.87	0.54	0.83
Unknown 1	-	-	0.38	-	-	-	-
Unknown 2	-	-	0.82	2.29	-	-	-
Unknown 3	-	0.91	1.8	3.9	5.2	5.1	5.0
Unknown 4	-	1.5	1.9	2.6	3.3	3.2	3.2
Methomyl methylol	-	0.54	1.5	2.0	1.9	2.0	2.0
Unknown 5a	-	-	-	-	0.40	-	-
Thiodicarb	-	83	70	51	26	14	12
Methomyl	-	6.6	14	24	38	46	47
Unknown 8	-	0.23	0.45	0.50	0.36	0.31	0.34
Unknown 9	-	-	0.12	-	-	-	-
Unknown 10	-	-	0.15	0.40	0.31	0.13	0.08
Methomyl oxime	-	0.38	0.63	0.92	1.2	1.6	1.7
Dark control							
Unknown 0	-	-	-	0.38	0.46	0.52	0.49
Unknown 1	0.54	0.47	-	0.28	-	-	-
Unknown 2	-	-	-	-	-	-	-
Unknown 3	0.27	0.24	-	1.3	1.8	2.2	2.3
Unknown 4	-	-	-	0.32	0.49	0.59	0.69
Methomyl methylol	-	-	-	1.1	1.0	1.1	1.1
Unknown 5a	-	-	-	-	0.33	-	-
Thiodicarb	97	96	-	82	75	68	67
Methomyl	1.71	1.91	-	11.2	17	22	24
Unknown 8	-	-	-	0.45	0.67	0.46	0.52
Unknown 9	-	-	-	-	-	-	-
Unknown 10	-	-	-	-	0.18	-	-
Methomyl oxime	-	-	-	0.83	1.1	1.3	1.5

The photodecomposition of thiodicarb in aqueous solution involved N-S cleavage to methomyl which was subsequently hydroxylated to methomyl methylol (minor) or hydrolyzed to methomyl oxime. Methomyl oxime was further degraded to acetonitrile and carbon dioxide.

Water sediment systems. The degradation of thiodicarb, applied at a rate of approximately 0.25 mg/kg water, was investigated in two systems over a period of 100 days at 20°C (Bieber, 1992).

Soil and water were collected from two areas northwest and north of Hamburg, Germany (referred to as “Krempe” and “Ohlau” systems). Incubation flasks were equipped with a device for the slow stirring of the water only and a trap for volatile components containing quartz wool impregnated with paraffin oil and soda-lime. The treated flasks were placed on an orbital shaker in the dark at 20°C. Thus aerobic conditions were achieved in the water and anaerobic conditions were found in parts of the sediment.

The recoveries of the applied radiocarbon at various sampling intervals were 86-99% from the Ohlau system (mean 91%) and 75-93% from the Krempe system (mean 86%).

Radioactivity in the water of both systems decreased slowly from 65% to <1% in the Krempe system and from 74% to <1% in the Ohlau system. The radioactivity retained in the sediment was approximately 10% at day 0 and increased slowly to approximately 50% (Ohlau system) and 30% (Krempe) on day 7, then decreased slowly again to about 15% on day 100.

Water and sediment samples were analysed by TLC. In both water and sediment, thiodicarb accounted for 1-2% of the ^{14}C at day 0 and was negligible in all further samples. Thiodicarb was degraded in both test systems to methomyl, which reached 50% and 17% of the applied dose in the

Ohlau and Krempe systems respectively, within one day. Methomyl levels decreased rapidly, becoming negligible by day 7. The levels of methomyl oxime were low (<5%) at all samplings. The final degradation product was carbon dioxide, which rose to represent more than 70% of the applied radioactivity by 100 days.

Some polar components which were present in water initially (about 20% and 14% of the applied dose in the Ohlau and Krempe systems respectively) decreased to negligible levels during the study, but approximately 15% of the applied radioactivity was retained in the sediment finally after having reached maxima at day 7 of about 50% and 30% of the applied dose in the Ohlau and Krempe systems respectively.

The degradation of methomyl from its maximum concentration was found to fit a square root first-order model in the Ohlau system and a first-order model in the Krempe system. The half-life was calculated to be 21 hours in the Ohlau system and 29 hours in the Krempe system, with a DT_{90} of 4 days in both systems. The half-life of thiodicarb was impossible to determine owing to the extremely rapid degradation.

The degradation of [^{14}C]methomyl was studied in two water/sediment systems, from Manningtree and Ongar, over a period of 44 days at 20°C (Oddy, 1999).

The Manningtree sediment had higher organic carbon, nitrogen and niomass contents, while the Ongar sediment had a much higher cation exchange capacity. The Manningtree sediment was classified as a sandy silt loam by the UK Agricultural Development and Advisory Service (ADAS) and as a loam by the USDA, and the Ongar sediment as a clay loam by ADAS and a sandy clay loam by USDA.

Incubation was in glass flasks, containing sediment to associated water in an average ratio of 1:7 in the Manningtree system and 1:5 in the Ongar system, maintained in the dark at 20°C ± 2°C. The water/sediment systems were equilibrated for approximately 4 weeks before adding 113 µg [14C]methomyl to the surface of the water, equivalent to a field application rate of 0.38 kg /ha.

Moist air was passed into the water layer in each flask at a constant rate and then through an ethylene glycol trap to capture organic volatiles and two potassium hydroxide traps to retain any evolved carbon dioxide.

Duplicate samples were taken at intervals for analysis. The water layer was decanted and the sediment layer extracted with methanol. Water and solvent extracts were analysed by HPLC, with certified reference standards for comparison.

Overall mean recoveries of ^{14}C were 87% and 90% from the Manningtree and Ongar systems respectively.

In both systems methomyl was rapidly degraded to CO_2 with levels reaching about 72% and 60% of the applied radioactivity after 31 and 41 days in the Manningtree and Ongar systems respectively. Dissolved carbon dioxide in the form of carbonate was also found in the water phases. Methomyl oxime reached 4% in the water phase and less than 1% in the sediment. It never exceeded 5% of the applied radioactivity in total.

Unextracted residues increased steadily to a maximum of about 16% and 19% of the applied radioactivity in the Manningtree and Ongar systems respectively, before declining to about 15% in both systems after 31 and 44 days.

Half-lives of methomyl calculated with a modelling program were 2.4 days in the Manningtree water and 5.7 days in the Ongar water. The DT_{90} values were 9 days and 11 days respectively.

In summary, in aerobic aquatic systems, thiodicarb is rapidly converted to methomyl which in turn is degraded to CO_2, with no other major products. Thiodicarb and methomyl are unlikely to persist in the aerobic aquatic environment. In or on soil, under aerobic or anaerobic conditions, thiodicarb is rapidly degraded to methomyl and then to acetonitrile and carbon dioxide.

Under sterile conditions in water, thiodicarb is more stable at lower than higher pH. It is hydrolysed to methomyl which is further degraded to methomyl oxime. Photolysis of aqueous solutions of thiodicarb yields methomyl and ultimately acetonitrile and carbon dioxide. The degradation of thiodicarb in the environment is shown in Figure 3.

METHODS OF RESIDUE ANALYSIS

Analytical methods

Hunt (1996) validated an analytical method, HPLC 3-96, for the determination of thiodicarb and methomyl in animal substrates at a limit of quantification of 0.02 mg/kg.

Residues are extracted by shaking homogenized samples with acetone/water (90:10). The extracts are purified by coagulation with ammonium chloride and phosphoric acid, followed by liquid-liquid partitioning and silica gel column chromatography. Quantification is by HPLC on a Zorbax phenyl column with fluorescence detection after post-column conversion to methylamine and derivatization. Typical retention times are 3.5 minutes for methomyl and 8.8 minutes for thiodicarb.

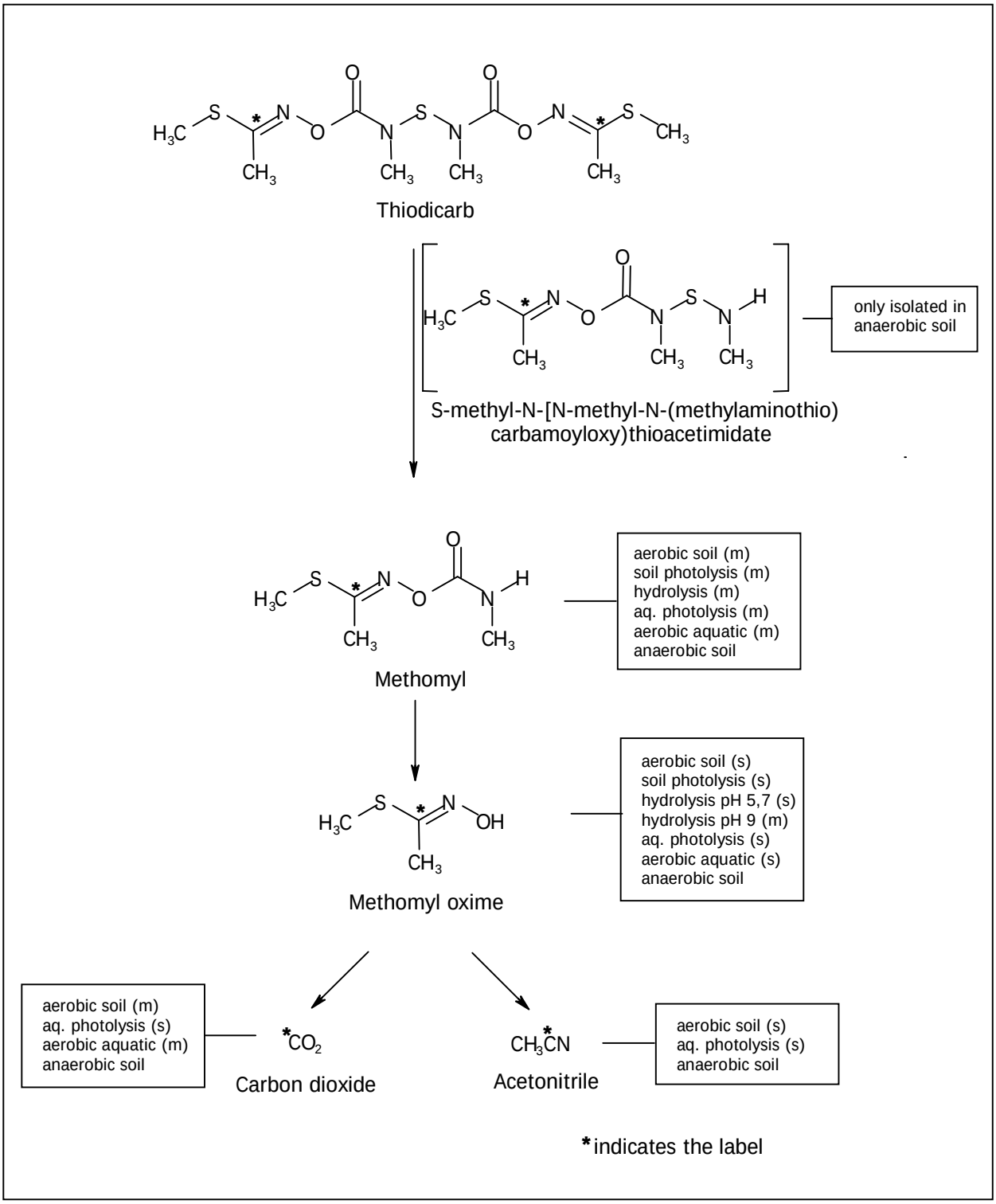
A total of 135 samples were analysed, 15 for each of the nine substrates milk, beef muscle, beef fat, beef liver, beef kidney, eggs, poultry muscle, poultry skin and fat and poultry liver. Control samples of each substrate were spiked at 0.02 and 0.10 mg/kg of thiodicarb or methomyl. The average recoveries are shown in Table 31.

Thiodicarb is very unstable in animal substrates, being degraded to methomyl during analysis. Residues of thiodicarb and methomyl were determined and reported as either total thiodicarb equivalents or total methomyl equivalents.

Table 31. Average recoveries of thiodicarb and methomyl from animal materials fortified at 0.02 and 0.10 mg/kg (Hunt, 1996).

Substrate (15 samples of each)	Thiodicarb recovery		Methomyl recovery	
	Mean %	SD %	Mean %	SD %
Milk	92	2	93	4
Beef muscle	96	9	94	3
Beef fat	82	6	99	2
Beef liver	68	5	88	11
Beef kidney	91	9	90	7
Eggs	85	6	86	2
Poultry muscle	92	2	95	3
Poultry skin and fat	80	5	98	11
Poultry liver	83	4	90	8

Figure 3. Degradation of thiodicarb in the environment.



s: minor product, <10% of the applied ^{14}C
m: major product, >10% of the applied ^{14}C
(note: although not indicated above, methomyl methylol was observed as a very minor product of aqueous photolysis)

Methods for plant commodities are similar to one another and rely upon base hydrolysis of thiodicarb and methomyl to methomyl oxime (MHTA) and determination of the MHTA by gas chromatography (Tew, 1992; Hunt and Langdon, 1982; US FDA, PAM II). These methods cannot distinguish between thiodicarb, methomyl and MHTA. In the general method SOP 90311 for thiodicarb determination developed in 1982 for a wide range of crops (Hunt and Langdon, 1982; Anon., 1982) the residues are extracted with a mixture of 9:1 acetone/water. A standard coagulation procedure is used to remove interfering co-extractives, and caustic hydrolysis converts thiodicarb and methomyl to methomyl oxime. The oxime is quantified by gas chromatography with a flame-photometric detector selective for sulfur-containing compounds.

The LOQ is 0.04 mg/kg for a 25 g sample. The average recoveries were 89% for thiodicarb and 93% for methomyl at several levels over a range of 0.04 to 10 mg/kg. The method was validated for almond hulls, almond kernels, apples, broccoli, cabbage, carrots, cauliflower, grapes, peanut hay, peanut hulls, peanut kernels, bell peppers, rice, soil (clay, clay loam, silt loam and sand), sorghum forage, sorghum grain, sorghum silage, spinach, tea, tobacco (flue-cured), wheat grain, wheat straw, cotton seed and cotton foliage.

A modification of the method, SOP 90321 (Tew, 1992), incorporates extraction of the parent and metabolites with acetone/methanol (90:10) or acetonitrile depending on the sample, and gel permeation chromatography (GPC) clean-up, before hydrolysis to methomyl oxime and quantification as above. The limit of quantification is about 0.02 mg/kg. Table 32 summarizes the recovery data.

Table 32. Recoveries from fortified samples by method SOP 90321 (Tew, 1992).

Sample	Fortification range (mg/kg)	No. of samples	Average recovery (%)	Standard deviation
Soya bean seed	0.02-1.0	35	89	11
Soya bean straw	2.8-60	11	83	8
Soya bean forage	4-60	14	74	5
Soya bean hay	12-60	14	90	17
Soya bean meal	0.04-1.0	15	99	10
Soya bean crude oil	0.04-1.0	15	90	8
Soya bean refined oil	0.04-1.0	15	86	9
Soya bean hulls	0.04-1.0	16	88	10
Soya bean soapstock	0.04-1.0	15	92	15
Soya bean grain dust	0.20	2	91	-
Cotton seed	0.02-1.2	26	89	11
Cotton straw	2.8-60	10	83	8
Cotton meal	0.04-1.0	16	93	18
Cotton crude oil	0.04-1.0	15	96	12
Cotton refined oil	0.04-1.0	15	90	12
Cotton soapstock	0.04-1.0	15	92	14
Sweet corn kernel	0.02-1.0	17	83	13
Sweet corn kernel + cob	0.02-1.0	16	102	12
Sweet corn cannery waste	0.02-1.0	16	103	7

This method was used with minor modifications for the analysis of tomatoes, sugar beet and Brussels sprouts (Moertl and Class, 1998). Mass spectrometric detection (GC-MS) was used for the determination of methomyl oxime instead of GLC with a flame photometric detector. Residues are extracted from the homogenized samples with acetone/water (9:1) and coagulation removes co-extractives. Residues in the aqueous extract are partitioned into methylene chloride, and after hydrolysis the methomyl oxime is again partitioned into methylene chloride. Methomyl oxime is quantified by measuring the sum of the characteristic ions m/e 58 + 88 + 105. The LOQ is 0.04 mg/kg. Recoveries from fortified samples of three different crops are shown in Table 33. A retention time shift problem was mentioned and thiodicarb recovery from Brussels sprouts was poor, 56%-66%.

Table 33. Recoveries from fortified control samples (Moertl and Class, 1998).

Crop	Fortification level (mg/kg)	thiodicarb		methomyl	
Tomato	0.04-0.20-0.4	73% ± 21%	n = 8	76% ± 22%	n = 8
Sugar beet	0.04-0.20-0.4	80% ± 12%	n = 7	72% ± 15%	n = 8
Brussels sprouts	0.04-0.4	60% ± 8%	n = 4	76% ± 11%	n = 4

An analytical method SOP-90318 was described for the determination of thiodicarb and methomyl in soils (Robinson, 1989). The residues are extracted with 50:50 acetone/water, then partitioned with methylene chloride. Quantification is by HPLC with post-column derivatization and fluorescence detection. The limit of quantification is 1 µg/kg (ppb) for a 50 g sample.

The method was validated with fortified soil samples over a range of 1 to 5000 µg/kg for methomyl and 1 to 2000 µg/kg for thiodicarb. The average recovery was 92% for both methomyl and thiodicarb.

In a comparison of conventional HPLC with LC-MS-MS in this method (Leonard, 1999) thiodicarb and methomyl were extracted from sandy loam and clay loam soil and partitioned as before, then analysed by HPLC with fluorescence detection. The samples were then analysed by LC-MS-MS.

Recoveries were determined with samples of sandy loam and clay loam soils from California and Iowa respectively fortified at 1 and 50 µg/kg. Recoveries by conventional HPLC were between 84% and 101% and averaged 91% ± 8% for methomyl and between 78% and 93% and averaged 86% ± 8% for thiodicarb. Very similar results were obtained from the same samples when analysed by LC-MS-MS. Recoveries of methomyl were between 82% and 91% and averaged 86% ± 5% and those of thiodicarb were between 81% and 95% and averaged 89% ± 7%.

Stability of residues in stored analytical samples

Field-treated samples of celery, head lettuce, leaf lettuce and spinach with quantifiable residues were re-analysed to determine the stability of thiodicarb during storage at -30°C (Hunt, 1988b). The results are given in Table 34.

Table 34. Stability of incurred residues of thiodicarb in leafy vegetables (Hunt, 1988b).

Crop	Celery				Head lettuce				Leaf lettuce				Spinach			
Days stored	0	152	0	235	0	176	0	499	0	100	0	202	0	162	0	486
mean residue (mg/kg)	17	17	5.1	4.6	16	14	1.4	1.1	3.2	3.1	17	14	22	16	1.9	0.99

The stability of thiodicarb in soya bean and its processed commodities during frozen storage was studied in North Carolina, USA (Lee, 1992a). Samples were fortified in duplicate and analysed for thiodicarb/methomyl after 1, 3, 6 and 12 months frozen storage at –15 ± 5°C by GLC with gel permeation clean-up. Analytical recoveries at 0.04 mg/kg were 95% for soya beans, 78% for hulls, 87% for meal, 90% for crude oil, 71% for refined oil and 76% for soapstock. The findings are shown in Table 35.

Table 35. Stability of thiodicarb/methomyl in soya beans and processed fractions stored frozen (Lee, 1992a).

Sample	Storage period (months)	% thiodicarb/methomyl remaining
Soya bean	0	91
	1	90
	3	92
	6	70
	12	56
Meal	0	100
	1	95
	3	86
	6	77
	12	60
Hulls	0	83
	1	77
	3	59
	6	79
	12	82
Crude oil	0	88
	1	75
	3	82
	6	92
	12	83
Refined oil	0	85
	1	69
	3	84
	6	83
	12	78
Soapstock	0	104

Sample	Storage period (months)	% thiodicarb/methomyl remaining
	1	95
	3	85
	6	44
	12	66

Samples of apples from field residue trials conducted in Virginia, USA in 1984 were first analysed 320 days after harvest by GLC with FPD, and again about 415 days later (Hunt, 1994). For 7-day PHI apples, the initial results were 1.5, 2.9 and 2.8 mg/kg, average 2.4 mg/kg. After the additional 415 days of storage, the corresponding values were 1.8, 3.0 and 2.8 mg/kg, average 2.5 mg/kg. For 14-day PHI apples, the initial results were 2.0, 2.0 and 1.3 mg/kg, average 1.8 mg/kg, and the corresponding final values 1.3, 1.9 and 0.89 mg/kg, average 1.4 mg/kg. The results were not corrected for concurrent analytical recoveries. It was concluded that residues of thiodicarb were stable in frozen apples for up to 14 months.

In a storage stability study on sorghum grain (Hunt, 1988e, 1989b) the field-treated grain was stored at -30°C and analysed 97, 166 and 473 days after harvest. The thiodicarb residue remained at 14 mg/kg during the 376 days between the first and third analyses.

In a companion study sorghum forage and stover were fortified at 40 and 45 mg/kg respectively and stored for approximately 6 months at -20°C (186 days for forage and 184 days for stover) (Hunt, 1989b). The results are shown in Table 36.

Table 36. Average thiodicarb residues remaining in fortified sorghum forage and stover after storage at -20°C (Hunt, 1989b).

Sample	Residue, mg/kg, after storage for				
	0 (initial sample)	1 month	2 months	3 months	6 months
Forage	40	39	33	30	33
Stover	44	44	39	38	37

The residues decreased during the 6 months of storage. The estimated half-life was calculated by first-order regression statistics to be 559 days in forage and 980 days in stover.

The stability of thiodicarb/methomyl in sweet corn and its processed commodities was determined by Lee (1991b). Samples fortified at 1 mg/kg were analysed after 0.5, 1, 3, 6, 9 and 12 months frozen storage at –15 ± 5°C by method SOP 90321, validated at 0.02 to 1.0 mg/kg for kernels, kernels plus cobs and cannery waste. The findings are shown in Table 37.

Table 37. Stability of thiodicarb/methomyl in sweet corn commodities (1 mg/kg fortification) during frozen storage (Lee, 1991b).

Sample	% of initial residue remaining after storage for (months)						
	0	0.5	1	3	6	9	12
Corn kernels	80	83	85	117	16, 57	8	56
Corn kernels + cobs	111	77	85	105	51	41	32
Cannery waste	101	94	82	113	61	66	75

A first-order kinetics model indicated half-lives of 86, 225 and 265 days for thiodicarb/methomyl in kernels, kernels plus cobs and cannery waste respectively.

In a similar study to determine the stability of thiodicarb on cotton seed and its processed commodities during storage at -15 ± 5°C (Lee, 1991a) samples were fortified with thiodicarb at a concentration of 1 mg/kg and analysed after 0, 1, 3, 6 and 12 months storage by method SOP 90321, validated at 0.04-1.0 mg/kg for all commodities. Results are shown in Table 38. Using a first-order model, the half-life of thiodicarb/methomyl was calculated to be 770 days in cotton seed, 386 days in meal and 828 days in hulls.

Table 38. Stability of thiodicarb/methomyl in fortified cotton seed commodities (1 mg/kg fortification) during frozen storage (Lee, 1991a).

Sample	% of initial residue remaining after storage for (months) [1]				
	0	1	3	6	12
Cotton seed	95	87	97	79	70
Meal	86	110	90	61	54
Hulls	94	104	106	83	76
Crude oil	90	106	91	102	87
Refined oil	98	104	106	83	76
Soapstock	87	88	88	80	98

[1] Average of duplicate samples

In a storage stability study on animal commodities (Davis *et al.*, 1996) control samples of milk, muscle, liver, kidney and fat were fortified with thiodicarb or methomyl at a level of 1 mg/kg and stored for up to 2 months at a nominal temperature of -20°C. Milk, muscle, kidney and fat samples were analysed at 0, 1 and 2 months; liver samples at 0 and 1 month and again in a separate experiment at 0 and 2 days. Analyses were by method HPLC 3-96. All control samples contained <0.1 mg/kg methomyl and thiodicarb. Results were not corrected for concurrent analytical recoveries.

Table 39. Stability of methomyl and thiodicarb in milk and tissues fortified at 1 mg/kg and stored frozen (Davis *et al.,* 1996).

Sample	Milk			Muscle			Kidney			Fat			Liver	
Storage (days)	0	31	62	0	31	60	0	43	69	0	32	63	0	2
% methomyl remaining	90	92	89	84	87	86	76	66	54	84	79	84	85, 86	0
% thiodicarb remaining	86	84	87	83	81	83	83	68	48	77	82	75	85, 63[1]	0

[1] As methomyl. No thiodicarb recovered.

Thiodicarb and methomyl appeared to be stable in milk, fat and muscle during 2 months of freezer storage. Both thiodicarb and methomyl decreased significantly in kidney, and in liver neither was detected after 2 days of storage.

Definition of the residue

The current definition is "sum of thiodicarb, methomyl and methyl hydroxythioacetimidate ('methomyl oxime'), expressed as thiodicarb". This definition is consistent with the analytes determined as methomyl oxime (MHTA) by the gas chromatography-based methods for thiodicarb. HPLC determines thiodicarb and methomyl separately and does not determine MHTA. This method has been used mainly for animal commodities to determine thiodicarb, but is the primary method for the analysis of plant commodities for methomyl.

Animal and plant metabolic studies have shown that thiodicarb is metabolized to methomyl, which is further degraded to carbon dioxide and acetonitrile. MHTA is a very minor metabolite, <0.5% in plants and absent in animals.

The recommended residue definition is "sum of thiodicarb and methomyl, expressed as methomyl". This recognizes that MHTA is a very minor metabolite and is not determined by some methods. Expressing the total residue as methomyl is consistent with combining the MRLs of thiodicarb and methomyl into a single list and recognizes that a significant proportion of the residue from the use of thiodicarb is methomyl. The practical effect is small, as the conversion factor from mg/kg thiodicarb to mg/kg methomyl is 0.92.

USE PATTERN

Tables 40-48 identify registered uses of thiodicarb. The Tables are based upon labels, and summaries and translations of labels, provided by the manufacturer. Formulation codes GB (granular bait) and RB (bait ready-to-use) are used interchangeably on labels, as are codes SG and WG.

Table 40. Registered uses on root and tuber vegetables.

Commodity	Country	Formulation	Application	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
Beet	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	2	--
	Chile	WG 800 g/kg	Spraying	0.8	--	--	14
Potatoes	Chile	WG 800 g/kg	Spraying	0.6	--	--	14
	Colombia	SC 375 g/l	Ground spray	0.38	0.1-0.19	--	--
	Ireland	RB 40 g/kg	Soil broadcast	0.2	--	--	--
	Ecuador	SC 375 g/l	Foliar spray	0.51	0.01-0.13	1	10
	Central America	SG 800 g/kg	Spray	0.24	--	--	7
		SC 375 g/l	Spray	1.9	--	--	7
		SG 800 g/kg	Spray	0.24	--	--	7
	Japan	WP 750g/kg	Spraying	1000-1500 dilution	--	5	7
	UK	RB 40 g/kg	Soil broadcast	0.2	--	1-3	21
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	21
Radish	Japan	RB 20 g/kg	Spray to base stem	0.8	--	2	45
		SC 320 g/l	Spraying	1000 dilution	--	2	21
Sugar beet	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	2	--
	Japan	SC 320 g/l	Spray	750 dilution	--	3	30
		WP 750 g/kg	Spray	1000-1500 dilution	--	3	30

Sweet potatoes	Japan	SC 320 g/l	Spraying	750 dilution	--	3	3
		WP 750g/kg	Spraying	1500 dilution	--	3	3
All crops	France	GB 40 g/kg	Soil broadcast	0.2	Not relevant	--	7

Table 41. Registered uses on leafy vegetables

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
Leafy vegetables	USA	SC 375 g/l	Spraying by air or by ground	0.84	0.225-1.87	([1])	14
Lettuce	Myanmar	SC 375 g/l	Foliar spraying	-0.93	0.23-2.07	--	14
	Japan	20 g/kg	Spraying to base stem	0.8	--	2	45
	Chile	WG 800 g/kg	Spraying	-0.8	--	--	14
Spinach	Chile	WG 800 g/kg	Spraying	0.8	--	--	14
Vegetables	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	3	21
	Norway	GB 40 g/kg	Soil broadcast	0.2	--	1	7
	Venezuela	SC 375 g/l	Spraying	0.56	--	--	15
	Western Africa	GB 40 g/kg	Soil broadcast	0.2	--	--	--
All crops	France	GB 40 g/kg	Soil broadcast	0.2	Not relevant	--	7

Table 42. Registered uses on Brassica vegetables.

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
Brassica crops	Australia	SC 375 g/l	Spray	0.75	--	--	7
		WG 800 g/kg	Spray	0.75	--	--	7
	Myanmar	SC 375 g/l	Foliar spray	0.93	--	--	7
Broccoli	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	3	21
	Chile	WG 800 g/kg	Spraying	0.8	--	--	7
	Central America	SG 800 g/kg	Spray	0.24	--	--	7
		SC 375 g/l	Spray	1.9	--	--	7
		SG 800 g/kg	Spray	0.24	--	--	7
	USA	SC 375 g/l	Air or ground spray	1.2	0.1-2.55(air) 0.225-0.575 (ground)	([1])	7
Brussels sprouts	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	6	21
	Chile	WG 800 g/kg	Spraying	0.8	--	--	7
Cabbage	China	WP 750 g/kg	Spray	0.75	0.17	--	--
	Central America	SG 800 g/kg	Spray	0.24	--	--	7
		SC 375 g/l	Spray	1.87	--	--	7

[1] Do not exceed 1.7 kg/ha per season

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
		SG 800 g/kg	Spray	0.24	--	--	7
	Chile	WG 800 g/kg	Spraying	0.8	--	--	7
	Ecuador	SC 375 g/l	Foliar	0.26	0.07	1	10
Cabbage	India	WP 750 g/kg	Spray	1	0.2	2-3([1])	7
	Japan	SC 320 g/l	Spraying	--	750-1000 dil.	4	7
		WP 750 g/kg	Spraying	--	100-1500 dil	4	7
	Pakistan	WG 800g/kg	Foliar	0.6	0.16-0.24	1	--
	Taiwan	WP 750 g/kg	Foliar spray	0.5	--	([2])	6
	USA	SC 375 g/l	air or ground Spray	1.2	0.1-2.55(air) 0.225-0.575 (ground)	([3])	7
Chinese cabbage	Japan	20 g/kg	Spraying to base stem	0.8	--	2	45
		SC 320 g/l	Spraying	--	750-1000 dil.	4	7
		WP 750 g/kg	Spraying	--	1000-1500 dil.	4	7
Cauliflower	Chile	WG 800 g/kg	Spraying	0.8	--	--	7
	Central America	SG 800 g/kg	spray	0.24	--	--	7
		SC 375 g/l	Spray	1.9	--	--	7
		SG 800 g/kg	Spray	0.24	--	--	7
	USA	SC 375 g/l	Air or ground spray	1.2	0.1-2.55(air) 0.225-0.575 (ground)	([4])	7
Vegetables	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	6	21
	Norway	GB 40 g/kg	Soil broadcast	0.2	--	1	7
	Western Africa	GB 40 g/kg	Soil broadcast	0.2	--	--	--
	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	6	21
All crops	France	GB 40 g/kg	Soil broadcast	0.2	Not relevant	--	7

Table 43. Registered uses on legume vegetables.

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
Beans	Brazil	FS 350 g/l	Seed treatment	0.52	--	1	--
	Ecuador	FS 350 g/l	Seed treatment	0.70	--	--	--
		SC 375 g/l	Foliar	0.23	0.05-0.08	1	15
	Central America	FS 350 g/l	Seed treatment	0.76	--	--	--
		FS 300 g/l	Seed treatment	0.3	--	--	--
	Paraguay	FS 350 g/l	Seed treatment	0.525	--	--	--

[1] 7 to 10 days interval depending on the pest intensity

[2] every 7 days

[3] Do not exceed 6.7 kg/ha per season

[4] Do not exceed 6.7 kg/ha per season

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
	Peru	FS 350 g/l	Seed treatment		--	--	28
		SC 375 g/l	Foliar		0.09-0.375	1	14
	Sri Lanka	SC 375 g/l	Spray	0.38	--	--	14
	Venezuela	FS 350 g/l	Seed treatment	0.7	--	--	--
Peas	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	1	--
Pigeon peas	Sri Lanka	SC 375 g/l	Spray	0.47	--	--	14
Pulses: Soya beans Mung beans, Chick-peas, Pigeon peas, Navy beans	Australia	SC 375 g/l	Aerial/ground	0.28	0.375-1.4	--	21
		WG 800 g/kg	Aerial/ground	0.28	0.375-1.4	--	21
	Myanmar	SC 375 g/l	Foliar application	0.46	0.08-0.5	--	21
Soya beans	Argentina	SC 375 g/l	Aerial/ ground	0.11	0.28-1.12 0.028-0.1125	1-2	20
		WG 800 g/kg	Aerial/ ground	0.092	0.2-0.95 0.02-0.095	1-2	20
		FS 350 g/l	Seed treatment	0.14	--	--	--
	Brazil	WG 800 g/kg	Aerial/ ground	0.056	0.56-1.12 (air) 0.028-0.056 (ground)	--	14
	Ecuador	FS 350 g/l	Seed treatment	0.70	--	--	--
		SC 375 g/l	Foliar	0.26	0.04-0.07	1	15
	Central America	SG 800 g/kg	spray	0.24	--	--	28
		SC 375 g/l	spray	1.9	--	--	28
		SG 800 g/kg	spray	0.24	--	--	28
		FS 350 g/l	Seed treatment	0.76	--	--	--
		FS 300 g/l	Seed treatment	0.3	--	--	--
	Indonesia	SC 375 g/l	Foliar spray	-0.3	0.02-0.08	--	14
		WP 750 g/kg	Spraying	1.4	0.15-0.22	2	14
	Japan	WP 750 g/kg	Spraying	750 dilution	--	2	14
Soya beans	Paraguay	WG 800 g/kg	Spray	0.12	--	--	14
	Mexico	SC 350 g/l	Seed treatment	0.88	--	--	--
	Thailand	SC 375 g/l	Foliar spray	0.21	0.11	1-2	28
	USA	SC 375 g/l	Spraying by air or by ground	0.84	0.6-8.4 (air) 0.14-0.42 (ground)	([1])	28
	Venezuela	SC 375 g/l	Spray	0.38	--	--	28
		FS 350 g/l	Seed treatment	0.7	--	--	--
All crops	France	GB 40 g/kg	Soil broadcast	0.2	Not relevant	--	7

[1] Do not exceed 3.4 kg/ha per season

Table 44. Registered uses on fruiting vegetables.

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
Sweet corn	USA	SC 375 g/l	Spraying by air or by ground	0.84	Max 2.94 (air) 0.4-3 (ground)	([1])	0
	Australia	SC 375 g/l	Spraying by air or by ground	0.75	1.12-1.5	--	7
		WG 800 g/kg	Spraying by air or by ground	0.75	1.12-3.75	--	7
		FS 500 g/l	Seed treatment	0.75	--	--	--
Tomato	Australia	SC 375 g/l	Spraying	0.52 (a)	0.13(a) 0.026 (b)	--	1
		WG 800 g/kg	Spraying	0.52 (a)	0.13 (a) 0.026 (b)	--	1
	Chile	WG 800 g/kg	Spraying	0.8	--	--	3
	Central America	SG 800 g/kg	Spray	0.24	--	--	7
		SC 375 g/l	Spray	1.9	--	--	7
		SG 800 g/kg	Spray	0.24	--	--	7
	Ecuador	FS 350 g/l	Seed treatment	2 l/q seeds	--	--	--
		SC 375 g/	Foliar	0.26	--	1	10
	Myanmar	SC 375 g/l	Foliar application	0.55	0.12-0.61	--	21
	Peru	SC 375 g/l	Spraying	0.75	--	1	14
	Spain	SC 375 g/l	Upward foliar spray	0.94	--	--	7
	Taiwan	SC 375 g/l	Foliar spray	0.56	--	([2])	3
		WP 750 g/kg	Foliar spray	0.25	--	--	3
	Thailand	SC 375 g/l	Foliar spray	--	0.11	NS	28

(a) low spray volume
(b) high spray volume
NS: not specified

Table 45. Registered uses on pome fruits.

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
Apple	Japan	SC 320 g/l	Spraying	750-1000 dil.	--	3	21
		WP 750 g/kg	Spraying	1000-1500 dil.	--	3	21
	South Korea	WP 750 g/kg	Foliar Spraying	3.4	0.075	3	21
	Romania	SC 375 g/l	Foliar spraying	0.56	0.038	--	15
Pear	Japan	SC 320 g/l	Spraying	750 dilution	--	3	7
		WP 750 g/kg	Spraying	1000-1500 dil.	--	3	7
Pome fruits	Chile	WG 800 g/kg	Spray	2	--	--	--
	Portugal	GB 40 g/kg	Soil broadcast	0.2	--	1	--

[1] Do not exceed 8.4 kg/ha per season, 1 to 7 day intervals.

[2] every 7 days

Table 46. Registered uses on small fruits and berries.

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
Grape	France	SC 375 g/l	Foliar spray	0.45	0.094-0.45	1-2	14
		SC 300 g/l	Upward foliar spray	0.45	0.094-0.45	1-2	45
	Greece	SC 375 g/l	Foliar spray	0.46	0.02-0.09	2-3	21
	Portugal	GB 40 g/kg	Soil broadcast	0.2	--	1	--
	Spain	SC 375 g/l	Foliar spray	0.75	--	--	21
	Taiwan	WP 750 g/kg	Foliar spray	0.5	0.01-0.02	([1])	14
	Myanmar	SC 375 g/l	Foliar application	0.93	0.2-1	--	7
	Romania	SC 375 g/l	Foliar spraying	0.38	0.038	--	15

Table 47. Registered uses on cereal grains.

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI, days
Barley	Ireland	RB 40 g/kg	Soil broadcast	0.2	--	--	--
	Norway	GB 40 g/kg	Soil broadcast	0.2	--	1	7
	UK	RB 40 g/kg	Admixture	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Admixture with seed	0.2	--	1	--
Cereals	Portugal	GB 40 g/kg	Soil broadcast	0.2	--	1	--
	Netherlands	GB 40 g/kg	Strew on the soil	0.2	--	2	--
Cereals (All crops)	France	GB 40 g/kg	Soil broadcast or admixture with seed	0.2	Not relevant	--	7
Cereals (except maize)	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	2	--
Cereals (winter)	Germany	GB 40 g/kg	Soil broadcast	0.2	--	1-2	--
Maize	Argentina	FS 350 g/l	Seed treatment	0.70	--	1	--
	Ecuador	FS 350 g/l	Seed treatment	0.70	--	--	--
		SC 375 g/l	Foliar spraying	0.38	--	1-2	15
	Central America	SG 800 g/kg	Spray	0.24	--	--	0
		SC 375 g/l	Spray	1.9	--	--	0
		SG 800 g/kg	Spray	0.24	--	--	0
		GR 75 g/kg	Soil broadcast	0.52	--	--	10
		FS 350 g/l	Seed treatment	0.76	--	--	--
		FS 300 g/l	Seed treatment	0.3	--	--	--
	Paraguay	WG 800 g/kg	Spray	0.8	--	--	14
		FS 350 g/l	Seed treatment	0.70	--	--	--
		FS 300 g/l	Seed treatment	0.60	--	--	--

[1] every 7 days

	Mexico	SC 350 g/l	Seed treatment	1.4	--	-	--
		SC 375 g/l	Aerial/ground	0.47	--	--	--
		SC 300 g/l	Seed treatment	1.5	--	--	--
	Philippines	FS 350 g/l	Seed treatment	0.49	--	--	--
	Turkey	WG 800 g/kg	Foliar spraying	0.72	--	--	28
Maize	Australia	SC 375 g/l	Spraying by air or ground	0.75	1.12 (air) 1.5 (ground)	--	7
		WG 800 g/kg	Spraying by air or ground	0.75	1.12 (air) 3.75 (ground)	--	7
		FS 500 g/l	Seed treatment	0.75	--	--	--
	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	2	--
	Brazil	FS 350 g/l	Seed treatment	0.7	--	1	--
		WG 800g/kg	Aerial/ground	0.1	0.5-1 (air) 0.03-0.0 (ground)	--	30
		300 g/l	Seed treatment	0.6	--	1	--
	Chile	FS 350 g/l	Seed treatment	1.0	--	--	--
		WG 800 g/kg	Spray	0.6	--	--	14
	Indonesia	SC 375 g/l	Foliar spray	0.77	0.154	--	14
		WP 750 g/kg	Seed treatment	1.5	-	--	--
	Myanmar	SC 375 g/l	Foliar application	0.93	--	--	7
	Peru	FS 350 g/l	Seed treatment	0.35	--	--	28
		SC 375 g/l	Spraying	0.38	0.14-0.09	1	14
	South Africa	SC 375 g/l	Ground or aerial spray	0.3	--	--	21
	Spain	SC 375 g/l	Upward foliar spray	0.94	--	--	21
	Venezuela	FS 300 g/kg	Seed treatment	0.75	--	--	--
		SC 375 g/l	Spray	0.38	--	--	28
		FS 320 g/l	Seed treatment	0.64	--	--	--
Oats	Ireland	RB 40 g/kg	Soil broadcast	0.2	--	--	--
	UK	RB 40 g/kg	Admixture	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Admixture with seed	0.2	--	1	--
Rice	Brazil	FS 350 g/l	Seed treatment	0.52	--	1	--
		300 g/l	Seed treatment	0.45	--	1	--
	Ecuador	FS 350 g/l	Seed treatment	0.70	--	--	--
		SC 375 g/l	Foliar spraying	0.38	0.05-0.125	1	15
	Japan	DP 30 g/kg	Spraying	1.2	--	3	30
	Paraguay	FS 350 g/l	Seed treatment	0.52	--	--	--
		FS 300 g/l	Seed treatment	0.45	--	--	--
Rye	Norway	GB 40 g/kg	Soil broadcast	0.2	--	1	7
Sorghum	Australia	FS 500 g/l	Seed treatment	0.5	--	--	--
	Colombia	FS 350 g/l	Seed treatment	0.52	--	--	--
		SC 375 g/l	Foliar pray	0.38	0.94-1.25	--	--

	Ecuador	SC 375 g/l	Foliar pray	0.38	--	1-2	15
	Central America	GR 75 g/kg	Soil broadcast	0.52	--	--	10
		FS 350g/l	Seed treatment	0.76	--	1	--
		FS 300 g/l	Seed treatment	0.3	--	--	--
	Mexico	FS 350 g/l	Seed treatment	1.4	--	--	--
		SC 300 g/l	Seed treatment	1.5	--	--	--
	Thailand	SC 375 g/l	Foliar spray	--	0.094	--	28
	Venezuela	SC 375 g/l	Spray	0.38	--	--	14
		FS 320 g/l	Seed treatment	0.64	--	--	--
Sweet corn	USA	SC 375 g/l	Spraying by air or by ground	0.84	Max 2.94 (air) 0.4-3 (ground)	([1])	0
	Australia	SC 375 g/l	Spraying by air or by ground	0.75	1.12-1.5	--	7
		WG 800 g/kg	Spraying by air or by ground	0.75	1.12-3.75	--	7
		FS 500 g/l	Seed treatment	0.75	--	--	--
Triticale	UK	RB 40 g/kg	Admixture with seed	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Admixture with seed	0.2	--	1	--
Wheat	Argentina	FS 350 g/l	Seed treatment	0.14		1	--
	Chile	FS 350 g/l	Seed treatment	0.14	--	--	--
	Ecuador	FS 350 g/l	Seed treatment	0.70	--	--	--
	Central America	FS 350 g/l	Seed treatment	0.76	--	--	--
		FS 300 g/l	Seed treatment	0.3	--	--	--
	Ireland	RB 40 g/kg	Soil broadcast	0.2	--	--	--
	Norway	GB 40 g/kg	Soil broadcast	0.2	--	1	7
	UK	RB 40 g/kg	Admixture	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Admixture with seed	0.2	--	1	--
Wheat (durum)	UK	RB 40 g/kg	Admixture with seed	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Admixture with seed	0.2	--	1	--

Table 48. Registered uses on oilseed.

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI , days
Cotton	Argentina	FS 350 g/l	Seed treatment	0.7	--	1	-
		SC 375 g/l	Aerial/ground	0.3	1.1-3 (air) 0.11-0.3 (ground)	1	20

[1] Do not exceed 8.4 kg/ha per season, 1 to 7 day intervals.

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI , days
		WG 800 g/kg	Aerial/ground	0.38	0.95-3.75 (air) 0.1-0.375 (ground)	1-2	20
	Australia	SC 375 g/l	Aerial/ground	0.94	3.75-4.7 (air) 1.5-1.9 (ground)	--	21
		WG 800 g/kg	Aerial/ground	0.96	3.75-4.8 (air) 1.5-1.9 (ground)	--	21
		FS 500 g/l	Seed treatment	0.5	--	--	--
		FS 400 g/l	Seed treatment	0.25	--	--	--
	China	SC 375 g/l	Spray	0.51	0.675-1.69	3	14
		WP 750 g/kg	Spray	0.51	0.675-1.69	3	14
	Colombia	FS 350 g/l	Seed treatment	0.70	--	--	--
		SC 375 g/l	Airway	0.56	0.7-1.86	--	--
	Ecuador	FS 350 g/l	Seed treatment	0.7	--	--	--
		SC 375 g/l	Foliar spraying	0.38	--	1	15
	Egypt	SC 375 g/l	Spraying	0.89	--	--	28
		WG 800 g/kg	Spraying	0.95	--	--	28
	Greece	WG 800 g/kg	Foliar spray	0.8	0.06-0.16	2-3	28
	Central America	SG 800 g/kg	Spray	-0.24	--	--	28
		SC 375 g/l	Spray	1.8	--	--	28
		SG 800 g/kg	Spray	0.24	--	--	28
		FS 300 g/l	Seed treatment	0.3	--	--	--
		FS 350 g/l	Seed treatment	0.76	--	--	--
	India	WP 750 g/kg	Spray	0.75	0.15	3 -4[1]	30
Cotton	Indonesia	SC 375 g/l	Foliar spray	0.38	0.04-0.08	--	14
		WP 750 g/kg	Foliar spray	1.5	0.19-0.5	--	14
	Iran	WG 800g/kg	Foliar spraying	-0.8	--	--	--
	Mexico	SC 350 g/l	Seed treatment	0.75	--	--	--
		SC 375 g/l	Foliar spraying	0.94	--	--	--
	Myanmar	SC 375 g/l	Foliar spraying	0.93	0.2-1	--	21
	Pakistan	WG 800g/kg	Spraying	0.9	0.27-0.36	1	--
	Paraguay	WG 800 g/kg	Spray	0.048	0.01-0.005	2-3	28
	Peru	FS 350 g/l	Seed treatment	0.35	--	--	28
		SC 375 g/l	Foliar spraying	0.094	0.01-0.02	1	14
	South Africa	SC 375 g/l	Ground or aerial spray	0.38	1.25 (air) 0.19 (ground)	--	21
	Spain	SC 375 g/l	Upward foliar spray	0.94	--	--	21
	Thailand	SC 375 g/l	Foliar spray	0.21	0.11	--	28
	Turkey	WG 800g/kg	Foliar spraying	0.72	--	--	28
	USA	SC 375 g/l	Spraying by air or by ground	1	0.1-5	--	28
	Zimbabwe	SC 375 g/l	Aerial/ground	0.41	1-8.2 (air) 0.14-0.41 (ground)	--	--
Oily crops	Portugal	GB 40 g/kg	Soil broadcast	0.2	--	1	--

[1] 10 to 15 days interval depending on the pest intensity

Commodity	Country	Formulation	Method	Rate kg ai/ha or /100 kg seed	Spray conc., kg ai/hl	No. of appl.	PHI , days
Oilseed rape	Ireland	GB 40 g/kg	Soil broadcast	0.2	--	--	--
	Belgium	GB 40 g/kg	Soil broadcast	0.2	--	2	--
	UK	RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
		RB 40 g/kg	Soil broadcast	0.2	--	1-3	--
Oilseed rape (winter)	Germany	GB 40 g/kg	Soil broadcast	0.2	--	1-2	--
	Netherlands	GB 40 g/kg	Soil broadcast	0.2	--	2	--
Oilseed rape (All crops)	France	GB 40 g/kg	Soil broadcast or admixture with seed	0.2	Not relevant	--	7

RESIDUES RESULTING FROM SUPERVISED TRIALS

Supervised field trials were reported for numerous commodities. Where results were given as ND (not detected) and a substantiated value for ND was not supplied, the ND was assigned 50% of the limit of quantification. Unless indicated otherwise, residues are the sum of thiodicarb and methomyl. Trials are listed in the following Tables. Residues from trials according to GAP are underlined and were used in estimating maximum residue levels.

Table no.	Commodity
49	Apples (foliar)
50	Grape (foliar)
51	Potato (bait)
52	Potato (foliar)
53	Sugar beet (bait)
54	Lettuce (bait)
55	Lettuce (foliar)
56	Lettuce (aerial)
57	Spinach (foliar)
58	Collards
59	Broccoli (foliar)
60	Broccoli (aerial)
61	Brussels sprouts (bait)
62	Cabbage (foliar)
63	Cabbage (aerial)
64	Cauliflower (foliar)
65	Cauliflower (aerial)
66	Garden peas (foliar)
67	Pea hay (foliar)
68	Chick-peas (foliar)
69	Chick-pea forage (foliar)
70	Chick-pea straw
71	Soya bean (foliar)
72	Soya bean (aerial)
73	Soya bean (seed treatment)
74	Soya bean forage (foliar)
75	Soya bean forage (seed treatment)
76	Soya bean hay (foliar)
77	Soya bean straw (foliar)
78	Tomato (foliar)

Table no.	Commodity
79	Sweet corn (kernel + cob with husk removed)(foliar)
80	Sweet corn (aerial)
81	Sweet corn (seed treatment)
82	Barley and wheat grain (bait)
83	Wheat (seed treatment)
84	Maize (foliar)
85	Maize (seed treatment)
86	Rice
87	Rice (seed treatment)
88	Sorghum (foliar)
89	Sorghum (seed treatment)
90	Barley and wheat forage (bait)
91	Barley and wheat forage (bait)
92	Barley and wheat straw (bait)
93	Barley and wheat forage (foliar)
94	Rice straw (foliar)
95	Sorghum forage (foliar)
96	Sorghum forage (seed treatment)
97	Sorghum straw (foliar)
98	Sorghum stover (foliar)
99	Sorghum stover (seed treatment)
100	Sweet corn fodder (foliar)
101	Sweet corn forage (foliar)
102	Sweet corn forage (seed treatment)
103	Cotton seed (foliar)
104	Cotton seed delinted (foliar)
105	Cotton seed (aerial)
106	Cotton seed (seed treatment)
107	Cotton leaves (foliar)

Table no.	Commodity
108	Cotton forage (foliar)
109	Cotton forage (aerial)
110	Cotton forage (seed treatment)

Table no.	Commodity
111	Rape seed (bait)
112	Rape forage (green) (bait)
113	Rape straw (bait)

Pome fruit

Supervised trials were conducted on apples in Australia, USA, Italy, Greece and Japan (Table 49). GAP exists only in Japan.

The samples from Japan were analysed by a GLC method, with an LOQ of 0.02 mg/kg and a stated limit of detection of 0.005 mg/kg (Anon., 2001a).

Table 49. Residues in apples (foliar application, ground equipment).

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Fennville, Michigan USA 1982	SC, 375 g/l	8	1.1		7 14 21	0.78, 0.64 0.74, 0.74 0.15, 0.30	Hunt, 1989a, File 40674, Project 804R10
Fennville, Michigan USA 1982	SC, 375 g/l	8	2.2		7 14 21	1.6, 1.3 3.0, 2.2 0.73, 0.66	Hunt, 1989a, File 40674, Project 804R10
Clayton, North Carolina USA 1982	SC, 375 g/l	8	1.1	0.11	3 7 14	0.60, 0.74 0.36, 0.56 0.22, 0.27	Hunt, 1989a, File 40674, Project 804R10
Clayton, North Carolina USA 1982	SC, 375 g/l	8	2.2	0.11	3 7 14	2.7, 2.2 2.6, 2.0 1.6, 0.80	Hunt, 1989a, File 40674, Project 804R10
Sodus, New York USA 1982	SC, 375 g/l	8	1.1	0.20	3 7 14 21	1.1 0.95 0.53 0.28	Hunt, 1989a, File 40674, Project 804R10
Sodus, New York USA 1982	SC, 375 g/l	8	2.2	0.20	3 7 14 21	3.6 1.2 0.78 0.77	Hunt, 1989a, File 40674, Project 804R10
Fennville, Michigan USA 1985	SC, 375 g/l	8	1.1	0.29	7 14 21	1.9 1.1 0.48	Hunt, 1989a, File 40674, Project 804R10
Sodus, New York USA 1985	SC, 375 g/l	8	1.1	0.03	7 14 21	0.64 0.50 0.70	Hunt, 1989a, File 40674, Project 804R10
Winchester, Virginia USA 1985	SC, 375 g/l	8	1.1		6 16 21	1.5 1.3 1.1	Hunt, 1989a, File 40674, Project 804R10
Kearneysville, West Virginia 1985	SC, 375 g/l	8	1.1	0.12	7 14 21	0.89 0.35 0.17	Hunt, 1989a, File 40674, Project 804R10
El Dorado, California USA 1986	SC, 375 g/l	1	1.1	0.08	90	0.20, 0.14	Hunt, 1989a, File 40674, Project 804R10
Manteca, California USA 1986	SC, 375 g/l	1	1.1	0.12	88	0.44, 0.78	Hunt, 1989a, File 40674, Project 804R10
Yakama, Washington USA 1986	SC, 375 g/l	1	1.1	0.04	92	0.16, 0.14, 0.19, 0.09	Hunt, 1989a, File 40674, Project 804R10
Manteca, California USA 1987	SC, 375 g/l	1	1.1	0.08	91	0.88, 0.84	Hunt, 1989a, File 40674, Project 804R10

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Bathurst, New South Wales Australia, 1982	SC 375 g/l	8	--	0.05	0 7 14 22	3.2 1.9 3.0 4.4 2.6 1.5 3.1 4.0 3.6 2.2 1.7 3.0 2.5 2.2 2.6 4.0	Hunt, 1983 Project No.20566 File No.1451V
Bathurst, New South Wales Australia, 1982	SC 375 g/l	8	--	0.1	0 7 14 22	9.2 6.5 6.9 9.6 6.8 5.8 6.2 6.0 6.7 4.4 5.7 7.8 5.7 3.8 4.3 7.0	Hunt, 1983 Project 20566 File 1451V
Corticella, Bologna Italy, 1989	SC 375 g/l	1	0.51	0.15	0 7 14 21 28 35	0.87 0.44 0.42 0.27 0.30 0.17	Muller 1990a AG/CRLD/AN/9015940
Naoussa Macedonia Greece, 1994	WG 800 g/kg	2	1.6		42	0.16 0.23	Richard and Muller, 1995b Study 94-689
Naoussa Macedonia Greece, 1994	WG 800 g/kg	2	1.6		17	0.22 0.22	Richard and Muller, 1995b Study 94-689
Naoussa Macedonia Greece, 1994	WG 800 g/kg	2	1.6		32	0.57 0.40	Richard and Muller, 1995b Study 94-689
GAP, Japan	SC 320 g/l	3		0.032-0.042	21		
Japan, 1990	SC 375 g/l	3	2.5	0.05	14 21 28	0.562 <u>0.676</u> 0.392	Anon., AventisCrop Science, 2001a Iwate Prefecture Study No.Saku3p-2-69
Japan, 1990	SC 375 g/l	3	2.5	0.05	14 21 28	0.472 <u>0.305</u> 0.346	Anon., AventisCrop Science, 2001a Nagano Prefecture Study No.Saku3p-2-69
Japan, 1990	SC 375 g/l	3	2.5	0.05	14 21 28	0.720 <u>0.612</u> 0.406	Anon., AventisCrop Science, 2001a Iwate Prefecture Study No.Saku3p-2-69
Japan, 1990	SC 375 g/l	3	2.5	0.05	14 21 28	0.680 <u>0.317</u> 0.662	Anon., AventisCrop Science, 2001a Nagano Prefecture Study No.Saku3p-2-69

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, Japan	WP 750 g/kg	3		0.05-0.075	21		
Japan, 1985	WP 750 g/kg	3	2.5	0.05	21 28 44	0.676 0.724 0.322	Anon., AventisCrop Science, 2001a Nagano Prefecture Saku61p-4-106
Japan, 1985	WP 750 g/kg	3	3.75	0.075	21 28 44	1.46 1.76 0.159	Anon., AventisCrop Science, 2001a Nagano Prefecture Saku61p-4-106
Japan, 1985	WP 750 g/kg	3	2.5	0.05	21 28 44	0.370 0.839 0.410	Anon., AventisCrop Science, 2001a Toyama Prefecture Saku61p-4-106
Japan, 1985	WP 750 g/kg	3	3.75	0.075	21 28 44	0.682 1.52 0.472	Anon., AventisCrop Science, 2001a Toyama Prefecture Saku61p-4-106
Japan, 1985	WP 750 g/kg	3	2.5	0.05	21 28 44	0.390 0.176 0.165	Anon., AventisCrop Science, 2001a Nagano Prefecture Nissan Chemical Industry
Japan, 1985	WP 750 g/kg	3	3.75	0.075	21 28 44	0.481 0.177 0.235	Anon., AventisCrop Science, 2001a Nagano Prefecture Nissan Chemical Industry
Japan, 1985	WP 750 g/kg	3	2.5	0.05	21 28 45	0.375 0.170 0.069	Anon., AventisCrop Science, 2001a Toyama Prefecture Nissan Chemical Industry
Japan, 1985	WP 750 g/kg	3	3.75	0.075	21 28 45	0.430 0.234 0.054	Anon., AventisCrop Science, 2001a Toyama Prefecture Nissan Chemical Industry
Japan, 1982	WP 750 g/kg	3	3.75	0.075	7 14 21	17.1 1.28 0.91	Anon., AventisCrop Science, 2001a Aomori Prefecture Saku57p-8-121
Japan, 1982	WP 750 g/kg	3	3.75	0.075	7 14 21	25.5 1.71 0.91	Anon., AventisCrop Science, 2001a Nagano Prefecture Saku57p-8-121
Japan, 1982	WP 750 g/kg	3	3.75	0.075	7 14 21	1.55 1.30 1.56	Anon., AventisCrop Science, 2001a Aomori Prefecture Saku57p-8-1216
Japan, 1982	WP 750 g/kg	3	3.75	0.075	7 14 21	0.622 0.712 0.403	Anon., AventisCrop Science, 2001a Nagano Prefecture Saku57p-8-1216

Small fruits and berries

Results of supervised trials on grapes in France, Spain and Italy are shown in Table 50.

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Table 50. Residues in grapes from foliar applications.

Location, year	Application				Residues		Reference/Comments
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, France	SC 375 g/l SC 300 g/l	1-2	0.375-0.45	0.094-0.45	14 45		SC 300 g/l also contains bifenthrin
Languedoc France, 1983	SC 375 g/l	1	0.38	0.038	1 7 14 20	1.2 0.9 0.7 0.5	Barciet, 1990
Languedoc France, 1983	SC 375 g/l	1	0.38	0.038	6	0.65 1.3	Barciet, 1990
Languedoc France, 1983	SC 375 g/l	1	0.45	0.045	6	1.3 2.8	Barciet, 1990
Languedoc France, 1981	SC 375 g/l	1	0.45 calculated	0.045 and 1000/ha	84	<0.02	Cooper and Mestres, 1982 UC 51-702
Languedoc France, 1981	SC 375 g/l	2	0.45	0.045	38	0.15	Cooper and Mestres, 1982 UC 51-702
Bordeaux region Burgundy France, 1982	SC 375 g/l	2	0.45	0.045	22	<0.02	Cooper and Mestres, 1982 UC 51-702
France, 1982	SC 375 g/l	1	0.45	0.045	60	0.04	Cooper and Mestres, 1982 UC 51-702
Colombiers Languedoc France, 1982	SC 375 g/l	1	0.3		0 1 2 4 7 14 20	0.9 1.2 0.9 0.7 0.9 0.7 0.5	Cooper and Mestres, 1983
Colombiers France, 1988	SC 375 g/l	2	0.38	0.12	35	0.66	Lusson and Muller, 1989a AG/CRLD/AN/8916392
Pouzolles France, 1988	SC 375 g/l	2	0.38	0.12	35	0.22	Lusson and Muller, 1989a AG/CRLD/AN/8916392
Coustellet France, 1988	SC 375 g/l	2	0.38	0.25	54	<0.08	Lusson and Muller, 1989a AG/CRLD/AN/8916392
Mazan France, 1992	SC 375 g/l	3	0.38	0.14	49	<0.05	Richard and Muller, 1994a Study No.92-147
Mazan France, 1992	SC 375 g/l	3	0.56	0.14	49	0.16	Richard and Muller, 1994a Study No.92-147
Pouzolles France, 1992	SC 375 g/l	3	0.38	0.12	45	<0.05	Richard and Muller, 1994a Study No.92-147
Pouzolles France, 1992	SC 375 g/l	3	0.56	0.18	45	0.11	Richard and Muller, 1994a Study No.92-147
Bram France, 1995	SC 300 g/l	2	0.30	0.023 0.062	40	0.14 0.44	Maestracci, 1997a Study 95-541
Caromb France, 1995	SC 300 g/l	2	0.31	0.10	49	0.10	Maestracci, 1997b Study 95-540
Marignac France, 1995	SC 300 g/l	2	0.30	0.20	31	0.32	Maestracci, 1997c Study 95-539
Corticella, Bologna Italy, 1989	SC 375 g/l	1	0.51	0.051	0 7 14 21 28 35	1.29 0.96 0.59 0.64 0.30 0.27	Muller, 1990b Ref. AG/CRLD/AN/9015941
GAP, Spain	SC 375 g/l	--	0.375-0.75		21		
Camarena Spain, 1993	SC 375 g/l	2	0.56	0.17	26	0.60 2.21	Richard and Muller, 1994b Study No.93-624
Requena Spain, 1993	SC 375 g/l	2	0.55	0.071	64	1.08 1.01	Richard and Muller, 1994b Study No.93-624

Root and tuber vegetables

Supervised trials were conducted on potatoes in the UK (Table 51) and Japan (Table 52) and on sugar beet in the UK (Table 53).

Table 51. Residues in potatoes from trials in the UK (bait application).

Location, year	Application				Residues		Reference
	Formulation	No	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, UK	RB 40 g/kg	1-3	0.2	--	21	--	
Swillington, 1992	GB 40 g/kg	3	0.2		9 34	<0.04 <0.04 <0.04 <0.04 <0.04 <0.04	Maycey *et al.*, 1993. Study No.P92/218 Doc No.200163
Todcaster, 1992	GB 40 g/kg	3	0.2		9 38	<0.04 <0.04 <0.04 <0.04 <0.04 <0.04	Maycey *et al.*, 1993. Study No.P92/218 Doc No.200163
Driffield, 1992	GB 40 g/kg	3	0.2		8 28	<0.04 <0.04 <0.04 <0.04 <0.04 <0.04	Maycey *et al.*, 1993. Study No.P92/218 Doc No.200163
Helmsley, 1993	GB 40 g/kg	3	0.2		7	<0.040 <0.040 <0.040	Anderson-Taylor, 1996a Study No.RES/93/007 Doc No.201126
Swillington, 1993	GB 40 g/kg	3	0.2		7	<0.040 <0.040 <0.040	Anderson-Taylor, 1996a Study No.RES/93/007 Doc No.201126
Wistow, 1993	GB 40 g/kg	3	0.2		7	<0.040 <0.040 <0.040	Anderson-Taylor, 1996a Study No.RES/93/007 Doc No.201126
Goole, 1993	GB 40 g/kg	3	0.2		7	<0.040 <0.040 <0.040	Anderson-Taylor, 1996a Study No.RES/93/007 Doc No.201126
North Walsham UK, 1994	GB 40 g/kg	3	0.2		7	<0.040 <0.040 <0.040	Anderson-Taylor, 1996b Study No.RES/94/007 Doc No.201127
Bourne, 1994	GB 40 g/kg	3	0.2		7	<0.040 <0.040 <0.040	Anderson-Taylor, 1996b Study No.RES/94/007 Doc No.201127
Great Barford UK, 1994	GB 40 g/kg	3	0.2		7	<0.040 <0.040 <0.040	Anderson-Taylor, 1996b Study No.RES/94/007 Doc No.201127
North Walsham UK, 1994	GB 40 g/kg	3	0.2		8	<0.040 <0.040 <0.040	Anderson-Taylor, 1996b Study No.RES/94/007 Doc No.201127

In the Japanese trials, tubers were analysed by GLC with FPD in the sulfur mode (limit of detection 0.008, 0.007 mg/kg; LOQ not reported).

Table 52. Residues in potatoes from trials in Japan, 1985.

Application				Residues		Reference
Formulation	No	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP Japan WP 750 g/kg	5	--	0.05-0.075	7		
WP 750 g/kg	5		0.075	7 14	<0.008 <0.008	Imose, 1986
WP 750 g/kg	5		0.075	7 14	<0.008 <0.008	Imose, 1986

Application				Residues		Reference
Formulation	No	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
WP 750 g/kg	5		0.075	7 14	<0.007 <0.007	Hayashi *et al.*, 1986
WP 750 g/kg	5		0.075	7 14	<0.007 <0.007	Hayashi *et al.*, 1986

Sugar beet roots from trials with granular bait in the UK were analysed by GC-MS (LOQ 0.04 mg/kg).

Table 53. Residues in sugar beet from trials in the UK, 1997.

Location	Application				Residues		Reference
	Formulation	No	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, Belgium	GB 40 g/kg	--	0.2	--	--	--	--
Hertfordshire	RB 40 g/kg	3	0.195		98	<0.040	Maestracci, 1998a, Study 97-732
Norfolk	RB 40 g/kg	3	0.195		101	<0.040	Maestracci, 1998a, Study 97-32
Cambridgeshire	RB 40 g/kg	3	0.195		101	<0.040	Maestracci, 1998a, Study 97-732
Ely	RB 40 g/kg	3	0.195		101	<0.040	Maestracci, 1998a, Study 97-732

Leafy vegetables (except Brassica vegetables)

Supervised trials were conducted on lettuce with granular bait treatment in Italy and France and with aerial and ground foliar application in Spain and the USA. Supervised trials were also conducted on spinach with ground foliar application in the USA.

In the French trials, lettuce heads were analysed by GC-MS (LOQ 0.04 mg/kg). Duplicate samples were taken at each site.

Table 54. Residues in lettuce (granular bait application).

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Italy, 1992	GB 40 g/kg	1	0.8	--	7	<0.04	Anon., 1992 Report No.0026
GAP, France	GB 40 g/kg	1-3	0.4		7		
Ingre, France, 2000	GB 40 g/kg	2	0.413	--	0 8 14 22	0.17 14 <0.04 <0.04 0.14 <0.04 <0.04 0.13 0.047	Gateaud and Yslan, 2001a Study No.00-561
Le Meillard, France, 2000	GB 40 g/kg	2	0.413	--	0 8 14 21	0.13 0.19 <0.04 0.048 <0.04 <0.04 <0.04 <0.04	Gateaud and Yslan, 2001a Study No.00-561
Ingre, France, 2000	GB 40 g/kg	2	0.413	--	21	0.14 0.14	Gateaud and Yslan, 2001b, Study No.00-562. Control: 0.056 mg/kg
Le Meillard, France, 2000	GB 40 g/kg	2	0.413	--	22	0.053 0.043	Gateaud and Yslan, 2001b Study No.00-562

Trials with foliar application to lettuce were reported from Spain and the USA. Lettuce heads were analysed by GLC (nitrogen FPD, LOQ 0.04 mg/kg).

Table 55. Residues in head lettuce (foliar spray application with ground equipment).

Location, year	Application				Residues		Reference
	Form.	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Spain, 1999	SC 375 g/l	3	0.94	0.141	0 7	9.4 7.3 1.5 2.4	Gateaud and Yslan, 2000. Study No.99-568
Spain, 1999	SC 375 g/l	3	0.941	0.231	0 7 15 22	27 25 3.0 3.5 0.50 0.25 0.082 0.14	Gateaud and Yslan, 2000. Study No.99-568
Spain, 1999	SC 375 g/l	3	0.85	0.257	0 7 14 21	21 26 8.9 10 2.4 2.3 0.93 1.2	Gateaud and Yslan, 2000. Study No.99-568
Spain, 1999	SC 375 g/l	3	0.941	0.221	0 7 14 21	12 14 1.1 0.96 0.12 0.16 0.041 0.073	Gateaud and Yslan, 2000. Study No.99-568
GAP, USA	SC 375 g/l	--	0.45-0.84		14		Do not exceed 1.7 kg ai/ha per season
Santa Maria, CA USA, 1985	SC 375 g/l	2	0.84	--	14	0.34 0.64 <u>0.71</u>	Langdon, 1987 Project No.804R10; File No.34768
Salinas, CA USA, 1985	SC 375 g/l	2	0.84	--	14	<u>0.12</u> <0.04	Langdon, 1987 Project No.804R10; File No.34768
Manteca, CA USA, 1985	SC 375 g/l	2	0.84	--	14	<u>13</u> 7.2 12	Langdon, 1987 Project No.804R10; File No.34768
Sanford, FL USA, 1985	SC 375 g/l	2	0.84	--	14	6.1 <u>7.7</u> 5.8 3.8 2.8 3.0	Langdon, 1987 Project No.804R10; File No.34768
Newton, IA USA, 1985	SC 375 g/l	2	0.84	--	14	0.28 <u>0.49</u> 0.36	Langdon, 1987 Project No.804R10; File No.34768
Marcellus, MI USA, 1985	SC 375 g/l	2	0.84	--	14	<u><0.04</u> <0.04 <0.04	Langdon, 1987 Project No.804R10; File No.34768
Wayside, MS USA, 1985	SC 375 g/l	2	0.84	--	14	<u>10</u> 7.7 7.9	Langdon, 1987 Project No.804R10; File No.34768

Clayton, NC USA, 1985	SC 375 g/l	2	0.84	--	14	3.6 2.0 6.2	Langdon, 1987 Project No.804R10; File No.34768
Phelps, NY USA, 1985	SC 375 g/l	2	0.84	--	14	18 17 17	Langdon, 1987 Project No.804R10; File No.34768
Westlaco, TX USA, 1985	SC 375 g/l	2	0.84	--	14	5.5 5.5 6.3	Langdon, 1987 Project No.804R10; File No.34768
Glendale, AZ USA, 1983-84	SC 375 g/l	4	0.84		14	0.17 0.10 0.25	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		14	0.11 0.21 0.20	
Hollister, CA USA, 1983	SC 375 g/l	4	0.84		7 14	2.7 2.4 3.0 1.7 1.7	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		7 14	1.7 0.37 0.06 0.06 1.8	
El Centro, CA USA, 1983-84	SC 375 g/l	4	0.84		14	1.8 3.2 2.4	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		14	1.5 1.4 1.7	
Arlington, WI USA, 1983	SC 375 g/l	4	0.84		14	0.24 0.34 0.24 0.20	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		14	0.24 0.34 0.14 0.48	
Manteca, CA USA, 1984	SC 375 g/l	4	0.84		14	0.07 0.06 0.06	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		14	0.14 0.14 0.13	
Sanford, FL USA, 1984	SC 375 g/l	4	0.84		14	1.2 1.1 1.3	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		14	1.7 1.5	
Sodus, NY USA, 1984-85	SC 375 g/l	4	0.84		14	0.08 0.06 0.09	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		14	0.19 0.17 0.11	
Westlaco, TX USA, 1984-85	SC 375 g/l	4	0.84		14	0.29 0.42 0.28	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		14	0.26 0.34 0.44	

Dome Valley, AZ USA, 1984-85	SC 375 g/l	4	0.84		15	0.63 0.96 0.96	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	4	0.84		15	0.96 1.4 1.9	
Santa Maria, CA USA, 1985	SC 375 g/l	2	0.84		14	0.13 0.35 0.27	Hunt, 1986a Project No.804R11 File No.34501
Salinas, CA USA, 1985	SC 375 g/l	2	0.84		14	2.6 0.73	Hunt, 1986a Project No.804R11 File No.34501
Manteca, CA USA, 1985	SC 375 g/l	2	0.84		14	17 8.6 16	Hunt, 1986a Project No.804R11 File No.34501
Newton, IA USA, 1985	SC 375 g/l	2	0.84		14	0.09 0.08 0.06	Hunt, 1986a Project No.804R11 File No.34501
Marcellus, MI USA, 1985	SC 375 g/l	2	0.84		14	0.05 0.05 0.07	Hunt, 1986a Project No.804R11 File No.34501
Wayside, MS USA, 1985	SC 375 g/l	2	0.84		14	≤0.04 <0.04 <0.04	Hunt, 1986a Project No.804R11 File No.34501
Clayton, CA USA, 1985	SC 375 g/l	2	0.84		14	≤0.04 <0.04 <0.04	Hunt, 1986a Project No.804R11 File No.34501
Godus, NY USA, 1985	SC 375 g/l	2	0.84		14	0.33 0.36 0.17	Hunt, 1986a Project No.804R11 File No.34501

Table 56. Residues in lettuce (aerial application), USA.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, US	SC 375 g/l	--	0.45-0.84		14		Do not exceed 1.7 kg ai/ha per season
Hollister, CA, 1983	SC 375 g/l	4	0.84		7 14	2.8 2.2 2.0	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	4	0.84		7 14	1.3 0.27 ≤0.05 <0.05 <0.05	
Dome Valley, AZ, 1984-85	SC 375 g/l	4	0.84		15	1.1 1.0 1.5	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	4	0.84		15	0.65 1.1 0.78	

In spinach trials in the USA, analyses were by GLC (nitrogen FPD, LOQ 0.04 mg/kg).

Table 57. Residues in spinach (foliar spray application), USA.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.45-0.84		14		Do not exceed 1.7 kg ai/ha per season
Santa Maria, CA, 1985	SC 375 g/l	2	0.84	--	14	3.2 3.4 3.5	Langdon, 1987 ProjectNo.804R10;File No.34768
Salinas, CA, 1985	SC 375 g/l	2	0.84	--	14	<0.04 0.21	Langdon, 1987 ProjectNo.804R10;File No.34768
Manteca, CA, 1985	SC 375 g/l	2	0.84	--	14	20 20 23 21 21 25	Langdon, 1987 ProjectNo.804R10;File No.34768
	SC 375 g/l + oil	2	0.84	--	14	22 25 18	
Sanford, FL, 1985	SC 375 g/l	2	0.84	--	14	2.2 4.1 3.9 1.0 2.5 1.9	Langdon, 1987 ProjectNo.804R10;File No.34768
Newton, IA, 1985	SC 375 g/l	2	0.84	--	14	0.04 <0.04 <0.04	Langdon, 1987 ProjectNo.804R10;File No.34768
Marcellus, MI, 1985	SC 375 g/l	2	0.84	--	14	<0.04 <0.04 0.04	Langdon, 1987 ProjectNo.804R10;File No.34768
Clayton, NC, 1985	SC 375 g/l	2	0.84	--	14	2.2 1.7 3.2	Langdon, 1987 ProjectNo.804R10;File No.34768 (Spinach growth not vigorous)
	SC 375 g/l + oil	2	0.84	--		0.57 0.74 0.85	
Phelps, NY, 1985	SC 375 g/l	2	0.84	--	14	0.94 1.0 0.99 0.23 0.31 0.15	Langdon, 1987 ProjectNo.804R10;File No.34768
Westlaco, TX, 1985	SC 375 g/l	2	0.84	--	14	6.6 10 12	Langdon, 1987 ProjectNo.804R10;File No.34768

Supervised trials on collards were reported from the USA.

Table 58. Residues in collards (foliar spray application), USA.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Tifton, GA 1988	SC 375 g/l	5	0.84		7 11 14 	0.33 1.4 2.0 1.4 0.88 1.1 0.91 1.9 0.70 0.89 <u>1.5</u> 0.11	Hunt, 1988a File No.40458 Project No.804R10
Tifton, GA 1988	SC 375 g/l	5	1.7		7 11 14 	2.8 3.5 3.1 3.1 0.98 1.3 0.16 0.88 0.34 <u>1.8</u> 0.62 1.3	Study No.40458 Project No.804R10

<u>Brassica vegetables</u>

Supervised trials were conducted on broccoli, Brussels sprouts, cabbage and cauliflower in the USA, Australia, the UK and The Netherlands.

Samples were analysed by GLC with FPD in the sulfur mode (LOQ 0.04 mg/kg in the USA, 0.02 mg/kg in Australia).

Table 59. Residues in broccoli (foliar spray application; ground equipment).

Location, year	Application				Residues		Reference/Comment
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.45-1.15		7		Do not exceed 6.7 kg ai/ha per season
Manteca, CA USA, 1982	SC 375 g/l	6	0.56	0.200	1 3 7 14	3.2 3.4 3.0 4.8 2.6 2.5 0.61 0.46 0.44 0.32 0.36 0.20	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	0.56	0.200	1 3 7 14	3.5 2.0 3.4 4.2 4.2 3.9 0.20 0.25 0.21 0.22 0.35 0.20	
Manteca, CA USA, 1982	SC 375 g/l	6	1.1	0.399	1 3 7 14	6.9 8.8 9.2 5.1 7.6 6.4 1.2 1.3 1.2 1.0 0.91 0.71	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	1.1	0.399	1 3 7 14	7.1 6.4 7.3 4.9 5.5 4.9 0.77 1.1 1.1 0.62 0.54 0.59	

Location, year	Application				Residues		Reference/Comment
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Newton, IA USA, 1982	SC 375 g/l	6	0.56	0.15	1 3 14 	5.9 3.6 6.5 4.8 0.23 0.44 <0.02 <0.02 <0.02	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	0.56	0.15	1 3 14 	7.8 8.4 7.6 2.4 4.2 11 0.04 0.03 0.05	
Newton, IA USA, 1982	SC 375 g/l	6	1.1	0.300	1 3 14 	16 9.5 7.5 3.6 3.7 1.2 0.13 0.11 <0.02	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	1.121	0.300	1 3 14 	23 9.2 13 7.1 5.9 5.9 0.03 0.16 0.10	
Wayside, MS USA, 1982	SC 375 g/l	6	0.56	0.35	1 3 7 14 	2.3 2.1 2.1 1.3 2.1 2.3 0.29 0.51 0.54 <0.02 <0.02 <0.02	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	0.56	0.346	1 3 7 14 	2.3 3.1 2.5 1.6 1.1 2.1 0.69 1.2 0.32 0.05 0.04 0.04	

Location, year	Application				Residues		Reference/Comment
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Wayside, MS USA, 1982-85	SC 375 g/l	6	1.1	0.700	1 3 7 14	7.2 8.1 7.2 3.1 4.6 3.5 2.0 <u>2.6</u> 2.4 0.09 0.11 0.07	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	0.56	0.346	1 3 7 14	2.3 3.1 2.5 1.6 1.1 2.1 0.69 1.2 0.32 0.05 0.04 0.04	
San Benito, CA USA, 1983	SC 375 g/l	6	1.1	0.40	7 14	4.2 <u>5.0</u> 3.3 0.67 0.46 0.60	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	1.1	0.400	7 14	1.5 1.5 <u>1.6</u> 0.20 0.15 0.11	
Weslaco, TX USA, 1985	SC 375 g/l	6	1.1	1.19	14	0.27 0.15 0.23	Hunt, 1986a Project No.804R10 File No.34501
GAP, Australia	SC 375 g/l	--	0.375-0.75		7		
Cambooya Australia, 1989	SC 375 g/l	5	0.525		3 7 11[1] 14	2.1 <u>0.14</u> 0.09 <0.04	Keats, 1990 Report No.AK/TN/AK905
Cambooya Australia, 1989	SC 375 g/l	5	0.52		3 7 11[1] 14	4.2 <u>0.33</u> 0.20 0.08	Keats, 1990 Report No.AK/TN/AK905

[1] 11 days after 4th application and 2 h before 5th (final) application.

Table 60. Residues in broccoli (aerial application), USA.

Location, year	Application				Residues		Reference
	Formulation.	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.45-1.15		7		Do not exceed 6.7 kg ai/ha per season
San Benito, CA, 1982	SC 375 g/l	6	1.1	NR	7 14	3.3 5.6 4.9 0.57 0.33 0.60	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	1.1	NR	7 14	1.8 1.9 1.2 0.20 0.14 0.17	

Brussels sprouts were analysed by a GLC method (LOQ 0.05 mg/kg) in supervised field trials in The Netherlands.

Table 61. Residues in Brussels sprouts (granular bait application).

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP (Belgium)	GB 40g/kg	6	0.2	21		
Thorrington UK, 1997-98	RB 40 g/kg	5	0.2	7	<0.04 <0.04	Maestracci, 1998b Study No.97-733
Freiston UK, 1997	RB 40 g/kg	5	0.2	7	<0.04 <0.04	Maestracci, 1998b Study No.97-733
Boston UK, 1997	RB 40 g/kg	5	0.2	7	<0.04 <0.04	Maestracci, 1998b Study No.97-733
Biggleswad UK, 1997	RB 40 g/kg	5	0.2	7	<0.04 <0.04	Maestracci, 1998b Study No.97-733
Smitshoek Netherlands, 1997	GB 40 g/kg	6	0.2	21	0.059 <0.050	Richard and Muller, 1995a. Study No.92-304
Achterzeedijk Netherlands, 1997	GB 40 g/kg	6	0.2	21	<0.050 <0.050	Richard and Muller, 1995a. Study No.92-304
Noordeinde Netherlands, 1997	GB 40 g/kg	6	0.2	21	<0.050 <0.050	Richard and Muller, 1995a. Study No.92-304
Hoogeveenseweg Netherlands, 1997	GB 40 g/kg	6	0.2	21	<0.050 <0.050	Richard and Muller, 1995a. Study No.92-304
Netherlands, 1998	GB 40 g/kg	6	0.19	21	<0.004 <0.004	Yslan and Baudet, 1999 Study No.98-747 Limit of detection: 0.004 mg/kg
Netherlands, 1998	GB 40 g/kg	6	0.19	21	<0.004 <0.004	Yslan and Baudet, 1999 Study No.98-747
Netherlands, 1998	GB 40 g/kg	6	0.19	21	<0.004 <0.004	Yslan and Baudet, 1999 Study No.98-747
Netherlands, 1998	GB 40 g/kg	6	0.19	21	<0.004 <0.004	Yslan and Baudet, 1999 Study No.98-747

Supervised trials on cabbages were conducted in the USA and Australia. Samples were analysed by GLC with an FPD (LOQ 0.04 mg/kg in the USA, 0.02 mg/kg in Australia).

Table 62. Residues in cabbage (foliar spray application).

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	1.15	7		Do not exceed 6.7 kg ai/ha per season
Montgomery, AL USA, 1982	SC 375 g/l	6	1.1	3 5 7	0.19 0.22 0.18 0.22 0.19 0.11 0.08 0.09 <u>0.12</u>	Hunt, 1986a Project No.804R11 File No.34501
Teloxsira Farms, CA USA, 1982	SC 375 g/l	6	1.1	3 5 7	5.8 6.1 4.7 8.6 4.4 5.4 2.5 <u>3.0</u> 1.5	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	6	1.1	3 5 7	9.6 5.4 6.9 3.9 7.3 11 1.9 <u>2.8</u> 2.3	
Sanford, FL USA, 1982	SC 375 g/l	6	1.1	3 5 7	1.4 2.4 1.8 2.1 2.7 3.0 2.0 1.8 <u>3.1</u>	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	6	1.1	3 5 7	4.7 3.5 3.2 3.5 3.6 4.1 <u>2.1</u> 0.37 1.7	
Phelps, NY USA, 1982	SC 375 g/l	6	1.1	3 5 7	1.2 0.77 1.6 0.70 0.88 1.1 1.2 0.44 <u>1.3</u>	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	6	1.1	3 5 7	1.2 1.7 2.1 2.0 2.0 1.8 <u>3.5</u> 1.0 1.2	

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Clayton, NC USA, 1982	SC 375 g/l	6	1.1	3 5 7	0.13 0.20 0.16 0.12 0.17 0.18 0.07 <0.05 0.08	Hunt, 1986a Project No.804R11 File No.34501
Wooster, OH USA, 1982	SC 375 g/l	5	1.1	3 5	1.1 2.8 3.0 0.56 1.7 5.0	Hunt, 1986a Project No.804R11 File No.34501
Rock Spring, PA USA, 1982	SC 375 g/l	6	1.1	3 5 7	4.0 1.5 1.4 0.45 0.15 0.09 0.08 0.04	Hunt, 1986a Project No.804R11 File No.34501
Arlington, WI USA, 1982	SC 375 g/l	6	1.1	3 5 7	0.74 0.51 0.23 1.1 0.61 0.18 0.20 0.53	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	6	1.1	3 5 7	0.55 0.79 0.91 0.16 0.75 0.58 0.14 0.51 1.2	
San Benito, CA USA, 1983	SC 375 g/l	6	1.1	7 14	3.4 2.4 3.8 0.41 0.21 0.24	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	6	1.1	7 14	1.9 1.7 2.7 0.13 0.07 0.10	
Wayside, MS USA, 1983	SC 375 g/l	6	1.1	7 14	0.76 0.75 0.73 0.22 0.28 0.32	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	6	1.1	7 14	0.73 0.97 0.88 0.21 0.19 0.21	

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Clayton, NC	SC 375 g/l	6	1.1	7 14	3.2 3.4 <u>4.3</u> 1.0 1.0 1.9	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	6	1.1	7 14	4.8 4.1 <u>5.3</u> 1.6 2.0 1.4	
Cleveland, Queensland Australia, 1989	SC 375 g/l	5	0.75	3 7 14	0.07 0.05 <0.04	Keats, 1989a AK/aw/ak89006
Cleveland, Queensland Australia, 1989	SC 375 g/l	5	1.5	3 7 14	0.13 0.07 0.05	Keats, 1989a AK/aw/ak89006

Table 63. Residues in cabbage (aerial application) from trials in the USA.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
US GAP	SC 375 g/l	--	0.45-1.15		7		Do not exceed 6.7 kg ai/ha per season
San Benito, CA, 1982	SC 375 g/l	6	1.1		7 14	4.9 2.3 <u>5.0</u> 0.37 0.28 0.47	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	6	1.1		7 14	<u>4.8</u> 1.9 1.1 0.10 0.22 0.13	

Supervised field trials on cauliflower were conducted in Australia and the USA. Samples were analysed by GLC with FPD (LOQ 0.04 mg/kg in the USA, 0.02 mg/kg in Australia).

Table 64. Residues in cauliflower (foliar spray application)

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.45-1.15		7		Do not exceed 6.7 kg ai/ha per season
Manteca, CA USA, 1982	SC 375 g/l	7	0.56	0.20	1 3 7 14	0.70 1.3 1.1 0.53 0.80 1.3 0.29 0.24 0.24 0.22 0.18 0.21	Hunt, 1986a Project No.804R11 File No.34501
	WG 800 g/kg	7	0.56	0.20	1 3 7 14	1.0 0.94 0.89 0.72 0.69 0.66 0.21 0.27 0.26 0.30 0.25 0.26	
Manteca, CA USA, 1982	SC 375 g/l	7	1.1	0.40	1 3 7 14	1.5 3.4 3.4 1.2 1.2 1.4 <u>0.64</u> 0.55 0.63 0.27 0.28 0.43	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	7	1.1	0.40	1 3 7 14	2.1 3.8 1.7 0.89 1.5 1.3 <u>0.71</u> 0.51 0.67 0.35 0.53 0.55	

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Wayside, MS USA, 1982	SC 375 g/l	6	0.56	0.346	1 3 7 14	2.2 2.6 2.0 2.6 1.8 1.3 1.3 1.3 1.3 0.16 0.15 0.14	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	0.56	0.346	1 3 7 14	3.4 2.3 3.7 3.4 2.3 2.8 1.5 1.7 1.7 0.38 0.29 0.23	
Wayside, MS USA, 1982	SC 375 g/l	6	1.1	0.69	1 3 7 14	3.8 5.4 4.9 5.5 4.5 3.2 <u>2.3</u> 2.2 1.8 0.64 0.70 0.34	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	1.1	0.69	1 3 7 14	4.1 5.3 5.0 3.3 3.0 4.2 1.4 1.7 <u>2.3</u> 0.51 0.49 0.45	
San Benito, CA USA, 1982	SC 375 g/l	6	1.1	0.400	7 14	0.36 <u>0.45</u> 0.33 <0.04 <0.04 ND	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	1.1	0.400	7 14	ND <u>0.09</u> 0.06 <0.04 ND ND	
Phelps, NY USA, 1982	SC 375 g/l	6	1.1	0.24	14	4.1 1.9 1.7	Hunt, 1986a Project No.804R10 File No.34501

Table 65. Residues in cauliflower (aerial application), USA.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.45-1.15		7		Do not exceed 6.7 kg ai/ha per season
San Benito, CA, 1982	SC 375 g/l	6	1.1		7 14	0.07 0.10 <u>0.16</u> <0.04 0.04 ND	Hunt, 1986a Project No.804R10 File No.34501
	WG 800 g/kg	6	1.1		7 14	0.10 <u>0.27</u> 0.06 ND <0.04 <0.04	

<u>Legume vegetables</u>

Supervised trials were conducted on peas and chick-peas in Australia and soya beans in Australia, Brazil and the USA.

In Australia, peas and hay from supervised field trials were analysed by GLC with an FPD (LOQ 0.02 mg/kg).

Table 66. Residues in peas (foliar spray application), Australia, 1990.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, Australia	SC 375 g/l	--	0.188-0.28		21		
Redland Bay Research Station	SC 375 g/l	1	0.19	--	28	<0.02	Keats, 1991, Report No. AK/JG/AK002
Redland Bay Research Station	SC 375 g/l	1	0.28	--	28	<0.02	Keats, 1991, Report No. AK/JG/AK002
Redland Bay Research Station	SC 375 g/l	1	0.38	--	28	<0.02	Keats, 1991, Report No. AK/JG/AK002
Redland Bay Research Station	SC 375 g/l	1	0.56	--	28	<0.02	Keats, 1991, Report No. AK/JG/AK002
Redland Bay Research Station	SC 375 g/l	1	1.1	--	28	0.04	Keats, 1991, Report No. AK/JG/AK002

Table 67. Residues in peas (hay), Australia, 1990.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Redland Bay Research Station,	SC 375 g/l	1	0.19	--	0 7 14 28	3.48 0.08 <0.02 <0.02	Keats, 1991, Report No. AK/JG/AK002
Redland Bay Research Station	SC 375 g/l	1	0.28	--	0 7 14 28	5.21 0.20 0.04 0.02	Keats, 1991, Report No. AK/JG/AK002
Redland Bay Research Station,	SC 375 g/l	1	0.38	--	0 7 14 28	5.82 0.36 0.05 <0.02	Keats, 1991, Report No. AK/JG/AK002

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Redland Bay Research Station,	SC 375 g/l	1	0.56	--	0 7 14 28	9.3 0.92 0.13 0.02	Keats, 1991, Report No. AK/JG/AK002
Redland Bay Research Station	SC 375 g/l	1	1.1	--	0 7 14 28	16.4 2.17 0.29 0.05	Keats, 1991, Report No. AK/JG/AK002

In trials in Australia, chick-peas, straw, foliage, stems and immature pods were analysed by a GLC method (LOQ 0.05 mg/kg for straw and peas, 0.02 mg/kg for foliage).

Table 68. Residues in chick-peas (foliar spray application, ground), Australia, 1986.

Location	Application				Interval (days)	Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl		PHI, days	mg/kg	
GAP, Australia	SC 375 g/l	--	0.188-0.28			21		
South East of Emerald Queensland	SC 375 g/l	2	0.56		15	38	<0.05	Clark and Shields, 1986, Ref. 1870/86/5
South East of Emerald Queensland	SC 375 g/l	2	1.1		15	38	<0.05	

Table 69. Residues in chick-pea foliage, stems and immature pods, Australia, 1986.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, Australia	SC 375 g/l	--	0.188-0.28		21		
South East of Emerald Queensland	SC 375 g/l	1	0.56		0 7 14	19 6.0 2.5	Clark and Shields, 1986, Ref. 1870/86/5
South East of Emerald Queensland	SC 375 g/l	2	0.56		0 7 14	45 23 21	
South East of Emerald Queensland	SC 375 g/l	1	1.1		0 7 14	51 16 12	
South East of Emerald Queensland	SC 375 g/l	2	1.1		0 7 14	52 44 96	

Table 70. Residues in chick-peas (straw), Australia, 1986.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
South East of Emerald Queensland	SC 375 g/l	2	0.56		38	1.4	Clark and Shields, 1986, Ref. 1870/86/5
South East of Emerald Queensland	SC 375 g/l	2	1.1		38	2.7	

Supervised field trials on soya beans were reported from Australia, Brazil and the USA. Seeds, forage, hay and straw were analysed by GLC (FPD in sulfur mode, LOQ 0.04 mg/kg) in the USA. In Brazil, seeds were analysed by HPLC (LOQ 0.1 mg/kg and LOD 0.05 mg/kg). In Australia, seeds were analysed by GLC (sulfur FPD, LOQ 0.04 mg/kg).

Table 71. Residues in soya bean seed (foliar spray application; ground equipment).

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.84		28		Do not exceed 3.4 kg/ha per season
East Baton Rouge, Louisiana USA, 1991	SC 375 g/l	4	0.84	--	28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Lonoke, AR USA, 1991	SC 375 g/l	4	0.841	--	28	0.05 <u>0.06</u> 0.05	Bird and Coffey, 1992 Project No.USA91L30
Washington, MS USA, 1991	SC 375 g/l	4	0.84	--	27	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Sibley, MN USA, 1991	SC 375 g/l	4	0.84	--	28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992
Martin, NC USA, 1991	SC 375 g/l	4	0.84	--	28	<u>≤0.04</u> <0.04 <0.04	Project No.USA91L30
Clay, MN USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992
Lancaster, PA USA, 1991	SC 375 g/l	4	0.94		27	0.10 0.06 <u>0.15</u>	Project No.USA91L30
Landry, LA USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992
Washington, MS USA, 1991	SC 375 g/l	4	0.84		29	<u>≤0.04</u> <0.04 <0.04	Project No.USA91L30
Des Moines, IA USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992
Des Moines, IA USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Project No.USA91L30
Shelby, MO USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Shelby, MO USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Arkansas, AR USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Hamilton, IN USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Hamilton, IN USA, 1991	SC 375 g/l	4	0.84		31	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Henderson, IL USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Henry, IL USA, 1991	SC 375 g/l	4	0.89		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Richmond, VA USA, 1991	SC 375 g/l	4	0.84		28	<u>≤0.04</u> <0.04 <0.04	Bird and Coffey, 1992 Project No.USA91L30
Geneseo, IL USA, 1990	SC 375 g/l	4	4.2		29	0.04	Lee, 1991c Project No.USA90L01 File No.41003

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, Australia	SC 375 g/l	--	0.188-0.28		21		
Patrick Estate, Lowood Australia, 1985	SC 375 g/l	1	0.75		35	ND (<0.02) ND ND ND	Hunt and Langdon, 1985
Patrick Estate, Lowood Australia, 1985	SC 375 g/l	1	1.9		35	ND (<0.02) ND ND ND	Hunt and Langdon, 1985
GAP, Brazil	WG 800 g/l	--	0.056		14		
Paulinia Brazil, 1996-1997	WG 800 g/kg	3	0.08		14	<0.05 <0.05 <0.05	Anon., 1997 CP-2480/97 Study No.006/97-PC
Paulinia Brazil, 1996-1997	WG 800 g/kg	3	0.16		14	<0.05 <0.05 <0.05	Anon., 1997 CP-2480/97 Study No.006/97-PC

Supervised field trials with aerial application were conducted in the USA. 2.3 l/ha of a commercially available emulsified vegetable oil was used in all cases.

Table 72. Residues in soya bean seed (aerial application), USA, 1987.

Location	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.84		28		Do not exceed 3.4 kg/ha per season
Proctor, AR	SC 375 g/l	2	0. 50		28	<0.04 <0.04 <0.04	Hunt, 1988d File No.40383 Project No.804R10
Midville, GA	SC 375 g/l	2	0. 50		31	<0.04 <0.04 <0.04	Hunt, 1988d File No.40383 Project No.804R10
Cohoama Country, MS	SC 375 g/l	2	0. 50		28	<0.04 <0.04 <0.04	Hunt, 1988d File No.40383 Project No.804R10
Leland, MS	SC 375 g/l	2	0. 50		27	<0.04 <0.04 <0.04	Hunt, 1988d File No.40383 Project No.804R10
Clarence, MO	SC 375 g/l	2	0. 50		31	ND (<0.02) ND ND	Hunt, 1988d File No.40383 Project No.804R10
Jamesville, NC	SC 375 g/l	2	0. 50		28	ND (<0.02) ND ND	Hunt, 1988d File No.40383 Project No.804R10
Plymouth, NC	SC 375 g/l	2	0. 50		28	<0.04 <0.04 <0.04	Hunt, 1988d File No.40383 Project No.804R10
Darlington Country, SC	SC 375 g/l	2	0. 50		28	0.05 <0.04 0.05	Hunt, 1988d File No.40383 Project No.804R10
Sulfolk, VA	SC 375 g/l	2	0. 50		28	<0.04 <0.04 <0.04	Hunt, 1988d File No.40383 Project No.804R10

Location	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Keller, VA	SC 375 g/l	2	0. 50		28	ND <0.04 <0.04	Hunt, 1988d File No.40383 Project No.804R10

Table 73. Residues in soya bean seed (seed treatment), USA, 1985.

Location	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Elberta, Al	900 g/kg	1	20	115	ND (<0.02)	Hunt, 1988c File No.40388 Project No.804R10
Buckley, IL	900 g/kg	1	20	155	ND ND	Hunt, 1988c File No.40388 Project No.804R10
Newton, IA	900 g/kg	1	20	149	ND ND ND ND	Hunt, 1988c File No.40388 Project No.804R10
Wayside, MS	900 g/kg	1	20	142	ND ND ND ND	Hunt, 1988c File No.40388 Project No.804R10
Clayton, NC	900 g/kg	1	20	206	ND ND ND ND	Hunt, 1988c File No.40388 Project No.804R10

Supervised field trials were conducted in the USA for residues in soya bean forage, hay and straw, but the labels forbid the use of these commodities as livestock feed.

Table 74. Residues in soya bean forage (foliar spray application), USA.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.28-0.84		--		Do not exceed 3.4 kg/ha per season
East Baton Rouge, LA	SC 375 g/l	4	0.84	--	0	16.0 8.6 11.9	Bird and Coffey, 1992 Project No.USA91L30
Lonoke, AR	SC 375 g/l	4	0.84	--	0	19.1 20.4 24.0	Bird and Coffey, 1992 Project No.USA91L30
Sibley, MN	SC 375 g/l	4	0.84	--	7	3.96 3.94 3.49	Bird and Coffey, 1992 Project No.USA91L30
Martin, NC	SC 375 g/l	4	0.84	--	0	16.2 13.4 22.0	Bird and Coffey, 1992 Project No.USA91L30
Richmond, VA	SC 375 g/l	4	0.84		0	10.6 23.2 23.0	Bird and Coffey, 1992 Project No.USA91L30
Clay, MN	SC 375 g/l	4	0.84		0	28.3 27.5 25.3	Bird and Coffey, 1992 Project No.USA91L30
Lancaster, PA	SC 375 g/l	4	0.94		0	35.7 19.8 25.8	Bird and Coffey, 1992 Project No.USA91L30
St Landry, LA	SC 375 g/l	4	0.84		0	26.7 32.2 29.6	Bird and Coffey, 1992 Project No.USA91L30

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Washington, MS	SC 375 g/l	4	0.84		0 29	26.4 27.4 22.2	Bird and Coffey, 1992 Project No.USA91L30
Des Moines, IA	SC 375 g/l	4	0.84		0	18.6 22.5 17.2	Bird and Coffey, 1992 Project No.USA91L30
Des Moines, IA	SC 375 g/l	4	0.84		0	25.6 14.0 11.9	Bird and Coffey, 1992 Project No.USA91L30
Shelby, MO	SC 375 g/l	4	0.84		0	22.0 19.5 22.5	Bird and Coffey, 1992 Project No.USA91L30
Shelby, MO	SC 375 g/l	4	0.84		0	29.6 16.3 20.7	Bird and Coffey, 1992 Project No.USA91L30
Arkansas, AR	SC 375 g/l	4	0.84		0	20.9 21.9 25.8	Bird and Coffey, 1992 Project No.USA91L30
Hamilton, IN	SC 375 g/l	4	0.84		0	17.5 15.8 21.8	Bird and Coffey, 1992 Project No.USA91L30
Hamilton, IN	SC 375 g/l	4	0.84		0	40.0 27.8 29.3	Bird and Coffey, 1992 Project No.USA91L30
Henderson, IL	SC 375 g/l	4	0.84		0	51.1 37.6 51.2	Bird and Coffey, 1992 Project No.USA91L30
Henry, IL	SC 375 g/l	4	0.8		0	23.1 23.0 24.3	Bird and Coffey, 1992 Project No.USA91L30
Richmond, VA	SC 375 g/l	4	0.84		3	8.36 20.5 15.5	Bird and Coffey, 1992 Project No.USA91L30

Table 75. Residues in soya bean forage (seed treatment), USA, 1985.

Location	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Clayton, NC	900 g/kg	1	20	14	21 22 31 25	Hunt, 1988c File No.40388 Project No.804R10
				21	7.6 6.9 3.9 2.1	
				35	0.70 0.78 0.76 0.76	
				68	ND ND ND ND	
				99	ND ND ND ND	

Table 76. Residues in soya bean hay (foliar spray application), USA, 1991.

Location	Application				Residues		Reference
	Form.	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.28-0.84	0.14-0.42	0-7		Do not exceed 3.4 kg/ha per season
East Baton Rouge, LA	SC 375 g/l	4	0.84		0	59.5 75.0 43.9	Bird and Coffey, 1992. Project No.USA91L30
Lonoke, AR	SC 375 g/l	4	0.84		5	85.0 50.2 64.7	Bird and Coffey, 1992. Project No.USA91L30
Sibley, MN	SC 375 g/l	4	0.84		11	7.19 7.18 4.73	Bird and Coffey, 1992. Project No.USA91L30
Martin, NC	SC 375 g/l	4	0.84		5	134.4 61.4 114.5	Bird and Coffey, 1992. Project No.USA91L30
Clay, MN	SC 375 g/l	4	0.84		0	21.2 30.4 37.6	Bird and Coffey, 1992. Project No.USA91L30
Lancaster, PA	SC 375 g/l	4	0.94		1	19.1 23.8 19.2	Bird and Coffey, 1992. Project No.USA91L30
St Landry, LA	SC 375 g/l	4	0.84		7	161.7 151.9 162.7	Bird and Coffey, 1992. Project No.USA91L30
Washington, MS	SC 375 g/l	4	0.84		2	47.2 45.2 50.8	Bird and Coffey, 1992. Project No.USA91L30
Des Moines, IA	SC 375 g/l	4	0.84		7	30.0 34.9 16.5	Bird and Coffey, 1992. Project No.USA91L30
Des Moines, IA	SC 375 g/l	4	0.84		7	35.6 20.3 78.9	Bird and Coffey, 1992. Project No.USA91L30
Shelby, MO	SC 375 g/l	4	0.84		4	17.7 25.7 12.3	Bird and Coffey, 1992. Project No.USA91L30
Shelby, MO	SC 375 g/l	4	0.84		4	17.6 10.5 22.6	Bird and Coffey, 1992. Project No.USA91L30

Location	Application				Residues		Reference
	Form.	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Arkansas, AR	SC 375 g/l	4	0.84		0	35.6 25.0 41.8	Bird and Coffey, 1992. Project No.USA91L30
Hamilton, IN	SC 375 g/l	4	0.84		0	65.9 31.9 63.5	Bird and Coffey, 1992. Project No.USA91L30
Hamilton, IN	SC 375 g/l	4	0.84		0	145.2 54.0 50.2	Bird and Coffey, 1992. Project No.USA91L30
Henderson, IL	SC 375 g/l	4	0.84		7	40.7 36.8 49.7	Bird and Coffey, 1992. Project No.USA91L30
Henry, IL	SC 375 g/l	4	0.89		3	31.1 27.2 27.7	Bird and Coffey, 1992. Project No.USA91L30
Richmond, VA	SC 375 g/l	4	0.84		3	13.1 25.3 17.7	Bird and Coffey, 1992. Project No.USA91L30

Table 77. Residues in soya bean straw (foliar spray application).

Location	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.28-0.84	0.14-0.42	28		Do not exceed 3.4 kg/ha per season
East Baton Rouge USA, 1991	SC 375 g/l	4	0.84	--	28	0.82 0.30 0.10	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Lonoke, AR USA, 1991	SC 375 g/l	4	0.84	--	28	0.71 0.65 1.61	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Washington, MS USA, 1991	SC 375 g/l	4	0.84	--	27	0.41 2.36 0.27	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Sibley, MN USA, 1991	SC 375 g/l	4	0.84	--	28	0.04 0.09 <0.04	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Martin, NC USA, 1991	SC 375 g/l	4	0.84	--	28	0.13 0.09 0.07	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Clay, MN USA, 1991	SC 375 g/l	4	0.84		28	0.51 0.19 0.28	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Lancaster, PA USA, 1991	SC 375 g/l	4	0.94		27	0.08 <0.04 0.05	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
St Landry, LA USA, 1991	SC 375 g/l	4	0.84		28	<0.04 0.04 0.04	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Washington, MS USA, 1991	SC 375 g/l	4	0.84		29	0.32 1.97 0.23	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Des Moines, IA USA, 1991	SC 375 g/l	4	0.84		28	0.28 0.20 0.24	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Des Moines, IA USA, 1991	SC 375 g/l	4	0.84		28	1.00 0.30 0.54	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Shelby, MO USA, 1991	SC 375 g/l	4	0.84		28	0.21 0.23 0.20	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Shelby, MO USA, 1991	SC 375 g/l	4	0.84		28	0.22 0.28 0.33	Bird and Coffey, 1992 File No.41215 Project No.USA91L30

Location	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Arkansas, AR USA, 1991	SC 375 g/l	4	0.84		28	<0.04 0.06 <0.04	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Hamilton, IN USA, 1991	SC 375 g/l	4	0.84		28	0.06 0.06 <0.04	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Hamilton, IN USA, 1991	SC 375 g/l	4	0.84		31	0.09 0.05 0.13	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Henderson, IL USA, 1991	SC 375 g/l	4	0.84		28	0.33 0.69 0.18	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Henry, IL USA, 1991	SC 375 g/l	4	0.89		28	0.20 0.27 0.42	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
Richmond, VA USA, 1991	SC 375 g/l	4	0.84		28	2.68 1.95 3.43	Bird and Coffey, 1992 File No.41215 Project No.USA91L30
GAP, Australia	SC 375 g/l	--	0.19-0.28		21		
Patrick Estate, Lowood Australia, 1985	SC 375 g/l	1	0.75		35	1.1 0.28 0.67 0.47	Hunt and Langdon, 1985
Patrick Estate, Lowood Australia, 1985	SC 375 g/l	1	1.9		35	0.11 0.11 0.26 0.09	Hunt and Langdon, 1985

Fruiting vegetables

Supervised trials were conducted in Australia, the USA and Spain on tomatoes. Samples in Australia were analysed by GLC with FPD (LOQ 0.04 mg/kg) and in Spain by GC-MS (LOQ 0.04 mg/kg).

Table 78. Residues in tomatoes and cherry tomatoes (foliar spray application).

Crop, Location, Year	Application				Interval (days)	Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl		PHI, days	mg/kg	
GAP, Australia	SC 375 g/l	--	0.524			1		
TOMATO Carpendale, Queensland Australia, 1985	SC 375 g/l	6	0.52	0.32	--	0 7 14 28 	0.18 0.73 0.14 0.16 0.33 0.14 <0.04 <0.04 <0.04 <0.04 <0.04 <0.04	Anon., 1986.
TOMATO Carpendale, Queensland Australia, 1985	SC 375 g/l	6	1.0	0.62	--	0 7 14 28 	1.6 0.42 0.47 0.70 050 0.32 0.05 <0.04 <0.04 <0.04 <0.04 <0.04	Anon., 1986.

Crop, Location, Year	Application				Interval (days)	Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl		PHI, days	mg/kg	
TOMATO Ma Ma Creek, Queensland Australia, 1985	SC 375 g/l	6	0.52	0.03	--	0 7 14 28 	<0.04 <u>0.09</u> <0.04 <0.04 0.09 0.04 0.13 <0.04 <0.04 <0.04 <0.04 <0.04	Anon., 1986.
TOMATO Ma Ma Creek, Queensland Australia, 1985	SC 375 g/l	6	1.0	0.05	--	7 14 28 	0.41 0.15 <0.04 <0.04 <0.04 <0.04 0.07 <0.04 <0.04	Anon., 1986.
TOMATO Clayton, NC USA, 1981	Larvin 500	6	1.1		7	1 3 7 14 	0.49 0.35 0.33 0.18 0.23 0.08 0.14 0.05 0.20 0.05 0.05 0.06	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Wayside, MS USA, 1981	Larvin 500	6	1.1		7	1 3 7 14 	0.10 0.06 0.09 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05 <0.05	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Manteca, CA USA, 1981	Larvin 500	6	1.1		7	1 3 7 14 	2.0 2.0 1.1 2.2 1.4 1.4 0.92 1.3 0.89 1.5 2.2 1.8	Hunt *et al.*, 1983 File No.32116 Project 804R11

Crop, Location, Year	Application				Interval (days)	Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl		PHI, days	mg/kg	
TOMATO Newton, Iowa USA, 1981	Larvin 500	6	1.1		7	1 3 7 14 	0.49 0.35 0.33 0.18 0.23 0.08 0.14 0.05 0.20 0.05 0.05 0.06	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Mendota, CA USA, 1981	SC 375 g/l	6	1.1			1 4 7 	0.06 0.04 0.04 0.05 0.04 0.06 0.11 0.07 0.06 0.09 0.04	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO California USA, 1981	SC 375 g/l	6	1.			1 3 7 	0.34 0.63 0.46 0.25 0.24 0.32 0.35 0.53 0.44	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Indiana USA, 1981	SC 375 g/l	6	1.1			1 4 7 	0.28 0.26 0.47 <0.02 0.24 0.20 0.17 0.12 0.10	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Montgomery, Al USA, 1981	SC 375 g/l	6	1.1			1 4 7 	0.24 0.24 0.24 0.07 0.08 0.06 0.07 0.07 0.08	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Pomona, CA USA, 1981	SC 375 g/l	6	1.1			1 4 7 	0.32 0.31 0.37 0.16 0.13 0.21 0.20 0.03 0.62	Hunt *et al.*, 1983 File No.32116 Project 804R11

Crop, Location, Year	Application				Interval (days)	Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl		PHI, days	mg/kg	
TOMATO Wooster, CA USA, 1981	SC 375 g/l	6	1.1			1 4 7 	0.30 0.49 0.64 0.52 0.52 0.39 0.28 0.35 1.0	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO California USA, 1981	SC 375 g/l	6	1.1			2	1.2 0.82 1.0 1.0	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO USA, 1981	SC 375 g/l	6	1.1			1 4 7 	0.02 0.03 <0.02 0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02 <0.02	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO USA, 1981	SC 375 g/l	6	1.1			1 4 7 	0.07 0.24 0.08 0.14 0.08 0.03 0.05 0.05 0.05	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO California USA, 1981	SC 375 g/l	6	1.1			1	0.13	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Mendota, CA USA, 1981	WG 800 g/kg	6	1.1		--	1 4 7 	0.06 0.04 0.04 0.04 0.04 0.04 0.04 0.06 0.08 0.04 0.09 0.07	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO California USA, 1981	WG 800 g/kg	6	1.1		--	0 3 7 	0.43 0.39 0.55 0.30 0.17 0.31 0.31 0.42 0.36	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Indiana USA, 1981	WG 800 g/kg	6	1.1		--	1 4 7 	0.16 0.34 0.37 0.04 0.21 0.13 0.09 0.12 0.12	Hunt *et al.*, 1983 File No.32116 Project 804R11

Crop, Location, Year	Application				Interval (days)	Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl		PHI, days	mg/kg	
TOMATO Pomona, CA USA, 1981	WG 800 g/kg	6	1.1		--	1 4 7	1.0 0.47 0.19 0.04 0.58 0.07 0.25 0.29 0.20	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO California USA, 1981	WG 800 g/kg	6	1.1		--	2	1.4 0.77 1.3 1.2	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO Florida USA, 1981	WG 800 g/kg	6	1.1		--	1 4 7	0.09 0.11 0.19 0.18 0.13 0.05 0.09 0.08 0.08	Hunt *et al.*, 1983 File No.32116 Project 804R11
TOMATO North Rose, NY USA, 1996	SC 375 g/l	6	1.1		7	1	<0.04 0.04	Kowite, 1998a File No.45559; Study No.96L10369
TOMATO San Juan Bautista, CA USA, 1996	SC 375 g/l	6	1.1		7	1	1.70 1.96	Kowite, 1998a File No.45559; Study No.96L10369
TOMATO Madera, CA USA, 1996	SC 375 g/l	6	1.1		7	1	0.26 0.18	Kowite, 1998a File No.45559; Study No.96L10369
CHERRY TOMATO Hickman, CA USA, 1996	SC 375 g/l	6	1.1		7	1	1.04 0.86	Kowite, 1998a File No.45559; Study No.96L10369
CHERRY TOMATO San Juan Bautista, CA USA, 1996	SC 375 g/l	6	1.1		7	1	2.36 2.48	Kowite, 1998a File No.45559; Study No.96L10369
CHERRY TOMATO Madera, CA USA, 1996	SC 375 g/l	6	1.1		7	1	0.99 0.89	Kowite, 1998a File No.45559; Study No.96L10369
TOMATO Hickman, CA USA, 1996	SC 375 g/l	6	1.1		7	1	0.88 0.80	Kowite, 1998a File No.45559; Study No.96L10369
GAP, Spain	SC 375 g/l	--	0.94			7		
TOMATO Roquetas, Almeria Spain, 1996-97 GLASSHOUSE	SC 375 g/l	3	0.94	0.070	15-22	7 14	<u>0.23</u> 0.21 0.38 0.30	Maestracci, 1998c Study No. 96-645
TOMATO Roquetas, Almeria Spain, 1996-97 GLASSHOUSE	SC 375 g/l	3	0.94	0.060	15-22	7 14	<u>0.18</u> 0.18 0.31 0.29	Maestracci, 1998c Study No. 96-645

Crop, Location, Year	Application				Interval (days)	Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl		PHI, days	mg/kg	
TOMATO Alginet, Valencia Spain, 1996-97 GLASSHOUSE	SC 375 g/l	2	0.94	0.064	21	7 14	0.06 0.05 0.06 0.05	Maestracci, 1998c Study No. 96-645
TOMATO La Canãda, Ameria Spain, 1996-97 GLASSHOUSE	SC 375 g/l	2	0.95	0.066	14	7 14	0.23 0.33 0.22 0.44	Maestracci, 1998d Study No. 97-680
TOMATO Alginet, Valencia Spain, 1996-97 GLASSHOUSE	SC 375 g/l	2	0.94	0.045	14	7 14	0.04 0.05 <0.04 <0.04	Maestracci, 1998d Study No. 97-680
TOMATO Ameria Spain, 1992-1993 GLASSHOUSE	SC 375 g/l	3	0.75	0.03	12-22	0 7 14 21 28	0.20 0.16 0.16 0.13 0.26	Richard and Muller, 1993 Study No. 91-242
TOMATO Ameria Spain, 1992-1993 GLASSHOUSE	SC 375 g/l	3	1.1	0.04	12-22	0 7 14 21 28	<0.05 0.13 0.23 0.16 0.18	Richard and Muller, 1993 Study No. 91-242
TOMATO Alginet Spain, 1992-1993 GLASSHOUSE	SC 375 g/l	3	0.75	0.11	10-11	0 7 14 21 28	0.15 0.08 <0.05 0.16 0.15	Richard and Muller, 1993 Study No. 91-242

Sweet corn. Supervised trials were conducted in the USA and Australia with ground foliar application, and in the USA with seed treatment application.

GAP in the USA for foliar application specifies 0.56-0.84 kg ai/ha, applied up to 8.4 kg ai/ha per season and a PHI of 0 days. Kernels, cobs and cannery waste were analysed by GLC with FPD (sulfur mode, LOQ 0.04 mg/kg). There is no GAP in the USA for seed treatment.

GAP in Australia for foliar application requires 0.56-0.75 kg ai/ha and a PHI of 7 days. Cobs and stalk were analysed by GLC with sulfur FPD (LOQ 0.02 mg/kg).

Table 79. Residues in sweet corn (kernel + cob with husk removed; foliar spray application).

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, Australia	SC 375 g/l	--	0.75		7		
Cowra, New South Wales, Australia, 1989	SC 375 g/l	2	0.52		18	<0.02	Keats, 1989d ak/aw/ak89005
Cowra, New South Wales, Australia, 1989	SC 375 g/l	2	1.05		18	<0.02	Keats, 1989d ak/aw/ak89005
GAP, USA	SC 375 g/l	--	0.84	0.40	0		Do not exceed 8.4 kg/ha per season
Lamberton, MN, USA, 1992	SC 375 g/l	10	0.84	0.45	0	<0.02	Lee, 1993 92-030; Project No.USA92L01 File No.44128. Retreatment interval 1 day.
Waterville, AR, USA, 1992	SC 375 g/l	10	0.84	0.82	0	0.07	Lee, 1993 92-098; Project No.USA92L01 File No.44128 Retreatment interval 1 day.

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Edcough, TX USA, 1977	WP 750 g/kg	8	1		1	0.02	Hunt, 1982, File No. 31094, Project No. 06570
Clayton, NC USA, 1978	WP 750 g/kg	16	1		1 3 7	<0.02 0.09 0.11	Hunt, 1982, File No. 31094, Project No. 06570
Edcough, TX USA, 1978	WP 750 g/kg	15	1		1 3 7	0.28 0.09 0.06	Hunt, 1982, File No. 31094, Project No. 06570
Newton, IA USA, 1981	WP 750 g/kg	10	0.84		0	<0.03 <0.03 <0.03	Hunt, 1982, File No. 31094, Project No. 06570
	SC 500 g/l	10	0.84		0 1 2	<0.03 <0.03 <0.03 <0.03 <0.03 <0.03 <0.03 <0.03 <0.03	
Clayton, NC USA, 1981	WP 750 g/kg	10	0.84		0	0.04 0.06 0.05	Hunt, 1982, File No. 31094, Project No. 06570
	SC 500 g/l	10	0.84		0 1 2	0.04 <0.04 0.13 0.06 0.07 0.08 0.07 0.08 0.06	
Sanford, FL USA, 1978	SC 500 g/l	10	0.84		1	0.19 0.82 0.39 0.29	Hunt, 1982, File No. 31094, Project No. 06570
Geneseo, IL USA, 1981	SC 500 g/l	10	0.84		0	<0.03 <0.03 <0.03	Hunt, 1982, File No. 31094, Project No. 06570
Salisbury, MD USA, 1981	SC 500 g/l	10	0.84		0	0.21 0.54 0.16	Hunt, 1982, File No. 31094, Project No. 06570
Hollandale, MN USA, 1981	SC 500 g/l	10	0.84		0	0.08 <0.03 0.06	Hunt, 1982, File No. 31094, Project No. 06570
Phelps, NY USA, 1981	SC 500 g/l	10	0.84		0	<0.03 <0.03 <0.03	Hunt, 1982, File No. 31094, Project No. 06570
Sanford, FL USA, 1981	SC 500 g/l	12	0.84		0 3 7	0.15 0.26 0.43 0.47 0..37 0.26 0.20 0.38 0.25	Hunt, 1982, File No. 31094, Project No. 06570
Donna, TX USA, 1981	SC 500 g/l	10	0.84		0	0.31 1.3 1.5	Hunt, 1982, File No. 31094, Project No. 06570
Kimberly, ID USA, 1981	SC 500 g/l	10	0.84		0	<0.03	Hunt, 1982, File No. 31094, Project No. 06570
Prosser, WA USA, 1981	SC 500 g/l	10	0.84		0	0.03 0.04 0.04	Hunt, 1982, File No. 31094, Project No. 06570

Location, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Manteca, CA USA, 1981	SC 500 g/l	10	0.84		0 1 2 	0.17 0.04 <u>0.22</u> 0.08 0.05 0.05 0.09 0.06 0.06	Hunt, 1982, File No. 31094, Project No. 06570

Table 80. Residues in sweet corn (kernel + cob with husk removed; simulated aerial application), USA, 1981.

Location	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.56-0.84	0.40	0		Do not exceed 8.4 kg/ha per season
Newton, IA	SC 500 g/l	10	0.84		0	<u><0.03</u> <0.03 <0.03	Hunt, 1982, File No. 31094, Project No. 06570
Clayton, NC	SC 500 g/l	10	0.84		0	<0.04 <u>0.09</u> <0.04	Hunt, 1982, File No. 31094, Project No. 06570
Manteca, CA	SC 500 g/l	10	0.84		0	0.03 <u>0.07</u> 0.03	

Table 81. Residues in sweet corn (kernel + cob with husk removed; seed treatment), USA, 1988.

Location	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Manteca, CA	SC 375 g/l	1	10	85	ND (<0.02) ND ND ND	Hunt, 1988i Project No.804R10 File No.40389
Manteca, CA	SC 375 g/l	1	10	81	ND ND ND ND	Project No.804R10 File No.40389
Newton, IA	SC 375 g/l	1	10	77	ND ND ND ND	Project No.804R10 File No.40389
Clayton, NC	SC 375 g/l	1	10	103	ND ND ND ND	Project No.804R10 File No.40389
Redfield, IA	SC 375 g/l + vitavax	1	10	81	ND ND ND ND	Project No.804R10 File No.40389
Manteca, CA	900 g/kg	1	20	76	ND ND ND ND	Project No.804R10 File No.40389
Newton, IA	900 g/kg	1	20	90	ND ND ND ND	Project No.804R10 File No.40389

Location	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Wayside, MS	900 g/kg	1	20	68	ND ND ND ND	Project No.804R10 File No.40389
Clayton, NC	900 g/kg	1	20	76	ND ND ND ND	Project No.804R10 File No.40389
Rochester, NY	900 g/kg	1	20	83	ND ND ND ND	Project No.804R10 File No.40389
Rochester, NY	900 g/kg	1	20	95	ND ND ND ND	Project No.804R10 File No.40389

Cereal grains

Barley and wheat. Supervised trials were conducted in the UK and Germany with granular bait applications. Grain and leaves were harvested and analysed by GLC (sulfur FPD, LOQs 0.05 and 0.04 mg/kg). Two other supervised trials in Brazil were with seed treatment applications.

Table 82. Residues in barley and wheat grain (granular bait application).

CROP Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, UK	RB 40 g/kg	1-3	0.2	--		
WINTER BARLEY Thirsk, North Yorkshire, UK, 1988	GB 40 g/kg	4	0.3	162	<0.04	Brockelsby *et al.*, 1989 Report No. 1295
WINTER BARLEY Chelmsford, Essex, UK, 1988	GB 40 g/kg	4	0.3	105	<0.04 <0.04	Brockelsby *et al.*, 1989 Report No. 1295
WINTER BARLEY Chapeltown, Derbyshire, UK, 1988	GB 40 g/kg	4	0.3	158	<0.04 <0.04	Brockelsby *et al.*, 1989 Report No. 1295
WINTER BARLEY West Hayes, Bedfordshire, UK, 1990	GB 40 g/kg	3	0.2	133	0.04 0.06 0.04	Brockelsby *et al.*, 1990a Report No. 1535
WINTER Eastwell, Leicestershire, UK, 1989-90	GB 40 g/kg	3	0.2	224	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No. 1535
WINTER BARLEY Abberley, Worcestershire, UK, 1989-90	GB 40 g/kg	3	0.2	175	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No. 1535
WHEAT Reepham, Lincolnshire, UK, 1989-90	GB 40 g/kg	3	0.2	245	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No. 1535
WHEAT Abberley, Worcestershire UK, 1989-90	GB 40 g/kg	3	0.2	182	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No. 1535
WHEAT West Hayes, Bedfordshire, UK 1990	GB 40g/kg	3	0.2	133	0.04 0.06 0.04	Brockelsby *et al.*, 1990a Report No. 1535
WHEAT Yeovil, Somerset K, 1987-88	GB 40 g/kg	4	0.3	165	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1989 Report No. 1295
WHEAT Northallerton, North Yorkshire, UK, 1987-88	GB 40 g/kg	3	0.4	179	<0.04	Brockelsby *et al.*, 1989 Report No. 1295
WHEAT Haywards Heath, Sussex, UK, 1987-88	GB 40 g/kg	4	0.3	123	<0.04 <0.04	Brockelsby *et al.*, 1989 Report No. 1295

CROP Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
WHEAT Salzhemmendorf Niedersachsen Germany, 1987-88	GB 40 g/kg	2	0.2	281	ND	Anon., 1992d BBA report No.05148
WHEAT Salzhemmendorf Niedersachsen Germany, 1987-88	GB 40 g/kg	2	0.2	101	ND	Anon., 1992d BBA report No.05149
WHEAT Schwaighofen Bayern, Germany, 1987-88	GB 40 g/kg	2	0.2	136	ND	Anon., 1992d BBA report No.03675
WINTER BARLEY Gerolfingen Germany, 1987-88	GB 40 g/kg	2	0.2	239	ND	Anon., 1992b BBA report No. 03725
WINTER BARLEY Gerolfingen Germany, 1987-88	GB 40 g/kg	2	0.2	93	ND	Anon., 1992b BBA report No. 05132

Table 83. Residues in wheat grain (seed treatment), Brazil, 1997-98.

Location	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Paulinia, Soa Paulo	SC 300 g/l	1	3	NR	< 0.2 <0.2 <0.2	Anon., 1998a Study No.CP-2557/98
Paulinia, Soa Paulo	SC 300 g/l	1	6	NR	< 0.2 <0.2 <0.2	Anon., 1998a Study No.CP-2557/98
Ponta Grossa	SC 300 g/l	1	3	NR	<0.2 <0.2 <0.2	Anon., 1998b Study No.001/98-PC
Ponta Grossa	SC 300 g/l	1	6	NR	<0.2 <0.2 <0.2	Anon., 1998b Study No.001/98-PC

Maize. Four supervised trials were conducted in Brazil with ground foliar and seed treatment applications, and in Australia one trial with foliar application only. Samples were analysed by GLC (sulfur FPD, LOQ 0.02 mg/kg) or HPLC (LOQ 0.1 mg/kg).

Table 84. Residues in maize grain (foliar application), Paulinia, Brazil, 1995.

Application			Residues		Reference
Formulation	No.	kg ai/ha	PHI, days	mg/kg	
WG 800g/l	--	0.1	30		
SC 375 g/l	1	0.18	30	≤0.1 <0.1	Anon., 1996, CP-2344/96, Study No. 049/95-PC
SC 375 g/l	1	0.35	30	≤0.1 <0.1	Anon., 1996, CP-2344/96, Study No. 049/95-PC
WDG 800 g/l	1	0.2	30	≤0.1 <0.1	Anon., 1996, CP-2344/96, Study No. 049/95-PC
WDG 800 g/l	1	0.4	30	≤0.1 <0.1	Anon., 1996, CP-2344/96, Study No. 049/95-PC
WG 800 g/l	1	0.2	30	≤0.1 <0.1	Anon., 1996, CP-2344/96, Study No. 049/95-PC
WG 800 g/l	1	0.4	30	≤0.1 <0.1	Anon., 1996, CP-2344/96, Study No. 049/95-PC

Table 85. Residues in maize grain (seed treatment)

Location, year	Application			Residues		Reference
	Formulation	No.	kg/tonne seeds	PHI, days	mg/kg	
GAP, Brazil	SC 375 g/l	1	7			
Emerald, Queensland, Australia, 1988	SC 375 g/l	1	7.5	120	<0.02 <0.02	Keats, 1989b, ak/am/ak89010
Londrina, Brazil, 1999-2000	SC 375 g/l	1	7.5	99	<u>≤0.10</u>	Anon., 2000
Londrina, Brazil, 1999-2000	SC 375 g/l	1	14	99	<u>≤0.10</u>	Anon., 2000
Paulinia, Brazil, 1999-2000	SC 375 g/l	1	7.5	90	<u>≤0.10</u>	Anon., 2000
Paulinia, Brazil, 1999-2000	SC 375 g/l	1	14	90	<u>≤0.10</u>	Anon., 2000

<u>Rice</u>. Supervised trials were conducted in Japan with ground foliar application. Grain was analysed by GLC (sulfur FPD, LOQ 0.25 mg/kg or 0.4 mg/kg, limit of detection 0.008 mg/kg). In four supervised trials in Brazil with seed treatment, grain was analysed by HPLC with an LOQ of 0.10 mg/kg.

Table 86. Residues in brown rice grain (foliar spray application), Japan, 1985.

Application			Residues		Reference
Formulation	No.	kg ai/ha	PHI, days	mg/kg	
DP 30 g/kg	3	0.9-1.2	30		
DP 30 g/kg	3	1.2	21 28 45	<0.25 (0.086) <u>≤0.25</u> (0.024) <0.008	Anon., 2001b Aventis CropScience Saku61p-2-55; Ibaraki Tokyo Kenbikyo Foundation LOQ 0.25 mg/kg
DP 30 g/kg	3	1.2	21 28 45	<0.25 (0.080) <u>≤0.25</u> (0.010) <0.008	Anon., 2001b Aventis CropScience Saku61p-2-55; Kochi Tokyo Kenbikyo Foundation LOQ 0.25 mg/kg
DP 30 g/kg	3	1.2	21 28 45	<0.4 (0.12) <u>≤0.4</u> (0.040) <0.008	Anon., 2001b Aventis CropScience Ibaraki Hokko Chemical Industry LOQ 0.4 mg/kg
DP 30 g/kg	3	1.2	21 28 45	<0.4 (0.10) <u>≤0.4</u> (0.041) <0.008	Anon., 2001b Aventis CropScience Saku61p-2-55; Kochi Hokko Chemical Industry LOQ 0.4 mg/kg

Table 87. Residues in rice grain (seed treatment), Brazil, 1999-2000.

Location	Application			Residues		Reference
	Formulation	No	kg/tonne seeds	PHI, days	mg/kg	
GAP, Brazil	SC 350 g/l	1	5.25	--		
Paulinia	SC 375 g/l	1	5.25	148	<u>≤0.10</u>	Anon., 2000c, No.2971/00
Paulinia	SC 375 g/l	1	10.5	148	<0.10	Anon., 2000c, No.2971/00
Rio Verde	SC 375 g/l	1	7.5	148	<0.10	Anon., 2000c, No.2972/00
Rio Verde	SC 375 g/l	1	14	148	<0.10	Anon., 2000c, No.2972/00

<u>Sorghum</u>. Supervised trials were conducted in the USA and Australia with ground foliar application, and in the USA also with seed treatment application. There is no GAP in the USA for foliar or seed treatment application and no GAP in Australia for foliar application.

Table 88. Residues in sorghum grain (foliar spray application).

Country, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Manteca, CA USA, 1982	SC 375 g/l	3	0.28	14 21 28	0.22 0.47 0.26 0.39 0.68 0.49 0.13 0.27 0.16	Hunt, 1988f Project No.804R10 File No.35252
	SC 375 g/l	3	0.56	14 21 28	1.6 1.5 1.5 1.4 2.0 1.3 0.46 0.79 0.55	
	SC 375 g/l	3	0.84	14 21 28	1.2 1.6 1.7 1.5 1.5 1.9 0.66 0.64 0.69	
Newton, IA USA, 1982	SC 375 g/l	3	0.28	14 21 28	0.18 0.24 0.44 0.15 0.06 <0.04 <0.04 0.06 <0.04 <0.04 <0.04 <0.04	Hunt, 1988f Project No.804R10 File No.35252
	SC 375 g/l	3	0.56	14 21 28	0.87 1.2 0.75 0.73 0.05 0.05 0.43 0.16 <0.04 <0.04 <0.04 <0.04	
	SC 375 g/l	3	0.84	14 21 28	1.4 0.94 1.2 0.66 0.12 0.06 0.10 0.17 <0.04 <0.04 <0.04 <0.04	

Country, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Wayside, MS USA, 1982	SC 375 g/l	3	0.28	14 21 28 	0.64 0.33 0.31 0.32 0.13 0.12 0.34 0.32 <0.04 <0.04 <0.04 0.20	Hunt, 1988f Project No.804R10 File No.35252
	SC 375 g/l	3	0.56	14 21 28 	0.58 0.57 2.0 0.96 0.38 0.30 0.81 0.88 <0.04 0.10 <0.04 <0.04	
	SC 375 g/l	3	0.84	14 21 28 	1.9 2.0 2.4 1.3 1.8 0.47 0.60 1.2 0.10 0.79 0.60 0.49	
Clayton, NC USA, 1982	SC 375 g/l	3	0.28	14 21 28 	ND (<0.02) ND ND ND ND ND ND ND ND	Hunt, 1988f Project No.804R10 File No.35252
	SC 375 g/l	3	0.56	14 21 28 	ND ND ND ND ND ND ND ND ND	
	SC 375 g/l	3	0.84	14 21 28 	ND ND ND ND ND ND ND ND ND	

Manteca, CA USA, 1983	SC 375 g/l	3	0.84	14	1.3 0.97 0.75	Hunt, 1988f Project No.804R10 File No.35252
Newton, IA USA, 1983	SC 375 g/l	3	0.84	13	4.5 5.7 6.6 6.1	Hunt, 1988f Project No.804R10 File No.35252
Manhattan, KS USA, 1983	SC 375 g/l	3	0.84	14	5.4 19 8.5	Hunt, 1988f Project No.804R10 File No.35252
Wayside, MS USA, 1983	SC 375 g/l	3	0.84	14	ND ND ND ND	Hunt, 1988f Project No.804R10 File No.35252
Clayton, NC USA, 1983	SC 375 g/l	3	0.84	14	0.86 0.68 0.69 1.4	Hunt, 1988f Project No.804R10 File No.35252
York, NE USA, 1983	SC 375 g/l	3	0.84	14	0.55 2.6 0.51	Hunt, 1988f Project No.804R10 File No.35252
Brookings, SD USA, 1983	SC 375 g/l	3	0.84	14	ND ND ND	Hunt, 1988f Project No.804R10 File No.35252
Burleson County, TX USA, 1983	SC 375 g/l	3	0.84	14	4.4 2.7 6.4	Hunt, 1988f Project No.804R10 File No.35252
Nobby, Darling Downs Queensland Australia, 1989	SC 375 g/l	1	0.49	215	<0.02 <0.02	Keats, 1989c, ak/am/ak89008
Emerald, Queensland Australia, 1989	SC 375 g/l	1	0.49	103	<0.02 <0.02 <0.02 <0.02	Keats, 1989c ak/am/ak89008

Table 89. Residues in sorghum grain (seed treatment), USA.

Location, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Wayside, MS 1984	SC 375 g/l	1	10	91	ND ND ND ND	Hunt, 1988g Project No.804R10 File No.40387
Newton, IA 1985	900 g/kg	1	10	145	ND ND ND ND	Hunt, 1988g Project No.804R10 File No.40387
Wayside, MS 1985	900 g/kg	1	20	99	ND <0.02 <0.02 ND	Hunt, 1988g Project No.804R10 File No.40387
Clayton, NC 1985	900 g/kg	1	20	113	ND ND ND ND	Hunt, 1988g Project No.804R10 File No.40387
Frisco, TX 1985	900 g/kg	1	20	170	<0.04	Hunt, 1988g Project No.804R10 File No.40387
Newton, IA 1985	900 g/kg + vitavax	1	20	145	ND ND ND ND	Hunt, 1988g Project No.804R10 File No.40387
Wayside, MS 1985	900 g/kg + vitavax	1	20	99	ND <0.02 <0.02 ND	Hunt, 1988g Project No.804R10 File No.40387

Location, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Clayton, NC 1985	900 g/kg + vitavax	1	20	113	ND ND ND ND	Hunt, 1988g Project No.804R10 File No.40387

Crops used as animal feed

Barley and wheat. Residues in barley and wheat plant parts used as feed are shown in Tables 90-93.

Table. 90 Residues in the whole plant (forage) of barley and wheat (granular bait application), Germany, 1987-88.

Crop, Location	Application			Part	Residues		Reference
	Formulation	No.	kg ai/ha		PHI, days	mg/kg	
GAP, UK	GB 40 g/kg	1-3	0.2		--		
WHEAT Salzhemmendorf Niedersachsen	GB 40 g/kg	2	0.2	Plant	49 161 194	ND (<0.02) ND ND	Anon., 1992d BBA report No.05148, 1992 LOQ 0.05 mg/kg
WHEAT Salzhemmendorf Niedersachsen	GB 40 g/kg	2	0.2	Plant	7 19 42	ND ND ND	Anon., 1992d BBA report No.05149, 1992
WHEAT Schwaighofen Bayern	GB 40 g/kg	2	0.2	Green Plant	7 38 76	ND ND ND	Anon., 1992d BBA report No.03675,1992
WINTER BARLEY Gerolfingen	GB 40 g/kg	2	0.2	Plant	6 143 193	ND (<0.02) ND ND	Anon., 1992b BBA report No. 03725, 1992 LOQ 0.05 mg/kg
WINTER BARLEY Gerolfingen	GB 40 g/kg	2	0.2	Plant	7 36	ND ND	Anon., 1992b BBA report No. 05132, 1992

Table 91. Residues in barley and wheat leaves (forage; granular bait application), UK.

CROP Country, year	Application			Part	Residues		Reference
	Formulation	No.	kg ai/ha		PHI, days	mg/kg	
GAP, UK	RB 40 g/kg	1-3	0.2		--		
WINTER BARLEY Trent, Dorset, 1988	GB 40 g/kg	4	0.3	Leaves	46	0.25	Brockelsby *et al.*, 1989 Report No. D.Ag.1295
WINTER BARLEY Thirsk, North Yorkshire, 1988	GB 40 g/kg	4	0.3	Leaves	0	<0.04	Brockelsby *et al.*, 1989 Report No. D.Ag.1295
WINTER BARLEY Chelmsford, Essex, 1988	GB 40 g/kg	4	0.3	Leaves	0	0.04	Brockelsby *et al.*, 1989 Report No. D.Ag.1295
WINTER BARLEY West Hayes, Bedfordshire, 1990	GB 40 g/kg	3	0.2	Leaves	42	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No. D.Ag.1535
WINTER BARLEY Eastwell, Leicestershire, 1989-90	GB 40 g/kg	3	0.2	Leaves	126	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No. D.Ag.1535
WHEAT Brentwood, Essex 1989-90	GB 40 g/kg	3	0.2	Leaves	84	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No.D.Ag.1535
WHEAT Reepham, Lincolnshire, 1989-90	GB 40 g/kg	3	0.2	Leaves	133	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No.D.Ag.1535
WHEAT Abberley, Worcestershire, 1989-90	GB 40 g/kg	3	0.2	Leaves	14	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No.D.Ag.1535

CROP Country, year	Application			Part	Residues		Reference
	Formulation	No.	kg ai/ha		PHI, days	mg/kg	
WHEAT Northallerton, North Yorkshire, 1987-88	GB 40 g/kg	3	0.4	Leaves	0	<0.04	Brockelsby *et al.*, 1989 Report No.D.Ag.1295
WHEAT Haywards Heath, Sussex, 1987-88	GB 40 g/kg	4	0.3	Leaves	0	0.21	Brockelsby *et al.*, 1989 Report No.D.Ag.1295
WHEAT West Midlands, Worcestershire, 1986	RB 40 g/kg	2	0.2	Leaves at 2 leaf stage	60	< 0.03 <0.03	Blundstone *et al.*, 1987 U.C.Project No.I60/03/11/86
WHEAT West Midlands, Worcestershire, 1986	RB 40 g/kg	2	0.4	Leaves at 2 leaf stage	60	ND ND	Blundstone *et al.*, 1987 U.C.Project No.I60/03/11/86
WHEAT West Midlands, Worcestershire, 1986	RB 40 g/kg	3	0.2	Leaves at 2 leaf stage	3	0.06 0.05	Blundstone *et al.*, 1987 U.C.Project No.I60/03/11/86
WHEAT West Midlands, Worcestershire, 1986	RB 40 g/kg	3	0.4	Leaves at 2 leaf stage	3	<0.03 ND	Blundstone *et al.*, 1987 U.C.Project No.I60/03/11/86

Table 92. Residues in barley and wheat straw (granular bait application).

CROP Country, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, UK	RB 40 g/kg	1-3	0.2	--		
WINTER BARLEY Trent, Dorset, UK, 1988	GB 40 g/kg	4	0.3	138	<0.04	Brockelsby *et al.*, 1989 Report No. D.Ag.1295
WINTER BARLEY Thirsk, North Yorkshire, UK, 1988	GB 40 g/kg	4	0.3	162	<0.04	Brockelsby *et al.*, 1989 Report No. D.Ag.1295
WINTER BARLEY Chelmsford, Essex, UK, 1988	GB 40 g/kg	4	0.3	105	<0.04 <0.04	Brockelsby *et al.*, 1989 Report No. D.Ag.1295
WINTER BARLEY Chapeltown, Derbyshire, UK, 1988	GB 40 g/kg	4	0.3	158	<0.04 <0.04	Brockelsby *et al.*, 1989 Report No. D.Ag.1295
WINTER BARLEY West Hayes, Bedfordshire, UK, 1990	GB 40 g/kg	3	0.2	133	0.24 0.16 0.13	Brockelsby *et al.*, 1990a Report No. D.Ag.1535
WINTER BARLEY Eastwell, Leicestershire, UK, 1989-90	GB 40 g/kg	3	0.2	224	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No. D.Ag.1535
WINTER BARLEY Abberley, Worcestershire, UK, 1989-90	GB 40 g/kg	3	0.2	175	<0.04 <0.04 <0.04	Brockelsby *et al.*, 1990a Report No. D.Ag.1535
WHEAT Salzhemmendorf, Niedersachsen Germany 1987-88	GB 40 g/kg	2	0.2	281	ND (<0.2)	Anon., 1992d BBA report No.05148, 1992. LOQ 0.5 mg/kg
WHEAT Salzhemmendorf, Niedersachsen Germany 1987-88	GB 40 g/kg	2	0.2	101	ND	Anon., 1992d BBA report No.05149, 1992
WHEAT Schwaighofen, Bayern Germany, 1987-88	GB 40 g/kg	2	0.2	136	ND	Anon., 1992d BBA report No.03675, 1992
WINTER BARLEY Gerolfingen Germany, 1987-88	GB 40 g/kg	2	0.2	239	ND (<0.2)	Anon., 1992b BBA report No. 03725, 1992 LOQ 0.5 mg/kg
WINTER BARLEY Gerolfingen, Germany, 1987-88	GB 40 g/kg	2	0.2	93	ND	Anon., 1992b BBA report No. 05132, 1992

Table 93. Residues in the whole plant of barley (forage; foliar application).

CROP Country, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
WINTER BARLEY Colombiers France, 1986	SC 375 g/l	1	0.25	7 15 45	<0.02 <0.02 <0.02	Mestre, 1986 ET/FB/AB/67

Rice. Four supervised trials were conducted in Japan with foliar application (see Table 86). GAP in Japan consists of a rate of 0.9-1.2 kg ai/ha, applied up to 3 times, and a PHI of 30 days. Straw was analysed by GLC (sulfur FPD, LOQ 0.5 to 1.0 mg/kg).

Table 94. Residues in rice straw (foliar application), Japan, 1985.

Application			Residues		Reference
Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, Japan, DP 30 g/kg	3	0.9-1.2	30		
DP 30 g/kg	3	1.2	21 28 45	0.76 0.62 0.19	Anon., 2001b Aventis CropScience 08-18 ASaku61p-2-55; Ibaraki Tokyo Kenbikyo Foundation LOQ 0.5 mg/kg
DP 30 g/kg	3	1.2	21 28 45	1.4 0.40 0.25	Anon., 2001b Aventis CropScience Saku61p-2-55; Kochi Tokyo Kenbikyo Foundation LOQ 0.5 mg/kg
DP 30 g/kg	3	1.2	21 28 45	1.0 0.69 0.25	Anon., 2001b Aventis CropScience Ibaraki Hokko Chemical Industry LOQ 1.0 mg/kg
DP 30 g/kg	3	1.2	21 28 45	1.6 0.50 0.36	Anon., 2001b Aventis CropScience Saku61p-2-55; Kochi Hokko Chemical Industry LOQ 1.0 mg/kg

Sorghum. Residues in plant parts used as animal feed are shown in Tables 95-99.

Table 95. Residues in sorghum forage (foliar application), USA.

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Manteca, CA 1982	SC 375 g/l	1	280	8	8.8 6.8 8.1	Hunt, 1988f Project No.804R10 File No.35252
Manteca, CA 1982	SC 375 g/l	1	560	8	18 6.0 26	Hunt, 1988f Project No.804R10 File No.35252
Manteca, CA 1982	SC 375 g/l	1	841	8	23 23 21	Hunt, 1988f Project No.804R10 File No.35252
Newton, IA 1982	SC 375 g/l	3	280	7	0.22 0.27 0.16 0.18	Hunt, 1988f Project No.804R10 File No.35252
Newton, IA 1982	SC 375 g/l	3	560	7	0.55 0.39 0.51 0.45	Hunt, 1988f Project No.804R10 File No.35252
Newton, IA 1982	SC 375 g/l	3	841	7	1.8 1.3 1.2 1.6	Hunt, 1988f Project No.804R10 File No.35252

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Wayside, MS 1982	SC 375 g/l	2	280	7	0.25 5.0 0.39 0.16	Hunt, 1988f Project No.804R10 File No.35252
Wayside, MS 1982	SC 375 g/l	2	560	7	0.59 0.62 0.35 0.68	Hunt, 1988f Project No.804R10 File No.35252
Wayside, MS 1982	SC 375 g/l	2	841	7	1.5 1.4 2.0 5.6	Hunt, 1988f Project No.804R10 File No.35252
Clayton, NC 1982	SC 375 g/l	3	280	7	0.12 0.03 0.03	Hunt, 1988f Project No.804R10 File No.35252
Clayton, NC 1982	SC 375 g/l	3	560	7	0.04 0.05 0.04	Hunt, 1988f Project No.804R10 File No.35252
Clayton, NC 1982	SC 375 g/l	3	841	7	0.05 0.09 0.15	Hunt, 1988f Project No.804R10 File No.35252
Manteca,CA 1983	SC 375 g/l	3	841	7	8.6 5.2 6.3	Hunt, 1988f Project No.804R10 File No.35252
Newton, IA 1983	SC 375 g/l	3	841	7	0.98 0.91 1.9 1.3	Hunt, 1988f Project No.804R10 File No.35252
Manhattan, KS 1983	SC 375 g/l	3	841	7	12 28 24	Hunt, 1988f Project No.804R10 File No.35252
Wayside, MS 1983	SC 375 g/l	1	841	7	0.52 0.46 0.41 0.45	Hunt, 1988f Project No.804R10 File No.35252
Clayton, NC 1983	SC 375 g/l	3	841	7	14 20 13 12	Hunt, 1988f Project No.804R10 File No.35252
York, NE 1983	SC 375 g/l	3	841	7	4.8 2.8 5.6	Hunt, 1988f Project No.804R10 File No.35252
Brookings, SD 1983	SC 375 g/l	3	841	7	ND ND ND	Hunt, 1099f Project No.804R10 File No.35252
Burleson County, TX 1983	SC 375 g/l	3	841	7	9.3 9.7 13	Hunt, 1988f Project No.804R10 File No.35252

Table 96. Residues in sorghum forage (seed treatment), USA.

Country, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Wayside, MS 1984	SC 375 g/l	1	10	29	0.08 0.11 0.09 0.02	Hunt, 1988g Project No.804R10 File No.40387
Newton, IA 1985	900 g/kg	1	10	27	0.04 0.03 0.07 0.11	Hunt, 1988g Project No.804R10 File No.40387

Country, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Clayton, NC 1985	900 g/kg	1	20	29	ND ND ND 0.03	Hunt, 1988g Project No.804R10 File No.40387
Frisco, TX 1985	900 g/kg	1	20	21	<0.04 <0.04	Hunt, 1988g Project No.804R10 File No.40387
Newton, IA 1985	900 g/kg + vitavax	1	20	27	0.02 0.04 0.05 0.04	Hunt, 1988g Project No.804R10 File No.40387
Clayton, NC 1985	900 g/kg + vitavax	1	20	29	ND ND <0.02 0.02	Hunt, 1988g Project No.804R10 File No.40387

Table 97. Residues in sorghum stubble straw (foliar application), Australia, 1989.

Location	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Emerald, Queensland	SC 375 g/l	1	0.49	103	<0.02 <0.02 <0.02 <0.02	Keats, 1989c ak/am/ak89008

Table 98. Residues in sorghum stover (foliar application), USA.

Country, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Manteca, CA 1982	SC 375 g/l	3	0.280	14 21 28 	6.7 8.0 6.7 11 6.7 8.6 8.8 5.4 5.1	Hunt, 1988f Project No.804R10 File No.35252
	SC 375 g/l	3	0.560	14 21 28 	13 19 12 16 17 12 10 9.8 9.4	
	SC 375 g/l	3	0.841	14 21 28 	22 30 24 28 38 27 18 25 29	

Country, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Newton, IA 1982	SC 375 g/l	3	0.280	14 21 28 	0.30 0.33 0.21 0.10 0.05 0.07 <0.04 0.18 <0.04 <0.04 <0.04 <0.04	Hunt, 1988f Project No.804R10 File No.35252
	SC 375 g/l	3	0.560	14 21 28 	0.80 0.71 0.47 0.85 0.14 0.12 0.07 0.11 <0.04 <0.04 <0.04 <0.04	
	SC 375 g/l	3	0.841	14 21 28 	0.50 1.3 1.2 0.95 0.11 0.28 0.17 0.14 <0.04 <0.04 0.17 0.11	
Wayside, MS 1982	SC 375 g/l	3	0.280	14 21 28 	0.82 0.29 0.47 0.43 0.12 0.07 <0.04 <0.04 <0.04 <0.04 <0.04 <0.04	Hunt, 1988f Project No.804R10 File No.35252
	SC 375 g/l	3	0.560	14 21 28 	0.50 0.93 0.51 0.57 0.12 0.07 0.05 0.11 <0.04 <0.04 <0.04 <0.04	

Country, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
	SC 375 g/l	3	0.841	14 21 28	1.1 1.0 0.84 1.3 0.64 0.47 0.61 0.45 0.30 0.30 0.37 0.31	
Clayton, NC 1982	SC 375 g/l	3	0.280	14 21 28	ND (<0.02) ND ND ND ND ND ND ND ND	Hunt, 1988f Project No.804R10 File No.35252
	SC 375 g/l	3	0.560	14 21 28	ND ND ND ND ND ND ND ND ND	
	SC 375 g/l	3	0.841	14 21 28	ND ND ND ND ND ND ND ND ND	
Manteca, CA 1983	SC 375 g/l	3	0.841	14	4.7 4.9 3.0	Hunt, 1988f Project No.804R10 File No.35252
Newton, IA 1983	SC 375 g/l	3	0.841	13	7.2 7.3 6.3 5.1	Hunt, 1988f Project No.804R10 File No.35252
Manhattan, KS 1983	SC 375 g/l	3	0.841	14	5.4 2.5 4.0	Hunt, 1988f Project No.804R10 File No.35252
Wayside, MS 1983	SC 375 g/l	3	0.841	14	<0.10 <0.10 <0.10 <0.10	Hunt, 1988f Project No.804R10 File No.35252
Clayton, NC 1983	SC 375 g/l	3	0.841	14	0.12 0.40 0.54 0.36	Hunt, 1988f Project No.804R10 File No.35252
York, NE 1983	SC 375 g/l	3	0.841	14	1.0 1.0 0.8	Hunt, 1988f Project No.804R10 File No.35252
Brookings, SD 1983	SC 375 g/l	3	0.841	14	0.11 0.11 <0.10	Hunt, 1988f Project No.804R10 File No.35252

Country, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Burleson County,TX 1983	SC 375 g/l	3	0.841	14	9.5 2.9 2.3	Hunt, 1988f Project No.804R10 File No.35252

Table 99. Residues in sorghum stover (seed treatment), USA.

Location, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Wayside, MS 1984	SC 375 g/l	1	10	91	ND (<0.01) ND ND ND	Hunt, 1988g, Project No.804R10 File No.40387
Newton, IA 1985	900 g/kg	1	10	145	ND ND ND ND	Hunt, 1988g, Project No.804R10 File No.40387
Wayside, MS 1985	900 g/kg	1	20	99	0.07 0.02 <0.02 <0.02	Hunt, 1988g, Project No.804R10 File No.40387
Clayton, NC 1985	900 g/kg	1	20	114	ND ND 0.03 <0.02	Hunt, 1988g, Project No.804R10 File No.40387
Frisco, TX 1985	900 g/kg	1	20	170	<0.04	Hunt, 1988g, Project No.804R10 File No.40387
Newton, IA 1985	900 g/kg + vitavax	1	20	145	ND ND ND ND	Hunt, 1988g, Project No.804R10 File No.40387
Wayside, MS 1985	900 g/kg + vitavax	1	20	99	<0.02 <0.02 <0.02 0.23	Hunt, 1988g, Project No.804R10 File No.40387
Clayton, NC 1985	900 g/kg + vitavax	1	20	114	ND 0.02 ND ND	Hunt, 1988g, Project No.804R10 File No.40387

Sweet corn. Residues in stalks and forage are shown in Tables 100-102.

Table 100. Residues in sweet corn fodder (stalk; foliar application), Australia, 1989.

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, Australia	SC 375 g/l	--	0.56-0.75	7		
Cowra, New South Wales	SC 375 g/l	2	0.525	20	0.02	Keats, 1989d ak/aw/ak89005
Cowra, New South Wales	SC 375 g/l	2	1.050	20	0.04	Keats, 1989d ak/aw/ak89005

Table 101. Residues in sweet corn forage (foliar application), USA, 1985.

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.84	21		Do not exceed 3.36 kg/ha per season
Santa Maria, CA	SC 375 g/l	4	0.84	7 14 21	22 ND 11	Hunt and Schwehr, 1987 Project No.804R10 File No.35293
Manteca, CA	SC 375 g/l	4	0.84	7 14 21	<0.05 <0.05 <0.05 5.8 9.9 5.4 18 13 11	Hunt and Schwehr, 1987 Project No.804R10 File No.35293
Sanford, FL	SC 375 g/l	4	0.84	7 14 21	0.96 1.2 1.1 ND 0.24 0.12 ND ND ND	Hunt and Schwehr, 1987 Project No.804R10 File No.35293
Geneseo, IL	SC 375 g/l	4	0.84	7 14 21	14 29 23 3.6 3.3 4.4 4.7 2.9 6.9	Hunt and Schwehr, 1987 Project No.804R10 File No.35293
Newton, IA	SC 375 g/l	4	0.84	7 14 21	1.3 0.78 2.2 1.0 2.1 2.4 1.1 0.51 0.37	Hunt and Schwehr, 1987 Project No.804R10 File No.35293
Haslett, MI	SC 375 g/l	4	0.84	7 14 21	2.9 4.4 7.6 <0.05 1.1 <0.05 <0.05 <0.05 <0.05	Hunt and Schwehr, 1987 Project No.804R10 File No.35293
Lamberton, MN	SC 375 g/l	4	0.84	7 14 21	0.18 <0.05 0.07 <0.05 <0.05 0.05 <0.05 0.06 <0.05	Hunt and Schwehr, 1987 Project No.804R10 File No.35293

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Wayside, MS	SC 375 g/l	4	0.84	7	13	Hunt and Schwehr, 1987
					20	Project No.804R10
					12	File No.35293
				14	0.45	
					0.32	
					0.28	
				21	0.21	
					2.3	
					0.19	
Bridgetown, NJ	SC 375 g/l	4	0.84	7	0.18	Hunt and Schwehr, 1987
					0.29	Project No.804R10
					0.16	File No.35293
				14	0.11	
					0.05	
					0.07	
				21	0.06	
					0.21	
					ND	
Phelps, NY	SC 375 g/l	4	0.84	7	0.26	Hunt and Schwehr, 1987
					0.32	Project No.804R10
					0.59	File No.35293
				14	0.09	
					0.26	
					0.42	
				21	0.06	
					0.11	
					0.16	
Clayton, NC	SC 375 g/l	4	0.84	7	0.69	Hunt and Schwehr, 1987
					0.90	Project No.804R10
					0.64	File No.35293
				14	0.54	
					0.62	
					1.1	
				21	0.54	
					0.24	
					0.56	
Prosser, WA	SC 375 g/l	4	0.84	7	7.3	Hunt and Schwehr, 1987
					8.3	Project No.804R10
					5.4	File No.35293
				14	1.0	
					5.2	
					0.87	
				21	5.1	
					5.2	
					2.9	

Table 102. Residues in sweet corn forage (seed treatment), USA, 1988.

Location, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Manteca, CA	SC 375 g/l	1	10	85	ND	Hunt, 1988i
					(<0.02)	Project No.804R10
					ND	File No.40389
					ND	
					0.05	
Redfield, IA	SC 375 g/l	1	10	81	ND	Hunt, 1988i
					ND	Project No.804R10
					ND	File No.40389
					ND	

Location, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Newton, IA	SC 375 g/l	1	10	13	0.44	Hunt, 1988i
					0.29	Project No.804R10
					0.34	File No.40389
					0.36	
				20	0.32	
					1.2	
					0.30	
					0.21	
				33	ND	
					ND	
					ND	
					ND	
				69	ND	
					ND	
					ND	
					ND	
				77	ND	
					ND	
					ND	
					0.05	
Redfield, IA	SC 375 g/l	1	10	69	ND	Hunt, 1988i
					ND	Project No.804R10
					ND	File No.40389
					ND	
Clayton, NC	SC 375 g/l + vitavax	1	10	81	ND	Hunt, 1988i
					ND	Project No.804R10
					ND	File No.40389
					ND	
Manteca, CA	900 g/kg	1	20	76	<0.05	Hunt, 1988i
					ND	Project No.804R10
					ND	File No.40389
					ND	
Newton, IA	900 g/kg	1	20	14	11	Hunt, 1988i
					9.0	Project No.804R10
					11	File No.40389
					9.2	
				21	2.9	
					3.5	
					2.6	
					1.2	
				35	0.07	
					<0.05	
					<0.05	
					<0.05	
				68	0.08	
					0.06	
					<0.05	
					ND	
				90	ND	
					ND	
					ND	
					ND	
Wayside, MS,	900 g/kg	1	20	68	0.27	Hunt, 1988i
					0.45	Project No.804R10
					0.25	File No.40389
					0.34	
Clayton, NC	900 g/kg	1	20	107	ND	Hunt, 1988i
					ND	Project No.804R10
					ND	File No.40389
					ND	
Rochester, NY	900 g/kg	1	20	83	ND	Hunt, 1988i
					ND	Project No.804R10
					ND	File No.40389
					ND	

Location, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Rochester, NY,	900 g/kg	1	20	95	ND ND ND ND	Hunt, 1988i Project No.804R10 File No.40389

Oilseed

Cotton. Supervised trials were conducted in Brazil, Australia, USA, Greece, Sudan and France with ground foliar application, and in the USA also some with aerial foliar application and others with seed treatment (no GAP). Grain, leaves, staple cotton and forage from the US trials were analysed by GLC (sulfur FPD, LOQ 0.04 mg/kg) and by CG-MS (limit of detection (LOD) 0.04 mg/kg). Cotton seeds from the Australian trials were analysed by GLC (sulfur FPD), and from the Greek trials by CG-MS (LOD 0.04 mg/kg and LOQ 0.10 mg/kg).

Table 103. Residues in cotton seed with linter (foliar application), Brazil, 1999-2000.

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Maracaju	WP 800 g/kg	1	0.24	7	<0.1	Anon., 2000 Study No.2988/00
Maracaju	WP 800 g/kg	1	0.48	7	<0.1	Anon., 2000 Study No.2988/00
Uberlândia,	WP 800 g/kg	1	0.24	7	<0.1	Anon., 2000 Study No.2988/00
Uberlândia	WP 800 g/kg	1	0.48	7	<0.1	Anon., 2000 Study No.2988/00

Table 104. Residues in cotton seed (foliar application).

Country, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
GAP, Australia	SC 375 g/l	--	0.75-0.84		21		
Wee Waa, North West New South Wales Australia, 1982	SC 375 g/l	6	0.84		0 7 14 21	0.30 0.15 0.08 0.05	Hunt and Langdon, 1983 Report No.1452V
Wee Waa, North West New South Wales Australia, 1982	SC 375 g/l	6	1.7		0 7 14 21	0.88 0.28 0.59 0.09	Hunt and Langdon, 1983 Report No.1452V
GAP, USA	SC 375 g/l	--	0.14-1.0		28		
El Centro, CA USA, 1988	SC 375 g/l	2	1.009		46	ND (<0.02) ND ND	Hunt, 1989c Project No.804R10 File No.40512
El Centro, CA USA, 1988	SC 375 g/l	2	1.009		46	ND ND ND	Hunt, 1989c Project No.804R10 File No.40512
Litchfield Park, AZ USA, 1988	SC 375 g/l	2	0.673		44	ND 0.04 <0.04	Hunt, 1989c Project No.804R10 File No.40512
Litchfield Park, AZ USA, 1988	SC 375 g/l	2	0.673		44	<0.04 <0.04 <0.04	Hunt, 1989c Project No.804R10 File No.40512
Fresno, CA USA, 1988	SC 375 g/l	2	1.009		33	0.10 0.06 0.04	Hunt, 1989c Project No.804R10 File No.40512
Poplar, CA USA, 1988	SC 375 g/l	2	1.009		45	<0.04 <0.04 <0.04	Hunt, 1989c Project No.804R10 File No.40512

Country, year	Application				Residues		Reference
	Formulation	No.	kg ai/ha	kg ai/hl	PHI, days	mg/kg	
Poplar, CA USA, 1988	SC 375 g/l	2	1.009		45	0.04 <0.04 <0.04	Hunt, 1989c Project No.804R10 File No.40512
El Campo, TX USA, 1998-99	SC 375 g/l	6	5.045		28	0.22	Lee, 1991d 90-047;, File No.40969 Study No.USA90L82
Caddo, OK USA, 1991	SC 375 g/l	6	1.022		28	<u>≤0.04</u> <0.04 <0.04	Lee, 1992b 91-005, Study No.USA91L82 File No.41198
Uvalde, TX USA, 1991	SC 375 g/l	6	1.026		28	<u>≤0.04</u> <0.04 <0.04	Lee, 1992b 91-007, Study No.USA91L82 File No.41198
Rapids, LA USA, 1991	SC 375 g/l	6	0.994		28	<u>≤0.04</u> <0.04 <0.04	Lee, 1992b 91-026, Study No.USA91L82 File No.41198
Lonoke, AR USA, 1991	SC 375 g/l	6	1.009		28	<u>≤0.04</u> <0.04 <0.04	Lee, 1992b 91-039, Study No.USA91L82 File No.41198
Hidalgo, TX USA, 1991	SC 375 g/l	6	1.007		28	<u>≤0.04</u> <0.04 <0.04	Lee, 1992b 91-071, Study No.USA91L82 File No.41198
Mitchell, GA USA, 1991	SC 375 g/l	6	1.035		28	0.08 0.07 <u>0.10</u>	Lee, 1992b 91-102, Study No.USA91L82 File No.41198
Washington, MS USA, 1991	SC 375 g/l	6	1.009		28	<u>≤0.04</u> <0.04 <0.04	Lee, 1992b 91-250, Study No.USA91L82 File No.41198
GAP, Greece	WG 800 g/kg	2-3	0.56-0.8		28		
Arma Viotas Greece, 1998	WG 800 g/kg	3	0.805		21 28	<0.04 <0.04 <0.04 <u>≤0.04</u>	Jendrzejczak and Yslan, 2000 99657, Study No.99-657
Thiva Viotia Greece, 1994	WG 800 g/kg	2	0.80	0.1	22	<u>≤0.05</u>	Richard and Muller, 1995c Study 94-692
Thiva Viotia Greece, 1994	WG 800 g/kg	1	0.80	0.1	42	<0.05	Richard and Muller, 1995c Study 94-692
GAP: Sudan (Egypt)	SC 375 g/l		0.89		28		
Arc Wad Me Sudan, 1998-99	WG 800 g/kg	3	0.571		61	0.59 0.33 0.23 <0.08	Lusson and Muller, 1989 b AG/CRLD/AN 8916381
GAP, Spain	SC 375 g/l		0.72		21		
Torre de la Reina, Seville Spain, 1993	SC 375 g/l	6	0.75		67	<0.05 <0.05	Richard and Muller, 1994c Study 93-625
Spain, 2000	WG 800 g/kg	1	0.24		7	<0.1	Anon., 2000d ESALQ/USP
Spain, 2000	WG 800 g/kg	1	0.48		7	<0.1	Anon., 2000e ESALQ/USP

Table 105. Residues in cotton seed (aerial application), USA, 1991.

Location	Application			Residues		Reference
	Form.	No.	kg ai/ha	PHI, days	mg/kg	
US GAP	SC 375 g/l	--	0.14-1.0	28		
Caddo, OK	SC 375 g/l	6	1.0	28	<0.04 <0.04 <0.04	Lee, 1992b 91-006, Study No.USA91L82 File No.41198
Uvalde, TX	SC 375 g/l	6	1.0	28	<0.04 <0.04 <0.04	Lee, 1992b 91-008, Study No.USA91L82 File No.41198
Rapids, LA	SC 375 g/l	6	1.0	28	<0.04 <0.04 <0.04	Lee, 1992b 91-027, Study No.USA91L82 File No.41198
Lonoke, AR	SC 375 g/l	6	1.0	28	0.05 0.04 0.10	Lee, 1992b 91-040, Study No.USA91L82 File No.41198
Hidalgo, TX	SC 375 g/l	6	1.0	28	<0.04 <0.04 0.09	Lee, 1992b 91-072, Study No.USA91L82 File No.41198
Mitchell, GA	SC 375 g/l	6	1.0	28	<0.04 <0.04 <0.04	Lee, 1992b 91-103, Study No.USA91L82 File No.41198
Washington, MS	SC 375 g/l	6	1.0	28	<0.04 <0.04 <0.04	Lee, 1992b 91-251, Study No.USA91L82 File No.41198

Table 106. Residues in cotton seed (seed treatment), USA.

Location, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Manteca, CA 1984	WG 800g/kg	1	5	144	ND ND ND ND	Hunt, 1988h Project No.804R10 File No.40386
Wayside, MS 1984	WG 800g/kg	1	5	137	ND ND ND ND	Hunt, 1988h Project No.804R10 File No.40386
Clayton, NC 1984	WG 800g/kg	1	5	101	ND ND ND ND	Hunt, 1988h Project No.804R10 File No.40386
Manteca, CA 1985	900 g/kg	1	20	161	ND ND ND ND	Hunt, 1988h Project No.804R10 File No.40386
Wayside, MS 1985	900 g/kg	1	20	151	ND ND ND ND	Hunt, 1988h Project No.804R10 File No.40386
Clayton, NC 1985	900 g/kg	1	20	168	ND ND ND ND	Hunt, 1988h Project No.804R10 File No.40386
Clayton, NC 1985	900 g/kg	1	20	189	ND ND ND ND	Hunt, 1988h Project No.804R10 File No.40386

Table 107. Residues in cotton leaves (foliar application), Sudan, 1998-99.

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
Arc Wad Medani	WG 800 g/kg	3	0.571	61	42 61 81 0.26	Lusson and Muller, 1989b AG/CRLD/AN 8916381

Table 108. Residues in cotton forage (foliar application), USA, 1991.

Location	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.14-1.0	28		
Caddo, OK	SC 375 g/l	6	1.022	0	25.4 30.7 24.5	Lee, 1992b 91-005, Study No.USA91L82 File No.41198
Uvalde, TX	SC 375 g/l	6	1.026	0	47.9 53.9 97.5	Lee, 1992b 91-007, Study No.USA91L82 File No.41198
Rapids, LA	SC 375 g/l	6	0.994	0	47.1 32.5 46.6	Lee, 1992b 91-026, Study No.USA91L82 File No.41198
Lonoke, AR	SC 375 g/l	6	1.009	0	57.6 37.5 43.4	Lee, 1992b 91-039, Study No.USA91L82 File No.41198
Hidalgo, TX	SC 375 g/l	6	1.007	0	172.2 168.7 193.6	Lee, 1992b 91-071, Study No.USA91L82 File No.41198
Mitchell, GA	SC 375 g/l	6	1.035	0	98.2 27.1 65.1	Lee, 1992b 91-102, Study No.USA91L82 File No.41198
Washington, MS	SC 375 g/l	6	1.009	0	28.4 32.1 30.6	Lee, 1992b 91-250, Study No.USA91L82 File No.41198

Table 109. Residues in cotton forage (aerial application), USA, 1991.

Location	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, USA	SC 375 g/l	--	0.14-1.0	28		
Caddo, OK	SC 375 g/l		1.000	0	27.2 31.9 14.7	Lee, 1992b 91-006, Study No.USA91L82 File No.41198
Uvalde, TX	SC 375 g/l		1.009	0	26.2 22.3 21.7	Lee, 1992b 91-008, Study No.USA91L82 File No.41198
Rapids, LA	SC 375 g/l		1.043	0	126.5 86.2 73.1	Lee, 1992b 91-027, Study No.USA91L82 File No.41198
Lonoke, AR	SC 375 g/l		1.009	0	33.0 33.8 34.1	Lee, 1992b 91-040, Study No.USA91L82 File No.41198

Hidalgo, TX	SC 375 g/l		1.007	0	91.1 99.1 106.8	Lee, 1992b 91-072 Study No.USA91L82 File No.41198
Mitchell, GA	SC 375 g/l		1.009	0	23.2 28.8 23.8	Lee, 1992b 91-103, Study No.USA91L82 File No.41198
Washington, MS	SC 375 g/l		1.009	0	116.5 93.3 81.6	Lee, 1992b, 91-251, Study No.USA91L82 File No.41198

Table 110. Residues in cotton forage (seed treatment), USA.

Location, year	Application			Residues		Reference
	Formulation	No.	kg/ton, seeds	PHI, days	mg/kg	
Clayton, NC 1984	SC 375 g/l	1	5	17 24 38 70 101 	0.21 0.28 0.27 0.29 0.25 0.46 0.72 0.60 ND (<0.02) ND ND ND ND ND ND ND ND ND ND ND	Hunt, 1988h Project No.804R10 File No.40386
Wayside, MS 1985	900 g/kg	1	20	19 26 40 75 103 	3.2 3.1 3.4 6.4 1.0 0.98 0.5 0.93 ND ND ND ND ND ND ND ND <0.04 <0.04 <0.04 <0.04	Hunt, 1988h Project No.804R10 File No.40386

Oilseed rape. Supervised trials were conducted in the UK and Germany with granular bait application. GAP in the UK and Germany specifies a rate of 0.2 kg ai/ha, applied up to 3 times. Seed, forage and straw were analysed by GLC with sulfur FPD (LOQ 0.04 mg/kg seed and forage in the UK; 0.05 mg/kg forage, 0.2 mg/kg straw, 0.1 mg/kg seed in Germany).

Table 111. Residues in rape seed (granular bait application).

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, UK	RB 40 g/kg	1-3	0.2	--		
Melton Mowbray Leicestershire UK, 1987-88	GB 40 g/kg	4	0.3	154	<0.04 <0.04	Brockelsby *et al.*, 1990b Report No.D.Ag.1439
Ongar; Essex UK, 1987-88	GB 40 g/kg	4	0.3	98	<0.04 <0.04	Brockelsby *et al.*, 1990b Report No.D.Ag.1439
Chelmsford; Essex UK, 1987-88	GB 40 g/kg	4	0.3	105	0.04 <0.04	Brockelsby *et al.*, 1990b Report No.D.Ag.1439
Abberley Worcestershire UK, 1989-90	GB 40 g/kg	1	0.2	322	≤0.04 <0.04 <0.04	Brockelsby *et al.*, 1990c Report No.D.Ag.1562
Stoney Stanton Leicestershire UK, 1989-90	GB 40 g/kg	1	0.2	322	0.05 <0.04 <0.04	Brockelsby *et al.*, 1990c Report No.D.Ag.1562
Dry Doddington Lincolnshire UK, 1989-90	GB 40 g/kg	1	0.2	280	≤0.04 <0.04 <0.04	Brockelsby *et al.*, 1990c Report No.D.Ag.1562
GAP, Germany	RB 40 g/kg	1-3	0.2			
Winseldorf Schleswig-Holstein Germany, 1988-89	GB 40 g/kg	2	0.2	259	ND (<0.05)	Anon., 1992c 2214 ; BBA report No. 05128
Winseldorf Schleswig-Holstein Germany, 1988-89	GB 40 g/kg	2	0.2	125	ND	Anon., 1992c 2214 ; BBA report No. 05051
Mönchneversdorf Schleswig-Holstein Germany, 1987-88	GB 40 g/kg	2	0.2	117	ND	Anon., 1992c 2431; BBA report No. 05107
Mönchneversdorf Schleswig-Holstein Germany, 1987-88	GB 40 g/kg	2	0.2	276	ND	Anon., 1992c 2431; BBA report No. 05071
Mohnesee Theiningsen Germany, 1987-88	GB 40 g/kg	2	0.2	117	ND	Anon., 1992c 4773 ; BBA report No. 05103
Mohnesee Theiningsen Germany, 1987-88	GB 40 g/kg	2	0.2	262	ND	Anon., 1992c 4773 ; BBA report No. 05076
Lentersheim,Bayern Germany, 1987-88	GB 40 g/kg	2	0.2	239	ND	Anon., 1992c 8821; BBA report No. 03724
Lentersheim,Bayern Germany, 1987-88	GB 40 g/kg	2	0.2	93	ND	Anon., 1992c 8821; BBA report No. 05133

Table 112. Residues in oilseed rape forage (granular bait application).

Location	Application			Residues		Reference
	Form.	No.	kg ai/ha	PHI, days	mg/kg	
GAP, UK	RB 40 g/kg	1-3	0.2			
Melton Mowbray Leicestershire, UK, 1987-88	GB 40 g/kg	4	0.3	56	≤0.04	Brockelsby *et al.*, 1990b Report No.D.Ag.1439
Ongar; Essex, UK, 1987-88	GB 40 g/kg	4	0.3	0	≤0.04	Brockelsby *et al.*, 1990b Report No.D.Ag.1439
Chelmsford; Essex, UK, 1987-88	GB 40 g/kg	4	0.3	0	≤0.04	Brockelsby *et al.*, 1990b Report No.D.Ag.1439

Location	Application			Residues		Reference
	Form.	No.	kg ai/ha	PHI, days	mg/kg	
GAP, Germany	RB 40 g/kg	1-3	0.2			
Winseldorf Schleswig-Holstein Germany, 1988-89	GB 40 g/kg	2	0.2	07 42 142 168	ND (<0.02) ND ND ND	Anon., 1992c 2214 ; BBA report No. 05128
Winseldorf Schleswig-Holstein Germany, 1988-89	GB 40 g/kg	2	0.2	7 20 45	ND ND ND	Anon., 1992c 2214 ; BBA report No. 05051
Mönchneversdorf Schleswig-Holstein Germany, 1987-88	GB 40 g/kg	2	0.2	06 15 37	ND ND ND	Anon., 1992c 2431; BBA report No. 05107
Mönchneversdorf Schleswig-Holstein Germany, 1987-88	GB 40 g/kg	2	0.2	6 26 156 176	ND ND ND ND	Anon., 1992c 2431; BBA report No. 05071
Mohnesee Theiningsen Germany, 1987-88	GB 40 g/kg	2	0.2	07 27	ND ND	Anon., 1992c 4773 ; BBA report No. 05103
Mohnesee Theiningsen Germany, 1987-88	GB 40 g/kg	2	0.2	7 136 157	ND ND ND	Anon., 1992c 4773 BBA report No. 05076
Lentersheim, Bayern Germany, 1987-88	GB 40 g/kg	2	0.2	14 151 161	ND ND ND	Anon., 1992c 8821; BBA report No. 03724
Lentersheim, Bayern Germany, 1987-88	GB 40 g/kg	2	0.2	7 36	ND ND	Anon., 1992c 8821; BBA report No. 05133

Table 113. Residues in oilseed rape straw (granular bait application), Germany.

Location, year	Application			Residues		Reference
	Formulation	No.	kg ai/ha	PHI, days	mg/kg	
GAP, Germany	RB 40 g/kg	1-3	0.2			
Winseldorf Schleswig-Holstein, 1988-89	GB 40 g/kg	2	0.2	259	ND (<0.1)	Anon., 1992c 2214 ; BBA report No. 05128
Winseldorf Schleswig-Holstein, 1988-89	GB 40 g/kg	2	0.2	125	ND	Anon., 1992c 2214 ; BBA report No. 05051
Mönchneversdorf Schleswig-Holstein, 1987-88	GB 40 g/kg	2	0.2	117	ND	Anon., 1992c 2431; BBA report No. 05107
Mönchneversdorf Schleswig-Holstein, 1987-88	GB 40 g/kg	2	0.2	276	ND	Anon., 1992c 2431; BBA report No. 05071
Mohnesee Theiningsen, 1987-88	GB 40 g/kg	2	0.2	117	ND	Anon., 1992c 4773 ; BBA report No. 05103
Mohnesee Theiningsen, 1987-88	GB 40 g/kg	2	0.2	262	ND	Anon., 1992c 4773 ; BBA report No. 05076
Lentersheim, Bayern, 1987-88	GB 40 g/kg	2	0.2	239	ND	Anon., 1992c 8821; BBA report No. 03724
Lentersheim, Bayern, 1987-88	GB 40 g/kg	2	0.2	93	ND	Anon., 1992c 8821; BBA report No. 05133

Animal feeding studies

In a cattle feeding study (Davis and Wilkes, 1994) eight lactating dairy cattle were randomly assigned to one of three groups. Group I was the control group and contained 2 animals. Groups II and III each contained 3 cows and were administered thiodicarb at dose levels equivalent to 350 ppm and 1050 ppm in the diet respectively by bolus for a period of 28 days. The 1050 ppm cows were dosed once a day for 12 days, and twice a day at half the quantity per dose for the remainder of the study. The dosing was changed to twice daily when cholinesterase inhibition was observed.

The cows were milked twice daily throughout the study except on day 28 when the high-dose cows were only milked once. Samples were immediately frozen. Milk samples for a given study day were defined as those taken at the evening milking of that day and next morning's milking. They were composited by mixing equal volumes from the two milkings. All samples were stored at a nominal temperature of -20°C.

Animals were slaughtered within 3 hours after the final dose and samples of muscle, fat, liver and kidney were collected and stored frozen for 0.5-2 months before extraction and clean-up. The storage stability study reported above indicates adequate stability over this period in all substrates except liver.

Samples were analysed by the HPLC method, with coagulation as a clean-up step. No quantifiable thiodicarb (<0.1 mg/kg) and no methomyl (<0.1 mg/kg) were found in any milk or tissue sample from the 1050 mg/kg dosing, nor in several milk samples analysed from the 350 ppm feeding level. At least one milk sample from the 1050 ppm feeding level on days 1, 7 and 25 showed unquantifiable residues, estimated at 0.02-0.03 mg/kg. A fat sample showed a residue estimated at 0.04 mg/kg, muscle 0.03 mg/kg, kidney 0.01 mg/kg, and liver 0.06 mg/kg, all from the 1050 ppm feeding level. The corresponding liver control sample yielded 0.09 mg/kg.

A poultry feeding study was not reported.

FATE OF RESIDUES IN STORAGE AND PROCESSING

In processing

Processing studies were conducted on soya beans, tomatoes, apples, sweet corn and cotton in the USA and grapes in France and Spain, as shown in the following Tables. The studies were conducted according to standard commercial practices in the respective countries and in all cases except one tomato study the residues were field-incurred. Processing factors are shown in Tables 114-119.

Table 114. Processing factors – soya beans.

Sample	Average residues (mg/kg)	Processing factors	Reference/Comments
Whole seed	0.04	1	Lee, 1991c Project USA90L01 File 41003 Illinois, USA Processing and analyses within 6 months of harvest. GLC method validated at 0.04 mg/kg for methomyl and thiodicarb in each sample.
Hulls	0.16	3.6	
Meal	<0.04	<1	
Refined oil	<0.04	<1	
Crude oil	<0.04	<1	
Soapstock	<0.04	<1	
Grain dust	1.24	29	

Table 115. Processing factors – tomatoes.

Processed fraction	Average residues (mg/kg)	Processing factors	Reference/Comment
Whole tomato	5.6	1	Hunt, 1986b Study No.804R11. File No.35032 Tomatoes sprayed in the laboratory with thiodicarb solution, allowed to dry for 24 h, then washed.
Whole tomato washed	0.62	0.11	
Wet pomace	1.5	0.27	

Dry pomace	3.9	0.70	
Juice	0.26	0.05	
Puree	<0.04	<0.01	
Paste	0.07	0.01	
Whole tomato	1.4		Kowite, 1998b Study No.96L10370. File No.45563 California, USA Tomatoes stored for 2 days at room temperature before processing. Maximum frozen storage 263 days.
Purée	<0.04	<0.03	
Paste	<0.04	<0.03	

Table 116. Processing factors – apples.

Sample	Residues (mg/kg) [1]	Processing factors	Reference/location
Apple	5.02, 7.2, 4.02 (5.4)	1	Avakian, 1991 Project No.S78AP01[2] North Carolina, USA
Fresh juice	0.67, 0.49, 0.59 (0.58)	0.11	
Canned juice	0.096, 0.055, 0.072 (0.074)	0.014	
Wet pomace	2.4, 1.4, 2.2 (2.0)	0.37	
Unwashed fruit	4.6	1	Hunt, 1989e Project No.804R10 File No.40657 Pennsylvania, USA
Washed fruit	1.4	0.30	
Juice	<0.02	<0.01	
Wet pomace	1.1	0.24	

[1] Replicate experiments. Average in parenthesis.
[2] Concurrent method recovery data (GLC) from 0.5 to 15 mg/kg fortifications. Apples stored for one week at 8-13°C before processing and one month at -20°C before analysis.

Table 117. Residues in grapes and wine.

Country, year	Application			Residues (mg/kg)				Reference
	Form.	No	kg ai/ha	PHI, days	Grapes	Wine	Processing factor	
France, 1988	SC 375 g/l	2	0.375	5	0.66	<0.08	0.12	Lusson and Muller, 1989a Study 8916392
France, 1988	SC 375 g/l	2	0.375	35	0.22	<0.08	0.36	
France, 1988	SC 375 g/l	2	0.375	54	<0.08	<0.08	-	
France, 1992	SC 375 g/l	3	0.563	49	0.16	<0.05	0.31	Richard and Muller, 1994a, Study 92-147
France, 1992	SC 375 g/l	3	0.563	45	0.11	<0.05	0.45	
France, 1995	SC 300 g/l	2	0.306	49	0.096	< 0.025 mg/l	0.31	Maestracci, 1997b Study 95-540
France, 1995	SC 300 g/l	2	0.30	31	0.32	0.15 mg/l	0.47	Maestracci, 1997c Study 95-539
Spain, 1993	SC 375 g/l	2	0.563	90	1.4	<0.05	0.036	Richard and Muller 1994b, Study 93-624; 9415899
Average							0.3	-

Table 118. Processing factors – sweet corn. [1]

Location	Kernels + Cob	Kernels	Cannery waste	Processing factor for waste	Reference
Minnesota, USA, 1992	<0.02	0.03	1.28	>64	Lee, 1993 Project No.USA92L01 File No.44128
Wisconsin, USA, 1992	0.07	0.06	5.46	78	

[1] Deviations from commercial practice: corn husked by hand; husked corn not washed.

Table 119. Processing factors – cotton.

Processed fraction	PHI (days)	Average residues (mg/kg)	Processing factors	Reference/Comment
Cotton seed (delinted)	28	0.184, 0.215 (0.20)	1	Lee, 1991d Study USA90L82. Texas. Seed held frozen for 3 months before processing. GLC method 90321 validated at 0.04 mg/kg. Processed fractions held frozen for up to 60 days before analysis.
Hulls	28	0.22	1.1	
Crude oil	28	<0.04	<0.2	
Refined oil	28	<0.04	<0.2	
Soapstock	28	0.06	0.31	
Meal	28	0.05	0.26	

Residues in the edible portion of food commodities

Table 120 Residues in lettuce (foliar application), USA.

Location. year	Application			Residues (mg/kg)			Reference
	Formulation	No	kg ai/ha	PHI, days	with wrapper leaves	without wrapper leaves	
GAP, USA	SC 375 g/l	--	0.45-0.84	14	Do not exceed 1.7 kg ai/ha per season		
Arizona, 1983-84	SC 375 g/l	4	0.841	14	0.17 0.10 0.25 mean 0.17	0.05 ND (<0.02) 0.04 mean 0.036 factor 0.21	Hunt, 1986a Project 804R10 File 34501
	WG 800 g/kg	4	0.841	14	0.11 0.21 0.20 mean 0.17	0.06 ND 0.06 0.047 factor 0.3	
California, 1983	SC 375 g/l	4	0.841	7 14	2.7 2.4 mean 2.6 3.0 1.7 1.7 mean 2.1	<0.05 0.07 0.06 mean 0.06 factor 0.02 <0.05 0.13 <0.05 0.077 factor 0.04	
	WG 800 g/kg	4	0.841	7 14	1.7 0.37 mean 1.0 0.06 0.06 1.8 mean 0.64	0.09 <0.05 <0.05 mean 0.063 factor 0.06 0.05 <0.05 <0.05 mean 0.05 factor 0.08	
California, 1983-84	SC 375 g/l	4	0.841	14	1.8 3.2 2.4 mean 2.5	0.04 0.04 0.04 mean 0.04 factor 0.02	
	WG 800 g/kg	4	0.841	14	1.5 1.4 1.7 mean 1.5	0.03 0.02 0.03 mean 0.027 factor 0.02	

Location. year	Application			Residues (mg/kg)			Reference
	Formulation	No	kg ai/ha	PHI, days	with wrapper leaves	without wrapper leaves	
Wisconsin, 1983	SC 375 g/l	4	0.841	14	0.24 0.34 0.24 0.20 mean 0.26	0.05 0.05 <0.05 mean 0.05 factor 0.2	
	WG 800 g/kg	4	0.841	14	0.24 0.34 0.14 0.48 mean 0.30	0.20 0.08 0.08 mean 0.12 factor 0.4	
California, 1984	SC 375 g/l	4	0.841	14	0.07 0.06 0.06 mean 0.063	<0.04 0.05 <0.04 mean 0.043 factor 0.7	
	WG 800 g/kg	4	0.841	14	0.14 0.14 0.13 mean 0.14	0.12 0.14 0.08 mean 0.11 factor 0.8	
Florida, 1984	SC 375 g/l	4	0.841	14	1.2 1.1 1.3 mean 1.2	0.08 0.06 0.11 mean 0.083 factor 0.07	
	WG 800 g/kg	4	0.841	14	1.7 1.5 mean 1.6	0.06 0.04 0.05 mean 0.05 factor 0.03	
New York, 1984-85	SC 375 g/l	4	0.841	14	0.08 0.06 0.09 mean 0.077	0.08 0.08 0.07 mean 0.077 factor 1	
	WG 800 g/kg	4	0.841	14	0.19 0.17 0.11 mean 0.16	0.06 0.08 0.08 mean 0.073 factor 0.4	
Texas, 1984-85	SC 375 g/l	4	0.841	14	0.29 0.42 0.28 mean 0.33	ND 0.04 0.04 mean 0.033 factor 0.1	
	WG 800 g/kg	4	0.841	14	0.26 0.34 0.44 mean 0.35	0.06 0.03 <0.04 mean 0.043 factor 0.1	
Arizona, 1984-85	SC 375 g/l	4	0.841	15	0.63 0.96 0.96 mean 0.85	ND ND ND mean <0.02 factor 0.02	
	WG 800 g/kg	4	0.841	15	0.96 1.4 1.9 mean 1.4	ND ND ND mean <0.02 factor 0.01	

The average factor for reduction of residue when removing the wrapper leaves from head lettuce is 0.2 (n = 20).

Table 121. Residues in lettuce (aerial application), USA.

Location , year	Application			Residues (mg/kg)			Reference
	Formulation	No	kg ai/ha	PHI, days	with wrapper leaves	without wrapper leaves	
GAP, USA	SC 375 g/l	--	0.45-0.84	14	Do not exceed 1.7 kg ai/ha per season		
California, 1983	SC 375 g/l	4	0.841	7 14	2.8 2.2 2.0	<0.05 0.05 0.06 0.15	Hunt, 1986a Project 804R10 File 34501
	WG 800 g/kg	4	0.841	7 14	1.3 0.27 <0.05 <0.05 <0.05	<0.05 <0.05 <0.05 <0.05	
Arizona, 1984-85	SC 375 g/l	4	0.841	15	1.1 1.0 1.5	ND ND ND	
	WG 800 g/kg	4	0.841	15	0.65 1.1 0.78	0.08 ND ND	

Table 122. Residues in cabbage (foliar application), USA.

Location, year	Application			Residues (mg/kg)			Reference
	Formulation	No	kg ai/ha	PHI, days	with wrapper leaves	without wrapper leaves	
GAP, USA	SC 375 g/l	--	0.45-1.15	7	Do not exceed 6.7 kg ai/ha per season		
Florida, 1982	SC 375 g/l	6	1.121	3	1.4 2.4 1.8 mean 1.9	0.34 0.22 0.50 mean 0.35 factor 0.2	Hunt, 1986a Project 804R10 File 34501
				5	2.1 2.7 3.0 mean 2.6	0.50 0.67 0.60 mean 0.59 factor 0.2	
				7	2.0 1.8 3.1 mean 2.3	0.37 0.23 0.54 mean 0.38 factor 0.2	
	WG 800 g/kg	6	1.121	3	4.7 3.5 3.2 mean 3.8	0.47 0.29 0.21 mean 0.32 factor 0.08	
				5	3.5 3.6 4.1 mean 3.7	0.93 1.0 1.2 mean 1.0	
				7	2.1 0.37 1.7 mean 1.4	0.54 2.9[1] 0.82 mean 0.68 factor 0.49	

Location, year	Application			Residues (mg/kg)			Reference
	Formulation	No	kg ai/ha	PHI, days	with wrapper leaves	without wrapper leaves	
New York, 1982	SC 375 g/l	6	1.121	3	1.2 0.77 1.6 mean 1.2	0.18 0.11 <0.03 mean 0.11 factor 0.1	
				5	0.70 0.88 1.1 mean 0.89	<0.03 0.04 <0.03 mean 0.033 factor 0.04	
				7	1.2 0.44 1.3 mean 0.98	<0.03 <0.03 <0.03 mean <0.03 factor 0.03	
	WG 800 g/kg	6	1.121	3	1.2 1.7 2.1 mean 2.0	0.05 <0.03 0.04 mean 0.04 factor 0.02	
				5	2.0 2.0 1.8 mean 1.9	<0.03 0.05 <0.03 mean 0.036 factor 0.02	
				7	3.5 1.0 1.2 mean 1.9	<0.03 ND <0.03 mean 0.04 factor 0.02	
Ohio, 1982	SC 375 g/l	5	1.121	3	1.1 2.8 3.0 mean 2.3	0.18 0.34 0.37 mean 0.30 factor 0.1	
				5	0.56 1.7 5.0 mean 2.4	0.25 0.21 0.22 mean 0.23 factor 0.09	
Pennsylvania, 1982	SC 375 g/l	6	1.121	3	4.0 1.5 1.4 mean 2.3	0.05 <0.03 ND mean 0.033 factor 0.01	
				5	0.45 0.15 0.09 mean 0.23	0.04 <0.03 ND mean 0.045 factor 0.2	
				7	0.08 0.04 mean 0.06	<0.03 <0.03 <0.03 mean <0.03 factor 0.5	

Location, year	Application			Residues (mg/kg)			Reference
	Formulation	No	kg ai/ha	PHI, days	with wrapper leaves	without wrapper leaves	
Wisconsin, 1982	SC 375 g/l	6	1.121	3	0.74 0.51 mean 0.62	0.19 0.05 mean 0.12 factor 0.2	
				5	0.23 1.1 0.61 mean 0.65	0.07 0.27 0.13 mean 0.16 factor 0.2	
				7	0.18 0.20 0.53 mean 0.30	0.17 0.07 0.09 0.07 mean 0.1 factor 0.3	
	WG 800 g/kg	6	1.121	3	0.55 0.79 0.91 mean 0.75	0.11 0.13 0.09 mean 0.11 factor 0.1	
				5	0.16 0.75 0.58 mean 0.50	0.11 0.25 0.18 mean 0.18 factor 0.4	
				7	0.14 0.51 1.2 mean 0.62	<0.05 0.18 mean 0.12 factor 0.2	
California, 1983	SC 375 g/l	6	1.121	7	3.4 2.4 3.8 mean 3.2	0.06 0.07 0.07 mean 0.67 factor 0.02	
				14	0.41 0.21 0.24 mean 0.29	0.06 0.12 0.07 mean 0.08 factor 0.3	
	WG 800 g/kg	6	1.121	7	1.9 1.7 2.7 mean 2.1	0.05 0.03 0.03 mean 0.037 factor 0.02	
				14	0.13 0.07 0.10 mean 0.10	0.11 0.08 ND mean 0.07 factor 0.7	

[1]Aberrant result. Excluded from calculation of mean.

Table 123. Residues in cabbage (aerial application), USA, 1982.

Location	Application			Residues (mg/kg)			Reference
	Formulation	No.	kg ai/ha	PHI, days	with wrapper leaves	without wrapper leaves	
California	SC 375 g/l	6	1.121	7	4.9	0.06	Hunt, 1986a Project 804R10 File 34501
					2.3	0.07	
					5.0	0.07	
				14	0.37	0.08	
					0.28	0.07	
					0.47	0.04	
	WG 800 g/kg	6	1.121	7	4.8	0.03	
					1.9	0.09	
					1.1	0.07	
				14	0.10	0.05	
					0.22	0.03	
					0.13	ND	

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The Carbamate Market Basket Survey Task Force (CMBSTF) sponsored a study to determine the level of certain *N*-methylcarbamate insecticide residues in single-serving samples of fresh fruits and vegetables available for consumption by the US population (Carringer, 2000). Thiodicarb was one of the insecticides of interest in this study. The commodities selected were head lettuce and broccoli.

The analytical method used to quantify residues was "Determination of Selected N-Methyl Carbamate Pesticides in Fruits and Vegetables", Morse Laboratories, Inc., SOP No. Meth-118 (Revision 2), December 22, 1998. The residues are extracted with a mixture of acetonitrile and water. The water and acetonitrile are partitioned by the addition of NaCl and an aliquot of the resulting acetonitrile phase is partitioned with hexane, followed by dichloromethane/salt water, then cleaned up on a Florisil solid phase extraction (SPE) cartridge. Broccoli samples require an additional carbon black SPE clean-up. The purified extract is concentrated and analysed by high-performance liquid chromatography with post-column derivatisation and fluorescence detection. The LOQ is 0.001 mg/kg. The method detection limit (MDL), confirmed by analysing samples fortified at one-third or one-half the LOQ, was 0.00033 mg/kg for thiodicarb in all commodities.

Sampling took place during 1999 and 2000, although most of the samples were gathered in 1999.

Table 124 summarizes the results. Of the approximately 400 samples analysed, 98%-100% have no residues above the LOQ of 0.001 mg/kg. The highest residue was 0.0022 mg/kg, in broccoli. Six samples were at or above the LOQ at the following levels: 0.0010 (1 sample), 0.0011 (2), 0.0012 (2), 0.0022 (1).

Table 124. Carbamate Market Basket Survey in the USA (1999-2000).

Analyte	Sample	No. of analyses	Residues <0.001 mg/kg		Residues ≥0.001 mg/kg		Range of residues (mg/kg)
			No.	%	No.	%	
Thiodicarb	Head Lettuce	399	399	100	0	0	ND - <0.001
	Broccoli	395	389	98	6	2	ND - 0.0022

NATIONAL MAXIMUM RESIDUE LIMITS

The following existing national MRLs were provided by the manufacturer.

Commodity	Country	MRL (mg/kg)
Beans	Brazil	0.1
Beans	Peru	0.1
Beans and peas	Taiwan	1
Beet leaves (Chard)	Europe	2
Beetroot	Europe	0.05
Brassica crops	Australia	1
Broccoli	USA	7
Brussels sprouts	Austria	0.2
Brussels sprouts	Belgium	1
Brussels sprouts	Europe	0.05*
Brussels sprouts	Germany	0.5
Cabbage	USA	7
Cauliflower	USA	7
Cereals	Europe	0.05
Corn	Argentina	-
Cotton	Argentina	0.4
Cotton	Brazil	0.1
Cotton	Europe	0.1
Cotton	Peru	0.4
Cotton seed	USA	0.4
Cotton seed oil	Australia	0.1
Cotton seed, hulls	USA	0.8
Grapes (table)	Europe	0.05*
Grapes (wine)	Europe	1
Leaf vegetables with small leaves	Taiwan	1
Leaf vegetables with wrapped leaves	Taiwan	1
Leafy vegetables	USA	35
Lettuce	Europe	2
Maize	Australia	0.1
Maize	Brazil	0.1
Maize	Europe	0.05
Maize	Peru	2
Melon vegetables	Taiwan	1
Oilseed rape	Europe	0.05*
Peach	Europe	0.2
Pome fruits	Europe	0.2
Potato	Europe	0.05*
Pulses	Europe	0.05*
Pulses (soya beans, Mung beans, chick-peas, pigeon peas, navy beans)	Australia	0.1
Radish	Europe	0.5
Rice	Brazil	0.1
Root vegetables	Taiwan	0.5
Small berries	Taiwan	0.5
Sorghum	Australia	0.5 (temporary)
Soya bean	Argentina	0.2
Soya bean	Brazil	0.1
Soya bean	Europe	0.1
Soya bean	USA	0.2
Soya bean hulls	USA	0.8
Spinach	Europe	2
Sunflower	Argentina	0.2
Sunflower	Australia	0.05
Sunflower	Europe	0.05

Sweet corn	USA	2
Sweet corn (corn-on-the-cob)	Australia	0.1
Tomato	Australia	2
Tomato	Europe	0.5
Tomato	Peru	0.2
Wheat	Argentina	-
Wheat	Brazil	0.2

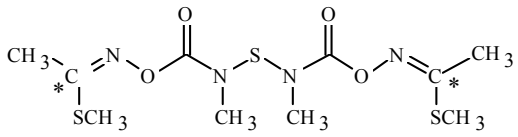
APPRAISAL

Thiodicarb is a carbamate insecticide and molluscicide. It is registered and used in agriculture and horticulture, against lepidoterous insects as a foliar treatment, as a molluscicide in the form of granular bait in various crops, and as a seed treatment.

Thiodicarb decomposes in plants and animals and in the environment to the insecticide methomyl. Currently, the MRLs for thiodicarb and methomyl are combined under methomyl.

Thiodicarb was evaluated by the FAO Panel of the JMPR in 1985, 1987 and 1988. The WHO Panel of the JMPR re-evaluated its toxicology in 2000.

Metabolism



[acetyl-1-^{14}C]Thiodicarb (designated [acetimide-^{14}C] thiodicarb in 2000 JMPR)
* Denotes ^{14}C carbons

The metabolism of thiodicarb in rats, monkeys, goats and chickens has been reported. Rats were given [acetyl-1-^{14}C]thiodicarb orally at 2 or 16 mg/kg bw in a single dose. More than 70% of the radiolabel was found in urine and respired gases over 7 days. The respired gases contained radiolabelled CO_2 and acetonitrile, and urine contained acetonitrile as a major metabolite and methomyl, methomyl oxime, methomyl sulfoxide and methomyl oxime sulfoxide as minor metabolites.

Cynomolgus monkeys were given a single oral dose of [acetyl-1-^{14}C]thiodicarb at 5 mg/kg bw. About 37% of the administered dose was eliminated in respired air over the first 48 h, consisting of 9% acetonitrile and 28% CO_2. Urine excreted over 168 h contained a combined total of 29% of the administered dose. Thiodicarb, methomyl and methomyl oxime were not detected in urine, blood or liver, while acetic acid was identified in liver.

The metabolic fate of [acetyl-1-^{14}C]thiodicarb in laying hens was studied after administration of a diet containing 15, 29 or 102 ppm for 21 days. The concentrations of residues achieved plateaux in egg white after 2 days and in egg yolk after 10 days. Thiodicarb and the potential metabolites methomyl, methomyl oxime, methomyl oxime sulfoxide and methomyl methylol were not detected in eggs, although low concentrations of acetonitrile (volatile) and acetamide (water-soluble) were found. In addition, considerable radiolabel was present as lipids (70% of the TRR) and other natural products due to incorporation of $^{14}CO_2$.

The concentrations of radiolabel in tissues of hens given 102 ppm were 4.2 mg/kg in muscle, 6.6 mg/kg in fat, 8.5 mg/kg in kidney and 10 mg/kg in liver. Acetonitrile and acetamide were identified in liver and muscle but not in abdominal fat. About 25% of the TRR in liver, 60% in muscle and 85% in fat was characterized as lipids. Methomyl, methomyl oxime, methomyl oxime sulfoxide and methomyl methylol were not found in any tissue.

The metabolic fate of [acetyl-1-^{14}C]thiodicarb was studied in two lactating goats after administration of 200 and 290 ppm per day for 7 days. The concentration of radiolabel reached maxima of 15 mg/kg and 20 mg/kg in the milk of the two goats on day 3. The goat at the higher dose became ill, and most of the results reported were for tissues from the goat at the lower dose.

After alkaline hydrolysis of water-soluble polar extracts, 32% of the TRR in liver was identified as acetonitrile, 6% as acetamide and 57% as acetic acid; 23% of the TRR in kidney was identified as acetonitrile, 10% as acetamide and 43% as acetic acid; and 72% of the TRR in muscle was acetonitrile, 14% was acetamide and 11% was acetic acid. No thiodicarb, methomyl or methomyl oxime was detected in any tissue.

The metabolites identified in fat were acetonitrile (10% of the TRR), acetamide (6% of the TRR) and saponifiable fatty acids and lipids (73% of the TRR). In milk, acetonitrile (29% of the TRR), lactose (11% of the TRR), saponifiable fatty acids and other lipids (32% of the TRR), palmitic and myristic acids and glycerol were identified. Some of the radiolabel in liver and kidney was also associated with amino acids and proteins.

The metabolism of [acetyl-1-^{14}C]thiodicarb in plants was studied in root crops (potato and carrot), in a fruiting vegetable (tomato), in cereal grain (wheat, maize and sweet corn) and in oilseed crops (cotton, soya bean and peanuts). When the radiolabelled compound was applied to the upper surface of the leaves of potato plants, < 0.2% of the administered dose migrated to the tubers, and 59% was found on the foliage. The constituents were thiodicarb (main), methomyl and methomyl oxime (trace).

[acetyl-1-^{14}C]Thiodicarb was applied to the upper surface of the leaves of 6-week-old carrot plants, and the carrots were harvested 28 days later. The aerial portions of the plants contained 90% of the applied radiolabel, and 0.06% was found in the roots. The following radiolabelled components were identified tentatively on the foliage: thiodicarb (79% of applied radiolabel), methomyl (8%), *N*-hydroxymethyl methomyl (0.18%) and methomyl oxime (0.09%).

[acetyl-1-^{14}C]Thiodicarb was also applied to the tops of tomato leaves at the time of flowering. The plants were maintained in a glasshouse, and the fruits were harvested at maturity. About 50% of the radiolabel was lost, perhaps as volatile compounds. About 49% was found on the foliage and about 0.45% on the tomatoes. The constituents on the foliage were identified as thiodicarb (78% of the TRR), methomyl (6%) and methomyl oxime (0.3%).

[acetyl-1-^{14}C]Thiodicarb was injected into the stems of 3-week-old sweet corn and wheat plants. The plants were maintained for 7 days and then harvested. The following metabolites were identified in both crops: thiodicarb (major), methomyl (major), methomyl oxime (very minor) and methomyl sulfoxide (very minor). About 30–50% of the radiolabel was unaccounted for.

A second study was performed with sweet corn, in which [acetyl-1-^{14}C]thiodicarb was painted onto leaves, ears and silk in one experiment and the leaves only in another. The plants were maintained for 7 days in a glasshouse and then harvested. Almost 70% of the applied radiolabel was unaccounted for. Only minute quantities were found in kernels plus cob. The concentrations on cobs and kernels were similar in the two experiments and were low (0.1–0.15% of the applied dose) in both cases. Over 70% of the radiolabel in the kernels could not be extracted. The metabolites identified on foliage (34% of the applied dose) were thiodicarb (20%), methomyl (6%) and methomyl oxime (trace to 0.3%).

In a study conducted with cotton plants, [^{14}C]thiodicarb was injected into the stem of 4–5-week-old cotton plants, which were maintained in a greenhouse and harvested 7, 14, 21 or 28 days after treatment. In a separate experiment, [^{14}C]thiodicarb was applied by stem injection and topical application to the tops of the leaves of 4-week-old cotton plants maintained in enclosed glass containers. Volatile compounds were collected in a series of traps at intervals of 1, 4 and 7 days after application and were identified as CO_2 and acetonitrile. In the injected plants (with no collection of volatile compounds), the percentage of the total applied radiolabel attributable to compounds soluble in organic solvents decreased from 53% on day 7 to 7% on day 28, whereas the percentage of water-soluble compounds increased from 12% to 21%. Throughout the experiment, 1–2% of the compounds could not be extracted. The compounds soluble in organic solvents were identified as thiodicarb, methomyl and methomyl oxime (trace). Methomyl was the major component on day 28.

The absorption, translocation and metabolism of thiodicarb in and on cotton after application to the leaf surface were investigated in a study in which a solution of [^{14}C]thiodicarb was spread onto the tops of the leaves of cotton plants at the flower bud stage at a rate equivalent to 1.1 kg ai/ha. The plants were maintained in a greenhouse until the bolls were mature, at which time they were harvested and the seeds de-linted. Senescent leaves were also collected for analysis. Lint and seed each contained < 0.1% of the applied dose, which was too little to allow adequate characterization. The senescent leaves were found to contain thiodicarb (22% of the TRR), methomyl (12% of the TRR), methomyl oxime (0.14% of the TRR) and methomyl methylol (0.5% of the TRR).

A similar experiment was performed with soya bean plants. A solution of [^{14}C]thiodicarb was spread onto the tops of the leaves of soya bean plants at the flower bud stage at a rate approximating 1.12 kg ai/ha. The plants were maintained in a greenhouse until the pods were mature, at which time they were harvested and the seeds separated from the hulls. Senescent leaves were also collected for analysis. Measurements of radiolabel in harvested seed and hull samples indicated activity representing 0.18–0.19% of the applied dose. The organic extract of soya bean leaves contained thiodicarb (85% of the applied radiolabel), methomyl (6%) and methomyl oxime (trace).

In a final study, [acetyl-1-^{14}C]thiodicarb was applied topically to peanut foliage four times at 7-day intervals at a rate of 1.1 kg ai/ha. The plants were harvested 21 days after the last treatment, and foliage, root, nut and shell were analysed separately. Of the applied radiolabel, 22% was in foliage, 0.2% in root, 0.5% in nut and 0.2% in nut shell. Almost 77% was unaccounted for and was presumably volatilized. The foliage contained thiodicarb and methomyl but no methomyl oxime or acetamide. Most (50–70%) of the radiolabelled residues in nut, shell and root could not be extracted. None of the components soluble in organic solvents could be identified.

The Meeting concluded that the metabolism of thiodicarb is adequately understood in both animals and plants. In animals, thiodicarb is converted to methomyl and, presumably via methomyl oxime, to CO_2, acetonitrile and acetamide. These may then be incorporated into natural products. Significantly, thiodicarb, methomyl and methomyl oxime are not found in tissues, eggs or milk. An analogous pathway exists in plants. Thiodicarb and its metabolites showed little tendency to translocate from the point of application. Thiodicarb is converted to methomyl and methomyl is metabolized to CO_2 and acetonitrile. At the point of application, e.g., foliage, the main soluble residue components are thiodicarb and methomyl. Volatile compounds often accounted for 50% or more of the residue. Traces of methomyl oxime, a potential intermediate to CO_2 and acetonitrile, were often found. The volatile compounds may be incorporated into natural products.

Environmental fate

Soil

A study of rotational crops was performed in sandy loam soil under confined conditions after application of [acetyl-1-^{14}C]thiodicarb at a rate of 6.7 kg ai/ha. After the soil had been tilled to a maximum depth of 10 cm, crops of mustard greens, radish and wheat were planted at intervals of 31,

125 and 364 days after treatment. Soil was analysed at the time of treatment and at the first plant-back interval (31 days). At day 0, 82% of the radiolabel was on thiodicarb; by day 31, thiodicarb accounted for 5% and methomyl for 47% of the radiolabel. Residues were found in crops planted 31 and 125 days after treatment but not in those with a 364-day plant-back (< 0.01 mg/kg). The concentrations of residues ranged from 0.11 mg/kg in radish root to 0.81 mg/kg in wheat straw at the 125-day plantback. At the 31-day plantback, the concentrations ranged from 0.48 mg/kg in wheat grain to 2.4 mg/kg in wheat straw.

When the crop matrices were extracted and analysed, the compounds identified included acetic acid released by acid hydrolysis, methomyl (maximum, 15% of the TRR), and methomyl oxime released by base hydrolysis (2–10% of the TRR). Most of the radiolabelled residues was not soluble in water or organic solvents and were found to be associated with natural products such as starch, proteins, pectins and lignin. At the 31-day plant-back, methomyl (from thiodicarb plus methomyl) was found at the following concentrations: wheat forage, 0.07 mg/kg; mustard greens, 0.12 mg/kg; radish tops, 0.14 mg/kg; and radish roots, 0.09 mg/kg. At the 125-day plant-back, the concentrations of methomyl were 0.02 mg/k in wheat forage, 0.04 mg/kg in mustard greens and 0.02 mg/kg in radish tops These values represented the LOQ of the analytical methods.

The Meeting concluded that thiodicarb and methomyl degradates persist in soil for at least 4 months and are taken up by plants and ultimately incorporated into natural products. The Meeting further concluded that, under typical GAP, ≥ 15% of the rate used in this study and field conditions, residues of methomyl and thiodicarb would not be quantifiable in rotational crops at intervals > 30 days.

Photolytic degradation of thiodicarb incubated under aerobic conditions at a temperature of 20 °C in a clay loam soil for 21 days was reported. In both irradiated and control soil, 50% of the radiolabelled material was lost as CO_2. Thiodicarb rapidly degraded to methomyl in both soils, the concentration of methomyl reaching a maximum of 80–90% of the applied dose on day 2. The concentration of thiodicarb declined slightly faster in the control than in the irradiated soil.

The Meeting concluded that sunlight has no net effect on the degradation of thiodicarb in soil.

In soil under aerobic conditions, thiodicarb degraded rapidly to methomyl, with a half-time based on first-order kinetics of 0.01–2.0 days, depending of soil type. The half-time of methomyl in sandy loam soil was 27 days. Methomyl oxime was found in the soil, at no more than about 3% of the applied dose. Over 60 days, the amount of radiolabel in sandy loam soil decreased to 42% of the applied dose, and the proportion of volatile compounds, identified as CO_2 and acetonitrile, increased to 53% of the applied dose. After 56 days, the radiolabel associated with volatile compounds represented 61% of the applied dose in clay loam with a high pH, 59% in clay loam at 20 °C and 40% in clay loam incubated at 10 °C. At the same time, the amount of unextractable radiolabel in the soils increased to a maximum of 35% of the applied dose.

The route and rate of degradation of [acetyl-1-^{14}C]thiodicarb under anaerobic conditions was studied in a soil–water mixture. The soil was flooded with deionized water and purged with nitrogen for 42–43 days before treatment to establish anaerobic conditions. A solution of [^{14}C]thiodicarb was applied to the water surface at a nominal application rate equivalent to 1 kg ai/ha. During incubation, the system was purged continuously with nitrogen to maintain anaerobic conditions. Within 16 min, the concentration of thiodicarb was < 1% of the applied dose. The concentration of methomyl was constant, at about 2% of the applied dose. An intermediate compound, *S*-methyl *N*-[*N*-methyl-*N*-(methylaminothio)-carbamoyloxy] thioacetamidate, was found at ≤ 17% of the applied dose. Acetonitrile was the ultimate degradate, accounting for 88% of the applied radiolabel after 4 h. No more than 16% of the applied radiolabel was associated with the soil at any time.

The adsorption and desorption properties of [acetyl-1-^{14}C]thiodicarb were characterized in four soil types. Thiodicarb had little mobility in clay soil, medium mobility in silt loam and sandy loam and high mobility in sandy soil.

The Meeting concluded that thiodicarb degrades within a few days to methomyl in soil under aerobic conditions and that methomyl degrades at a much slower rate. About 50% of methomyl is degraded to volatile compounds over 60 days in various soil types. Under anaerobic conditions in a water–soil mixture, thiodicarb degraded in < 4 h to acetonitrile. The Meeting further concluded that thiodicarb has little mobility in clay but is increasingly mobile in soils containing loam and sand. Some leaching of thiodicarb/methomyl can be expected in soil types other than clay.

Water–sediment systems

The hydrolysis of [acetyl-1-^{14}C]thiodicarb was determined at various pHs in sterile aqueous buffer solutions. After 7 days at pH 7 and pH 5, 92–94% of the applied radiolabel remained as thiodicarb. At pH 9, however, < 1% remained, with 54% methomyl and 32% methomyl oxime. The methomyl underwent degradation at pH 9, resulting in 77% methomyl oxime and 19% methomyl after 30 days.

The photodegradation of [acetyl-1-^{14}C]thiodicarb was tested in a buffered solution at pH 6 under natural sunlight. After 23 days, the solution of a control sample kept in the dark contained 98% of the applied radiolabel, consisting of 67% thiodicarb and 24% methomyl, whereas the irradiated solution contained only 72% of the applied radiolabel, consisting of 12% thiodicarb and 47% methomyl. CO_2 and acetonitrile accounted for 4.4% and 15%, respectively, of the applied radiolabel in the irradiated solution. No volatile compounds arose from the solution maintained in the dark. The time to 50% degradation was calculated to be 8 days in sunlight and 37 days in the dark.

The degradation of thiodicarb, applied at a rate of approximately 0.25 mg/kg of water, was investigated in two water–sediment systems under aerobic conditions over 100 days at 20 °C. The concentration of radiolabel in the water phase of both systems decreased slowly to < 1% of that applied, whereas that in the sediments increased from 10% on day 0 to 30–50% and then decreased to about 15%. Thiodicarb disappeared in both phases within < 1 day. Methomyl accounted for 50% and 17% of the applied dose in the two systems by day 2. Within 100 days, CO_2 accounted for 70% of the applied radiolabel in both systems. The half-time of methomyl was calculated as 20–30 h.

The Meeting concluded that thiodicarb is unstable at alkaline pH, decomposing to methomyl, which is converted to methomyl oxime. The Meeting further concluded that thiodicarb photodegrades in water, with a half-time of 8 days, and that it is rapidly converted to methomyl in water–sediment systems, with ultimate conversion to CO_2.

Methods of analysis

An HPLC method with post-column derivatization and a fluorescence detector has been validated for the determination of thiodicarb and methomyl in milk, muscle, kidney, liver and eggs, with an LOQ of 0.02 mg/kg for both thiodicarb and methomyl. Thiodicarb is unstable in animal matrices and degrades to methomyl.

GLC methods exist for the determination of thiodicarb, methomyl and methomyl oxime as methomyl oxime in plant commodities. The commodity is extracted with acetone and water and treated with a coagulation mixture to remove co-extractives. Caustic hydrolysis is used to convert both thiodicarb and methomyl to methomyl oxime, which is quantified by GLC with a flame photometric detector in the sulfur mode. Variations have been developed, including the use of GPC for extract clean-up, capillary GC columns and MS detectors. The LOD is either 0.02 or 0.04 mg/kg, depending on the exact procedure.

The Meeting concluded that adequate analytical methods exist for the determination of thiodicarb and methomyl in and on plant and animal commodities for the purposes of data collection and for monitoring and enforcing MRLs.

Stability of residues in stored analytical samples

Data were presented on the stability of thiodicarb and methomyl under frozen storage (–20 °C) in celery, head lettuce, leaf lettuce, spinach, soya bean, soya bean processed commodities (meal, hulls, oil, soapstock), apples, sorghum grain, sorghum forage, sweet corn, cotton seed, cotton seed processed commodities (meal, hulls, oil, soapstock), milk and ruminant muscle, kidney, fat and liver.

Thiodicarb plus methomyl was stable in celery, head lettuce and leaf lettuce for at least 7 months, but a significant proportion (30%) was lost in spinach after 5 months. Thiodicarb plus methomyl was stable in soya beans and cotton seed and its processed commodities, except cotton seed soapstock (50% loss) and cotton seed meal (40% loss), for 6 months. Thiodicarb and methomyl were stable in apples for at least 14 months and in sorghum grain for at least 13 months; however, thiodicarb plus methomyl showed a continuous decline on sorghum forage and stover, with a 20% loss over 6 months. Thiodicarb and methomyl were stable on frozen sweet corn (cob plus kernel) for 3 months but showed significant loss thereafter. Thiodicarb and methomyl were each stable in frozen milk, muscle and fat for 2 months but unstable in kidney (50% loss in 2 months, 20% in 1 month), disappearing from liver within 2 days.

The Meeting concluded that the stability of thiodicarb plus methomyl in frozen plant commodities is variable, 6 months generally being the longest desired storage interval. The Meeting further concluded that animal commodities, except liver and kidney, may be stored for 2 months, whereas kidney should be stored no more than 1 month, and liver is not amenable to standard frozen storage (see report item on methomyl).

Definition of the residue

The studies of animal and plant metabolism showed that thiodicarb is converted to methomyl and that methomyl is metabolized primarily to CO_2, acetonitrile and acetamide (animals only). The simpler metabolic products may then be incorporated into natural products, particularly in plants. Thiodicarb/methomyl showed no tendency to bioaccumulate in animal matrices. The P_{ow} of 1.6 indicates no tendency to accumulate in fat. Moreover, the studies of plant metabolism showed that thiodicarb/methomyl has a low tendency to migrate from the point of application, i.e. is not systemic. In both animals and plants, methomyl oxime appeared as a minor metabolite, representing < 1% in most cases, and is probably the intermediate in the metabolism of methomyl.

The analytical method for animal commodities allows determination of both thiodicarb and methomyl but not methomyl oxime. The method for plant commodities does not allow a distinction between thiodicarb and methomyl but reflects the sum of thiodicarb, methomyl and methomyl oxime as methomyl oxime.

The Meeting concluded that the residue definition for both plant and animal commodities should be the sum of thiodicarb and methomyl, expressed as methomyl. This definition takes into account the fact that methomyl oxime is a very minor metabolite and is not determined in some methods. Expression of the total residue as methomyl is consistent with the combination of the MRLs for thiodicarb and methomyl and reflects the fact that a significant portion of the residue found after use of thiodicarb is methomyl. The practical effect is small, as the conversion factor from mg/kg thiodicarb to mg/kg methomyl is 0.92.

Results of supervised field trials

The results of supervised field trial studies were presented for apple, grapes, potato, sugar beet roots, head lettuce, spinach, broccoli, Brussels sprouts, cabbage, cauliflower, collards, chick-peas, garden (green) peas, pea hay, soya beans, soya bean forage and hay and straw, tomato, barley grain, wheat grain, maize grain, sweet corn, rice grain, sorghum grain, barely forage and straw, wheat forage and straw, rice straw, sorghum forage and straw and stover, sweet corn forage, cotton seed, cotton forage, rape seed grain and rape seed forage (green) and straw.

As relevant GAP was not available for sorghum grain, this commodity was not considered further.

Generally, information on moisture content was not available for the forages, stovers and fodders, and the default values for per cent dry matter presented in Appendix IX of the *FAO Manual* were used.

Supervised trials on apple were conducted in Australia, Italy, Greece, Japan and the USA. The only relevant GAP is that of Japan, in which a wettable powder formulation of 750 g ai/kg may be applied three times at a maximum rate of 0.075 kg ai/hl, with a 21-day PHI, or a suspension concentrate formulation of 320 g/l may be applied three times at a rate of 0.043 kg ai/hl, with a 21-day PHI. In eight trials conducted at GAP with the wettable powder formulation, the ranked order of concentrations of residues was: 0.40, 0.43, 0.48, 0.68, 0.91 (2), 1.5 and 1.6 mg/kg. In four trials conducted at GAP with the suspension concentrate formulation, the ranked order of concentrations of residues was: 0.30, 0.32, 0.61 and 0.68. The data from the 12 trials may be combined, as they appear to represent the same population, as follows (ranked order, median underlined): 0.30, 0.32, 0.40, 0.43, 0.48, 0.61, 0.68 (2), 0.91 (2), 1.5 and 1.6 mg/kg (see report item on methomyl).

Supervised trials on grapes were conducted in France, Italy and Spain. The two trials in Spain were not conducted at the GAP of Spain (0.75 kg ai/ha; PHI, 21 days). The one trial in Italy and two in France were conducted at the GAP of France (0.45 kg ai/ha; PHI, 14 days), as no GAP was available for Italy. The ranked order of concentrations of residues was: 0.59 and 0.7 (2) mg/kg (see report item on methomyl).

Supervised trials on potato were presented from Japan and the UK. The trials in Japan involved five foliar applications at a GAP rate of 0.075 kg ai/hl and a 7-day PHI. In four trials conducted at GAP, no residues were detected: < 0.007 mg/kg (2) and < 0.008 mg/kg (2). The trials reported from the UK involved bait application to soil at 0.2 kg ai/ha and a PHI of 21 days. In the 11 trials at GAP, the concentrations of residues were: < 0.04 mg/kg (11) (see report item on methomyl).

Four supervised trials on sugar beet were reported from the UK. The GAP of Belgium requires application of a bait to soil at 0.2 kg ai/ha with no specified PHI. All the concentrations of residues in or on beet roots were < 0.040 mg/kg. The Meeting concluded that the number of trials was insufficient to permit estimation of a maximum residue level or an STMR value. The Meeting further agreed to withdraw the recommended MRL for sugar beet (0.1 mg/kg).

Supervised trials were conducted on lettuce, head, involving application of a granular bait to soil in Italy (no GAP) and France (0.8 kg ai/ha; PHI, 7 days) and aerial and ground foliar application in Spain (no GAP) and the USA (0.84 kg ai/ha; PHI, 14 days). In two trials in France at GAP, the concentrations of residues were 0.048 and 0.14 mg/kg. In 36 trials in the USA at GAP, the ranked order of concentrations of residues was: < 0.04 (3), 0.07 (2), 0.09 (2), 0.12, 0.14, 0.19, 0.21, 0.25, 0.34, 0.35, 0.36, 0.42, 0.44, 0.48, 0.49, 0.71, 0.96, 1.2, 1.7 (2), 1.8, 1.9, 2.6, 3.0, 3.2, 6.2, 6.3, 7.7, 10, 13, 17 and 18 mg/kg (see report item on methomyl).

Supervised field trials on spinach were conducted in the USA. In the nine trials at GAP (foliar spray at 0.84 kg ai/ha; PHI, 14 days), the ranked order of concentrations of residues was: 0.04 (2), 0.21, 1.0, 3.2, 3.5, 4.1, 12 and 25 mg/kg (see report item on methomyl).

Supervised field trials were conducted on collards in the USA (GAP: suspension concentrate, 0.84 kg ai/ha; PHI, 14 days). In two trials conducted under maximum conditions, the concentrations of residues were 1.5 and 1.8 mg/kg. The Meeting concluded that the number of trials was insufficient to permit estimation of a maximum residue level or STMR value but decided to combine the data with those for leafy vegetables treated with methomyl (see report item on methomyl).

Supervised field trials were conducted on broccoli in the USA. The GAP for foliar application (ground or aerial) is 1.2 kg ai/ha; with a 7-day PHI. The ranked order of concentrations of residues in the seven trials at GAP was: 1.1, 1.3, 1.6, 1.9, 2.6, 5.0 and 5.6 mg/kg. Two trials were reported from Australia, but they were not conducted at GAP (0.75 kg ai/ha; PHI, 7 days) (see report item on methomyl).

Supervised field trials were conducted on Brussels sprouts in The Netherlands and the UK (GAP of Belgium: bait application, 40 g ai/kg, 0.2 kg ai/ha; PHI, 21 days). In eight trials conducted at maximum GAP, the ranked order of concentrations of residues was < 0.04 (4), ≤ 0.05 (3) and 0.059 mg/kg. The Meeting decided to establish a group maximum residue level for Brassica vegetables based on foliar application, which results in higher concentrations of residues (see report item on methomyl).

Supervised trials were conducted on foliar application to cabbage in Australia (no GAP) and the USA (1.2 kg ai/ha; PHI, 7 days). In 19 trials at GAP, the ranked order of concentrations of residues was: 0.08 (2), 0.12, 0.53, 0.76, 0.97, 1.2, 1.3, 2.1, 2.7, 2.8, 3.0, 3.1, 3.5, 3.8, 4.3, 4.8, 5.0 and 5.3 mg/kg (see report item on methomyl).

Supervised field trials with foliar application to cauliflower were conducted in the USA (GAP: 1.2 kg ai/ha; PHI, 7 days). In eight trials at GAP, the ranked order of concentrations of residues was: 0.09, 0.16, 0.27, 0.45, 0.64, 0.71 and 2.3 (2) mg/kg (see report item on methomyl).

Supervised trials were conducted on garden peas in Australia. None of the trials met GAP (0.28 kg ai/h; PHI, 21 days), and the Meeting agreed to recommend withdrawal of the MRL for peas, shelled (succulent seeds) (0.5 mg/kg).

Supervised trials of foliar application to chick-pea were conducted in Australia. None of the trials met GAP (0.28 kg ai/ha; PHI, 21 day PHI).

Supervised field trials were conducted on foliar treatment of soya beans in Australia (0.28 kg ai/ha; PHI, 28 day), Brazil (0.06 kg ai/ha; PHI, 14 days) and the USA (0.84 kg ai/ha; PHI, 28 days). None of the trials in Australia and Brazil met GAP. In 19 trials with foliar ground application in the USA that were at GAP, the ranked order of concentrations of residues was: < 0.04 (17), 0.06 and 0.15 mg/kg.

The Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR value of 0.04 mg/kg and a highest residue of 0.15 mg/kg. The Meeting agreed to maintain the current recommended MRL for soya bean (dry) (0.2 mg/kg).

Supervised field trials on tomato were conducted in Australia (0.52 kg ai/ha; PHI, 1 day), Spain (0.94 kg ai/ha; PHI, 7 days) and the USA (no GAP). The trials in Spain were conducted in glasshouses. In two trials in Australia that met GAP, the concentrations of residues were 0.09 and 0.73 mg/kg. In nine trials in Spain at GAP, the ranked order of concentrations was: 0.05, 0.06, 0.08, 0.09, 0.13, 0.16, 0.18, 0.23 (2), 0.33 and 0.73 mg/kg (see report item on methomyl).

Supervised field trials were conducted on sweet corn in the USA (0.84 kg ai/ha; no PHI) and Australia (0.75 kg ai/ha; PHI, 7 days) with foliar application. None of the trials in Australia met GAP. In 22 trials in the USA at GAP, the ranked order of concentrations of residues was: < 0.02, 0.02, < 0.03 (6), < 0.04, 0.04, 0.06, 0.07 (2), 0.08, 0.11, 0.13, 0.22, 0.28, 0.43, 0.54, 0.82 and 1.5 mg/kg. Additional trials of seed treatment were presented, but there is no GAP for this application in the USA. Field trials were also conducted with methomyl, and the Meeting decided to combine the data sets (see report item on methomyl).

Supervised field trials of the application of granular bait to barley were conducted in Germany (0.2 kg ai/ha; PHI not specified) and the UK (at drilling and before the second node is detectable; 0.2 kg ai/ha; PHI not specified). In two trials in Germany and six in the UK at GAP, the ranked order of residues was: < 0.02 (2), ≤ 0.04 (5) and 0.06 mg/kg. Trials with foliar application of methomyl resulted in higher concentrations (see report item on methomyl).

Supervised field trials of the application of granular bait to wheat were conducted in Germany (0.2 kg ai/ha; PHI not specified) and the UK (at drilling and before the second node is detectable; 0.2 kg ai/ha; PHI not specified). In three trials in Germany and six in the UK at GAP, the ranked order of concentrations of residues was < 0.02 (3), < 0.04 (5) and 0.06 mg/kg. Supervised field trials conducted with methomyl yielded higher values (see report item on methomyl). Supervised field trials with seed treatment were provided from Brazil, but no GAP was reported and the LOQ was unacceptably high, at 0.2 mg/kg.

Supervised field trials on maize were conducted in Brazil with both foliar (0.1 kg ai/ha; PHI, 30 days) and seed treatment (7 kg/t of seeds). The trials with foliar application were conducted at an excessive rate (two to four times GAP), but the concentrations of residues were < 0.1 (6) mg/kg. In four trials of seed treatment at GAP the concentration was < 0.1 mg/kg. The Meeting concluded that data from the two uses could not be combined. The data for foliar treatment were combined with similar data in trials with methomyl (see the report item on methomyl).

Supervised trials on rice were conducted in Japan with foliar application (1.2 kg ai/ha; PHI, 30 days). In four trials at GAP, the ranked order of concentrations of residues was: < 0.25 (2) and < 0.4 (2) mg/kg. Supervised trials were conducted in Brazil with seed treatment of rice (5.3 kg/t of seed). The one trial at GAP and three trials at exaggerated rates (1.4–2.7 times GAP) yielded no quantifiable residues; the concentrations of residues were < 0.10 (4) mg/kg. The Meeting considered that the data from foliar and seed treatments could not be combined, and that the numbers of trials for the two treatments were insufficient to permit estimation of a maximum residue level or an STMR value. Furthermore, the LOQ of the analytical method for rice grain in Japan was unacceptably high.

Supervised field trials on barley forage were conducted in Germany (0.2 kg ai/ha) and the UK (early season, bait application; 0.2 kg ai/ha). In seven trials at GAP, the ranked order of concentrations of residues was: < 0.02 (2), ≤ 0.04 (3), 0.04 and 0.25 mg/kg. Barley forage is not a recognized animal feed commodity.

Supervised field trials were conducted on wheat forage in Germany (0.2 kg ai/ha) and the UK (0.2 kg ai/ha). In 12 trials at GAP, the ranked order of concentrations of residues was: < 0.02 (4), ≤ 0.04 (4), < 0.03 (2), 0.06 and 0.21 mg/kg. Trials conducted with methomyl by foliar application yielded higher concentrations (see report item on methomyl).

Supervised field trials on barley straw were conducted in Germany (0.2 kg ai/ha) and the UK (0.2 kg ai/ha). In nine trials at GAP, the ranked order of concentrations of residues was: ≤ 0.04 (6), < 0.2 (2) and 0.24 mg/kg (see report item on methomyl).

Supervised field trials on wheat straw were conducted in Germany (0.2 kg ai/ha). In three trials at GAP, the concentration of residues was < 0.2 mg/kg. The Meeting decided that the number of trials was insufficient to permit estimation of a maximum residue level or an STMR value (see report item on methomyl).

Supervised field trials were conducted on rice straw in Japan. In four trials at GAP (1.2 kg ai/ha; PHI, 30 days), the ranked order of concentrations of residues was: < 0.5, 0.62, and ≤ 1 (2) mg/kg. The Meeting decided to combine these data with those for cereal grain straw treated with methomyl (see report item on methomyl).

Supervised trials on sweet corn fodder were conducted in Australia, but none was at GAP (0.75 kg ai/ha; PHI, 7 days).

Supervised trials were conducted on foliar application to sweet corn forage in the USA. In 12 trials at GAP (0.84 kg ai/ha; PHI, 21 days), the ranked order of concentrations of residues was: < 0.02, < 0.05, 0.06, 0.16, 0.21, 0.56, 1.1, 2.3, 5.2, 6.9, 11 and 18 mg/kg. Trials were also conducted with methomyl, and the Meeting decided to combine the results (see report item on methomyl).

Supervised trials on cotton seed were conducted in Australia (GAP: 0.84 kg ai/ha; PHI, 21 days), Brazil (no GAP), Greece (GAP: 0.8 kg ai/ha; PHI, 28 days), Spain (0.94 kg ai/ha; PHI, 21 days), the Sudan (no GAP) and the USA (1 kg ai/ha; PHI, 28 days) by foliar application. None of the trials in Brazil corresponded to the GAP of Argentina (0.38 kg ai/ha; PHI, 20 days), and none of the trials in Spain was at GAP. One trial in Australia, two in Greece and 15 in the USA were at GAP; the ranked order of concentrations of residues was: ≤ 0.04 mg/kg (12), < 0.05, 0.05, 0.09 and 0.10 (3) mg/kg. Supervised trials were also conducted with methomyl, and the Meeting decided to combine the values (see report item on methomyl). Supervised field trials were presented on seed treatment in the USA, but there is no GAP for this application.

Many supervised field trials on cotton forage were conducted in the USA, but none was at GAP (1.0 kg ai/ha; PHI, 28 days), and cotton forage is not a recognized feed item, either in the USA or in the *FAO Manual*.

Supervised field trials on rape seed were conducted in Germany (0.2 kg ai/ha; no PHI) and the UK (soil broadcast up to and including stem extension stage; 0.2 kg ai/ha; no PHI) with granular bait. In 14 trials at GAP, the ranked order of concentrations of residues in rape seed was: < 0.04 (4), 0.04, ≤ 0.05 (8) and 0.05 mg/kg. The Meeting estimated a maximum residue level of 0.05 mg/kg and an STMR value of 0.05 mg/kg.

Supervised field trials on rape seed forage (green) were conducted in Germany (winter rape, soil broadcast; 0.2 kg ai/ha; no PHI) and the UK (soil broadcast, up to an including stem extension stage; 0.2 kg ai/ha; no PHI) with granular bait. In 11 trials at GAP, the ranked order of concentrations of residues was: ≤ 0.02 (8) and < 0.04 (3) mg/kg. Applying the default value for dry matter of 30%, the Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR value of 0.07 mg/kg and a highest residue of 0.13 mg/kg. Data were also presented for rape seed straw, but this is not a recognized feed commodity.

Fate of residues during processing

Studies were conducted on the processing of grapes in France and Spain and of soya, tomato, apple, sweet corn and cotton in the USA. The studies were conducted according to standard commercial practices. One study on tomato was rejected because samples in which residues had been incurred in the field were not used.

The processing factors and the maximum residue levels and STMR-P and HR-P values resulting from application of the factor to the estimated maximum residue levels and STMR values presented above and in the report item on methomyl are summarized in the latter.

Residues in animal commodities

No studies were provided on poultry, but the study of the nature of the residue in poultry was conducted after feeding concentrations ≤ 102 ppm for 21 days. The concentrations of residues reached a plateau in eggs within 10 days. Thiodicarb, methomyl and methomyl oxime were not detected in eggs or tissues, at an estimated LOD of about 0.005 mg/kg. As poultry diet contains a maximum of 2 ppm thiodicarb/methomyl (see report item on methomyl), quantifiable amounts of thiodicarb and/or methomyl in poultry commodities are unlikely. For methomyl plus thiodicarb, the Meeting estimated maximum residue levels of 0.02(*) mg/kg in meat, 0.02(*) mg/kg in eggs and 0.02(*) mg/kg in edible offal. Furthermore, the Meeting estimated STMR and highest residue values of 0.00 mg/kg for edible offal, meat and eggs (see report item on methomyl).

In a study in lactating dairy cattle in the USA, thiodicarb was administered orally for 28 consecutive days at 350 or 1050 ppm, as measured from actual feed consumption. Milk, liver, kidney, fat and muscle from cows at 1050 ppm contained no thiodicarb (LOQ 0.1 mg/kg) and no methomyl (LOQ 0.1 mg/kg). The vast majority of samples contained no detectable residues. Thiodicarb/methomyl was detected at 0.02 mg/kg in one milk sample on day 1 and at 0.02 mg/kg on day 25, in one muscle sample at about 0.03 mg/kg, in one liver sample at 0.06 mg/kg and in one kidney sample at 0.01 mg/kg from cows at 1050 ppm. A control sample of liver contained 0.09 mg/kg.

The Meeting estimated the dietary burden of thiodicarb for farm animals on the basis of the diets listed in Appendix IX of the *FAO Manual*. As the data for methomyl and thiodicarb were combined for estimating maximum residue levels and STMR values, a single diet applies to both. The dietary calculations are given in the report item on methomyl. The dietary burden of beef and dairy cattle was estimated to be 28 ppm. No residue (< 0.1 mg/kg) was found in meat, milk, fat, kidney or liver from cattle fed at 350 or 1050 ppm. Thiodicarb/methomyl was detected in a few samples at 0.02–0.03 mg/kg from cattle at 1050 ppm. Residues of thiodicarb/methomyl will therefore not be quantifiable in ruminant commodities. For methomyl plus thiodicarb, the Meeting estimated a maximum residue level of 0.02 (*) mg/kg for meat, 0.02 (*) mg/kg for milk and 0.02 (*) mg/kg for edible offal (see the report item on methomyl). The estimates for meat and milk confirm the existing values.

The average daily dietary burden of thiodicarb for ruminants is a fraction of the maximum daily burden. Thus, the STMR values for meat and milk were estimated to be 0.000 mg/kg. The highest residues for meat and milk were estimated to be 0.000 mg/kg, as there is no reasonable expectation of residues (see report item on methomyl).

RECOMMENDATIONS

On the basis of the data from supervised trials and studies of processing, the Meeting concluded that the concentrations of residues listed in the evaluation of methomyl are suitable for establishing MRLs and for assessing IEDIs and IESTIs.

Definition of residue (for compliance with MRLs and for estimation of dietary intake): sum of thiodicarb and methomyl, expressed as methomyl.

Dietary risk assessment

See evaluation of methomyl.

REFERENCES

Anderson-Taylor, K.E. 1996a. Molluscicides: Thiodicarb: Residue in potatoes treated with EXP 60392B Year 2. UK 1993. Rhône Poulenc Agriculture Ltd. Study RES/93/007. Unpublished.

Anderson- Taylor, K.E. 1996b. Molluscicides: Thiodicarb: Residue in potatoes treated with EXP 60392D Year 3. UK 1994. Rhône Poulenc Agriculture Ltd.. Study RES/94/007 - Document n°201127. Unpublished.

Andrawes, N.R. and Bailey, R.H. 1979. Metabolism of acetyl-1-^{14}C LARVIN in the rat. Union Carbide Agricultural Products Co. File No.26925, UCC Project No. 814C50. Unpublished.

Andrawes, N.R. and Bailey, R.H. 1980. Fate of Acetyl-1-^{14}C Thiodicarb in Laying Chickens under Continuous Feeding Conditions. Union Carbide Agricultural Products Co. File No.27414, Project No. 814C50. Unpublished.

Anon. 1982. Larvin® Thiodicarb/Methomyl: General Method of Residue Analysis by Gas Chromatography. Union Carbide Agricultural Products Co. No reference. Unpublished.

Anon. 1986. Thiodicarb residues on Australian tomatoes. Trial 1985. Colorado Analytics Research Development Corp, Colorado Springs, CO, USA. Unpublished.

Anon. 1992a. Determinazione di residu di thiodicarb su ortaggi. Neotron richerche chimiche biochimiche e microbiologiche. No. 0026 . Unpublished.

Anon. 1992b. Residues of thiodicarb in winter barley. Seasons 1987-88 and 1988-89. BBA reports No.03725, 05132 and 06753. Unpublished.

Anon. 1992c. Residues of thiodicarb in winter oil seed rape. Germany Seasons 1987-88 and 1988-89. BBA reports No. 03724, 05071, 05076, 05103, 05107, 05133, 05051 and 05128 . Unpublished.

Anon. 1992d. Residues of thiodicarb in winter wheat. Seasons 1987-88 and 1988-89. BBA reports No.05148, 05149 and 03675. Unpublished.

Anon. 1996. Determinação de Resíduos de Thiodicarb em grãos de milho. Laboratório de Ecotoxicologica - CENA -USP. Piracicaba, 1996. CP-2344/96 DE 11.06.96. Certificado de análise de Thiodicarb a nível de resíduo na cultura. De milho. CPP - Rhodia SP. Unpublished.

Anon. 1997. Certificado de análise de Thiodicarb a nível de resíduo na cultura. de soja. CPP - Rhodia SP . CP-2479/97 DE 01.07.97. Unpublished.

Anon. 1998a. Certificado de análise de Thiodicarb a nível de resíduo em cultura de trigo. CPP - Rhodia - SP. Estudo CP-2557/98 de 03.03.98. Unpublished.

Anon. 1998b. Determinação de Resíduos de Thiodicarb na Cultura de Trigo. Laboratório de Ecotoxicologica - CENA - USP. Piracicaba, 1998. Unpublished.

Anon. 2000a. Laudo Técnico de Análise de Resíduos de Semevin 350 RPA/ Bonanza em milho/Ensaio A Determinação de Resíduos de Semevin 350 RPA/BONANZA em milho. ESALQ - USP de 21/06/00. Unpublished.

Anon. 2000b. Laudo Técnico de Análise de Resíduos de Semevin 350 RPA/ Bonanza em milho/Ensaio B Determinação de Resíduos de Semevin 350 RPA/BONANZA em milho. ESALQ - USP de 21/06/00. Unpublished.

Anon. 2000c. Laudo Técnico de Análise de Resíduos de Semevin 350 RPA/ Bonanza em arroz/Ensaio B Determinação de Resíduos de Semevin 350 RPA/BONANZA em arroz. ESALQ - USP de 21/06/00. Unpublished.

Anon. 2000d. Laudo Técnico de análises de resíduos de Larvin 800 WG em algodão/Ensaio A. ESALQ/USP, Piracicaba - SP, 2000. Unpublished.

Anon. 2000e. Laudo Técnico de análises de resíduos de Larvin 800 WG em algodão/Ensaio B. ESALQ/USP, Piracicaba - SP., 2000. Unpublished.

Anon. 2001a. AventisCrop Science. Larvin Thiodicarb: Review of Residue data on Apple - Trials conducted in Japan. 1983-1991. No references. Unpublished.

Anon. 2001b. AventisCrop Science. Larvin Thiodicarb: Review of Residue data on Rice - Trials conducted in Japan. 1983-1986. No references. Unpublished.

Avakian, M.D. 1991. Thiodicarb: Magnitude of the residue processed food/feed apples North Carolina. Environmental Technologies Institute. ETI Report No. S78AP01. Unpublished.

Barciet, F. 1990. Memo and data only: LARVIN / Vignes - vin: residue data from 1981, '82, '83. Rhône-Poulenc Agro, Lyon. Unpublished.

Bascou, J.P. 1999. Thiodicarb - Henry's Law Constant. Rhône-Poulenc Agro. Lab Project ID File No. 40161. Unpublished.

Bieber, W-D. 1992. Degradation of Thiodicarb in Aerobic Aquatic Environment. NATEC Institute. Project No.NA 91 1034. Unpublished.

Bird, R. and Coffey, J.S. 1992. Magnitude of thiodicarb residue on soybean raw agricultural commodities after ground application of LARVIN® brand 3.2 thiodicarb insecticide/ovicide. Environmental Technologies Institute, Inc. Study No.USA91L30. Unpublished.

Blundstone, H.A.W, Rose, D.J., Robertson, A. 1987. Determination of Thiodicarb residues in winter wheat plants. Camden Food Preservation Research Association. Project No.I60/03/11/86.

Brockelsby, C.H., Maycey, P.A. and Savage, E.A. 1989. Insecticides : Thiodicarb: Residue studies on cereals. UK 1987/88. Rhône Poulenc Agriculture Ltd. Report No.1295. Unpublished.

Brockelsby, C.H., Maycey, P.A. and Savage, E.A. 1990a. Insecticides : Thiodicarb: Residue studies on cereals. UK 1989/90. Rhône Poulenc Agriculture Ltd. Report No.1535. Unpublished.

Brockelsby, C.H., Maycey, P.A. and Savage, E.A. 1990b. Molluscicides: Thiodicarb: Residue studies on oil seed rape. UK 1987/88. Rhône-Poulenc Agriculture Ltd. Report No.1439. Unpublished.

Brockelsby, C.H., Maycey, P.A. and Savage, E.A.1990c. Molluscicides: Thiodicarb: Residue studies on oil seed rape. UK 1989/90. Rhône-Poulenc Agriculture Ltd. Report No.1562. Unpublished.

Burr, C.M. 2000. [^{14}C]-Thiodicarb: Rate of Degradation in Three Soils at 20°C and One Soil at 10°C. Aventis CropScience UK Ltd. Lab. Project No. 16966. Document No. 202508. Unpublished

Carringer, S.J.. 2000. Carbamate Insecticide Market Basket Survey. The Carringer , Inc., Apex, NC 27502. Study No. TCI-99-001. Unpublished

Clark, D.V. and Shields, R. 1986. LARVIN® 375 thiodicarb insecticide residues in chick peas. Australia, 1987. Analysis. Analchem Consultants Pty, Ltd. Lab Ref. No. 1870/86/5. Unpublished.

Clarke, D.E. 2000. [^{14}C]-Thiodicarb: Anaerobic Soil Degradation. Aventis CropScience UK Ltd. Lab. Project No. 18237. Document No. 202645. Unpublished

Cookinham, J. 1999ab. Thiodicarb - Partition Coefficient. Midwest Research Institute. Lab Project ID: 5385-F(03) Unpublished.

Cookinham, J. 1999b. Thiodicarb - Dissociation Constant. Midwest Research Institute. Lab Project ID: 5385-F(02. Unpublished.

Cookinham, J. 1999c. Thiodicarb - Water Solubility. Midwest Research Institute. Lab Project ID: 5385-F(04. Unpublished.

Cooper, J.F. and Mestres, R. 1982. Résidus de thiodicarbe dans les raisins - Résultats des essais La Littorale. Réf. UC 51-702. Université de Montpellier. Unpublished.

Cooper, J.F. and Mestres, R. 1983. Courbe de dégradation du thiodicarbe sur vigne. Université de Montpellier. Unpublished.

Cranor, W. 1991. Soil Adsorption / Desorption with ^{14}C-Thiodicarb. ABC. ABC Report No. 37453. Unpublished.

Davis, C.W. and Wilkes, L.C. 1994. Thiodicarb: Magnitude of residues in meat and milk of lactating dairy cows. Analytical Development Corporation. Project No.1456, RPAC study No. US94L01R. Unpublished.

Davis, C.W., Wilkes, L.C. and Norris, F.A. 1996. Supplemental submission of freezer storage stability for Thiodicarb: Magnitude of residues in meat and milk of lactating dairy cows. Analytical Development Corporation. ADC Project No.1456, RPAC Study No. US94L01R- SS. Unpublished.

Doble, M.L., Hatcher, G., Oddy, A.M. 2000. [^{14}C]-Thiodicarb Photodegradation in soil. Aventis CropScience UK Ltd. Lab. Project No. 18280. Document No. 202587. Unpublished.

Feung, C.S. and Blanton, C.S. 1986a. Larvin® Metabolism of Thiodicarb in corn: Evaluation of acetamide as a potential metabolite. Union Carbide Agricultural Products Co. File No.34962, Project No. 804R10 Unpublished.

Feung, C.S. and Blanton, C.S. 1986b. LARVIN®: Metabolism of Thiodicarb in peanut: Evaluation of acetamide as a potential metabolite. Union Carbide Agricultural Products Co. File No.35231, Project No. 804R10. Unpublished.

Feung, C.S. and Blanton, C.S. 1987. LARVIN® Photolysis of Thiodicarb in Aqueous Buffered Solution. Rhône-Poulenc Ag Company. Lab Project ID: 804R14. File No. 40254 Unpublished.

Feung, C.S. and Chancey, E.L. 1977a. Metabolism of UC 51762 in cotton plant. Union Carbide Agricultural Products Co. File No.23370, Project No. 814C21. Unpublished.

Feung, C.S. and Chancey, E.L. 1977b. Metabolism of UC 51762 in corn and wheat plant. Union Carbide Agricultural Products Co. File No.24168, Project No. 814C21. Unpublished.

Feung, C.S. and Chancey, E.L. 1978a. Absorption, Translocation and Metabolism of UC 51762 in Cotton Plants and Seeds. Union Carbide Agricultural Products Co. File No.24948, Project No. 814C21. Unpublished.

Feung, C.S. and Chancey, E.L. 1978b. Absorption, translocation and metabolism of UC 51762 in the carrot plant. Union Carbide Agricultural Products Co. File No.24758, Project No. 814C21. Unpublished.

Feung, C.S. and Chancey, E.L. 1979a. Disposition and Metabolism of UC 51762 in the senescent soybean foliage and seeds. Union Carbide Agricultural Products Co. File No.25817, Project No. 814C21. Unpublished.

Feung, C.S. and Chancey, E.L. 1979b. Disposition and Metabolism of UC 51762 in tomato foliage and fruits. Union Carbide Agricultural Products Co. File No.26055, Project No. 814C21. Unpublished.

Feung, C.S. and Chancey, E.L. 1979c. Disposition and metabolism of UC 51762 in potato tuber and foliage. Union Carbide Agricultural Products Co. File No.26927, Project No. 814C50. Unpublished.

Feung, C.S. and Jeffs, R.A. 1986. Larvin® Metabolism of Thiodicarb in tomatoes: Evaluation of acetamide as a potential metabolite. Union Carbide Agricultural Products Co. File No.34315, Project No. 804R10. Unpublished.

Feung, C.S. and Weisbach, P.J. 1991a. Aerobic Soil Metabolism of Thiodicarb. Rhône-Poulenc Ag Company. File No.41068, Project No: EC-91-142. Unpublished.

Feung, C.S. and Weisbach, P.J. 1991b. Hydrolysis of Thiodicarb in Aqueous Buffer Solutions. Rhône-Poulenc Ag Company. Lab Project ID: EC-91-143 Unpublished report

Feung, C.S., College, P.R. and Chancey, E.L. 1980. Studies on the Disposition of ^{14}C-Thiodicarb in Lactating Cows. Union Carbide Agricultural Products Co. File No.27350, Project No. 814C50. Unpublished.

François, J.M. 1999. Thiodicarb -Determination of the Explosion Properties - Flammability, ability for self heating and oxidizing properties. Safety process Laboratory, Rhône-Poulenc Industrialisation. RP Ind. No.99-303-SEC. Unpublished.

Gateaud, L. and Yslan, F. 2000. Thiodicarb and metabolites (methomyl and methomyl oxime) formulation EXP06298B(SC) South/Spain/1998-1999 - 4 Decline study trials - Residues in Lettuce (leaf. Aventis CropScience Lyon/lD .Study No.99-568. Unpublished.

Gateaud, L. and Yslan, F. 2001a. Thiodicarb and metabolites (methomyl and methomyl oxime) formulation EXP60392E (GB) North /France/2000 - 2 Decline study trials - Residues in Lettuce (leaf. Aventis CropScience Lyon/lD .Study No.00-561. Unpublished.

Gateaud, L. and Yslan, F. 2001b. Thiodicarb and metabolites (methomyl and methomyl oxime) formulation EXP60392E (GB) North /France/2000 - 2 Harvest Trials - Residues in Lettuce (leaf. Aventis CropScience Lyon/lD .Study No.00-562. Unpublished.

Hanlon, C.M. and Norris, K.J. 1991. Metabolism of ^{14}C-Thiodicarb in Lactating Goats. Analytical Development Corporation. Lab Project No. 1245-1, Sponsor Project No. EC-91-170. Unpublished.

Hanlon, C.M. 1994. Metabolism of ^{14}C -thiodicarb in lactating goats, Sponsor Project EC-91-170 - Supplemental Report to MRID 42919601. Analytical Development Corporation. Lab Project No. 1245-1, Supplemental report. Unpublished.

Hawkins, D.R., Mayo, B.C., Pollard, A.D. and Haynes, L.M. 1993. ^{14}C-Thiodicarb: The metabolism in monkeys. Huntingdon Research Center Ltd. HRC Report No. HRC/RNP 398/921553. Unpublished.

Hiles, R.A. 1987. The metabolism of Thiodicarb (acetyl-1-^{14}C) in albino rats. Hazleton Laboratories America, Inc. Project No. HLA 6224-100. Unpublished.

Huhtanen, K. and Dorough, H.W. 1976. Isomerization and Beckman rearrangement reactions in the metabolism of Methomyl in rats. Union Carbide Agricultural Products Co. *Pesticide Biochem. Physiol*., 6 : 571-583.

Hunt, T.W 1982. Thiodicarb residues in sweet corn and sweet corn cannery waste -section D- Union Carbide agricultural products company. File No.31094, Project No.06570 Part 2. Unpublished.

Hunt, T.W. 1983. LARVIN® thiodicarb residues in 1982 Australian apples. Union Carbide Agricultural Products Co. Project No. 20566, File 1451V. Unpublished.

Hunt, T.W. 1986a. LARVIN® thiodicarb : Insecticide - section D-residues broccoli, cabbage, cauliflower, head lettuce, almonds. US 1982-1985. Union Carbide Agricultural Products Co. File No.34501, Project No. 804R11. Unpublished.

Hunt, T.W. 1986b. Thiodicarb insecticide tomato processing study. Union Carbide Agricultural Products Co. File No.35032, Project No. 804R11. Unpublished.

Hunt, T.W. 1988a. Thiodicarb insecticide residue report collards. US 1988. Rhône-Poulenc Ag Company. File No.40458, Project No. 804R10. Unpublished.

Hunt, T.W. 1988b. Thiodicarb insecticide -section D-residues in leafy vegetables: additional data to support MRID#401704-01. Rhône-Poulenc Ag Company. File No.40365, Project No. 804R10. Unpublished.

Hunt, T.W. 1988c. Thiodicarb insecticide soybean seed treatment. Rhône-Poulenc Ag Company. File No.40388, Project No. 804R10. Unpublished.

Hunt, T.W. 1988d. Thiodicarb insecticide - Section D-residues soybean 1 GPA. Rhône-Poulenc Ag Company. File No.40383, Project No. 804R10. Unpublished.

Hunt, T.W. 1988e. Thiodicarb insecticide - section D-residues sorghum; additional data. Rhône Poulenc Ag Company. File No.40312, Project No. 804R10. Unpublished.

Hunt, T.W. 1988f. Thiodicarb insecticide residues sorghum; raw agricultural and processed fraction. Union Carbide Agricultural Products company, Inc. Project No. 804R10, File No.35252. Unpublished.

Hunt, T.W. 1988g. Thiodicarb insecticide sorghum seed treatment. Rhône Poulenc Ag Company. File No.40387, Project No. 804R10. Unpublished.

Hunt, T.W. 1988h. Thiodicarb insecticide residues cotton seed treatment. Rhône-Poulenc Ag Company. File No.40386, Project No. 804R10. Unpublished.

Hunt, T.W. 1988i. Thiodicarb insecticide -section D-residues sweet corn seed treatment. Rhône Poulenc Ag Company. File No.40389, Project No. 804R10. Unpublished.

Hunt, T.W. 1989a.Thiodicarb insecticide - section D-residues apples. Crop field trials. Rhône-Poulenc Ag Company .File No.40674, Project No. 804R10. Unpublished.

Hunt, T.W. 1989b. Thiodicarb insecticide -section D-residues sorghum; additional data to support MRID 40204202. Rhône Poulenc Ag Company. File No.40571, Project No. 804R10. Unpublished.

Hunt, T.W. 1989c. Thiodicarb insecticide residue report CA/AZ cotton. Rhône-Poulenc Ag Company. File No.40512, Project No. 804R10. Unpublished.

Hunt, T.W. 1989d. Thiodicarb insecticide -section D-residues cotton. Additional data to support MRID 410191-01. Rhône-Poulenc Ag Company. File No.40585, Project No. 804R10. Unpublished.

Hunt, T.W. 1989e.Thiodicarb insecticide apple processing. Rhône-Poulenc Ag Company.File No.40657, Project No. 804R10. Unpublished.

Hunt, T.W. 1994. Storage stability of thiodicarb in apples. Additional data to support pesticide petition OF3833, MRID 41347101 Rhône-Poulenc Ag Company. File No.44219. Unpublished.

Hunt, T.W. 1996. Validation of the Method for Quantification of Thiodicarb and its Metabolite, Methomyl in Animal Substrates. Rhône-Poulenc Ag Company. RP Study No. EC96 331. Unpublished.

Hunt, T.W. and Langdon, T.R. 1982. General method for the determination of thiodicarb residues. Union Carbide Agricultural Products Co. File No.30715, Project No. 06570. Unpublished.

Hunt, T.W. and Langdon, T.R. 1983. LARVIN® thiodicarb residues in 1982 Australian cottonseed. Union Carbide Agricultural Products Co. Report No. 1452V. Unpublished.

Hunt, T.W. and Langdon, T.R. 1985. LARVIN® thiodicarb residues in 1985 Australian soybean seed and straw. Union Carbide Agricultural Products Co. Unpublished.

Hunt, T.W. and Schwehr, R.D. 1987. Thiodicarb Insecticide. Section D - Residues Sweet Corn Forage. Union Carbide agricultural products company. File No.35293, Project No.804R10. Unpublished.

Hunt, T.W., Langdon, T.R. and Myers, W.R. 1983. LARVIN® thiodicarb - Section D - residues tomatoes. US 1981-1982. Union Carbide Agricultural Products Co. File No.32116, Project No. 804R11. Unpublished.

Imose, J. 1986. Report on the results of pesticide residue analysis, Larvin research project. Aburahi Lab., Shionogi and Co. Ltd. Unpublished

Jendrzejczak, N. and Yslan, F. 2000. Thiodicarb and metabolites (methomyl and methomyl oxime) formulation EXP06297A (WG) South/Greece/1999-1 Harvest trial - Residues in cotton (seed. Aventis CropScience Lyon/lD. Study No.99-657. Unpublished.

Jordan, R.C. and Wyatt, D. 1994. Confined Accumulation Study on Rotational Crops with ^{14}C-Thiodicarb. American Agricultural Services, Inc. and Analytical Developmental Corporation. ADC Project 1270, AASI Trial No. 91-02-14C-CR, Study No. EC-91-156. Unpublished.

Keats, A. 1989a. Thiodicarb residue studies in cabbage. Australia 1989. Rhône Poulenc Rural Australia. Study No. ak/aw/ak89006. Unpublished.

Keats, A. 1989b. Thiodicarb: residue studies in Maize. Australia 1989. Rône-Poulenc Rural Australia Pty, Ltd. Study No. ak/am/ak89010. Unpublished.

Keats, A. 1989c. Thiodicarb residue studies in sorghum. Australia 1989. Rhône Poulenc Rural Australia. Study No. ak/am/ak89008. Unpublished.

Keats, A. 1989d. Thiodicarb residue studies in sweet corn. Australia 1989. Rhône Poulenc Rural Australia. Study No. ak/aw/ak89005. Unpublished.

Keats, A. 1990. Thiodicarb residue studies in broccoli. Australia 1990. Rhône Poulenc Rural Australia. Study No.AK/TN/AK905. Unpublished.

Keats, A. 1991. Thiodicarb residue studies in pea and pea hay. Australia 1991. Rhône-Poulenc Rural Australia. Study No. AK/JG/AK002. Unpublished.

Kowite, W.J. 1998a. LARVIN® 3.2: Magnitude of thiodicarb residues in/on fresh market cherry and large tomato RAC. Rhône-Poulenc Ag Company. File No.45559, Study No. 96L10369. Unpublished.

Kowite, W.J. 1998b. LARVIN® 3.2: Magnitude of thiodicarb residues in tomato processing fractions. Rhône-Poulenc Ag Company. File No.45563, Study No. 96L10370. Unpublished.

Langdon, T.R 1987. Thiodicarb insecticide -section D-residues leafy vegetables. Union Carbide Agricultural Products Co. File No.34768, Project No. 804R10. Unpublished.

Lee II, R.E. 1991a. Thiodicarb: Stability of thiodicarb on cottonseed and cottonseed processed commodities during frozen storage. Rhône-Poulenc Ag Company. File No.41038, Study No. EC-90-118. Unpublished.

Lee II, R.E. 1991b. Thiodicarb: Stability of thiodicarb on sweet corn and sweet corn processed commodities during frozen storage. Rhône-Poulenc Ag Company. File No.41037, Study No. EC-90-127. Unpublished.

Lee II, R.E. 1991c. Magnitude of thiodicarb residues on soybeans and processed commodities after ground application of LARVIN® 3.2 brand thiodicarb insecticide/ovicide. Rhône-Poulenc Ag Company. File No.41003, Study No.USA90L01. Unpublished.

Lee II, R.E. 1991d. Thiodicarb: Magnitude of thiodicarb residues on cottonseed and processed commodities after ground applications of LARVIN® 3.2 brand thiodicarb insecticide/ovicide. Rhône-Poulenc Ag Company. File No.40969, Study No. USA90L82. Unpublished.

Lee II, R.E. 1992a. Thiodicarb: Stability of thiodicarb on soybean and soybean processed commodities during frozen storage. Rhône-Poulenc Ag Company. File No.41039, Study No. EC-90-119

Lee II, R.E. 1992b. Magnitude of thiodicarb residues on cotton raw agricultural commodities after ground and aerial applications of LARVIN® brand 3.2 thiodicarb insecticide/ovicide. Rhône-Poulenc Ag Company. File No.41198, Study No. USA91L82. Unpublished.

Lee II, R.E. 1993. Thiodicarb residues in sweet corn and sweet corn cannery waste following ground applications of LARVIN® brand 3.2 thiodicarb insecticide/ovicide. Rhône Poulenc Ag Company. File No.44128, Study No.USA92L01. Unpublished.

Leonard, M.S.1999. Validation of "HPLC Method of Analysis for Thiodicarb and Methomyl in Soil". Rhône-Poulenc Ag Company. Study No. US99L17644. Unpublished

Lipscomb, W.T. 1987. Solubility Testing of Thiodicarb. Rhône-Poulenc Ag Company. Lab Project ID: 804F10, File No. 40163. Unpublished.

Lusson, R. and Muller, M.A. 1989a. Thiodicarbe : Formulation LARVIN® SC, Essais France 1988. Résidus dans le raisin, le vin, et la lie. Rhône-Poulenc Agro, Lyon. Ref. AG/CRLD/AN/8916392

Lusson, R. and Muller, M.A. 1989b. Thiodicarbe - Formulation LARVIN 80DF (WG) - Essais Soudan 1988. Résidus dans les graines et les feuilles de coton ainsi que dans le sol. Rhône-Poulenc Agro, Lyon. Ref. AG/CRLD/AN/8916381. Unpublished.

Maestracci, M. 1997a. Bifenthrin-Thiodicarb: Formulation EXP61108A (SC), trial France 1995. Residues in grapes. Rhône-Poulenc Agro, Lyon. Study No. 95-541. Unpublished.

Maestracci, M. 1997b. Bifenthrin-Thiodicarb: Formulation EXP61108A (SC), trial France 1995. Residues in grapes and wine. Rhône-Poulenc Agro, Lyon. Study No. 95-540. Unpublished.

Maestracci, M. 1997c. Bifenthrin-Thiodicarb: Formulation EXP61108A (SC), trial France 1995-1996. Residues in grapes, must, wine and alcohol. Rhône-Poulenc Agro, Lyon. Study No. 95-539. Unpublished.

Maestracci, M. 1998a. Thiodicarb and metabolites (methomyl and methomyl oxime): Formulation EXP60392D (RB), trials UK 1997. Residues in sugar beet. Rhône Poulenc Agro, Lyon. Study No. 97-732. Unpublished.

Maestracci, M. 1998b. Thiodicarb and metabolites (methomyl and methomyl oxime): Formulation EXP60392D (RB), trials UK 1997. Residues in Brussels sprouts. Rhône Poulenc Agro, Lyon. Study No. 97-733. Unpublished.

Maestracci, M. 1998c. Thiodicarb and metabolites (methomyl and methomyl oxime): Formulation EXP06298A(SC), trials Spain 1996-1997. Residues in tomato (greenhouse), decline study. Rhône-Poulenc Agro, Lyon. Study No. 96-645. Unpublished.

Maestracci, M. 1998d. Thiodicarb and metabolites (methomyl and methomyl oxime): Formulation EXP06298A(SC), trials Spain 1997. Residues in tomato (in greenhouse. Rhône-Poulenc Agro, Lyon. Study No. 97-680. Unpublished.

Maycey, P.A., McDonald, A.J. and Savage, E.A. 1993. Molluscicides: Thiodicarb: Residue studies on potatoes. UK 1992. Rhone Poulenc Agriculture Ltd. Study No. P92/218. Document n°200163 Unpublished.

Mestre, R. 1986. Residues of thiodicarb on sunflower, barley and lettuce. La Littorale; ET/FB/AB/67. Unpublished.

Moertl, S. and Class, T. 1998. Determination of Thiodicarb in Tomato, Sugar Beet and Brussels Sprout: Detailed Description of Analytical Method with Validation Results. PTRL Europe. Report B 250-4 G, PTRL Europe Study No. P 250 G, RP Study No. 97-140. Unpublished.

Muller, M.A. 1990a. Thiodicarbe Formulation MINAVIN (SC) essais Italie 1989, Résidus dans la pomme (étude de décroissance. Rhône-Poulenc Agro. Ref. AG/CRLD/AN/9015940. Unpublished.

Muller, M.A. 1990b. Thiodicarbe Formulation MINAVIN (SC) essais Italie 1989, Résidus dans le raisin (étude de décroissance. Rhône-Poulenc Agro, Lyon. Ref. AG/CRLD/AN/9015941. Unpublished.

Oddy, A.M. 1999. [^{14}C]-Methomyl: Degradation in Two Water Sediment Systems. Rhône-Poulenc Agriculture Ltd. RPA Study No.14854. Unpublished.

Richard, M. and Muller, M.A. 1993. Thiodicarbe et ses métabolites (methomyl et methomyl oxime): Formulation EXP06298A (SC), essais Espagne 1992-1993. Résidus dans la tomate (étude décroissance en plein air et en serre. Rhône-Poulenc Agro, Lyon. Study No. 91-242. Unpublished.

Richard, M. and Muller, M.A. 1994a. Thiodicarbe et ses métabolites (methomyl et methomyl oxime): Formulation EXP06298A (SC), essais France 1992. Résidus dans le raisin et le vin. Rhône-Poulenc Agro, Lyon. Study No. 92-147. Unpublished.

Richard, M. and Muller, M.A. 1994b. Thiodicarbe et ses métabolites (methomyl et methomyl oxime): Formulation EXP06298A (SC), essais Espagne 1993. Résidus dans le raisin et le vin. Rhône-Poulenc Agro, Lyon. Study No. 93-624. Unpublished.

Richard, M. and Muller, M.A. 1994c. Thiodicarbe et ses métabolites (methomyl et methomyl oxime): Formulation EXP06298A (SC), essais Espagne 1993. Résidus dans le coton (fibre et graine. Rhône-Poulenc Agro, Lyon. Study No. 93-625. Unpublished.

Richard, M. and Muller, M.A. 1995a. Thiodicarb and metabolites (methomyl and methomyl oxime): Formulation EXP60392B (GB), The Netherlands 1992. Residues in Brussels sprouts. Rhône Poulenc Agro, Lyon. Study No. 92-304. Unpublished.

Richard, M. and Muller, M.A. 1995b. Thiodicarb and metabolites (methomyl and methomyl oxime): Formulation EXP06297A (WG), Greece 1994. Residues in apple. Rhône-Poulenc Agro. Study No. 94-689. Unpublished.

Richard, M. and Muller, M.A. 1995c. Thiodicarb and metabolites (methomyl and methomyl oxime): Formulation EXP06297A (WG), Greece 1994. Residues in cotton (grain. Rhône-Poulenc Agro, Lyon. Study No. 94-692. Unpublished.

Robinson, T.W. 1989. HPLC Method of Analysis for Thiodicarb and Methomyl in soil. Rhône-Poulenc Ag Company.SOP-90318. Unpublished

Robles, J.M. and Bascou J.P. 2000. Thiodicarb : Physical Characteristics. Rhône-Poulenc Agro. Study n°99-195. Unpublished.

Schweitzer, M.G. 1993. Vapor Pressure of Thiodicarb. Battelle. Battelle Study No. SC920210. Unpublished.

South, P.L. and Pitts, G.E. 1993. Stability of Thiodicarb. Battelle. Battelle Study No. SC930255. Unpublished.

Tew, E.L. 1992. An analytical method for the quantification of Thiodicarb and its metabolite Methomyl in/on agricultural crops. Rhône-Poulenc Ag Company. File No.41230. Unpublished.

Toshio Hayashi et al 1986. Report on the results of pesticide residue analysis. Japan Medical Foods Association; JPPA No. Plant86P-2-58. Unpublished

Yslan, F. and Baudet, L. 1999. Thiodicarb and metabolite (Methomyl and Methomyl oxime) : Residue in Brussels sprout. Formulation EXP60392D(GB. North/The Netherland/1998. 4 harvest trials. Rhône Poulenc Agro, Lyon. Study No.98-747. Unpublished.

[1] 7 to 10 days interval depending on the pest intensity

[1] every 7 days

[1] Do not exceed 6.7 kg/ha per season

[1] Do not exceed 6.7 kg/ha per season

[1] Do not exceed 3.4 kg/ha per season

[1] Do not exceed 8.4 kg/ha per season, 1 to 7 day intervals.

[1] every 7 days

[1] every 7 days

[1] Do not exceed 8.4 kg/ha per season, 1 to 7 day intervals.

[1] 10 to 15 days interval depending on the pest intensity

2