PHOSALONE (060)

EXPLANATION

Phosalone, a phosphorodithioate acaricide and insecticide, was evaluated for residues by the JMPR in 1972, 1975 and 1976. It was scheduled for re-evaluation in the CCPR periodic review programme by the 1993 JMPR. The toxicology was reviewed at the 1993 JMPR, resulting in the ADI being lowered from 0.006 mg/kg bw to 0.001 mg/kg bw. The WHO estimated that current MRLs result in the TMDI being 509% of the ADI and no reduction factors were found. The scheduled residue review was postponed until the present Meeting because data could not be provided in time for review in 1993.

The Meeting received data from the manufacturer (Rhône-Poulenc), as well as information on GAP from Canada, Germany, The Netherlands and Spain and limited residue data from Germany and The Netherlands. In response to an inquiry, the manufacturer informed the Meeting that no information was available on the stability of stored analytical samples, the fate of residues during processing, or residues in the edible portions of foods.

IDENTITY

ISO common name: phosalone

Chemical name

IUPAC:*S*-6-chloro-2,3-dihydro-2-oxobenzoxazol-3-ylmethyl *O,O*-diethyl phosphorodithioate

CA: *S*-[(6-chloro-2-oxo-3(2*H*)-benzoxazolyl)methyl] *O,O*-diethyl phosphorodithioate

CAS No.: [2310-17-0]

CIPAC No: 109

Synonyms: Rubitox; Zolone; RP-11974; NPH-1091

Structural formula:

Molecular formula: $C_{12}H_{15}ClNO_4PS_2$

Molecular weight: 367.81

Physical and chemical properties

Pure active ingredient

Vapour pressure: $<0.5 \times 10^{-6}$ mm Hg at 25°C, static method (Guyot and Mede, 1988); 4.6×10^{-7} mm Hg at 25°C, gas saturation method, OECD protocol 104 (Guyot *et al*., 1989).

Melting point: No information was provided. Literature references: 45-48°C (1972 JMPR Evaluations); 45-47°C (Farm Chemicals Handbook, 1992); 48°C (Pesticide Manual, 8th Ed.).

Octanol/water partition coefficient:

Log P = 3.77 or $K_{ow} = 5.89 \times 10^3$ by reversed phase LC (Rhône-Poulenc 1987)
Log P = 4.37 or $K_{ow} = 2.38 \times 10^4$ (Bowman and Sans, 1983)
Log P = 4.3 or $K_{ow} = 1.99 \times 10^4$ (Chiow *et al*., 1977).

Pesticides with Log P_{OW} >4 are likely to be fat-soluble (1991 JMPR Report).

Solubility (at 20°C):

1.7 mg/l water (Laurent and Buys, 1975).

Literature references:
10 mg/l water (1972 JMPR; Pesticide Manual, 8th ed.)
200 g/l methanol, ethanol (1972 JMPR)
1000 g/l acetone, acetonitrile, benzene, chloroform, cyclohexanone, dioxane, ethyl acetate, methylene chloride, methyl ethyl ketone, toluene, xylene (1972 JMPR).

Specific gravity: No information was provided.

Hydrolysis: Stable (<10% degradation after 4 weeks) in water at pH 5 and 7. Half-life 9 days at pH 9 (Laurent and Buys, 1975; see also Metabolism and Environmental Fate, Environmental fate in water/sediment systems).

Photolysis: No information was provided.

Technical material

Purity: 94.61% (one determination of one lot) (Charbassol, 1988).

Melting range: No information was provided.
Stability: No information was provided.

Formulations:

35 EC: 350 g/l emulsion concentrate
30 & 25 WP: 300 and 250 g/kg wettable powders
SC: 500 g/l suspension concentrate
Dust: 25, 30 or 40 g/kg

Mixed formulations:

SC: 400 g/l phosalone and 35 g/l teflubenzuron
EC: 225 g/l phosalone and 62.5 g/l cypermethrin
240 g/l phosalone and 60 g/l pirimicarb
?: 260 g/l phosalone and 200 g/l parathion-methyl

METABOLISM AND ENVIRONMENTAL FATE

Information was available on the fate of residues in food and in the environment. Relevant metabolites and phosalone-related chemical structures are identified in Table 1.

Table 1. Metabolites and degradation products associated with phosalone.

Code	Chemical name	Common or other name	Structure	Found in
I	*S*-6-chloro-2,3-dihydro-2-oxobenzoxazol-3-ylmethyl *O,O*-diethyl phosphorodithioate	Phosalone, RP 11974		rat cow goat apple sorghum soil
II	*S*-6-chloro-2,3-dihydro-2-oxobenzoxazol-3-ylmethyl *O,O*-diethyl phosphorothioate	Phosalone oxon, O-analogue, RP 12244		rat goat apple (leaves) sorghum soil
III	6-chloro-3-mercaptomethyl-2-oxobenzoxazole	Thiol, mercapto, RP 14450, U-6, M-4		rat cow goat
IV	6-chloro-3-methylthiomethyl-2-oxobenzoxazole	Sulphide, RP 19914		rat goat
V	6-chloro-3-methylsulphinylmethyl-2-oxobenzoxazole	Sulphoxide, RP 19889		rat goat

Code	Chemical name	Common or other name	Structure	Found in
VI	6-chloro-3-methylsulphonylmethyl-2-oxobenzoxazole	Sulphone, RP 19888		rat goat
VII	6-chloro-2-oxobenzoxazoline 6-chlorobenzoxazolone	Aglycone, RP 11881, chlorobenzoxazolone		goat apple (leaves) sorghum soil
VIII	6-chloro-3-(*N*-glucosyl)-2-oxobenzoxazoline (6-chloro-2-benzoxazolon-3-yl)-1-glucopyranose	Glycoside, RP 20650		goat? sorghum soil
IX	2-amino-5-chlorophenol	chloroaminophenol, CAMP, RP 18709, U-5B, M-2		cow goat sorghum soil
X	3*H*-2-amino-7-chloro-phenoxazin-3-one	Phenoxazone, RP 18726		goat soil
XI	Bis(*O,O*-diethylphosphorothioyl) disulphide.	Disulphide, disulphide of diethyldithiophosphoric acid	$(C_2H_5O)_2PS\text{-}S\text{-}S\text{-}SP(OC_2H_5)_2$	soil
XII	*O,O*-diethyl *S*-hydrogen phosphorodithioate	diethyldithiophosphoric acid, R.P. 5961	$(C_2H_5O)_2PS\text{-}SH$	soil rat
XIII	*O,O*-diethyl *S*-hydrogen phosphorothioate	diethylthiolophosphoric acid	$(C_2H_5O)_2PO\text{-}SH$	rat soil
XIV	*O,O*-diethyl *O*-hydrogen phosphorothioate	diethylthionophosphoric acid, R.P. 5961	$(C_2H_5O)_2PS\text{-}OH$	

Animal metabolism

Metabolism in animals is characterized by rapid elimination of phosalone and its metabolites in the urine and faeces, mainly the urine. Although in most tissues most of the residue has not been identified, the available data indicate oxidation of phosalone to its oxygen analogue, cleavage of phosalone to yield *O,O*-diethyl dithiophosphoric acid (XII) and of the oxon to the corresponding thiolic acid (XIII), and of both phosalone and its oxon to 2-oxo-3-mercaptomethyl-6-chlorobenzoxazole (III). The diethylthionophosphoric acid (XIV) may be formed either by direct oxidation of (XII) or by hydrolysis of phosalone oxon to the thiolic acid (XIII) and immediate isomerization to the thiono acid (Demoras and Fournel, 1968; Demoras *et al.*, 1966). A metabolic pathway in animals can be proposed by combining the findings of metabolism studies, mainly on rats and goats (Figure 1).

Laboratory animals. The transformation of phosalone in rats has been described (Demoras and Fournel, 1968; Demoras, 1979; Smith *et al.*, 1988, 1991; WHO, 1993). Phosalone was reported to be oxidized in the rat to its oxon (II) and both of these converted to the putative intermediate thiol (III), with the phosphorus portion of phosalone forming diethyldithiophosphoric acid and that of the oxon forming diethylthiophosphoric acid. The thiol is presumably subsequently transformed sequentially into the sulphide (VI), sulphoxide (V) and sulphone (VI).

The residues in the faeces were mostly phosalone. Although most of the metabolites were excreted, phosalone, its oxon (in mouse liver), and the sulphide, sulphoxide and sulphone were identified in the tissues of rats and/or mice and the thio- and dithiophosphoric acids in urine, faeces and whole animals. The thiol (III) was not identified, but was presumed to exist. Proportionally the sulphoxide was the highest tissue residue. Approximately 70 and 20% of the label was excreted in the urine and faeces respectively after 72 hours. The metabolic pathway in mice and rabbits was considered to be similar.

Ruminants

A goat metabolism study and studies of the disposition of [^{14}C]phosalone in lactating cows were provided as well as feeding trials with unlabelled phosalone which are discussed in the section "Residues resulting from supervised trials".

Cows. In one disposition study 926 mg (491 ìCi) of [^{14}C]phosalone uniformly labelled in the aromatic ring (0.53 ìCi/mg specific activity) was administered via a fistula directly into the rumen of a 530 kg lactating Holstein cow whose daily dietary ration was approximately 8.2 kg (whether this was the total feed intake was not clear). Urine and faeces samples were collected over a 100-hour period, and the cow was milked in the morning and evening each day after feeding (Craine, 1974a). The total recovery of ^{14}C from the experiment was 100.1%, with urine containing 93.7%, faeces 6.1% and milk 0.3%. The lack of radioactive $C0_2$ in the urine suggested that no degradation of the benzene ring had occurred.

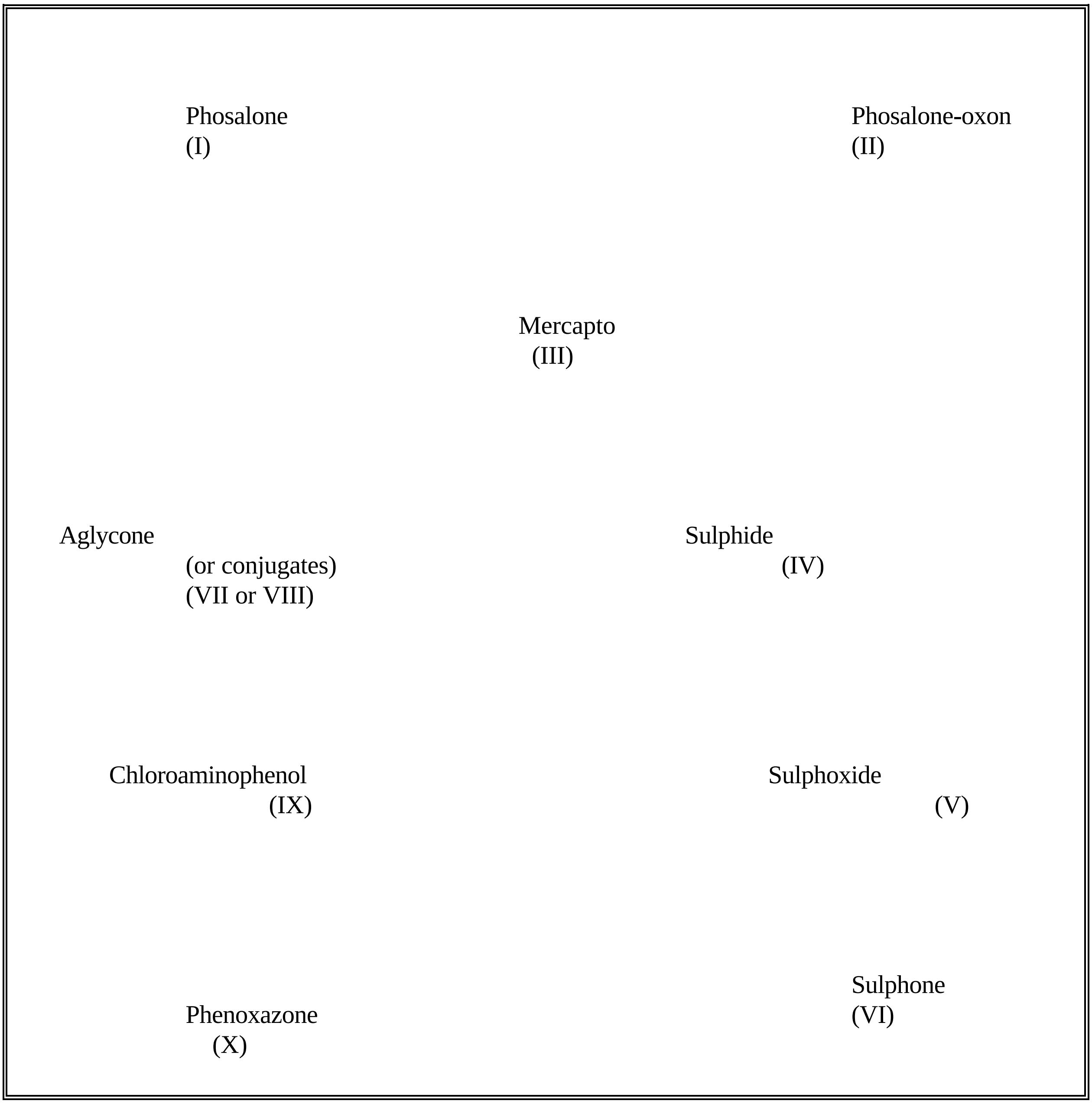
Significant residues occurred in the urine within one hour of dosing, excretion remaining steady after 2 hours and beginning to decrease after approximately 40 hours; 78.8% of the dose was excreted after 48 hours and 93% within approximately 100 hours. Residues were not measurable in the faeces until 16 hours after dosing. Residues in the milk were measurable at the first milking (8 hours after dosing), with maximum residues of 0.13 mg/kg phosalone equivalent.

A repeat study was conducted with a different cow using phosalone with a higher proportion (>99%) of the labelled compound and an increased specific activity (2.57 ìCi) (Craine, 1974b). The

Figure 1. Proposed Metabolic Pathway for Phosalone in Animals

---> diethylthionophosphoric acid <--------
(XIV)

diethyldithiophosphoric acid diethylthiolophosphoric acid
(XII) (XIII)



results were almost identical in terms of residue distribution and very similar in other respects. The highest residues in the milk were 0.19 mg/kg. Separation of the cream from the milk revealed that ≤10% of the milk residue was in the cream.

The analyses of the urine and milk from the two cow studies (Craine, 1974a,b) are included in a separate study (Craine, 1975). Solvent partitioning, TLC, autoradiography, derivatization, and GC-MS were used in the analyses. The urine extracts showed 9 TLC zones, some of which comprised more than one compound, giving a total of at least 14 compounds. Most of the ^{14}C was found in zones designated as U-2 (2-12% of the total radioactivity), U-5 (15-40%), U-6 (30-42%) and U-9 14-23%). Fraction U-5 was found to contain two compounds, one of which was unidentified and unstable and was degraded to 2-

amino-5-chlorophenol (CAMP). Zone U-6 was found to contain 6-chloro-3-mercaptomethyl-2-oxobenzoxazole (the thiol).

Extracts of skimmed milk gave 6 TLC zones representing 9 compounds. The main zones were designated M-2 (41% of the total), M-3 (13.9%), M-4 (11.9%) and M-6 (15.7%), although M-6 comprised three compounds none of which was considered to exceed 10% of the total. Zones M-2 and M-4 were found to contain 2-amino-5-chlorophenol and the thiol respectively. Zone M-3 was not analysed.

Goats. In a goat metabolism study (Witkonton *et al.*, 1980) a lactating goat was fed aromatic ring-labelled [^{14}C]phosalone in five gelatin capsules, each containing phosalone giving 2.5 x 10^9 dpm (1124 ìCi) and having a specific activity of 7,032 dpm/ìg = 3.2 ìCi/mg. The capsules were given twice daily for two days and once on the 3rd day. The feeding equivalent was 957 ppm phosalone based on the total administered dose (1.78 x 10^6 ìg) and the total feed consumed (1860 g). The main reported purpose of the study was to validate an analytical method. The validation is considered later.

The preparation for TLC consisted in extraction of milk with acetone and tissues with 1:1 acetone/methanol, followed by acetonitrile/hexane partition. The acetonitrile extracts (representing 63.3% of the total radioactive residue (TR) in the milk; 74.5% of the TR in fat; 52.5% in muscle; 71.5% in liver, and 93.2% in kidney) were analysed by TLC. Urine was collected, milk was taken twice daily and pooled, and tissue samples were taken after the final milking on the last day. The total radioactivity was measured and the residues characterized or identified. The urine residues accounted for approximately 61% of the applied radioactivity.

A variety of techniques was used for the analyses, including total combustion, scintillation counting, solvent partitioning, acid hydrolysis, column chromatography, mass spectrometry, derivatization, GLC, and radiometric TLC. The results are summarized in Table 2.

Table 2. ^{14}C-residues in goat tissues and milk from feeding labelled phosalone (TLC Analyses) (Witkonton *et al.*, 1980).

Compound and code no. (see Table 1)	Kidney 13.1 mg/kg[1]		Liver 12.6 mg/kg[1]		Fat 1.02 mg/kg[1]		Muscle 0.27 mg/kg[1]		Milk 0.86 mg/kg[2]	
	mg/kg	%TR	mg/kg	%TR	mg/kg	%TR	mg/kg	%TR	mg/kg	%TR
Phosalone (I)	0.185	1.4	0.41	3.3	0.118	11.6	0.003	1.1	0.005	0.6
Sulphide (IV)	0.347	2.6	3.75	29.9	0.122	12.0	0.006	2.2	0.005	0.6
Thiol (III)	0.014	0.1	0.08	0.6	0.008	0.8	0.002	0.7	0.002	0.2
Oxon (II)	0.006		0.07	0.6	0.009	0.9	0.002	0.7	0.008	
Sulphone (VI)	0.000		0.1	0.8						
Phenoxazone (X)	0.000		0.11	0.9	0.000		0.000	0.0	0.000	
Aglycone (VII)	0.16	0.1	0.17	1.35	0.01	1.0	0.002	0.7	0.003	0.3
Sulphoxide (V)	0.075	0.6	2.6	20.7	0.093	9.2	0.029	10.8	0.019	2.2
CAMP (IX)	0.102	0.8	0.06	0.5	0.008	0.8	0.002	0.7	0.003	0.3
Glycoside region	0.32		0.09	0.7	≤0.04		0.004	1.5	0.008	0.9
Total identified or characterized		5.6	7.4	59.3		36.3		18.4	0.045	5.1

Compound and code no. (see Table 1)	Kidney 13.1 mg/kg[1]		Liver 12.6 mg/kg[1]		Fat 1.02 mg/kg[1]		Muscle 0.27 mg/kg[1]		Milk 0.86 mg/kg[2]	
	mg/kg	%TR	mg/kg	%TR	mg/kg	%TR	mg/kg	%TR	mg/kg	%TR
TLC origin	≤2.7		≤0.26		≤0.05		≤0.08		≤0.21	

[1] Total by oxidative combustion and scintillation counting
[2] Total by direct liquid scintillation counting

The total identified or characterized residue in each tissue as a proportion of the TR found in the tissue in the samples from the goat study by GLC methods described under methods of analysis showed broadly good agreement with the corresponding results found by TLC: milk 10.6% of the TR; liver 57.2%; muscle 7.5%, fat 33.7% and kidney 4.8%. The low recovery of identified compounds was thought to be due to unidentified and unmeasured water-soluble conjugates formed as part of the detoxification process. It was suggested that these might be reduced to undetectable levels by appropriate withdrawal periods.

Pigs. The disposition of [^{14}C]phosalone from skin applications to pigs has been studied by Craine (1974c). A 13.7 mg (23.6 ìCi) dose of [^{14}C]phenyl-labelled phosalone was applied to a 144 cm^2 section of de-haired skin on an 11-kg male Yorkshire pig. Urine and faeces were collected at intervals for 48 hours and skin and tissue samples taken after slaughter. ^{14}C was not measurable in the faeces or blood, or in fat tissues outside the application zone. 73.6% of the applied dose was found in ethanolic washes of the application zone and adjacent areas. 22.9% of the applied dose was found in the skin of the application zone, 1.3% in a retaining strap and 1.7% in the urine. Trace residues were found in the liver and kidney, and in fat underneath the application zone, all <0.07 mg/kg phosalone equivalent.

No metabolism studies on hens were provided.

Plant metabolism

A translocation study on potatoes and metabolism studies on sorghum and apples were provided. Although only about 50% of the radioactivity in the sorghum foliage and <0.1-22% in the grain was identified in the metabolism studies, the available information suggests a plant metabolic process similar to one of the routes of metabolism proposed in Figure 1 for animals. The pathway indicated in plants is some conversion of phosalone (I) to its oxon (II), and of both to the aglycone (VII), thence to the glycoside (VIII) and finally to chloroaminophenol (IX). No attempts were made in the plant studies to determine the other metabolites identified in animal studies (thiol (III), sulphide (IV), sulphoxide (V), sulphone (VI) and phenoxazone (X)), but only the apple study was conducted after the more definitive of the animal metabolism studies. Phenoxazone has also been identified as a degradation product in soil (see "Fate of residues in soil").

Potatoes. A translocation study was conducted on potted potato plants grown in a greenhouse (Ambrosi and Demoras, 1974). [^{14}C]phosalone labelled in the benzoxazolone ring was applied by syringe to the tops of individual leaves, either approximately 16 ìg in 50% acetone solution or 12 ìg in aqueous emulsion. The treated leaves, petioles, leaf parts below the treated area, the rest of the aerial parts, roots, tubers (at the 3rd sampling) and soil were sampled at 2, 7 and 14 days after treatment. Extraction was with 1:1 acetone/water. Samples were counted for radioactivity and from the 3rd sampling analysed by TLC.

After two days 97% of the applied radioactivity was recovered from the whole plant and after 3 days 87.8%. At each sampling ≥99.5% of the recovered residue was in the area of application. The lower total recovery from plants on the third day was attributed to lower extraction in the aqueous emulsion experiment and a postulated uptake into natural plant constituents. Less than 0.1% of the applied radioactivity was found in the soil, and <0.01% was detected in the tubers after 14 days. TLC analyses were said to show most of the recovered residue as phosalone, with possibly some oxon.

Sorghum. The metabolism of phosalone in sorghum has been elucidated by Wargo *et al.* (1978) on the basis of greenhouse studies (included as an appendix) conducted at Michigan State University (Penner, 1977). [^{14}C]phosalone, labelled uniformly in the aromatic ring, of 91.7% purity and with a specific activity of 50,114 dpm/ìg was formulated as an EC for spray application in two separate application regimens.

In one experiment 90 sorghum plants (one plant/flat after thinning) of 25 to 50 cm height (before flowering) were sprayed at a rate equivalent to 3.4 kg ai/ha and a field equivalent of 95 l water/ha. In the second, 60 plants were treated at the flowering stage at a field rate equivalent of 1.8 kg ai/ha and 95 l/ha. Fresh plant parts (whole plant above ground, roots, flowerhead, fodder and seedheads) were sampled at intervals ranging from 1 day (both studies) to 92 days (second study) or 132 days (first study). Some whole-plant radioautograms were developed as a measure of ^{14}C coverage and some indication of the degree of any translocation. Total residues in the plant parts were determined by combustion.

Each plant part was extracted with acetone/water (80:20). ^{14}C was measured in the filter cake and the acetone evaporated from the extract which was then partitioned with dichloromethane. The aqueous fraction was further partitioned with ethyl acetate. Both the dichloromethane and ethyl acetate fractions were counted for total radioactivity and the components of the residues characterized by TLC. The remaining aqueous layer was subjected to acid hydrolysis and again partitioned with ethyl acetate. The aqueous and ethyl acetate extract fractions from this partition were counted for ^{14}C and the ethyl acetate extract analysed by TLC. Standard TLC reference solutions of phosalone, its oxon, the glycoside, aglycone and chloroaminophenol were used for comparison.

Residues were identified by similar R$_F$s to the standards in two of three solvent systems, except those of chloroaminophenol which were too low for confirmation. The dichloromethane fraction contained phosalone and its oxon, which were separated on a Florisil column and confirmed by GC-MS. The ethyl acetate fraction contained the glycoside, which was purified on a Florisil column and confirmed by a colour reaction with 2,6-dichloro-*N*-chloro-*p*-benzoquinone monoimine. The ethyl acetate extract of the acid hydrolysate contained the aglycone, which was also purified on a Florisil column and confirmed by 2-dimensional TLC.

The residues in the foliage increased or decreased with time, depending on the treatment and the compound:

	% of Residue			
	Treatment 1 (10-50 cm ht.)		Treatment 2 (flowering stage)	
	day 1	day 134	day 1	day 92
phosalone	82.7	44.1	85	26.5
phosalone oxon	1.2	2.7	0.5	3.7
glycoside	1.1	2.6	0.9	1.4
aglycone	0.75	4.6	1.1	16.7
unknown R_F 0.52	0.5	5	0.25	7.7
unknown R_F 0.35	0.3	3	1.03	6.1

The residues in sorghum at harvest are shown in Table 3.

Table 3. Distribution of ^{14}C-phosalone activity in fresh sorghum at harvest[1] (Wargo *et al.*, 1978).

Residues	Foliage				Grain				Glume = hulls = seed head less grain)			
	Treatment 1[2]		Treatment 2[3]		Treatment 1		Treatment 2		Treatment 1		Treatment 2	
	mg/kg[4]	% of total ^{14}C	mg/kg[4]	% of total ^{14}C	mg/kg[4]	% of total ^{14}C	mg/kg[4]	% of total ^{14}C	mg/kg[4]	% of total ^{14}C	mg/kg[4]	% of total ^{14}C
Extractable ^{14}C	34.0	83.3	36.2	79.3	0.01	7.6	3.1	58	0.04	22	27.5	55
Bound ^{14}C	6.8	16.7	9.4	20.7	0.1	92.4	2.3	42	0.12	78.4	22.6	45.4
Total ^{14}C	40.8	100	45.6	100	0.11	100	5.4[6]	100	0.16	100	50	100
Total ^{14}C on dry-matter basis	147		164		0.13		6.2[7]		0.18		58	
Identified compounds	21.7	53.3	22.0	48.3	<0.01	<0.01	1.1	21.5	<0.01	<0.1	11.7	23.4
Phosalone (I)	18	44.2	12.1	26.5			0.9	16.9			7.6	15.3
Oxon (II)	1.1	2.7	1.7	3.7			0.18	3.4			0.8	1.6
Glycoside (VIII)	0.72	1.8	0.63	1.4			0.06	1.2			0.73	1.5
Aglycone (VII)	1.9	4.6	7.6	16.7	<0.01				<0.01		2.5	5
CAMP (IX)	0.0	0.0	0.0	0.0			0.0	0.0			0.09	0.18
Unidentified residues	18.4	45.9	23.1	50.6		100	4.1	72.9	0.16	100	37.6	75.7
R_F 0.35[5]	1.3	3.1	2.8	6.1			0.3	5.7			0.8	1.7
R_F 0.52[5]	2	5	3.5	7.7			0.4	7.3			5.5	11.1
At TLC origin	0.5	1.2	0.5	1			0.14	0.73			0.3	0.58
Other ^{14}C on TLC plate	5.6	14.6	2.9	6.4	0.008	7.6	0.6	10.4	0.03	18.3	4.3	8.6
^{14}C in water after extraction	2.2	5.3	4	8.7	<0.001	0	0.36	6.8	0.005	3.3	4.1	8.3
Bound ^{14}C	6.8	16.7	9.4	20.7	0.1	92.4	2.3	42	0.12	78.4	22.6	45.4
% of total ^{14}C accounted for		99.2		98.9		100		94.4		100		99.3

[1] Harvest 134 days after treatment 1 and 92 days after treatment 2

[2] Spraying equivalent to 3.4 kg ai/ha at 25 to 50 cm growth height

[3] Spraying equivalent to 1.7 kg ai/ha at flowering

[4] All residues, whether identified or not, expressed as mg/kg phosalone

[5] Reaction with diazomethane and trifluoroacetic anhydride suggested the presence of phenolic compounds.

[6] Reduced to 2.6 mg/kg when washed with water [7] Reduced to 3.0 mg/kg when washed with water

Several points can be noted from Table 3. About 80% of the ^{14}C was extractable from the foliage regardless of whether phosalone was applied at 25-50 cm height or at the flowering stage. However, extraction was low from grain (7.6%) and glume (22%) after the earlier treatments, and low but better (about 55%) after treatment at flowering. The major identified residues in foliage in decreasing order were phosalone, the aglycone, phosalone oxon and the glycoside, from both the earlier and later treatments. Little identified or unidentified residue was found in the grain from the earlier treatment, while phosalone was the major residue found from the later treatment, followed by the oxon and then the glycoside. A reduction of about half of the residue in grain by washing with water suggests a surface residue caused by contamination when separating the grain from the hulls. A similar profile was found in the glume (seedhead less grain) after the later treatment, except that CAMP was tentatively identified in the glume and the aglycone was the second most abundant residue.

The major characterized but unidentified metabolites designated R_F 0.52 and R_F 0.35 had phenolic properties, since they were extractable from an acid medium and reacted with trifluoroacetic anhydride and diazomethane. The remaining extractable residue consisted of approximately 18 compounds (11% of the total residue), no one of which was more than 1% of the total.

The water-soluble metabolites of phosalone were isolated from sorghum in a follow-up study (Craig *et al.*, 1980c). It could not be used because alternate pages were missing and photographs of TLC plates were illegible.

Apples. Selected fruit and leaves on two apple trees (red delicious) were brush-treated in the field with benzene-labelled [^{14}C]phosalone (3.3 dpm/mg specific activity) at a rate equivalent to 3.4 kg ai/ha (Kimmel *et al.*, 1990). One tree (trial 1) was treated while the fruit were immature and sampled 14 days after treatment. The second (trial 2) was treated when the fruit were almost mature and sampled at harvest 24 days after treatment. Leaves and apples were first rinsed (leaves with methanol, apples with 25% methanol in water) and the rinsed samples extracted with methanol. All fractions were analysed by TLC and HPLC using as standards phosalone (I), its oxon (II), the aglycone (VII), benzoxazolone (i.e. VII without chlorine), and the thiol (III) for identification. Acceptable recoveries of ^{14}C were obtained by methanol extraction, as follows.

	Recovery of ^{14}C, %	
	Trial 1	Trial 2
Apple leaf	98.8	91.6
Apple peel	89.9	88.8
Apple pulp	73.0	104.1

Residues of ^{14}C in the apples and apple leaves at harvest are shown in Table 4. After 14 days the total ^{14}C in the leaves plus apples was about 85% of that applied and after 24 days about 50%. The losses of ^{14}C were not accounted for. No rainfall was recorded for the test period. In the apples harvested after 24 days approximately 97% of the ^{14}C was in or on the peel (<1% could be removed with a methanol/water rinse) and about 3% was in the pulp. In contrast, about 59% of the ^{14}C in the leaves after 24 days could be removed with a methanol rinse. Residues after 24 days (trial 2) were predictably lower than those after 14 days (trial 1). TLC analysis indicated that about half of the low pulp residue was phosalone *per se*.

Table 4. ^{14}C residues in methanol extracts or rinses of apple leaf and fruit samples (Kimmel *et al.*, 1990).

Sample	% of applied ^{14}C		[^{14}C]phosalone equivalent, mg/kg	
	Trial 1[1]	Trial 2[2]	Trial 1	Trial 2
Leaf rinse	37.3	21.6	511	453
Leaf extract	28.4	15.2	389	318
Subtotal	65.7	36.8		
Apple rinse	0.2	0.1	0.2	0.2
Apple peel extract	18.8	11.2	79.9	58.1
Apple pulp extract	0.3	0.4	0.6	0.9
Subtotal	19.3	11.7		
Total recovery	85	48.5		
% Loss	15	51.5		

[1] Equivalent of 3.4 kg ai/ha brushed on to selected leaves and immature fruit; harvest 14 days after treatment

[2] As trial I, but separate tree, almost mature fruit treated; harvest 24 days after treatment

The samples were analysed by TLC and HPLC. The analyses gave comparable qualitative and quantitative results, but resolution was better with HPLC. The residues found in the extracted fractions are shown below (Table 5). Most of the determinations were by HPLC. Those by TLC are in parentheses.

Table 5. ^{14}C residues separated by HPLC or (TLC)[1], in apples and apple leaves after treatment of apple trees with [^{14}C]phosalone (Kimmel *et at.*, 1990).

Sample, residue	^{14}C of residue as % of ^{14}C in each sample		Residues, mg/kg	
	Trial 1[2]	Trial 2[2]	Trial 1	Trial 2
Leaf rinse				
Phosalone (I)	95.9	91.2	490	413
Phosalone oxon (II)	1.3	2.5	6.7	11.3
Aglycone (VII)	0.5	0.5	2.5	2.1
Unknowns	1.9	5.0	9.8	22.4
Polar products	0.3	0.9	1.7	4.0
Leaf extract				
Phosalone	(87.5)	78.8	(329)	230
Phosalone oxon	--	1.6	---	4.6
Aglycone	(6.9)[3]	2.3	(25.8)[3]	6.8
Unknowns		4.4	---	12.8
Polar Products	(5.7)	12.9	(21.2)	37.6
Peel extract				
Phosalone	(91.7)	93.8	(67.1)	52.6
Unknowns	(2.3)	1.0	(1.7)	0.6
Polar Products	(6.0)	5.2	(4.4)	2.9
Pulp extract				
Phosalone	Not analysed	(51.1)	Not analysed	(0.42)
Unknowns		(16.3)		(0.14)
Polar Products (at TLC origin)		(32.6)		(0.27)

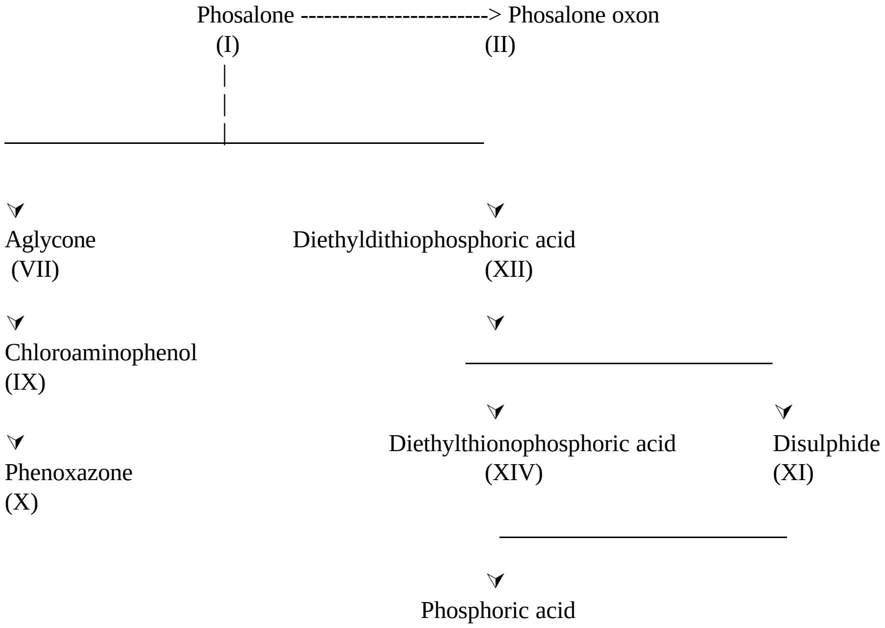
[1] TLC analyses in parentheses. Included only if there were no HPLC analyses. [2] Trials described in footnotes to Table 4

[3] TLC region for phosalone oxon and 6-chlorobenzoxazolone which were not separated

Environmental fate in soil

Several studies have reported the fate of phosalone in soil. The available information shows similar degradation patterns to those in plants and animals, although the diethyldithiophosphoric acid (XII) appears to be converted to the disulphide (XI) only in soil (Demoras *et al.*, 1968). Degradation is similar under aerobic and anaerobic conditions and soil microbes play a major role in the degradation. The microbes' function does not appear to be significantly affected. Half-lives of phosalone are typically about 2 days, varying according to the conditions.

The proposed degradation in soil is as follows.



The degradation of the oxon is similar, except that hydrolysis yields the diethylthiolophosphoric acid (XIII) which would be isomerized to the thiono acid (XIV).

The degradation of phosalone has been investigated in sterile and normal Beauce soil with powder formulations, applied at an equivalent of 3 or 6 kg/ha incorporated into the top 2 cm (Demoras *et al.*, 1966). The residues in dry soil after application at 6 kg/ha decreased rapidly:

Day	1	7	14	21
Residue, mg/kg	8.8	3.3	2.1	1

From these and other data the half-life was 1 to 2 days under the experimental conditions (22 ± 4°C). Residues in the 2 cm layer below the top 2 cm were <0.5 mg/kg.

Analysis of soils with initial phosalone concentrations of 20 mg/kg by a colorimetric analytical procedure showed a rapid decrease of residues in normal soil and a slow decrease in sterile soil, indicating the importance of soil micro-organisms in the degradation of phosalone (Table 6).

Table 6. Dissipation of phosalone residues in sterile and normal soils (Demoras *et al.*, 1966).

Soil	Residue, mg/kg, at interval (days) between treatment and analysis		
	1	7	14
Sterile	18.5	17	16
Normal	12.5	2.2	0.6

The major unidentified degradation product appeared to be a phenoxazone by IR analysis, and was postulated to result from degradation of the aglycone VII to the chloroaminophenol IX, then conversion to the phenoxazone X.

In another study a 10% preparation of phosalone on talc was incorporated into a 2 cm layer of soil and residues were measured at intervals (Demoras *et al.*, 1967, 1968). The authors presented a detailed discussion of possible degradation routes (desethylation, oxidation and hydrolysis) and compared this to their findings. Desethylation under natural conditions was considered improbable, a view supported by the lack of reports of desethyl residues having been detected in soil or plants. Hydrolysis and oxidation were shown to occur in laboratory experiments. The residues of toxicological concern were considered to be phosalone itself and its oxon which was found in small quantities (<10% of the residue), and which is degraded even more rapidly than phosalone. The authors also reported formation of the phenoxazone from the benzoxazolone. They noted uptake by plants and the formation of glycosides, but the degradation products were not expected to accumulate significantly in soil or plants. The major compounds found in soil 7 days after treatment at a rate of 30 mg phosalone/kg were as follows.

	mg/kg
Phosalone (I)	16
Disulphide (XI)	1.2
Phosalone oxon (II)	1.0
Phenoxazone (X)	0.8
Diethyldithiophosphoric acid (XII)	0.6
Diethylthionophosphoric acid (XIV)	0.4

Similar results, shown below, were reported with non-sterile soil in a later study with ^{14}C-labelled phosalone where the phenoxazone (X) was also identified by IR analysis (Demoras and Ambrosi, 1975).

	% of total radioactive residue at interval (days)				
	1	3	7	10	17
phosalone	80.8	36.1	4.2	2.1	<0.2
phenoxazone (X)	2.3	20.3	13.3	5.9	2.0
polar extractable radioactivity	4.4	7.5	14.6	22.1	19.2
unextracted radioactivity	12.4	36.1	67.9	69.9	78.7

Again, the half-life of phosalone under the glasshouse conditions (18-22°C, light 5000 lux) was about 2 days and that of the phenoxazone 4-5 days, while polar materials and unextracted radioactivity continued to increase with time. Results similar to these were reported for flooded and moist Matapeake loam and Monmouth fine sandy soils, where the half-life ranged from 3 to 7 days (Ambrosi *et al.*, 1977). Phosalone and the phenoxazone were confirmed by MS. Measurement of evolved CO_2 indicated less than 10% cleavage of the benzoxazolone ring.

The degradation of phosalone by soil microbes and physico-chemical processes during 30 days and the potential for effects on microbial function were also investigated in loam and loamy sand soils in flasks under dark aerobic, anaerobic and sterile conditions (Spare and Isbister, 1979). The soils were fortified with [^{14}C]phosalone at 10 mg/kg on a dry weight basis.

No significant change in microbial function that could be attributed to the presence of phosalone was observed under any of the conditions, as determined by the measurement of nitrification, nitrogen fixation, starch and cellulose degradation, and protein degradation (mineralization). For characterization of the products by TLC soil was extracted with acetone/water (80:20), the acetone was evaporated and the aqueous phase partitioned successively with dichloromethane and ethyl acetate. Reference standards included phosalone (I), its oxon (II) the glycoside VIII, the aglycone VII, the aminophenol IX and the phenoxazone X. Identifications were by TLC in two solvent systems.

No ^{14}C volatiles were detected and <0.4% of the ^{14}C was recovered as CO_2 under either aerobic or anaerobic conditions. The bound residues as a proportion of the dose at the beginning and end of the experiment were as follows.

California loam				New Jersey loamy sand			
aerobic		anaerobic		aerobic		anaerobic	
1 day	30 days	1 day	30 days	3 days	30 days	3 days	30 days
2.1%	41%	28%	48%	70.6%	79%	60%	91%

The ^{14}C in individual residues after 30 days as a proportion of the initial ^{14}C is shown in Table 7.

Table 7. Distribution of ^{14}C residues extracted from soil after 30 days as a percentage of applied ^{14}C, as found by TLC (Spare and Isbister, 1979).

Compound or fraction	California loam			New Jersey loamy sand		
	Aerobic	Anaerobic	Sterile	Aerobic	Anaerobic	Sterile
phosalone	17.6	39.4	46.7	4.3	3.7	43.5
phosalone oxon	1.8	2.9	---	0.64	0.96	2.9
aglycone	0.28	0.54	---	0.71	1.28	3.9
phenoxazone	Traces reported under some experimental conditions					
chloroaminophenol	0.15	0.29	---	0.11	0.21	0.45
glycoside	---	0.1	---	---	0.03	---
polar	0.05	0.16	0.02	0.04	0.1	0.01
other unknown	0.1	---	---	0.03	0.15	0.16
remainder	0.44	0.71	1.5	0.37	0.5	0.74
Total	20.4	44.1	48.2	6.2	6.9	51.7
Unextracted (by difference)	79.6	55.9	51.8	93	93.1	48.3

None of the identified degradation products except the oxon were measurable until the 30th day in the non-sterile California loam (aerobic or anaerobic) soil. In the New Jersey loamy sand only the aglycone was measurable before thirty days.

Environmental fate in water/sediment systems

Hydrolysis

The solubility and stability of phosalone in water at pH 5, 7 and 9 have been investigated (Laurent and Buys, 1975). The solubility was determined by suspending 50 g phosalone in 500 ml water, stirring, heating to 50°C and cooling to 20°C or agitating 1 g/100 ml water with ultra-sound. Phosalone was determined in dichloromethane extracts of the filtered solutions by GLC. The solubility was estimated to be about 1.7 mg/kg at 20°C.

Little degradation (<10%) was observed during 28 days at pH 5 or 7 at 20°C. At pH 9 degradation was more pronounced, giving a half-life of about 9 days. Over 15 degradation products were poorly resolved by TLC and only three were identified. Samples were hydrolysed in aqueous methanol in order to obtain sufficient material for characterization. The two major identified products were the aglycone VII and the phenoxazone X, both identified by IR and/or co-chromatography. Phosalone oxon was the third identified compound and was one of only two of the products which was reported to exhibit anticholinesterase properties; the other was found only at trace levels and could not be identified.

Sediment systems

No information was provided.

METHODS OF RESIDUE ANALYSIS

A number of analytical methods are available for the determination of phosalone and/or its metabolites in various matrices. Methods for phosalone and its oxon are similar, but there are significant differences according to the matrix. Methods reported to the Meeting include a number of general "enforcement" methods (cited but not provided), as well as separate methods for the determination of phosalone in plants, animals, soil and water and in the technical grade product. The cited enforcement methods are listed in the references under "Enforcement, 1994 JMPR".

The method for determining phosalone in the technical material is based on GLC with thermal conductivity detection and with di(2-ethylhexyl) sebacate as an internal standard. At 238°C the retention times on a 10% silicone SE 30 column is 2.8 min for phosalone and 6.3 minutes for the internal standard (Charbassol, 1988).

For convenience, the more general methods are described first, followed by those intended specifically for plants, animals, soil and water.

Plants, animals and soil

A general method has been described for the determination of phosalone *per se* in a variety of crop matrices and soil (Demoras and Laurent, 1980). Plants, milk, fatty crops and oil are extracted with acetone, citrus fruits with pentane and/or ethyl acetate, and cereal grains with dichloromethane/methanol (93:7). A 7.6 pH buffer is added for extraction of plants, and dry ice for milk, meat and fatty crops and oils. Extracts are concentrated and partitioned with dichloromethane (crops generally, milk, soil and cereal grains) or acetonitrile/hexane (meat, citrus, fatty crops and oils) and dried with sodium sulphate.

If further clean-up is necessary the samples are taken to dryness, dissolved in dichloromethane and chromatographed on a Florisil column with 95:5 v/v dichloromethane/ethyl acetate. The eluate is taken to dryness, redissolved and analysed by GLC with EC or thermionic detection. Identities can be confirmed by TLC on silica gel. The limit of "detection" was reported to be 0.01-0.02 mg/kg for most crops, 0.05 mg/kg for rape seed, olive oil and citrus, and 0.03 mg/kg for meat and milk. Information was not provided to allow the independent confirmation of detection limits or limits of determination (no control values, actual recovery data or sample chromatograms).

Plants

An improved version of "Rhodia Method 54-A" has been described for the determination of phosalone and its oxygen analogue in raw agricultural commodities (Guardigli, 1974). Samples are ground or chopped (ground with dry ice and sodium sulphate if they have a high moisture content). Non-oily crops are extracted with acetone and oily crops with acetonitrile.

Clean-up is by liquid-liquid partition and Florisil column chromatography, the partition steps being different for oily and non-oily crops. The acetone extract of non-oily crops is concentrated and partitioned with 10% ethyl acetate in dichloromethane. The organic phase is evaporated for transfer of the residue to a Florisil column. The extract of oily crops is concentrated and partitioned with 2:1 acetonitrile/hexane. After backwashing, the hexane is discarded and the acetonitrile layer partitioned with 10% ethyl acetate in dichloromethane. The ethyl acetate/dichloromethane phase is concentrated to dryness. Residues from both oily and non-oily crops are transferred to a Florisil column with benzene. Phosalone is eluted with 2.5% ethyl acetate in benzene and subsequently the oxon with 2.5% methanol in benzene. Both are determined by GLC on a 3% OV-17 chromatographic column with EC detection.

The authors considered the limits of "detection" to be approximately 0.05 mg/kg for phosalone and 0.1 mg/kg for the oxon. Reported recoveries averaged 94% for phosalone and 89% for the oxon, at fortification levels ranging from 0.05 to 20 mg/kg for phosalone and 0.2 to 0.5 mg/kg for the oxon. Recovery data were provided for a wide variety of crops. Results at the lower levels are summarized below.

Crop	Recovery, %, at fortification level, mg/kg		
	0.2	0.1	0.05
almond hulls	92.7		
almond shells		90	
almond nut-meat	98.7		85
artichoke hearts	93.7		
broccoli		93.7	
cabbage		104	
cauliflower		98.5	
cherries	90.9	102	
lettuce		96.1	
peaches	90.9		
pecan shells	93.5		
pecan nut-meat	93		
potato	93.8	96.4	90
sorghum plants	94.9	108	
sorghum seedheads	97.5		
sorghum straw	93.9		
tomato	94.9		

Fewer results were reported for the oxon: alfalfa plants 84.2% at 0.2 mg/kg; broccoli 84.3% at 0.25 mg/kg; cauliflower heads 85.1% at 0.2 mg/kg; pecan forage (?) 97.6% at 0.2 mg/kg and spinach 86.3-93.6% at 0.5 mg/kg. The Meeting could not independently estimate the limits of detection or determination on the basis of data provided (no control values, no sample chromatograms).

Methods have also been described for the determination of phosalone *per se* specifically in cereals (Laurent and Chabassol, 1981), grapes (Laurent and Chabassol, 1982) and apples (Laurent and Chabassol, 1983). The Meeting could not review the method for cereals because pages were missing from the submission. It appears to be similar to the determination of cereals in the general method described above (Demoras and Laurent, 1980). Grapes are extracted with acetone and after evaporation of the acetone clean-up is by dichloromethane/water partition. After concentration, residues are determined by GLC on a 5% silicone SE 30 column fitted with a thermionic detector. The method submitted for apples was identical to that for grapes. Reported recoveries were about 100% and the reported "limit of detection" about 0.02 mg/kg. No information was provided to allow independent confirmation of the reported recoveries and limits of detection (no sample chromatograms, control values, or actual recovery data).

Animals

Three analytical methods developed for the measurement of phosalone and its metabolites in animal products were used in the animal feeding trials provided. One, a modification of Rhodia Method 54-C (Witkonton *et al.*, 1979, reproduced as Appendix II of Craig *et al.* 1980a), was developed for the determination of phosalone and its oxon. It is similar to Method 54-C (Guardigli, 1973) except that the initial extraction solvent has been changed from ethyl acetate to acetone for milk and to 1:1 acetone/methanol for tissues, and the benzene component of the Florisil chromatographic elution solvent has been replaced. After evaporation of the acetone the aqueous milk sample is partitioned with acetonitrile and hexane, the hexane layer is discarded, and the aqueous acetonitrile concentrated. Tissue extracts are also concentrated and partitioned with ethyl acetate, which in turn is concentrated, partitioned with acetonitrile and hexane, and the hexane discarded. The remaining tissue extract is concentrated. The concentrated milk and tissue aqueous fractions are diluted with a sodium sulphate solution and the residues partitioned into hexane. The hexane fraction is transferred to a Florisil column; phosalone is eluted with 12% ethyl acetate and the oxon with 2:8:90 acetone/methanol/hexane. The final determination is by electron-capture gas chromatography.

After omitting extremely high and low values, the recoveries of phosalone from cow tissues fortified at 0.01 to 0.5 mg/kg ranged from 93 to 120%, and from milk at 0.001 to 0.1 mg/kg levels from 80 to 112%. Recoveries of the oxon from tissues were 70-150% at 0.02 to 0.5 mg/kg fortification levels and those from milk were 68-138% (mean approximately 105%) at 0.01 and 0.1 mg/kg. On the basis of "typical" chromatograms, normal control values and realistic limits of determination of phosalone and its oxon in milk would appear to be as shown below.

Sample	Phosalone, mg/kg		Oxon, mg/kg	
	LOD	Control	LOD	Control
Milk	0.002-0.005	<0.001	0.01-0.02	<0.01
Liver	0.02-0.05	<0.01	0.1	<0.04
Other tissues	0.02	≤0.01	0.1	≤0.02

Samples from the metabolism study on goats were used for validation of the modified Method 54-C (Witkonton *et al.*, 1980). Radioactivity was measured at each step of the procedure. The results for phosalone and its oxon by the two methods were very close except in liver, where the GLC method gave substantially higher results (interference by co-extractives was suspected).

Although at least 85% of the radioactivity in the milk and tissues was extractable, measurement of radioactivity at each step of 54-C during the validation studies (and the TLC studies) indicated that most of it was not from phosalone or its oxon. Additional analytical methods were therefore developed for the determination of other compounds containing the chlorobenzoxazole moiety and of chloroaminophenol (Craig *et al.*, 1980b). One method was for the determination of the free aglycone (chlorobenzoxazolone, VII), chloroaminophenol (IX) and the thiol (III) in milk and a second for the determination of the sulphone VI, its precursors IV and V, and the water-soluble conjugates of chlorobenzoxazolone in milk and tissues.

For the determination of unconjugated chlorobenzoxazolone, chloroaminophenol and the thiol in milk extraction and acetylation are combined by refluxing with acetic anhydride and ethyl acetate in a Soxhlet extractor. The extract is concentrated and the acetylated residues extracted with hexane for determination by comparison with the individually acetylated standards. Recoveries/controls at 0.05-0.1 mg/kg levels were: chlorobenzoxazolone 67-112%/<0.01mg/kg; chloroaminophenol 52-61%/<0.02 mg/kg and the thiol 67-100%/<0.05 mg/kg. On the basis of typical chromatograms a reasonable limit of determination would appear to be approximately 0.1 mg/kg for each of the free metabolites.

For the determination of compounds IV-VI and the chlorobenzoxazolone conjugates milk is extracted with acetone and tissues with 1:1 acetone methanol. The organic solvents are evaporated from both extracts and the remaining aqueous solution is partitioned with ethyl acetate. The aqueous fraction contains the water-soluble conjugates of chlorobenzoxazolone and the ethyl acetate fraction the sulphone and its precursors. The conjugates are hydrolysed to benzoxazolone by refluxing with HCl, the benzoxazolone is extracted with ethyl acetate and the concentrated extract is refluxed with acetic anhydride to form the acetyl derivative. This is determined by electron-capture gas chromatography, using acetylated chlorobenzoxazolone as a standard. Recoveries from milk averaged 102% at 0.01 and 0.1 mg/kg levels (control <0.005 mg/kg). Recoveries from tissues at 0.1 mg/kg ranged from 55-69% for fat to 63-90% for liver (average for all tissues 73%, controls <0.01 mg/kg).

The ethyl acetate fraction from the original partition is used to determine the sulphone and its precursors. For tissues this is further partitioned with acetonitrile/hexane. The acetonitrile or ethyl acetate is evaporated and the residues oxidized to the sulphone (VI) by reaction with peracetic acid. The sulphone is determined by electron-capture GLC. Average recoveries at 0.01-0.1 mg/kg levels were 97% for milk and ranged from 67% (in brain) to 83% in tissues (controls were ≤0.02 mg/kg).

Typical chromatograms indicated that reasonable limits of determination would be approximately as follows.

Sample	Sulphone, mg/kg		chlorobenzoxalone conjugates, mg/kg	
	LOD	Controls	LOD	Controls
Tissues	0.05-0.1	≤0.02	0.02-0.05	<0.01
Milk	0.05	<0.005	0.01-0.02	<0.005

The sulphone determination was also validated with samples from the goat metabolism study. The results by GLC agreed reasonably well with the measurements of ^{14}C in TLC spots. Expressed as mg/kg by GLC/TLC they were: milk 0.03/0.017; muscle 0.014/0.025; kidney 0.3/0.3; liver 5.1/4.5. Analyses for conjugates were not compared.

Soil

In a method for the determination of phosalone and its major degradation product in soil, the phenoxazone X (Macchia and Piznik, 1976), air-dried soil is extracted with 1:1:2 benzene/ethyl acetate/acetone, and the filter cake is extracted with methanol. The combined extracts are concentrated and cleaned up on an automated gel-permeation chromatographic system using Bio-beads SX-3 and elution with benzene. Separate aliquots are used for the determination of phosalone by GLC with an ECD and the phenoxazone by TLC. Analytical recoveries of phosalone were 83-90% for sandy loam, silty loam and organic loam soils fortified at 0.5 mg/kg and 80-83% for phenoxazone in these soils fortified at 0.1 mg/kg.

Water

A method has been reported for the determination of phosalone in water (Maycey and Outram, 1988). It involves hexane extraction and elution from a Florisil "Sep-Pak" column with 15% ethyl acetate in hexane. The eluate is taken to dryness and the phosalone determined by EC GLC. Analytical recoveries of approximately 100% are reported at fortification levels of 0.1-10 ìg/l with controls of ≤0.02 ìg/l.

Stability of pesticide residues in stored analytical samples

The Meeting was informed that no information was available (Chabrol, 1994).

USE PATTERN

Summarized information on GAP and labels for numerous crops were provided to the Meeting. The information which is relevant to the available residue data is shown in Tables 8 to 10, which refer to vegetables, fruits, and cereals, oilseeds, nuts and tea respectively.

Table 8. Nationally approved and registered uses of phosalone on vegetables.

Crop	Country	Application			PHI, days	Notes
		Form.	Rate, kg ai/ha (g ai/hl)	No.		
Beet	France	EC	0.5	*	15	* Not specified
		WP	0.45	*	15	
Cabbage	Denmark	Flo (=SC?)	(250)	1	-	
	France	Flo	0.6	?	15	
	India	EC	0.4	-	-	
	Romania	EC	(50-70)	-	-	
Cauliflower	India	EC	0.6	-	-	
Brussels sprouts	Netherlands	SC	0.6	4	14	
Cucumber	Japan	EC	(35-50)	2	3	
	Portugal	WP	2 (200)	2-3	21	
Lettuce	France	Flo	0.6		15	
Melons (same for watermelon)	Portugal	WP	2 (200)	2-3	21	Last treatment at 3 wk. growth stage
	Denmark	Flo	1 (250)			
Potato	Austria	EC	0.5-0.7	-	21	
	Denmark	Flo	1 (250)	-	-	
	France	EC	0.6	-	15	
		Flo	0.6	-	15	
		WP	0.6	-	15	
	Greece	WP	0.6	2	15	
		EC	0.5-0.7	2	21	
	Italy	EC	0.3 (60)	2	21	
		WP	0.3 (60)	2	21	
(ware and starch)	Netherlands	SC	0.45	1	28	
(ware, starch & seed)		SC	0.6	1	28	
	Poland	EC	0.5-0.7	-	-	
	Portugal	WP	2	2-3	21	
	Romania	EC	0.7	-	-	
Spinach	Denmark	Flo	(1500)	-	-	
Tomato	India	EC	0.6	*	*	*Not specified
	Portugal	WP	2 (200)	*	21	*At 10-12-day intervals
	Thailand	EC	(200)	-	14	
Vegetables	China	EC	0.6-1 (200-280)	2	10	
	Greece	WP	(45-60)	1-2	15	

Crop	Country	Application			PHI, days	Notes
		Form.	Rate, kg ai/ha (g ai/hl)	No.		
		EC	(53-62)	1-2	21	
	Italy	EC & WP	0.3-0.5 (60)	1-2	21	
	Spain	EC	(53-70)	-	7*	*Artichokes, green beans, tomatoes
					15**	**Other vegetables
Watermelon	Portugal	WP	2 (200)	2-3	21	

Table 9. Nationally approved and registered uses of phosalone on fruits.

Crop	Country	Application			PHI, days	Notes
		Form.	Rate, kg ai/ha (g ai/hl)	No.		
Grapes	Austria	EC	(52-70)	-	21	
	Canada	WP & SC	1 (30-60)	≥1	14	
	France	EC	0.6	-	15	
		Flo (=SC?)	0.6	-	15	
		WP	0.6	-	15	
	Germany	WP	(60)	2	*	*Last treatment at full flowering. PHI depends on growth
	Italy	EC & WP	0.6 (60)	2	21	Last treatment in August
	Spain	WP	(60)	2	15	
Pome fruit	Germany	WP	0.9 (60)	4	42	Apply in summer at flowering, beginning of attack
		EC	0.8-1 (0.05-0.07)	4	42	
Apples	Canada	WP	1-1.15 (30-45)	-	1	
		SC	1-1.15 (30-62)	-	1	
	Denmark	Flo (= SC?)	1-1.3 (250-312)	-	28	
	France	EC	0.65 (60)	-	15	
		WP	(60)	-	15	
	Italy	EC & WP	0.6-1 (60)	1	21	
	Netherlands	SC	0.72 (60)	1	*	*Last application June 15
		EC	0.6-0.9 (60)	1		
(deciduous fruit)	Spain	EC	(53-70)	-	15	
(fruit)		PM (= WP?)	(60)	-	15	
(pip fruit)	Switzerland	EC	0.8-1.1 (52)	-	30	
Pears	Canada	WP	1-1.5 (30-45)	-	7	
		EC	1-1.5 (30-62)	-	7	

Crop	Country	Application			PHI, days	Notes
		Form.	Rate, kg ai/ha (g ai/hl)	No.		
	Denmark	Flo	1-1.3 (250-312)	-	28	
	Italy	EC & WP	0.6-1 (60)	1	21	
	Netherlands	SC	0.72 (60)	1	*	*Last application June 15
		EC	0.6-0.72 (60)			
(see Apples)	Spain					
(see Apples)	Switzerland					
Citrus fruit	Japan	EC	(35-50)	2	100	
	Spain	WP	(60)	-	15	
	Thailand	EC	(150-200)	-	14	
Stone fruit	Austria	EC	(50-70)	-	21	(fruits)
	France	Flo	(60)	-	15	
	Italy	EC	0.6-0.8 (60)	2-4	21	
		WP	0.6-0.8 (60)	2-3	21	
(fruit trees)	Spain	WP	(50)	-	15	
	Switzerland	EC	0.8-1.1 (52)	-	30	
Cherry	Canada	WP	1-1.5 (30-45)	-	7	
		SC	1-1.5 (30-45)	≥1	7	
	Denmark	Flo (=SC?)	1-1.3 (250-312)	-	28	
	Germany	WP	(60)	2	35	Last treatment at full flowering
		EC	2.3-3 (7-52)	2	35	Last treatment at full flowering
Peach	Canada	WP & SC	1-1.5 (30-45)	≥1	7	
	Portugal	WP	2 (200)	2-3	21	
Plums	Canada	WP	1-1.5 (30-60)	≥1	14	(plums and prunes) Thorough tree coverage
		SC	1-1.5 (30-60)	≥1	14	
	France	Flo (=SC?)	(70)	-	-	
	Germany	WP	(60)	2	35	Last treatment at full flowering
		EC	2.3-3 (7-52)	2	35	Last treatment at full flowering
	Portugal	WP	2 (200)	2-3	21	
	UK	EC	0.7 (33-340)	3	21	
Berries						
Raspberries	Austria	EC	(52-70)	-	21	(fruit)
(small fruit)	Poland	EC	(40-60)	-	-	
	Switzerland	EC	0.8-1 (52)	-	21	
Strawberries	Austria					(see raspberries)
(see small fruit)	Poland					

Crop	Country	Application			PHI, days	Notes
		Form.	Rate, kg ai/ha (g ai/hl)	No.		
	CSFR	EC	0.7	-	-	

Table 10. Nationally approved or registered uses of phosalone on cereals, nuts, oilseed and tea.

Crop	Country	Application			PHI, days	Notes
		Form.	Rate, kg ai/ha (g ai/hl)	No.		
Nuts						
Almonds	France	Flo	(60-75)			
Chestnuts	France	Flo	(60)			
Hazelnut	France	Flo	(60)			
	Turkey	EC	(70)			
Walnut	France	Flo	(60)			
Pistachio	Turkey	EC	(70)	2-3	15	
		WP	(60)	1	15	
Oilseed rape	CSFR	EC	0.7			
(Brassica oil crops)	France	EC	1.1-1.2			
		Flo	0.6-1		15	
		WP	1.1-1.2			
	Germany	EC	1.1 (60)	2	28	Last treatment at full flowering
	Hungary	EC	0.6			
	Netherlands	SC	1.3	1		Last treatment in May, mid-flowering
	Poland	EC	0.5-1.2			
	Sweden	Flo	1 (500?)			
(oilseed)	Switzerland	EC	0.5-1		21	
	UK	EC	0.7	3	21	End of flowering
Cereals	Bulgaria	EC	(70)			
	Denmark	Flo	0.8-1 (375-500)			
	France	Flo	0.6		15	
(Rice)	India	EC	0.4-0.6			
(Sorghum, wheat)	India	EC	0.4			
	Italy	EC	0.5 (60)		21	Last treatment in May
	Morocco	EC	(40-50)			
	Norway	Flo	1 (500?)			
	Spain (winter cereals &	WP	(60)			

Crop	Country	Application			PHI, days	Notes
		Form.	Rate, kg ai/ha (g ai/hl)	No.		
	maize)					
	Sweden	Flo	1 (500?)			
	Switzerland	EC	0.5-0.7		21	
Tea	India	EC	0.3-0.8		*	*No PHI cited (label almost illegible) but 1970-71 residue reports (ref. 55) state that a 10-day interval complied with agricultural practice at that time
	Japan	EC	(35-50)	1	7	
	Taiwan	EC	0.4-0.5 (35-133)	*	21	Treat shoots at 10-14 day intervals

RESIDUES RESULTING FROM SUPERVISED TRIALS

Data were available from supervised trials on a variety of vegetables, fruits, grains, nuts, oilseed rape, hops and tea, and from feeding trials on cows. Underlined residues in Tables 11-21 are from trials according to GAP.

Table No.	Commodities
11.	Vegetables (artichokes, beans, beets, broccoli, cabbage, cauliflower, Brussels sprouts, cucumbers, lettuce, melons, potatoes, spinach, tomatoes)
12.	Apples
13.	Pears
14.	Stone fruit (apricots, cherries, peaches, nectarines, plums, dry prunes)
15.	Grapes
16.	Oranges
17.	Lemons and grapefruit
18.	Strawberries and raspberries
19.	Nuts
20.	Cereals and oilseed rape
21.	Cotton and hops
22.	Tea
23.	Phosalone and its oxon in cow tissues
24.	Total phosalone sulphone metabolite residues in cow tissues
25.	Phosalone and its oxon in cow milk
26.	Total sulphone, free and conjugated chlorobenzoxazolone, chloroaminophenol and thiol in cow milk

Residues in plants

Vegetables (Table 11)

Artichokes. Data were available from two supervised trials in 1968 in France and one 1972-3 trial in the United States. No information was provided on GAP for artichokes in France, although GAP for "vegetables" was available for Italy and Spain.

Beans. Data were available from two 1968 supervised trials on French beans in France and summary data from one 1965 trial in the USA. No relevant information on GAP was provided except for "vegetables" in other countries.

Beets. Data were available for two 1968 supervised trials in France. French GAP requires 0.5 kg ai/ha and a 15-day PHI. The residues at approximately the GAP application rate and PHI were 0.1 and 0.2 mg/kg.

Broccoli. Substantial summarized data were available from supervised trials in the USA in 1964-5 and 1972-3 with multiple applications ranging from 1.1 to 36 kg ai/ha. Residues ranged from <0.05 to 3.9 mg/kg after 21 days. No relevant information on GAP was provided.

Brussels sprouts. Data were available from one trial in the USA (summarized) and 5 trials in The Netherlands. Maximum residues were 0.6 mg/kg in the latter at the GAP application rate and PHI, from WP formulations. The information on GAP referred to SC uses.

Cabbage. Summaries were available from supervised trials in 1964-5 and 1972-3 in the USA with multiple applications at two or more times the registered rate in France, the only other country for which limited data were available. No GAP was provided for the USA. The highest residue in the French trial approximating GAP (0.6 kg/ha, 15-day PHI) was 1.2 mg/kg and in the US trials 0.07 mg/kg after 14 days from twice the French GAP rate.

Cauliflower. Summaries were available from six supervised trials in 1972-3 in the USA and two in 1973 in France. No information on GAP was available for these countries.

Cucumbers. Summaries were available from 7 supervised trials in the USA and data from two trials in France (both showing 0.02 mg/kg after 14 days). Information on GAP was available for Portugal and Japan.

Lettuce. Data were available from 9 supervised trials in the USA and 6 in France but only 2 of the French trials, which were according to GAP, were fully reported. These showed residues of 0.6 and 0.7 mg/kg. No relevant US GAP was provided.

Melons and watermelon. Limited trials data were available from France and the USA, together with information on Portuguese GAP.

Potatoes. Data were available from 10 trials in Canada and 2 in France and summary data from 22 in the USA. French GAP requires a 0.6 kg ai/ha application rate and a PHI of 15 days. GAP was not reported for Canada or the USA. In all trials residues at PHIs of ≤15 days from application rates at or exceeding the French GAP rate were below the current CXL of 0.1 mg/kg. This level was exceeded in only one US trial, from an application rate 10 times that which is GAP in France.

Spinach. Summary data were available from 3 supervised trials in the USA. Information on GAP specifically for spinach was known only for Denmark, although GAP for "vegetables" was available from other countries.

Tomatoes. Results of supervised trials in 1972-3 in the USA and 1968-73 in France and a published report on four 1978-9 trials in India were available. Residues in the Indian trials were 0.3 to 0.5 mg/kg 7 days after applications approximating the Indian GAP rate of 0.6 kg ai/ha, but the Indian PHI was not known. The application rates in the four French trials, 0.4 to 0.7 kg ai/ha (40-70 g ai/hl), are comparable to Spanish and Italian GAP rates for tomatoes and vegetables respectively, but there were only two results

(0.2 and 0.3 mg/kg) at the shorter (Spanish) PHI of 7 days.

Table 11. Residues of phosalone on vegetables resulting from supervised trials. All US trials were reported as summaries only.

Crop, Country, State, Year (variety)	Application			Residues, mg/kg, at intervals (days) after last application								Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)									
							0	4	7	14	17	
Artichokes				Notes								
France 1968	EC	1	0.35 (60)	a			2.1	1.3				1,2,3
		2	0.9 (40+80)	a			4.5	1.9				1,2,3
												5
1973	EC	1	0.34 (65)	b							0.07	5
			0.52 (130)	b							0.13	
											Control <0.01	
					1	3	7	10	14	21		
USA 1972-3	EC	6	2.2 (2400) (aerial)	c	5.7	5.5	3.1	3	2.4	0.9		4
Beans					0	2	6	13	49			
USA (French	EC	1	0.6	d					<0.1			3,7,8
beans, seed) 1978			(60)					Control	<0.1			
France (green												1,2,3
haricot, bean+pod)	EC	1	0.4(40)	a,d	1.8	0.7	0.3	0.09				
1968		1	0.6(60)	a,d	2.5	0.8	0.4	0.2				
Beets (edible)					0	3		7		14		
France 1968	EC	1	0.4	a,d	0.3	0.3		0.1		0.1		1,2,3
			0.6	a,d	1	0.8		0.2		0.2		
Broccoli				0	1	3	5	7	14	21		
USA NY 1964	EC	2	(30)	6.1 3.6	1.8	<0.2						3,7,8
			(60)	6.3 5.8	2.7	0.6						
			(120)	16.1	8.5	5.5	5.3	EC determination				
								Controls ≤0.05				
TX 1972-3	EC	5	1.1 (2400)	0.24		0.06		0.06	<0.05	<0.05		4
			2.2 (4800)	1.4	2.1		0.5	0.07	0.09			
			4.5 (9600)	4.6	4.5		2.3	0.3	0.1			
OR 1972-3	EC	5	1.1 (100)	3.8 3.8	1.7		0.8	0.06	<0.05			
			2.2 (200)	8.4 7.9	1.8		1.2	0.14	<0.05			
			4.5 (400)	14	9.2	7		5.4	2.8	0.4		
SC 1972-3	EC	6	9 (1925)	43		31		4.5	3.6	1.9		
			18 (3850)	204		83		80	30	2.4		
			36 (7700)	281		242		110	36	4		
TX 1972-3	EC	5	1.1 (2400)	13		8.2		3.2	0.5	<0.05		
			2.2 (4800)	19		14		5.3	0.7	<0.05		
			4.5 (9600)	45		40		9.7	5.5	<0.05		
Brussels sprouts					0		3	4	7	14		
USA, NY 1965	EC	6	(60)		2.1				1.2	0.7		3,7
					Controls <0.1		EC GLC determination					
Netherlands 1970	WP	4	0.6		0.6			0.4	0.3	0.4		3
	WP	3	0.6		0.8		1.2		0.7	0.2		
1970	WP	3	0.6	a	0.9 (1 d.)					0.6		9
	WP	4	0.6	a	0.8					0.6		
	WP	4	0.6	a						0.2		
				Controls ≤0.1								
Cabbage												3,7,8
USA WI 1964	EC	1	(60-120)		<0.2 mg/kg at 68 days							
1965	EC	1	(60)		<0.1 mg/kg at 1, 8, 62 days							

Crop, Country, State, Year (variety)	Application			Residues, mg/kg, at intervals (days) after last application								Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)									
			(120)	<0.1 mg/kg at 0, 7, 50 days								
Cabbage				0		1	3 5	7	14	21		
USA, AZ 1965	EC	5	(120)	1.4				0.9		<0.1		3,7,8
AZ 1972-3											(26 d.)	
	EC	5	1.1(340)	0.7		0.5		1.2?	0.7	0.2		4
	EC	5	2.5(680)	0.4?			1.1		2.1	2.1	1.1	
	EC	3	4.5 (1360)	8.2		4.2		4.4	2	1.8		
OR 1972-3d	EC	5	1.5(100)	8.6	8.3	0.9		0.7	0.6	0.05		
	EC	5	2.5(200)	10		15?	1.8		0.8	0.7	0.1	
	EC	5	4.5(400)	13		16?	7.7		3.7	2.8	0.2	
						0		2	6	13	21	
France Green	EC	1	0.4	a		4.7		0.8	0.5	0.3		1,3
cabbage 1968			0.6	a		7.2		2	1.2	1.2		
Sauerkraut 1972 (P. Blaesheim)	liq.	2-4	0.5-0.8 (120-200)	≤0.02 mg/kg at 67-89 days								3
Germany (Sauerkraut) 1973	WP	1	0.36(60)								0.04	6
										Control	≤0.02	
Cauliflower				0		1		3	7	14	21	
USA, OR 1972-3	EC	5	1.1(100)	5.1	3.9		0.8	0.08	0.05	<0.05		
			2.2(200)	8.9	8.1		1.2	0.3	0.08	0.05		4
			4.5(400)	9.9	9.4		2.3	0.5	0.1	<0.05		
WI 1972-3												
	EC	6	1.1(120)	0.5			0.1	0.07	<0.05	<0.05		
			2.2(240)	0.3			2?	0.4	<0.05	<0.05		
France 1973			4.5(480)	0.3?				3.6	2	<0.05	<0.05	
				EC-GLC determination, LOD 0.05 mg/kg								
	EC	2	0.7(70)								0.1 (20 days)	
	EC	1	0.65							0.06 (16 days)		
Cucumber				0		1	2	3	7	14	28	
USA, MD 1965	EC	3	(60)	0.5	0.2		0.3	<0.1	<0.1	<0.1		3,7,8
				Controls <0.1			EC GLC determination			(13 days)		
												4
AR 1972-3	SE	4	1.1(600)	0.2		0.07		<0.05				
			3.3 (1800)	0.3		0.12		0.1				
SC 1972-3	SE	4	1.1(240)	1.1			0.9	0.2	0.05	<0.05		4
			3.3(720)	2.2			1.4	0.9	0.4	0.12		
				EC-GLC determination, LOD 0.05 mg/kg								
USA CA 1972-3	SE	4	1.1(300)	2.4			0.8	0.2	<0.05	<0.05		4
			3.3(900)	9.8			7.2	3.7	(16 days)	0.3		
				EC GLC determination, LOD 0.05 mg/kg								
					0		4		8		14	
France 1968	WP	1	0.6(30)	a	0.2		0.1		0.02		0.02	1,2,3
			1.2(60)	a	0.4		0.12		0.03		0.02	
				GLC EC determination. Results corrected for 80% recovery								
Lettuce					0		7		26			
USA, AZ 1965	spray	5	(120)		7.1		2.8		0.6			7,8
				Controls <0.1 mg/kg, corrected for 84% recovery								
FLA 1972-3				0		7		10	14	21	28	
	SE	9	0.3(30)	2.5	0.2		<0.05	<0.05	<0.05			4
			1.7(170)	27		0.9		0.1	0.08	<0.05		

Crop, Country, State, Year (variety)	Application			Residues, mg/kg, at intervals (days) after last application							Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)								
			3.4(340)	87	1.9		0.2	0.3	0.2		
				EC GLC determination, LOD 0.05 mg/kg							
CA 1972-3	SE	3	0.6(150)	11	4			0.4	<0.05	<0.05	4
			1.7(450)	58	8.6			2.5	0.05	<0.05	
					<0.1 oxon			<0.1 oxon			
			3.3(900)	172	9.6			3	0.1	0.1	
					0.2 oxon			<0..1 oxon			
				EC GLC determination, LOD 0.05 mg/kg phosalone 0.1 mg/kg oxon							
				0	5		10	13	20		
CA 1972-3	SE	3	1.1(600)	11	7.7		2.9	1	0.5		4
			3.3 (1800)	45	39		21	7	4.9		
							0.2 oxon	<0.1 oxon	<0.1 oxon		
				EC GLC determination, LOD 0.05 mg/kg phosalone 0.1 mg/kg oxon							
					0		2	6	13		
France 1968	spray	1	0.4(40)	a	10		3.1	2.1	0.6		1,2,3
(Reine de Mai)			0.6(60)	a	12		5.3	3.2	0.7		
				EC GLC, corrected for recovery							
					28		35	39	57		
1972 (Mildura)	EC	1	0.5(50)	d	0.04						
(Aurelia)			0.3(50)	d				0.03			3
(Aurelia)			0.3(50)	d			0.03				
(v. unkwn.)			0.7(75)						≤0.02		
				GLC, thermionic detector, >90% recovery							
Melons											
Cantaloupe				0	1		2	4	7	15	
France 1968	WP	1	0.3(30)	0.06				0.08	<0.05	<0.05	1,2,3
			0.6(60)	0.2			0.09	<0.05	<0.05		
				Control <0.05, GLC determination (corrected for >90% recovery)							
USA, AR 1972-3	SE	4	1.1(600)	<0.05			0.06		<0.05		4
(v. golden b.)			3.3 (1800)	0.4		0.08		0.07			
				EC GLC determination, LOD 0.05 mg/kg							
Watermelon											
USA, AR 1972-3	SE	4	1.1(600)	<0.05			<0.05		<0.05		4
			3.3 (1800)	<0.05			<0.05		<0.05		
				EC GLC determination, LOD 0.05 mg/kg							
Potatoes											
France, 1968	EC	1	0.4,0.6 (40,60)	a <0.05 mg/kg 7 days after application							1,2,3
USA, FL 1971	EC	5	0.6 or 2.2	<0.03 mg/kg at intervals 0 to 28 days <0.03 mg/kg controls, EC GLC determination							3,4,7,8
				0	3	7/9	14/16	17/21	26/30	44	
OH 1971	EC	5	1.1	≤0.03 all intervals to 44 days							
			5.6	<0.06		0.11	0.13		0.04	<0.03	
				Controls <0.03							
MI 1971	EC	5	1.1 or 5.6	0.03 all intervals to 34 days, controls <0.03							
MI 1971	EC	5	0.6, 2.3 or 5.6	0.03 all intervals to 26 days, controls <0.03							
WA 1971	EC	4	2.3 or 5.6	<0.03 all intervals to 34 days							

Crop, Country, State, Year (variety)	Application			Residues, mg/kg, at intervals (days) after last application							Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)								
				0	7	17	21	30/34	50-55	62	
WI 1965	EC	3	(60)	≤0.1 all intervals to 54 days							
1966	EC	2	1.1							<0.03	
ME 1969	EC	2 or 3	0.6 or 1.1						<0.05		
		5									
CA 1971	EC	1	1.7					<0.03			
ME 1965			(30 or 60)					<0.1 Control <0.1			
		1									
WI 1964	EC		(120?)					<0.1 Control <0.1			
		7									
VA 1965	EC		(60)			<0.1 Control <0.1					
		4									
MI 1968	EC		0.6(48)		<0.05						
		5			<0.05						
NY 1968	EC		1.1(120)								
		6									
Australia, 1968	EC	6	0.6		<0.02 Control <0.02						
			0.6		<0.02 (9 days) Control <0.02						3,
		1-3									11
Canada, 1977	EC		1.2-1.8	<0.02 mg/kg at 8, 11, 14, 16 and 51 days; 3 sites, 10 trials; Method Demoras, Laurent and Buys, 1973							12
Spinach				0	3		7		14	21	
USA 1972-3	SE	5	1.1 (2400)	13	8.2		3.2		0.5	<0.05	4
			2.2 (4800)	19	14		5.3		0.7	<0.05	
							<0.1 oxon		<0.1 oxon	<0.1 oxon	
			4.5 (9600)	45	40		9.7		6	<0.05	
							<0.1 oxon		<0.1 oxon	<0.1 oxon	
Tomato											
USA				0	2	3	5	10	14/15	26	
NJ, 1965	EC	4	(60)		0.5		0.2	0.1	<0.1		3,7,8
			(120)		0.6		0.4	0.2	<0.1		
				Control <0.1, corrected for 84% recovery							
											3,7,8
VA, 1966	EC	1	(120)	0.9				<0.1			
				Control <0.1, corrected for 92% recovery							
				0	1	2	3	5	14/15	26	
USA NH 1972-3	SE	9	0.3(100)			0.14	0.06	0.06		<0.05	4
			1.7(560)			0.9	0.7	0.4		<0.05	
				EC GLC determination, LOD 0.05 mg/kg							
CA 1972-3	SE	4	0.5(150)	0.4	0.5	0.3	0.2	<0.05			4
			1.7(450)	0.8	0.9	0.7	0.7	0.1			
			3.6(900)	1.9	1.3	1.1	1.2	0.9			
							<0.1*	<0.1*	<0.1* * = oxon		
				EC GLC determination, LOD 0.05 mg/kg phosalone							
				0.1 mg/kg oxon							
MI 1972-3	SE	5	0.6(100)		0.2		0.5	0.3 (7 d.)	0.07		4
			1.7(300)		0.5		1.5	0.5	0.4		
				EC GLC determination, LOD 0.05 mg/kg							

Crop, Country, State, Year (variety)	Application			Residues, mg/kg, at intervals (days) after last application									Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)										
FL 1972-3	SE	7	0.6(60)			0.2		0.03	<0.05	<0.05 (9 days)			4
			1.8(180)			1.5		0.8	0.5	0.1 (9 days)			
			3.6(360)					1.6	1	0.1 (9 days)			
				0		1	3	6/7	10	14	21		
CA 1972-3	SE	3	1.1(300)	1		0.7	0.6	0.4		0.1			4
			3l3(900)	1.6	1.4	1.2	0.7		0.6				
				EC GLC determination, LOD 0.05 mg/kg									
CA 1972-3	SE	3	1.1(240)	0.7	0.7	0.5	0.5	0.4	0.2	0.08			4
			2.2(480)	1.7	1.9	1.4	1.2	0.8	0.8	0.4			
				EC GLC determination, LOD 0.05 mg/kg									
												42	
OH 1972-3	SE	5	1.7(270)	0.5	0.5	0.8	0.9				0.3		
			3.6(560)	2		1	1.5	1.1				0.5	
				EC GLC determination, LOD 0.05 mg/kg									
							3	5	8			34	
NH 1972-3	SE	8	2.3(50)				0.08	0.08	<0.05			<0.05	4
			4.5(100)				0.3	0.4	0.3			<0.05	
				EC GLC determination, LOD 0.05 mg/kg									
				0		1	3	7	14	21			
TX 1973	EC	5	8.4	0.5		0.4	0.3	0.06	<0.05				13
CA 1974	EC	5	8.4(300)	0.6	0.5	0.1							
NJ 1974	EC	5	8.4(180)			0.6	0.1	0.09					
DE 1974	EC	7	12(360)	0.6	0.4	0.2							
				0		1	3	7	14	21			
CA 1974	WP	5	8.4(300)	0.5	0.4	0.3	All controls <0.05 mg/kg						
France,													
Vaucluse 1973	SE	4	0.7(70)						0.3 (15 days)				5
								Control	≤0.01				
S.& Marne 1968[a]	EC	1	0.4(40)	0.4		0.2	0.2	0.06					1,2,3
			0.6(60)	0.5		0.5	0.3	0.09					
				EC determination, corrected for recoveries									
												26	
Francfort 1973	WP	1	0.35(60)									0.7	6
				GLC, thermionic detector					Control		≤0.03		
				0		1	2	3/4	7	10	Half-life (days)		
India 1978	EC	1	0.44	1.4	0.9	0.7	0.4						14
1979-80	EC	1	0.4(50)	7.4	5.2		1.5	0.3	N.D.*	1.5			15
			0.6(75)	8.4	5.7		1.7	0.5	N.D.	1.7			
			0.7(100)	10.7		7.4		2.5	0.9	N.D.		1.9	
										*Detection level not specified			

a. Biological method gave similar results to GLC, but only GLC are shown. b. GLC with thermionic detector
c. Summary only, no field report. GLC. 0.05 mg/kg "sensitivity" reported. No controls. d. Results are mean of 2 extraction procedures.

Pome Fruit (Tables 12 and 13)

Apple (Table 12). Data were available from approximately 150 supervised trials in 10 countries but mainly only summaries of trials before 1980, often in the 1960s. The better described trials were in France, Germany, Italy, and The Netherlands, and those most closely approximating GAP were in France and Germany.

French GAP requires 0.65 kg ai/ha for EC formulations and 60 g ai/hl and a 15-day PHI (number of applications unspecified) for both EC and WP. The French trials in 1986-89 were with SC and most of the others with liquid formulations ("emulsionable solution" in 1970). The maximum residues from 4-7 SC applications at WP/EC GAP application rates in 14 trials after 14 days or more were 1.9 mg/kg, and most were over 1 mg/kg. Most of the trials with liquid formulations were also at application rates close to WP and EC GAP, but no results were available at PHIs as short as 15 days. The highest residue was 3.8 mg/kg after 28 days.

German GAP allows up to 4 applications at 0.9 kg ai/ha (60 g ai/hl) of WP and up to 1 kg ai/ha (70 g ai/hl) of EC, both with a 42-day PHI. Two trials under GAP conditions with an SC formulation showed ≤0.2 mg/kg after 42 days. There were 9 trials with WP which approximated GAP. The highest residue in these was 1.3 mg/kg after 35-42 days, discounting two trials with only summary data. Although the GAP PHI is 42 days, the results indicate that little decrease would be expected between 35 and 42 days after treatment.

GAP in The Netherlands allows single applications of SC at 0.72 kg ai/ha and EC at 0.6-0.9 kg ai/ha, both at 60 g ai/hl. No PHI is specified. One trial in 1970 with 4 applications of an SP formulation resulted in residues up to 2.5 mg/kg after 21 days.

Italian GAP allows single applications up to 1 kg ai/ha (60 g ai/hl) of EC or WP formulations with a 21-day PHI. Residues after 21 days from 5 applications at 0.6 or 9 at 1.2 kg ai/ha were 0.7-1 mg/kg. Spanish GAP allows 60 g ai/hl of WP and 53-70 g ai/hl of EC formulations with a 15-day PHI, the number of applications unspecified. Three applications at 0.6 kg ai/ha in three trials resulted in residues of 5.4 mg/kg after 8 days and 1.6 mg/kg after 33 days. No results were available at the 15-day GAP PHI.

Table 12. Residues of phosalone in apples resulting from supervised trials.

Country, Year, Variety	Form.	No.	Rate, kg ai/ha (g ai/hl)	Residues, mg/kg, at intervals (days) after last application[1]	
				1 4 721 374550	
Canada 1967[2]	EC	8	(60)	2.42.2	73,74
McIntosh				3.22.1	
	WP	8	(60)	2.71.2	
				2.12.4	
	EC	8	(60)	3.22.6	
	WP	8	(60)	2.91.3	
	EC	7	(60)	2	
	WP	7	(60)	1.3	
				2.3	
	EC	8	(60)	1.7	
				60/616475	
Northern spy	EC	8	(60)	2.4	
	WP	8	(60)	1.3	
	EC	7	(60)	1	

Country, Year, Variety	Form.	No.	Rate, kg ai/ha (g ai/hl)	Residues, mg/kg, at intervals (days) after last application[1]	
	WP	7	(60)	0.9	
McIntosh	EC	8	(60)	2.5	
	WP	8	(60)	2	
Red delicious	EC	7	(60)	3.1	
	WP	7	(60)	3.6	
	EC	7	(60)	2.3	
	WP	7	(60)	1.3	
				1 4 72127	
1968[2] McIntosh	WP	10	(30)	2.1 1.4 1.2	
			(45)	3.4 2.8 2.6	74
	WP	9	(30)	1.30.9	
			(45)	1.42.1	
Greening	WP	9	(30)	2 1.3 1.2	
			(45)	2.5 1.8 2.1	
	WP	8	(30)	1.30.6	
			(45)	1.70.9	
				No control values	
				6 2127 42	
Denmark 1966[2]	EC	1	(70)	0.5 0.40.40.1	77
			(140)	1.3 0.40.60.2	
				6 2127424758	
France 1973[2]	EC	3	0.6(60)	0.3	62
G. delicious				GLC with thermionic detectorControl≤0.03	
	WP	5	0.6(60)	0.4	63
				Control ≤0.03	

[1] Where separate analyses by GLC and biological methods were reported, only GLC results are shown. Where only a summary of GLC and biological results was reported, results are shown as "GLC/biol.".
[2] Only summary data provided.

France 1967[2]				16 25 28/29 34/35 47 56/57 90/93	
Golden Delicious	WP	4	0.5 (96 or 160)	0.1 GLC/biol.	65
1972[2]	WP	1	0.5(60)	0.3 GLC thermionic	
B. Hardy	WP	3	0.5(60)	0.05 GLC thermionic	
1969[2]	liq.	4	0.6(40)	0.6 GLC GLC/biol.	
R. des Renin.			0.8(60)	0.8	
1969[2] G. Delic.	WP	4	0.5(40)	0.9 GLC GLC/biol.	
			0.7(60)	1.2	
1969[2]	liq.	7	0.4(40)	0.9 GLC/biol.	
			0.6(60)	1.8	
1967[2] Wine sap	EC	5	0.5(60)	1.3 GLC/biol.	
Canada blanc			0.5(60)	1.1	
1969[2] Starkrimson	liq.	7	0.4(40)	0.8 GLC/biol.	
			0.6(60)	1.5	
1969[2] G. delic.	WP	4	0.7(60)	0.6 GLC/biol.	
1967[2] G. delic.	spray	3	0.6(60)	0.3 GLC/biol.	
1967[2] G. delic.	liq.	4	0.7(88)	0.8 GLC/biol.	
France 1967[2]	liq.	3	0.7(116)	0.7 GLC/biol.	65
G. delicious	liq.	3	0.7(61)	1.2 (20 days) GLC/biol.	
				0 3 7 14/15 37/38 65	
1968[2]	liq.	1	0.6(60)	0.3 0.5 0.2 0.2 GLC/biol.	
Canada blanc			0.6(600)	0.5 0.5 0.4 0.2	
1968[2] P. Triumph	WP	5	0.6(60)	1 1.5 0.8 0.4 GLC/biol.	
			0.6(600)	0.9 1.2 0.9 0.3	
1967[2] G. delicious	spray	5	0.9(60)	GLC/biol. 0.8	

Country, Year, Variety	Form.	No.	Rate, kg ai/ha (g ai/hl)	Residues, mg/kg, at intervals (days) after last application[1]					
								(75 days)	
1969[2] Canada gris	WP	4	1.1(145)				GLC/biol.	0.2	
1967[2] Ca. blanc	WP+liq.	3+2	1.1(70)				1.6 GLC/biol.		
1967[2] G. delicious	liq.	3	0.9(438)				0.2 GLC/biol.		
	liq.	6	1.1(70)			0.4 GLC/biol.			
				9	15				
1974[2] Plein	WP	5	0.9(60)		0.9				64
Haie fruit	WP	4	0.7(60)	0.08					
France 1989				Days	Surface	Pulp	Total		
Touraine G. Delic.	SC	5	0.7	0	0.5	1.4	1.9		84[3]
				14	0.3	1.1	1.4		
				23	0.09	1.2	1.3	control 0.03	
		5	0.7	0	0.08	1.7	2.6		
				14	0.4	1.4	1.6		
				23	0.2	1.3	1.5		
							control	≤0.9?	
Bouches du		6	0.6	1	0.4	1.6	2		
Rhin.Granny				14	0.1	1.2	1.3		
				23	0.09	1.2	1.3		
							control	≤0.5	
Nimms, Golden		6	0.67	17	0.03	0.8	0.8		
				17	0.002	0.1	0.1		
				17	0.06	0.8	0.9		
				17	0.03	0.8	0.9		
				17	0.05	0.7	0.8		
				17	0.002	0.07	0.07		
							control	0.03-0.6	
			0.67	17	0.2	1.5	1.7		
				17	0.1	1.3	1.4		
				17	0.2	1.6	1.8		
Touraine, G. Del.							control	≤0.1	
		5	0.7	23	0.1	1.3	1.4		
Vaucluse, G. Del.			0.7	23	0.03	0.7	0.8		
		7	0.6	23	0.2	1.7	1.9		
Charentes, G. Del.							control	0.1	
		5	0.6	79	<0.001	0.6	0.6		
		4	0.6	77	<0.002	1.3	1.3		
		5	0.6	41	<0.001	1.5	1.5		
		5	0.6	50	<0.001	1.3	1.3		
1970							control	<0.01	
Emerainville				19	28	29/30	58		
Canada blanc	liq.	7	0.4(40)		1				79
	(emuls-		0.6(40)		3.2				
	ionable		0.8(40)		3.8				
	solution)		0.8(40)		3.4				
			0.4(26)		2.3				
			0.6(40)		1.5				
			0.8(53)		2.5				
		8	0.4(27)		1.4				79
		8	0.6(40)		3				
		5	0.4(27)		1.3				
1986 Vaucluse, G. delicious		5	0.6(40)		1.6				
	SC	7	0.5(40)	2					80
1988 Vaucluse, G.		10	0.24(40)				2.3		
delicious				(control 0.3, GLC, LOD 0.05 mg/kg)					
Charente Maritime	SC	4	0.8			1.1			81

Country, Year, Variety	Form.	No.	Rate, kg ai/ha (g ai/hl)	Residues, mg/kg, at intervals (days) after last application[1]	
		2	0.4	0.6	

[3] In reference 84, results were obtained by two extraction methods. Since extraction B gave consistently lower results than extraction A, only results from A are shown.

				0	7	14	21	28	35	42 (GAP)		
Germany 1977	WP		1.2(60)	1.7	1.3	1.3	1	1	0.9			66
				1.8	1 1	1.2	1.1	0.7				
				4.2	3.5	3.9	2.7	2.8	1.3			
				Controls ≤0.05, GLC determ. recoveries ≥100%								
1982[2]	WP	4	0.9(60)	2.2	1.8	1.9	0.8	1.1	0.9	0.8		67
				3.8	2.6	2.6	2.1	2.2	1.3	1.4		
				Controls ≤0.03, recoveries ≥80%								
1980	WP	4	1.4	3.9	0.1	0.8	0.7	0.3	0.3	0.06		68
				Controls <0.05 mg/kg; sampling-to-analysis 6 mo.								
1975	WP	3	0.9(60)	4.4	0.1	0.05	0.05					69
		2	(60)	1.8	2.9	1	0.9	0.2	0.2			
		3	1.1(60)	1.6	0.9	0.7	0.6					
		3	(60)	0.9	1 0.8	0.6	0.3	0.5				
		3	(60)	2	1.1	0.7	0.7	0.9	0.8			
		2	(60)	0.8	0.5	0.5	0.4	0.2	0.2			
				Controls ≤0.1; GLC thermionic determ., recoveries ≥90%								
1980	SC	4	0.9	0.6	1 0.8	1.1	0.3	0.3	0.2			70
				0.6	0.4	0.7	0.1	<0.02	<0.02	<0.02		
					(2 days) Controls ≤0.08; LOD 0.02							
					Sampling to analysis 5-6 months							
											83	
1989	WP	2	0.2								0.03	84[1]
				0	7	14	21	28	35	42		
Italy 1989	EC	5	0.6				0.7					83,84[1]
		5	0.6				0.9					
		9	1.2				1					
					Controls ≤0.2, 103% recov.							82
	EC	4	0.6	1.6	1	1.1	0.5	0.4	0.6			
				control ≤0.05; sample to analysis 3-4 mo.								
Netherlands 1970[2]	WP	3-4	1.2-1.5	2.7		1	2.1					72
				1.6		1.5	2.5					
				0	4	7	14	21				
1970	SP	4	1.3(75)	2.4	3	2.3	3.2	2.5				72a
				1.9	2.6	1.9	2.4	2.1				
				Controls <0.02, not corrected for 83% recov., GLC thermionic determ.								
						8	33	36 (3 separate trials)				
Spain 1989	WP	3	0.6			5.4	1.6	1.5				84
				Controls 0.03		0.5	0.3					
						5	10	20	35(H)			
Switzerland 1966[2]	WP	2	(60)			0.9	0.7	1	0.6			75,77

[1] In reference 84, results were obtained by two extraction methods. Since extraction B gave consistently lower results than extraction A, only results from A are shown.

				1	8	15	21	29	39 (H = harvest)		

Country, Year, Variety	Form.	No.	Rate, kg ai/ha (g ai/hl)	Residues, mg/kg, at intervals (days) after last application[1]								
S. Africa 1968[2]				All Ref. 71 values except days 34 and 54 = mean of three samples								
	WP	7	(34)	2.8	1.9	1.7	1.4	1.4	1.1			71
	EC	7	(34)	2.8	1.8	1.9	1.3	1	0.9			
				24	34	42	54(H)	57	70	84(H)		
	WP	5	(34)			1.1		1	1.1	0.7(H)		
	EC	5				1.4		1.5	0.9	0.9		
	WP	6		2	1.5		2					
	EC	6		1.6	1.6		0.4					
				24	27(H)		38	42(H)	51	62(H)	66(H)	
	WP	5	(34)	1.1			1.4		0.9		0.6	
	EC	5		0.9			1		0.9		0.8	
	WP	7								0.8		
	EC	7								0.5		
	WP	5						1.4				
	EC	5						1.2				
	WP	6			1							
	EC	6			1.1							
USA[2]				0137/8	14/15		21/22	28/29	30-32		63	
VA 1965	WP	5	(60)	5.132.4			2.2 (H))					76,78
MI 1965	WP	6	1	0.30.1			<0.1(H)					
OR 1965	WP	2	1	3.8(H)								
OR 1965	EC	2	1.6	3.9(H)								
VA 1964	EC	6	(30)	8.17.3	3.8		3.9					
NY 1965	EC	1	(30)	21.81.3	1.1							
		5	(30)	7.14.2	2.8		2.4					
			(60)	8.26.1	6		5.3					
			(30)	2.3 (36 d.=H)								
			(60)	4.7 (36 d.=H)								
		6	(30)	6.75.7			4.2					
				(4 d.)								
		1	(90)	1.1	0.8(H)							
				(16 d.)								
ME 1964	EC	1	(30)	2.92.1			1.2	1.1				
				(9 d.)			(18 d.)					
		8	45	2.21.6			0.7	1.4 (32 d.=H)				
				(12 d.)			(26 d.)					
				0171417	21		24	28/31	48			
CO 1967	EC	3	(50)	22.21.1	0.6			0.4				
				(67 d.=H)								
			(90)	4.33.7			3.3					
PA 1965	EC	4	(90)	6.35.1	4.2			3.1		2.4(H)		
MI 1964	EC	5	(60)	2.110.2					0.1(H)			
		6	(30)	8.17.3	3.8			3.9				
		7-8	(30)	1.20.3	0.1			0.1		0.1		
		6	1	10.60.6(H)								
		6	2	2.9(H)*								
		6	2	2.1(H)*								
				014721								
		3	6	10.90.7	0.6			0.3*		*6 l/ha spray		

In refs. 62, 75, 77, 79, 80 and 81 the field trials details were in French. No information on sampling to analysis intervals or storage stability, and no details of analytical methods. No pome fruit processing data?

Pears (Table 13). Summary data were available from trials in France, Denmark, the USA and South Africa, mainly in the 1960s.

Table 13. Residues of phosalone in pears resulting from supervised trials. All summary reports only.

Country, State/Year, Variety	Application			Residues, mg/kg, at intervals (days) after last application (H = harvest)	Ref
	Form	No	Rate, kg ai/ha (g ai/hl)		
France 1972	WP	3	0.48(60)	0 1 3 7 11 13 15 56	
B. Hardy				<0.05 (H) GLC	65
1968	WP	5	0.6(60)	1 1.5 0.8 0.4(H)	65
P. Triumph			0.6(600)	0.9 1.2 0.9 0.3(H)	
				Means of GLC and biological; individual results not reported	
1963	EC	5	0.75(60)	0.5 0.3 0.3) analytical method	77
Williams	WP	5	0.5(40)	0.5 0.2 0.2) not specified	
P. Triumph	EC	5	0.5(60)	(0.4(H)	
	WP	5	0.5(60)	Mean of colorimetric (0.2(H)	
	EC	5	0.3(40)	and bioassays (0.4(H)	
	WP	5	0.3(40)	(0.2(H) 51	
1988	SC	4	0.8	0.3	81
				Control <0.01	
				HPLC determ. with UV detector	
				6 14 21	
Denmark	EC	1	(70)	1.1 0.4)	77
1966		1	(140)	1.5 0.6) Mean of colorimetric and GLC analyses	
USA, OR 1970 Anjou	EC+WP	3	2.5 + 5.5 + 5 (45)	1.3(H) (oxon <0.1)	85
	EC+WP	3	2.5 + 5.5 + 5 (90)	1.8(H) (oxon <0.1)	
OR 1970 Anjou	EC+WP	4	1.7+2.5+ 1.7+3 (5)	1.2(H) (oxon <0.1)	
		4	1.7+2.5+ 1.7+5.9 (90)	1.6(H) (oxon <0.1)	
				EC GLC determination for phosalone	
				0 7 13 20/21 32 38 63 102	
NY 1965	EC	1	(60)	3 2.7 1.5 0.8	
Bartlett			(30)	2.3 1.3 0.9 0.6 colorimetric determination	76, 78
MI 1965 Bartlett	EC	3	(60)	0.5 colorimetric determination	
NY 1964	EC	1	(60)	(0.5(H)	
				colorimetric determination (0.1(H)	
				(0.5(H)	
MI 1964 Bartlett	EC	7	(60)	colorimetric determination 0.1	
WA 1965	WP	2	(100)	5.1 1.1 0.8 0.1(H)	
Bartlett				colorimetric determination	
OR 1966	EC	1	(180)	4.5 0.2(H)	

Country, State/Year, Variety	Application			Residues, mg/kg, at intervals (days) after last application (H = harvest)				Ref
	Form	No	Rate, kg ai/ha (g ai/hl)					
Anjou				colorimetric determination				
				17	26	28	32	71
South Africa	WP	5	(34)			1(H)		
1968	EC	5				0.8(H)		
Packman								
1968	WP	6	(34)				0.5(H)	
Packman	EC	6					0.6(H)	
1968	WP	6	(34)		0.5(H)			
B. Chretien	EC				0.4(H)			
1968	WP	6	(34)	0.7(H)				
B. Chretien	EC			0.8(H)				
				All Ref. 71 results are means of three samples by EC GLC				

Stone Fruit (Table 14).

Data were available for apricots, cherries, peaches, plums and prunes.

Apricots. Summary data were available from 1970-71 supervised trials in the USA. No relevant GAP was reported.

Cherries. Data were available from supervised trials in 1973 in Germany and summary data from Canada (1969), France (1963) and the USA (1964-70). GAP for cherries in Germany and Canada and for stone fruit in France and other European countries was reported. The highest residues (mg/kg) approximately reflecting GAP were France 0.4, Canada 3 to 5, Germany 1.1 and the USA (from trials according to Canadian GAP) 6.3.

Peaches. Supervised trials data were available from Canada (1969), France (1970-86), Italy (1989) and the USA (1966-70). Information on GAP for peaches was available from Canada and Portugal and for stone fruit from France, Switzerland and Italy. The highest residues (mg/kg) from trials approximating GAP were Canada 1.1-2.2, France (French, Swiss or Italian GAP) 1.2-3, Italy 0.13, USA 0.9 (from WP applications. Most US trials were with EC applications, for which no GAP was provided).

Plums. Data were available from more than 30 trials in five countries during 1966-77, in which the residues (mg/kg) from trials which approximated GAP were France 0.3 from an EC formulation after 36 days (GAP was given for SC, with no PHI), UK 0.6-1.8, Canada 1.3-4.1, Germany 1.2, and USA 0.1 (from a trial according to Canadian GAP). Residues in dried plums suggest approximately a two-fold concentration from the loss of water.

Table 14. Residues of phosalone in stone fruit resulting from supervised trials. All the German trials, the French trials on peaches in 1973, 1986 and 1988 and on plums in 1973, and the Italian trial on peaches in 1989 were fully reported. All others in summary only.

Crop, Country, State, Year, Variety	Application			Residues, mg/kg, intervals (days) after last application (H = harvest)									Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)										
				0	7	14	21 (H = harvest)						
Apricots													
USA CA 1971	SE	2	4.2+2.8(90+60)	6.5	4	2.6	1.5						85
		2	4.2+5.6(90+120)	12	8.9	5.8	3.4						
CA 1970	SE	3	4.2+2.8+ 1.7 (90+60+60)	4.8	2.4	0.6(H) 2.6 dehydrated							
		3	4.2+2.8+2.5 (90+60+90)	5.6	3.3	3 (H) 11 dehydrated, oxon <0.1 mg/kg							
CA 1970	SE	2	2.5+3.2 (90+60)	2.2	1.8	1.2 (H)							
			2.5+4.8(90+60)	3.3	2.8	1.3 (H) oxon <0.1 mg/kg							
CA 1970	SE	1	2.1(60)	1.1	0.8	0.3	0.3						
			3.2(90)	2.5	1.6	1.5	0.6						
CA 1970	SE	2	2.1(60)	1.9	1	0.7 (H)							
			3.2((90)	4.4	1.8	1							
				All determinations by EC-GLC, recoveries ≥93%									
Cherries				1	7/8	17/18							
France	EC	1	(60)	0.8	0.8	0.3 mean colorimetric/ bioassay							77
1963 Marmot						0.4 (H) "							
1964 Bigar. d'Edelf.	EC	1	(60)										
Canada 1969				1	8								
sweet cherry	WP	4	(30)	2.9, 2.7	1.2, 0.8	)							74,94
sour cherry		4	(30)	5.3, 4.8	2.8, 2.8	)							
						) Replicate plots, corrected for							
sweet cherry		4	(45)	2, 2.7	2, 1.9	) 89% recovery; EC-GLC determ.							
sour cherry		4	(45)	4.8, 6.7	4.3, 4.9	) No control values							
Germany 1973	EC	1	(52.5)	21 (GAP-35)									
Garibaldi				0.5 2 trees									95
Primavera				0.4 1 tree									
Hedelfingen				0.5 2 trees									
				Controls ≤0.01 95% recovery, therm. GLC determ.									
1975				0	7	14	21	28	35	42			
Schattmor.	WP	3	0.9(60)	4.4		0.13 0.05	0.05		Control ≤0.03				69
Schn. Knorp.	WP	2	(60)	1.8	2.9	1	0.9	0.2	0.2 Control ≤0.1				
Schattmor.	WP	4	1.2(60)	4.5		12	7.5	2.6	1.1	0.4 Control ≤0.05 EC-GLC determ.			66
USA 1968				0	1	2	4/5	7/8	14/15	21	29	70	
MI, Montmor.	EC	3	1.7(60)			9.3		6.3	5.9(H)				
WA, Bing	EC	3	1.5(45)		9.1		5.9	5 (H)					
			2.3(60)		15		9.7	8.7(H)					
OR, Bing	WP	1	4.5(60)									<0.05(H)	

				0	1	2	4/5	7/8	14/15	21	29	
Cherry USA, 1968												
OR Royal Anne	EC	3	2.5(60)	6	5.6			4.4	1.9(H)			85
1969, R. Anne	WP	1	6.7(120)			5						
Lambert						6						
R. Anne		1	13(240)			6						
Lambert						11						
Bing	EC	3	3.4(60)		5.7		3.1	3.2	0.9(H)			
	WP	3	3.4(60)		4.7		4.8	3.7	0.3(H)			
				Determination by EC-GLC, no controls								
OR 1967, Bing	EC	3	(60)	6.1	5			3	2.5	1.6		
OR R. Anne	EC	3	(90)	8.3	6.1			5.6	4			
				Colorimetric determination, no controls								
MI 1970,	EC	3	2.5(90)		21			8.2	3.8		1.1	
MI							oxon	<0.1	<0.1	<0.1		
1964 sweet cherry												
	EC	4	(60)	10	11	11(3 days)		8.2	2	0.1(H)		76,
MI sour cherry								(6 days)	(13 days)			78
	EC	4	(90)			11(3 days)		6	4	0.8 (19 days)		
				Colorimetric determination, no controls								

				0	7/8	10/11	12/13	14/15	17/19	21	23/26	34/36	
Peaches													
France 1986													
Vivian	EC*	4	0.5-0.6(60)						1.4 (Ital. GAP)				89
Sudanel	EC*	9	0.6(43)				1.2						
Michellini	EC*	5	1-1.2((60)						3				
	(*emul- sionable conc.)			Controls ≤0.07; Plots 2-3 trees, GLC LOD 0.05									
Vivian	SC	4	0.5-0.6 (60)						1.2				90
Sudanel	SC	9	0.8(60)				1						
Michellini	SC	5	1-1.2(60)						1.6				
				Controls ≤0.07; Plots 2-3 trees, GLC LOD 0.05									
1988													
Vivian	SC	4	0.6(60)			1.4							81
Michellini	SC	6	1(60)		3								
				Controls 0.08, 0.5. Determination by FPD-GLC. Plots 2-3 trees									
1970				0	7/8	10/11	12/13	14/15	17/19	21	23/26	34/36	
Michellini	EC	1	0.3(60)							≤0.1	≤0.05,		86
1964												≤0.05	
Dugelay	EC	1	0.5(60)		1.9			0.7					77,
1970													86
Redskin	EC	1	0.7(235)			0.4					0.3(23 days)		86
											≤0.01(26 days)		
Fairhaven	EC	1	0.9(60)					0.3			0.2 (24 days)		
											0.1 (28 days)		
											0.08 (31 days)		
Michellini	EC	2	0.3+0.5(60)	0.7			0.3	0.2					
			0.3+0.7(60+80)	0.9			0.7	0.4					
				Average of EC-GLC and biological methods									

				0	10	12	14	15	17	19	
1970											
Redskin	EC	2	0.7+0.5(235+175)	1.2		0.6		0.4			86
	EC	2	0.7(235)	0.9		0.5		0.6			
Fairhaven	EC	2	0.9+0.4 (60+100)	0.7	0.2		0.2		0.2 (Ital GAP)		
		2	0.9+0.6 (60+150)	1.8	0.3		0.4		0.5		
			0.9 (60+225)	2.4	0.8		0.5		0.3		
1973											87
Red Haven	EC	3	0.4(117)	0.4 (control ≤0.01 EC-GLC)							
Coronet	EC	2	0.6(200)	(7 days)						0.5	
Red Haven	EC	3	0.8(70)			0.7 (13 days)					
Redskin	EC	5	0.7(53)							0.8(18 days)	
J. Hale	EC	5	0.7(70)							0.9(18 days)	

Elberta, J. Hale	EC	6	0.7(70)	2.1	
				1973 French trials Controls ≤0.01 GLC/thermionic	
				0 7 13/14 21 28/30	
Italy 1989				0.4 0.3 0.2 0.13 0.13	91
Star.gold	EC	1	1.2(60)	Controls 0.03-0.09; GLC/thermionic; LOD 0.01 half-life ca. 12 days. Plot 4 trees.	
USA 1968				0 7 13/14 21 28/30 35	
VA, Elberta	EC	5	(60)	3.8 3.6 0.5 0.4(H)	85,
	WP	5		2.7 0.5 0.5 0.3(H)	86
	EC	5	4(120)	11 - 0.6 0.7(H)	
	WP	5		3 1.7 0.5 0.5(H)	
1969					
CA, Halford	EC	2	0.5+0.6(15)	0.6 0.5	
			1+1.2(30)	1.2 1	
			2+2.4(60)	3.4 2.8 2.5 2.3(H)	
CA, Halford	EC	1	2(60)	7.9 5.8 4.8 1	
	EC	1	1.7(60)	10 5.1 4.5 1.4 0.8(H)	
	EC	2	0.4+0.6 (15)	1.1 1	
			0.8+1.2 (30)	3.9 2.1	
			1.7+2.4 (60)	8.2 4.3 3 2.6(H) oxon <0.1 all intervals	
CA, Loving	EC	7	0.4(70)	2 (19 days)	
1966					
VA, Elberta	EC	5	0.6(60)	3.8 2.2 0.9	76,
				(40 days)	78
					85,
					86
				0 7 13/14 21 28/30	
USA 1970					
WA, J. Hales	WP	1	1.7(45)	0.9(H) (Canadian GAP)	
			4.5(120)	1.4(H) oxon <0.1	85,
MI, Redhaven	EC	3	3.4(90)	0.7	86
				1.2	
				0.9(H)	
CA, J. Cling	EC	1	2.8(60)	0.6(H) 0.3(H) (separate trials)	
				1 8(H)	
Canada 1969	WP	5	(30)	2.2, 1.9, Corrected for 83% recovery;	74,
Loving				2.1 1.1 EC-GLC determ.	88
		5	(45)	2.8, 1.6, Dual plots	
				4.3 2.2 Controls ≤0.02	
Nectarines				11(H)	
USA 1970	EC	2	1.1(60)	0.2	
CA, Sept. Grand	WP	2		0.3	85,
	EC	2	1.7(90)	0.8	86
	WP	2		0.5	
Plums (fresh prunes)				0 6/7 14/16 20/21 24/26 36 45	
France 1973	SE	6	0.9(60)	0.3 (fresh plums)	87
INRA 711				0.8 (dried)	
				GLC/thermionic determination, 95% recovery. No controls	
UK 1968, Y. Egg					
	liq.	1	(30)	Plot A 1.2, 1.8 unripe fruit	92
Czar				Plot B 1.2, 1 unripe fruit	
		1	(30)	Plot A 0.3, 0.2	
Victoria				Plot B 0.6, 0.6	
		2	(30)	1.1, 1.2,	
				1.1 0.8	
				Other Ref. 92 data (3 applications at 0.4-1 mg/kg) could not be aligned with PHIs. Controls ≤0.04; EC-GLC determination; Data uncorrected for 109% recoveries.	
Canada 1970					
Italian	WP	5	(30)	2.4, 1.3,)	
				1.7 1.8(H)) GLC thermionic determ. No	74
			(45)	1.3, 3.4,) controls; 89% recovery	

				2.2 2.7(H))	
Bluefree	WP	5	(30)	6.1, 1.6,)	
				6.1 2.2(H)) GLC thermionic determ. No	
			(45)	10, 4.1,) controls; 90% recovery	
				9 3.2(H))	
1971				0 6 14 21 28	
Italian	WP	3	(60)	5.4, 7.1 7.3, 8.4 3.6, 3.8 1.1, 1.4 1.5, 2	93
				Duplicate plots. Stones removed. Corrected for 94% recovery, except 0.01 to 0.23 mg/kg controls corrected for 60% if under 0.18 mg/kg. EC-GLC determination.	
Germany 1977					
Hauszwetsch.				0 14 21 28 35 42 47 59	
Auerbauer	WP	2	1.2(60)	2.2 1.7 0.5? 1.5 1.2 1.3 Control ≤0.05	66
	WP	2	1.2(60)	2.7 0.7 0.2 0.2 0.14 0.2 Control 0.1	
USA 1966 "prunes"				EC-GLC determin.	
Fellenburg					76,
Fellenburg					78,
Unspecified	EC	1	(60)	1.2	85
	EC	1	(90)	3.8 (29 days)	
			(60)	1.4	
				Ref. 76 colorimetric determ.	

				0	1	7	13/14	21	28	42	
Plums cont'd USA											
1968 NY,	WP	1	2(60)	0.6		0.3			0.1(H) (Canadian GAP)		85
Fellenburg		1	3(90)	0.7		0.4			0.1(H)		
1969 CA, French	EC	1	0.9(30)	1.6		1.4	1.2	1.2	1.1	0.3(H)	
			1.7(60)	5.6		5.6	4.9	4.4	4.2	0.5(H)	
CA, French	EC	1	2(30)	2.1		1.5	1.4	1.2	0.6(H)		
			4(60)	3.3		3.2	3	2.9	2(H)		
1970 MI, Stan.	EC	3	3.4(450)				4.1				
WA, Italian	EC	1	2.5(45)				0.4				
			6.7(120)				0.6				
1969									130		
OR, Italian	EC	1	3.4(60)					fresh	0.2		85
								dry	0.3		
	WP	1	3.4(60)					fresh	0.05		
								dry	0.1		
1967				0	7	14	21	28	44	48(H)	
OR, Italian	EC	2	(90)	3	2.2	1.8	1.1			1.1 fresh	85
										2.6 dry	
					14(H)		30(H)		44(H)		
1970					Fresh	Dry	Fresh		Fresh		
OR, Italian	EC	3	2.1(45)		2.7	2					85
	WP				1.2	1.2					
	EC	3	2.1+2x4.2		5.7	5.9					
	WP		(45+90)		1.8	1.8					
1971 CA, French	EC	3	4.7(120) +3.1(60)+1.9(60)		5.9	11.4					
1970 CA, French	EC	1	2.8(60)		1.6		0.4 (separate trials)				
CA, Santa Rosa	EC	1	5(60)						0.2		
				Ref. 85 plum analyses by EC-GLC unless indicated otherwise							

Grapes (Table 15). Residue data were available from 52 supervised trials in France, Germany, Italy, Canada, Switzerland, South Africa and the USA (Table 15). Relevant information on GAP was available for France, Germany, Italy and Canada (Table 9).

The highest residues from trials approximating reported GAP were France 0.4 mg/kg, Germany 1.6, Canada 5.0, Switzerland 4.2 (Italian GAP) and the USA 2.6 mg/kg (Canadian GAP). The 1969 South African trials did not accord with reported GAP. Results from five additional German trials on wine grapes (Bacchus and Müller-Thurgau) in 1981 (Germany, 1993) were supplied, but because only a summary table was provided they were not considered further and are not included in Table 15. Residues from these trials did not exceed 0.6 mg/kg after PHIs of 4 to 113 days from 2 applications of a WP at rates of 6 to 24 g ai/hl (GAP allows up to 60 g ai/hl).

Table 15. Residues of phosalone in grapes resulting from supervised trials. All except 1989 trials in France, Germany and Italy reported in summary form only.

Crop, Country, State, Year, Variety	Application			Residues, mg/kg, intervals (days) after last application (H = harvest)								Ref.
	Form	No.	Rate, kg ai/ha (g ai/hl)									
France 1989				0	1	7/8	14/15		32		49	
Gamay	SC	3	1(250)						0.15			84
Ugni Blanc		2	0.6(300)								0.22	
						GLC/thermionic determ., no controls						
1964	EC	1	0.6(363)	0.7		0.4	0.4) Mean, 6 analyses by					77
Gamay	WP	1	0.6(363)(low vol)	0.6		0.4	0.4) colorimetric and bioassay					
Germany 1989				0	14	23	28	42	64	77	90	
Scheurebe	WP	2	0.4+1.1(180)						0.5			84
1974 Riesling	WP	2	0.72(60)	16			8.6	5.1			1(H)	
1975 Riesling	WP	2	1.2(60)	13			5.2	3.1		1.6		97
	WP	3	1.2(60)	7.8	6.3	7.1						
				Ref. 97 and 98 controls ≤0.03 mg/kg. Means of duplicate GLC/thermionic determinations								98
Italy 1989												
Bologna	EC	1	0.6(60)					0.2				84,
Pavia	EC	1						0.2				101
Pavia	EC	1						0.5				
				GLC/thermionic determ.			Control	<0.01 Recoveries 90%				
Canada 1970				0	1	7/8	14/15	21	32	36		
Agawan	WP	5	(36)			4.2,	1.8,					74, 99
						3.5	1.7					
			(54)		7.3,	6.2,	4.2,			4.0,		
					9.5	6	4.3			5.0 (H)		
			(72)		6.3,	4.4,	6.7,			3.9,		
					6.9	5.9	6.3			5.1		
Concord	WP	5	(36)				2.7,					
							3					
			(54)		5.4,		4.4,					
					6.3		4.7					
			(72)		9.2,		7.5,					
					11		10					
						8						74,
Agawan	WP	1	(45)			2.6, 4.3						100
						2.9, 3.6						
						2.9, 3.4						
				Ref. 74, 99 & 100 controls ≤0.02. Results are from duplicate plots, corrected for 86% recoveries; EC-GLC determ.								
Switzerland 1966				5	10	20	35	70(H)				
	EC	1	(53)	10	8	4.6	1.1	0.3				75, 77
				(12) (10)	(4.2)	(1.1)	(0.3)					
	WP	1	(60)	8	4.2	1.5	0.9	0.2				
				(7.2)	(4.5)	(1.3)	(0.8)	(0.2) (Controls <0.05)				
				Ref. 77 results mean of 4-6 colorimetric/bioassay analyses. Those in parentheses are colorimetric alone.								

Crop, Country, Year	Application			Residues, mg/kg, at intervals (days) after last application (H = harvest)								Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)									
				41	61	73						
South Africa 1969												
Barlinka site 1	WP	3	(45)	2, 1.8, 1.1								96
	EC	3	(40)	0.5, 0.9 0.7								
Barlinka site 2	WP	3	(45)		0.4, 0.08, 0.4, 0.2							
	EC	3	(40)		0.3, 0.3, 0.2, 0.2							
Almeria site 3	WP	3	(45)			0.1, 0.4, 0.3						
	EC	3	(40)			0.1, 0.07, 0.1						
				Ref. 96 results are from replicate plots and are corrected for 79% recovery.								
USA 1965				0	6	14	21	28	42			76, 78
NY, Concord	WP	3	2	8	7.3	6.2	5	4.2	3.8			
		3	1	4.3	3.5	2.6	2.4	1.8	1.9			
				Colorimetric determ.								
1964												
MI, Concord	EC	1	1	4	2.2 (4 days)	2.6						
USA 1967				7	14	35	42					76, 78
CA, Emperor	10% powder	1	1.8	0.6	0.5	0.4(H)	GLC determ.					76, 78
			3.6	4	0.9	0.7(H)						
			7.2	2.9	1.5	1.3(H)						
CA, Thompson seedless	EC	2	4.5				2.8(H) GLC determ.					
		2	2.2				0.7(H)					
1970				0	1	3	6	14	17	21/22	60	
CA, Emperor	EC	1	2.3(1400)					1.2(H)				
CA, Ribier	EC	1	2.2(850)	5.7			4.1	3.3		1.7		
		1	4.5(1700)	9.7			9.5	8.4		7.3		
CA, Thompson seedless	EC	1	3.4(1200)							0.8, 1.2 (H)		
CA, Thompson	WP	1	4.5(240) 9(480)								0.5(H) 2.6(H)	
CA, Emperor	WP	2	4.5 (1700)						1.6 (H)			

Crop, Country, Year	Application			Residues, mg/kg, at intervals (days) after last application (H = harvest)	Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)		
			9(3600)	2.2(H)	
CA, Emperor	WP	2	4.5(200)	6.6 (H)	
			9(400)	12 (H) (oxon 0.2)	
WA, Concord	EC	3	2.4(50)	5 5.3 4.7 3(H)	
MI, Concord	EC	3	1.7(150)	1.5, 1.3, 1.7(H)	
				Ref. 85 results by EC-GLC determination	
USA 1970				14	
MI, Champion	EC	3	2.5(135)	1.2, 1.9, 1.2 (H)	85
CA, White Riesling	WP	2	9.6+19(960)	35(H)	
	WP	2	19+9.6 (1920+480)	20 (H)	
CA, Zinfandel	WP	2	9.6+19(960)	24 (H) (oxon 0.5)	
	WP	2	19+9.6(1920+ 480)	18 (H) (oxon 0.3)	
				Ref. 85 results by EC-GLC determination	

Citrus fruit (Table 16 oranges; Table 17 lemons and grapefruit)

Summary data were available from 5 trials on oranges, 2 trials on lemons and 1 trial on grapefruit in 1966-1969 in the USA, as well as summary information on processed fractions. Information on GAP was available for Thailand, Japan and Spain. Only one trial was according to (Spanish) GAP. It gave residues in unwashed orange fruit of 0.2 mg/kg after 14 days (0.3 mg/kg after 235 days). The residues in whole unwashed lemons in a trial approximating Spanish GAP were 1.2 mg/kg after 10 days, 0.3 mg/kg after 25 days and 0.1 mg/kg at harvest after 100 days. The Spanish PHI is 14 days. In the single trial on grapefruit, residues in the whole fruit were 0.2 and 0.3 mg/kg after 10 and 30 days respectively and 0.1 mg/kg after 100 days (at harvest). The Spanish PHI is again 14 days.

The results of processing shown in Tables 16 and 17 are discussed under "Fate of residues in processing" later in this monograph.

Table 16. Residues of phosalone in oranges resulting from supervised trials in the USA. All EC formulations. All analyses by EC-GLC. Summary reports only. (All ref.78).

Year, State, Variety	Application		Residues, mg/kg at intervals (days) after last application						
	No.	Rate, kg ai/ha (g ai/hl)	PHI, days	Washed			Unwashed		
				Whole fruit	Peel	Pulp	Whole fruit	Peel	Pulp
1966 FL. Lue G. Gong	2	8.8 (60)	235	0.17			0.3	0.8	<0.1
1969 CA. Valencia[1]	1	2.5 (45)	14	0.18	0.3	0.1	0.18	0.34	<0.1
		5 (90)		0.9	1.6	<0.1	1.5	7.1	<0.1
1969 CA. Valencia	1	5 (90)	0				2	12	<0.1
			8				1.3	9.5	<0.1
			14				1.5	7.1	<0.1
			28				0.53	3.5	<0.1
1969 CA, Valencia	1	21 (22.5)	35				2.8	1.5	2.8[3]
	1	21 (22.5)	3					3.3	0.1
			7					4.2	0.1
			14					4.2	0.1
			21					4.3	-
			30					3.0	ND
			35					2.8	
			45					2.5	-
			79					1.3	ND
			105					1.0	-

[1] Fruit from these trials were processed with the results shown in Table 28.

Table 17. Residues of phosalone in lemons and grapefruit resulting from supervised trials in the USA. All single applications by EC-GLC. Summary reports only. (All ref. 78).

Crop, Year, Variety	Rate, kg ai/ha (g ai/hl)	Residues, mg/kg, at intervals (days) after last application (H = harvest)				
			Washed whole fruit	Unwashed		
				whole fruit	peel	pulp
Lemon		Days				
1966 CA, Eureka	31(135)	0	2.3	4	8.5	<0.1
		15	2.3	--	7.8	<0.1
		31	2	4	6.2	<0.1
		73(H)	0.8	2.4	2.4	<0.1
CA, ?						
	8.4(45)	3		3.2	6.6	<0.1
		10		1.2	2.3	<0.1
		25		0.3	0.6	<0.1
		50		0.2	0.3	<0.1

Grapefruit USA 1967 AZ, Ruby Blush		100(H)		0.1	0.1	<0.1
	1.7(60)	0	0.8	0.8	1.4	<0.1
		10	0.3	0.2	0.7	<0.1
		30	0.2	0.3	0.09	<0.1
		60	<0.1	<0.1	<0.1	<0.1
		100(H)	<0.1	0.1	<0.1	<0.1

Raspberries and Strawberries (Table 18). Reports were available from the USA (two trials), the UK (one trial) and Germany (1 trial) on strawberries and from the USA (1 trial) on raspberries. Information was received on GAP in Austria (for fruit), Poland (for small fruit) and the CSFR.

Table 18. Residues of phosalone in raspberries and strawberries resulting from supervised trials. Summary reports only.

Crop, Country, Year, (State)	Application			Residues, mg/kg, intervals (days) after last application (H = harvest)							Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)								
Raspberries					1	4	8	11	18		
USA 1965 (MD)	EC	1	0.6(60)		6.5	0.9	0.3	≤0.1	<0.1		7-8, 85
				Controls <0.1, 86% recovery. Determination by spectrophotometry.							
Strawberries				0	3	7	10	17	27	44	
USA 1965 (MD)	Unspec.	1	(60)	2.8	2.1	0.7	0.3	<0.1	(controls 0.1)		7-8
	Unspec.	1	(330)						1	<0.1	
									(control 0.1)		
					14	21	26		28	35	
UK 1968	EC	2	0.6(31)				0.07, 0.08 0.05 *				
				*Replicate plots; Controls ≤0.01 mg/kg. Determination by EC-GLC							102
					2.3	1.4			0.8	0.2	
Germany 1974	WP	2	1.2(60)	Controls ≤0.05. Determ. by GLC/thermionic							103

Nuts (Table 19). Summary data were provided from supervised trials in the USA on almonds, hazelnuts, walnuts and pecans in 1967-70. Information on GAP was available from France for almonds, chestnuts and hazelnuts and from Turkey for hazelnuts (Table 9). In 5 US trials on almonds residues in the dry nuts were <0.05 mg/kg at all PHis of ≥ one week and in the dry husks up to 16 mg/kg at 35 days after EC applications at rates roughly comparable to French GAP for "Flo" formulations.

Residues of <0.05 mg/kg were also reported in the meat of hazelnuts from 6 US trials according to Turkish GAP on a g ai/hl basis, although multiple applications were made whereas one application is allowed in Turkey. Residues were up to 3.7 mg/kg in hazelnut shells. Residues in walnut kernels in four US trials were reported as <0.1 mg/kg from EC applications at rates comparable to French GAP for "Flo" formulations. Residues in walnut shells were up to 35 mg/kg. Residues were <0.1 mg/kg in the meat of pecans in 7 US trials from applications up to 5 kg ai/ha (120 g ai/hl), but no GAP was reported for pecans.

Table 19. Residues of phosalone in nuts resulting from supervised trials in the USA.

Crop, Year, State	Application	Residues, mg/kg, intervals (days) after last application (H = harvest)	Ref.

	Form.	No.	Rate, kg ai/ha (g ai/hl)		Whole nuts	Husks		Nut meat			
Almonds 1969				Days	Green	Dry	Green	Dry	Green	Dry	
CA	EC	1	1.7(60)	0	5.5	14					
				7	4.4	11					85
				14	2.5	6.4					
				21			5.5	25		<0.05	
				28			4.5	19		<0.05	
				35			15	16 (oxon 0.5)		<0.05	
CA	EC	1	2(60)	0	6.2	20					
				8	5.8	16				<0.05	
				14	2.6	7				<0.05	
				19			5.6	16		<0.05	
				0	5.5	21					
CA	EC	1	2.2(60)	7						<0.05	
				14						<0.05	
				21	6.5	14				<0.05	
				35			3.1	13			
										<0.05<0.05	
				63(H)	0.05	<0.05	5.6	6.1	<0.05	<0.05	
CA	EC	1	2.8(120)					(oxon 0.6)			
				0	4.6	16					
				7	3.3	12					
				14	3.4	9.6					
				21(H)	2.8	2.8	35	39	0.1		
CA	EC	1 or 2	4.4					(oxon 0.1)			
				0	7.7	39					
				7	6.4	29					
				14	4.9	22					
				21					<0.05		
				40(H)			50	54	<0.05		
								(oxon 0.8)			
				All Ref. 85 phosalone analyses by EC-GLC; ≥90% recoveries; oxon analyses by CCM							
Hazelnuts 1970				Days	Whole nuts		Husk		Nut-meat		
OR	EC	2	4.8(70)	62(H)	0.3						
OR	EC	2	4.8(70)	62(H)	0.5			1.1		<0.05	85
	EC	4	2.1(45)	15(H)	0.5			0.8		<0.05	
OR	WP	4	2.1(45)	15(H)	0.5			1		<0.05	
	EC	2+2	2.1(45)+ 4.2(90)	15(H)	1			3.7		<0.05	
OR			2.1(45)+4.2(90)								
	WP	2+2		15(H)	0.7			2.3		<0.05	
				EC-GLC determination							
Walnuts				Days		Whole		Shell		Nut-meat	
CA 1967	EC	1	2.2(60)	60(H)						<0.1	78
CA 1968	EC	1	2.2	48(H)		<0.1				<0.1	
CA 1969	EC	2	2+1.7	36(H)						<0.1	
CA 1969	EC	2	1.7	14(H)						<0.1	
				28(H)						<0.1	
CA 1969	EC	1	6.7(480)	14(H)				35		0.1	
				EC-GLC determination							
Pecans				Days				Shell		Nut-meat	
AL 1967	EC	8	0.6(87)	66(H)				0.2		<0.1	78
AL 1969	EC	9	0.28(30)	28(H)						<0.1	
			0.56(60)	28(H)						<0.1	
			1.1(120)	28(H)				<0.1		<0.1	
SC 1969 Stuart	EC	5	5(120)	95(H)				<0.1		<0.1	

Crop, Year, State	Application			Residues, mg/kg, intervals (days) after last application (H = harvest)				Ref.
	Form.	No.	Rate, kg ai/ha (g ai/hl)	Whole nuts	Husks	Nut meat		
						<0.1	<0.1	
GA 1969 Sucress	EC	3	0.6(60)	120(H)			<0.1	
	EC	1	0.8(60)	198(H)			<0.1	
				EC-GLC determination				

Cereals (Table 20). Data were available on one US trial on sorghum in 1964, showing 0.2 mg/kg in the seed, and three French trials on wheat in 1967. GAP was available for European countries and India. Residues in the French wheat seeds were ≤0.1 mg/kg after 25-44 days. The French PHI for cereals is 25 days.

Rape (Table 20). Summary reports were available from 7 1966 trials in France, 3 1968-9 trials in Denmark and one in 1975 trial in Poland. Three 1974 trials in Germany were more fully described. Information on GAP was available for France (for Brassica oil crops), Germany and Poland as well as other European countries (not Denmark), but no French PHI was specified for the EC formulation used in the French trials. In these the residues in the seed were 0.1 mg/kg after 27 and 77-78 days from GAP rates. Residues from applications according to GAP were ≤0.1 mg/kg in the Danish trials after 57 days, the Polish trial after 48 days and two of the German trials after 28-35 days. In the third German trial the residues were 0.3 mg/kg after 28 days and 0.4 mg/kg at harvest after 35 days.

Table 20. Residues of phosalone in cereals and oilseed rape resulting from supervised trials. All except the German trials reported as summaries.

Crop, Country,Year	Application			Residues, mg/kg, intervals (days) after last application (H = harvest)						Ref.
	Form.	No.	Rate, kg i/ha (g ai/hl)				108			
Sorghum										
USA TX 1964	EC	2	1				0.2 seed			21
							0.2 leaves and stem			
				Colorimetric analyses						
Wheat					6	11	18	25(H)		
France 1967	EC	1	0.6(8.5)	whole plt	10	11	9.2	6.7		20,21
				seed				0.1		
		1	0.6(300)	whole plt	11	9.2	7.2	7.2		
				seed				0.1		20,21
					0	7	15	30	44(H)	
	EC	1	0.6(60)	whole plt	18	4.5	2.3	0.6	0.3	
				seed					<0.05	
				physico-chemical (undefined) and bioassay determinations						
Oilseed Rape				Days	Seed	Oil				
France 1966	EC	1	0.6	78(H)	0.1	<0.5				20,21
			1.2	78(H)	0.1	<0.5				
			2.4	78(H)	0.1	<0.5 Colorimetric and bioassay				
	EC	3	1	77(H)	0.1	<0.5				
			2.4	77(H)	0.1	<0.5 Colorimetric and bioassay				
					Whole seedhead[1]			Seed		
					9	24		27	42	
	EC	1	0.7		3.7	0.3		<0.1	<0.1	20,21
		1	1.4		11	0.8		<0.1	<0.1	

				Colorimetric and GLC determination (means of 6 analyses)	
Denmark 1968	EC	1	2.1(10000) (aerial)	<0.1 in seed after 28 days(H) GLC and bioassay (means of 4 determinations	20,21
1969	Dust EC	1 1	0.8 1.1 (195)	≤ 0.03 in seed after 57 days (both trials) EC-GLC determination	41
Poland 1975	EC	1	1.2(400)	≤ 0.1 in seed after 48 days (same in control) GLC/thermionic determination	42
				Days Grain	
Germany 1974	EC	2	1.1(175)	34(H) ≤0.05	43
	EC	2	1.1(175)	27(H) ≤0.05	
				35 ≤0.05	
	EC	2	1.1(260)	28 0.3	
				35(H) 0.4	
				Controls ≤0.1 GLC/thermionic determination. Recoveries ≥86% from grain	

[1] This is assumed to be the meaning of the French "siliques entières"

Cotton (Table 21). Residues were ≤0.1 mg/kg in seed (delinted or not delinted), meal and crude oil from cotton harvested 6 to 57 days after applications up to 1.5 and even 4 kg ai/ha.

Hops (Table 21). In two US trials in 1966 residues were 1.2 and 1.7 mg/kg in green cones and <0.1 mg/kg in dry cones 28 days after applications up to 2 kg ai/ha.

Tea (Table 22). Data were available from four 1967-9 trials in Pakistan and 6 1970-71 trials in India. Information on GAP was available for Taiwan, Japan and India (Table 9). In the Pakistan trials residues did not exceed 0.8 mg/kg in green tea 5 days after applications at rates comparable to Indian GAP or, according to a summary report, 0.3 mg/kg in manufactured tea after 7 days. Residues in a tea infusion were 0.004 mg/kg.

The Indian trials were at application rates in accordance with Indian GAP. Although the information on GAP did not specify a PHI, the trials reports indicate that a 10-day PHI was normal practice at the time of the trials. This is consistent with the reported 7-day PHI for Japan. Some of the trials in Tocklai (northern India) were in dry weather. Treatment in the dry season is not considered GAP in the north because the harvest is suspended as there is little plant growth. In the other Indian trials that approximated GAP residues of phosalone and phosalone oxon in green tea were up to 0.06 and 0.1 mg/kg respectively after 10 days and 0.5 and 0.1 mg/kg after 7 days, but the oxon controls were also ≤0.1 mg/kg. Phosalone and oxon residues in processed (dry) tea from GAP treatments in dry weather in southern India were up to 7.2 and 1.1 mg/kg respectively after 8 days and 3.8 and 0.6 mg/kg after 10 days. In the northern trials in wet conditions residues of phosalone were 0.07-0.09 mg/kg and of the oxon ≤0.05 mg/kg in both treated and control samples. The results of the northern trials in dry weather (not GAP) indicate concentration factors from green to processed tea of about 6 for phosalone and possibly up to about 10 for its oxon.

Phosalone and oxon residues in infused tea from treatments in the wet season according to GAP were up to 1 and 0.8 ìg/l respectively after a 10-day PHI and in the dry season 12 and 10 ìg/l at 8 days and 1.6 and 1.5 ìg/l at 15 days.

Table 21. Residues of phosalone in cotton and hops resulting from supervised trials in the USA. All EC formulations. Summary data only.

Crop, Year, State	Application	Residues, mg/kg, intervals (days) after last application	Ref.

			(H = harvest)	
	No.	Rate, kg ai/ha (g ai/hl)		
Hops 1966 WA Yamika c.	 2 2	 2+1 2+1	28 Green Cones / Dried Cones (dried 3 days) 1.7 / <0.1 1.2 / <0.1 Mean of 4 colorimetric analyses	 21
Cotton seed 1964-6 TX, AR, CA (7 trials, 6 varieties)	6-13	1-1.5 (6 trials) 1-4(1 trial)	0.1 mg/kg in seed (delinted or not) harvested after 6, 14, 15,17, 34, 39 and 57 days PHI, and in the meal and crude oil from the seed samples. All colorimetric analyses	21

Table 22. Residues of phosalone in tea resulting from supervised trials. All single applications of EC. Underlined residues are from trials according to Indian GAP.

Country, Year, Conditions	Appl. rate, kg ai/ha (g ai/hl)	PHI, days	Residue, mg/kg				Residue, ìg/l		Ref.
			Green tea		Processed tea		Infusion		
			Phos.	Oxon	Phos.	Oxon	Phos.	Oxon	
Pakistan	0.4 (30)	0	4.8, 5.2						53
1969		1	14, 11						
		3	1.1, 0.9						
		5	0.3, 0.6						
			Controls ≤0.07. Mean recovery 88%. GLC						
	0.7 (60)	0	14, 17						
		1	16, <0.02[b]						
		3	6.1, 10						
		5	0.3, 0.8						
1967[a]	0.4 (30)[c]	3	1.5						54
		7	0.27				4[d]		
	0.7 (60)[c]	3	0.7						
		7	0.2						
			Controls ≤0.04						
India	0.4 (440)	0	75	≤0.2					55,56
1971		3	4.2	0.4					
Toklai		7	0.4, 0.3	≤0.1					
(N. India)		10	≤0.04, ≤0.05	≤0.1	0.08, 0.07	≤0.05	1, 0.8	≤0.8	
Wet	0.7 (700)	0	76	0.4					
weather		3	7,5	0.7					
		7	0.5, 0.4	≤0.1					
		10	0.06, 0.06	≤0.1	0.09, 0.07	≤0.05	0.7, 0.6	≤0.8	
		Controls	≤0.03	≤0.1	≤0.04	≤0.05			
Dry	0.4 (440)[f]	0	40, 24	0.2					55
weather		3	59, 75	1.7					
		7	13, 10	0.6					
		10	2.6, 2.7	0.3	16, 19	3.2, 2.9	34, 42[e]	15, 17[e]	
	0.7 (700)[f]	0	97, 103	0.4					
		3	68, 92	2.5					
		7	16, 41	2					
		10	7.5, 14	0.3, 0.8	50, 53	6.5, 1.3	95, 92[e]	29, 25[e]	
		Controls	≤0.05	≤0.05	6	1.3			55
1970	0.6	1	1		185	3			
Chichona		5	-	-	36	2.1			
(S. India)		8	-	-	7.2	1.1			
		10	-	-	2.8	≤0.05			

Country, Year, Conditions	Appl. rate, kg ai/ha (g ai/hl)	PHI, days	Residue, mg/kg				Residue, ìg/l		Ref.
			Green tea		Processed tea		Infusion		
			Phos.	Oxon	Phos.	Oxon	Phos.	Oxon	
		15	-	-	0.4	≤0.05			
		Controls			≤0.05	<0.5			
	0.7	1	-	-	169	3	280	23	55
		5	-	-	41	3.1	34	24	
		8	-	-	6	0.9	12	10	
		10	-	-	3.8	0.6	10	2	
		15	-	-	0.4	≤0.1	1.6	≤1.5	
		Controls			≤0.03	≤0.1			

[a] Summary data only

[b] Result suggests untreated sample

[c] Residues suggest application rates were reversed, but they are listed as reported in Ref. 54. Manufacturer's summary lists residues as 1.5, 0.27, 0.72 and 0.18 mg/kg at 0, 1, 3 and 5 days after application and only the 0.4 kg ai/ha rate. This seems more logical, but ref. 54 lists two application rates (0.03% and 0.06%), with residues from each at 3 and 7 days (supported by recorded rates, treatment dates and picking dates) as shown in this Table.

[d] 15 g of manufactured tea containing 1.5 mg/kg phosalone was infused with 1000 ml boiling water for 15 minutes and the infusion analysed for phosalone by GLC after extraction with dichloromethane. A control showed 0.008 mg/kg

[e] 7.5 g processed tea simmered 15 minutes in 500 ml boiling water

[f] Not considered GAP, since harvest is suspended during the dry season in these northern sites (little plant growth)

Residues in Animals (Tables 22-25)

Cows. The potential for residues in the milk and tissues of dairy cows was investigated by feeding unlabelled technical phosalone (96% pure) in gelatin capsules for 28 days at rates equivalents to 500, 200 and 100 ppm in the diet (Craig *et al*., 1980a), half of the dose in the morning and half in the evening. Animals were slaughtered for tissue samples on the last day of dosing, and 14 and 21 days later. Milk samples were taken in the morning and evening at 11 intervals during the 28 days, and on the 29th day. Tissue and milk samples were analysed for phosalone and its oxygen analogue by the modified method 54C (see "Methods of residue analysis" above). Determinations were by EC-GLC.

Although animal metabolism studies have not identified all the radiolabelled residues, phosalone, the sulphide (IV) (Table 1) and the sulphoxide V were identified as significant proportions of the residue in tissues and milk, together with lesser amounts of other metabolites. Phosalone was a significant residue only in the fat. Because residues in animals were expected to consist largely of compounds other than phosalone and its oxon, the tissue and milk samples were also analysed by other analytical methods (Craig *et al.*, 1980b) for other chlorobenzoxazoles and chloroaminophenol. In particular, milk was analysed for the free aglycone VII, chloroaminophenol (IX), and the thiol III and both milk and tissues for combined sulphide, sulphoxide and sulphone (as "total sulphone") and for water-soluble conjugates of chlorobenzoxazolone.

No chlorobenzoxazolone conjugates were detected in the tissues of cows fed phosalone at the 100 or 200 ppm dietary levels. At the 500 ppm level <0.01 mg/kg was detected in muscle, spleen, brain, heart and kidney and <0.02 mg/kg in fat, while <0.01 and 0.02 mg/kg were reported in the livers of the two cows tested. Recoveries from tissues at the 0.1 mg/kg fortification level were 55-91% with an average of approximately 73%.

Residues of phosalone and the oxon in tissues are shown in Table 23 and total residues measured as the sulphone in Table 24. At the 100 ppm feeding level phosalone residues were ≤0.023 mg/kg in the fat, ≤0.07 mg/kg in the liver and (by inference from residues at the higher feeding levels) <0.01 mg/kg in other tissues.

Low total sulphone residues were found in the tissues of cows at each of the three feeding levels. At the 100 ppm level they were ≤0.05 mg/kg in all tissues (<0.01 mg/kg in muscle) except liver where they were 0.2 mg/kg.

The maximum combined residues of phosalone, its oxon and "total sulphone" at the 100 ppm feeding level were therefore about 0.03 mg/kg in the fat, 0.3 mg/kg in the liver, 0.05 mg/kg in the kidneys and <0.05 mg/kg in other tissues. The tissues were not analysed for the aglycone VII, chloroaminophenol (IX) or the thiol III as was milk.

Table 23. Residues of phosalone and its oxon in cow tissues resulting from the feeding of phosalone at rates equivalent to 500, 200 and 100 ppm in the diet (Craig *et al.*, 1980a).

Tissue	Days after end of feeding	Feeding level 500 ppm		Feeding level 200 ppm	Feeding level 100 ppm
		Residues, mg/kg			
		Phosalone	Oxon	Phosalone	Oxon
Fat	0	0.07	<0.02	0.07	0.010
	0	0.14	<0.02	0.04	0.023
	14	0.04		<0.01	<0.01
	21	<0.01		<0.01	<0.01
	Control	<0.01			
	Recovery	93-115%	98-123%		
Liver	0	0.04	<0.04	0.1	0.06
	0	0.18	<0.04	0.03	0.07
	14	<0.01		<0.01	<0.01
	21	<0.01		<0.01	<0.01
	Control	<0.01			
	Recovery	94-107%	148%		
Kidney	0	0.013,	<0.02	<0.01	-
	0	0.016	<0.02	<0.01	-
	14	0.013		<0.01	-
	21	--		<0.01	-
	Control	<0.01	91-143%		
	Recovery	100-119%			
Heart	0	0.015	<0.02	<0.01	-
	0	0.03	<0.02	0.011	-
	14	<0.01		<0.01	-
	21	0.013		<0.01	-
	Control	<0.01			-
	Recovery	104-136%	86-94%		
Muscle (B. Femoris)	0	<0.01	<0.02	-	-
	0	<0.01	<0.02	-	-
	14	<0.01		-	-
	21	<0.01		-	-
	Control	<0.01	150%		
	Recovery	90-91%			
Muscle (L.	0	<0.01	<0.02	-	-

Tissue	Days after end of feeding	Feeding level 500 ppm		Feeding level 200 ppm	Feeding level 100 ppm
		Residues, mg/kg			
		Phosalone	Oxon	Phosalone	Oxon
Dorsi)	0	0.02	<0.02	-	-
	14	<0.01		-	-
	21	<0.01		-	-
	Control	<0.01			
	Recovery	100-104%	100%		
Brain	0	<0.025	<0.025	-	-
	0	<0.025	<0.025	-	-
	14	<0.025		-	-
	21	<0.025		-	-
	Control	<0.025			
	Recovery	111-122%	100-112%		
Spleen	0	<0.01	<0.02	-	-
	0	<0.01	<0.02	-	-
	14	<0.01		-	-
	21	<0.01		-	-
	Control	<0.01			
	Recovery	95-102%	80-104%		

Table 24. Residues determined as total sulphone (mg/kg) in cow tissues (Craig *et al*, 1980a).

Tissue	Days after end of feeding	Feeding level, ppm		
		500	200	100
Fat	1	0.05	0.06, 0.07	0.03, 0.03
	1	0.06, 0.07	0.03	0.06, 0.04, 0.03
	14	<0.02	-	-
	21	<0.02	-	-
	Control	<0.02		
	Recovery	67% at 0.01		
Liver	1	0.22	0.2, 0.2	0.2
	1	0.24, 0.22	0.07	0.2, 0.2, 0.2
	14	<0.01	-	-
	21	<0.01	-	-
	Control	<0.01		
	Recovery	82% at 0.01		
Kidney	1	0.05	0.04, 0.05	0.04, 0.04
	1	0.06, 0.06	0.014	0.05, 0.04, 0.04
	14	<0.01, <0.01	-	-
	21	-	-	-
	Control	<0.01		
	Recovery	60 & 89% at 0.01		
Heart	1	0.02	0.01, 0.02	0.01, 0.01
	1	0.02, 0.02	0.01	0.02
	14	<0.01	-	-
	21	-	-	-
	Control	<0.01		

Tissue	Days after end of feeding	Feeding level, ppm		
		500	200	100
	Recovery	76 & 95% at 0.01		
Muscle (B. Femoris, L. Dorsi)	Same	<0.01		
Brain	1	0.02, 0.02	0.02	<0.01
	1	0.02	0.01	0.03
	14	<0.01	-	-
	21	-	-	-
	Control	<0.01		
	Recovery	72% at 0.01		
Spleen	1	<0.01	0.014, 0.015	0.03
	1	0.02, 0.03	<0.01, 0.01	0.02
	14	<0.01, <0.01	-	-
	21	<0.01	-	-
	Control	<0.01		
	Recovery	60 & 95% at 0.01		

Table 25 shows the residues of phosalone and its oxon in milk at days 1, 16, 28 and 19 (dosing stopped on day 28) at the three feeding levels. Table 25 shows the total sulphone in milk at all feeding levels, and free and conjugated chlorobenzoxazolone, chloroaminophenol (CAMP) and the thiol conjugates were found at the 200 ppm feeding level and at the 500 ppm feeding level (≤0.05 mg/kg of the conjugate found at 200 ppm feeding level).

At the 100 ppm feeding level residues of phosalone plus its oxon were 0.007 mg/kg (found on day 19 and total sulphone residues were ≤0.04 mg/kg giving a combined maximum of 0.05 mg/kg. The combined residues decreased to about 0.02 mg/kg after a one-day withdrawal period. The free and conjugated chlorobenzoxazolone, chloroaminophenol and thiol metabolites did not individually exceed 0.05 mg/kg after 7 days at the 500 ppm feeding level.

Table 25. Residues of phosalone and its oxon in cow milk resulting from the feeding of phosalone at 500, 200 and 100 ppm in the diet (Craig *et al*, 1980a).

Day	Residue, mg/kg[1], feeding level (ppm)			
	500		200	100
	Phosalone	Oxon	Phosalone	Phosalone
1 a.m.	<0.001	<0.01	<0.005	<0.001
	(0.001)	(<0.01)	(0.02)	(<0.001)
p.m.	0.004		<0.002	<0.001
	(0.005)	all dosing intervals	(0.003)	(0.001)
16 a.m.	0.008		0.003	<0.002
	(0.02)		(0.006)	(0.003)
p.m.	0.005		0.005	0.002
	(0.007)		(0.013)	(0.003)
28 a.m.	--		--	--

Day	Residue, mg/kg[1], feeding level (ppm)			
	500		200	100
	Phosalone	Oxon	Phosalone	Phosalone
	--		--	--
p.m.	0.008		0.005	<0.002 [3]
	(0.01)		(0.006	(0.004) [3]
29 (withdrawal)				
a.m.	0.006			
	(0.009)		0.005	<0.002
p.m.	0.005[2]		(0.01)	(0.003)
	(0.008)[2]		0.006 b (0.009) [2]	0.005 [2] 0.007 [2]

[1] Residues are means from 4 different cows at each dosing level, with maximum levels in parentheses. Days 1, 16 and 28 taken as representative of 11 actual sampling days.

[2] 2 cows only

[3] 3 cows only

Table 26. Residues of total sulphone, free and conjugated chlorobenzoxazolone (CBZ), chloroaminophenol (CAMP) and thiol in cow milk resulting from the feeding of phosalone (Craig *et al*, 1980a).

Day	Residue, mg/kg[1], feeding level (ppm)			
	500		200	100
	Phosalone	Oxon	Phosalone	Phosalone
Total sulphone				
1	0.05 [2]		-	-
	(0.04-0.06)		-	-
7	0.08		0.06	0.03 [3]
	(0.04-0.12)		(0.05-0.08)	(0.02-0.04)
13	0.07		0.06	0.03
	(0.04-0.013)		(0.05-0.07)	(0.02-0.04)
27	0.04		0.05	0.02
	(0.03-0.06)		(0.03-0.14)	(0.01-0.03)
1 Day withdrawal	0.04		0.03 [2]	0.01[4]
	(0.03-0.04) [3]			(0.007, 0.014)
Controls				
Recoveries	<0.005			
	108% at 0.01 fortification			

Day	Residue, mg/kg, at 500 ppm feeding level			
	CBZ		CAMP	Thiol
	Free	Conjugated[5]		
1	-	<0.005[2]	-	-
7	<0.01	<0.006 (<0.005-0.008)	<0.02	<0.05
13	<0.01	<0.007 (<0.005-0.012)	<0.02	<0.05
27	<0.01	<0.006 (<0.005-0.008)	<0.02	<0.05
One day withdrawal	<0.01[3]	<0.006 (<0.005-0.007)	0.02[4]	
Controls	<0.01	<0.005	<0.02	<0.05
Recoveries	112% at 0.05 fortification	102% at 0.01 fortification	60% at 0.05 fortification	80-100% at 0.05 fortification

[1] Unless indicated otherwise results are means, with range in parentheses, of a.m. and p.m. milkings of 4 cows

[2] One cow

[3] Three cows

[4] Two cows

[5] At 200 ppm dietary level CBZ conjugate levels averaged <0.005 mg/kg with a range of <0.005-0.007 mg/kg over all intervals

Chickens. The potential for residues in chicken tissues and eggs has been investigated by feeding phosalone in the diet at 10, 30 and 100 ppm for four weeks (Craine and Kiggens, 1979). Several pages were missing from the submitted report, mainly from the protocol. Tissue and eggs were analysed for total sulphone and conjugates of the aglycone. The results are shown in Table 27. Residues of the aglycone conjugates were <0.006 mg/kg in eggs at 30 ppm and ≤0.03 mg/kg in all tissues at 100 ppm. Total sulphone residues were detected at all feeding levels; the highest residues (extracted from Table 27) during the four-week feeding were as follows.

	mg/kg sulphone ppm phosalone in feed		
Sample	100	30	10
gizzard	9	-	0.7
kidney	13	3	0.09
liver	15	4.3	1.3
heart	6.9	1.5	0.5
leg	8.6	2.3	0.7
breast	3.2	0.8	0.3
skin (& adhering fat)	18	3.3	1.1
eggs	7.2	1.3	0.5

In most tissues the total sulphone residues appear to have reached their highest level before the end of the feeding period but those in eggs, and to a lesser extent skin, from the 100 ppm feeding level continued to increase throughout.

Table 27. Residues of total sulphone and conjugated aglycone in chicken tissues from the *ad libitum* feeding of feed containing phosalone at 10, 30 and 100 ppm for 4 weeks, followed by a 4-week withdrawal period. Results not corrected for recovery (Craine and Kiggins, 1979).

Sample Week of treatment or withdrawal	100 ppm	30 ppm	10 ppm

		Sulphone	Aglycone	Sulphone	Aglycone	Sulphone	Aglycone
Gizzard							
Trt. week	2	9	all <0.01	-	-	0.7	-
	3	-		-	-	-	-
	4	5.1		-	-	0.2	-
Wdrwl wk	1	1.2	all <0.01	-	-	all <0.05	
	2	0.06		-	-		-
	3	-		-	-		-
	4	<0.05		-	-		-
Control		<0.05	-	-	-	<0.05	-
Recovery, %[1]		78-111	69	-	-	55-111	- -
Kidney							
Trt. week	2	13	0.01	3	-	<0.05	-
	3	-	-	-	-	-	-
	4	8.5	0.02	2.1	-	0.09	-
Wdrwl wk	1	2	-	0.4	-	all <0.05	-
	2	0.08	<0.01	<0.05	-		-
	3	-	-	-	-		-
	4	<0.05	<0.01	<0.05	-	<0.05	-
Control		<0.05	<0.01	<0.05	-	68-90	-
Recovery, %[1]		68-90	67-74	68-90	-		-
Liver							
Trt. week	2	13	<0.01	4.3	-	1.3	-
	3	-	-	4	-	-	-
	4	15	<0.01	2	-	1.1	-
Wdrwl wk	1	0.9	-	0.3	-	<0.05	-
	2	0.4	<0.01	0.07	-	<0.05	-
	3	-	-	-	-	-	-
	4	0.2	<0.01	<0.05	-	<0.05	-
Control		<0.05	<0.01	<0.05	-	<0.05	-
Recovery, %[1]		82-93	-	82-93	-	82-93	-
Heart							
Trt. week	2	6.4	0.02	1.3	-	0.5	-
	3	-	-	-	-	-	-
	4	6.9	0.03	1.5	-	0.3	-
Wdrwl wk	1	1.2	-	0.2	-	<0.05	-
	2	0.1	0.01	<0.05	-	<0.05	-
	3	-	-	-	-	-	-
	4	<0.05	<0.01	<0.05	-	<0.05	-
Control		<0.05	<0.01	<0.05	-	<0.05	-
Recovery, %[1]		88-115	69-73	89-115	-	89-115	-

Sample Week of treatment or withdrawal	100 ppm		30 ppm		10 ppm	
	Sulphone	Aglycone	Sulphone	Aglycone	Sulphone	Aglycone
Leg						
Trt. week 2	8.4	all <0.01	2.3	-	0.7	-
3	4.9		1.5	-	0.3	-
4	8.6		2.2	-	0.6	-
		all <0.01				
Wdrwl wk 1	0.9		0.1	-	<0.05	-
2	0.08		<0.05	-	<0.05	-
3	-	<0.01	-	-	-	-
4	<0.05	55	<0.05	-	<0.05	-
Control	<0.05		<0.05	-	<0.05	-
Recovery, %[1]	83-87		83-87	-	83-87	-
Breast						
Trt. week 2	1.9	0.01	0.3	-	0.05	-
3	3.2	-	0.8	-	0.3	-
4	1.8	<0.01	0.7	-	0.1	-
Wdrwl wk. 1	0.7	-	0.09	-	<0.05	-
2	<0.05	<0.01	<0.05	-	<0.05	-
3	-	-	-	-	-	-
4	0.07	<0.01	<0.05	-	<0.05	-
Control	<0.05	<0.01	<0.05	-	<0.05	-
Recovery, %[1]	75	46	75	-	75	-
Skin (& adhering fat)						
Trt. week 2	12	<0.01	2.8	-	0.8	-
3	15	-	3.2	-	0.4	-
4	18	<0.01	3.3	-	1.1	-
Wdrwl wk. 1	2.3	-	0.09	-	<0.05	-
2	<0.05	<0.01	<0.05	-	<0.05	-
3	-	-	-	-	-	-
4	0.1	<0.01	<0.05	-	<0.05	-
Control	<0.05	<0.01	<0.05	-	<0.05	-
Recovery, %[1]	87-92	46	87-92	-	87-92	-
Eggs						
Trt. week 1	0.1	<0.005	0.2	<0.005	0.3	-
2	0.4	<0.005	1	<0.005	0.4	-
3	2.7	0.006	1.3		0.5	-
4	6.9		1.2	<0.006	0.5	-
Wdrwl wk. 1	7.2	all <0.005	0.7	<0.005	0.3	-
2	0.6		0.06	<0.005	0.01	-

Sample Week of treatment or withdrawal	100 ppm		30 ppm		10 ppm	
	Sulphone	Aglycone	Sulphone	Aglycone	Sulphone	Aglycone
3	0.01		0.005	<0.005	<0.005	-
4	<0.005	<0.005	<0.005	<0.005	0.008	-
Control	<0.005		<0.005	<0.005	<0.005	-
Recovery, %	107[2]	65-97[1]	92[2]	102[2]	99[2]	-

[1] At 0.1 mg/kg of aglycone, 0.1 mg/kg each of sulphide, sulphoxide and sulphone

[2] At 0.1 mg/kg of aglycone, 0.01 mg/kg each of sulphide, sulphoxide and sulphone

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No information was provided.

In processing

The Meeting was informed that no information was available (Chabrol, 1994), although some results of citrus and tea processing were included with the supervised trials data.

Citrus fruit. Oranges from trials in California (Table 16) were processed with the results shown in Table 28.

Table 28. Fate of residues in processed Valencia oranges in California, 1969 (Ref. 78).

Sample	Residue, mg/kg, at application rate					
	2.5 kg ai/ha, 45 g ai/hl		5 kg ai/ha, 90 g ai/hl		21 kg ai/ha, 22.5 g ai/hl	
	Unwashed	Washed	Unwashed	Washed	Unwashed	Washed
Whole fruit	0.18	0.18	1.5	0.9		
Peel	0.34	0.3	7.1	1.6	2.8	
Peel after juice extraction						1.5
Ground peel		0.36		0.9		
Pulp	<0.1	0.1	<0.1	<0.1		
Pulp after juice extraction		<0.1		<0.1		
Pulp, dried and ground		1.7		4.2		
Dried pulp for cattle feed						2.8
Juice, simple extraction		<0.1		<0.1		
Juice, expressed		0.5		2		
Emulsion of essential oils		<0.1		<0.1		
Essential oils from fruit washings	20		34			

Sample	Residue, mg/kg, at application rate					
	2.5 kg ai/ha, 45 g ai/hl		5 kg ai/ha, 90 g ai/hl		21 kg ai/ha, 22.5 g ai/hl	
	Unwashed	Washed	Unwashed	Washed	Unwashed	Washed
Molasses		0.9		1.5		

The following approximate concentration/reduction factors for residues in oranges during processing can be derived from Table 28.

Orange	Factor
Unwashed peel/unwashed fruit	2-5
Washed whole fruit/unwashed fruit	0.6-1
Unwashed pulp/unwashed fruit	0.05-0.5
Molasses/washed whole fruit	2-5
Expressed juice/washed fruit	2.5
Extracted juice/washed fruit	<0.5
Dried ground pulp/washed whole fruit	5-10
Essential oils/washed whole fruit	40-100

Similar results were obtained for lemons and grapefruit.

Tea. Processing green tea to dry, manufactured tea concentrated residues of phosalone and its oxon about sixfold (Table 22), although high oxon controls make interpretation difficult. Residues of phosalone and its oxon in infusions of tea treated in accordance with GAP were about 12 and 10 ìg/l respectively.

Residues in the edible portion of food commodities

The Meeting was informed that no information was available (Chabrol, 1994), but the results of processing summarized above are relevant.

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

No information was provided.

NATIONAL MAXIMUM RESIDUE LIMITS

The following national MRLs for phosalone were provided to the Meeting.

Country	Crop(s)	MRL (mg/kg)
Australia*		
	Peaches	3
	Pears, apples	2.5
	Fat of meat and edible offal of goat, sheep	1
Austria		
	Fruit	2
	Cabbage	1
	Oilseed rape, cereals, potatoes	0.1
	Other crops	0.02
Belgium		
	Pip fruit, peaches, hops	2
	Other fruit	0.02
	Root and tuber vegetables, olives	0.1
	Other vegetables	1
	Others	0 (0.02)
Brazil		
	Peas, tomatoes, citrus	1
	Grapes	0.5
	Cotton, watermelon, wheat, dry beans, peanuts, soya beans	0.1
Canada		
	Artichokes	15
	Apricots (dried)	12
	Cherries	6
	Apples, plums (fresh prunes), grapes	5
	Peaches	4
	Pears	2
	Citrus fruit	1.5
Denmark		
	Apples, pears, peaches, plums, bananas, carobs, figs, kiwifruit, melons, passion fruit, pawpaws, persimmons, pineapples, pomegranates	2
	Citrus, strawberries, grapes, leafy vegetables, other vegetables (artichokes, asparagus, peas, cabbages, tomatoes)	1
	Potatoes, carrots, beetroot,black radish, celeriac, horseradish, Jerusalem artichokes, onions, radishes, swedes, turnips, vipersgrass	0.1
France		
	Citrus, strawberries, vegetables	1
	Pip fruit, peaches, grapes	2

Country	Crop(s)	MRL (mg/kg)
	Root vegetables, olives	0.1
	Other fruit and vegetables	1
Germany		
	Pome fruit, peaches	2
	Vegetables, other fruit	1
	Oilseed rape, bird rape	0.5
	Olives, root vegetables	0.1
	Other crops	0.05
Hungary		
	Pears	2
	Cherries, peaches, plums, apples, grapes, strawberries	1
	Oilseed rape, potatoes	0.1
Italy		
	Pome fruit, stone fruit, citrus, strawberries, grapes, vegetables	1
	Sunflower seeds	0.5
	Potatoes, sugar beet, fodder	0.2
	Cereals	0.1
	Olives, root vegetables	0.1
Japan		
	Tea	2
	Apples, pears, citrus, watermelons, melons	1
	Cucumbers, egg plants	0.5
	Potatoes	0.1
Luxembourg		
	Pip fruit, peaches	2
	Vegetables (root), olives	0.1
	Other fruit and vegetables	1
	Others	0.05
Netherlands (parent compound)		
	Pome fruit, peaches, hops	2
	Other vegetables and fruit	1
	Tuber and root vegetables, olives, oilseed rape 0.1	0.1
	Potatoes, caraway, sheep meat	0.05 (LOD)
	Sheep fat	0.5
	Other	0 (0.005)
South Africa		

Country	Crop(s)	MRL (mg/kg)
	Apples, pears	2
Spain		
	Vegetables, cereal grains, cotton seed	1
	Fruit	2
	Roots, potatoes, other	0.1
Switzerland		
	Fruit	2
	Cereals, rape	0.1
UK		
	Apples, peaches, pears, nectarines	2
	Beans, blackcurrants, brassicas, bananas, celery, citrus fruit, cucumbers, grapes, leeks, lettuce, mushrooms, onions, peas, plums, strawberries, raspberries, tomatoes	1
	Carrots, potatoes, swedes, turnips	0.1
USA**		
	Dried apple pomace	85
	Almond hulls	50
	Dried grape pomace	45
	Dried prunes	40
	Artichokes	25
	Raisins	20
	Apricots, cherries, nectarines, peaches, plums (fresh prunes)	15
	Citrus pulp	12
	Apples, grapes, pears	10
	Tea	8
	Citrus fruit	3
	Meat, fat and meat by products of cattle, goats, hogs, horses and sheep	0.25
	Potatoes, nuts	0.1

* No longer registered, but MRLs remain until withdrawn
** No current US food uses

APPRAISAL

Phosalone, a phosphorodithioate acaricide and insecticide, was evaluated in 1972, 1975 and 1976. Re-evaluation under the periodic review programme was scheduled for the 1993 JMPR. The JMPR periodic review of the toxicology was conducted in 1993, resulting in the ADI being lowered from 0.006 mg/kg bw to 0.001 mg/kg bw. The scheduled residue review was postponed until 1994 because data could not be provided in time for review by the 1993 Meeting.

The present periodic review was conducted on the basis of data from the manufacturer as well as information on GAP from Canada, Germany, The Netherlands and Spain and limited residue data from Germany and The Netherlands. In response to an inquiry, the manufacturer informed the Meeting that no information was available on the stability of stored analytical samples, the effects of processing, or the residues in the edible portions of foods.

Metabolism in animals is characterized by rapid elimination of phosalone and its metabolites in the urine and faeces, mainly in urine. While a high percentage of the residue has not been identified in most tissues, the available data indicate oxidation to phosalone oxygen analogue, cleavage of phosalone to yield *O,O*-diethyldithiophosphoric acid (and of the oxon to give the corresponding thiolic acid) and both phosalone and its oxon to 2-oxo-3-mercaptomethyl-6-chlorobenzoxazole (the thiol metabolite). Diethylthionophosphoric acid may be formed by direct oxidation of the dithiophosphoric acid and/or by hydrolysis of the oxon to the thiolic acid and immediate isomerization to the thiono acid.

The rapid elimination of ^{14}C in the urine and faeces of cows is consistent with findings in the rat and elimination via the urine in the later goat and pig metabolism studies. Although it was reported that essentially all the ^{14}C was eliminated in the urine, faeces and milk, no tissues were analysed. The fact that residues were found in milk suggested the possibility of low tissue residues. That would be consistent with the trace residues found in rat tissues and in the goat metabolism study. No reference was made to residues of the phosphorus-containing metabolites and their disposition.

The fact that higher residues were found in skimmed milk than in cream suggests that the main residues in milk were not phosalone or its oxygen analogue, since the octanol/water partition coefficient of phosalone indicates that it is fat-soluble. The residue partition in milk was confirmed in later goat metabolism studies.

In a metabolism study in goats 5 doses of [^{14}C]phosalone were given, each at about twice the level of the single dose in the two studies on cows. The specific activity in the goat study was comparable to the higher of the specific activities in the cow studies. The higher doses facilitated the identification and measurement of tissue and milk residues. For example, the total milk residues in the goat study were 5 times those in the cow studies.

About 80-97% of the total ^{14}C was organo-extractable. 59% of ^{14}C residue in the liver was identified, but only 6-36% in the other tissues and only 5.1% in the milk. The sulphide and sulphoxide metabolites accounted for approximately 30 and 21% of the total radioactivity in the liver. In fat, although only 18.4% was identified, approximately equivalent amounts of phosalone and the sulphide metabolite were reported and only slightly less of the sulphoxide. In muscle the sulphoxide was the major identified residue followed by the sulphide, and in milk the sulphoxide was the major compound identified.

The high percentage of ^{14}C excreted in the urine is consistent with that found in other animals. No reference was made to the fate of the phosphorus portion of the molecule nor to attempts, except by acid hydrolysis, at the further separation and identification of the unidentified residues (e.g. enzymatic hydrolysis). In several separative analytical steps significant levels of ^{14}C were counted in discarded fractions. No evidence was presented to support the suggestion that unidentified residues were water-soluble conjugates which would be reduced to undetectable levels by the end of a withdrawal period. It can be concluded that the fate of phosalone in goats has been partially elucidated.

The study of phosalone disposition in pigs after applications to the skin was too short to draw conclusions about the long-term storage potential. The limited exposure area would also not be sufficient to show the total potential exposure. It was sufficient to show that absorption through the skin is relatively slow.

The fate of phosalone residues in plants was investigated in potatoes, sorghum and apples. Although the available information suggests that plant metabolism is similar to that proposed for animals (formation of phosalone oxon, the aglycone, aglycone conjugates and chloroaminophenol), plant samples were not analysed for other metabolites found in animals (the thiol, sulphide, sulphoxide, sulphone or phenoxazone).

No evidence was provided in a potato translocation study to support the suggestion that ^{14}C was incorporated into normal plant constituents. The available information was not presented in such a way as to allow an independent conclusion on the nature of the reported residues (phosalone and its oxon). The overall evidence suggests little if any translocation from the surface 14 days after application, although residues became less extractable with time.

Sorghum metabolism studies showed decreasing residues of phosalone and increasing levels of identified and unidentified metabolites from the day of application to the harvest of mature plants. No significant translocation of residues was observed. There appears to be little likelihood of grain residues arising from early spray treatments (before flowering), but residues may occur from treatments at or after the flowering stage. The identified metabolites comprised phosalone, its oxon, the glycoside, aglycone and aminophenol. In foliage, grain and glume (seed head less grain) phosalone was the major identified residue, although after later stage applications the aglycone was a close second. These metabolites accounted at most for 53, 22 and 23% of the total residues in the foliage, grain and glumes respectively.

Over 90% of the residues in the grain from early applications and about half of that from applications at flowering was bound. There appear to have been no attempts to identify the substantial bound residues in this or other plant parts. Two major unidentified metabolites, phenolic in character, accounted for approximately 13% of the residue in foliage, grain and glumes. The remaining extractable residue (11% of the total) consisted of approximately 18 compounds, no one of which was more than 1% of the total residue. A follow-up report on the nature of the water-soluble metabolites in sorghum could not be reviewed as the even-numbered pages were omitted and photographs of TLC plates were not legible. The study should be submitted again for the next rescheduled review of phosalone.

While a significant account of information has been provided on the fate of residues in sorghum, approximately 50 to 75% of the residue has not been identified and, even of the named compounds, only the identities of phosalone and its oxon have been confirmed by more definitive methods such as GC-MS. The levels of the two major unidentified metabolites appear to be similar to those of phosalone and its oxon. Analytical standards of other potential metabolites, some of which have been identified in animal metabolism studies, appear not to have been used in the sorghum investigations. It can be concluded that the metabolism of phosalone in sorghum has been partially elucidated.

Up to 50% of the [^{14}C]phosalone applied to apple trees (apples plus leaves) was lost after 24 days, although the fate of the lost compound was not determined. No rainfall occurred during the test period. About 97% of the radioactivity associated with the apples was found in or on the peel and approximately 90% of that was extractable with methanol. About 90-94% of the peel residue was shown to be unchanged phosalone. Other products on the apples were not identified, although analyses of apple leaves revealed the presence of low levels of phosalone oxon (<3% of the leaf rinse or extract) and the aglycone 6-chlorobenzoxazolone (<5% of the leaf rinse or extract).

While CXLs have previously been established for a variety of fruits (citrus, pome fruit, stone fruit, small fruits and berries), vegetables (fruiting, tuber, root, leafy, brassica) and nuts, the fate of residues has only been partially elucidated in apples, sorghum and potatoes. The Meeting recommended that in any future studies to supplement the available information in support of continued or new phosalone limits, consideration should be given to determining the fate of residues in other types of crops such as a fruiting vegetable and/or leafy vegetables.

The fate of residues in soil shows similarities to that in plants and animals. A notable difference is the detection of low levels of the disulphide metabolite, $(C_2H_5O)_2PS\text{-}S\text{-}S\text{-}SP(OC_2H_5)_2$ in soil. The half-life of phosalone in soil is typically of the order of 2 days, but depends on the conditions. None of the usual leaching studies were conducted under field conditions nor were results of typical adsorption/desorption experiments available.

Numerous analytical methods have been developed for the determination of phosalone, its oxon and other metabolites in a variety of substrates including plant and animal products, soil and water. Most methods are based on extraction with polar organic solvents, liquid-liquid organic solvent partition and separation and clean-up on Florisil. Hydrolysis and derivatization are used for some metabolites. Most methods for plants emphasize the measurement of phosalone and its oxygen analogue and those for animal products include methods for the total sulphone metabolites and total chlorobenzoxazolone water-soluble metabolites in milk and tissues and for the free aglycone, free chloroaminophenol and free the thiol metabolite in milk. Determination is generally by GLC with EC and/or thermionic detection.

Generally the reported limits of "detection" for phosalone were 0.01-0.02 mg/kg for most crops (0.03-0.05 mg/kg for oily crops), and were usually higher for the oxon. The reports of analytical methods for plants and of general methods of analysis did not include sufficient information to permit an independent estimate of the limits of determination, but they could be independently estimated for those analytical methods for the parent and metabolites in animal matrices which were used in the animal feeding trials. These were validated by radiometric techniques and should be suitable for enforcement purposes.

Reasonable limits of determination would be 0.005 mg/kg for phosalone in milk and 0.02-0.05 mg/kg in animal tissues, 0.02 mg/kg for the oxon in milk, 0.1 mg/kg for the oxon in tissues and for the free aglycone, aminophenol and the thiol metabolite in milk, 0.05-0.1 mg/kg for total sulphones in milk and tissues, and 0.01-0.02 mg/kg for total benzoxazolone metabolites in milk and 0.02-0.05 mg/kg in tissues.

Data on residue trials were available for a variety of crops, but most of the substantial, relatively old, US data were only summaries and therefore of limited use. Phosalone is no longer registered for food uses in the USA. The remaining data were in many cased also quite old and often not in English, although in most cases English summaries from the individual reports were provided. Often the analytical procedures used were not specified and few representative chromatograms were provided, although control values and percentage recoveries often were. Frequently data summaries gave averaged results from biological and chemical assay methods. Where distinguishable, the monograph includes only the chemical results, as these would be more relevant to current standards and enforcement methods.

Fruits

Apples. Data were available from approximately 150 supervised trials in 10 countries, although mainly only summaries from trials before 1980, many of them in the 1960s. Some of the results were presented only as averages of analyses by GLC and biological methods. Because of the lack of detail and lack of confidence in the older results, the Meeting concluded that over two thirds of the data were not suitable for consideration (although all are summarized in the monograph). In particular, data from trials in Canada, Denmark, South Africa, Switzerland, the USA, and more than half of those in France were given little consideration. Even in other, generally more recent, studies sample chromatograms, control values, details of the analytical methods, and information on sampling-to-analysis intervals or storage conditions were provided in only a few cases. Often even if the information was supplied it was not in English, and no information was available on the storage stability of analytical samples or on processing.

In considering MRLs the Meeting placed most emphasis on the more recent French and German supervised trials, although the data could be closely matched to GAP only for 8 German trials with WP formulations which resulted in maximum residues of 1.3 mg/kg. The highest residues from approximately 20 French trials which approximated GAP for EC and WP formulations were 1.9 mg/kg for an SC formulation and 3.8 mg/kg for an emulsifiable solution formulation. No relevant French GAP was reported for formulations other than WP and EC, although the application rates in the French SC trials approximated SC GAP in The Netherlands.

The available data suggest that the CXL of 5 mg/kg for apples would not be exceeded. However, because critical supporting information (e.g. interval from sampling to analysis, storage stability data etc.) for the German and French trials was generally lacking, the Meeting could not with confidence confirm this limit and recommended its withdrawal. Future consideration would require critical supporting information and preferably additional, more recent, data from trials according to GAP.

Pears. The data were summaries of trials in the 1960s, almost none of which were according to reported GAP. Many of the analyses were by colorimetric or biological methods. The Meeting concluded that the data were not suitable for estimating a maximum residue level and recommended withdrawal of the 2 mg/kg CXL for pears.

Summary data on apricots without relevant GAP gave an insufficient basis for recommending an MRL.

Cherries. Data were available from supervised trials in four countries and residues were 6.3 mg/kg in trials which approximated GAP. However, because data from Canada, France and the USA were from pre-1971 trials and mostly summaries with few details of the trials, analyses, or sample handling and storage, the Meeting did not consider them suitable for recommending or confirming MRLs by current standards. Residues in the French trials were determined by colorimetric or biological methods. While more detail was available for some of the 1973-75 German trials (maximum residue 1.1 mg/kg from trials according to GAP), important supporting information (e.g. interval from sampling to analysis, stability of stored samples) was often lacking. The Meeting recommended withdrawal of the current 10 mg/kg CXL.

Data were available from supervised trials on peaches in 4 countries, although older data from the USA and Canada (and some of the French data) had too few details to serve as a basis for recommending MRLs. Summary data for apricots and nectarines were not suitable for supporting the data on peaches.

A total of about 5 supervised peach trials from two countries reasonably reflect GAP and are acceptably documented. Residues in the better documented, more recent, French trials which approximated GAP were 3 mg/kg. Residues were 0.13 mg/kg in the Italian trial which was also relatively well described. Although the storage conditions for the analytical samples were adequate in the French and Italian trials (for periods of 8-9 months and 2-3 months respectively) no storage stability data were provided, and sample chromatograms were available only for the Italian trial. While the available data suggest that the 5 mg/kg CXL for peaches may not be exceeded, the Meeting did not consider that the adequately reported results were sufficient to support a limit and recommended its withdrawal. Future consideration would require additional well-documented data from trials according to GAP, with information on intervals and storage conditions from sampling to analysis and on the storage stability of analytical samples.

Although data were available from over 30 supervised trials on plums in five countries, all but the most recent (two 1977 German trials) were mostly summaries with few details of critical supporting information such as sample chromatograms, method validation, plot sizes, sampling to analysis intervals, and stability on storage. Information on sample handling and storage conditions was provided in only a few cases. Much of this supporting information was lacking even for the German trials, although analytical storage conditions were described. While the substantial quantity of data (maximum residue 4.1 mg/kg from an application according to GAP) suggests that the CXL of 5 mg/kg would not be exceeded by treatments according to current GAP, because of the shortcomings in the data the Meeting could not confirm it and recommended that the CXL should be withdrawn.

While data on grapes were available from 55 supervised trials in 7 countries, the data from South Africa and Italy could not be related to reported GAP. Most of the remaining data were summary reports from trials in 1970 or earlier. Generally there is a lack of detailed information, although in a few cases the analytical storage conditions were described. Critical information such as intervals from sampling to analysis, storage stability data, control values and details of analytical methods (control values, sample chromatograms) was generally missing, although in most cases laboratory storage conditions were provided. With a few exceptions, studies did not include information on plot sizes. There was no information on residues in processed products.

Summary data from trials in France (1964) and Switzerland (1966) in accordance with reported GAP indicate maximum residues of 0.4 and 4.2 mg/kg respectively, but residues were determined by outdated colorimetric methods. Because most application rates were exaggerated, only one of the 25 US trials could be related to Canadian GAP: the trial was in 1964 and showed a maximum residue of 2.6 mg/kg. Canadian trials in 1970 in accordance with GAP yielded a maximum residue of 5 mg/kg. Three of four German trials in 1974 or 1975 approximated GAP, with a maximum residue of 1.6 mg/kg.

The available data suggest that the CXL of 5 mg/kg for grapes would not be exceeded from current uses. However, because most of the data were from old summary reports and generally lacked the detail required by current standards, the Meeting could not with confidence confirm the CXL and recommended that it should be withdrawn.

Limited summary data were available on citrus fruits from supervised trials in 1966-69 on oranges (5 trials), lemons (2) and grapefruit (1) in the USA. Information on GAP was available for Spain, Thailand and Japan but it could not be compared with most of the trials data. The summary data were not accompanied by trial details, sample handling and storage conditions or intervals, information on storage stability, or other critical supporting information. The Meeting was unable to confirm the CXL of 1 mg/kg for citrus fruits on the basis of the available information and recommended that it should be withdrawn.

The highest residues in strawberries were 1.4 mg/kg in the one German trial using a WP formulation according to Austrian GAP for EC formulations and <0.1 mg/kg in US trials after 17 days at similar rates with an EC formulation. Residues were <0.1 mg/kg in the single UK trial after 26 days. One trial on raspberries in the USA according to the GAP of European countries resulted in residues of <0.1 mg/kg. Because only summary reports were available, critical supporting information was lacking, and a good match could not be made between the limited data and GAP, the Meeting could not with confidence confirm the CXL of 1 mg/kg for strawberries and recommended that it should be withdrawn.

Vegetables

Artichokes. There were data from two 1968 French trials and one 1972-3 United States trial, without relevant information on GAP. There was therefore no basis for recommending a limit for artichokes.

Beans. There were data from two 1968 French trials and one 1965 United States trial and again no relevant information on GAP. The Meeting could not estimate a maximum residue level.

Two results (0.2 mg/kg) from two very old trials on beetroots in one country were not enough to support a limit. The Meeting recommended withdrawal of the CXL of 2 mg/kg.

Broccoli. There was no US GAP and only relatively old summary data. There is therefore no basis for retaining the current CXL.

Marginally sufficient data on Brussels sprouts were from trials at the GAP rate and PHI of The Netherlands. Maximum residues of 0.6 mg/kg from the use of WP formulations in accordance with Netherlands GAP for SC formulations suggest that the current 1 mg/kg CXL would not be exceeded. However, without any data for the SC formulation for comparison with the supervised trials data for WP formulations, the Meeting could not with confidence confirm the current CXL and recommended that it should be withdrawn.

Cabbages. There was no US GAP, only summary US data and only two results (France, 1968, green cabbage 0.3 and 1.2 mg/kg) which reflected GAP. The Meeting concluded that the data were insufficient to confirm the current 1 mg/kg CXL and recommended that it should be withdrawn.

Because there were only two results from trials on cauliflowers in one country with summary data from another and relevant information on GAP was lacking, the Meeting concluded that the available data were too limited to recommend an MRL.

Cucumbers. There were summary data from one country, two trials from another (with residues of 0.02 mg/kg from applications according to GAP) and information on GAP of questionable use from two others. The Meeting concluded that this was not a sufficient basis to confirm the CXL of 1 mg/kg and recommended that it should be withdrawn.

Although there were substantial data on lettuce, only two of 15 trials included more than summary information. Only two results (the higher at 0.7 mg/kg) could be related to French GAP. The Meeting recommended that the 1 mg/kg CXL for head lettuce should be withdrawn.

Limited data on melons and watermelons and the lack of relevant information on GAP precluded the estimation of a maximum residue level.

Because no residue data were provided in support of the 1 mg/kg CXL for peas the Meeting recommended that it should be withdrawn.

Only two old supervised trials from France on potatoes (residues <0.05 mg/kg) could be strictly related to GAP, although in 12 additional old trials in Australia and Canada residues were well below 0.1 mg/kg from application rates which were often much higher than the GAP rate of France. Although the more substantial US data also showed residues below 0.1 mg/kg and usually below 0.03 mg/kg, this information was of limited use because only summaries were provided and because there is no GAP in the USA. The Meeting considered the limited old data insufficient to support an MRL and recommended withdrawal of the current CXL (0.1* mg/kg).

There were data from 3 supervised trials on spinach in one country without any relevant information on GAP an insufficient basis to propose an MRL for spinach.

Data on tomatoes from 26 supervised trials in 1972-73 in the USA were of limited use because only summaries were available and the trials were not according to GAP. Maximum residues were 0.9 mg/kg in four Indian trials approximating GAP or 0.3 mg/kg in two French trials according to Spanish GAP. Because data from trials according to GAP are limited (4-5 trials) for such a major crop, the Meeting recommended withdrawal of the CXL of 1 mg/kg. The Meeting also noted the need for processing studies if a limit should be proposed in the future.

Other crops

Although summary data from 7 US trials on cotton seed in 1964-6 with the residues determined colorimetrically indicated that there were no residues (<0.1 mg/kg) in delinted or non-delinted cotton seed, meal or crude oil, the lack of information on GAP and of critical supporting information precluded the estimation of a maximum residue level.

Summary data on hops from two 1966 trials with colorimetric determination of the residues in one country suggested that residues would not exceed 0.1 mg/kg in dry cones or 2 mg/kg in green cones, but again the lack of information on GAP and on supporting details precluded confirmation of the 2 mg/kg CXL for dry hops. The Meeting recommended that it should be withdrawn.

Summary data on nuts from 1967-70 supervised trials in the USA suggest that residues of phosalone in the meats of almonds, hazelnuts, walnuts and pecans may not exceed 0.1 mg/kg from applications roughly approximating European GAP, if the linking of US trials to European GAP is justified. No data were available for chestnuts to confirm the current 0.1 mg/kg CXL and no GAP was provided for pecans. Because only summary data were available without critical supporting information and because European GAP could not be closely matched with the conditions of the field trials, no limits could be recommended for almonds, hazelnuts, or walnuts. Because no GAP was provided for pecans the CXL of 0.1 mg/kg could not be confirmed even if supporting critical information had been provided. The Meeting therefore recommended that the CXLs of 0.1 mg/kg for chestnuts and pecans should be withdrawn.

Summary data from one 1964 trial on sorghum in the USA with colorimetrically determined residues and from three 1967 trials on wheat in France with physico-bioassay analyses did not provide a suitable basis for the Meeting to estimate a maximum residue level for either commodity.

Oilseed rape. The results of two 1974 German trials, one 1975 Polish trial, five 1966 French trials and two 1968-9 Danish trials according to GAP suggest that residues in oilseed rape from authorised treatments would not exceed the 0.1 mg/kg CXL. One German trial according to GAP in 1974 with residues up to 0.4 mg/kg was an exception. The French data suggest residues in the oil of <0.5 mg/kg.

While the quantity of data is adequate, the Meeting noted with concern that only summaries were available for the French trials, and in 5 of the 7 trials residues were determined by outdated colorimetric and bioassay methods. Residues were also determined partly by bioassay in one of the three Danish trials (1968) which was again only reported in summary form. The other two included more trial details, but not other critical information such as sampling-to-analysis intervals and storage stability. The Polish trial report was also a limited summary. Only the reports of the German trials gave substantial trial details, but even these did not include important supporting information such as the interval from sampling to analysis, sample chromatograms, storage stability information or more than a citation of the analytical method used.

Because only the results of the three German trials could be used with some confidence, because one of these trials suggested that residues could exceed 0.1 mg/kg from applications according to GAP and because even in these trials important supporting information was lacking, the Meeting could not support the current 0.1* mg/kg CXL and recommended its withdrawal.

Four Indian and one Pakistani trials on tea according to GAP indicate that residues of phosalone and phosalone oxon in green tea would not exceed 0.5 and 0.1 mg/kg respectively after 7 days or 0.06 and 0.1 mg/kg after 10 days under higher rainfall conditions. Although data reflective of GAP for green leaves under dry weather conditions were not available, a comparison of residues in processed tea from trials according to GAP in wet and dry weather conditions clearly shows significantly higher residues from applications in dry weather. Residues of phosalone/phosalone oxon on processed (dry) tea would not be expected to exceed 7.2/1.1 mg/kg after 8 days in dry weather or 0.09/0.05 mg/kg after 10 days in wet weather. The concentration factor for green tea to dry tea is approximately 6. Residues of phosalone/phosalone oxon in beverage tea infused from dry tea could be expected to be of the order of 12/10 µg/l.

Although the data were old and somewhat limited, the Meeting considered that most of the studies were among the better documented phosalone residue trials and that they might provide a basis for recommending a limit for tea (dry). From the available data the Meeting would not expect residues in dry manufactured tea to exceed 10 mg/kg from GAP applications. However, the main concern is the lack of information on the interval from sampling to analysis and on the stability of the analytical samples during storage. The Meeting was also concerned by the lack of information on the Indian PHI, since most of the data were from Indian trials. On the weight of the evidence the Meeting agreed not to estimate a maximum residue level for tea at present.

Animals

MRLs have not previously been recommended for phosalone in animal products except 0.5 mg/kg in sheep fat and 0.05 mg/kg (limit of determination) in sheep meat resulting from veterinary use, which have now become CXLs. Because no current information was provided for the periodic review of this use, the Meeting recommended that these limits should be withdrawn.

Feeding trials have been conducted with dairy cattle and chickens. In cows, a trial at a dietary feeding level of 100 ppm indicated that the total residues of phosalone and its oxon and sulphone metabolites are unlikely to exceed 0.3 mg/kg in the liver or 0.05 mg/kg in other tissues at this feeding level.

A study on chickens showed sulphone residues in all tissues and eggs. At the lowest feeding level of 10 ppm, total sulphone metabolite residues were of the order of 0.5 to 1 mg/kg in all tissues except kidney where the maximum was 0.1 mg/kg. However, the results at the highest feeding level of 100 ppm suggest that residues might not have reached their maximum in eggs and skin by the end of the 4-week feeding period. Because parts of the study were missing in the submission the Meeting could not draw any final conclusions. It recommended that the complete study be again submitted with any future submissions to the JMPR on phosalone.

The crops with current CXLs most likely to contribute to phosalone residues in animal tissues or milk include apples (CXL 5 mg/kg), citrus fruits (1 mg/kg), tomatoes (1 mg/kg) and perhaps grapes (5 mg/kg). Limited information on citrus processing indicates a concentration factor of 5-10 from fruit to dry pomace. No data were provided on residue levels in the dry pomace of apples, grapes or tomatoes.

Because the data were insufficient to confirm the CXLs for raw agricultural products used as animal feed (or for feed items that could be derived therefrom) the Meeting was unable to estimate maximum residue levels for animal products.

No information was available on the fate of residues in storage, in stored analytical samples, or in foods in commerce or at consumption. Only limited data were available on processing and on residues in the edible portion of food commodities (citrus, tea).

The Meeting recommended the withdrawal of the existing MRLs, as shown below.

RECOMMENDATIONS

Owing to inadequacies in the available information the Meeting concluded that the existing CXLs for phosalone, listed below, should be withdrawn.

Commodity		Recommended MRL or ERL (mg/kg)	
CCN	Name	New	Previous
FP 0226	Apple	W[1]	5
VR 0574	Beetroot	W	2
VB 0400	Broccoli	W	1
VB 0402	Brussels sprouts	W	1
VB 0041	Cabbages, head	W	1
FS 0013	Cherries	W	10
TN 0664	Chestnuts	W	0.1*
FC 0001	Citrus fruits	W	1
VC 0424	Cucumber	W	1
FB 0269	Grapes	W	5
DH 1100	Hops, dry	W	2
VL 0482	Lettuce, head	W	1
FS 0247	Peach	W	5
FP 0230	Pear	W	2
VP 0063	Peas	W	1
TN 0672	Pecan	W	1
FS 0014	Plums (incl. prunes)	W	5
VR 0589	Potato		

Commodity		Recommended MRL or ERL (mg/kg)	
CCN	Name	New	Previous
SO 0495	Rape seed	W	0.1*
MF 0822	Sheep fat	W	0.1*
MM 0822	Sheep meat	W	0.5 V
FB 0275	Strawberry	W	0.05* V
VO 0448	Tomato	W	1

* At or about the limit of determination
[1] Withdrawal recommended

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