PHOSALONE

EXPLANATION

Phosalone, a phosphorodithioate acaricide and insecticide, was re-evaluated by the 1994 JMPR in the CCPR Periodic Review Programme. That Meeting concluded that the existing CXLs for phosalone should be withdrawn, owing to inadequacies in the available information on storage stability and the effects of processing. The 1996 CCPR was informed that new residue data on apples, citrus fruits, grapes and potatoes would become available in 1999, and decided to maintain the CXLs for these commodities for four years pending the evaluation by the 1999 JMPR. The 1999 CCPR recommended withdrawal of the CXLs for citrus fruits, grapes and potato, however, as they were no longer supported. The toxicology was reviewed at the 1997 JMPR which allocated an ADI of 0.02 mg/kg bw.

The Meeting received new or revised information on physical and chemical properties, metabolic and environmental fate, analytical methods, stability of analytical samples, use patterns, supervised trials, effect of processing apples, and national MRLs.

Physical and chemical properties

The Meeting received the revised information on physical and chemical properties shown below.

Pure active ingredient

Octanol/water partition coefficient:	log P_{ow} = 4.01 at 20°C (Cousin, 1995)
Solubility:	in water 1.4 mg/l at 20°C (Cousin, 1997a)
Photolysis:	in water at pH 5, decomposition is very rapid (half-life 15-20 min) (Laurent *et al.*, 1977). The quantum yield (Φ_{300}) at 300 nm for phosalone in aqueous solution was determined to be 0.19 (Boinay, 1994).

Technical material

Purity:	930 g/kg (minimum)
Melting range:	42 to 48°C
Specific gravity:	1.338 g/ml at 20°C (Pollard, 1987)
Vapour pressure:	4.57 x 10^{-7} mn Hg at 25°C (Hoffman, 1989)
Solubility:	in organic solvents at 20°C: (Cousin, 1997a) acetone >1000 g/l dichloromethane >1000 g/l ethyl acetate >1000 g/l n-heptane 26.3 g/l toluene >1000 g/l methanol >1000 g/l

n-octanol 266.8 g/l

Stability: Not highly flammable, not autoflammable, not explosive (Fillion, 1997), does not have oxidizing potential (Cousin, 1997b)

Formulations Emulsifiable concentrate, wettable powder, flowable

METABOLISM AND ENVIRONMENTAL FATE

Plant metabolism

The Meeting received studies on apples (Kimmel *et al*., 1990) and grapes (Periasamy *et al*., 1995).

Apples. Apple trees were brushed with [*phenyl*-^{14}C]phosalone on the surface of individual leaves and fruits at a rate equivalent to 3.3 - 3.5 kg ai/ha, grossly in excess of the recommended application rate. Single applications were made at different times to each of two trees. On the first tree the chemical was applied to all immature fruits (3.8 cm diameter) and to leaves on 3 selected branches, and samples were taken 14 days after application. On the second the compound was applied to all fruits (6.5-7.5 cm diameter; about 4 weeks before maturity) and leaves on three selected branches and samples were taken 14 days and 24 days after application. Only the final samples were analysed because the later harvest would be likely to identify more extensive metabolism. Apples and leaves were rinsed, and extracted with methanol. The extractable residues were characterized by TLC and HPLC.

In the leaves, 57% of the total radioactive residue (TRR) was contained in the rinse fractions with 43% in the homogenized leaf (Table 1). In contrast, the apple fruits showed <1% of the radioactivity in the rinse fraction, 1-2% in the pulp, and 97-98% in the peel.

Table 1. Total radioactive residues in apple leaves and fruits.

Treatment	Sample		^{14}C, mg/kg as phosalone	% of TRR
Application to immature fruit (Treatment I)	Leaf	Rinse	511	57
		Leaf homogenate	389	43
		TRR	**900**	**100**
	Fruit	Rinse	0.21	0.26
		Peel	80	99
		Pulp	0.63	0.78
		TRR	**80.84**	**100.04**
Application to near mature fruit (Treatment II)	Leaf	Rinse	453	59
		Leaf homogenate	318	41
		TRR	**771**	**100**
	Fruit	Rinse	0.16	0.27
		Peel	58	98
		Pulp	0.87	1.5
		TRR	**59.03**	**99.77**

Table 2 shows the efficiency of the methanol extraction.

Table 2. Recovery of [^{14}C]phosalone from apples.

Sample	Treatment I	Treatment II

	Methanol extractable, %[1]	Unextractable, %[1]	Methanol extractable, %	Unextractable, %
Apple leaf	99	3.4	92	8.4
Apple peel	90	8.2	89	3.2
Apple pulp	73	NA[2]	104	4.9

[1] Based on total dpm determined by combustion of sample before extraction
[2] Not analysed

Phosalone was only metabolized to a limited extent. Residues were quantitatively extracted and phosalone accounted for 75-92%. Phosalone oxon and 6-chlorobenzoxazolone were found at low levels (2-7%) in leaf extracts and rinses (Table 3).

Table 3. Distribution and identity of residues in apples following application of [^{14}C]phosalone.

Sample	Treatment I		Treatment II	
	% of TRR	mg/kg as phosalone	% of TRR	mg/kg as phosalone
Leaf rinse	100	511	100	453
Phosalone	86	441	83	376
Phosalone oxon	3.5	18	2.2	10
6-chlorobenzoxazolone	4.5	23	6.4	29
Unknowns	3.7	19	5.1	23
Polar products	2.2	11	3.3	15
Leaf extract	100	376	100	291
Phosalone	88	329	75	219
Phosalone oxon & 6-chlorobenzoxazolone	6.9	26	5.6	16
Polar products	5.6	21	19	56
Peel extract	100	73.1	100	55.8
Phosalone	92	67	88	49
Unknowns	2.3	1.7	3.9	2.2
Polar products	6.0	4.4	8.2	4.6
Pulp extract	not analysed		101	0.83
Phosalone			51	0.42
Unknowns			17	0.14
Polar products			33	0.27

Grapes. Grape bunches on two vines were treated with phenyl-labelled [^{14}C]phosalone at the equivalent rate of 2.1 kg ai/ha, either as a single application 23 days before harvest or as two applications each at 1.05 kg ai/ha 23 and 9 days before harvest. This was highly atypical practice, resulting in exaggerated application. The spray was directed on the bunches of grapes, with foliage held away from the bunches. After harvest, grapes were rinsed and separated into juice and pulp, which were analysed. Samples were characterized by TLC and HPLC. Polar fractions in the juice and pulp were analysed by derivatization and chemical and enzymatic hydrolysis. The limit of detection for combustion radioassay and for HPLC analysis of the solvent-extractable residues was <0.01 mg/kg.

Most of the radioactivity was found in the pulp with very low amounts in the rinse or juice There was no difference in distribution between the two application schemes (Table 4).

Table 4. [^{14}C]phosalone residues in grapes.

Sample	1 application		2 applications	
	mg/kg as phosalone	% of TRR	mg/kg as phosalone	% of TRR
Rinse	0.51	1.8	0.45	1.6

Juice	0.87	3.1	0.70	2.6
Pulp	26	95	26	96

The results of the analysis of the rinses, juice and pulp are shown in Table 5. The parent phosalone was the major residue in each fraction. Examination of the more polar group of metabolites in the juice and pulp samples by chemical and enzymatic hydrolysis indicated that the metabolites were mainly glycosides.

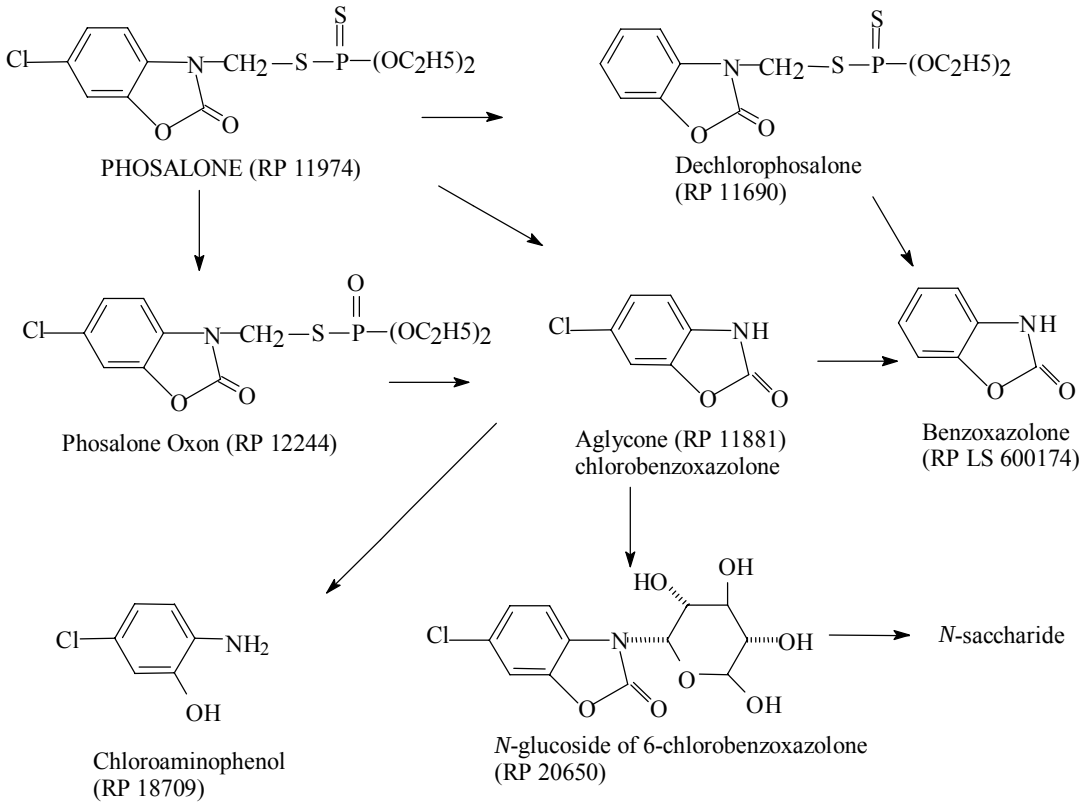
Table 5. Distribution of [^{14}C]phosalone and its metabolites in grapes.

Compound	Rinse		Juice		Pulp		Total	
	mg/kg[1]	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]	% of TRR	mg/kg[1]	% of TRR
1 Application								
Phosalone	0.18	0.65	0.39	1.4	24	86	24.6	88
Phosalone oxon	0.14	0.5	0.016	0.06	0.19	0.68	0.35	1.3
Benzoxazolone			0.019	0.07	0.01	0.04	0.029	0.1
6- chlorobenzoxazolone	0.007	0.03	0.005	0.02	0.008	0.03	0.02	0.07
2-amino-5-chlorophenol					0.027	0.1	0.027	0.1
Dechloro-phosalone					0.055	0.2	0.055	0.2
N-glucoside of chlorobenzoxazolone			0.057	0.21	0.036	0.13	0.093	0.33
N-saccharide of chlorobenzoxazolone			0.17	0.61	0.031	0.11	0.2	0.72
Unknowns A-1			0.061	0.22	0.013	0.05	0.074	0.27
Unknowns A-2					0.048	0.17	0.048	0.17
2 Applications								
Phosalone	0.06	0.22	0.33	1.2	27	100	27.4	102
Phosalone oxon	0.17	0.63	0.034	0.13	0.34	1.3	0.54	2.02
Benzoxazolone	0.005	0.02	0.033	0.12			0.038	0.14
6- chlorobenzoxazolone	0.009	0.03			0.049	0.18	0.058	0.22
2-amino-5-chlorophenol					0.019	0.07	0.019	0.07
Dechloro-phosalone					0.028	0.10	0.028	0.10
N-glucoside of chlorobenzoxazolone			0.049	0.18	0.024	0.09	0.073	0.27
N-saccharide of chlorobenzoxazolone			0.08	0.30	0.02	0.07	0.100	0.37
Unknowns A-1			0.029	0.11	0.008	0.03	0.037	0.14
Unknowns A-2					0.032	0.12	0.032	0.12

[1]As phosalone

The metabolism of phosalone in grapes involves dechlorination, oxidation, hydrolysis and conjugation. Several potential metabolites were specifically looked for but not found. These included phenoxazone, 6-chloro-3-mercaptomethylbenzoxazolone, 6-chloro-3-methylsulfinylmethylbenzoxa - zolone, 6-chloro-3-methylthiomethylbenzoxazolone, 6-chloro-3-methylsulfonylmethylbenzoxa- zolone, and other dephosphorylated derivatives of chlorobenzoxazolone.

Figure 1. Proposed metabolic pathways of phosalone in plants.



Environmental fate in soil, water and air

Photolysis in water

Laurent *et al*. (1977) studied the photodegradation of phosalone at a concentration of 1 mg/l in water buffered at pH 5 at 25°C. Phosalone is most stable to hydrolysis at pH 5 (less than 10% degradation after one month). The light source was a mercury vapour arc, with wavelengths below 290 nm filtered out. The power in the UV band (290-400nm) was about 9 W. Degradation kinetics were examined in both distilled water and water buffered to pH 5 using technical phosalone (not radiolabelled), with levels determined by GLC with an ECD. Phosalone was found to decompose very rapidly, with a half-life of 15-20 minutes in both distilled water and the buffered solution. The addition of acetone (2%) reduced the degradation rate by a factor of 1.5 to 2 but also changed the quantity and nature of the degradation products.

Photolytic degradation products were determined using [^{14}C]phosalone in buffered solution (pH 5) without acetone. Samples were irradiated for times estimated to result in 25%, 50% and 75% degradation. The irradiated solutions were extracted with dichloromethane and ethyl acetate. The overall recovery was >96%. In the solution with 75% degradation (confirmed by GLC analysis) 0.25% of the radioactivity was recovered in the traps for volatiles and 4.4% from the residual aqueous phase.The organic extract contained 92.2% of the applied radioactivity as phosalone, hydroxy-phosalone, and about 20 other degradation products none of which exceeded 5% (0.05 mg/kg as phosalone equivalents). Analyses were by TLC in various systems with liquid scintillation counting (LSC) and confirmation of identities by IR, MS and NMR.

Samples in buffer solution with acetone were irradiated for times estimated to result in 25%, 40% and 68% degradation. The overall recovery was >85%. In the solution with 68% degradation (confirmed by GLC) 14.5% of the radioactivity was recovered in the volatile traps and 20.7% from the residual aqueous phase. The minor products hydroxy-phosalone, phosalone oxon and 6-

chlorobenzoxazolone were found in the organic extract, which contained 50.5% of the applied radioactivity.

Boinay (1994) reported the quantum yield at 300 nm of the direct photolysis of phosalone in aqueous solution, determined with an actinometer. The concentration of the test solution was 5.21 mg/l (1.409 x 10^{-5} mol/l). The Δn and Na values were calculated to be 2.751 x 10^{-9} mol/sec and 2.08 x 10^{-8} Einstein/l/sec for irradiation for 0-0.5 h in the first assay, giving a calculated quantum yield of 0.13. In the second assay, Δn and Na were calculated to be 2.273 x 10^{-9} mol/sec and 9.185 x 10^{-9} Einstein/l/sec and the quantum yield (Φ_{300}) was 0.25, giving a mean value of 0.19.

Maestracci (1995) calculated the environmental photolytic half-life of phosalone in a natural aquatic system. The molar extinction coefficient in the range 292.5-800 nm was determined by Jendrzejczac (1994). The half-life of phosalone in a natural aquatic system in Europe (52° north latitude) was calculated to range from 49 hours in July to 1354 hours in December.

Photolysis in air

Maestracci (1994) estimated the rate of photochemical transformation of phosalone in the troposphere, the main reaction being between hydroxy radicals and the phosphorus-containing group. The estimated reaction constant at 298°K was 9.34 x 10^{-3} s^{-1} which corresponds to a half-life of about 74 daylight seconds.

Biodegradability

Muttzall and Hanstveit (1989) reported the inherent biodegradability of phosalone which was determined in the modified Sturm test (OECD-TG 301B, 1981) during a six-week period in an activated sludge taken from an oxidation ditch. Because of the low water solubility of phosalone, [^{14}C]phosalone, applied at concentrations of 1 and 2 mg/l, was used in addition to unlabelled phosalone in order to measure the degradation adequately. Radioactivity in the CO_2 traps was determined at 1, 2, 3, 4, and 6 weeks. About 20% of the initial radioactivity were detected as $^{14}CO_2$ at the end of the sixth week.

Aerobic degradation in soil

Skinner and Jao (1995) reported the aerobic degradation of [^{14}C]phosalone in German standard soil 2.2 (a loamy sand, pH 5.6, 0.62% organic matter). Phosalone was applied at an initial concentration of 1 μg/g soil (equivalent to 1 kg ai/ha) and samples were maintained in the dark at 20 ± 2°C at 40% of maximum water holding capacity for up to 45 days. The overall recovery of ^{14}C was 99.8 ± 2.9%. Phosalone was degraded rapidly with a half-life of 2.9 days. Production of $^{14}CO_2$ continued throughout the sampling and CO_2 was the most abundant single product, representing 4% of the dose. The total of all degradation products in the extract accounted for 5.0% of the applied dose. Phenoxazone was observed only in day 30 and day 45 extracts and represented ≤ 0.7% of the applied dose, but its presence indicates the transient existence of its precursors in the degradation pathway. Some minor products, 6-chlorobenzoxazole, 2-amino-5-chlorophenol and phosalone oxon, were detected, all representing ≤2% of the applied dose. Unextracted radiocarbon increased to an average of 84.7% by day 30, then decreased to 80.6% at 45 days. Bound residues were characterized; none of the individual components in the fulvic acid, hymatomelanic acid, or humin fractions accounted for more than 10% of the applied dose.

The rate of aerobic degradation of [^{14}C]phosalone was investigated at an initial concentration of 1 mg/kg (1 kg ai/ha) in four soils from Germany, one of which was used to establish the degradation pathways in aerobic soil. Soil samples were maintained in the dark at 20 ± 2°C and at 40% of maximum water holding capacity Incubation was continued until two consecutive samplings showed that less than 10% of the applied phosalone remained intact, which was between 42 and 45

days. All samples were assayed by LSC, extracted, and the residues characterized and quantified by TLC and HPLC. Phosalone was found to be degraded rapidly in all four soils (Table 6).

Table 6. Degradation rates of phosalone in four soils under aerobic conditions.

	DT-50 (days)	DT-90 (days)
Standard Soil 2.1	4.1	16.5
Standard Soil 2.2	2.9	30
Standard Soil 2.3	0.8	19.2
German Field Soil	0.8	13.3

At the final sampling the largest single component in the combined initial extracts in any of the soils accounted for 4.7% of the applied dose (on day 28 in Standard Soil 2.1). Phenoxazone was detected by HPLC in all three soils but did not exceed 1.5% of the applied dose. Radioactivity from the three precursors of phenoxazone represented a total of ≤2.0%. CO_2 accounted for 3.5 to 5.9% of the applied dose at the end of the study. The unextractable radiocarbon increased over time, rising to between 74.4 and 84.6% of the applied dose. No organic volatiles were found at any time (Table 7).

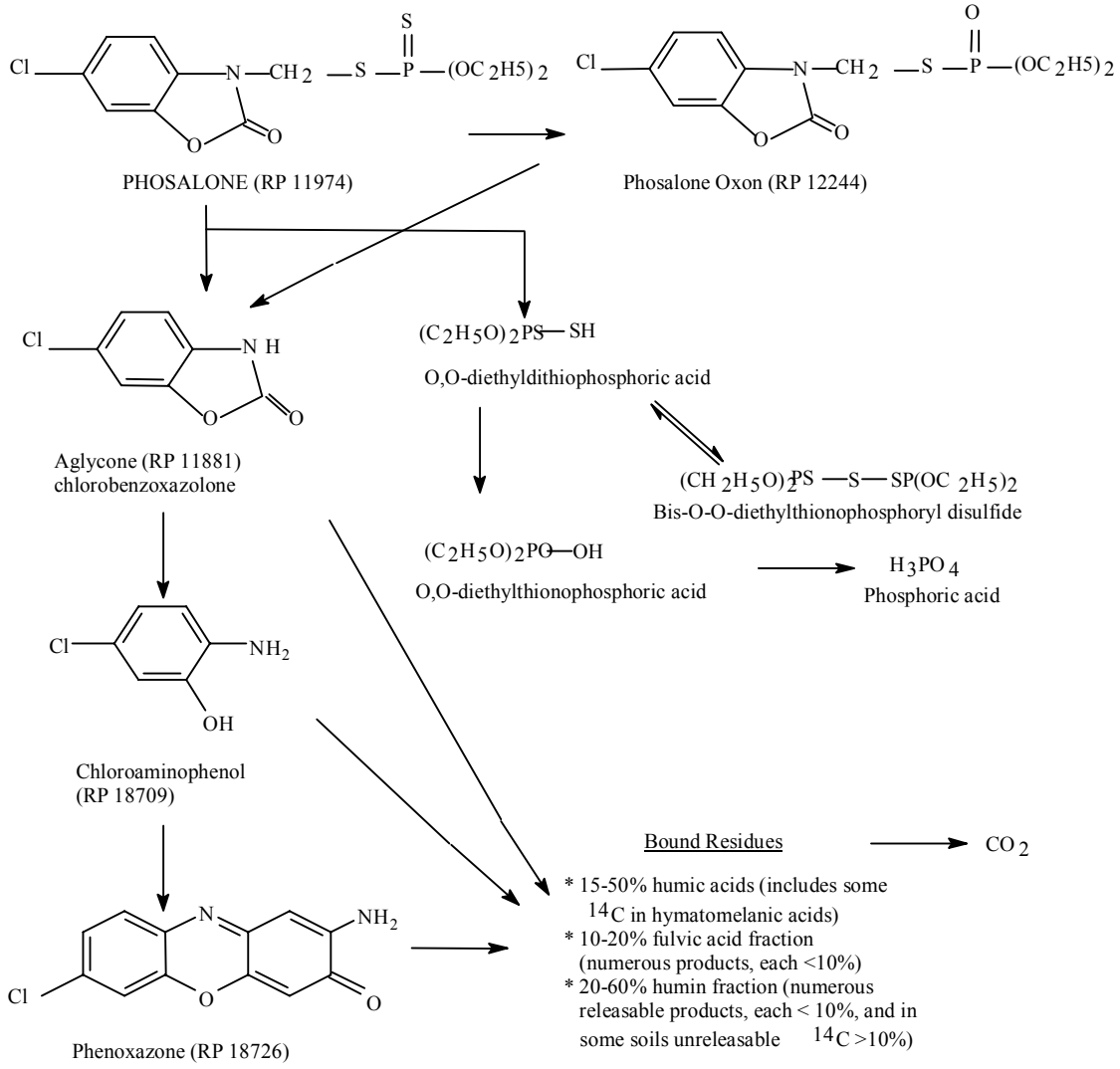
Table 7. Distribution of radioactivity in soil after 45 days.

	% of applied dose					
Soil	Phosalone	Other extracted	Unextracted	$^{14}CO_2$	Other volatiles	Total recovery
Speyer 2.1	4.3	14.8	74.4	3.5	0.0	97.0
Speyer 2.2	7.4	5.0	80.6	4.0	0.0	96.9
Speyer 2.3	5.6	7.8	76.0	4.8	0.0	94.2
Sandy Loam	4.5	3.9	84.6	5.9	0.0	98.9

Unextracted soil residues from the last sampling were characterized. The results were similar to those reported in the degradation study with no single component accounting for more than 10% of the applied ^{14}C.

Proposed degradation pathways are shown in Figure 2.

Figure 2. Proposed degradation pathways of phosalone in soil under aerobic conditions.



Anaerobic degradation in soil

Clarke (1999) reported the anaerobic degradation of [1-*phenyl*-^{14}C]phosalone in sandy loam soil (ADAS and USDA classifications) incubated at 20°C in the dark.

Soil was flooded and purged with nitrogen for 39 days before treatment to establish anaerobic conditions. Phosalone was applied to the water surface at a rate equivalent to 1.04 kg ai/ha and samples were incubated for up to 77 days. Additional flasks were treated with [^{14}C]phosalone at a rate equivalent to 1.0 kg ai/ha after 97 days incubation under anaerobic conditions to confirm the rates of degradation in the water phase and transfer to the soil phase, which were found to be extremely rapid in the early samples. Radioactivity was rapidly transferred to the soil, with only 14% of the applied ^{14}C remaining in the water after 7 days, and 2% after 77 days. Degradation products were identified by HPLC co-chromatography with certified reference standards and identities were confirmed, where possible, by mass spectral analysis. Overall recoveries ranged from 90 to 100%. In the water phase, phosalone was rapidly degraded to 2-amino-5-chlorophenol (chloroaminophenol), which reached a maximum of 20% of the applied radioactivity after 3 days and then decreased. Low levels of 6-chloro-3*H*-benzoxazol-2-one, the precursor of chloroaminophenol, were detected up to 3 days at 0.5-3.5% of the applied radioactivity. In the soil phase, phosalone was rapidly degraded to chloroaminophenol, which reached a maximum of 8% of the applied radioactivity at day 14, and 6-chlorobenzoxazolone

which reached a maximum of 2% at day 56. No significant levels of volatile compounds were found (<0.5% of the applied ^{14}C after 77 days). Seven-day samples containing residues which were not extracted by solvents were subjected to further treatment to determine whether any additional radioactivity could be released. Ammonia reflux released an additional 14% of the radioactivity. No phosalone was present but there were 4 components, two of which were identified as chloroaminophenol and 6-chlorobenzoxazolone accounting for 2% of the applied dose, and 2 unknowns at 4 and 6%. Additional analysis of the remaining soil to determine the distribution of the unextracted radioactivity showed that 13% of the applied radioactivity was associated with humin, 21% with humic acid and 9% with fulvic acid. The KIM modelling programme (Schering AG) was used to calculate degradation values for phosalone and chloroaminophenol and showed a fit criterion of 0.996.

Table 8. Degradation rates of phosalone under anaerobic conditions.

	phosalone		2-amino-5-chlorophenol	
	DT-50, days	DT-90, days	DT-50, days	DT-90, days
Water phase	0.10	1.65	10.15	38.42
Soil phase	4.33	37.93	nc	nc
Entire system	1.82	25.47	29.10	nc

nc: could not be calculated

Phosalone was rapidly taken up by the soil phase and readily degraded in both soil and water. Chloroaminophenol was also readily degraded in anaerobic water and in the system as a whole. Its rate of degradation in soil could not be calculated. The routes of degradation in anaerobic and aerobic soil were similar.

Soil adsorption/desorption

Skinner (1995) reported an adsorption/desorption study of [^{14}C]phosalone in four soils (sandy loam, silty clay loam, loam and clay) by the batch equilibrium method (OECD-TG 106 1981).

[^{14}C]phosalone was applied at concentrations of 0.082, 0.149, 0.424 and 1.0 mg/kg in the silty clay, loam, and clay soils and at 0.076, 0.152, 0.445, and 1.02 mg/kg in the sandy loam. The overall ^{14}C balance for all soils and application rates was 99.5 ± 3.9%. Samples were analysed by TLC and HPLC. Phosalone accounted for >95% of the total radiocarbon in adsorption and desorption solutions of sandy loam and loam soils and >94% in soil pellet extracts of all soils. Phosalone was stable during both adsorption and desorption in sandy loam and loam soils, but was degraded in silty clay loam during adsorption with the formulation of degradation products in solution, and degraded in clay too rapidly to allow quantitative adsorption/desorption measurements. No individual product in any solution or soil extract exceeded 8.5% of the applied radiocarbon. The formation of bound residues was significant in silty clay loam and clay.

The adsorption and desorption coefficients (K_d and K_{oc}) were determined (Table 9). An adsorption isotherm could not be obtained for the clay soil owing to the degradation of phosalone during the adsorptive phase. The adsorption K_d values were found to correlate roughly with the organic carbon content of the soil. The overall average K_{oc} value for adsorption to the three soils for which it could be calculated was 2060. On the basis of the K_{oc} values phosalone is predicted to have only slight to low mobility in soils.

Table 9. Soil characteristics and Freundlich adsorption/desorption constants.

Soil	Organic carbon	Adsorption		First Desorption		Second Desorption	
	%	K_d	K_{oc}	K_d	K_{oc}	K_d	K_{oc}
sandy loam	0.84	22.5	2680	40.0	4760	38.4	4570
silty clay loam	0.71	6.2	870	NA		NA	
loam	1.33	35.1	2640	53.8	4050	51.8	3890
clay	2.95	NA		NA		NA	

NA = not available owing to degradation

Environmental fate in water/sediment systems

Muttzall and de Kreuk (1987) and Muttzall (1995) reported the degradation of phosalone in two water/sediment systems, one from a river and one from a ditch, in biometer flasks over a 12-week period. [^{14}C]phosalone was added to the system at 1.0 mg/l. The level of radioactivity in the water phase decreased from 42% to 10% between week 0 and week 12 in the river system and from 13 to 2% in the ditch system. Radioactivity was mainly associated with the sediment fraction and was rapidly bound. After 1 week in the river system, 22% of the applied ^{14}C was extractable from solids and 59% was bound, 44% and 47% in the ditch system. By the end of the study, 68% and 65% of the applied radioactivity was in bound residues in the river and ditch systems respectively, and $^{14}CO_2$ accounted for 7.9% and 6.9%. Characterization of the products in the aqueous phase showed that phosalone was rapidly degraded, and four unidentified compounds were found. These did not account for more than 10% of the applied radioactivity in either system at the end of the study. The main component in the river system was a polar compound, accounting for 7% of the applied radioactivity, which was not detected in the ditch system. In the sediment phase the main compound found was phosalone, but it did not account for >6% of the applied ^{14}C by the end of the study The 4 compounds found in the water were not present or were at levels ≤1%. Phosalone was rapidly degraded in both the aqueous and sediment phases of both systems.

Bioaccumulation in aquatic organisms

Forbis *et al*. (1986) reported a dynamic 42-day study to evaluate the bioconcentration of [^{14}C]phosalone by bluegill sunfish in a flow-through proportional dilution system which maintained an average concentration of phosalone of 0.93 ± 0.24 µg/l, close to the nominal 1.0 µg/l, throughout the 28-day uptake period. There was a 14-day depuration period. [^{14}C]phosalone was found to be stable in a preliminary equilibration study where 86-109% of the radioactivity was extracted from the water, of which 96% was phosalone and the remainder phosalone oxon.

The minimum quantifiable levels were about 1.5 µg/kg in edible and inedible tissues and whole fish, and 0.34 µg/l in the water. Recoveries from tissues averaged 97-99%.

At the end of the 28-day uptake phase the residues were 0.073, 0.26 and 0.18 µg/g phosalone equivalents in edible tissue, inedible tissue, and whole fish respectively. Residues decreased very rapidly during the depuration phase.

The uptake rate constant (K_1) for whole fish was 0.18 ± 0.02 mg/kg fish/mg/l water/day, and the time to reach 90% of a steady state was 2.4 ± 0.3 days. The depuration rate constant (K_2) was 0.98 ± 0.1, with a half-life for depuration of 0.71 ± 0.08 days. The steady-state bioconcentration factor (BCF) for whole fish was 180 ± 30 according to the non-linear two-compartment kinetic modelling computer programme BIOFAC. During days 14-28 of the uptake phase, the BCF values ranged from 280 to 300 for viscera, 78 to 85 for edible tissue, and 190 to 200 for whole fish.

METHODS OF RESIDUE ANALYSIS

The Meeting received information on methods for the determination of phosalone in crops and soil.

Crops

Residues are generally extracted by macerating with acetone/water 80/20 and cleaned up by partition with dichloromethane. The final extract is concentrated and analysed by gas chromatography.

CNG An No. 4174. This method was used in most supervised trials on crops. Fifty g samples are steeped in water and extracted with acetone. The extract is partitioned with dichloromethane and the final extract is concentrated and analysed by gas chromatography with an NP detector.

In some field trials the procedure was modified by extracting with acetone/water (80/20) instead of steeping in water. The final residue was dissolved in toluene. The LOD is 0.05 mg/kg.

CNG An No. 4765E. This method is used for the determination of phosalone and phosalone oxon. Extraction with acetone is followed by partitioning between water and dichloromethane. Residues are determined by gas-liquid chromatography with an FPD. The LOD is 0.02 mg/kg for both phosalone and phosalone oxon.

CNG An Nos. 4432E and 4698E. Samples are ground with acetone/water (80/20) and partitioned with dichloromethane. The final extract is analysed by gas chromatography with thermionic detection (NPD). The limit of determination is 0.05 mg/kg. The method was validated by analysing untreated control samples and samples spiked at 0.05, 0.25, 0.5, 1 and 2 mg/kg. The mean recoveries from each sample at each level were between 75 and 109% with relative standard deviations less than 20% (Table 10), confirming the repeatability of the method (Gabereau, 1997).

Table 10. Recoveries of phosalone from rape seed, barley and apples.

Commodity	Fortification, mg/kg	No. of analyses	Recovery, %	Mean recovery, %	SD %
Rape seed	0.05	3	82 ,84, 86		
	0.25	3	80, 89, 104	83	9
	1	3	73, 75, 76		
Barley	0.05	3	102, 109, 117		
	0.25	3	99, 100, 100	99	9
	1	3	88, 89, 91		
Apple	0.05	3	86, 88, 94		
	0.5	3	102, 103, 108	98	9
	2	3	99, 101, 101		

Ref. 97-95 (AR 148-97). This is the same as the previous method except that an FPD is used instead of an NPD with confirmation by selective ion-monitoring GC-MS (Bourgade and Yslan, 1988). The limit of determination is 0.05 mg/kg (Table 11).

Table 11. Recoveries of phosalone from apples and rape seed.

Commodity	Fortification, mg/kg	No. of analyses	Recovery, %	Mean recovery, %	SD %
Apple	0.05	3	73, 74, 81	82	6
	0.5	3	86, 87, 89		2
Rape seed	0.5	3	72, 87, 88	82	10

The official method of the Japan Environmental Agency consists in extraction with acetone, partitioning with dichloromethane, and clean-up by Florisil column chromatography with benzene as the eluting solvent. The final extract is analysed by gas chromatography with an NP or FP detector. The limit of detection is 0.01 mg/kg.

The Meeting received information on the multi-residue methods developed by the USFDA (Anon., 1997).

Table 12. Analytical methods used for analysing supervised field trial samples.

Crop	Analytical methods	Reference
Apple	Ref. 4698 (3 Feb 1983)	REF. 25
Apple	see study no. 89-10	REF. 26
Apple	CNG An No 4432E	REF. 27
Apple	CNG An No 4174	REF. 28
Apple	CNG An No 4174	REF. 29
Apple	CNG An No 4174	REF. 30
Apple	CNG An No 4174	REF. 31
Apple	CNG An No 4174	REF. 32
Apple	Japan EA official	REF. 33
Apple	CNG An No 4174	REF. 34
Apple	CNG An No 4432E	REF. 62
Pear	CNG An No 4174	REF. 34
Pear	CNG An No 4174	REF. 35
Pear	modified Japan EA official	REF. 36
Cherry	CNG An No 4698E	REF. 37
Cherry	CNG An No 4174	REF. 38
Cherry	CNG An No 4174	REF. 39
Peach	Ref. 4698 3.Feb. ‘83	REF. 40
Peach	CNG An No 4174	REF. 41
Peach	CNG An No 4174	REF. 42
Peach	CNG An No 4174	REF. 43
Peach	CNG An No 4174	REF. 44
Peach	modified Japan EA official	REF. 45
Peach	Ref. 97-95 (AR 148-97)	REF. 63
Peach	Ref. 97-95 (AR 148-97)	REF. 64
Peach	Ref. 97-95 (AR 148-97)	REF. 65
J-Apricot	modified Japan EA official	REF. 46
Almond	CNG An No 4765E	REF. 47
Almond	CNG An No 4174	REF. 48
Almond	CNG An No 4174	REF. 49
Almond	CNG An No 4174	REF. 50
Almond	CNG An No 4174	REF. 51
Hazelnut	CNG An No 4765E	REF. 52
Hazelnut	CNG An No 4174	REF. 53
Hazelnut	CNG An No 4174	REF. 54
Hazelnut	CNG An No 4174	REF. 55
Hazelnut	CNG An No 4174	REF. 56
Walnut	CNG An No 4765E	REF. 57
Walnut	CNG An No 4174	REF. 58
Walnut	CNG An No 4174	REF. 59
Walnut	CNG An No 4174	REF. 60
Walnut	CNG An No 4174	REF. 61

Soil

Le Gren (1997) described a method for the determination of phosalone and its potential phenoxazone degradation product (2-amino-7-chlorophenoxazin-3-one) in soil. Samples are extracted by distilling with a mixture of water and acetone, and cleaned up on a diol mini-cartridge. Quantification is by GLC with electron capture detection. The limit of determination is 0.02 mg/kg for each compound. The method was validated on four European soils (Chazay, Manningtree, Sevilla, and Bologna) by spiking control samples at 0.02, 0.2, and 2 mg/kg. Individual recoveries ranged from 96 to 115% for phosalone and 68 to 113% for the phenoxazone. The mean recoveries of phosalone and the phenoxazone at each level ranged from 80 to 110% (Table 13).

Table 13. Recoveries of phosalone and aminochlorophenoxazone from soil.

Fortification, mg/kg	Soil	Recovery, %					
		Phosalone			Phenoxazone		
		Individual	mean	SD, %	Individual	mean	SD, %
0.02	Manningtree	107,96,110			102,107,111		
	Sevilla	105,103,101	**102**	**4**	85,110,92	**98**	**15**
	Bologna	101,100,101			110,113,111		
	Chazay	98,103,100			70,83,84		
0.2	Manningtree	110,115,111			70,83,100		
	Sevilla	111,111,111	**110**	**2**	89,81,89	**85**	**15**
	Bologna	108,113,111			94,104,103		
	Chazay	111,104,110			71,70,70		
2	Manningtree	107,106,108			93,77,71		
	Sevilla	102,102,102	**105**	**3**	79,79,84	**80**	**9**
	Bologna	103,101,104			73,68,81		
	Chazay	100,109,111			83,87,87		

Stability of residues in stored analytical samples

Quintelas (1998) reported stability studies on selected crops which showed phosalone to be stable for up to 24 months in macerated samples at -18°C.

Peaches and almonds were spiked with phosalone at 0.5 mg/kg and stored at -18°C for 1 and 3 months. The analytical method was AR 148-97. The mean recoveries after 1 and 3 months respectively were 102 and 94% from peaches and 81 and 79% from almonds. These values were all higher than the initial recoveries at 0.5 mg/kg.

Table 14. Stability of phosalone on peaches and almonds stored at -18°C.

Sample	Storage, months	Fortification, mg/kg	Mean recovery, %	Range, %
Peach	0	0.05	81.5	80-83
	0	0.5	74.5	71-78
	1	0.5	102	93-109
	3	0.5	94	91-96
Almond	0	0.05	89.5	83-96
	0	0.5	77	75-79
	1	0.5	80.7	73-92
	3	0.5	79	72-85

Almonds, apples and cherries containing incurred residues from field studies were analysed after storage at -18°C for 19 to 24 months by method AR 148-97. Recoveries of phosalone were 66 and 77% from almonds, 67 and 70% from apples and 113 and 133% from cherries (Table 15).

Table 15. Stabilities of incurred residues of phosalone in almonds, apples and cherries stored at -18°C.

Sample	Storage, months	Initial residue, mg/kg	Recovery, %	Mean, %
Almond	23	3.39	66	71.5
		4.36	77	
Apple	19	0.75	67	68.5
		0.66	70	
Cherry	24	0.46	133	123
		0.72	113	

Definition of the residue

A metabolism study on apples showed that phosalone was the predominant residue (75-92%) with phosalone oxon at low levels (2-7%) in extracts and rinses of fruit and leaves. In grapes phosalone was also the major residue in the fruit (≥88%) and the oxon was at a very low level (1-2%).

The Meeting concluded that the current definition of the residue as phosalone is suitable both for compliance with MRLs and the estimation of dietary intake.

USE PATTERN

The Meeting received updated information on the registered uses of phosalone on selected crops against leaf eater, leaf roller, tree fly, etc. The information is summarized in Table 16.

Table 16 Registered uses of phosalone in pome fruit, stone fruit and tree nuts. All foliar applications.

Crop	Country	Form	Application			PHI, days
			Rate, kg ai/ha	Spray conc., kg ai/hl	No.	
Almond	EU [3]	EC350 g/l	0.75		3	70
Almond	EU [3]	SC500g/l	0.75		3	70
Almond	France [2]	EC350 g/l		0.06-0.07	N	70
Almond	France [2]	SC500 g/l		0.06-0.075	N	70
Apple	Algeria	EC350 g/l	0.6		N	N
Apple	Belgium [2]	SC500 g/l	0.5-0.75	0.05-0.075	N[1]	28
Apple	Canada	SC500 g/l	1.0-1.5	0.03-0.045	3	30
Apple	EU [3]	EC350 g/l	0.6-0.9	0.06	3	28
Apple	EU [3]	WP300 g/l	0.6-0.9	0.06	3	28
Apple	France [2]	EC350 g/l		0.06	N	14
Apple	France [2]	SC500 g/l		0.06	N	15
Apple	Italy [2]	EC350 g/l		0.05-0.07	N	21
Apple	Japan	EC350 g/l		0.023-0.035	2	45
Apple	Japan	EC200 g/l + DDVP		0.02-0.025	2	45
Apple	Netherlands	SC500 g/l	0.6-0.9	0.06	2	28
Apple	Russia	EC350 g/l	0.7-1.4		2	30 (south)
Apple	Russia	EC350 g/l	0.7-1.4		2	40 (north)
Apple	Turkey	EC350 g/l		0.07	N	15
Apple	Ukraine	EC350 g/l	0.7-1.4		2	40
Apricot	EU [3]	EC350 g/l	0.6-0.9	0.06	3	28
Apricot	EU [3]	SC500g/l	0.6-0.9	0.06	3	28
Apricot	France [2]	EC350 g/l		0.06	N	14
Apricot	France [2]	SC500 g/l		0.06	N	15
Apricot	Italy [2]	EC350 g/l		0.05-0.07	N	21
Apricot	Japan	EC200 g/l + DDVP		0.02	2	45
Apricot	Russia	EC350 g/l	0.7-0.84		1	45
Apricot	Ukraine	EC350 g/l	0.7-0.84		1	45
Cherry	Canada	SC500 g/l	1.0-1.5	0.03-0.045	3	14
Cherry	EU [3]	EC350 g/l	0.9	0.06	2	15
Cherry	EU [3]	WP300 g/l	0.9	0.06	2	15
Cherry	France [2]	EC350 g/l		0.06	N	14
Cherry	France [2]	SC500 g/l		0.06	N	15
Cherry	Italy [2]	EC350 g/l		0.05-0.07	N	21
Cherry	Russia	EC350 g/l	0.28-0.98		2	40
Cherry	Turkey	EC350 g/l		0.06	N	15
Cherry	Ukraine	EC350 g/l	0.28-0.98		2	40
Fruit trees	Austria	EC350 g/l		0.05-0.07	N	21
Fruit trees	Croatia	EC350 g/l	7-8.75	0.07-0.875	3	35
Fruit trees	Greece	EC350 g/l		0.05-0.07	N	21
Fruit trees	Hungary	EC350 g/l	0.61		N	21
Fruit trees	Iran	EC350 g/l	N	0.05	N	15
Fruit trees	Iraq	EC350 g/l		0.04-0.06	N	15

Crop	Country	Form	Application			PHI, days
			Rate, kg ai/ha	Spray conc., kg ai/hl	No.	
Fruit trees	Kuwait	SC500 g/l		0.06	N	14
Fruit trees	Morocco	EC350 g/l		0.04-0.05	N	15
Fruit trees	Morocco	WP300 g/l		0.045-0.06	N	15
Fruit trees	Poland	EC350 g/l	0.63-0.91	0.06-0.19	2	15
Fruit trees	Spain	WP300 g/l		0.06	N	15
Fruit trees	Tunisia	WP300 g/l		0.04-0.06	N	15
Hazelnut	EU [3]	EC350 g/l	0.6		3	28
Hazelnut	EU [3]	SC500g/l	0.6		3	28
Hazelnut	France [2]	EC350 g/l		0.06	N	21
Hazelnut	France [2]	SC500 g/l		0.06	N	21
Hazelnut	Turkey	EC350 g/l		0.07	N	15
Peach	Canada	SC500 g/l	1.0-1.5	0.03-0.045	3	30
Peach	EU [3]	EC350 g/l	0.6-0.9	0.06	3	28
Peach	EU [3]	SC500g/l	0.6-0.9	0.06	3	28
Peach	France [2]	EC350 g/l		0.06	N	14
Peach	France [2]	SC500 g/l		0.06	N	15
Peach	Italy [2]	EC350 g/l		0.05-0.07	N	21
Peach	Japan	EC200 g/l + DDVP		0.02	2	14
Peach	Russia	EC350 g/l	0.56-0.84		1-2	30-40
Peach	Ukraine	EC350 g/l	0.56-0.84		1-2	30-40
Pear	Algeria	EC350 g/l	0.6		N	N
Pear	Belgium [2]	SC500 g/l	0.5-0.75	0.05-0.075	N[1]	28
Pear	Canada	SC500 g/l	1.0-1.5	0.03-0.045	3	30
Pear	EU [3]	EC350 g/l	0.6-0.9	0.06	3	28
Pear	EU [3]	WP300 g/l	0.6-0.9	0.06	3	28
Pear	France [2]	EC350 g/l		0.06	N	14
Pear	France [2]	SC500 g/l		0.06	N	15
Pear	Italy	EC350 g/l		0.05-0.07	N	21
Pear	Japan	EC350 g/l		0.023-0.035	2	45
Pear	Japan	EC200 g/l + DDVP		0.02	2	45
Pear	Netherlands	SC500 g/l	0.6-0.72	0.06	2	28
Pear	Russia	EC350 g/l	0.7-1.4		2	30 (south)
Pear	Russia	EC350 g/l	0.7-1.4		2	40 (north)
Pear	Taiwan	WP300 g/l	0.3-0.6		1	15
Pear	Ukraine	EC350 g/l	0.7-1.4		2	40
Plum	Canada	SC500 g/l	1.0-1.5	0.03-0.0625	3	30
Plum	EU [3]	EC350 g/l	0.6-0.9	0.06	3	28
Plum	EU [3]	SC500g/l	0.6-0.9	0.06	3	28
Plum	France [2]	EC350 g/l		0.06-0.07	N	14
Plum	France [2]	SC500 g/l		0.06-0.07	N	15
Plum	Italy [2]	EC350 g/l		0.05-0.07	N	21
Plum	Russia	EC350 g/l	0.28-0.98		2	40
Plum	Turkey	EC350 g/l		0.07	N	15
Plum	Ukraine	EC350 g/l	0.28-0.98		2	40
Pome fruit	Slovak Republic	EC350 g/l		0.07	N	21
Pome fruit	Switzerland	EC350 g/l	0.79-1.05	0.05	N	30
Prune	Canada	SC500 g/l	1.0-1.5	0.03-0.0625	3	30
Stone fruit	Algeria	EC350 g/l	0.6		N	N
Stone fruit	Czech Republic	EC350 g/l		0.07	N	21
Stone fruit	EU	EC350 g/l	0.9	0.06	3	28
Stone fruit	EU	WP300 g/l	0.9	0.06	3	28
Stone fruit	Slovak Republic	EC350 g/l		0.07	N	21
Stone fruit	Switzerland	EC350 g/l	0.79-1.05		N	N
Walnut	EU [3]	EC350 g/l	0.6			
Walnut	EU [3]	SC500g/l	0.6			
Walnut	France	EC350 g/l		0.06	N	21
Walnut	France	SC500 g/l		0.06	N	21

N: not specified
In France, standard orchard spray volume is 10 hl/ha.
[1] Used in integrated pest management (IPM) programmes, rarely exceeding 2 applications
[2] Currently registered national labels for countries within the EU Member States will be revised in the future to reflect GAP to be supported at the EU level
[3] Use pattern envisaged at EU level in anticipation of the future registration requirements.

RESIDUES RESULTING FROM SUPERVISED TRIALS

Information received on supervised field trials on apples, pears, peaches, Japanese apricots, cherries, almonds, hazelnuts and walnuts is summarized in Tables 17-24.

Table 17	*Apples.* Italy, France, Japan, Germany
Table 18	*Pears.* Spain, Japan
Table 19	*Cherries*. France
Table 20	*Peaches*. Italy, France, Spain, Japan
Table 21	*Japanese Apricots*. Japan
Table 22	*Almonds*. France
Table 23	*Hazelnut.* France
Table 24	*Walnuts*. France

Where residues were not detected they are shown as below the limit of determination (LOD), e.g. <0.01 mg/kg.

References to supervised trials will be found in the second (numerical) reference list.

Apples. Phosalone was applied by experimental backpack sprayers in France and Germany and by pressure sprayers in Italy and Japan. Plots in the French trials were 3 to 6 trees and field samples of twelve fruit or >2 kg were stored frozen for 2 to 10 months before analysis. In Italy plots were 4 to 10 trees, field samples were twelve fruit, and storage was 1 to 4 months before analysis. In Germany 4 trees per plot and samples of 12 apples with storage for 5 to 7 months before analysis. In Japan only one tree was treated and 4 to 6 kg samples were stored for 3 months before analysis.

Table 17. Phosalone residues in apples resulting from supervised trials in Italy, France, Japan and Germany. Analyses of replicate field samples are shown separately. Double underlined residues are from treatments according to GAP.

Country, Year, Location	Form.	Application				Variety	PHI days	Residues mg/kg		Ref./Report no.
		No.	kg ai/ha	water l/ha	kg ai/hl			Mean	Individual	
Italy, 1989	EC 232 g/l	4	0.6	1000	0.06	Rome Beauty	0 7 14 21 28 35	1.6 1.0 1.1[1] 0.5 0.4 0.6[2]		Ref. 25 AG/CRLD/AN/8916827
Italy, 1989	EC 24%	5	0.6	1000	0.06	Rome Beauty	21	0.65[2]		Ref. 26 AG/CRLD/AN/9015907
		5	0.6	1000	0.06	Red Chief	21	0.85[2]		
		9	1.2	2000	0.06	Red Chief	21	0.96[2]		
France, 1995 Saran	SC 500 g/l	1	0.88	563	0.16	Golden	2 h 3 7 14		0.69,0.72 0.67,1.3 0.54,1.4 0.78,1.1	Ref. 27 R&D/CRLD/AN/kd/9616621
Souastre		1	0.88	560	0.16	Idared	2 h 3 7		1.1,1.4 1.2,2.6 1.3,1.9	

Country, Year, Location	Form.	Application				Variety	PHI days	Residues mg/kg		Ref./Report no.
		No.	kg ai/ha	water l/ha	kg ai/hl			Mean	Individual	
							14		0.49,1.6	
St. Laurent d`Agng		1	0.87	583	0.15	Elstar	2 h 3 7 14		1.0,1.4 1.2,1.8 1.7,1.6 2.5,1.5	
Lavaur		1	0.88	1143	0.08	Grany	2 h 3 8 14		0.71,0.45 0.54,0.54 0.49,0.58 0.38,0.17	
France, (South) 1996 Beaucaire	EC 350 g/l	3	0.61	983	0.06	Golden	16 22 31 45	0.70 0.68 0.74[1,2] 0.58		Ref. 28 R&D/CRLD/AN/dbe/9 716155
France (North), 1996 Tigy	EC 350 g/l	3	0.6	810	0.07	Idared	14 19 29 44	0.66[1] 0.52 0.45 0.37		Ref. 29 R&D/CRLD/AN/vt/97 16613
France (South), 1996 Seyssuel		3	0.61	833	0.07	Golden	15 21 30 45	0.99 1.1 1.5[1,2] 1.1		
France (North), 1997 Crops Nuds	EC 350 g/l	3	0.74	545	0.14	Indaine	27		1.0,1.2	Ref. 30 R&D/CRLD/AN/vt/98 15382
France (South), 1997 Eyragues		3	0.74	872	0.08	Golden	27		0.82,0.87	
France (North), 1997 Crops Nuds	EC 350 g/l	3	0.73	538	0.14	Indaine	2 h 11 18 24 31 38	1.4 1.0 1.0 0.7 0.6 0.43		Ref. 31 R&D/CRLD/AN/dbe/9 815324
France (South), 1997 Seyssuel		3	0.61	740	0.08	Golden	2 h 14 21 28 35 42	0.93 0.88 0.77 0.63 0.65 0.64		
Italy, 1997 Bologna	WP 291 g/kg	3	0.90	1500	0.06	Golden delicious	21 28	0.88 0.91[2]		Ref. 32 R&D/CRLD/AN/vt/98 15916
Japan, 1979 Fuku-shima	EC 35%	2(3)[3]	1.8	5000	0.035	Starking Delicious	45	0.42		Ref. 33
							60	0.20		
		3(4)[3]					45 60	0.52 0.59		
Nagano		2	1.8	5000	0.035	Fuji	43 57	0.58 0.52		
		3					43 57	0.82 0.78		
Germany (North), 1997 Berlin[4]	WP 304 g/kg	3			0.06	Jonathan	0 7 14 21 28	3.0 2.7 2.5 0.97 0.84		Ref. 34 R&D/CRLD/AN/msa/9 816476

Country, Year, Location	Form.	Application				Variety	PHI days	Residues mg/kg		Ref./Report no.
		No.	kg ai/ha	water l/ha	kg ai/hl			Mean	Individual	
							35	0.063		
Germany (North), 1997 Sollingen		3	0.92	1515	0.06	Jonagold	0 15 21 28 35 42	0.73 0.43 0.45 <u>0.46</u>[1] 0.35 0.29		

[1] According to French GAP
[2] According to Italian GAP
[3] 2 or 3 treatments were intended but one support treatment was also applied, owing to rainfall
[4] This trial was reported to have been at the GAP spray concentration of 0.06 kg ai/hl but the measurement of the spray volume, which was close to 4 l/tree, was suspect

<u>Pears</u>. Phosalone was applied by experimental backpack sprayers in Spain and Germany and by pressure sprayers in Japan. The plot sizes were 16 m^2 (4 trees) in Japan, 25.6 m^2 (4 trees) in Spain and 70 m^2 (4 trees) in Germany. Field samples were twelve pears in Germany and Spain and 4 kg in Japan, and were stored in the freezer for 1 to 7 months before analysis.

Table 18. Phosalone residues in pears resulting from supervised trials in Germany, Spain and Japan. Underlined and double underlined residues are from treatments according to GAP. Double underlined residues were used to estimate maximum residue and STMR levels.

Country, Year	Form.	Application				Variety	PHI, days	Mean residues, mg/kg	Ref./Report no.
		No.	kg ai/ha	water l/ha	kg ai/h				
Germany, 1997	WP	3	0.89	1485	0.061	Conference	29	0.49	Ref. 34 R&D/CRLD/AN/msa/9816476
							35	0.63	
Spain, 1997	WP 300g/kg	3	0.90	1250	0.072	Conference	0 15 21 30 45	0.93 <u>0.38</u>[1] <u>0.36</u>[2] 0.23 0.13	Ref. 35 R&D/CRLD/AN/vt/9815818
Japan, 1989	EC 35%	2	1.75	5000	0.035	Kousui	45 60	<u>0.11</u>[3] 0.033	Ref. 36
		2	2.19	6250	0.035	Cyojuro	45 60	<u>0.29</u>[3] 0.099	

[1] According to French GAP
[2] According to Italian GAP
[3] According to Japanese GAP

<u>Cherries</u>. Trees were sprayed with an experimental atomizer with spray boom, backpack and tree sprayers in supervised trials in France. Plots sizes ranged from 12.75 m^2 to 81 m^2 and 500 to 1000g of field samples were collected and stored frozen for 3 to 9 months before analysis.

Table 19. Phosalone residues in cherries resulting from supervised trials in France. Analyses on replicate field samples in each trial are shown separately. Double underlined residues are from treatments according to GAP and were used to estimate maximum residue and STMR levels.

Year, Location	Form.	Application				Sample (Variety)	PHI, days	Residues, mg/kg	Ref./Report no.
		No.	kg ai/ha	Water, l/ha	kg ai/hl				
1994 St.Didier sous	SC 500g/l	3	0.75	769	0.098	fruit with stone (Starking)	14	<u>1.2, 1.4</u>	Ref. 37 R&D/CRLD/An/fb/9515875

Year, Location	Form.	Application				Sample (Variety)	PHI, days	Residues, mg/kg	Ref./Report no.
		No.	kg ai/ha	Water, l/ha	kg ai/hl				
		3	1.1	769	0.15	fruit with stone (Starking)	14	1.6, 3.4	
Blauvac		3	0.74	497	0.15	fruit with stone (Van)	14	1.6, 1.3	
		3	1.1	505	0.22	fruit with stone (Van)	14	1.8, 2.5	
St. Simplice		3	0.87	993	0.088	fruit with stone (Sumbeurs)	11	1.5, 1.8	
		3	1.4	1345	0.1	fruit with stone (Sumbeurs)	11	1.5, 3.1	
(South),1996	SC 500g/l	1	0.6	300	0.2	fruit with stone (Sunburst)	11	0.34, 0.37	Ref. 38 R&D/CRLD/AN /dbe/9716189
Meilhan							16 21	0.59, 0.23 0.23, 0.17	
		2	0.6	300	0.2	fruit with stone (Sunburst)	11 16 21	0.61, 0.78 0.46, 0.72 0.43, 0.41	
(North), 1996 Mezieres		1	0.59	803	0.073	fruit with stone (Burlat)	8 14 18	0.51, 0.5 0.35, 0.21 0.47, 0.32	
		2	0.59	801	0.073	fruit with stone (Burlat)	8 14 18	0.53, 0.6 0.45, 0.31 0.13, 0.33	
(North), 1996 Olivet		1	0.58	653	0.089	fruit with stone (Duroni 3)	9 15 23	0.58, 0.69 0.26, 0.26 0.08, 0.15	
		2	0.58	650	0.089	fruit with stone (Duroni 3)	9 15 23	0.60, 0.63 0.23, 0.22 0.14, 0.08	
(South),1996 St. Didier		1	0.60	500	0.12	fruit with stone (Burlat)	10 17 21	0.33, 0.33 0.18, 0.16 0.15, 0.08	
		2	0.60	416	0.14	fruit with stone (Burlat)	10 17 21	0.45, 0.6 0.15, 0.26 0.12, 0.2	
(South),1996		1	0.60	667	0.09	fruit with stone (Duroni 2)	15	0.22, 0.28	
Blcuvac							20 26	0.14, 0.3 0.10, 0.14	
		2	0.60	667	0.09	fruit with stone (Duroni 2)	15 20 26	0.27, 0.42 0.17, 0.22 0.14, 0.17	
(South),1996 Belcaste		1	0.60	400	0.15	fruit with stone (Stark)	11 14 20	0.17, 0.16 0.15, 0.6 0.53, 0.22	
		2	0.60	400	0.15	fruit with stone (Stark)	11 14 20	0.62, 0.56 0.37, 0.46 0.15, 0.36	
(North),1997 Villeveque	EC 353g/l	1	0.56	935	0.06	fruit (Lapins)	10 15 21	0.36, 0.24 0.36, 0.18 0.11, 0.12	Ref. 39 R&D/CRLD/AN /vt/9815381
		2	0.58	965	0.06	fruit (Lapins)	10 15	0.36, 0.32 0.29 ,0.2	
							21	0.13, 0.13	

Peaches. Trees were sprayed with a hand-gun motor pump and pressure spray in Italian and Japanese trials, and with backpack sprayers and air blast sprayers in France. Plots were 4 to 10 trees in Italy, 45 to 90 m^2 with 2 to 6 trees in France, 100 m^2 in Spain and 1 or 2 trees in Japan. Field samples of 500 g or 12 peaches were stored frozen for 2 to 6 months before analysis.

Table 20. Phosalone residues in peaches resulting from supervised trials in Italy, France, Spain and Japan. Replicate individual residues are from replicate field samples. Double underlined residues are from treatments according to GAP and were used to estimate maximum residue and STMR levels.

Country, Year	Form.	Application				Sample (Variety)	PHI days	Residues, mg/kg		Ref.
		No.	Kg ai/ha	waterl /ha	kg ai/hl			Mean	Individual	
Italy, 1989	EC 232 g/l	1	1.2	2000	0.06	Fruit (Starkred Gold)	0 7 14 21 28	0.4 0.3 <u>0.16</u>[1] <u>0.13</u>[2] 0.13		Ref. 40 AG/CRLD/AN/ 8916828
France, 1997 (South)	EC 350 g/l	3	0.74	563	0.13	Fruit without stone (Alexandra)	2 h 14 21 28 35		1.5,1.6 0.80,0.52 0.57,0.61 0.53,0.76 0.54,0.49	Ref. 41 R&D/CRLD/AN /dbe/9815342
						Fruit (whole)	2h 14 21 28 35		1.4, 1.5 <u>0.73</u>[1], 0.48 0.53, 0.57 0.48, <u>0.68</u>[2] 0.5, 0.45	calculation
France, 1997 (South) Ste Bazeille	EC 350 g/l	3	0.61	288	0.21	Fruit without stone (Royal glory) Fruit (whole)	26 26		0.44,0.29 0.37,0.28 0.41, 0.27 0.35, 0.26	Ref. 42 R&D/CRLD/AN /vt/9815817 calculation
Italy, 1997	WP 291 g/kg	3	0.88	1500	0.06	Fruit (whole) (Duchessa d'Este) Pulp	0 14 21 28 35 35	3.7 <u>1.5</u>[1] <u>1.4</u>[2] 0.71 0.45 0.49	calculation	Ref. 43 R&D/CRLD/AN /dbe/9815848
Spain, 1997	WP 300 g/kg	3	0.9	800	0.11	Fruit without stone (Red Top) Fruit (whole)	28 28	0.54 0.51	calculation	Ref. 44 R&D/CRLD/AN /dbe/9815325
Japan, 1982 Fuku-shima	EC 20%	2	0.8	4000	0.02	Fruit without stone & peeled (Ohkubo)	15 30 45	0.09 0.02 <0.01		Ref. 45
						Peel	15 30 45	8.92 3.93 1.20		
						Fruit without stone	15	2.3	calculation	
							30 45	0.88 0.25		
Ishikawa						Fruit without stone & peeled (Ohkubo)	15 30 45	0.04 0.02 <0.01		
						Peel	15 30 45	5.43 3.62 1.02		
						Fruit without stone	15 30 45	1.33 0.63 0.17	calculation	

Country, Year	Form.	Application				Sample (Variety)	PHI days	Residues, mg/kg		Ref.
		No.	Kg ai/ha	waterl /ha	kg ai/hl			Mean	Individual	
France, 1998 (South) Subirats	WP 298 g/kg	3	0.89	800	0.11	Fruit without stone (Baby Gold 6)	0 15 21 30 45	0.72 0.50 0.27 0.35 0.49		Ref. 63 R&D/CRLD/AN /mr/9915513
						Fruit (whole)	0 15 21 30 45	0.65 --- 0.25 0.32 0.45	calculation	
France, 1998 (South) Charly	EC 350 g/l	3	0.76	351	0.22	Fruit without stone (Redwing) Fruit (whole)	26 26		0.24,0.68 <u>0.22</u>, <u>0.63</u>[2]	Ref. 64 R&D/CRLD/AN /mr/9915763 calculation
France, 1998 (South)	EC 350 g/l	3	0.76	571	0.13	Fruit without stone (Manon)	2 h 10 17 24 31	 0.33 0.12	0.81,0.54 0.30,0.35 0.13,0.37	Ref. 65 R&D/CRLD/AN /mr/9915759
						Fruit (whole)	2 h		0.78, 0.52	calculation
							10 17 24 31	 0.31 0.11	0.29, 0.33 <u>0.12</u>, <u>0.35</u>[1]	

[1] According to French GAP
[2] According to Italian GAP

<u>Apricots.</u> Trees were treated by motor pump sprayers in Japan. Plots consisted of 2 and 3 trees and 4 kg field samples were stored frozen for 2 to 3 months before analysis.

Table 21. Phosalone residues in Japanese apricots resulting from a supervised trial in Japan. Underlined residues are from treatments according to GAP.

Year	Form.	Application				Sample (Variety)	PHI, days	Residues, mg/kg	Ref./Report no.
		No.	kg ai/ha	water l/ha	kg ai/hl				
1982	EC 20%	2	0.8	4000	0.02	Fruit without stone (Shiro Kaga)	21 32 47	0.046 0.044 <u>0.009</u>	Ref. 46
		2	0.8	6000	0.02	Fruit without stone (Seigyoku)	21 30 45	0.724 0.698 <u>0.005</u>	

<u>Almonds.</u> Trees were treated with phosalone by experimental atomizer with spray boom and backpack sprayers in supervised trials in France. Plots sizes ranged from 105 m^2 to 140 m^2 with 3 or 4 trees/plot. Field samples of 1000 g were collected and stored frozen for 3 to 8 months before analysis.

Table 22a. Phosalone residues in almonds resulting from supervised trials in Southern France. Analyses of replicate field samples in each trial are shown separately. Double underlined residues are from treatments according to GAP and were used to estimate maximum residue and STMR levels.

Location Year	Form	Application				Sample (Variety)	PHI days	Residues, mg/kg	Ref./Report no.
		No.	kg ai/ha	Water l/ha	kg ai/hl				
1994 St Gilles	SC 500g/l	3	0.75	143	0.52	nut without husk (Ferragnes)	27	<0.02, <0.02	Ref. 47 R&D/CRLD/AN/bd/9516513
		3	1.1	143	0.79		27	<0.02, <0.02	
Ventenac		3	0.75	857	0.09		14	<0.02, <0.02	
		3	1.1	857	0.13		14	0.022, 0.028	
1996 St.Gilles	EC	3	0.77	215	0.36	kernel (Lauranne)	78	<u>≤0.05, ≤0.05</u>	Ref. 48 R&D/CRLD/AN/dbe/9716150
1996 St.Gilles	EC	3	0.77	215	0.36	shell & kernel (Ferragnes) kernel	2 h 29 42 78	4.1, 4.6 2.6, 3.2 3.2, 2.0 <u>≤0.05, ≤0.05</u>	Ref. 49 R&D/CRLD/AN/dbe/9716168
1996 Ventenac	SC 500g/l	3	0.75	571	0.13	nut (Ferragnes)	76	<u>≤0.05</u>, <0.05	Ref. 50 R&D/CRLD/AN/kd/9716028
1996 St.Gilles	SC 500h/l	3	0.75	215	0.35	nut (Ferragnes) kernel	2 h 29 42 78	4.7, 4.5 4.1, 4.4 3.4, 4.4 <u>≤0.05, 0.074</u>	Ref. 51 R&D/CRLD/AN/dbe/9716188

Table 22b. Residues of phosalone oxon in almonds.

Year, location	Form.	Application				Sample (Variety)	PHI days	Residues, mg/kg	Ref./Report no.
		No.	kg ai/ha	water l/ha	kg ai/hl				
1994 St Gilles	SC 500g/l	3	0.75	143	0.52	nut without husk (Ferragnes)	27	<0.02	Ref. 47 R&D/CRLD/AN/bd/9516513
		3	1.1	143	0.79		27	<0.02	
		3	0.75	857	0.09		14	<0.02	
		3	1.1	857	0.13		14	<0.02	

<u>Hazelnuts</u>. Trees were sprayed with phosalone by experimental atomizer with spray boom and backpack sprayers in supervised trials in France. Plots sizes were 45 m^2 to 108 m^2 with 3 to 8 trees/plot. Field samples of 1000g (900g in one trial) were stored frozen for 6 to 8 months before analysis.

Table 23a. Phosalone residues in hazelnuts resulting from supervised trials in Southern France. Analyses of replicate field samples in each trial are shown separately. Double underlined residues are from treatments according to GAP and were used to estimate maximum residue and STMR levels.

Year, location	Form.	Application	Sample	PHI,	Residues, mg/kg	Ref./Report no.

		No.	kg ai/ha	Water l/ha	kg ai/hl	(Variety)	days		
1994 Guitinieres	SC 500g/l	3	0.75	200	0.38	without cup (Ennis)	13	0.026, 0.039	Ref. 52 R&D/CRLD/AN/bd/9515930
		3	1.1	200	0.56		13	0.077, 0.067	
Pollionnay		3	0.75	800	0.09	without cup (Merveille de Bollwiller)	14	0.071, 0.036	
		3	1.1	933	0.12		14	0.079, 0.068	
1996 Pollionnay	SC 500g/l	2	0.6	778	0.08	nuts (Merveille de Bollwiller) kernel without shell	2 h 7 14 21	7.2,6.0 0.061, 0.064 <0.05(2) <u>≤0.05</u>(2)	Ref. 53 R&D/CRLD/AN/dbe/9716158
1996 Puechoursi	EC 350g/l	2	0.61	556	0.11	kernel without shell (Fertile de Coutard)	20	<u>≤0.05</u>(2)	Ref. 54 R&D/CRLD/AN/dbe/9716157
1996 Villesequelande	SC 500g/l	2	0.60	667	0.09	kernel without shell (Merveille de Bollwiller)	22	<u>≤0.05</u>(2)	Ref. 55 R&D/CRLD/AN/dbe/9716159
1996 Guitineres	EC	2	0.61	800	0.08	nuts (Ennis) kernel without shell	2 h 7 13 21	2.3,2.7 0.82,0.76 0.63,0.70 <u>≤0.05</u> (2)	Ref. 56 R&D/CRLD/AN/dbe/9716229

Table 23b Residues of phosalone oxon in hazelnuts.

Country Year	Form.	Application				Sample (Variety)	PHI, days	Residues, mg/kg	Ref./Report no.
		No.	kg ai/ha	Water l/ha	kg ai/hl				
France, 1994	SC	3	0.75	200	0.38	without cup (Ennis)	13	<0.02	Ref. 52 R&D/CRLD/AN/bd/9515930
			1.1	200	0.56		13	<0.02	
			0.75	800	0.09	without cup	14	<0.02	
			1.1	933	0.12		14	<0.02	

<u>Walnuts</u>. Trees were sprayed with phosalone by experimental atomizer with spray boom and backpack sprayers in supervised trials in France. Plots sizes were 49 m^2 to 400 m^2 with 3 to 4 trees/plot. Field samples of 1000 g (1.5 kg in one trial) were stored frozen for 5 to 11 months before analysis.

Table 24. Phosalone residues in walnuts resulting from supervised trials in Southern France. Analyses of replicate field samples in each trial are shown separately. Double underlined residues are from treatments according to GAP and were used to estimate maximum residue and STMR levels.

Year Location	Form.	Application				Sample (Variety)	PHI, days	Residues, mg/kg	Ref./Report no.
		No.	kg ai/ha	Water, l/ha	kg ai/hl				
1994 Barret	SC 500g/l	3	0.75	146	0.51	kernel (Franquette)	14	<0.02(2)	Ref. 57 R&D/CRLD/AN/bd/ 9515931
		3	1.1	150	0.75		14	<0.02(2)	
Chatte		3	0.75	530	0.14	kernel (Chico)	14	0.18,0.14	
		3	1.1	530	0.21		14	0.13,0.15	
1996 Marches	EC 350g/l	4	0.41	232	0.18	kernel (Franquette)	2 h	<0.05(2)	Ref. 58 R&D/CRLD/AN/vt/9 716585
						whole walnut (nut, shell & hull)	7	1.3,1.8	Ref. 58 R&D/CRLD/AN/981 7001
						whole walnut (nut, shell & hull)	14	0.81,0.89	Ref. 58 R&D/CRLD/AN/981 7001
						kernel	21	<0.05, 0.055	Ref. 58 R&D/CRLD/AN/vt/9 716585
1996 Marches	SC 500g/l	4	0.40	233	0.17	kernel (nut) (Franquette)	21	0.19,0.09 0.16,0.09	Ref. 59 R&D/CRLD/AN/vt/9 716568
1996 La Lustre	EC 350g/l	4	0.61	141	0.43	kernel (nut) (Franquette)	28	<0.05(2)	Ref. 60 R&D/CRLD/AN/vt/9 716561
1996 La Lustre	SC 500g/l	4	0.61	141	0.43	kernel (nut) (Franquette)	2 h	<0.05(2)	Ref. 61 R&D/CRLD/AN/dbe /9716595
							7	<0.05, 0.052	
							14	<u>≤0.05</u>(2)	
							28	<0.05(2)	

RESIDUES IN STORAGE AND PROCESSING

In storage

No information.

In processing

<u>Apples</u>. In a processing trial according to commercial practice (Maestracci, 1999) apples were treated four times at an application rate of 0.6 kg ai/ha with 300 l/ha of an SC formulation. Samples taken 76 days after the last application were processed into compote and pulp.

To produce compote, the apples were peeled by rotating knives, cooked for 10 min. at 100 °C, then stored in holding tanks at 80°C for 1 to 3 h. Samples were filtered, combined with sugar etc. and heated first at 105°C, then at 92°C, before packing as infant food.

About 86% of the phosalone was lost during processing into compote (Table 25).

Table 25. Effect of processing on phosalone residues in apples.

Sample	Mean residue, mg/kg	Processing factor
Whole apple	0.19	1.00

Sample	Mean residue, mg/kg	Processing factor
Peeled apple	0.13	0.68
Unpeeled, washed apple	0.20	1.05
Boiled pulp from peeled apple	0.030	0.16
Boiled pulp from unpeeled washed apple	0.055	0.29
Compote from whole apple	0.026	0.14
Compote from unpeeled washed apple	0.054	0.28

NATIONAL MAXIMUM RESIDUE LIMITS

The Meeting was informed of the national MRLs shown in the Table below.

Country	MRL, mg/kg	Commodity
Austria	1	Fruit except pome fruit and peach
	2	Peach
Belgium	2	Peach, pome fruit
Belarus	0.2	Pome and stone fruit
Bulgaria	2	Apple
Canada	2	Pear
	4	Apricot, peach
	5	Apple, plum
	6	Cherry
	12	Apricot (dried)
Croatia	2	Fruit
Czech Republic	2	Apple, stone fruit
Finland	2	Peach, pome fruit
France	0.1	Almond, hazelnut
	0.2	Walnut
	2	Pome fruit, stone fruit (including cherry)
Hungary	1	Fruit
India	0.5	Fruit except pears and citrus fruit
	2	Pear
Italy	2	Peach, pome fruit
Japan	1	Fruit (general)
Luxemburg	2	Peach, pome fruit
Macedonia	2	Apple, peach
Moldavia	0.2	Pome and stone fruit
Netherlands	1	Fruit except peach and pome fruit
	2	Peach, pome fruit
Poland	0.1	Fruit except strawberry, citrus fruit
Russia	0.2	Pome and stone fruit
Spain	2	Pome and stone fruit
Sweden	2	Peach, pome fruit
Switzerland	2	Peach, pome fruit
Taiwan	1	Pear
Turkey	0.5	Peach, plum, cherry
UK	2	Peach, pome fruit
Yugoslavia	2	Apple, peach

APPRAISAL

Phosalone was the subject of a periodic review of residues by the 1994 JMPR. That Meeting concluded that the existing CXLs for phosalone should be withdrawn, owing to inadequacies in the available information. The CCPR decided to maintain the CXLs for 4 years. A periodic review of the toxicology was carried out by the 1997 JMPR, which allocated an ADI of 0-0.02 mg/kg bw.

The Meeting received new or revised information on physical and chemical properties, metabolic and environmental fate, analytical methods, stability of analytical samples, use patterns, supervised trials, apple processing, and national MRLs. A new determination of the octanol/water partition coefficient gave a value of log P_{ow} = 4.01 at 20°C.

Plant metabolism

Phosalone was typically the main residue; phosalone oxon and 6-chlorobenzoxazolone were found at low levels in apples. In grapes, phosalone was the main residue in the juice and constituted more than 95% of the residue in the pulp.

Environmental fate

Photolysis. In water at pH 5, decomposition is very rapid (half-life 15-20 minutes). The quantum yield at 300 nm for phosalone in aqueous solution was determined to be 0.19. In the troposhere the estimated reaction constant at 298° K is 9.34 x 10^{-3} s^{-1} which corresponds to a half-life of about 74 daylight seconds.

Biodegradability. In active sludge about 20% of the initial radioactivity of [^{14}C]phosalone was detected as $^{14}CO_2$ after six weeks.

Aerobic degradation in soil. Phosalone was degraded rapidly with a half-life of 2.9 days. Unextracted radiocarbon increased to an average of 85% by 30 days. The product phenoxazone was observed but did not exceed 1.5% of the applied radioactivity.

Anaerobic degradation in soil. In the water phase, phosalone was rapidly degraded to 2-amino-5-chlorophenol which reached a maximum of 20% of the applied radioactivity after 3 days, then decreased. In the soil phase phosalone was also rapidly degraded, producing chloroaminophenol which reached a maximum of 8% of the applied radioactivity after 14 days.

Soil adsorption/desorption was studied in sandy loam, silty clay loam, loam and clay. The average K_{oc} value for adsorption on the three loam soils was 2060. Degradation on clay was too rapid to measure adsorption. Phosalone is predicted to have slight to low mobility in soils.

Environmental fate in water/sediment systems. In river and ditch systems 68% and 65% of the applied radioactivity was bound after 12 weeks. In the aqueous phases four degradation products were found but not identified. They did not exceed 10% of the applied radioactivity. In the sediment phosalone was the main residue, but did not exceed 6% of the applied radioactivity.

Bioaccumulation. A dynamic 42-day study was conducted to determine the uptake of radiolabelled phosalone by bluegill sunfish. The uptake rate constant for whole fish was 0.18 (± 0.02) mg/kg fish/mg/l water/day. The bio-concentration factors were 280 to 300 for viscera, 78 to 85 for edible tissue and 190 to 200 for whole fish.

Analytical methods. In general residues are extracted with acetone/water and cleaned up by liquid-liquid partition with dichloromethane. The final extract is concentrated and analysed by GLC with EC, NP or FP detection. There are several variants.

A number of residue analytical methods were described in the 1994 JMPR monograph. The LODs for plants were generally 0.05 mg/kg with an ECD, FPD or NPD.

In a more recent method for phosalone and phosalone oxon the residues are extracted with acetone and cleaned up by partitioning between water and dichloromethane. Quantification is with an FPD. The LODs are 0.02 mg/kg for both compounds.

Stability of residues on stored analytical samples. Fortified peaches and almonds were stored frozen at −8°C for 1 and 3 months. Recoveries after storage were 94 to 102% from peaches and 79 to 81% from almonds. Almonds, apples and cherries with incurred residues were stored at −18°C for 19 to 24 months. The recovery of phosalone was 66 to 77% from almonds, 67 to 70% from apples and 113 to 133% from cherries.

Definition of the residue

The current definition of the residue is "phosalone". A metabolism study on apples showed that phosalone was the predominant residue (75%-92%) with only 2%-7% of the oxon. In grapes phosalone was about 90% or more of the residue with only 1-2% of the oxon. The Meeting concluded that the current residue definition is suitable both for compliance with MRLs and for the estimation of dietary intake.

The octanol-water partition coefficient (log P_{ow} = 4.01) suggests fat-solubility. The Meeting concluded that phosalone is fat-soluble.

Residues resulting from supervised trials

Pome fruits. Phosalone may be used at 0.06 kg ai/hl (0.6 kg ai/ha, standard orchard spray volume: 1000 l/ha) on apples and pears in France with a PHI of 14 days for EC, 15 days for SC. The residues in apples and pears from 5 French, 1 German, 1 Italian and 1 Spanish trials meeting these conditions were 1.1, 0.38, 0.74, 0.66, 1.5, 1.0, 0.46 and 0.38 mg/kg.

In Italy, phosalone may be used at 0.05-0.07 kg ai/hl on apples and pears with a PHI of 21 days. Several of the trials complied with both French and Italian GAP. The residues from 5 Italian, 3 French and 1 Spanish trials meeting these conditions were 0.6, 0.65, 0.85, 0.96, 0.74, 0.52, 1.5, 1.0, 0.91 and 0.36 mg/kg.

Phosalone residues in apples and pears from the 5 Italian, 5 French, 1 German and 1 Spanish trials in rank order (median underlined) were 0.38 (2), 0.46, 0.65, 0.66, 0.74, 0.85, 0.91, 0.96, 1.0, 1.1 and 1.5 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.8 mg/kg for phosalone in pome fruits.

Stone fruits. Phosalone may be used on cherries and peaches in France at 0.06 kg ai/hl (0.6 kg ai/ha) with a PHI of 14 days. The residues from 16 trials on cherries and 4 on peaches meeting these conditions were 0.18-1.6 mg/kg and 0.16-1.5 mg/kg respectively. The trials on peaches also complied with Italian GAP: 0.06-0.07 kg ai/hl with a PHI of 21 days. The residues in peaches from 2 Italian and 4 French trials meeting these conditions were 0.13, 0.68, 1.4, 0.45, 0.63 and 0.31 mg/kg.

The Meeting concluded that the residues in cherries and peaches belonged to the same population.

The residues in cherries and peaches from the French and Italian trials in rank order (median underlined) were 0.16, 0.18, 0.23, 0.26(2), 0.29, 0.3, 0.35, 0.36, 0.42, 0.45 (2), 0.46, 0.47, 0.59, 0.6, 0.63, 0.72, 0.73, 1.4, 1.5 and 1.6 mg/kg.

Apricots. Phosalone may be used at 0.02 kg ai/hl on Japanese apricots in Japan with a PHI of 45 days. The residues in Japanese apricots from 2 trials in Japan meeting these conditions were 0.005 and 0.009 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg and an STMR of 0.45 mg/kg for phosalone on stone fruits.

Tree nuts. Phosalone may be used on almonds in France at 0.06-0.075 kg ai/hl (0.60-0.75 kg ai/ha) with a PHI of 70 days. The residues in almonds from 6 French trials meeting these conditions in rank order (median underlined) were <0.02 (2), <0.05 (3) and 0.074 mg/kg.

The Meeting estimated a maximum residue level of 0.1 mg/kg and an STMR of 0.05 mg/kg for phosalone in almonds.

Phosalone may be used on hazelnuts and walnuts in France at 0.06 kg ai/hl (0.60 kg ai/kg) with a PHI of 21 days. The residues in hazelnuts from 4 French trials and in walnuts from 1 French trial meeting these conditions were all <0.05 mg/kg.

The Meeting estimated a maximum residue level of 0.05* mg/kg and an STMR of 0.05 mg/kg for phosalone in hazelnuts and walnuts.

Processing

Apples were processed to compote with a processing factor of 0.14. As the STMR for pome fruits is 0.8 mg/kg the Meeting estimated an STMR of 0.1 mg/kg for phosalone in apple compote.

RECOMMENDATIONS

On the basis of data from supervised trials the Meeting estimated the maximum residue levels and STMRs listed below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue for compliance with MRLs and for estimation of dietary intake: phosalone. The residue is fat-soluble.

Commodity		MRL, mg/kg		STMR, mg/kg
CCN	Name	New	Previous	
TN 0660	Almonds	0.1	-	0.05
	Apple compote		-	0.1
TN 0666	Hazelnuts	0.05*	-	0.05
FP 0009	Pome fruits	2	-[1]	0.8
FS 0012	Stone fruits	2	-	0.45
TN 0678	Walnuts	0.05*	-	0.05

[1]A CXL of 5 mg/kg for apple was recommended for withdrawal by the 1994 JMPR

The international estimated short-term intake (IESTI) for phosalone was calculated for the commodities for which maximum residue levels and STMRs were estimated and for which consumption data were available. The results are shown in Annex IV of the 1999 JMPR Report. The IESTI varied from 0 to 0.034 mg/kg bw for the general population and from 0 to 0.118 mg/kg bw for children. As no acute reference dose was established, the acute risk assessment for phosalone was not finalized.

DIETARY RISK ASSESSMENT

Chronic intake

Six STMRs were estimated for phosalone. There were consumption data for the 5 main commodities which were used for the dietary intake calculation. The results are shown in Annex III.

The International Estimated Daily Intakes for the 5 GEMS/Food regional diets, based on estimated STMRs, were in the range of 0-4% of ADI. The Meeting concluded that the intake of residues of phosalone from uses that have been considered by the JMPR is unlikely to present a public health concern.

Acute intake

The international estimated short-term intake (IESTI) for phosalone was calculated for the commodities for which maximum residue levels and STMRs were estimated and for which consumption data were available. The results are shown in Annex IV. The IESTI varied from 0 to 0.034 mg/kg bw for the general population and from 0 to 0.118 mg/kg bw for children. As no acute reference dose was established, the acute risk assessment for phosalone was not finalized.

REFERENCES

References are listed alphabetically and then also numerically in order of their occurrence in the monograph.

1. Alphabetical list

Anon. 1980. Report on pesticide residue analysis. Official trials. Phosalone on apple Chuo University, 18 pp.

Anon. 1982. Report on pesticide residue analysis. Official trials. Phosalone on peach from application of Ranbeck (phosalone 20% + dichlorvos 40%). Japan Food Analysis Federation, 2 pp.

Anon. 1982. Report on pesticide residue analysis. Official trials. Phosalone on Japanese apricot from application of Ranbeck (phosalone 20% + dichlorvos 40%). Azabu University, 2 pp.

Anon. 1989. Report on pesticide residue analysis. Official trials. Phosalone on Japanese pear. Japan Food Analysis Federation, 16 pp.

Anon. 1997. Pesticide Analytical Manual Vol. I. Multiresidue methods, 3rd edition, revised Oct. 1997 US Food and Drug Administration (selected pp).

Boinay, P. 1994. Determination of the direct phototransformation of phosalone in water. Study Number 94-092 ENH/ENV CRIT Document No. CP/MAN/ENH/338/94/0393. Report R&D/CRLD/AN/bd 9416379. Rhône-Poulenc Industrialisation, 29 pp.

Bourgade, C., and Yslan, F. 1998. Phosalone: confirmatory methods for the determination of residues in plants. RP Analytical Method. Reference AR 159-97 RP. Report No. R&D/CRLD/AN/ 9815432. Rhône-Poulenc Agro (CRLD), 24 pp.

Clarke, D.E. Residue1999. [14C]-Phosalone: anaerobic soil degradation. RPAL. Study No. 14543, RPA Document No. 201926. Rhône-Poulenc Agriculture Ltd., 156 pp.

Cousin, J.A. 1995. Phosalone Active Ingredient Octanol/water partition coefficient. Study No. 94-161, Report No. R&D/CRLD/AN/9515083. Rhône-Poulenc Agro (CRLD), 16 pp.

Cousin, J.A. 1997. Phosalone active ingredient Water and solvent solubility RPA Study No. 97-09, Report

No. R&D/CRLD/AN/ 9715915. Rhône-Poulenc Agro (CRLD), 25 pp.

Cousin, J.A. 1997. Phosalone active ingredient - oxidizing properties. Report No. R&D/CRLD/AN/ 9716048, 1 page.

Fillion, J. 1997. Determination of the explosion properties, flammability, ability for self heating of technical phosalone RPA Study No 97-137-SEC. Rhône-Poulenc Agro (CRLD), 13 pp.

Forbis, A.D., Schoen, L.T., and Stuerman, L. 1986. Uptake, depuration and bioconcentration of 14C-phosalone by bluegill sunfish (Lepomis macrochirus) ABC report no. 43283 Analytical Bio-Chemistry Laboratories, Inc., 74 pp.

Gabereau, M. 1997. Phosalone Analytical method for the determination of residues in plants. Study No. 97-97-18. RPA Analytical Method Reference AR 148-97. RP Report No. R&D/CRLD/AN/ 9716342. Rhône-Poulenc Agro (CRLD), 40 pp.

Hoffman, M.J. 1989. Vapor pressure determination of phosalone. Project No. HLA 6001-362. Hazleton Laboratories America, 70 pp.

Jendrzejzac. 1994. Molar extinction coefficient of phosalone, 290-800 nm.

Kimmel, E.C. 1990. A metabolism study with 14C-phosalone on apples (Pyrus malus). PTRL-W project no. PTRL-W 181-W. RPAC Study No EC-89-026 PTRL West, Inc., 89 pp.

Laurent, M., and Buys, M. 1975. Phosalone Solubility and stability in water. Report No. R.P./R.D./C.N.G. An no. 2793. Rhône-Poulenc (CRV), 21 pp.

Laurent, M., Buys, M., and Chabassol, Y. 1977. Phosalone Photodegradation in the water-dissolved state. Report No. RP/RD/CNG/AN/3131. Rhône-Poulenc (CRV), 29 pp.

Le Gren, I. 1997. Phosalone and its metabolite RP 18726: analytical method for the determination of residues in soil. RPA Method ref. AR 141-97. Doc. No. R&D/CRLD/AN/vt/9715716. Rhône-Poulenc Agro (CRLD), 55 pp.

Lusson, R., and Muller, M.A. 1990. Phosalone - Zolone 24 L(EC) Formulation Tests in Italy 1989. Residues in leaves and fruit of apples. Report No. AG/CRLD/AN/9015907 (In French). Rhône-Poulenc Agro (CRLD), 16 pp.

Maestracci, M.P. 1994. Phosalone - estimation of the rate of photochemical transformation in the atmosphere under tropospheric conditions. Study No. 94-123, Document No. R&D/CRLD/AN/9416675. Rhône-Poulenc Agro (CRLD), 14 pp.

Maestracci, M. 1995. Calculation of environmental photolytic half-life of phosalone in water. Report No. R&D/CRLD/AN/9515814. Rhône-Poulenc Agro (CRLD), 14 pp.

Maestracci, M. 1997a. Phosalone Formulation EXP 06027A (SC). Trials France 1995. Residues in apples. Decline study RPA Study No. 95-568. Report No. R&D/CRLD/AN/kd/9616621. Rhône-Poulenc Agro (CRLD), 47 pp.

Maestracci, M. 1997b. Phosalone Formulation EXP 05531D (EC). Trial France 1996. Residues in apple. Decline study (reverse curve). RPA Study No. 96-775 RPA Report No. R&D/CRLD/AN/dbe/ 9716155. Rhône-Poulenc Agro (CRLD), 62 pp. Unpublished report.

Maestracci, M. 1997c. Phosalone Formulation EXP 05531D (EC). Trials France 1996. Residues in apple. Decline study. RPA Study No. 96-776. RPA Report No. R&D/CRLD/AN/vt/ 9716613. Rhône-Poulenc Agro (CRLD), 50 pp. Unpublished report.

Maestracci, M. 1997d. Phosalone Formulation EXP 06027B (SC). Trials France 1996. Residues in cherry. Decline study. RPA Study No. 96-542. Report No. R&D/CRLD/AN/dbe/ 9716189. Rhône-Poulenc Agro (CRLD), 70 pp.

Maestracci, M. 1997e. Phosalone Formulation EXP 05531D (EC). Trial France 1996. Residues in almond. RPA Study No. 96-528. RPA Report No. R&D/CRLD/AN/dbe/ 9716150. Rhône-Poulenc Agro (CRLD), 44 pp.

Maestracci, M. 1997f. Phosalone Formulation EXP 05531D (EC). Trial France 1996. Residues in almond. Decline study. RPA Study No. 96-530. Report No. R&D/CRLD/AN/dbe/ 9716168. Rhône-Poulenc Agro (CRLD), 47 pp.

Maestracci, M 1997g. Phosalone Formulation EXP 06027B (SC). Trial France 1996. Residues in almond. RPA Study No. 96-525. Report No. R&D/CRLD/AN/kd/ 9716028. Rhône-Poulenc Agro (CRLD), 44 pp.

Maestracci, M. 1997h. Phosalone Formulation EXP 06027B (SC). Trial France 1996. Residues in almond. Decline study. RPA Study No. 96-529. RPA Report No. R&D/CRLD/AN/dbe/ 9716188. Rhône-Poulenc Agro (CRLD), 47 pp.

Maestracci, M 1997i. Phosalone Formulation EXP 06027B (SC). Trial France 1996. Residues in hazelnut. Decline study. RPA Study No. 96-551. RPA Report No. R&D/CRLD/AN/dbe/ 9716158. Rhône-Poulenc Agro (CRLD), 48 pp.

Maestracci, M. 1997j. Phosalone Formulation EXP 05531D (SC). Trial France 1996. Residues in hazelnut. RPA Study No. 96-550. RPA Report No. R&D/CRLD/AN/dbe/ 9716157. Rhône-Poulenc Agro (CRLD), 44 pp.

Maestracci, M. 1997k. Phosalone Formulation EXP 06027B (SC). Trial France 1996. Residues in hazelnut.

RPA Study No. 96-549. RPA Report No. R&D/CRLD/AN/dbe/ 9716159. Rhône-Poulenc Agro (CRLD), 43 pp.

Maestracci, M. 1997l. Phosalone Formulation EXP 05531D (SC). Trial France 1996. Residues in hazelnut. Decline study. RPA Study No. 96-548. RPA Report No. R&D/CRLD/AN/dbe/ 9716229. Rhône-Poulenc Agro (CRLD), 47 pp.

Maestracci, M. 1997m. Phosalone Formulation EXP 05531D (EC). Trial France 1996. Residues in walnut. Decline study. RPA Study No. 96-546. RPA Report No. R&D/CRLD/AN/vt/ 9716585. Report Amendment 1 as R&D/CRLD/AN/ 9817001 (5 January 1999). Rhône-Poulenc Agro (CRLD), 49 pp.

Maestracci, M. 1997n. Phosalone Formulation EXP 06027B (SC). Trial France 1996. Residues in walnut. RPA Study No. 96-545. RPA Report No. R&D/CRLD/AN/vt/ 9716568. Rhône-Poulenc Agro (CRLD), 44 pp.

Maestracci, M. 1997o. Phosalone Formulation EXP 05531D (EC). Trial France 1996. Residues in walnut. RPA Study No. 96-544. RPA Report No. R&D/CRLD/AN/vt/ 9716561. Rhône-Poulenc Agro (CRLD), 44 pp.

Maestracci, M. 1997p. Phosalone Formulation EXP 06027B (SC). Trial France 1996. Residues in walnut. Decline study. RPA Study no. 96-547. RPA Report No. R&D/CRLD/AN/dbe/ 9716595. Rhône-Poulenc Agro (CRLD), 45 pp.

Maestracci, M. 1998a. Phosalone Formulation EXP 05531D (EC). Trial France 1997. Residues in apple. RPA Study No. 97-602 RP. Report No. R&D/CRLD/AN/vt/ 9815382. Rhône-Poulenc Agro (CRLD), 43 pp

Maestracci, M. 1998b. Phosalone Formulation EXP 05531D (EC). Trial France 1997. Residues in apple. Reverse curve. RPA Study No. 97-601 RP. Report No. R&D/CRLD/AN/dbe/ 9815324. Rhône-Poulenc Agro (CRLD), 53 pp.

Maestracci, M. 1998c. Phosalone Formulation EXP 60308B (WP). Trial Italy 1997. Residues in apple. RPA Study No. 97-624. RPA Report No. R&D/CRLD/AN/vt/ 9815916. Rhône-Poulenc Agro (CRLD), 74 pp.

Maestracci, M. 1998d. Phosalone Formulation EXP 60308B (WP). Trials Germany 1997. Residues in apple and in pear. Reverse curve. Study No. 97-767. RPA Report No. R&D/CRLD/AN/msa/ 9816476. Rhône-Poulenc Agro (CRLD), 116 pp.

Maestracci, M. 1998e. Phosalone Formulation EXP 60308B (WP). Trial Spain 1997. Residues in pear. Reverse curve. RPA Study No. 97-702. RPA Report No. R&D/CRLD/AN/vt/ 9815818. Rhône-Poulenc Agro (CRLD), 44 pp.

Maestracci, M. 1998f. Phosalone Formulation EXP 05531D (EC). Trial France 1997. Residues in cherry. Decline study. RPA Study No. 97-554 RPA. Report No. R&D/CRLD/AN/vt/ 9815381. Rhône-Poulenc Agro (CRLD), 70 pp.

Maestracci, M. 1998g. Phosalone Formulation EXP 05531D (EC). Trial France 1997. Residues in peach. Reverse curve. RPA Study No. 97-522 RP. Report No. R&D/CRLD/AN/dbe/ 9815342. Rhône-Poulenc Agro (CRLD), 44 pp.

Maestracci, M. 1998h. Phosalone Formulation EXP 05531D (EC). Trial France 1997. Residues in peach. RPA Study No. 97-523. RPA Report No. R&D/CRLD/AN/vt/ 9815817. Rhône-Poulenc Agro (CRLD), 45 pp.

Maestracci, M. 1998i. Phosalone Formulation EXP 60308 (WP). Trial Italy 1997. Residues in peach. Decline study. RPA Study No. 97-622. RPA Report No. R&D/CRLD/AN/dbe/ 9815848. Rhône-Poulenc Agro (CRLD), 77 pp.

Maestracci, M. 1998j. Phosalone Formulation EXP 60308 (WP). Trial Spain 1997. Residues in peach. RP. Study No. 97-701. RPA Report No. R&D/CRLD/AN/dbe/ 9815325. Rhône-Poulenc Agro (CRLD), 42 pp.

Maestracci, M. 1999. Phosalone Formulation EXP 06027B (SC). Residues in apple and processed products. Trial France 1996. RPA Study No. 96-694. Report R&D/CRLD/AN/dbe/ 9816999. Rhône-Poulenc Agro (CRLD), 51 pp.

Muller, M.A. 1989. Phosalone - Zolone 25 L (EC) Formulation Tests in Italy, 1989. Residues in apples. Decline curves. Report No. AG/CRLD/AN/8916827 (in French). Rhône-Poulenc Agro (CRLD), 18 pp.

Muller, M.A. 1989. Phosalone - Zolone 25 L (EC) Formulation Tests in Italy, 1989. Residues in peaches. Decline curves. Report No. AG/CRLD/AN/8916828. Rhône-Poulenc Agro (CRLD), 17 pp.

Muller, M.A. 1995a. Phosalone et oxo-phosalone (metabolite). Formulation EXP 06207A (SC). Essais France 1994. Residus dans la cerise. Etude No. 94-545. Report R&D/ CRLD/ An/ fb/ 9515875 (in French). Rhône-Poulenc Agro (CRLD), 51 pp.

Muller, M.A. 1995b. Phosalone et oxo-phosalone (metabolite). Formulation EXP 06027A (SC). Essais France 1994. Residus dans l'amande. Etude No. 94-543 Rapport R&D/CRLD/AN/bd/ 9516513 (in French). Rhône-Poulenc Agro (CRLD), 48 pp.

Muller, M.A. 1995c. Phosalone et oxo-phosalone (metabolite). Formulation EXP06027A (SC). Essais France 1994. Residus dans la noisette. Etude No. 94-547. Report No. R&D/CRLD/AN/bd/ 9515930 (in French). Rhône-Poulenc Agro (CRLD), 47 pp.

Muller, M.A. 1995d. Phosalone et oxo-phosalone (metabolite). Formulation EXP06027A (SC). Essais

France 1994. Residus dans la noix (amande). Etude No. 94-548. Report No. R&D/CRLD/AN/bd/ 9515931 (in French). Rhône-Poulenc Agro (CRLD), 47 pp.

Muttzall, P.I. 1995. Biodegradation of phosalone in a water/sediment system. Supplement to TNO report R87/245a (22 Sept 1987). TNO Study No. MTB-185 TNO Institute of Environmental Sciences, 8 pp.

Muttzall, P.I., and Hanstveit, A.O. 1989. Biodegradability of phosalone according to OECD Guideline 301B (modified Sturm test). TNO report no. R 88/428. Netherlands Organization for Applied Scientific Research, 29 pp.

Muttzall, P.I., and de Kreuk, J.F. 1987. Biodegradation of phosalone in a water/sediment system. TNO. report no. R 87/245a. TNO Institute of Environmental Sciences, 34 pp.

Periasamy, R., Kimmel, E.C., and Toia, R.F. 1995. A metabolism study with [14C]-phosalone on grapes. PTRL. Report No. 385W-1 PTRL West, Inc., 309 pp.

Pollard, J.W. 1987 Specific gravity determination of technical grade phosalone insecticide. Project No. 785F10. File No. 40156. Rhône-Poulenc Ag. Co., 2 pp.

Quintelas, G. 1998. Stability study of phosalone in fruits after storage in a freezer at a temperature near -18°C. Intermediary results. RPAC study no. 98-162. Defitraces. Study RPA/98-072 Defitraces, 25 pp.

Skinner, W. 1995. Soil adsorption/desorption of [14C]Phosalone by the Batch Equilibrium Method (OECD 106, 1981). PTRL. Report No. 498W-1. PTRL West, Inc., 134 pp.

Skinner, W., and Jao, N. 1995. Aerobic Soil Metabolism of [14C]Phosalone. PTRL. Report No. 497W-1. PTRL West, Inc. 138 pp

2. Numerical list

1 Cousin, J.A. 23 Jan 1995. Phosalone Active Ingredient. Octanol/water partition coefficient. Study No. 94-161, Report No. R&D/CRLD/AN/9515083. Rhone-Poulenc Agro (CRLD). 16 pp.

2 Cousin, J. 23 Jun 1997. Phosalone active ingredient. Water and solvent solubility. RPA Study No. 97-09, Report No. R&D/CRLD/AN/ 9715915. Rhone-Poulenc Agro (CRLD). 25 pp.

3 Laurent, M., and Buys, M. 30 Jul 1975. Phosalone. Solubility and stability in water. Report No. R.P./R.D./C.N.G. An no. 2793. Rhone-Poulenc (CRV). 21 pp.

4 Laurent, M., Buys, M., and Chabassol, Y. 3 Mar 1977. Phosalone. Photodegradation in the water-dissolved state. Report No. RP/RD/CNG/AN/3131. Rhone-Poulenc (CRV). 29 pp.

5 Boinay, P. 15 Sep 1994. Determination of the direct phototransformation of phosalone in water. Study Number 94-092 ENH/ENV. CRIT Document No. CP/MAN/ENH/338/94/0393. Report R&D/CRLD/AN/bd 9416379. Rhone-Poulenc Industrialisation. 29 pp.

6 Pollard, J.W. 27 Oct 1987. Specific gravity determination of technical grade phosalone insecticide. Project No. 785F10. File No. 40156. Rhone-Poulenc Ag Co. 2 pp.

7 Hoffman, M.J. 31 Jan 1989. Vapor pressure determination of phosalone. Project No. HLA 6001-362. Hazleton Laboratories America. 70 pp.

8 Fillion, J. 6 May 1997. Determination of the explosion properties, flammability, ability for self heating of technical phosalone. RPA Study No 97-137-SEC. Rhone-Poulenc Agro (CRLD). 13 pp.

9 Cousin, J. 9 Jul 1997. Phosalone active ingredient - oxidizing properties. Report No. R&D/CRLD/AN/ 9716048. 1 pp.

10 Kimmel, E.C. 20 Dec 1990. A metabolism study with 14C- phosalone on apples (Pyrus malus). PTRL-W project no. PTRL-W 181-W. RPAC Study No. EC-89-026. PTRL West, Inc. 89 pp.

11 Periasamy, R., Kimmel, E.C., and Toia, R.F. 13 Dec 1995. A metabolism study with [14C]-phosalone on grapes. PTRL Report No. 385W-1. PTRL West, Inc. 309 pp.

12 Maestracci, M. 31 May 1995. Calculation of environmental photolytic half-life of phosalone in water. Report No. R&D/ CRLD/ AN/ 9515814. Rhone-Poulenc Agro (CRLD). 14 pp.

13 Maestracci, M.P. 18 Nov 1994. Phosalone - estimation of the rate of photochemical transformation in the atmosphere under tropospheric conditions. Study No. 94-123, Document No. R&D/CRLD/AN/9416675. Rhone-Poulenc Agro (CRLD). 14 pp.

14 Muttzall, P.I., and Hanstveit, A.O. 16 Jan 1989. Biodegradability of phosalone according to OECD Guideline 301B (modified sturm-test). TNO report no R 88/428. Netherlands Organization for Applied Scientific Research. 29 pp.

15 Skinner, W., and Jao, N. 9 Jun 1995. Aerobic Soil Metabolism of [14C]Phosalone. PTRL Report No. 497W-1. PTRL West, Inc. 138 pp.

16 Clarke, D.E. 28 Jan 1999. [14C]-Phosalone: anaerobic soil degradation. RPAL Study No. 14543, RPA Document No. 201926. Rhone-Poulenc Agriculture, Ltd. 156 pp.

17 Skinner, W. 27 Mar 1995. Soil adsorption/desorption of [14C]Phosalone by the Batch Equilibrium Method (OECD 106, 1981). PTRL Report No. 498W-1. PTRL West, Inc. 134 pp.

18 Muttzall, P.I., and de Kreuk, J.F. 22 Sep 1987. Biodegradation of phosalone in a water/sediment system. TNO Report No. R 87/245a. TNO Institute of Environmental Sciences. 34 pp.

19 Muttzall, P.I. 26 Jan 1995. Biodegradation of phosalone in a water/sediment system. Supplement to TNO report R87/245a (22 Sept 1987). TNO Study No. MTB-185. TNO Institute of Environmental Sciences. 8 pp.

20 Forbis, A.D., Schoen, L.T., and Stuerman, L. 17 Oct 1986. Uptake, depuration and bioconcentration of 14C-phosalone by bluegill sunfish (Lepomis macrochirus). ABC report no. 43283. Analytical Bio-Chemistry Laboratories, Inc. 74 pp.

21 Gabereau, M. 3 Sep 1997. Phosalone. Analytical method for the determination of residues in plants. Study No. 97-97-18. RPA Analytical Method Reference AR 148-97. RP Report No. R&D/CRLD/AN/ 9716342. Rhone-Poulenc Agro (CRLD). 40 pp.

22 Bourgade, C., and Yslan, F. 3 Apr 1998. Phosalone: confirmatory methods for the determination of residues in plants. RP Analytical Method Reference AR 159-97. RP Report No. R&D/CRLD/AN/ 9815432. Rhone-Poulenc Agro (CRLD). 24 pp.

23 Le Gren, I. 30 May 1997. Phosalone and its metabolite RP 18726: analytical method for the determination of residues in soil. RPA Method ref. AR 141-97. Doc No. R&D/CRLD/AN/vt/9715716. Rhone-Poulenc Agro (CRLD). 55 pp.

24 Quintelas, G. 29 Dec 1998. Stability study of phosalone in fruits after storage in a freezer at a temperature near -18C. Intermediary results. RPAC study no. 98-162. Defitraces Study RPA/98-072. Defitraces. 25 pp.

25 Muller, M.A. 28 Nov 1989. Phosalone - Zolone 25 L (EC) Formulation. Tests in Italy, 1989. Residues in apples. Decline curves. Report No. AG/CRLD/AN/8916827. (In French). Rhone-Poulenc Agro (CRLD). 18 pp.

26 Lusson, R., and Muller, M.A. 18 Jun 1990. Phosalone - Zolone 24 L(EC) Formulation. Tests in Italy 1989. Residues in leaves and fruit of apples. Report No. AG/CRLD/AN/9015907. (In French). Rhone-Poulenc Agro (CRLD). 16 pp.

27 Maestracci, M. 9 Jan 1997. Phosalone. Formulation EXP 06027A (SC). Trials France 1995. Residues in apples. Decline study. RPA Study No. 95-568. Report No. R&D/CRLD/AN/kd/9616621. Rhone-Poulenc Agro (CRLD). 47 pp.

28 Maestracci, M. 1 Sep 1997. Phosalone. Formulation EXP 05531D (EC). Trial France 1996. Residues in apple. Decline study (reverse curve). RPA Study No. 96-775. RPA Report No. R&D/CRLD/AN/dbe/ 9716155. Rhone-Poulenc Agro (CRLD). 62 pp. Unpublished report submitted by Rhone-Poulenc

29 Maestracci, M. 21 Nov 1997. Phosalone. Formulation EXP 05531D (EC). Trials France 1996. Residues in apple. Decline study. RPA Study No. 96-776. RPA Report No. R&D/CRLD/AN/vt/ 9716613. Rhone-Poulenc Agro (CRLD). 50 pp. Unpublished report submitted by Rhone-Poulenc

30 Maestracci, M. 30 Mar 1998. Phosalone. Formulation EXP 05531D (EC). Trial France 1997. Residues in apple. RPA Study No. 97-602. RP Report No. R&D/CRLD/AN/vt/ 9815382. Rhone-Poulenc Agro (CRLD). 43 pp. Unpublished report submitted by Rhone-Poulenc

31 Maestracci, M. 3 Apr 1998. Phosalone. Formulation EXP 05531D (EC). Trial France 1997. Residues in apple. Reverse curve. RPA Study No. 97-601. RP Report No. R&D/CRLD/AN/dbe/ 9815324. Rhone-Poulenc Agro (CRLD). 53 pp. Unpublished report submitted by Rhone-Poulenc

32 Maestracci, M. 18 Jun 1998. Phosalone. Formulation EXP 60308B (WP). Trial Italy 1997. Residues in apple. RPA Study No. 97-624. RPA Report No. R&D/CRLD/AN/vt/ 9815916. Rhone-Poulenc Agro (CRLD). 74 pp.

33 Anon. 1980. Report on pesticide residue analysis. Official trials. Phosalone on apple. Chuo University. 18 pp.

34 Maestracci, M. 16 Dec 1998. Phosalone. Formulation EXP 60308B (WP). Trials Germany 1997. Residues in apple and in pear. Reverse curve. Study No. 97-767. RPA Report No. R&D/CRLD/AN/msa/ 9816476. Rhone-Poulenc Agro (CRLD). 116 pp.

35 Maestracci, M. 18 Jun 1998. Phosalone. Formulation EXP 60308B (WP). Trial Spain 1997. Residues in pear. Reverse curve. RPA Study No. 97-702. RPA Report No. R&D/CRLD/AN/vt/ 9815818. Rhone-Poulenc Agro (CRLD). 44 pp.

36 Anon. 1989. Report on pesticide residue analysis. Official trials. Phosalone on Japanese pear. Japan Food Analysis Federation. 16 pp.

37 Muller, M.A. 28 Jun 1995. Phosalone et oxo-phosalone (metabolite). Formulation EXP 06207A (SC). Essais France 1994. Residus dans la cerise. Etude No. 94-545. Report R&D/ CRLD/ An/ fb/ 9515875. (in French). Rhone-Poulenc Agro (CRLD). 51 pp.

38 Maestracci, M. 16 Sep 1997. Phosalone. Formulation EXP 06027B (SC). Trials France 1996. Residues in cherry. Decline study. RPA Study No. 96-542. Report No. R&D/CRLD/AN/dbe/ 9716189. Rhone-Poulenc Agro (CRLD). 70 pp.

39 Maestracci, M. 24 Apr 1998. Phosalone. Formulation EXP 05531D (EC). Trial France 1997. Residues in cherry. Decline study. RPA Study No. 97-554. RPA Report No. R&D/CRLD/AN/vt/ 9815381. Rhone-Poulenc Agro (CRLD). 70 pp.

40 Muller, M.A. 28 Nov 1989. Phosalone - Zolone 25 L (EC) Formulation. Tests in Italy, 1989. Residues in peaches. Decline curves. Report No. AG/CRLD/AN/8916828. Rhone-Poulenc Agro (CRLD). 17 pp.

41 Maestracci, M. 1 Apr 1998. Phosalone. Formulation EXP 05531D (EC). Trial France 1997. Residues in peach. Reverse curve. RPA Study No. 97-522. RP Report No. R&D/CRLD/AN/dbe/ 9815342. Rhone-Poulenc Agro (CRLD). 44 pp.

42 Maestracci, M. 21 Aug 1998. Phosalone. Formulation EXP 05531D (EC). Trial France 1997. Residues in peach. RPA Study No. 97-523. RPA Report No. R&D/CRLD/AN/vt/ 9815817. Rhone-Poulenc Agro (CRLD). 45 pp.

43 Maestracci, M. 30 Jul 1998. Phosalone. Formulation EXP 60308 (WP). Trial Italy 1997. Residues in peach. Decline study. RPA Study No. 97-622. RPA Report No. R&D/CRLD/AN/dbe/ 9815848. Rhone-Poulenc Agro (CRLD). 77 pp.

44 Maestracci, M. 11 Sep 1998. Phosalone. Formulation EXP 60308 (WP). Trial Spain 1997. Residues in peach. RP Study No. 97-701. RPA Report No. R&D/CRLD/AN/dbe/ 9815325. Rhone- oulenc Agro (CRLD). 42 pp.

45 Anon. 1982. Report on pesticide residue analysis. Official trials. Phosalone on peach from application of Ranbeck (phosalone 20% + dichlorvos 40%). Japan Food Analysis Federation. 2 pp.

46 Anon. 1982. Report on pesticide residue analysis. Official trials. Phosalone on Japanese apricot from application of Ranbeck (phosalone 20% + dichlorvos 40%). Azabu University. 2 pp.

47 Muller, M.A. 15 Nov 1995. Phosalone et oxo-phosalone (metabolite). Formulation EXP 06027A (SC). Essais France 1994. Residus dans l'amande. Etude No. 94-543. Rapport R&D/CRLD/AN/bd/ 9516513. (In French). Rhone-Poulenc Agro (CRLD). 48 pp.

48 Maestracci, M. 1 Sep 1997. Phosalone. Formulation EXP 05531D (EC). Trial France 1996. Residues in almond. RPA Study No. 96-528. RPA Report No. R&D/CRLD/AN/dbe/ 9716150. Rhone-Poulenc Agro (CRLD). 44 pp.

49 Maestracci, M. 5 Sep 1997. Phosalone. Formulation EXP 05531D (EC). Trial France 1996. Residues in almond. Decline study. RPA Study No. 96-530. Report No. R&D/CRLD/AN/dbe/ 9716168. Rhone-Poulenc Agro (CRLD). 47 pp.

50 Maestracci, M. 28 Aug 1997. Phosalone. Formulation EXP 06027B (SC). Trial France 1996. Residues in almond. RPA Study No. 96-525. Report No. R&D/CRLD/AN/kd/ 9716028. Rhone-Poulenc Agro (CRLD). 44 pp.

51 Maestracci, M. 5 Sep 1997. Phosalone. Formulation EXP 06027B (SC). Trial France 1996. Residues in almond. Decline study. RPA Study No. 96-529. RPA Report No. R&D/CRLD/AN/dbe/ 9716188. Rhone-Poulenc Agro (CRLD). 47 pp.

52 Muller, M.A. 10 Jul 1995. Phosalone et oxo-phosalone (metabolite). Formulation EXP06027A (SC). Essais France 1994. Residus dans la noisette. Etude No. 94-547. Report No. R&D/CRLD/AN/bd/ 9515930. (In French). Rhone-Poulenc Agro (CRLD). 47 pp.

53 Maestracci, M. 1 Sep 1997. Phosalone. Formulation EXP 06027B (SC). Trial France 1996. Residues in hazelnut. Decline study. RPA Study No. 96-551. RPA Report No. R&D/CRLD/AN/dbe/ 9716158. Rhone-Poulenc Agro (CRLD). 48 pp.

54 Maestracci, M. 5 Sep 1997. Phosalone. Formulation EXP 05531D (SC). Trial France 1996. Residues in hazelnut. RPA Study No. 96-550. RPA Report No. R&D/CRLD/AN/dbe/ 9716157. Rhone-Poulenc Agro (CRLD). 44 pp.

55 Maestracci, M. 5 Sep 1997. Phosalone. Formulation EXP 06027B (SC). Trial France 1996. Residues in hazelnut. RPA Study No. 96-549. RPA Report No. R&D/CRLD/AN/dbe/ 9716159. Rhone-Poulenc Agro (CRLD). 43 pp.

56 Maestracci, M. 5 Sep 1997. Phosalone. Formulation EXP 05531D (SC). Trial France 1996. Residues in hazelnut. Decline study RPA Study No. 96-548. RPA Report No. R&D/CRLD/AN/dbe/ 9716229. Rhone-Poulenc Agro (CRLD). 47 pp.

57 Muller, M.A. 10 Jul 1995. Phosalone et oxo-phosalone (metabolite). Formulation EXP06027A (SC). Essais France 1994. Residus dan la noix (amande). Etude No. 94-548. Report No. R&D/CRLD/AN/bd/ 9515931. (In French). Rhone-Poulenc Agro (CRLD). 47 pp.

58 Maestracci, M. 7 Nov 1997. Phosalone. Formulation EXP 05531D (EC). Trial France 1996. Residues in walnut. Decline study. RPA Study No. 96-546. RPA Report No. R&D/CRLD/AN/vt/ 9716585. Report Amendment 1 as R&D/CRLD/AN/ 9817001 (5 January 1999). Rhone-Poulenc Agro (CRLD). 49 pp.

59 Maestracci, M. 7 Nov 1997. Phosalone. Formulation EXP 06027B (SC). Trial France 1996. Residues in walnut. RPA Study No. 96-545. RPA Report No. R&D/CRLD/AN/vt/ 9716568. Rhone- oulenc Agro (CRLD). 44 pp.

60 Maestracci, M. 7 Nov 1997. Phosalone. Formulation EXP 05531D (EC). Trial France 1996. Residues in walnut. RPA Study No. 96-544. RPA Report No. R&D/CRLD/AN/vt/ 9716561. Rhone-Poulenc Agro (CRLD). 44 pp.

61 Maestracci, M. 7 Nov 1997. Phosalone. Formulation EXP 06027B (SC). Trial France 1996. Residues in walnut. Decline study. RPA Study no. 96-547. RPA Report No. R&D/CRLD/AN/dbe/ 9716595. Rhone-Poulenc Agro (CRLD). 45 pp.

62 Maestracci, M. 6 Jan 1999. Phosalone. Formulation EXP 06027B (SC). Residues in apple and processed products. Trial France 1996. RPA Study No. 96-694. Report R&D/CRLD/AN/dbe/ 9816999. Rhone-Poulenc Agro (CRLD). 51 pp.