

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 That Meeting concluded that the existing CXLs for phosalone should be withdrawn, owing to inadequacies in the available information on storage stability and the effects of processing.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 <i>et al.</i> , 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: | 4.57×10^{-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*-¹⁴C]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 |
| | | Leaf homogenate | 389 |
| | | TRR | 900 |
| | Fruit | Rinse | 0.21 |
| | | Peel | 80 |
| | | Pulp | 0.63 |
| | | TRR | 80.84 |
| Application to near mature fruit (Treatment II) | Leaf | Rinse | 453 |
| | | Leaf homogenate | 318 |
| | | TRR | 771 |
| | Fruit | Rinse | 0.16 |
| | | Peel | 58 |
| | | Pulp | 0.87 |
| | | TRR | 59.03 |

Table 2 shows the efficiency of the methanol extraction.

Table 2. Recovery of [¹⁴C]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 [¹⁴C]phosalone.

| Sample | Treatment I | | Treatment II | |
|--|--------------|--------------------|--------------|--------------------|
| | % of TRR | mg/kg as phosalone | % of TRR | mg/kg as phosalone |
| <u>Leaf rinse</u> | 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 |
| <u>Leaf extract</u> | 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 |
| <u>Peel extract</u> | 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 |
| <u>Pulp extract</u> | 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 [¹⁴C]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. [¹⁴C]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.

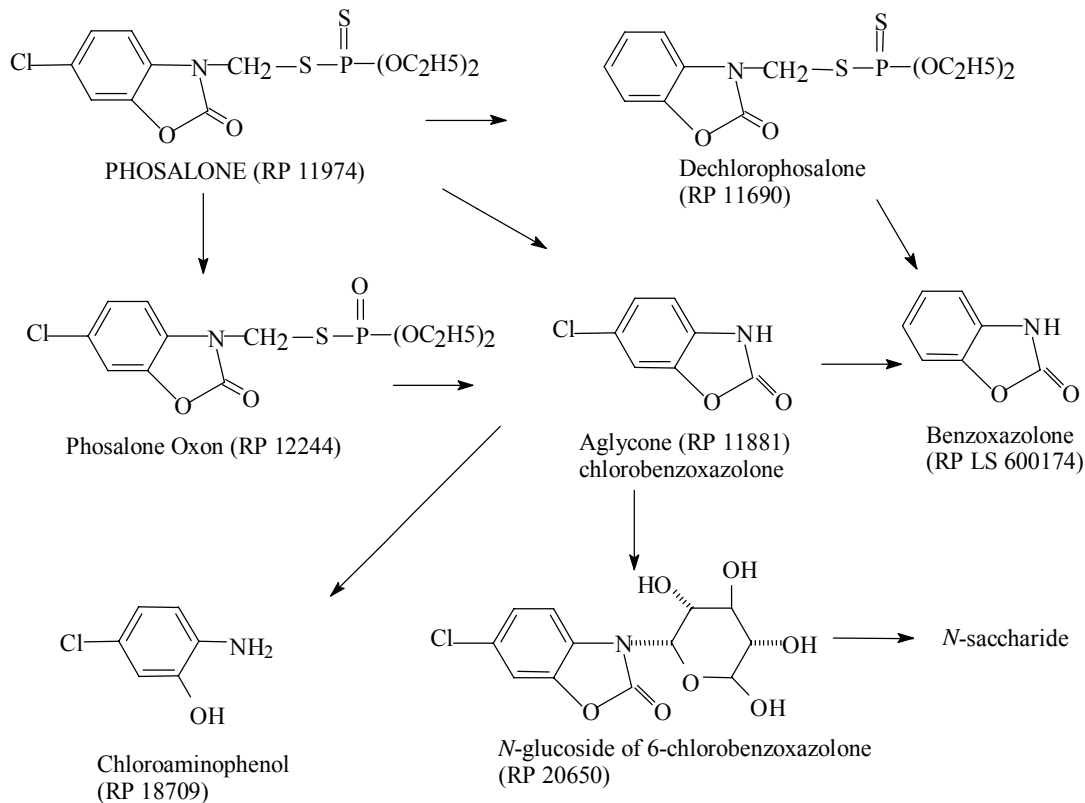
Table 5. Distribution of [¹⁴C]phosalone and its metabolites in grapes.

| 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 |
| <i>N</i> -glucoside of chlorobenzoxazolone | | | 0.057 | 0.21 | 0.036 | 0.13 | 0.093 | 0.33 |
| <i>N</i> -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 |
| <i>N</i> -glucoside of chlorobenzoxazolone | | | 0.049 | 0.18 | 0.024 | 0.09 | 0.073 | 0.27 |
| <i>N</i> -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 |

¹As 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-methylsulfinylmethylbenzoxazolone, 6-chloro-3-methylthiomethylbenzoxazolone, 6-chloro-3-methylsulfonylmethylbenzoxazolone, 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 [¹⁴C]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 (1.409×10^{-5} mol/l). The Δn and N_a values were calculated to be 2.751×10^{-9} mol/sec and 2.08×10^{-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 N_a were calculated to be 2.273×10^{-9} mol/sec and 9.185×10^{-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 Jendrzeczac (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 $9.34 \times 10^{-3} \text{ s}^{-1}$ which corresponds to a half-life of about 74 daylight seconds.

Biodegradability

Mutzall 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 $^{14}\text{CO}_2$ 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 $\mu\text{g/g}$ soil (equivalent to 1 kg ai/ha) and samples were maintained in the dark at $20 \pm 2^\circ\text{C}$ at 40% of maximum water holding capacity for up to 45 days. The overall recovery of ^{14}C was $99.8 \pm 2.9\%$. Phosalone was degraded rapidly with a half-life of 2.9 days. Production of $^{14}\text{CO}_2$ 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 $\leq 0.7\%$ 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 $\leq 2\%$ 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 $20 \pm 2^\circ\text{C}$ 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 $\leq 2.0\%$. CO₂ 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).

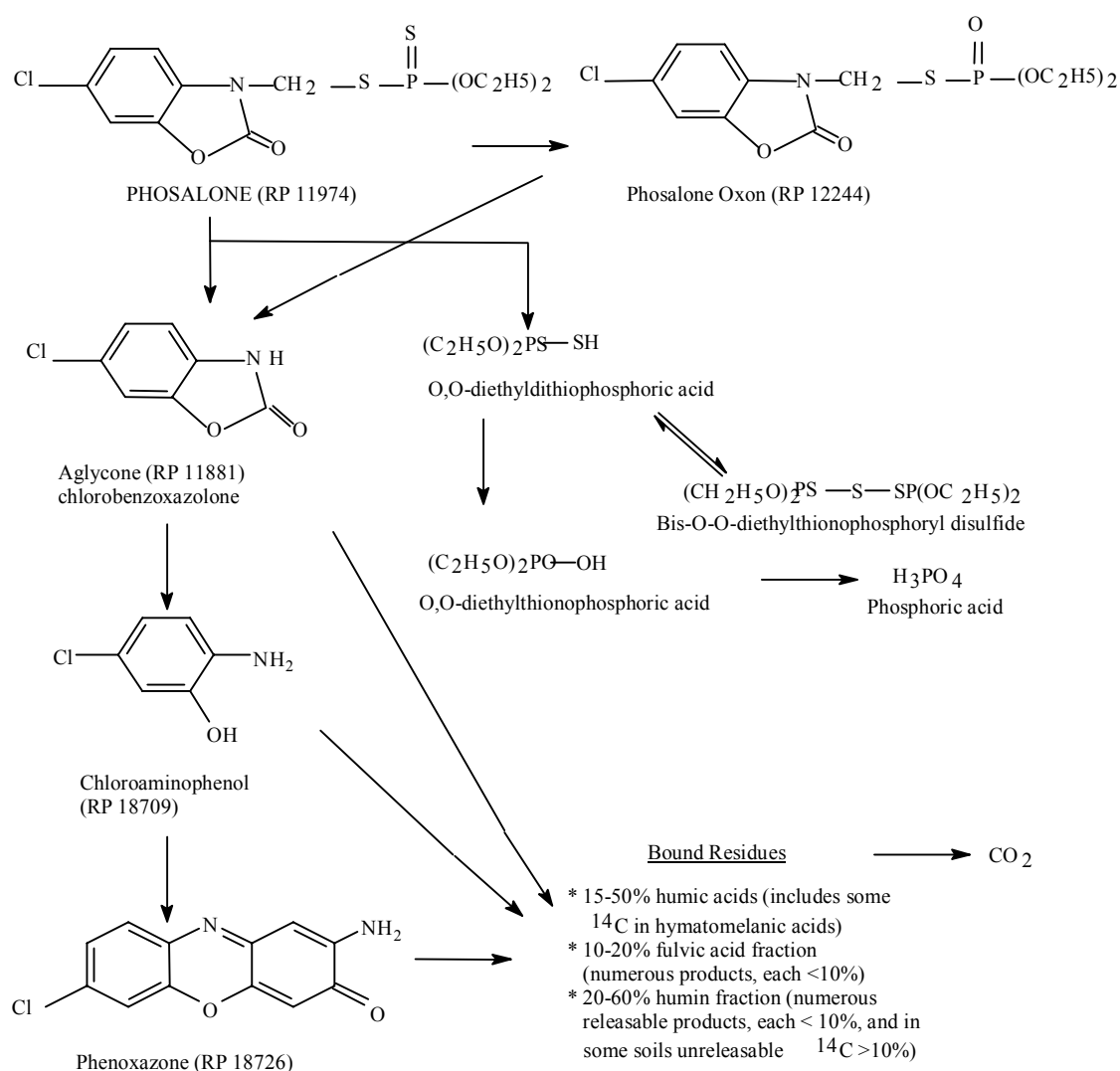
Table 7. Distribution of radioactivity in soil after 45 days.

| Soil | % of applied dose | | | | | Total recovery |
|------------|-------------------|-----------------|-------------|-------------------------------|-----------------|----------------|
| | Phosalone | Other extracted | Unextracted | ¹⁴ CO ₂ | Other volatiles | |
| 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 ¹⁴C.

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-¹⁴C]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 [¹⁴C]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 ¹⁴C 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-3H-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 $99.5 \pm 3.9\%$. 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. [¹⁴C]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 ¹⁴C 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 ¹⁴CO₂ 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 ¹⁴C 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 [¹⁴C]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. [¹⁴C]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 (K₁) 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 (K₂) 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 | 83 | 9 |
| | 0.25 | 3 | 80, 89, 104 | | |
| | 1 | 3 | 73, 75, 76 | | |
| Barley | 0.05 | 3 | 102, 109, 117 | 99 | 9 |
| | 0.25 | 3 | 99, 100, 100 | | |
| | 1 | 3 | 88, 89, 91 | | |
| Apple | 0.05 | 3 | 86, 88, 94 | 98 | 9 |
| | 0.5 | 3 | 102, 103, 108 | | |
| | 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 | | |
| 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 USDA (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 | 4 | 102,107,111 | 98 | 15 |
| | Sevilla | 105,103,101 | | | 85,110,92 | | |
| | Bologna | 101,100,101 | | | 110,113,111 | | |
| | Chazay | 98,103,100 | | | 70,83,84 | | |
| 0.2 | Manningtree | 110,115,111 | 110 | 2 | 70,83,100 | 85 | 15 |
| | Sevilla | 111,111,111 | | | 89,81,89 | | |
| | Bologna | 108,113,111 | | | 94,104,103 | | |
| | Chazay | 111,104,110 | | | 71,70,70 | | |
| 2 | Manningtree | 107,106,108 | 105 | 3 | 93,77,71 | 80 | 9 |
| | Sevilla | 102,102,102 | | | 79,79,84 | | |
| | 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 ($\geq 88\%$) 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 | <i>Apples</i> . Italy, France, Japan, Germany |
| Table 18 | <i>Pears</i> . Spain, Japan |
| Table 19 | <i>Cherries</i> . France |
| Table 20 | <i>Peaches</i> . Italy, France, Spain, Japan |
| Table 21 | <i>Japanese Apricots</i> . Japan |
| Table 22 | <i>Almonds</i> . France |
| Table 23 | <i>Hazelnut</i> . France |
| Table 24 | <i>Walnuts</i> . 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/ha | | | Mean | Individual | |
| Italy, 1989 | EC 232 g/l | 4 | 0.6 | 1000 | 0.06 | Rome Beauty | 0 | 1.6 | | Ref. 25 AG/CRLD/AN/891682 7 |
| | | | | | | | 7 | 1.0 | | |
| | | | | | | | 14 | <u>1.1</u> ¹ | | |
| | | | | | | | 21 | 0.5 | | |
| | | | | | | | 28 | 0.4 | | |
| 35 | 0.6 ² | | | | | | | | | |
| Italy, 1989 | EC 24% | 5 | 0.6 | 1000 | 0.06 | Rome Beauty | 21 | <u>0.65</u> ² | | Ref. 26 AG/CRLD/AN/901590 7 |
| | | | | | | | 5 | <u>0.85</u> ² | | |
| | | | | | | | 9 | <u>0.96</u> ² | | |
| France, 1995 Saran | SC 500 g/l | 1 | 0.88 | 563 | 0.16 | Golden | 2 h | 0.69,0.72 | | Ref. 27 R&D/CRLD/AN/kd/96 16621 |
| | | | | | | | 3 | 0.67,1.3 | | |
| | | | | | | | 7 | 0.54,1.4 | | |
| | | | | | | | 14 | 0.78,1.1 | | |
| Souastre | | 1 | 0.88 | 560 | 0.16 | Idared | 2 h | 1.1,1.4 | | |
| | | | | | | | 3 | 1.2,2.6 | | |
| | | | | | | | 7 | 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 <u>0.38,0.17</u> | |
| France, (South) 1996 Beaucaire | EC 350 g/l | 3 | 0.61 | 983 | 0.06 | Golden | 16 22 31 45 | 0.70 0.68 <u>0.74</u> ^{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 | <u>0.66</u> 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 <u>1.5</u> ^{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 <u>0.91</u> 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 60 | 0.42 0.20 | | Ref. 33 |
| | | 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 | 0.73 | | |
| | | | | | | | 15 | 0.43 | | |
| | | | | | | | 21 | 0.45 | | |
| | | | | | | | 28 | <u>0.46</u> ¹ | | |
| | | | | | | | 35 | 0.35 | | |
| | | | | | | | 42 | 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

Pears. Phosalone was applied by experimental backpack sprayers in Spain and Germany and by pressure sprayers in Japan. The plot sizes were 16 m² (4 trees) in Japan, 25.6 m² (4 trees) in Spain and 70 m² (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 | 0.93 | Ref. 35 R&D/CRLD/AN/vt/9815818 |
| | | | | | | | 15 | <u>0.38</u> ¹ | |
| | | | | | | | 21 | <u>0.36</u> ² | |
| | | | | | | | 30 | 0.23 | |
| | | | | | | | 45 | 0.13 | |
| Japan, 1989 | EC 35% | 2 | 1.75 | 5000 | 0.035 | Kousui | 45 | <u>0.11</u> ³ | Ref. 36 |
| | | | | | | | 60 | 0.033 | |
| | | 2 | 2.19 | 6250 | 0.035 | Cyojuro | 45 | <u>0.29</u> ³ | |
| | | | | | | | 60 | 0.099 | |

¹ According to French GAP

² According to Italian GAP

³ According to Japanese GAP

Cherries. 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 m² to 81 m² 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 | <u>1.6, 1.3</u> | |
| | | 3 | 1.1 | 505 | 0.22 | fruit with stone (Van) | 14 | 1.8, 2.5 | |
| St. Simplicie | | 3 | 0.87 | 993 | 0.088 | fruit with stone (Sumbours) | 11 | 1.5, 1.8 | |
| | | 3 | 1.4 | 1345 | 0.1 | fruit with stone (Sumbours) | 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 | <u>0.59, 0.23</u> <u>0.23, 0.17</u> | |
| | | 2 | 0.6 | 300 | 0.2 | fruit with stone (Sunburst) | 11 16 21 | 0.61, 0.78 <u>0.46, 0.72</u> 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 <u>0.47, 0.32</u> | |
| | | 2 | 0.59 | 801 | 0.073 | fruit with stone (Burlat) | 8 14 18 | 0.53, 0.6 <u>0.45, 0.31</u> 0.13, <u>0.33</u> | |
| (North), 1996 Olivet | | 1 | 0.58 | 653 | 0.089 | fruit with stone (Duroi 3) | 9 15 23 | 0.58, 0.69 <u>0.26, 0.26</u> 0.08, 0.15 | |
| | | 2 | 0.58 | 650 | 0.089 | fruit with stone (Duroi 3) | 9 15 23 | 0.60, 0.63 <u>0.23, 0.22</u> 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 <u>0.18, 0.16</u> 0.15, 0.08 | |
| | | 2 | 0.60 | 416 | 0.14 | fruit with stone (Burlat) | 10 17 21 | 0.45, 0.6 <u>0.15, 0.26</u> 0.12, 0.2 | |
| (South),1996 | | 1 | 0.60 | 667 | 0.09 | fruit with stone (Duroi 2) | 15 | <u>0.22, 0.28</u> | |
| Blcuvac | | | | | | | 20 26 | 0.14, <u>0.3</u> 0.10, 0.14 | |
| | | 2 | 0.60 | 667 | 0.09 | fruit with stone (Duroi 2) | 15 20 26 | <u>0.27, 0.42</u> 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, <u>0.6</u> <u>0.53, 0.22</u> | |
| | | 2 | 0.60 | 400 | 0.15 | fruit with stone (Stark) | 11 14 20 | 0.62, 0.56 <u>0.37, 0.46</u> 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 <u>0.36, 0.18</u> 0.11, 0.12 | Ref. 39 R&D/CRLD/AN /vt/9815381 |
| | | 2 | 0.58 | 965 | 0.06 | fruit (Lapins) | 10 15 21 | 0.36, 0.32 <u>0.29, 0.2</u> 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 m² with 2 to 6 trees in France, 100 m² 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 | 0.4 | | Ref. 40 AG/CRLD/AN/ 8916828 | |
| | | | | | | | 7 | 0.3 | | | |
| | | | | | | | 14 | <u>0.16¹</u> | | | |
| | | | | | | | 21 | <u>0.13²</u> | | | |
| | | | | | | | 28 | 0.13 | | | |
| France, 1997 (South) | EC 350 g/l | 3 | 0.74 | 563 | 0.13 | Fruit without stone (Alexandra) | 2 h | | 1.5, 1.6 | Ref. 41 R&D/CRLD/AN /dbe/9815342 | |
| | | | | | | | 14 | | 0.80, 0.52 | | |
| | | | | | | | 21 | | 0.57, 0.61 | | |
| | | | | | | | 28 | | 0.53, 0.76 | | |
| | | | | | | | 35 | | 0.54, 0.49 | | |
| | | | | | | Fruit (whole) | 2h | | 1.4, 1.5 | calculation | |
| | | | | | | | 14 | | <u>0.73¹</u> , 0.48 | | |
| | | | | | | | 21 | | 0.53, 0.57 | | |
| | | | | | | | 28 | | 0.48, <u>0.68²</u> | | |
| | | | | | | | 35 | | 0.5, 0.45 | | |
| France, 1997 (South) Ste Bazeille | EC 350 g/l | 3 | 0.61 | 288 | 0.21 | Fruit without stone (Royal glory) | 26 | | 0.44, 0.29 | Ref. 42 R&D/CRLD/AN /vt/9815817 calculation | |
| | | | | | | | 26 | | 0.37, 0.28 | | |
| Italy, 1997 | WP 291 g/kg | 3 | 0.88 | 1500 | 0.06 | Fruit (whole) (Duchessa d'Este) | 0 | 3.7 | calculation | Ref. 43 R&D/CRLD/AN /dbe/9815848 | |
| | | | | | | | 14 | <u>1.5¹</u> | | | |
| | | | | | | | 21 | <u>1.4²</u> | | | |
| | | | | | | | 28 | 0.71 | | | |
| | | | | | | | 35 | 0.45 | | | |
| Spain, 1997 | WP 300 g/kg | 3 | 0.9 | 800 | 0.11 | Fruit without stone (Red Top) | 28 | 0.54 | calculation | Ref. 44 R&D/CRLD/AN /dbe/9815325 | |
| | | | | | | | 28 | 0.51 | | | |
| Japan, 1982 Fukushima | EC 20% | 2 | 0.8 | 4000 | 0.02 | Fruit without stone & peeled (Ohkubo) | 15 | 0.09 | | Ref. 45 | |
| | | | | | | | 30 | 0.02 | | | |
| | | | | | | | 45 | <0.01 | | | |
| | | | | | | | Peel | 15 | | | 8.92 |
| | | | | | | | | 30 | | | 3.93 |
| 45 | 1.20 | | | | | | | | | | |
| | | | | | | Fruit without stone | 15 | 2.3 | calculation | | |
| | | | | | | | 30 | 0.88 | | | |
| Ishikawa | | | | | | Fruit without stone & peeled (Ohkubo) | 15 | 0.04 | | | |
| | | | | | | | 30 | 0.02 | | | |
| | | | | | | | 45 | <0.01 | | | |
| | | | | | | | Peel | 15 | | | 5.43 |
| | | | | | | | | 30 | | | 3.62 |
| 45 | 1.02 | | | | | | | | | | |
| | | | | | | Fruit without stone | 15 | 1.33 | calculation | | |
| | | | | | | | 30 | 0.63 | | | |
| | | | | | | | 45 | 0.17 | | | |

| 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 | 0.72 | | Ref. 63 R&D/CRLD/AN /mr/9915513 |
| | | | | | | | 15 | 0.50 | | |
| | | | | | | | 21 | 0.27 | | |
| | | | | | | | 30 | 0.35 | | |
| | | | | | | | 45 | 0.49 | | |
| | | | | | Fruit (whole) | 0 | 0.65 | calculation | | |
| | | | | | | 15 | --- | | | |
| | | | | | | 21 | 0.25 | | | |
| | | | | | | 30 | 0.32 | | | |
| | | | | | | 45 | 0.45 | | | |
| France, 1998 (South) Charly | EC 350 g/l | 3 | 0.76 | 351 | 0.22 | Fruit without stone (Redwing) | 26 | | 0.24,0.68 | Ref. 64 R&D/CRLD/AN /mr/9915763 calculation |
| | | | | | | Fruit (whole) | 26 | | | |
| France, 1998 (South) | EC 350 g/l | 3 | 0.76 | 571 | 0.13 | Fruit without stone (Manon) | 2 h | | 0.81,0.54 0.30,0.35 0.13,0.37 | Ref. 65 R&D/CRLD/AN /mr/9915759 |
| | | | | | | | 10 | | | |
| | | | | | | | 17 | | | |
| | | | | | | | 24 | 0.33 | | |
| | | | | | | | 31 | 0.12 | | |
| | | | | | Fruit (whole) | 2 h | | 0.78, 0.52 | calculation | |
| | | | | | | 10 | | | | |
| | | | | | | 10 | | 0.29, 0.33 <u>0.12</u> , <u>0.35</u> ¹ | | |
| | | | | | | 17 | | | | |
| | | | | | | 24 | 0.31 | | | |
| | | | | | | 31 | 0.11 | | | |

¹ According to French GAP

² According to Italian GAP

Apricots. 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 | 0.046 | Ref. 46 |
| | | | | | | | 32 | 0.044 | |
| | | | | | | | 47 | <u>0.009</u> | |
| | | 2 | 0.8 | 6000 | 0.02 | Fruit without stone (Seigyoku) | 21 | 0.724 | |
| | | | | | | | 30 | 0.698 | |
| | | | | | | | 45 | <u>0.005</u> | |

Almonds. Trees were treated with phosalone by experimental atomizer with spray boom and backpack sprayers in supervised trials in France. Plots sizes ranged from 105 m² to 140 m² 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/b d/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</u> , <u><0.05</u> | Ref. 48 R&D/CRLD/AN/d be/9716150 |
| 1996 St.Gilles | EC | 3 | 0.77 | 215 | 0.36 | shell & kernel (Ferragnes) | 2 h 29 42 78 | 4.1, 4.6 2.6, 3.2 3.2, 2.0 <u><0.05</u> , <u><0.05</u> | Ref. 49 R&D/CRLD/AN/d be/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/k d/9716028 |
| 1996 St.Gilles | SC 500h/l | 3 | 0.75 | 215 | 0.35 | nut (Ferragnes) | 2 h 29 42 78 | 4.7, 4.5 4.1, 4.4 3.4, 4.4 <u><0.05</u> , <u>0.074</u> | Ref. 51 R&D/CRLD/AN/d be/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 | |

Hazelnuts. Trees were sprayed with phosalone by experimental atomizer with spray boom and backpack sprayers in supervised trials in France. Plots sizes were 45 m² to 108 m² 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/A N/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) | 2 h | 7.2,6.0 | Ref. 53 R&D/CRLD/A N/dbe/9716158 |
| | | | | | | kernel without shell | 7 | 0.061, 0.064 | |
| | | | | | | | 14 | <0.05(2) | |
| | | | | | | | 21 | <0.05(2) | |
| 1996 Puechoursi | EC 350g/l | 2 | 0.61 | 556 | 0.11 | kernel without shell (Fertile de Coutard) | 20 | <0.05(2) | Ref. 54 R&D/CRLD/A N/dbe/9716157 |
| 1996 Villesequelande | SC 500g/l | 2 | 0.60 | 667 | 0.09 | kernel without shell (Merveille de Bollwiller) | 22 | <0.05(2) | Ref. 55 R&D/CRLD/A N/dbe/9716159 |
| 1996 Guitinieres | EC | 2 | 0.61 | 800 | 0.08 | nuts (Ennis) | 2 h | 2.3,2.7 | Ref. 56 R&D/CRLD/A N/dbe/9716229 |
| | | | | | | | 7 | 0.82,0.76 | |
| | | | | | | | 13 | 0.63,0.70 | |
| | | | | | | kernel without shell | 21 | <0.05 (2) | |

Table 23b Residues of phosalone oxon in hazelnuts.

| Country | 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/95 15930 |
| | | | 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 | |

Walnuts. Trees were sprayed with phosalone by experimental atomizer with spray boom and backpack sprayers in supervised trials in France. Plots sizes were 49 m² to 400 m² 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 Ref. 58 R&D/CRLD/AN/981 7001 Ref. 58 R&D/CRLD/AN/981 7001 Ref. 58 R&D/CRLD/AN/vt/9 716585 | |
| | | | | | | whole walnut (nut, shell & hull) | 7 | | | 1.3,1.8 |
| | | | | | | whole walnut (nut, shell & hull) | 14 | | | 0.81,0.89 |
| | | | | | | kernel | 21 | | | <0.05, 0.055 |
| 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 | | | <0.05(2) |
| | | | | | | | 28 | | | <0.05(2) |

RESIDUES IN STORAGE AND PROCESSING

In storage

No information.

In processing

Apples. 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 troposphere the estimated reaction constant at 298° K is $9.34 \times 10^{-3} \text{ s}^{-1}$ which corresponds to a half-life of about 74 daylight seconds.

Biodegradability. In active sludge about 20% of the initial radioactivity of [¹⁴C]phosalone was detected as ¹⁴CO₂ 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 $0.18 (\pm 0.02) \text{ mg/kg fish/mg/l water/day}$. 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 |

¹A 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.

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