PROFENOFOS (171)

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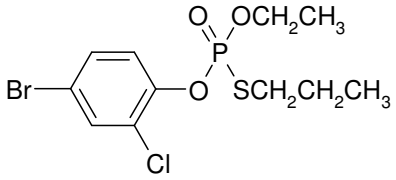
EXPLANATION

Profenofos, an organophosphorus insecticide, was listed in the Periodic Re-Evaluation Program at the 39th Session of the CCPR for residue evaluation by the 2008 JMPR. Residue and analytical aspects of profenofos were evaluated by the JMPR in 1990, 1992, 1994 and 1995. The toxicological review was conducted in 2007, when an ADI of 0–0.03 mg/kg bw and an ARfD of 1 mg/kg bw were established.

The Meeting received information on animal and plant metabolism, environmental fate, analytical method, GAP, storage stability, processing study and residue trial data on cabbage, cotton, watermelon, chili pepper, tomato and soybean.

GAP information on cotton was submitted by Australia. GAP information and residue trial result on mango and mangosteen were also provided by Thailand.

IDENTITY

ISO common name:	Profenofos
Manufacturer's code Number:	CGA15324, OMS2004
Other code numbers:	CAS No: 41198-08-7
	CIPAC No: 461
Chemical name:	IUPAC: *O*-4-bromo-2-chlorophenyl *O*-ethyl *S*-propyl phosphorothioate
	CA: *O*-(4-bromo-2-chlorophenyl) *O*-ethyl *S*-propyl phosphorothioate
Formulae:	$C_{11}H_{15}BrClO_3PS$
Molecular mass	373.6 g/mol
Structural formula:	

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Property	Results	Ref
Purity	Minimum 99.0%	
Appearance	Clear liquid	Das, 1999
Odour	Weak odour, like cooked onion	Das, 1999
Colour	Colourless	Das, 1999
Vapour pressure	1.24×10^{-4} Pa at 25 °C (extrapolated)	Rordorf 1988, OECD 104
Melting/freezing point	-76 °C (Freezing)	Geoffroy 1999, EEC A.1
Relative density	1.46 at 20°C	Das, 1999, EEC A.3
Octanol-water partition coefficient	Log Kow = 4.44 at 25 °C	Laster, 1991, OECD 107
Solubility in water at 22°C	28 mg/L at pH 6.9	Jäkel, 1987, OECD 105

Property	Results	Ref
Dissociation constant	No dissociation constant in accessible pH range (0.6 to 12.0)	Hörmann, 1999, OECD 112

Technical material

Property	Results	Ref
Purity	Minimum 91.4%	
Appearance	Clear liquid	Das, 1999
Odour	Weak odour, like cooked onion	Das, 1999
Colour	Light brown	Das, 1999
Solubility in organic solvents at 25 °C	n-Hexane: completely miscible n-Octanol: completely miscible Toluene: completely miscible Ethanol: completely miscible Dichloromethane: completely miscible Ethyl acetate: completely miscible Acetone: completely miscible Methanol: completely miscible	Kettner, 1999 Rodler, 1991
Stability:	No thermal effect found between room temperature and 150 °C	Angly, 1999, OECD 113

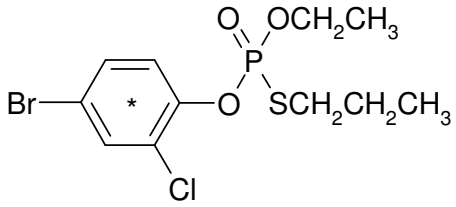
FORMULATION

Emulsifiable concentrate (EC) in various concentrations alone or in combination with cypermethrin. For profenofos, pesticide specifications were established for TC, EC and UL formulations through the Joint FAO/WHO Meeting on Pesticide Specifications (JMPS), and published as FAO Specifications and Evaluations for Agricultural Pesticides compounds in 1998.

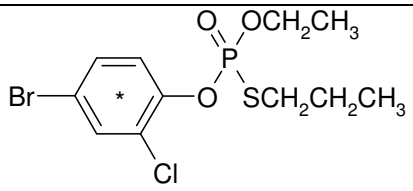
(http://www.fao.org/ag/agp/agpp/Pesticid/Default.htm)

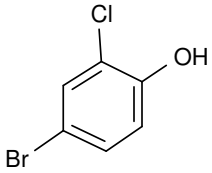
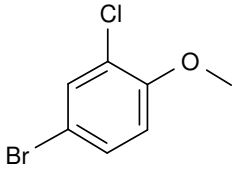
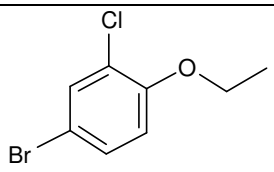
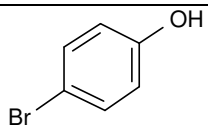
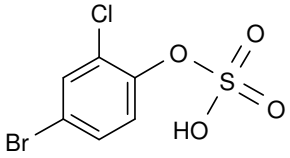
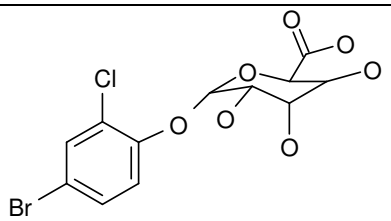
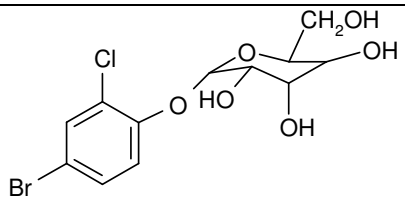
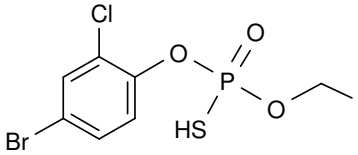
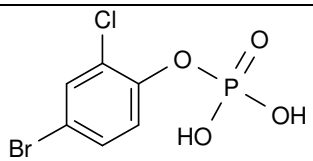
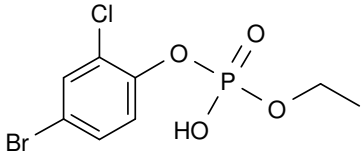
METABOLISM AND ENVIRONMENTAL FATE

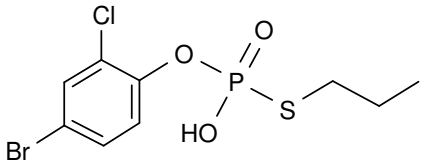
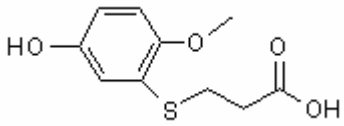
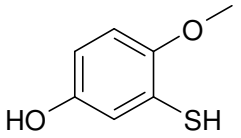
The metabolism of profenofos in animals and plants was investigated using [phenyl-UL-^{14}C]-profenofos.



The following table shows the structures, codes and names of profenofos and related metabolites referred to within this document.

Name/Code number	Chemical structure	Chemical name
Profenofos CGA 15324		*O*-4-bromo-2-chlorophenyl *O*-ethyl S-propyl phosphorothioate (IUPAC) *O*-(4-bromo-2-chlorophenyl) *O*-ethyl S-propyl phosphorothioate (CAS)

Name/Code number	Chemical structure	Chemical name
CGA 55960 C 13565		4-bromo-2-chlorophenol (IUPAC)
BCPME CGA236844		4-bromo-2-chlorophenylmethylether
BCPEE		4-bromo-2-chlorophenylethylether
CGA 19941		4-bromophenol
CGA55960 sulfate		Sulfuric acid mono-(4-bromo-2-chloro-phenyl) ester
CGA55960		glucuronide of 4-bromo-2-chloro-phenol (in rat metabolism report this metabolite is referred to as CGA55163)
CGA55162		Glucoside of 4-bromo-2-chloro-phenol
CGA 65867		Thiophosphoric acid O-(4-bromo-2-chloro-phenyl) ester O'-ethyl ester
CGA 47195		O-(4-bromo-2-chloro-phenyl) monophosphate
CGA 47196		O-ethyl-O-(2-chloro-4-bromo-phenyl)-phosphate

Name/Code number	Chemical structure	Chemical name
CGA 47197		O-(2-chloro-4-bromo-phenyl)-S-n-propyl-thiophosphate
THPME		2-thioethylenecarboxy-4-hydroxyphenyl methyl ether
MHPME		2-mercapto-4-hydroxyphenylmethylether

Animal metabolism

The Meeting received information on the fate of orally dosed profenofos in lactating goats and in laying hens. Metabolism in laboratory animals (rats) was summarized and evaluated by the WHO panel of the JMPR in 2007.

Lactating goat

Study 1

In a study by Cassidy and Thomas (1976; CGA15324/0899), a goat was administered once a day for 9 consecutive days with [phenyl-^{14}C]-profenofos in gelatin capsules at a level of 5 ppm in feed. The total recovery of radioactivity was 97.8% with 85.0% of it being excreted in the urine and 4.4% in the faeces. The metabolites were not identified. .

Study 2

In a study by Ritter (1994; CGA15324/1397), [phenyl-^{14}C]-profenofos was administered orally in gelatin capsules to two lactating goats once a day for 4 consecutive days. The test material of 150 mg was dosed at levels equivalent to 100 ppm in the feed.

During the dosing period, urine and faeces samples were collected once a day in the morning and milk samples were collected twice a day in the morning and afternoon. A blood sample was taken from each goat just prior to sacrifice. Both goats were sacrificed approximately 6 hours after the final dose. Leg muscle, omental fat, perirenal fat, kidneys, liver, bile and tenderloin were taken for analysis.

Radioactivity in milk and urine was analysed directly by liquid scintillation counting (LSC). Faeces and tissue were homogenized and analysed by combustion radioanalysis and LSC. Samples were initially extracted with acetonitrile, and unextracted liver and kidney residues incubated with protease and then extracted with buffer and acetonitrile. Final extracts were analysed using HPLC and TLC. Confirmation of the identity of the metabolites was obtained by mass spectrometry.

The total material balance averaged 71.6%. Most of the dose was excreted in the urine, where an average of 69.4% of the dosed radioactivity was recovered. The faeces contained 1.46% dose and the milk 0.12% dose. The radioactivity balance is shown in Table 1.

Table 1 Recovery of Radioactivity from Goats after Oral Administration of [^{14}C]-Profenofos (% of Total Administered Radioactivity)

	Goat 1 (% TAR)	Goat 2 (%TAR)	Average (%TAR)
Urine	59.26	79.44	69.35
Faeces	1.70	1.22	1.46
Milk am total	0.07	0.08	0.04
Milk pm total	0.04	0.03	0.08
Muscle	0.12	0.21	0.17
Liver	0.05	0.06	0.06
Kidney	0.04	0.04	0.04
Fat	0.01	0.02	0.02
Blood	0.28	0.52	0.40
Total Recovery	61.57	81.62	71.60

TAR – total administered radioactivity

The total radioactive residues (TRR), expressed as mg/kg equivalents of profenofos, measured in the various tissues are presented in Table 2.

Table 2 Total radioactive residues in goat tissues at sacrifice following oral administration of [^{14}C]-profenofos

	mg/kg profenofos equivalents	
	Goat 1	Goat 2
Muscle	0.046	0.073
Liver	0.422	0.514
Kidney	2.500	2.316
Fat	0.053	0.069
Blood	0.586	0.986

The total radioactive residue in the collected milk is presented in Table 3.

Table 3 Total Radioactive Residues in Goat Milk at following Oral Administration of [^{14}C]-Profenofos

	mg/kg profenofos equivalents					
	Goat 1			Goat 2		
	PM milk	AM milk	Mean	PM milk	AM milk	Mean
Day 1	0.315	0.140	0.228	0.306	0.088	0.197
Day 2	0.376	0.170	0.273	0.401	0.072	0.237
Day 3	0.435	0.210	0.323	0.369	0.097	0.233
Day 4	0.310	NS	-	0.405	NS	-

NS = No Sample Taken

Milk taken on day 4 and tissues and fat samples were extracted with acetonitrile. Greater than 90% of the TRR in milk and fat went into acetonitrile phase. Slightly less than 90% (88–89%) was extracted from muscle; however, the unextracted residue represented less than 0.01 mg/kg, and was not characterized further.

The post-extraction solids from liver and kidney were extracted with aqueous buffer and acetonitrile after treatment of protease. Most of the residues in the unextracted liver solids were extracted by an aqueous buffer and 4% of the TRR were extracted into acetonitrile.

Metabolites in tissues and milk were identified by comparison in at least two chromatographic systems with radiolabelled metabolites obtained from the goat urine and from the urine of rats dosed with [^{14}C]-profenofos during a rat metabolism study (Report No.ABR-92026).

The major metabolites identified in tissues, milk and urine is summarized in Table 4.

Table 4 Summary of metabolite identification in tissues, milk and urine

	Milk day 4	Liver	Kidney	Muscle	Fat	Urine day 4
	Goat 2	Goat 2	Goat 2	Goat 2	Goat 2	Goat 2
TRR (mg/kg)	0.405	0.514	2.316	0.073	0.069	-
Metabolite fractions as a percentage of TRR (mg/kg profenofos equivalents)						
Profenofos	-	10 (0.049)	-		44 (0.03)	-
CGA55960 glucuronide	-	8 (0.041)	28 (0.65)	2 (0.002)	-	15 (36)
CGA55960 Sulfate	85 (0.345)	-	40 (0.93)	56 (0.041)	11 (0.008)	74 (175)
CGA55960	12 (0.047)	25 (0.04)	22 (0.52)	4 (0.003)	29 (0.02)	1 (1)
CGA47196	-	3 (0.013)	-	2 (0.001)	-	-
Unknown	7 (0.027)	8 (0.038)	4 (0.1)	21 (0.015)	5 (0.003)	8 (19)
Total (%)	104	54 [a]	94	85	89	98

[a] 90% TRR taking into account evaporation during concentration at neutral pH and degradation at pH 12.

Metabolism of profenofos in the goat proceeded mainly via hydrolysis of the phosphate group to form 4-bromo-2-chlorophenol (CGA55960) and its sulphate and glucuronic acid conjugates. Unmetabolized profenofos was the major compound in fat, and was also present in liver. CGA55960 sulphate and CGA55960 glucuronide were the major metabolites in urine, kidney and milk.

The metabolism of profenofos in the lactating goat follows the same major pathways as in the rat described in the toxicology section of the 2007 Report of the JMPR (see page 210).

Laying hen

Study 1

In a study by Ballantine, Marco and Oakes (1986; CGA15324/0900), [phenyl-^{14}C]-profenofos were administered orally as capsules containing ground corns to two white leghorn hens for 14 consecutive days. The hens were dosed at a rate of 5.0 ppm based on an average daily feed consumption. A majority of the radioactivity was eliminated in the excreta accounted for 81–85% of the total dose. The tissues and blood only accounted for 0.02% and 0.01% of the dose, respectively. The study does not include any significant identification of the residues.

Study 2

In a study by Ritter (1994; CGA15324/1398), [phenyl-^{14}C]-profenofos was administered orally in gelatin capsules to two groups of five laying hens once a day for 8 consecutive days. One group was dosed with level equivalent to 1 ppm profenofos in the feed; the other group was dosed with level equivalent to 10 ppm in the feed.

During the dosing period, egg and excreta samples were collected once a day. A blood sample was drawn just prior to sacrifice. All the hens were sacrificed 6 to 8 h after the last dose. The liver, kidneys, peritoneal fat, skin with attached fat, bile, and lean meat (breast and leg muscle) were collected during necropsy.

A composite sample for each tissue, egg white (day 7) and egg yolk (day 7) was generated by pooling samples from all the hens within a dose group. Samples were extracted by acetonitrile/methanol, and extract solutions were analysed by TLC. After extraction with organic solvents, bound residues in the liver and 1 ppm dosed egg yolks were released by incubation with protease or lipase and further acetonitrile extraction. Solutions were further purified by column chromatography or TLC and analysed by GC/MS.

The total average recovery of the administered radioactivity at the 1 mg/kg dose level was 94.6%. The excreta accounted for 93.3% of the dose, with an average of 1% in the tissues and 0.3% in the eggs.

The total recovery at the 10 mg/kg dose level averaged 89.4%, with 88.5% in the excreta, 0.7% in the tissues, and 0.2% in the eggs. Individual tissue residues were approximately 10-fold higher than those from the 1 mg/kg dose group.

Bile was not radioanalysed as it was unobtainable from 2 hens and available amounts were too small for normal radioanalysis procedures from the other hens. The distribution of radioactivity and total residue of two dose group are presented in Tables 5 and 6.

Table 5 Summary of distribution of radioactivity in hen tissues, egg and excreta at 1 mg/kg Dose

	Fat	Muscle	Liver	Kidney[a]	Blood[a]	Skin/ Fat[1]	Egg yolk day 7	Egg white[a] Day 7	Excreta[a]
TRR (mg/kg)	0.005	0.003	0.023	0.116	0.050	0.014	0.031	0.002	
% of dose	0.01	0.12	0.13	0.13	0.49	0.07	0.05	0.01	93.3
Acetonitrile/ Methanol	90%	94%	88%				61%		
Non extracted (PES)	10%	5%	12%				39%		
Characterisation of PES									
Protease treatment									
Aqueous buffer			10%				<1%		
Acetonitrile			4%				2%		
Lipase treatment									
Aqueous buffer							14%		
Acetonitrile							18%		

[a] no metabolite characterisation carried out.

Table 6 Summary of distribution of radioactivity in hen tissues, egg and excreta at 10 mg/kg Dose

	Fat	Muscle	Liver	Kidney [a]	Blood [a]	Skin/ Fat [a]	Egg yolk day 7	Egg white day 7	Excreta
TRR (mg/kg)	0.044	0.030	0.280	1.321	0.396	0.095	0.434	0.029	
% of dose	0.02	0.10	0.10	0.14	0.38	0.05	0.07	0.01	88.5
Acetonitrile/ Methanol	89%	92%	79%				93%	98%	96%
Non extracted (PES)	11%	8%	21%				7%	2%	

	Fat	Muscle	Liver	Kidney [a]	Blood [a]	Skin/ Fat [a]	Egg yolk day 7	Egg white day 7	Excreta
Characterisation of PES									
Protease treatment									
Aqueous buffer			12%						
Acetonitrile			1%						
Non-extracted			8%						

[a] no metabolite characterisation carried out.

N/A Not analysed

Total extractable residues represented 89 – 98% TRR in all tissues and eggs except liver where 79% TRR was extracted with acetonitrile/methanol and a further 13% TRR was extracted after protease treatment.

The total contribution of major metabolites of two dose groups in tissues, egg and excreta is summarized in the tables below.

Table 7 Summary of Metabolite Identification in tissues, egg and excreta at 1 mg/kg Dose.

	Fat	Muscle	Liver	Egg Yolk	Egg White[a]	Excreta
				day 7	day 7	
TRR (mg/kg)	0.005	0.003	0.023	0.031	0.002	Na
Metabolite fractions as a percentage of TRR (mg/kg profenofos equivalents)						
Profenofos	-	-	-	-		
CGA55960 glucuronide	-	-	-	-		
CGA55960 Sulfate	13% (0.001)	85% (0.003)	-	88% (0.027)		
CGA55960	77% (0.004)	9% (0.0003)	75% (0.017)	-		
Unknown	-	-	-	-		
Total (%)	90%	94%	75%	88%		

[a] the 1 mg/kg egg whites were not extracted.

Na - not analysed

- not found

Table 8 Summary of Metabolite Identification in tissues, egg and excreta at 10 mg/kg Dose.

	Fat	Muscle	Liver	Egg Yolk	Egg White	Excreta
				day 7	day 7	
TRR (mg/kg)	0.044	0.030	0.280	0.434	0.029	
Metabolite fractions as a percentage of TRR (mg/kg profenofos equivalents)						
Profenofos	-	-		-	-	4%
CGA55960 glucuronide	-	-	4% (0.01)	-	-	-
CGA55960 Sulphate	-	75% (0.023)	-	93% (0.40)	98% (0.028)	-
CGA55960	89% (0.039)	17% (0.005)	71% (0.20)	-	-	91%

	Fat	Muscle	Liver	Egg Yolk	Egg White	Excreta
				day 7	day 7	
Unknown	-	-	-	-	-	4%
Total (%)	89%	92%	75%	93%	98%	95%

- not found

Metabolism of profenofos in the chicken proceeded mainly via hydrolysis of the phosphate ester to form 4-bromo-2-chlorophenol (CGA55960) and its sulfate conjugate. No unmetabolized profenofos was detected in the tissues or eggs. The dose level had little effect on the metabolite profiles in the tissues.

The metabolism of profenofos in hen follows similar pathways to those in the rat and goat.

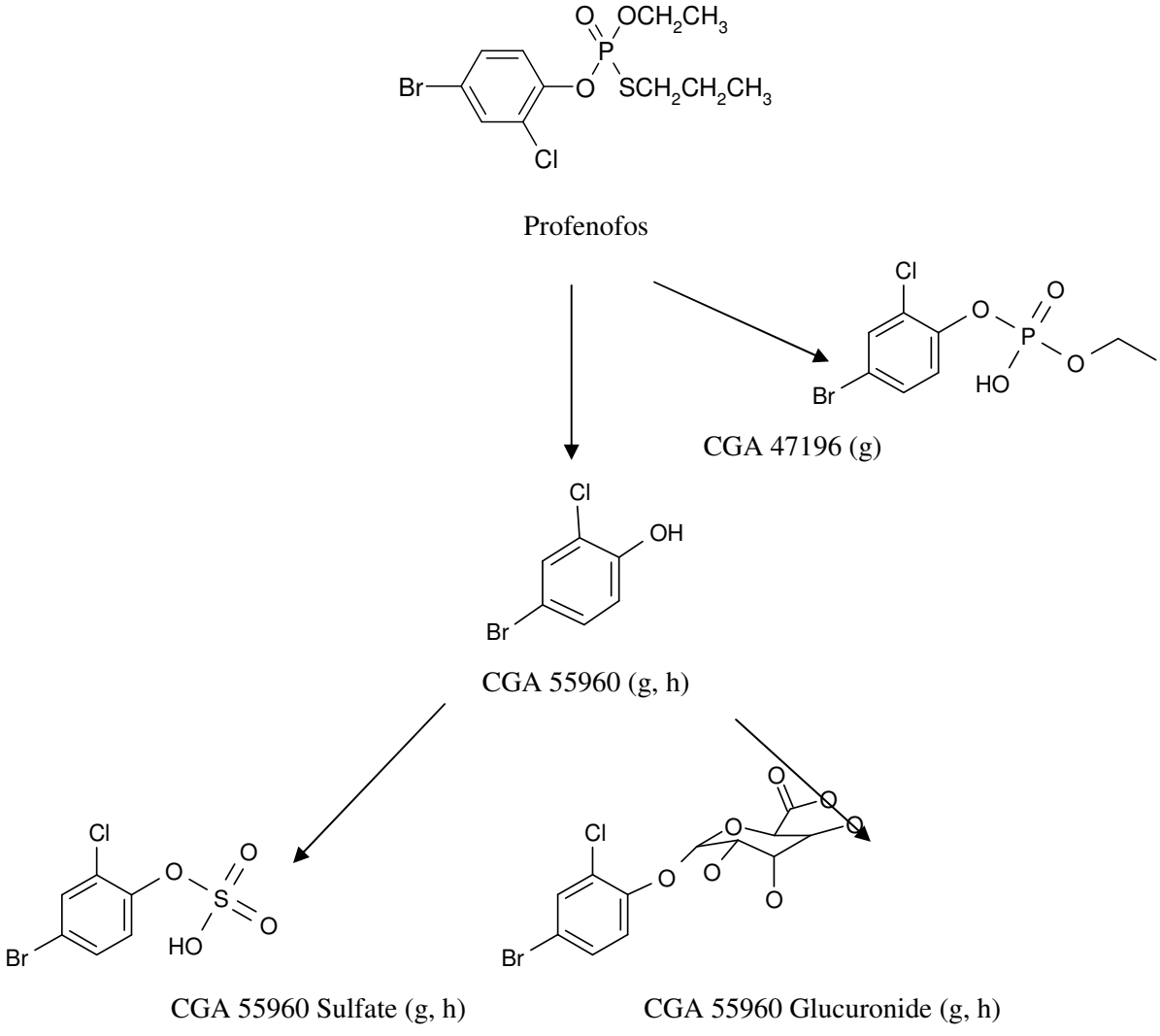


Figure 2: Proposed metabolic pathway of profenofos in goat and hen.

NB. g, h in parentheses mean the compound was only in goat or hen, respectively.

Plant metabolism

The Meeting received plant metabolism studies on cotton, Brussels sprouts, lettuce and tomatoes.

Cotton

Study 1

Ten week old cotton plants (variety Coker 310) grown in a greenhouse (Fischer, Ross and Cassidy, 1975; CGA15324/0234) were sprayed with an ethanol solution of [U-^{14}C -phenyl]-profenofos, once at a rate equivalent to 1.7 kg ai/ha. Each plant was treated with approximately 1.5 times the single application rate in order to simulate a multiple application of pesticide and maximize metabolites

Sample of leaves and stems were collected at 0 day and 6 weeks after treatment and samples of leaves and stems, fibre and seed samples were collected at 12 weeks after treatment. Samples were homogenized with dry ice in a mill and combusted to determine the total radioactivity.

The TRR for leaves and stems of cotton after treatment was determined to be 21.0 mg/kg equivalent to [U-^{14}C -phenyl]-profenofos. The level of radioactivity decreased to 1.1 mg/kg and 0.6 mg/kg in leaves and stems at 6 and 12 weeks, respectively. The fibre and seed at 12 weeks after treatment were analysed only for total radioactivity. The radioactivity in the fibre and seed at 12 weeks was 0.03 mg/kg and 0.06 mg/kg.

Samples of leaves and stems were extracted to give organic, aqueous and unextracted fractions. The organic and aqueous fractions were carried out using two dimensional TLC for quantitative analysis. Immediately after treatment the majority (90.7%) of the TRR was extracted with organic solvent, decreasing to 50.9% at 6 weeks and 38.3% at 12 weeks. The majority (53.3%) of the TRR at 12 weeks represented aqueous soluble components. The aqueous-soluble residues mainly comprised acidic components (43.0% of the TRR).

Identification of the components of the radioactive residue by TLC and GC showed that at 12 weeks after treatment the residues in the leaves and stems comprised parent profenofos (12.5% of the TRR) and CGA55960 (26.4% of the TRR).

Table 9 Identification of metabolites of [^{14}C]-profenofos in cotton plants

Interval after treatment (weeks)	0	6	12
Extractable Radioactivity (mg/kg)	19.89	1.01	0.55
Metabolite fractions as a percentage of TRR			
Profenofos	88.5	49.7	12.5
CGA55960	3.3	10.2	26.4
Unresolved [a]	2.9	31.9	52.8
Total	94.7	91.8	91.7

[a] upon digestion with strong base and acid this fraction was determined to be 95% CGA55960.

The data indicated that the major polar metabolites were sugar acid conjugates of CGA55960.

Study 2

Field cotton (variety Stoneville 213) grown for 10 weeks in a Bosket silt loam soil in Mississippi USA (Simoneaux, Thomas and Cassidy, 1976; CGA15324/0235) was sprayed over-the-top with [U-^{14}C -phenyl]-profenofos. Three consecutive treatments were made at 2-weeks intervals at the rate of 2.2 kg ai/ha per treatment. The ground portions of cotton plants were harvested following application at 10, 12 and 14 weeks after planting. Two additional harvests were taken at 17 and 21 (at maturity) weeks. The mature harvest was separated into leaves, seeds and fibre.

Plant samples were homogenized with dry ice in a mill, and subsamples were combusted. After the final spraying, 14 weeks after planting, the radioactivity in leaves and stems was 60.1 mg/kg equivalent to [U-^{14}C -phenyl]-profenofos. At maturity, 21 weeks after planting, 8.3 mg/kg was in the leaves, 0.4 mg/kg in the seeds and 0.2 mg/kg in the cotton fibre. Biphasic extractions were done on all leaf and/or stem samples to give organic, polar and unextractable fractions. In addition, the samples were extracted with methanol/water. However, seed samples were first refluxed in hexane to remove the oil prior to extraction.

The distribution of radioactivity in the aqueous and organic fractions was determined by two dimensional TLC and GC. Analysis of releasable CGA55960 in mature leaves and seeds was conducted using sequential acid and base hydrolysis.

Immediately after application the majority (81.2%, 78.8% and 78.9% of the TRR for 10, 12 and 14 weeks old plants respectively) of the radioactivity in leaves and stems was extracted with organic solvent, decreasing to 32.9% at 3 weeks after the final application. Organic soluble residues accounted for 34.6% of the TRR in leaves, while 48.3% of the TRR was represented aqueous soluble components.

In mature leaves, the parent profenofos amounted to 31.5% of the TRR. For all samples, free CGA 55960 remained at 2–4% of the TRR in whole plants and in mature leaves. Mature seed contained 1.2% of CGA 55960. A single major polar metabolite (Metabolite B) and a minor one (Metabolite A) were found in mature cotton. Metabolite B was 30.9% of TRR in leaves and 14.8% of TRR in seeds, Metabolite A was 0.1% in leaves and 3.2% in seeds. Hydrolyses of extractable radioactivity from mature leaves and seeds released 69.3% and 37.6% of the TRR as CGA 55960, respectively. A majority of the extractable metabolites contain this moiety, a few percent as CGA 55960 itself but most of it is released from Metabolite B.

Based on ionic character, comparison to available standards, and mass spectra obtained on peracetylated material, Metabolite B was identified as a glucose conjugate of CGA55960.

Table 10 Identification in Cotton at Maturity following Application of [^{14}C]-Profenofos.

Interval after treatment weeks	7	
Age of cotton (weeks)	21	
	Leaves	Seed
TRR (mg/kg) a	8.27	0.35
% TRR		
Profenofos	31.5	-
CGA55960	1.7	1.2
Metabolite Ab	0.1	3.2
Glucose conjugate of CGA55960	30.9	14.8
Unknowns in the extractable fraction	17.7	27.9
Non-extracted	12.1	48.6
Total	94.0	95.7

[a] in profenofos equivalents

[b] metabolite not identified further

Study 3

The field grown cotton (variety DPL-51) in Mississippi USA (Sanson, 1994; CGA15324/1394) was treated with 6 foliar spray treatments of formulated [U-^{14}C -phenyl]-profenofos (radiochemical purity 98.6%) on a weekly basis at a rate of 2.2 kg ai/ha for a total of 6.7 kg ai/ha. This is the maximum annual treatment rate allowed for cotton. A control plot was treated similarly with formulation only. Plant samples were collected at various stages of plant maturity. Whole plant sample were collected immediately before the fourth application (5 days after the third application). Crop samples were

collected at 61 days (mature bolls) and 83 days (remaining mature bolls and stalks) after the last application. Mature harvest bolls were separated into lint and cotton seed.

Based on the combustion radioassay of homogenized cotton samples, immature and mature stalks contained total radio residues (TRR) of 37.6 mg/kg and 13.5 mg/kg, respectively. Mature seed and lint contained respective TRR amounting to 0.66 mg/kg and 1.5 mg/kg.

Samples were extracted with methanol/water followed by partitioning of the aqueous fraction with chloroform. The cotton seed was initially refluxed with hexane to remove oils prior to extraction. Metabolites were identified using TLC, HPLC, NMR and MS. Residues of immature and mature stalk were almost quantitatively extractable (approximately 85% TRR). Two methods were employed to release unextracted residues from the seed. The first consisted of a methanol/water reflux followed by sequential base and acid hydrolysis. The resulting aqueous fraction was partitioned with organic solvents. The second extraction method consisted of enzyme treatment, solubilisation with surfactant, 0.5 M acid treatment, 0.5 M base treatment, 6.0 M acid reflux and 6.0 M base reflux.

Table 11 Identification of metabolites of profenofos in cotton

Crop part	Immature Stalk		Mature Stalk		Seed	
TRR (mg/kg)	37.5		13.5		0.66	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Hexane reflux					11.8	0.077
Aqueous fraction	54.2	20.4	48.6	6.6	19.9	0.131
Chloroform fraction	46.6	17.5	32.5	4.4	1.9	0.013
Post Extraction Solid (PES)	5.8	2.2	13.7	1.9	57.4	0.376
Total	106.6		94.8		91	
Identification						
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Profenofos	33.9	12.7	28.5	3.9	6.5	0.042
CGA55960	2.2	0.82	6.3	0.86	2.1	0.014
Bromochloroanisole	0.8	0.29	0.7	0.09	1.2	0.008
CGA55960 polysaccharide	6.3	2.4	15.1	2.1	1.4	0.009
CGA55960 glucosyl sulfate	33.8	12.7	31.0	4.2	17.3	0.11
CGA55162	7.4	2.8	3.3	0.45	0.4	0.002
Total	84.4	31.6	84.9	11.5	28.9	0.19

The treatment that resulted in the largest amount of radioactivity released from unextracted residue of seed samples were cellulose enzymatic and base hydrolyses. The extracts were characterized, but identification was not successful due to co-extractants and the interference of the base solvent.

The results of the study show that profenofos was metabolized to bromochlorophenol which can be further conjugated to sugars and be likely incorporated into structural components of the plant cell wall.

Brussels sprouts

Brussels sprouts (variety Frigor Star) grown in Switzerland (Gross, 1975; CGA15324/0233) were treated with [U-^{14}C -phenyl]-profenofos. Three applications were made to Brussels sprouts at a rate of 1.1 kg ai/ha per application. Applications were conducted at 25% maturity of the sprouts and repeated twice at two-week intervals. The amount applied and the number of the treatments were identical to those recommended for the practice. Samples were taken 2 h after each treatment, before the second and third treatment and 3 weeks after the last treatment (at usual harvest time).

The total radioactivity of each plant part was determined by direct combustion. Distribution of radioactivity in the plants was determined following exhaustive extraction with methanol/water followed by partitioning with hexane and further separation by TLC. Aqueous and organic fractions were purified and characterized using TLC, GC and MS. Further acid hydrolysis of plant material followed by TLC and GC analysis was conducted to determine the presence of conjugate with the intact CGA 55960 moiety.

At harvest, three weeks after the last application, 3.6 mg/kg and 0.3 mg/kg [U-^{14}C -phenyl]-profenofos equivalents were found in the leaves/stems and sprouts, respectively. More than 90% of the profenofos applied was degraded within two to three weeks after each application. At harvest only 1.9% of the total radioactivity in the plants was in form of profenofos. CGA 55960 in the non-conjugated form, was characterized by TLC and GC as a transient metabolite. It amounted to 1.2% and 0.4% of the TRR in leaves/stems and sprouts, respectively. The polar metabolites represented with 0.6% in the sprouts and 74.2% in the leaves/stems the main radioactive fraction. This fraction from the leaves/stems contained three minor metabolites and in addition two major metabolites too. Their structure was elucidated by MS of their peracetyl and pertrimethylsilyl derivatives. The first major metabolite representing 35.8% of the TRR consisted of the β-D-glucopyranoside of CGA55960. The second major metabolite representing 29.5% of the TRR was shown to be the conjugate of CGA55960 with a disaccharide, possibly gentiobiose.

In addition, all three minor metabolites also formed CGA 55960 during chemical hydrolysis indicating the presence of other types of conjugates with the intact phenol-moiety. One of these metabolites degraded during re-chromatography on TLC to the β-D-glucopyranoside of CGA 55960. The methanol/water insoluble radioactivity increased slightly during the application period but decreased later and accounted for 21.7% of the total plant radioactivity. It was shown by chemical hydrolysis that at least half of this radioactivity still contained the intact CGA55960 moiety.

Table 12 Identification of metabolite of profenofos in Brussels sprouts

Characterisation of Radioactivity	Brussels Sprouts			
	Days after last application			
	Leaves/stems		Sprouts	
	21 days		21 days	
TRR (mg/kg)	3.6		0.3	
	%TRR	mg/kg	%TRR	mg/kg
Profenofos	1.9	0.068	-	-
CGA55960	1.2	0.043	0.4	0.0012
CGA55960 Polysaccharide conjugate	29.5	1.06	0.6	0.0018
CGA55960 Monosaccharide conjugate	35.8	1.29		
Unextracted residue	21.3	0.76	0.4	0.0012
Total	90.2		1.4	

Profenofos was rapidly degraded following application to Brussels sprouts, with 0.3 mg/kg profenofos equivalents in the sprouts at maturity. Parent profenofos was present at 1.9% of the TRR in the leaves/stems but was not found in the sprouts.

Lettuce

Five week old lettuce leaves (Fisher and Cassidy, 1974; CGA15324/0237) were streaked with an ethanolic solution of [U-^{14}C -phenyl]-profenofos. Two leaves per plant were treated by smearing 1 mg of material evenly over each leaf surface. Each plant had 4-5 well developed leaves, and the second and third leaves from top were the ones streaked. Lettuce leaves were sampled at 0, 7, 14 and 21 days after treatment. Only treated leaves were taken from each plant.

Plant samples were homogenized with dry ice in a mill and 150–200 mg samples combusted. Biphasic extractions of samples gave organic, polar and non-extractable fractions. Further ionic characterization of the polar fraction was carried out. Quantitative distribution of the radioactivity was determined by TLC followed by radioassay using LSC.

Immediately after application, the TRR in lettuce represented 310 mg/kg equivalent to profenofos and 98.2% of which was extractable. The level of radioactivity decreased to 169 mg/kg at 14 days and essentially remained constant to 21 days after the last application. Of this radioactivity, 89.3%, 93.3% and 83.9% was extractable at 7, 14 and 21 days respectively. The majority of the radioactive residues over 21 day period represented organo-soluble components. The polar fraction contained mostly acidic metabolites.

The organic and polar fraction were analysed by TLC. At each sampling interval the majority of the radioactive residue was identified as the parent compound. There was no further significant metabolism.

Table 13 Identification of metabolites of profenofos in lettuce

Characterisation of Radioactivity[a]	Lettuce Leaves [b]			
	Days after last application			
	0	7	14	21
TRR (mg/kg)	310	325	168	171
Radioactive distribution	% TRR	% TRR	% TRR	% TRR
Organic	95.2	67.6	69.8	72.9
Polar	3.0	21.7	23.5	11.0
Non-extractable	1.8	10.8	6.7	16.1
Total	100.0	100.1	100.0	100.0
Extractable Radioactivity (mg/kg)	305	291	157	144
	% TRR	% TRR	% TRR	% TRR
Profenofos	91.8	64.8	68.1	61.1
CGA55960	0.9	2.2	0.8	10.3
Unresolved	0.6	19.9	23.0	10.0
Total	93.3	86.9	91.9	81.4

[a] the total radioactivity (mg/kg) is not relevant as the TRR was not obtained under practical conditions.

[b] leaves were streaked to get high initial residues of 310 mg/kg - this treatment does not represent practice.

Tomatoes

The metabolism of profenofos was studied in field grown tomato plants (Sanmeier, 2003; CGA15324/1830). Tomato plants (variety Cristall F1) were first seeded in the greenhouse and then transplanted to the selected field plot (2 m × 3 m) in two rows of three plants. To protect the plants from rain and to avoid wash off of radioactivity after treatment, a plastic cover was provided. The tomato plants were treated with [U-^{14}C -phenyl]-profenofos at a rate of 722 g ai/ha. The treatment was repeated at a rate of 822 g ai/ha and 806 g ai/ha one and two weeks later, respectively. Mature tomatoes as well as tomato leaves were harvested just after the third treatment, and 4, 7 and 14 days later.

Tomatoes were dipped for 30 seconds in methanol and then rinsed with further methanol. The combined washings were radioassayed and were used for TLC-analysis. Washed tomatoes and leaves were homogenized and were taken for combustion to determine the total radioactivity. Aliquots of the homogenized plant material were extracted with methanol/water and the combined extracts were used for TLC-analysis. Thereafter, the non-extractable radioactivity was determined by combustion of dry aliquots of the extracted plant material. Further isolation of metabolites was possible through cellulose and glucosidase treatment. Acid hydrolysis (3 M HCl, 65 °C/18h) of the water phase fractions was

conducted to determine the presence of conjugates with the intact CGA55960-moiety. Characterisation and identification of the metabolites was carried out using LC-MS and LC-NMR.

Total radioactive residues in tomatoes were 1.78 mg/kg, 1.91 mg/kg, 2.07 mg/kg, and 1.06 mg/kg at 0, 4, 7 and 14 days after the last application. About 42% of the TRR was washed off the tomatoes harvested just after treatment by rinsing with methanol. Only minor amounts of radioactivity were found in the washings 4, 7 and 14 days later (15.0%, 10.1% and 5.5% respectively). Nevertheless it was shown by autoradiography of tomatoes harvested 14 days after the last treatment that a considerable amount of the tomato radioactivity was associated with the cuticle, despite the low amount found to be removable by methanol. Unchanged parent profenofos was found as major fraction in tomatoes at all time points. Metabolite fractions I_7 , I_{11}, I_{15} (CGA55960) were found as major fractions. All other metabolite fractions were $\leqq$ 2%. Quantification of the metabolites in tomato fruits is summarized in the Table below.

Table 14 Identification of metabolites in tomato fruit

Interval	1		2		3		4	
Days after 3rd treatment	0		4		7		14	
	% TRR[a]	mg/kg [b]	% TRR[a]	mg/kg [b]	% TRR[a]	mg/kg [b]	% TRR[a]	mg/kg [b]
Metabolite Fractions I_1 (start)	0.2	0.0042	0.4	0.0074	0.3	0.0072	1.1	0.012
I_2	0.4	0.0070	0.4	0.0075	0.7	0.0138	2.0	0.021
I_3	n.d.	n.d.	0.4	0.0073	0.2	0.0035	1.0	0.011
I_4	0.3	0.0056	0.2	0.0035	0.8	0.0169	1.5	0.016
I_5	n.d.	n.d.			0.3	0.0066	1.1	0.011
I_6	0.2	0.0038	0.4	0.0082	0.5	0.0101	1.0	0.011
I_8 (hexose-pentose-pentose conjugate of CGA55960)	0.3	0.0056	0.5	0.0097	1.3	0.028	5.2	0.055
I_8	n.d.					n.d.		
I_9	0.3	0.0050	0.4	0.0068	0.4	0.0093	1.1	0.012
I_{10} (hexose-hexose conjugate of CGA55960)	0.5	0.009	1.1	0.021	1.0	0.021	2.0	0.021
I_{11} (hexose-pentose conjugate of CGA55960)	0.8	0.015	1.5	0.029	2.3	0.048	6.6	0.070
I_{12} (CGA47196)	0.1	0.0011	0.1	0.0012	0.1	0.0012	< 0.1	0.0004
I_{13} (CGA47197)	0.1	0.0017	0.2	0.0041	0.2	0.0047	< 0.1	0.0002
I_{14} (CGA55162)	0.2	0.0040	0.3	0.0057	0.2	0.0038	0.6	0.006
I_{15} (CGA55960)	1.4	0.025	1.1	0.021	1.7	0.035	3.6	0.038
I_{16} (Profenofos)	95.9	1.7	91.9	1.8	82.5	1.7	63.4	0.67
Unresolved	5.4	0.096	3.4	0.066	2.0	0.042	9.0	0.095
Sub. Total	106	1.9	102	2.0	94.7	2.0	99.1	1.0
NE [c]	0.1	0.0018	0.2	0.0038	0.2	0.0041	0.5	0.0053
Total	106	1.8[d]	103	1.9[d]	94.9	2.1[d]	99.6	1.1[d]

n.d. - not detected

[a] - % of the total radioactivity found in tomato, determined by the sum of surface and penetrated radioactivity.

[b] - in equivalents of CGA15324

[c] - NE: non extractable residues

[d] - TRR determined by combustion

Total radioactive residues in leaves decreased from 61.6 mg/kg just after the third treatment to 29.1 mg/kg at day 14. Profenofos accounted for 71.8%, 35.4%, 18.1% and 6.3%, 0, 4, 7 and 14 days after last treatment, respectively. Major metabolite fractions in leaves were I_{15} (CGA55960) and I_{10} (hexose-hexane conjugate of CGA55960). No other metabolite fraction amounted to $\geqq$ 10% at any time point. Quantification of the metabolites in tomato leaves is summarized in the table below.

Various minor tomato metabolite fractions amounted to only 1% to 2% of the TRR or about 0.01 mg/kg to 0.02 mg/kg. Nevertheless some attempts to characterize these minor water soluble fractions were made. In a preliminary experiment it was shown by hydrolysis of the water soluble radioactivity with 3 N HCl (65 °C/18 h) that all fractions contained the CGA55960 moiety, as this compound was the only fraction released by hydrolysis. Based on the available results from chemical hydrolysis as well as from enzyme cleavage it was concluded that essentially all fractions consisted of sugar conjugate of CGA55960. In many cases these conjugates may be complex conjugates consisting of various sugar moieties.

Table 15 Identification of metabolites in tomato leaves

Interval	1		2		3		4	
Days after 3rd treatment	0		4		7		14	
	% TRR[a]	mg/kg[b]	% TRR[a]	mg/kg[b]	% TRR[a]	mg/kg[b]	% TRR[a]	mg/kg[b]
Metabolite Fractions I_1 (start)	1.3	0.79	3.6	1.2	4.5	2.4	8.1	2.4
I_2	1.7	1.0	3.7	1.3	7.8	4.1	7.0	2.0
I_3	0.9	0.55	1.5	0.53	1.9	1.0	2.3	0.67
I_4	1.6	0.99	2.9	1.0	5.0	2.6	3.8	1.1
I_5	1.1	0.65	2.1	0.74	1.8	0.95	1.7	0.50
I_6	0.6	0.35	2.1	0.73	4.7	2.5	5.6	1.6
I_8 (hexose-pentose-pentose conjugate of CGA55960)								
I_8	0.5	0.29	2.3	0.82	2.7	1.4	4.7	1.4
I_9	0.7	0.43	1.8	0.64	2.0	1.1	1.7	0.51
I_{10} (hexose-hexose conjugate of CGA55960)	5.9	3.7	14.7	5.1	24.2	12.7	7.3	2.1
I_{11} (hexose-pentose conjugate of CGA55960)	1.6	1.0	6.4	2.3	3.1	1.6	1.1	0.32
I_{12} (CGA47196)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
I_{13} (CGA47197)	0.4	0.24	1.3	0.46	1.6	0.87	1.6	0.48
I_{14} (CGA55162)	0.3	0.16	1.5	0.53	0.8	0.40	0.5	0.14
I_{15} (CGA55960)	3,1	1.9	6.2	2.2	9.4	4.9	19.6	5.7
I_{16} (Profenofos)	71.8	44.2	35.4	12.4	18.1	9.5	6.3	1.8
Unresolved	5.2	3.2	7.9	2.7	11.5	6.0	15.6	4.6
Sub. Total	96.6	59.4	93.5	32.7	99.1	52.1	86.9	25.3
NE[c]	1.7	1.0	3.6	1.3	4.6	2.4	13.6	4.0
Total	98.2	61.6[d]	97.1	34.9[d]	103	52.6[d]	100	29.1[d]

n.d. not detected

[a] in % of the total radioactivity found in tomato, determined by the sum of surface and penetrated radioactivity.

[b] in equivalents of CGA15324

[c] NE: non extractable residues

[d] TRR determined by combustion

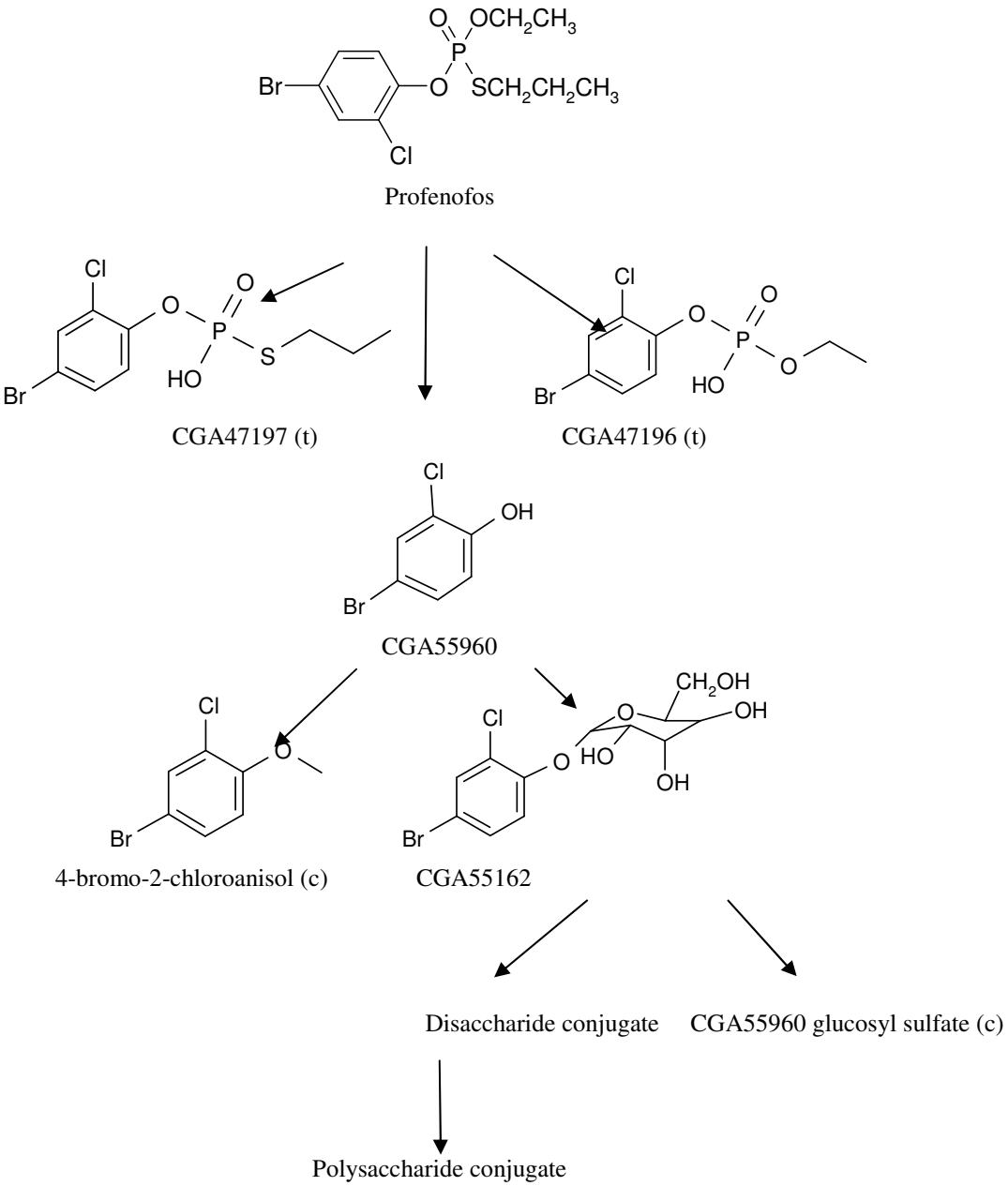


Figure 3 Proposed metabolic pathway of profenofos in plants

NB. c, b, t in parentheses mean the compound was only in cotton, Brussels sprouts or tomato, respectively.

Environmental fate in soil

The Meeting received information on aerobic and anaerobic soil metabolism of profenofos and its degradation products, a rotational crop study as well as the studies on photolysis in soil, dissipation, absorption, mobility in soil, column leaching and fate in water-sediment systems, water and air. Since profenofos is intended for protection of vegetables and supervised trials were conducted with vegetables, studies on soil metabolism and rotational crops are relevant for the current review.

Aerobic soil metabolism

Study 1

The degradation in soil of profenofos was studied for up to 12 weeks with the aid of [U-^{14}C -phenyl]-profenofos (Laanio, 1975: CGA15324/0206). Physical characteristics of the soil are presented below.

Soil parameter	Stein soil
pH (H_2O)	7.5
Organic matter (%)	3.4
Classification (USDA)	Clay loam

For each experiment 232 g (dry weight equivalent) samples of soil were adjusted to 25% moisture content by addition of distilled water or by air drying. Solutions of the radiochemical in water:ethanol (9:1) were applied drop wise to the surface of the soil in each flask to give 4 mg ai/kg. The soil was then mixed to give a uniform distribution of radiochemical. Treated soils were incubated under a 14:10 light/dark cycle at 21 ± 2 °C. The flasks were ventilated with a pump and evolved [^{14}C]-CO_2 trapped with 2M NaOH (40 mL). Four experiments were performed:

- incubation under aerobic/non-sterile conditions for 4 weeks
- incubation under aerobic/non-sterile conditions for 12 weeks
- incubation under aerobic/non-sterile conditions for the first 4 weeks, thereafter anaerobic/non-sterile conditions for 8 weeks (aged anaerobic)
- incubation under aerobic/autoclaved conditions for 12 weeks

The apparatus was ventilated twice daily for one hour with air (Experiments 1, 2, 4 and Experiment 3 during the first 4 weeks) or nitrogen (Experiment 3 during the last 8 weeks). Analysis of the NaOH traps was conducted weekly.

Soils were extracted by methanol/water (8:2) solution. The soil debris was then re-extracted by Soxhlet with methanol for 16 h. Radioactivity in the soil extracts was quantified by LSC. The extracts were analysed by TLC autoradiography and GC.

Four extractable degradation products were detected. Mineralization of profenofos occurred in the aerobic experiment over the 12 week incubation. The concentration of residual [U-^{14}C -phenyl]-profenofos at 0, 28 and 84 days after the application was fitted to a simple first order degradation curve. DT_{50} was estimated at 4.7 days and DT90 at 15.6 days. Results of the study with autoclaved soil indicated that the activity of soil microorganisms is essential for oxidation of the phenyl ring to CO_2. Evolution of [^{14}C]-CO_2 was slow and low levels of non-extractable radioactivity were found.

Table 16 Distribution and recovery of profenofos in soil

	Aerobic 4 weeks	Aerobic 12 weeks	Aerobic 4 weeks then anaerobic 8 weeks	Autoclaved aerobic 12 weeks
	(% of applied radioactivity)			
Profenofos	1.6	0.1	0.0	-
Unknown polar	0.3	0.6	1.2	-
CCGA 55960	1.8	0.5	0.9	65.1
Unknown polar	4.3	1.8	2.2	4.8
Unextracted residue	73.1	68.2	65.2	18.1
CO2	17.1	25.6	19.8	1.1
Total Recovery	98.2	96.8	89.3	89.1

Profenofos degraded rapidly with mineralization and formation of unextracted residues under aerobic soil conditions. Anaerobic incubation for 8 weeks following 4 weeks of aerobic degradation did not modify this degradation pattern. In sterilized soil cleavage of the phenol phosphorous ester bond of profenofos proceeded via chemical hydrolysis, with accumulation of CGA 55960 and formation of unextracted residues.

Study 2

The metabolism of [U-^{14}C -phenyl]-profenofos under aerobic soil conditions was studied (Das, 1992: CGA15324/1176). Physical characteristics of the soil are presented below.

Soil parameter	California soil
pH (H_2O)	7.8
Organic matter (%)	1.5
CEC ($mmol_c$/100 g)	8.3
Classification (USDA)	Sandy loam
Clay (%)	7.0
Silt (%)	25.0
Sand (%)	68.0

A California sandy loam soil was prepared at 75% of its moisture capacity at 0.33 bar and was transferred to individual 25 ml capacity glass test vessels. Each test vessel contained 5 g of soil (dry weight basis). Control vessels contained soil that was sterilized by exposure to gamma radiation. [U-^{14}C -phenyl]-profenofos was applied to the soils at a concentration of 10.9 mg/kg. The test vessels were incubated in the dark at 25 ± 1 °C. The aerobic condition of the soil and the presence of aerobic microorganisms were verified at the beginning and at the end of the study. The moisture content of the test vessels was verified gravimetrically at each sampling. Duplicate test vessels were obtained at 0, 1, 2, 3, 4, 5, 9, 30, 60, 90, 120, 180, 270 and 360 days. Sterile test vessels were sampled at 0, 2 and 360 days. The headspace of each test vessel was purged through a series of traps comprised of polyurethane foam, ethylene glycol and 1 N potassium hydroxide solution.

The trapping solutions and polyurethane foam extract were assayed by LSC, and the polyurethane foam extract was analysed by HPLC for the parent and degradates. The soils were sequentially extracted with 3 different solvent systems and the extracts were analysed by HPLC. The unextractable radioactivity was determined by combustion analysis. Material balance was 98.9 ± 2.7% under non-sterile conditions and 99.7 ± 2.3% under sterile conditions. The chemical identities of the parent and its degradates in the extracts of soil and polyurethane foam were established by HPLC, GC and MS.

[^{14}C]-profenofos concentration declined rapidly to 37.4% of the applied radioactivity (AR) in 3 days and 9.8% by 9 days. Concomitantly, the formation of its major metabolite CGA55960 increased to 49.3% of the AR in 3 days and to a maximum of 79.7% in 60 days. Another important metabolite, 4-bromo-2-chlorophenyl ethyl ether (BCPEE) gradually reached a maximum level of 42.6% of the AR by 360 days. Both of these major metabolites were volatile in nature and were recovered from the volatile traps and organic solvent soil extracts. The level of evolved [^{14}C]-CO_2 during the study period reached a maximum level of 0.4% of the AR at 3 days. The unextractable radioactivity reached a maximum value of 10.2% of the AR by 360 days. Under sterile conditions, the abiotic breakdown yielded CGA55960 as the only degradate, reaching a maximum level of 93.9% of the AR by 360 days.

Table 17 Distribution and recovery of radioactivity of profenofos in soil under aerobic conditions

Day	THPME[a]	MHPME[b]	CP-G[c]	CGA55960	BCPME[d]	BCPEE[e]	Parent	Other[f]	CO_2	Bound[g]	Total
	% of applied radioactivity										

Day	THPME[a]	MHPME[b]	CP-G[c]	CGA55960	BCPME[d]	BCPEE[e]	Parent	Other[f]	CO_2	Bound[g]	Total
0	< 0.1	< 0.1	< 0.1	4.2	< 0.1	< 0.1	92.8	3.6	-	1.0	101.6
1	< 0.1	< 0.1	< 0.1	11.4	< 0.1	< 0.1	87.6	2.0	0.2	2.6	103.8
2	2.0	0.4	0.1	35.0	< 0.1	0.1	57.6	2.1	0.2	2.4	99.9
3	2.6	1.3	0.3	49.3	< 0.1	0.5	37.4	3.6	0.4	2.7	98.1
4	3.2	0.7	0.2	58.3	< 0.1	1.8	28.0	2.6	0.4	3.4	98.6
5	2.1	0.8	0.1	72.5	< 0.1	2.0	15.5	2.1	0.2	1.9	97.2
9	3.7	0.6	< 0.1	59.0	< 0.1	12.8	9.8	3.4	0.4	6.0	95.7
30	5.5	4.3	1.9	74.5	< 0.1	9.6	< 0.1	2.7	0.2	3.2	101.9
60	3.0	2.3	0.5	79.7	0.1	5.1	< 0.1	2.5	< 0.1	6.2	99.4
90	3.0	0.7	0.7	73.3	0.1	12.9	< 0.1	2.3	< 0.1	6.0	99.0
120	5.6	1.8	0.8	78.9	0.7	3.9	< 0.1	2.4	0.1	6.0	100.2
180	9.9	3.9	0.1	63.1	2.4	9.0	< 0.1	3.4	< 0.1	4.5	96.3
270	10.0	0.4	< 0.1	32.0	0.8	41.7	< 0.1	1.7	< 0.1	10.0	96.6
360	6.0	1.8	< 0.1	31.7	1.8	42.6	< 0.1	1.6	< 0.1	10.2	95.7
STERILE											
0	< 0.1	< 0.1	< 0.1	2.7	< 0.1	< 0.1	97.1	1.8	-	0.7	102.3
2	< 0.1	< 0.1	< 0.1	37.4	< 0.1	< 0.1	58.6	3.5	-	0.1	99.6
360	< 0.1	< 0.1	< 0.1	93.9	< 0.1	< 0.1	< 0.1	1.3	< 0.1	2.0	97.2

[a] 2-thioethylenecarboxy-4-hydroxyphenyl methyl ether

[b] 2-mercapto-4-hydroxyphenyl methyl ether

[c] chlorophenol-glucose conjugate

[d] 4-bromo-2-chlorophenyl methyl ether

[e] 4-bromo-2-chlorophenyl ethyl ether

[f] unresolved fractions

[g] unextracted radioactivity

Profenofos was metabolized rapidly in soil under aerobic conditions. The calculated average DT50 under non-sterile conditions was 1.9 days.

Anaerobic soil metabolism

The metabolism of [U-^{14}C -phenyl]-profenofos under anaerobic soil conditions was studied (Das, 1991: CGA15324/1175). Physical characteristics of the soil used are presented in the table below.

Soil parameters	California soil
pH (H_2O)	7.8
Organic matter (%)	1.5
Classification (USDA)	Sandy loam
Clay (%)	7.0
Silt (%)	25.0
Sand (%)	68.0

A California sandy loam soil was prepared at 75% of its moisture capacity at 0.33 bar and was transferred to individual 25 ml capacity glass test vessels. Each test vessel contained 5 g of soil (dry weight basis). Control vessels contained soil that was sterilized by exposure to gamma radiation. [U-^{14}C -phenyl]-profenofos was applied to the soils at a concentration of 10.9 mg/kg. The test vessels were incubated in the dark at 25 ± 1°C. The anaerobic condition of the soil and the presence of anaerobic microorganisms were verified after the establishment of anaerobic conditions and at the end

of the study. The moisture content of the test vessels was verified gravimetrically at each sampling. After 2 days of aerobic incubation, anaerobic conditions were established by purging the test vessels with nitrogen. Duplicate non-sterile test vessels were sampled at 0, 1, 2, 3, 7, 30 and 60 days after conversion to anaerobic conditions. Duplicate sterile test vessels were sampled at 0, and 60 days after conversion to anaerobic conditions. The headspace of each test vessel was purged through a series of traps comprised of polyurethane foam, ethylene glycol and 1 N potassium hydroxide solution.

The trapping solutions and polyurethane foam extract were assayed by LSC. The polyurethane foam extract was analysed by HPLC for the parent and degradates. The soils were sequentially extracted with 3 different solvent systems and the extracts were subjected to HPLC analyses. The unextractable radioactivity was determined by combustion analysis. Material balance was 100.0 ± 2.2%. The chemical identities of the parent and its degradates in the extracts of soil and polyurethane foam were established by HPLC and were confirmed by GC-MS.

[^{14}C]-profenofos concentration declined rapidly from an initial level of 57.6% of the dose at beginning of anaerobic conditions to a low level of 10.0% in 7 days and to a final level of 1.5% by the end of 60 days anaerobic incubation. Concomitantly, the formation of its phenolic metabolite CGA55960 increased to 35.0% to 78.6% of the AR in 7 days, and reached a maximum level of 82.5% by the end of 60 days anaerobic incubation. CGA55960 was the only major metabolite found in the study. The phenolic derivative BCPEE reached a maximum level of 2.7% of the AR by 2 days. There was no significant evolution of CO_2 during the study period. In the case of sterile samples, the polar compounds were never formed, suggesting microbial involvement in the formation of the polar compounds (conjugates) under non-sterile conditions.

Table 18 Distribution and Metabolites of Profenofos in soil under anaerobic conditions

Days	Profenofos	CGA 55960	BCPEE	Polar metabolites	Others	CO_2	N.E.	Total
	% of applied radioactivity							
Non Sterile								
Aerobic								
0	92.8	4.2	< 0.1	< 0.1	3.6	--	1.0	101.6
1	87.6	11.4	< 0.1	< 0.1	2.0	0.2	2.6	103.8
Anaerobic								
0	57.6	35.0	0.1	2.2	2.4	0.2	2.4	99.9
1	37.0	50.0	2.0	3.3	3.3	< 0.1	3.2	98.8
2	28.1	60.4	2.7	3.0	2.8	0.2	3.0	100.2
3	19.8	72.3	2.7	1.3	2.2	< 0.1	2.3	100.6
7	10.0	78.6	1.9	2.4	2.8	0.1	3.6	99.4
30	2.9	78.4	2.0	5.8	3.2	< 0.1	4.8	97.1
60	1.5	82.5	2.7	3.0	4.0	< 0.1	4.4	98.1
Sterile								
Anaerobic								
0	58.6	37.4	< 0.1	< 0.1	3.4	--	0.1	99.5
60	1.3	89.1	1.3	< 0.1	2.6	< 0.1	0.2	98.0

N.E. – non-extractable

Profenofos was metabolized rapidly in soil under anaerobic conditions. The calculated DT_{50} was 2.9 days.

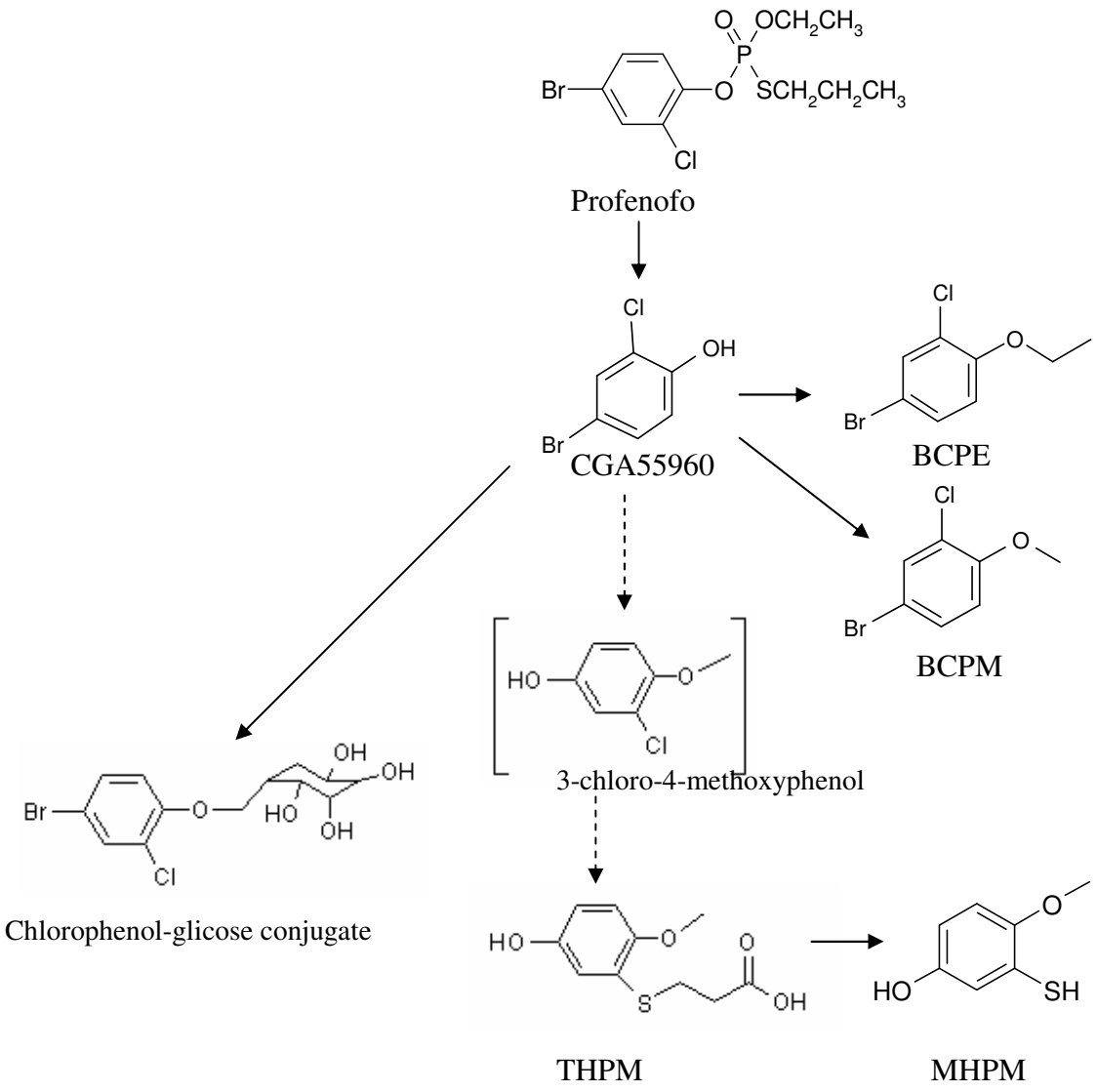


Figure 4: Proposed metabolic pathway of profenofos in soil

Residues in succeeding crops

Study 1

In a confined rotational crop study conducted in the field (Peffer, 1998; CGA15324/1601), a representative leafy vegetable (mustard), root crop (radishes) and small grain (wheat) were planted in the treated soil at 30, 60, 90, 180 and 365 days after test substance application. [U-^{14}C -phenyl]-profenofos as an 8E formulation was applied to bare ground plots at California. The test substance was applied at the maximum seasonal use rate of 6.7 kg ai/ha.

For each planting interval, the leafy vegetable and root crops were harvested at maturity, and the small grain crop was harvested at quarter maturity, half maturity and full maturity. The root crop was separated into root and leaves. The mature small grain crop was separated into straw (including grain husks) and grain. For each planting interval, soil samples were taken prior to each application, after application, at planting, and at the harvest of the mature small grain.

Plant samples were homogenized, extracted with methanol/water, and then partitioned with hexane/heptane. The aqueous fractions were further partitioned using chloroform. All of the fractions were concentrated and analysed by HPLC. The post-extraction solids were subjected to hydrolysis procedures involving cellulase and protease treatments, followed by 0.1 M HCl, 6 M HCl, 0.1 M NaOH and 6 M NaOH hydrolysis. The cellulase solubles were further partitioned with ethyl acetate,

organic fractions were profiled using two dimensional TLC and the aqueous fractions analysed by HPLC. The results are shown in the following table.

Crops planted at the 30-day interval had residues of 0.026–0.157 mg/kg of the TRR. Residue levels were slightly lower at the 60, 90 and 180-day intervals. At the 365-day interval, residue levels decreased for all samples.

Table 19 Total radioactive residues determined in rotational crops

Plant back interval	Plant part	Total Radioactive Residues (mg/kg)		
		Quarter mature	Half mature	Mature
30 days	Mustard - leaves	-	-	0.095
30 days	Radish – leaves	-	-	0.157
30 days	Radish – roots	-	-	0.101
30 days	Wheat - forage	0.067	0.026	-
30 days	Wheat – straw	-	-	0.056
30 days	Wheat – grain	-	-	0.050
60 days	Mustard - leaves	-	-	0.141
60 days	Radish – leaves	-	-	0.079
60 days	Radish – roots	-	-	0.051
60 days	Wheat – forage	0.091	0.040	-
60 days	Wheat – straw	-	-	0.082
60 days	Wheat – grain	-	-	0.070
90 days	Mustard - leaves	-	-	0.041
90 days	Radish – leaves	-	-	0.048
90 days	Radish – roots	-	-	0.046
90 days	Wheat – forage	0.063	0.032	-
90 days	Wheat – straw	-	-	0.042
90 days	Wheat – grain	-	-	0.060
180 days	Mustard - leaves	-	-	0.089
180 days	Radish – leaves	-	-	0.045
180 days	Radish – roots	-	-	0.094
180 days	Wheat – forage	0.031	0.025	-
180 days	Wheat – straw	-	-	0.082
180 days	Wheat – grain	-	-	0.054
365 days	Mustard - leaves	-	-	0.009
365 days	Radish – leaves	-	-	0.013
365 days	Radish – roots	-	-	0.008
365 days	Wheat – forage	0.022	0.017	-
365 days	Wheat – straw	-	-	0.030
365 days	Wheat – grain	-	-	0.030

Intact profenofos was positively identified only in 30-day radishes mature root, at very low levels (0.001 mg/kg). CGA55960 was identified as the major organosoluble residue in 30-day samples, representing a maximum of 3.4% of the TRR (0.005 mg/kg). Other minor metabolites that were identified or characterized were CGA47196, CGA47197, CGA55162 and CGA236844.

For all planting intervals, the majority of the residues in rotational crops were in the post-extraction solids (PES, 41.8–106.2% of the TRR) and aqueous-soluble fraction (2.7–53.7% of the TRR). The aqueous soluble residues were characterized as a mixture of neutral, basic and acidic components. A total hydrolysis method that was applied to representative 30-day and 60-day aqueous samples converted only minor amounts to the common moiety CGA55960 (0.8–6.0% of the TRR). The neutral aqueous fraction contained very polar components that were characterized as monosaccharides and other sugars. The acidic aqueous fraction contained similar polar peaks as the neutral fraction, and two minor acidic metabolites that were characterized but not identified since both represented ≤0.005 mg/kg.

The post-extraction solids from 14 samples were subjected to sequential hydrolysis techniques of increasing severity. The largest amount of radioactivity was released by the initial treatment with cellulase/ cellobiase. The released radioactivity was highly polar, and only minor percentages partitioned into ethyl acetate. Additional radioactivity was released by treatment with protease, suggesting incorporation of radiolabel into proteins. Further hydrolysis with weak and strong acid, and finally weak and strong base, released additional amounts of radioactivity, so that the final PES contained only 0.3–7.9% of the TRR.

The PES samples from 60-day mature wheat straw were subjected to a procedure for the isolation of purified cellulose and crude lignin. The purified cellulose fraction contained 19.0% of the TRR, and the lignin fraction contained 20.2% of the TRR. To further confirm the identification of ^{14}C-cellulose as a major residue, a portion was hydrolysed with acid and derivatised with phenylhydrazine. The phenylosazones of [^{14}C]-glucose that was liberated from [^{14}C]-cellulose and those from the glucose standard has identical mass spectra. This confirmed the identification of [^{14}C]-glucose and [^{14}C]-cellulose as major residues in the rotational crops.

Study 2

Residues of profenofos in rotational crops were investigated following application to soil in Switzerland (Altenburger, 1978; CGA15324/1054). Profenofos was applied once as a Curacron G5 at a rate of 5 kg ai/ha. The results are shown below.

Plant part	PHI (days)	Profenofos Residue (mg/kg)
Lettuce	42	< 0.02
Lettuce	329	< 0.06 (total residue)
Carrot	411	< 0.06 (total residue)
Wheat – grain	405	< 0.06 (total residue)
Wheat – straw	405	< 0.1 (total residue)

Total residues determined as CGA55960 and calculated as profenofos.

No residues above the determination limits were found in any of the crop.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The Meeting received description and validation data for analytical methods for residue of profenofos in agricultural commodities, animal tissues, milk and eggs. Recovery data are summarized in Tables 20 and 21.

The principle of most methods involves a solvent extraction step such as methanol, methanol/water followed by partition steps with non-polar solvent such as benzene, hexane, and clean up steps on silica or florisil column chromatography. The final solution is analysed by GC with NPD, FPD or ECD detection.

Method REM 12/84 was developed for analysis of profenofos and cypermethrin residues in a range of crops (apple, pear and tomato) with high water content (Altenburger, 1984, CGA15324/0038). None of the compounds interfered with the analysis of profenofos at 0.02 mg/kg.

DFG Method S 19 (extended revision) was subject to an independent laboratory validation for the analysis of profenofos residues in meat, milk and eggs (Steinhauer, 2002, SYN-0111V). No significant background interferences were observed and recoveries between 70 and 110 were generally achieved.

Fruits and vegetables (Blass, 1973, REM 28/73)

Analyte:	profenofos	GC-NPD	Method : REM 28/73
LOQ:	0.03 mg/kg		
Description	Crop samples are extracted with methanol and an aliquot partitioned with dichloromethane. Following drying of the extract with anhydrous sodium sulphate and transfer into benzene, the solution is cleaned up on silica. The final solution is analysed using gas chromatography with nitrogen-phosphorus detection (GC-NPD)		

Fruits and vegetables (Altenburger, 1981, REM 16/81)

Analyte:	profenofos	GC-NPD	Method : REM 16/81
LOQ:	0.02 mg/kg		
Description	Crop samples are extracted with methanol and an aliquot partitioned with dichloromethane. Following drying of the extract with anhydrous sodium sulphate and transfer into hexane, the solution is cleaned up on silica. The final solution is analysed using gas chromatography with nitrogen-phosphorus detection (GC-NPD).		

Citrus (Altenburger, 1982, REM 15/82)

Analyte:	profenofos	GC-NPD	Method : REM 15/82
LOQ:	0.02 mg/kg		
Description	Crop samples are extracted with methanol and an aliquot diluted with water and partitioned with hexane. The final extract is analysed using gas chromatography with nitrogen-phosphorus detection (GC-NPD).		

Cotton seed (1977)

Analyte:	profenofos	GC-KCl flame thermionic detector	R & D Analytical Procedure No. 154
LOQ:	0.01 mg/kg		
Description	Crop samples are extracted using Soxhlet apparatus with methanol; an aliquot is diluted with water and partitioned with dichloromethane, transferred into hexane and back partitioned with acetonitrile. The final extract is analysed using gas chromatography with KCl Flame thermionic detection.		

Pome fruit and tomato (Altenburger, 1984, REM 12/84)

Analyte:	profenofos	GC-NPD	Method : REM 12/84
LOQ:	0.02 mg/kg		
Description	Crop samples are extracted with methanol and an aliquot diluted with water/brine and partitioned with hexane. Following a silica clean-up the final extract is analysed using gas chromatography with nitrogen-phosphorus detection (GC-NPD).		

Cotton seed, cabbage, apple, sweet corn and tobacco (Smith, 1975, AG-282)

Analyte:	profenofos	GC-FPD or ECD	Method: AG-282

LOQ: 0.05 mg/kg

Description Crop samples are extracted with methanol/water and a concentrated aliquot partitioned with benzene. Following drying of the extract with anhydrous sodium sulphate the solution is cleaned up on silica. The final solution is analysed using gas chromatography with phosphorus specific flame photometric detection (GC-FPD) or electron capture detection (GC-ECD).

Fruits and vegetables (Kühne-Thu, 1989, REM 119.01)

Analyte:	profenofos	GC-NPD or ECD	Method : REM 119.01

LOQ: 0.02 mg/kg

Description Crop samples are extracted with methanol/water and an aliquot diluted with water and saturated sodium chloride and partitioned with toluene. The final extract is either subjected to silica or florisil column clean up or analysed directly using gas chromatography with nitrogen-phosphorus detection (GC-NPD) or electron capture detection (GC-ECD).

Water-containing vegetables (Altenburger, 1978, REM 16/78)

Analyte:	profenofos as CGA55960	GC-ECD	Method: REM 16/78

LOQ: 0.06 mg/kg

Description Crop and soil samples are subjected to a base hydrolysis in an aqueous sodium hydroxide solution followed by an acid hydrolysis using an aqueous solution of hydrochloric acid, combined with a steam/distillation extraction according to Bleidner. The organic extract is cleaned up by partitioning of the residues into aqueous base and re-partitioning into toluene under acidic conditions. Residues of profenofos are determined by gas chromatography using electron capture detection (GC-ECD).

Oil-containing crops (Altenburger, 1979, REM 6/79)

Analyte:	profenofos as CGA55960	GC-ECD	Method: REM 6/79

LOQ: 0.06 mg/kg

Description Crop samples are subjected to a base hydrolysis in an aqueous sodium hydroxide solution followed by an acid hydrolysis using an aqueous solution of hydrochloric acid, combined with a steam/distillation extraction according to Bleidner. The organic extract is cleaned up by partitioning of the residues into aqueous base and re-partitioning into toluene under acidic conditions. Residues of profenofos are determined by gas chromatography using electron capture detection (GC-ECD).

Crops with high oil content (Ross J A and Ross RD, 1975, AG-283)

Analyte:	profenofos as CGA55960	GC-ECD	Method: AG-283

LOQ: 0.05 mg/kg

Description Samples are extracted with methanol/water (95:5 v/v). An aliquot of this extract is concentrated and refluxed with 6M HCl for 2 hours. Then following dilution the extract is subjected to a base hydrolysis. The extract is then filtered, acidified and extracted with isooctane/hexane, and this extract is then subjected to a silica column clean up. The final solution is analysed using gas chromatography with electron capture detection (GC-ECD).

Cotton seed (Yokley, 1990, AG-322A)

Analyte:	profenofos as CGA55960	GC-ECD	Method: AG-322A

LOQ: 0.05 mg/kg

Description Cotton seed, hulls and meal samples are extracted through refluxing with methanol:water and then following filtering the extract is subjected to a base hydrolysis followed by an acid hydrolysis in the presence of isooctane. The extract is then partitioned into hexane and this extract is then subjected to a silica column clean up. The final solution is analysed using gas chromatography with electron capture detection (GC-ECD).

Cotton seed oil samples are extracted through partitioning acetonitrile refluxing with methanol:water and then following filtering the extract is subjected to a base hydrolysis followed by an acid hydrolysis in the presence of isooctane. The extract is then partitioned into hexane and this extract is then subjected to a silica column clean up. The final solution is analysed using gas chromatography with electron capture detection (GC-ECD).

Cotton seed soapstock samples are extracted through dissolving in water and partitioning with benzene. The extract is transferred into hexane and liquid partitioned with acetonitrile. The extract is then subjected to a base hydrolysis followed by an acid hydrolysis in the presence of isooctane. The extract is then partitioned into hexane and this extract is then subjected to a silica column clean up. The final solution is analysed using gas chromatography with electron capture detection (GC-ECD).

Animal tissue, blood, eggs and milk (Smith, 1976, AG-297)

Analyte:	profenofos	GC-FPD or ECD	Method: REM AG-297

LOQ: 0.05 mg/kg for fat, muscle, liver and kidney. 0.02 mg/kg for blood and eggs. 0.01 mg/kg for milk.

Description Residues of profenofos are extracted from animal tissues, blood, milk and eggs into methanol. Following filtering, the extract is concentrated to an aqueous suspension and partitioned into benzene using a sodium chloride solution. Samples are then cleaned-up using silica gel chromatography (chicken liver requires an additional Florisil column clean up), and analysed by gas chromatography using flame photometric phosphorus specific detection (FPD) or electron capture detection (ECD).

Residues of profenofos are extracted from fat samples into hexane. Following filtering, an aliquot of the extract is partitioned with hexane/acetonitrile to remove oils and fats and the acetonitrile fraction evaporated to dryness and re-dissolved in benzene. Samples are then cleaned-up using silica gel chromatography followed by a Florisil column, and analysed by gas chromatography using flame photometric phosphorus specific detection (FPD) or electron capture detection (ECD).

Plant material and foodstuffs of animal origin (Pelz, 2002, DFG Method S 19)

Analyte:	profenofos	GC-FPD or	Method: S 19 (Extended

ECD revision)

LOQ: 0.02 mg/kg for plant material. 0.01 mg/kg for foodstuffs of animal origin.

Description Residues of profenofos are extracted from plant and animal matrices (exception cotton seed) following extraction module E1(acetone/water (2:1 v/v) followed by a liquid/liquid partition using ethyl acetate/cyclohexane (1:1 v/v) plus sodium chloride solution); cotton seed was extracted using extraction module E7 (acetonitrile/acetone (9:1 v/v) with Calfro E and Celite). Samples were cleanup up using gel permeation chromatography (GPC) followed by final determination using gas chromatography with flame photometric detection (GC-FPD).

Validation Method validation testing included linearity of response and specificity.

Animal tissues, blood, milk and eggs (Smith, 1976, AG-298)

Analyte: profenofos as CGA55960 GC-ECD Method: REM AG-298

LOQ: 0.05 mg/kg for tissues, blood, fat and eggs. 0.02 mg/kg for milk.

Description Residues of profenofos are extracted from animal tissues, blood, milk and eggs into methanol, and from fat samples into hexane followed by a partition into acetonitrile. Aliquots from the methanol or acetonitrile extracts are evaporated to dryness; samples of cow muscle, blood and milk are subjected to a further hexane partition. All samples are subjected to an acid hydrolysis in an aqueous hydrochloric acid solution followed by a base hydrolysis using an aqueous sodium hydroxide solution. The solutions are filtered, acidified and partitioned into isooctane/hexane; the organic extracts are cleaned up using silica gel chromatography. Residues of CGA55960 are determined by gas chromatography using electron capture detection (GC-ECD).

Animal tissues, blood, milk and eggs (Yokley, 1990, AG-326A)

Analyte: profenofos as CGA55960 GC-ECD Method: REM AG-326A

LOQ: 0.05 mg/kg for tissues and eggs. 0.02 mg/kg for milk.

Description Residues of profenofos are extracted from animal tissues, blood, milk and eggs into methanol, and from fat samples into hexane followed by a partition into acetonitrile. Aliquots from the methanol or acetonitrile extracts are evaporated to dryness; samples of cow muscle, blood and milk are subjected to a further hexane partition. All samples are subjected to a base hydrolysis in an aqueous sodium hydroxide solution followed by an acid hydrolysis using an aqueous hydrochloric acid solution. The solutions are partitioned into isooctane/hexane; the organic extracts are cleaned up using silica gel chromatography. Residues of CGA55960 are determined by gas chromatography using electron capture detection (GC-ECD).

Table 20 Analytical recoveries for spiked profenofos in agricultural commodities

Commodity	Analyte	Spiked level (mg/kg)	Recovery (%)		N	Method
			Values	Mean		
Apple	Profenofos	0.2	97, 95	96	2	REM 28/73
		1.0	96, 95	96	2	
Apple	Profenofos	0.05	94	N/A	1	REM 12/84
		0.50	89	N/A	1	
Apple	Profenofos	0.05	68, 95	82	2	AG-282 GC-ECD
		0.10	80-100	N/A	N/A	
Apple	Profenofos	0.05	50, 85	68	2	AG-282 GC-FPD
		0.10	74-92	N/A	N/A	

Commodity	Analyte	Spiked level (mg/kg)	Recovery (%)		N	Method
			Values	Mean		
Citrus Pulp	Profenofos	0.05	Mean reported	108	3	REM 15/82
		0.50		101	4	
Citrus Peel	Profenofos	0.05	Mean reported	113	4	REM 15/82
		0.50		101	4	
Citrus Whole fruit	Profenofos	0.05	Mean reported	103	1	REM 15/82
		0.50		118	1	
Grapes	Profenofos as CGA55960	0.2	110, 102, 102, 105	105	4	REM 16/78
		1.0	93, 90, 95, 93	93	4	
Kiwi	Profenofos	0.40	112	N/A	1	REM 119.01
		4.0	124	N/A	1	
Orange	Profenofos	0.02	112, 115, 121, 108, 121	115	5	DFG Method S 19 GC-FPD
		0.20	95, 71, 99, 98, 101	93	5	
Orange	Profenofos	0.02	116, 112, 126, 107, 119	116	5	DFG Method S 19 GC-MSD
		0.20	112, 84, 115, 110, 114	107	5	
Pear	Profenofos	0.05	96	N/A	1	REM 12/84
		0.50	92	N/A	1	
Brussels Sprouts	Profenofos	0.05	112	N/A	1	REM 16/81
		0.50	91	N/A	1	
Brussels sprouts	Profenofos as CGA55960	0.2	114, 104	109	2	REM 16/78
		1.0	83, 81	82	2	
Cabbage	Profenofos	0.05	114	N/A	1	REM 16/81
		0.50	88	N/A	1	
Cabbage	Profenofos	0.05	65	N/A	1	AG-282 GC-ECD
		0.10	80	N/A	1	
Cabbage	Profenofos	0.05	90	N/A	1	AG-282 GC-FPD
		0.10	88	N/A	1	
Cabbages	Profenofos as CGA55960	0.2	103, 99	101	2	REM 16/78
		1.0	83, 90	87	2	
Cauliflower	Profenofos	0.2	99, 93	96	2	REM 28/73
		1.0	98, 97	98	2	
Cauliflower	Profenofos	0.05	75	N/A	1	REM 16/81
		0.50	104	N/A	1	
Cauliflowers	Profenofos as CGA55960	0.2	100, 109	105	2	REM 16/78
		1.0	122, 114	118	2	
Cucumber	Profenofos	0.05	106	N/A	1	REM 16/81
		0.50	101	N/A	1	
Cucumber	Profenofos as CGA55960	0.2	117, 110	114	2	REM 16/78
		1.0	101, 107	104	2	
Lettuce	Profenofos	0.2	91, 86	89	2	REM 28/73
		1.0	97, 91	94	2	
Lettuce	Profenofos	0.05	96	N/A	1	REM 16/81
		0.50	98	N/A	1	
Pepper	Profenofos	0.05	102	N/A	1	REM 16/81
		0.50	87	N/A	1	

Commodity	Analyte	Spiked level (mg/kg)	Recovery (%)		N	Method
			Values	Mean		
Salad	Profenofos as CGA55960	0.2	84, 84, 72, 69	77	4	REM 16/78
		1.0	85, 77, 100, 97	90	4	
Spinach	Profenofos	0.10	75, 70	73	2	REM 119.01
Tomato	Profenofos	0.05	120	N/A	1	REM 16/81
		0.50	95	N/A	1	
Tomato	Profenofos	0.05	110	N/A	1	REM 12/84
		0.50	95	N/A	1	
Tomato	Profenofos as CGA55960	0.2	120, 93	107	2	REM 16/78
		1.0	108, 71	90	2	
Carrots	Profenofos as CGA55960	0.2	105, 105	105	2	REM 16/78
		1.0	96, 98	97	2	
Potato	Profenofos	0.05	92	N/A	1	REM 16/81
		0.50	94	N/A	1	
Potato	Profenofos	0.02	105, 96, 108, 107, 104	104	5	DFG Method S 19 GC-FPD
		0.20	108, 107, 109, 102, 106	106	5	
Potato	Profenofos	0.02	112, 99, 111, 103, 107	106	5	DFG Method S 19 GC-MSD
		0.20	118, 118, 121, 115, 114	117	5	
Beans with Pods	Profenofos	0.02	107, 104, 111, 108, 110	108	5	DFG Method S 19 GC-FPD
		0.20	110, 106, 108, 109, 89	104	5	
Beans with Pods	Profenofos	0.02	113, 118, 115, 109, 123	116	5	DFG Method S 19 GC-MSD
		0.20	120, 113, 117, 116, 96	112	5	
Maize grain	Profenofos	0.05	104	N/A	1	REM 16/81
		0.50	103	N/A	1	
Sweet corn Ears	Profenofos	0.05	82	N/A	1	AG-282 GC-ECD
		0.10	81	N/A	1	
Sweet corn Ears	Profenofos	0.05	114	N/A	1	AG-282 GC-FPD
		0.10	88	N/A	1	
Sweet corn Fodder	Profenofos	1.0	112	N/A	1	AG-282 GC-ECD
		3.0	103	N/A	1	
Sweet corn Fodder	Profenofos	1.0	89	N/A	1	AG-282 GC-FPD
		3.0	84	N/A	1	
Wheat grain	Profenofos as CGA55960	0.2	Mean reported	82.5	4	REM 6/79
		1.0	Mean reported	89.8	4	
Wheat straw	Profenofos as CGA55960	0.2	71, 76	74	2	REM 6/79
		1.0	79, 79	79	2	
Olive	Profenofos	0.05	88	N/A	1	REM 16/81
		0.50	81	N/A	1	
Cotton seed	Profenofos	0.1-0.2	Mean reported	100	3	R & D Analytical Procedure 154
		1		100	4	
Cotton seed	Profenofos	0.05	62, 72	67	2	AG-282 GC-ECD
		0.10	76, 88	82	2	
Cotton seed	Profenofos	0.05	62. 80	71	2	AG-282 GC-FPD
		0.10	67, 82	75	2	
Cotton seed oil	Profenofos as	0.2	95, 89	92	2	REM 6/79

Commodity	Analyte	Spiked level (mg/kg)	Recovery (%)		N	Method
			Values	Mean		
Crude	CGA55960	1.0	Mean reported	93.3	4	
Cotton seed oil Refined	Profenofos as CGA55960	0.2	Mean reported	80.2	4	REM 6/79
		1.0	Mean reported	76.3	4	
Cotton seed Cake	Profenofos as CGA55960	0.2	106, 123	115	2	REM 6/79
		1.0	93, 99	96	2	
Cotton seed	Profenofos as CGA55960	0.05	82	N/A	1	AG-283
		0.10	71	N/A	1	
Cotton seed	Profenofos as CGA55960	0.20	106, 101	104	2	AG-322A
		0.40	110, 98, 97	102	3	
		1.0	95, 101, 101, 95, 101	99	5	
		1.5	104	N/A	1	
Cotton seed	Profenofos	0.02	136, 118, 118, 102, 117	118	5	DFG Method S 19 GC-FPD
		0.20	116, 98, 114, 103, 104	107	5	
Cotton seed	Profenofos	0.02	140, 131, 124, 98, 124	123	5	DFG Method S 19 GC-MSD
		0.20	118, 107, 112, 103, 96	107	5	
Tobacco	Profenofos	0.05	105	N/A	1	REM 16/81
		0.50	100	N/A	1	
Tobacco	Profenofos	1.0	92	N/A	1	AG-282 GC-ECD
Tobacco	Profenofos	1.0	73	N/A	1	AG-282 GC-FPD

N/A - Not applicable

Table 21 Analytical recoveries for spiked profenofos in animal tissues

Commodity	Analyte	Spiked level (mg/kg)	Recovery (%)		n	Method
			Values	Mean		
Cow tissue (round)	Profenofos	0.05	105, 72, 95	91	3	AG-297
Cow tissue (round)	Profenofos as CGA55960	0.05	85, 65, 96	82	3	AG-298
Cow tissue (round)	Profenofos as CGA55960	0.05	84, 65, 96	82	3	AG-326A
Cow tissue (loin)	Profenofos	0.05	55, 95	N/A	2	AG-297
Cow tissue (loin)	Profenofos as CGA55960	0.05	103, 72	88	2	AG-298
Cow tissue (loin)	Profenofos as CGA55960	0.05	103, 72	88	2	AG-326A
Cow fat (omental)	Profenofos	0.05	94, 76	85	2	AG-297
		0.10	96, 77	87	2	
Cow fat (omental)	Profenofos as CGA55960	0.05	74, 76	75	2	AG-298
		0.10	72, 74	73	2	
Cow fat (omental)	Profenofos as CGA55960	0.05	76, 74	75	2	AG-326A
		0.10	72, 74	73	2	
Cow fat (perirenal)	Profenofos	0.05	100	N/A	1	AG-297

Commodity	Analyte	Spiked level (mg/kg)	Recovery (%)		n	Method
			Values	Mean		
Cow fat (perirenal)	Profenofos as CGA55960	0.05	72	N/A	1	AG-298
Cow fat (perirenal)	Profenofos as CGA55960	0.05	72	N/A	1	AG-326A
Cow kidney	Profenofos	0.05	80	N/A	1	AG-297
		0.10	110	N/A	1	
Cow kidney	Profenofos as CGA55960	0.05	89	N/A	1	AG-298
		0.10	72	N/A	1	
Cow kidney	Profenofos as CGA55960	0.05	89, 78, 78	82	3	AG-326A
		0.10	72, 73, 78	74	3	
Cow liver	Profenofos	0.05	87	N/A	1	AG-297
		0.10	98	N/A	1	
Cow liver	Profenofos as CGA55960	0.05	76	N/A	1	AG-298
		0.10	79	N/A	1	
Cow liver	Profenofos as CGA55960	0.05	76	N/A	1	AG-326A
		0.10	79	N/A	1	
Cow blood	Profenofos	0.02	82	N/A	1	AG-297
		0.05	95	N/A	1	
Cow blood	Profenofos as CGA55960	0.05	74	N/A	1	AG-298
Cow blood	Profenofos as CGA55960	0.01	74	N/A	1	AG-326A
Chicken tissue (thigh)	Profenofos	0.05	96	N/A	1	AG-297
Chicken tissue (thigh)	Profenofos as CGA55960	0.05	98	N/A	1	AG-298
Chicken tissue (thigh)	Profenofos as CGA55960	0.05	98	N/A	1	AG-326A
Chicken tissue (breast)	Profenofos	0.05	110	N/A	1	AG-297
Chicken tissue (breast)	Profenofos as CGA55960	0.05	73	N/A	1	AG-298
Chicken tissue (breast)	Profenofos as CGA55960	0.05	73	N/A	1	AG-326A
Chicken tissue (liver)	Profenofos	0.05	101	N/A	1	AG-297
Chicken tissue (liver)	Profenofos as CGA55960	0.05	65	N/A	1	AG-298
Chicken tissue (liver)	Profenofos as CGA55960	0.05	65	N/A	1	AG-326A
Chicken fat	Profenofos	0.05	93	N/A	1	AG-297
Chicken fat	Profenofos as CGA55960	0.05	78	N/A	1	AG-298
Chicken fat	Profenofos as CGA55960	0.05	68	N/A	1	AG-326A
Meat	Profenofos	0.01	102, 101, 95, 103	100	5	DFG Method S 19 GC-FPD
		0.10	95, 96, 97, 100, 98	97	5	

Commodity	Analyte	Spiked level (mg/kg)	Recovery (%)		n	Method
			Values	Mean		
Meat	Profenofos	0.01	92, 100, 90, 90	93	5	DFG Method S 19 GC-MSD
		0.10	91, 96, 102, 98, 94	96	5	
Milk	Profenofos	0.01	81, 65	73	2	AG-297
		0.02	81, 65	73	2	
Milk	Profenofos	0.01	96, 96, 96, 99, 93	96	5	DFG Method S 19 GC-FPD
		0.10	95, 90, 100, 100, 101	97	5	
Milk	Profenofos	0.01	91, 83, 87, 95, 95	90	5	DFG Method S 19 GC-MSD
		0.10	94, 99, 104, 102, 106	101	5	
Milk	Profenofos as CGA55960	0.02	70, 58	64	2	AG-298
		0.04	79	N/A	1	
Milk	Profenofos as CGA55960	0.02	60, 60	60	2	AG-326A
		0.04	63, 75	69	2	
Eggs	Profenofos	0.02	65	N/A	1	AG-297
		0.05	85	N/A	1	
Eggs	Profenofos	0.01	95, 114, 107, 95, 121	106	5	DFG Method S 19 GC-FPD
		0.10	90, 92, 75, 83, 81	84	5	
Eggs	Profenofos	0.01	84, 97, 97, 91, 86	91	5	DFG Method S 19 GC-MSD
		0.10	82, 88, 74, 77, 75	79	5	
Eggs	Profenofos as CGA55960	0.05	73	N/A	1	AG-298
Eggs	Profenofos as CGA55960	0.02	73	N/A	1	AG-326A

N/A - Not applicable

Stability of Pesticide Residues in Stored Analytical Samples

The Meeting received information on the freezer storage stability of profenofos residues and of total residues of profenofos determined as CGA55960 in cotton seed and grapes. Residues were generally stable.

Cottonseed

The stability of profenofos in cottonseed stored frozen was evaluated fortifying homogenized cottonseed with 1.0 mg/kg of profenofos (Smith, 1976; GAAC-76034). The samples were stored in a freezer at -15°C. Duplicate, fortified samples were analysed for profenofos using method AG-282 (Smith, 1975) at 0-day, 1-month, 2-month and 3-month intervals. A control and freshly fortified (1.0 mg/kg) recovery sample were run at each sampling intervals.

Field- treated cottonseed samples were also stored in a freezer at -15°C for 12 months. These samples were analysed for profenofos and total residues of profenofos (determined as CGA55960) using AG-282 and AG-283 (Ross JA and Ross RD, 1975).

The result of the study showed that profenofos and its metabolites convertible toCGA55960 were stable for at least 1 year if stored in a freezer at -15°C.

Table 22 Storage stability of profenofos in cottonseed

Matrix	Storage interval	Fortification, mg/kg	Residue remaining, mg/kg	Average % remaining
Cottonseed (Fortified)	0 day	1.0	0.92, 0.92	92
	1 month	1.0	1.0, 1.1	105

Matrix	Storage interval	Fortification, mg/kg	Residue remaining, mg/kg	Average % remaining
	2 month	1.0	0.85, 0.88	87
	3 month	1.0	1.0, 1.1	105
Matrix	Storage interval, days	Application rate, kg ai/ha	% Remaining of profenofos	% Remaining of total residues of profenofos
Cottonseed (Field-treated)	371	0.56 × 6	217	84
	371	1.12 × 6	- -	102
	405	0.56 × 6	-	145
	405	1.12 × 6	67	100

Residues are corrected for procedural recoveries < 100%. – :not analysed

The stability of profenofos in cottonseed that had been treated with profenofos using the Curacron 8E formulation was evaluated (Hayworth, 1993; ABR-93029). Samples of weathered cottonseed and processed sample consisting of hulls, crude oil and soapstock were analysed on 0-day and stored under freezer storage conditions (approximately -20°C). These samples were analysed for total residues of profenofos determined as CGA55960 using AG-322A (Yokley, 1990) at various intervals for up to 24 months. A control sample and procedural recovery sample were run at each sampling intervals. The result of the study showed that total residues of profenofos were stable in cottonseed and hulls under freezer storage conditions for a minimum of 24 months, in crude oil for a minimum of 22 months, and in soapstock for a minimum of 23 months.

Table 23 Storage stability of total profenofos residues in cottonseed

Matrix	Storage interval	Procedural recoveries %	Residues remaining, mg/kg	Average % remaining
Cottonseed	0 day	90	1.9, 1.9	-
	3 months	81	3.0, 2.5	147
	9 months	94, 120, 96	1.3, 1.5, 1.4, 1.3, 1.7, 1.6	68
	14 months	109	1.3, 1.3	68
	17 months	95	1.3, 1.2	68
	24 months	94	1.2, 1.3	68
Cottonseed hulls	0 day	121	1.5, 1.5	-
	3 months	119	1.3, 1.6	100
	10 months	104, 136	1.1, 1.6, 1.2, 1.6	93
	14 months	97	1.3, 1.3	87
	17 months	93	1.1, 1.0	73
	24 months	105	1.2, 1.1	80
Crude oil	0 day	90	0.14, 0.15	-
	2 months	80	0.14, 0.15	100
	9 months	66	0.12, 0.13	87
	13 months	57	0.08, 0.11	67
	16 months	72	0.10, 0.11	73
	22 months	84	0.11, 0.11	73
Soapstock	0 day	101	0.55, 0.53	-
	3 months	95	0.65, 0.62	119
	9 months	65	0.72, 0.93	154
	14 months	65	1.2, 0.85	185
	21 months	101	0.78	144
	23 months	83	0.88, 0.89	89

Residues are corrected for procedural recoveries < 100%.

Grapes

A freezer storage stability study was conducted to confirm validity of residue results of profenofos in grapes. Treated, residue containing 0-day grape samples and untreated samples, fortified with profenofos at 5.0 mg/kg were placed in frozen storage at –20 °C for 18 months. Duplicate samples of both types were analysed for profenofos using method REM 28/73 (Blass, 1973) at 0-day, 6-month, 12-month and 18-month intervals. At the sampling interval control and freshly fortified recovery samples (1.0 mg/kg) were run, but no procedural recovery data were available to confirm the performance of the analytical method in each occasion. The results of the study showed profenofos to be stable in grapes under freezer storage conditions for a minimum of 18 months.

Table 24 Storage stability of profenofos in grapes

Matrix	Storage interval	Residue remaining, mg/kg	Average % remaining
Grapes (Field sample)	0 day	6.75, 6.75	100
	6 month	5.70, 5.75	85
	12 month	5.50, 5.25	80
	18 month	5.13, 4.81	74
Grapes (Fortified sample)	0 day	5.00, 5.06	101
	6 month	4.44, 4.50	90
	12 month	4.31, 4.44	88
	18 month	4.50, 3.94	85

Residues are not corrected for recovery values.

Animal commodities

The Meeting received information on the stability of residues of profenofos and metabolite CGA55960 in animal tissues, milk and eggs when stored at freezer temperatures for 1 year. Residues were generally stable.

The stability of profenofos and its major metabolite CGA55960 was studied in meat, liver, milk and eggs (Eudy, 1993-1994, ABR-93033). Control samples of beef muscle, liver and eggs were fortified with profenofos at 0.5 mg/kg and control samples of milk were fortified at 0.2 mg/kg. Samples were stored under freezer storage conditions (approximately –20 °C). These samples were analysed for total residues of profenofos determined as CGA55960 using method AG-326A (Yokley, 1990) at 0-day, 1-month, 1.5-month, 6-month and 12-month intervals. Each analysis set contained the procedural recovery samples and the storage stability samples. The result of the study showed total residues of profenofos to be stable in beef muscle, liver, milk and egg samples stored under freezer storage conditions for a minimum of 12 months.

Table 25 Storage stability of total profenofos residues in animal commodities

Matrix	Storage interval, days	Procedural recoveries %	Residue remaining, mg/kg	Average % remaining
Beef muscle	0	114, 118	0.62, 0.59	122
	46	129, 102	0.66, 0.68	134
	60	102, 100	0.54, 0.62	116
	178	90, 115	0.54, 0.53	108
	367	96, 83	0.48, 0.53	102
Beef liver	0	114, 112	0.55, 0.62	118
	42	130, 124	0.59, 0.55	114
	59	112, 123	0.51, 0.47	98

Matrix	Storage interval, days	Procedural recoveries %	Residue remaining, mg/kg	Average % remaining
	179	126, 128	0.62, 0.59	122
	361	86, 90	0.44, 0.40	84
Milk	0	119, 116	0.22, 0.25	120
	46	82, 109	0.22, 0.23	115
	60	113, 114	0.21, 0.20	105
	186	101, 114	0.23, 0.24	115
	362	113, 123	0.25, 0.20	115
Eggs	0	103, 105	0.52, 0.53	106
	42	106, 107	0.58, 0.59	118
	59	113, 116	0.54, 0.54	108
	180	104, 107	0.52, 0.58	110
	367	69, 69	0.56, 0.53	110

Residues are corrected for procedural recoveries.

USE PATTERNS

The Meeting received labels from many countries in North America, Latin America, Asia and Australia. It also received labels from other countries but they were not used in the review since they were either not related to the supervised trials provided or written in languages other than English or Spanish. Pests controlled are summarized in Table 26.

Information of registered formulations, application methods and dosage rate of profenofos for uses on the crops for which supervised trial data were provided is summarized in Table 27. On most of labels provided there are instructions about application intervals.

Table 26 Information on pests controlled by profenofos

Crop	Pests controlled
Mango	Thrips
Mangosteen	Chili thrips
Cabbage and other crucifer	Diamondback moths, Cabbage worms, Loopers, Aphids
Tomato	Fruitworm, Armyworms, Cutworms
Watermelon	Thrips
Beans	Bean flies, Aphids
Soybeans/Mung beans	Corn earworms, Leafminers
Cotton	Thrips, Bollworms, Budworms, Aphids, Plant bugs, Lygus bugs, Leaf perforator, Armyworms, Mites, Whitefly

Table 27 Registered uses of profenofos relevant to the review

Crop	Country	Formulation		Application of profenofos					PHI days
		Type	Conc. of profenofos (conc. of other ingredient)	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hL	Max number	
Mango	Thailand	EC	500 mg/L	Foliar	0.005-0.0075[a]	10 [b]	0.05-0.075	7 days intervals	21
Mangosteen	Thailand	EC	500 mg/L	Foliar	0.023-0.030 [a]	15-20 [b]	0.15	14 days intervals	21

Crop	Country	Formulation		Application of profenofos					PHI days
		Type	Conc. of profenofos (conc. of other ingredient)	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hL	Max number	
Cabbage	Brazil	EC	400 g/L (40 g/L cypermethrin)	Foliar	0.24	600	0.04	Depends on the intensity	14
Cabbage	Indonesia	EC	500 g/L	Foliar	0.38-0.6	500-800	0.075	Depends on the intensity	
Cabbage	Philippines	EC	500 g/L	Foliar	0.5-0.75	500	0.1-0.15	NS	7
Cabbage	South Africa	EC	500 g/L	Foliar	0.38 0.5	300-500 200-300		7-10 days intervals	7
Water melon	Brazil	EC	400 g/L (40 g/L cypermethrin)	Foliar	0.24	600	0.04	Depends on the intensity	4
Water melon	Indonesia	EC	500 g/L	Foliar	0.38-0.6 0.25-0.8	500-800	0.075 0.05-0.1	NS	NS
Water melon	Philippines	EC	500 g/L	Foliar	0.5-0.75	500	0.1-0.15	NS	7
Pepper (Chilli)	Indonesia	EC	500 g/L	Foliar	0.38-1.2 0.13-0.4	500-800	0.075-0.15 0.025-0.05	Depends on the intensity	
Tomato	Brazil	EC	400 g/L (40 g/L cypermethrin)	Foliar	0.27-0.36 0.2-0.36 0.25-0.5 0.35-0.5 0.3	900 500-900 500-1000 700-1000 1000	0.03-0.04 0.04 0.05 0.05 0.03	Depends on the intensity	10
Tomato	Chile	EC	720 g/L	Foliar	0.43-0.58 0.58-0.72			NS	21
Tomato	Indonesia	EC	500 g/L	Foliar	0.38-1.2	500-800	0.075-0.15	Depends on the intensity	
Tomato	Philippines	EC	500 g/L	Foliar	0.5-0.75	500	0.1-0.15	NS	7
Tomato	South Africa	EC	500 g/L	Foliar	0.38-0.5 0.25-0.38 0.5-0.75	500-1500 250-500		NS	4
Soybean	Brazil	EC	400 g/L (40 g/L cypermethrin)	Foliar	0.04-0.048	80-200		NS	30
Soybean	Brazil	EC	500 g/L	Foliar	0.08-0.1			NS	21
Soybean	Philippines	EC	500 g/L	Foliar	0.5-0.75	500	0.1-0.15	NS	7

Crop	Country	Formulation		Application of profenofos					PHI days
		Type	Conc. of profenofos (conc. of other ingredient)	Method	Rate kg ai/ha	Volume L/ha	Spray conc. kg ai/hL	Max number	
Cotton	Australia	EC	250 g/L	Foliar	0.25-1.0			NS[c]	28
Cotton	Australia	EC	500 g/L	Foliar	0.25-1.0			NS	28
Cotton	Brazil	EC	400 g/L (40 g/L cypermethrin)	Foliar	0.4 0.06-0.1	80-200		NS	20
Cotton	Brazil	EC	500 g/L	Foliar	0.1-0.15 0.25-0.5			NS	15
Cotton	Indonesia	EC	500 g/L	Foliar	0.5-0.8	500-800	0.1	Depends on the intensity	
Cotton	Colombia	EC	500 g/L	Foliar	0.75-1.0			NS	14
Cotton	Philippines	EC	500 g/L	Foliar	0.5-0.75	500	0.1-0.15	NS	7
Cotton	South Africa	EC	500 g/L	Foliar Aerial	0.55 0.75 0.83	100 100 30	0.55 0.75	NS	21
Cotton	USA	EC	730 g/l	Foliar/Aerial	0.14-0.86 [d]	47/9.4		2-4 [e]	14

[a] The unit of rate is kg ai/tree

[b] The unit of volume is l/tree

[c] NS = Not specified on the label

[d] For lepidopteran pests only, may be used 0.86 kg ai/ha per application with a maximum of 2 applications per season

[e] Not apply more than 4.3 kg ai/ha per season

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised field trials for the following crops.

Commodity	Group	Table No.
Mango	Tropical fruits, inedible peel	Table 28
Mangosteen		Table 29
Cabbages	Brassica vegetables	Table 30
Watermelon	Fruiting vegetables, Cucurbits	Table 31
Chilli peppers	Fruiting vegetables, other than cucurbits	Table 32
Tomatoes		Table 33
Soybean	Pulses	Table 34
Cotton seed	Oilseeds	Table 35
Cotton meal, hulls, gin trash	Feeds	Table 36

In these tables, application rates are reported for profenofos only. Unless otherwise noted, the residue concentrations being reported are also for profenofos. When residues were not detected they are shown as below the LOQ (e.g., < 0.01 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures or, for residues near the LOQ, to one significant figure. Residue values from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and STMRs. Those results included in the evaluation are double underlined.

Laboratory reports included method validation with procedural recoveries from spiking at residue levels similar to those occurring in samples from the supervised trials. Date of analyses or duration of residue sample storage were also provided. Although trials included control plots, no control data are recorded in the tables except where residues in control samples exceeded the LOQ. Residue data are unadjusted for percent recovery.

Conditions of the supervised residue trials were generally well reported in detailed field reports. Most field reports provided data on the sprayers used, plot size, field sample size and sampling date.

Mango

A total of six field trials on mango were conducted in Thailand. The studies conducted used the EC formulation containing 500 g/L profenofos. The GAP for mango in Thailand is based on multiple applications (7 days intervals) of profenofos with a PHI of 21 days. The use rate is 0.075 kg ai/hL with the intended kg ai/ha varying with water volume according to the height of mango tree. The maximum volume of 10 L/tree is recommended for mango tree of 5 m or higher. Smaller tree requires less spray volume. The supervised mango trials conducted in Thailand were performed with 4 applications of profenofos at 0.075 kg ai/hL in support of a PHI of 21 days. In all trials, mature fruits were present at the last application, thus giving a worst-case potential for residue in the RAC.

The analytical method was validated with analyses by spiking control samples with profenofos at fortification levels ranging from 0.05 to 0.5 mg/kg. The limit of quantification (LOQ) was 0.01 mg/kg.

Table 28 Profenofos residues in mango from supervised trials in Thailand

Country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	g ai/tree	kg ai/hL	water, L/tree	no.			
GAP, Thailand	EC		0.075	10		21		
Thailand, 2004 (Namdokmai)	EC	3.8	0.075	5	4	0 1 5 7 10 14 21	0.88 0.83 0.20 0.07 0.05 0.04 < 0.01	Chukiatwatana, 2004, PRO-MG-01
Thailand, 2004 (Namdokmai)	EC	3	0.075	4	4	0 1 5 7 10 14 21	1.2 0.89 0.62 0.41 0.32 0.10 0.05	Chukiatwatana,2004, PRO-MG-02
Thailand, 2004 (Namdokmai)	EC	1.5	0.075	2	4	0 1 5 7 10 14 21	1.2 1.0 0.53 0.50 0.41 0.23 0.06	Chukiatwatana,2005, PRO-MG-03
Thailand, 2004 (Namdokmai)	EC	1.9	0.075	2.5	4	0 2 5 7	1.4 1.0 0.75 0.67	Chukiatwatana,2005, PRO-MG-04

Country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	g ai/tree	kg ai/hL	water, L/tree	no.			
						10	0.53	
						14	0.27	
						21	0.05	
Thailand, 2005 (Namdokmai)	EC	1.5	0.075	2	4	0	1.1	Chukiatwatana,2006, PRO-MG-05
						1	0.80	
						5	0.59	
						7	0.38	
						14	0.08	
						21	0.07	
Thailand, 2006 (Namdokmai)	EC	2.6	0.075	3.5	4	0	2.2	Chukiatwatana,2006, PRO-MG-06
						2	1.4	
						7	0.31	
						10	0.15	
						14	0.07	
						21	0.06	

Mangosteen

Four field trials were conducted in Thailand using an EC formulation containing 500 g/L profenofos. The GAP for mango in Thailand is based on multiple applications (14 days intervals) of profenofos with a PHI of 21 days. The use rate is 0.15 kg ai/hL varying with water volume according to the height of mangosteen tree. The volume of 15–20 l/tree is recommended for mangosteen tree of 8 m or higher, using high pressure pump sprayer. Smaller tree requires less spray volume. The supervised mangosteen trials conducted in Thailand were performed with 3 applications of profenofos at 0.15 kg ai/hL in support of a PHI of 21 days.

The analytical method was validated with analyses by spiking control samples with profenofos at fortification levels ranging from 0.05 to 0.5 mg/kg. The limit of quantification (LOQ) was 0.05 mg/kg.

Table 29 Profenofos residues in mangosteen from supervised trials in Thailand

Country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	g ai/tree	kg ai/hL	water, L/tree	no.			
GAP, Thailand	EC		0.15	20		21		
Thailand, 2004	EC	30	0.15	20	3	0	1.2	Suvaparp, 2004, PRO-MGS-01
						1	2.5	
						3	2.2	
						5	3.3	
						7	2.4	
						10	2.7	
						14	2.8	
						21	2.3	
Thailand, 2005	EC	30	0.15	20	3	0	2.9	Suvaparp, 2005, PRO-MGS-02
						1	2.9	
						3	2.5	
						5	1.7	
						7	1.3	
						10	1.7	
						14	1.8	
						21	1.9	

Country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	g ai/tree	kg ai/hL	water, L/tree	no.			
Thailand, 2006	EC	1.7	0.15	11	3	0 7 14 21 28	3.9 3.7 2.9 3.7 3.5	Suvaparp, 2006, PRO-MGS-03
Thailand, 2007	EC	1.7	0.15	11	3	0 7 14 21 28	2.9 2.0 2.0 1.9 1.7	Suvaparp, 2007, PRO-MGS-04

Cabbages

Twenty supervised trials on cabbages were conducted using an EC formulation containing 500 g/L profenofos. Two trials were conducted in Italy using an EC formulation containing 233 g/L profenofos and two trials in Canada using an EC formulation containing 720 g/L.

Table 30 Profenofos residues in cabbages from supervised trials in Australia, Canada, Germany, Italy, South Africa and Switzerland

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]		Ref
	Form	kg ai/ ha	kg ai/hL	water, L/ha	no.		profenofos	Total profenofos[c]	
Australia, 1978 (Bullhead)	EC	0.5		900-1000	6	Pre-treat 1 5 8 11 15 22	0.89, 0.51, 0.54, 0.89, 0.80 5.2, 7.5, 5.5, 4.5, 7.6 2.1, 1.9, 1.6, 1.6, 1.5 0.40, 0.90, 0.54, 0.38, 0.41 0.16. 0.35, 0.38, 0.57, 0.25 0.08, 0.10, 0.23, 0.05, 0.15 < 0.01, < 0.01, < 0.01, < 0.01, 0.03		Bull, 1978, 78/4/665
Australia, 1978 (Bullhead)	EC	0.5		900-1000	6	Pre-treat 1 5 8 11 15 < 0.01,	0.99, 0.43 4.7, 4.3, 5.8 1.0, 1.3, 1.1 0.26, 0.44, 0.45 0.09, 0.16, 0.18 0.05, 0.01, 0.04		Bull, 1978, 78/4/665

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]		Ref
	Form	kg ai/ ha	kg ai/hL	water, L/ha	no .		profenofos	Total profenofos[c]	
						22	< 0.01, 0.02		
Canada, 1978 (Penn State Ballhead)	EC	0.48	0.06	800	3	1 7	0.50 0.52		Altenburger, 1979, RVA 104/79
Canada, 1979 (Penn State Ballhead)	EC	0.5	0.06	800	3	1 7 14 21	0.82 0.20 0.06 0.02	1.17 0.30 0.17 0.07	Altenburger, 1980, RVA 1151/79
Canada, 1977 (Penn State Ballhead)	EC	0.5	0.06		3	21	< 0.02		Altenburger, 1978, RVA 80/78
Germany, 1979 (Vertus)	EC	0.4		600	3	Pre-treat 0 7 10 14 21 28	0.05 c: 0.02 2.82 0.26 0.21 0.02 < 0.02 < 0.02		Altenburger, 1979, RVA 1066/79
Germany, 1981 (Vorbote)	EC	0.75 0.4 0.4		400	3	Pre-treat 0 11 14 21 28	1.5 c: 0.10 22.6 0.3 0.3 0.03 0.02	1.5 c: 0.08 25.6 1.1 1.0 0.2 0.04	Altenburger, 1981, RVA 1171/81
Germany, 1981 (Eisenkopf)	EC	1.5 0.8 0.8		600	3	Pre-treat 0 10 14 21 28	2.1 12.9 2.7 1.9 0.25 0.12	3.5 17.4 5.6 4.2 1.9 1.3	Altenburger, 1981, RVA 1173/81
Germany, 1981 (Allrot)	EC	0.75 0.4 0.4		400	3	Pre-treat 0 11 14 21 28	0.5 21 2 0.7 0.1 < 0.02	1.7 c: 0.07 33 2.5 2.3 0.7 0.16c:0.04	Altenburger, 1981, RVA 1175/81
Germany, 1981 (Allrot)	EC	0.75 0.4 0,4		600	3	Pre-treat 0 10 14 21 28	0.12 3.1 0.6 0.5 0.3 0.08	0.97 4.5 1.2 1.2 1.2 0.27	Altenburger, 1981, RVA 1176/81
Germany, 1981 (Marner Dauerwirsin	EC	1.5 0.8 0.8		400	3	Pre-treat 0 9	0.72 6.47 0.80		Bischoff, 1981, (CGA15324/

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]		Ref
	Form	kg ai/ ha	kg ai/hL	water, L/ha	no .		profenofos	Total profenofos[c]	
g)						13 20 27	1.01 0.25 0.17		0349)
Italy, 1985 (Hybrid)	EC	0.5	0.05	1000	1	0 7 14 21	0.66 0.21 0.13 0.04		Kühne, 1986, 1113/85
Italy, 1985 (Hybrid)	EC	0.5	0.05	1000	1	0 7 14 21	1.1 0.80 0.13 0.05		Kühne, 1986, 1112/85
South Africa, 1975 (Gloria Osena)	EC	0.5			3	0 1 2 4 8	2.0 0.30 0.18 0.08 < 0.02		Altenburger, 1976, RVA 352/76
South Africa, 1977 (Hercules)	EC	0.5			10	Pre-treat 0 4 8 18 25	0.24 0.63 c: 0.12 0.22 0.24 0.05 0.02 c: 0.04		Altenburger, 1978, RVA 91/78
South Africa, 1977 (Stonehead)	EC	0.5			2	Pre-treat 0 1 2 4 8 16	0.06 0.46 0.44 0.14 0.23 0.06 0.09		Altenburger, 1978, RVA 92/78
South Africa, 1978 (Hercules)	EC	0.5	0.25	200	3	Pre-treat 0 2 4 8 16	0.75 1.48 c: 0.13 0.72 1.16 0.13 < 0.02		Altenburger, 1979, RVA 117/79
South Africa, 1977 (Hercules)	EC	1.0			1	0 4 8 18 25	1.6 c: 0.10 2.1 0.13 0.13 0.03		Altenburger, 1978, RVA 89/78
South Africa, 1977 (Stonehead)	EC	1.0			1	0 1 2 4 8 16	0.72 0.61 0.42 0.54 0.04 0.04		Altenburger, 1978, RVA 90/78
South Africa, 1977 (Hercules)	EC	1.0	0.5	200	3	Pre-treat 0 2 4	0.63 c: 0.13 3.72 1.68 2.10		Altenburger, 1979, RVA 116/79

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]		Ref
	Form	kg ai/ ha	kg ai/hL	water, L/ha	no .		profenofos	Total profenofos[c]	
						8 16	0.62 < 0.02		
Switzerland, 1979 (Marner Frühknopf)	EC	0.75 0.4 0.4		600	3	Pre-treat 0 7 9 14 21 28	0.08 3.02 0.51 0.23 0.07 0.02 < 0.02		Altenburger, 1979, RVA 1063/79
Switzerland, 1979 (Marner Dauerwirsing)	EC	0.75 0.4 0.4		600	3	Pre-treat 0 5 9 14 21 28	0.08 1.71 1.40 0.25 < 0.02 < 0.02 < 0.02		Altenburger, 1979, RVA 1064/79
Switzerland, 1981 (Marner Dauerwirsing)	EC	0.75 0.4 0.4		600	3	Pre-treat 0 9 14 21 28	0.09 2.0 0.1 0.02 < 0.02 < 0.02		Altenburger, 1981, RVA 1174/81
Switzerland, 1981 (Idena)	EC	0.75 0.4 0.4		1000	3	Pre-treat 0 9 14 21 28	0.09 1.2 0.05 0.02 < 0.02 < 0.02		Altenburger, 1981, RVA 1177/81

Pre-treat: sample before last application

c: sample from control plot

total residue of profenofos equivalents determined as CGA55960

Watermelon

Six supervised field trials were conducted in the Philippines using either an EC formulation containing 500 g/L profenofos or an EC formulation containing 400 g/L profenofos and 40 g/L cypermethrin.

Water volume varied according to the growth stage of the crop at the time of applications. In all trials, the growth stage of the crop at last application was early stage of maturity.

Table 31 Profenofos residues in watermelon from supervised trials in Philippines

Country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Philippines, 1982 (Sugar Baby)	EC	0.28 × 3 0.35 × 1 0.49 × 3	0.05	555 694 972	7	13	< 0.02	Altenburger, 1983, RVA 1225/81
Philippines, 1982 (Sugar Baby)	EC	0.21 × 3 0.26 × 1 0.36 × 3	0.037	555 694 972	7	13	< 0.02	Altenburger, 1983, RVA 1224/81
Philippines, 1982 (Sugar Baby)	EC	0.42 × 3 0.52 × 1 0.73 × 3	0.075	555 694 972	7	13	< 0.02	Altenburger, 1983, RVA 1226/81
Philippines, 1982 (Sugar Baby)	EC	0.12 × 3 0.15 × 1 0.21 × 3	0.022	555 694 972	7	13	< 0.02	Altenburger, 1983, RVA 1227/81
Philippines, 1982 (Sugar Baby)	EC	0.24 × 3 0.31 × 1 0.43 × 3	0.014	555 694 972	7	13	< 0.02	Altenburger, 1983, RVA 1228/81
Philippines, 1982 (Sugar Baby)	EC	0.37 × 3 0.46 × 1 0.64 × 3	0.066	555 694 972	7	13	< 0.02	Altenburger, 1983, RVA 1229/81

Chilli peppers

Twelve supervised trials on chilli peppers were conducted using an EC formulation containing 500 g/L profenofos.

Table 32 Profenofos residues in chilli peppers from supervised trials in Indonesia, Malaysia and Spain

Country, year (variety)	Application					PHI Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Indonesia, 1985 (local)	EC	0.6-0.96	0.12	500-800	10	14 21	4.7, 6.0 1.5, 1.4	Altenburger, 1986, 1035/85
Indonesia, 1985 (local)	EC	0.4-0.64	0.08	500-800	10	14 21	1.1, 1.2 0.28, 0.17	Altenburger, 1986, 1034/85
Indonesia, 1983 (local)	EC	0.6, 0.75-	0.15	400,	9	7	4.5 c: 0.02	Altenburger, 1984,

Country, year (variety)	Application					PHI Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
		1.2		500-800			4.6	RVA 1017/83
Indonesia, 1983 (local)	EC	0.3, 0.38-0.6	0.075	400, 500-800	9	7	3.4 c: 0.02 3.2	Altenburger, 1984, RVA 1018/83
Malaysia, 1993 (local)	EC	0.52	0.05	1042	5	0 3 7 14 21 28	5.7 c: 0.05 4.5 0.53 0.21 0.05 0.08	Kühne-Thu, 1994, 1142/93 (unwashed)
						0 3 7 14 21 28	5.8 4.1 0.93 0.14 0.07 0.18	(washed)
Malaysia, 1993 (local)	EC	0.78	0.075	1042	5	0 3 7 14 21 28	4.2 c: 0.23 6.7 1.3 0.53 0.29 0.39 c: 0.08	Kühne-Thu, 1994, 1143/93 (unwashed)
						0 3 7 14 21 28	5.4 4.1 1.4 0.64 0.20 0.54	(washed)
Malaysia, 1991 (MC 4)	EC	0.64	0.075		5	0 3 7 14 21	5.4 c: 0.02 5.0 4.4 4.1 2.1	Neppel, 1992, 1125/91
Malaysia, 1991 (MC 4)	EC	1.2	0.15		5	0 3 7 14 21	9.4 c: 0.04 12 8.5 8.6 5.4	Heineman, 1992, 1126/91
Spain, 1984 (Sonar)	EC	0.83	0.075	1110	1	7 22	2.6 c: 0.05 0.54 c: 0.07	Altenburger, 1984, RVA 1041/84
Spain, 1984 (Glovi)	EC	1.1	0.075	1500	1	7 21	1.9 c: 0.05 1.4 c: 0.07	Altenburger, 1984, RVA 1043/84
Spain, 1984 (Gedeon)	EC	1.7	0.075	2220	1	7 22	2.1 c: 0.05 1.3 c: 0.07	Altenburger, 1984, RVA 1040/84
Spain, 1984 (P-728)	EC	1.1	0.075	1500	1	7 21	2.4 c: 0.05 1.3 c: 0.07	Altenburger, 1984, RVA 1042/84

c: sample from control plot

Tomatoes

Fifteen trials on tomatoes were conducted in Europe using either an EC formulation containing 500 g/L profenofos or an EC formulation containing 400 g/L. Thirty one trials in South Africa and ten trials in other countries were conducted using an EC formulation containing 500 g/L.

Table 33 Profenofos residues in tomatoes from supervised trials in Australia, Denmark, France, Indonesia, Israel, Italy, South Africa, Spain and Switzerland

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Australia, 1975 (Napoli)	EC	0.75		388	4	0 7 14	1.2 0.96 0.48	Bull, 1976
Australia, 1976 (Roma)	EC	0.75		700	4	Pre (-1) 1 4 7 14	0.20 0.72 0.97 0.22 0.44	
Australia, 1975	EC	0.75		388	4	0 7 14	1.2 0.96 0.48	Bull, 1978, 78/11/692
Australia, 1976	EC	0.5		700	4	Pre (-1) 1 4 7 14	0.16 0.37 0.39 0.12 0.03	
	EC	0.75		700	4	Pre (-1) 1 4 7 14	0.20 0.72 0.97 0.22 0.44	
Australia, 1978	EC	1.0		520	3	5	0.57	
Australia, 1977	EC	to run-off			12	1 3 7 14 21	0.55 0.44 0.21 0.11 0.05	
Denmark, 1982 (Ida)	EC	1.3	0.04	3000	1	2 3 5 7	0.13 0.07 0.09 0.11	Altenburger, 1985, RVA 1182/83
France, 1977 (Luca)	EC	to run-off	0.03		3	0 4 8 12 16 20	0.31 0.40 0.11 0.11 0.09 0.07	1977, LABO 21.77
France, 1978 (Saint-Pierre)	EC	0.4	0.04		4	0 8 14 21 28	0.34 0.08 0.05 0.03 < 0.02	Tournayre, 1978, 41/78

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Indonesia, 1985 (Ratna)	EC	0.80, 0.96, 1.1, 1.3	0.16	500, 600, 700, 800	8	1 7 15	1.6 0.48 < 0.02	Kühne, 1986, 1241/85
Indonesia, 1985 (Ratna)	EC	0.40, 0.48, 0.56, 0.64	0.08	500, 600, 700, 800	8	1 7 15	1.3 0.41 0.04	Kühne, 1986, 1240/85
Israel	EC	1.0		300	5	14 30	0.55, 0.43, 0.83, 0.28 < 0.05, < 0.05, < 0.05, 0.12	CGA15324/ 0750
Italy, 1977 (Ventura)	EC	to run-off	0.05		1	11	0.80	Altenburger, 1978, RVA 77/78
Italy, 1982	EC	0.1	0.01	1000	1	19	0.10 u: 0.07	Altenburger, 1984, RVA 1211/82
South Africa, 1979 (Heinz)	EC	0.5	0.05	1000	4	Pre-treat 0 2 4 8 16 32	0.60 3.2 2.3 2.4 0.56 0.40 0.03	Altenburger, 1980, RVA 1017/79
South Africa, 1979 (Heinz)	EC	0.5	0.25	200	4	Pre-treat 0 2 4 8 16 32	1.28 2.0 2.4 1.8 0.53 0.38 0.04	Altenburger, 1980, RVA 1027/79
South Africa, 1985 (Rodate)	EC	0.5	0.16	320	3	Pre (-4) 0 2 4 7 14 21	0.14 0.20 0.18 0.13 0.22 0.18 0.09	Kühne, 1986, 1233/85
South Africa, 1985 (Rodate)	EC	0.75	0.23	320	3	Pre (-4) 0 2 4 7 14 21	0.26 0.15 0.51 0.23 0.39 0.31 0.12	Kühne, 1986, 1234/85
South Africa, 1987 (Floridate)	EC	0.38	0.063	600	4	Pre-treat 0 1 2 3 4 5	0.05 0.33 c: 0.03 0.21 0.15 0.39 0.20 0.17	Kühne, 1988, 1239/86

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
						7 10	0.10 0.10	
South Africa, 1987 (Floridate)	EC	0.38	0.063	600	4	Pre-treat 0 1 2 3 4 5 7 10	0.05 0.38 c: 0.03 0.40 0.04 0.29 0.17 0.44 0.28 0.39	Kühne, 1988, 1240/86
South Africa, 1975	EC	0.75	0.15		6	11	0.36	Altenburger, 1975, RVA 298/75
					7	10	0.12	
South Africa, 1977 (Roodeplaat)	EC	0.5	0.17		1	0 2 4 8 16 32	0.13 0.11 0.17 0.07 0.05 < 0.02 c: 0.04	Altenburger, 1977, RVA 65/77
South Africa, 1977 (Roodeplaat)	EC	1.0	0.33		1	0 2 4 8 16 32	0.18 0.24 0.18 0.17 0.05 < 0.02 c: 0.04	Altenburger, 1977, RVA 66/77
South Africa, 1977 (Letaba)	EC	1.5			12	0 2 4 8 16 32	4.3 c: 0.02 2.1 2.2 2.4 2.1 1.2	Altenburger, 1978, RVA 76/78
South Africa, 1977 (Letaba)	EC	0.75			12	0 2 4 8 16 32	0.74 0.94 0.81 0.48 0.57 0.39	Altenburger, 1978, RVA 88/78
South Africa, 1978 (Heinz)	EC	0.75	0.05		4	Pre (-1) 0 2 4 8 16	0.18 c: 0.11 1.3 2.0 1.9 1.8 0.95	Altenburger, 1978, RVA 120/78
South Africa, 1978 (Heinz)	EC	1	0.075		4	Pre (-1) 0 2 4 8 16	2.5 4.1 4.6 4.7 4.0 3.3	Altenburger, 1978, RVA 121/78

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
South Africa, 1979 (Mooketsi)	EC	1.3	0.1	1250	1	0 2 4 8 16 33	0.30 c: 0.11 0.62 0.40 0.12 0.04 0.03	Altenburger, 1980, RVA 1008/79
South Africa, 1979 (Heinz)	EC	1.0	0.1	1000	1	0 2 4 8 16 32	3.2 2.4 1.3 0.50 0.30 0.03	Altenburger, 1980, RVA 1009/79
South Africa, 1979 (Mooketsi)	EC	0.63	0.05	1250	1	0 2 4 8 16 33	0.40 c: 0.11 0.17 0.15 0.10 0.07 0.05	Altenburger, 1980, RVA 1010/79
South Africa, 1979 (Heinz)	EC	0.5	0.05	1000	1	0 2 4 8 16 32	1.6 1.0 0.58 0.62 0.05 0.06	Altenburger, 1980, RVA 1011/79
South Africa, 1979 (Mooketsi)	EC	0.31	0.025	1250	1	0 2 4 8 16 33	0.19 c: 0.11 0.09 0.07 0.09 0.02 0.12	Altenburger, 1980, RVA 1012/79
South Africa, 1979 (Heinz)	EC	0.25	0.025	1000	1	0 2 4 8 16 32	0.52 0.22 0.27 0.19 0.05 0.03	Altenburger, 1980, RVA 1013/79
South Africa, 1979 (Mooketsi)	EC	0.7 × 1 1.0 × 2 1.3 × 3	0.1	700 1000 1250	6	Pre-treat 0 2 4 8 16 33	0.28 1.8 c: 0.11 1,1 0.82 0.30 0.13 0.07	Altenburger, 1980, RVA 1014/79
South Africa, 1979 (Heinz)	EC	1.0	0.1	1000	4	Pre-treat 0 2 4 8 16 32	1.8 8.6 4.7 4.2 3.0 0.52 0.07	Altenburger, 1980, RVA 1015/79

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
South Africa, 1979 (Mooketsi)	EC	0.35 × 1 0.5 × 2 0.63 × 3	0.05	700 1000 1250	6	Pre-treat 0 2 4 8 16 33	0.24 0.74 c: 0.11 0.52 0.42 0.38 0.11 0.04	Altenburger, 1980, RVA 1016/79
South Africa, 1979 (Mooketsi)	EC	0.18 × 1 0.25 × 2 0.31 × 3	0.025	700 1000 1250	6	Pre-treat 0 2 4 8 16 33	0.10 0.25 c: 0.11 0.21 0.12 0.08 0.04 < 0.02	Altenburger, 1980, RVA 1018/79
South Africa, 1979 (Heinz)	EC	0.25	0.025	1000	4	Pre-treat 0 2 4 8 16 32	0.71 0.88 0.40 0.35 0.38 0.13 0.02	Altenburger, 1980, RVA 1019/79
South Africa, 1979 (Mooketsi)	EC	1.25	0.5	250	1	0 2 4 8 16 33	0.66 c: 0.11 0.39 0.29 0.25 0.09 0.04	Altenburger, 1980, RVA 1020/79
South Africa, 1979 (Heinz)	EC	1.0	0.5	200	1	0 2 4 8 16 32	1.14 0.76 0.40 0.22 0.03 0.04	Altenburger, 1980, RVA 1021/79
South Africa, 1979 (Mooketsi)	EC	0.63	0.25	250	1	0 2 4 8 16 33	0.64 c: 0.11 0.24 0.17 0.08 0.08 0.02	Altenburger, 1980, RVA 1022/79
South Africa, 1979 (Heinz)	EC	0.5	0.25	200	1	0 2 4 8 16 32	0.94 0.74 0.42 0.26 0.04 < 0.02	Altenburger, 1980, RVA 1023/79
South Africa, 1979 (Mooketsi)	EC	0.7 × 1 1.0 × 2 1.3 × 3	0.5	140 200 250	6	Pre-treat 0 2 4 8 16	0.35 0.76 c: 0.11 0.54 0.55 0.11 0.07	Altenburger, 1980, RVA 1024/79

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
						33	< 0.02	
South Africa, 1979 (Heinz)	EC	1.0	0.5	200	4	Pre-treat 0 2 4 8 16 32	1.6 2.8 2.0 1.8 1.1 0.51 0.03	Altenburger, 1980, RVA 1025/79
South Africa, 1979 (Mooketsi)	EC	0.35 × 1 0.5 × 2 0.63 × 3	0.25	140 200 250	6	Pre-treat 0 2 4 8 16 33	0.18 0.56 c: 0.11 0.18 0.23 0.06 < 0.02 < 0.02	Altenburger, 1980, RVA 1026/79
Spain, 1977 (Muchamiel)	EC	0.75	0.15		3	15	0.07 c: 0.03	Altenburger, 1978, RVA 30/78
Spain, 1977 (C-35)	EC	0.75	0.1		3	12	0.16	Altenburger, 1978, RVA 31/78
Switzerland, 1977 (Montfavet H 63)	EC	to run-off	0.03		3	0 5 8 12 16 20 26	0.96 0.56 0.39 0.19 0.12 0.06 0.06	Altenburger, 1977, RVA 69/77
Switzerland, 1977 (Lonay (VD) Switzerland)	EC	to run-off	0.03		3	0 4 8 11 15 21	0.80 0.38 0.36 0.30 0.09 0.05	Altenburger, 1977, RVA 70/77
Switzerland, 1974 (Montfavet H 6315)	EC	to run-off	0.04		1	0 2 7 14 21 28	1.1 0.84 0.53 0.34 0.23 0.21	Blass, 1974, RVA 176/74
Switzerland, 1975 (Montfavet M 63/5)	EC	to run-off	0.04		1	0 7 14 21 28	1.95 0.85 0.39 0.25 0.19	Altenburger, 1976, RVA 244/76
Switzerland, 1978 (Montfavet H 63-5 F 1)	EC	0.6	0.06	1000	3	0 3 7 14 21 28	1.6 0.82 0.73 0.37 0.15 0.11	Altenburger, 1979, RVA 53/79

Country, year (variety)	Application					PHI Days[a]	Residues, mg/kg[b]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Switzerland, 1978 (Momtfavet H 63-5 F 1)	EC	0.4	0.04	1000	4	0 7 11 16 21 28	0.51 0.15 0.10 0.10 0.07 0.02	Altenburger, 1979, RVA 57/79
Switzerland, 1978 (Montfavet H 63-5 F 1)	EC	0.6	0.06	1000	3	0 3 7 14 21 28	1.6 0.54 0.51 0.38 0.22 0.08	Altenburger, 1979, RVA 52/79
Switzerland, 1979 (Montfavet H 63-4)	EC	0.4	0.04	1000	3	0 4 7 14 21 28	1.1 0.52 0.68 0.44 0.18 0.44	Altenburger, 1979, RVA 1030/79

[a] Pre-treat: sample before last application

[b] c: sample from control plot

Soya bean

A total of nineteen supervised trials on soya bean were conducted using an EC formulation containing 500 g/L profenofos. Sixteen trials were conducted in South America and three trials in Indonesia.

Table 34 Profenofos residues in soya bean from supervised trials in Brazil, Indonesia and Mexico

country, year (variety)	Application					PHI Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Brazil, 1983 (Vicoja)	EC	0.2	0.13	150	1	20	< 0.02	Altenburger, 1983, RVA 1110/83
		0.4	0.27	150	1	20	< 0.02	
Brazil, 1983 (Parana)	EC	0.3	0.2	150	1	20	< 0.02	Altenburger, 1983, RVA 1111/83
		0.6	0.4	150	1	20	< 0.02	
Brazil, 2000 (FT 2002)	EC	0.075			2	21 35	≤ 0.02, < 0.02 < 0.02, < 0.02	Riberio, 2000, QO-18/21
		0.15			2	21 35	0.05, 0.09 0.06, 0.03	
Brazil, 2000 (JAC 8.2)	EC	0.075			2	21 35	0.04, 0.04 c: 0.03 0.02, 0.03	Riberio, 2000, QO-17/21
		0.15			2	21 35	0.07, 0.09 c: 0.03 0.06, 0.04 c: 0.02	
Brazil, 2001 (Conquista)	EC	0.075			2	21 35	< 0.02 < 0.02	Trevizan, 2002, M00094-CVG
		0.15			2	21 35	< 0.02 < 0.02	

country, year (variety)	Application					PHI Days	Residues, mg/kg [a]	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Brazil, 2001 (Spring)	EC	0.075			2	20 35	< 0.02 < 0.02	Trevizan, 2002, M00094-JJB
		0.15			2	20 35	< 0.02 < 0.02	
Brazil, 1976 (Santa Rosa)	EC	0.25			2	76	< 0.02 (oil: < 0.04)	Altenburger, 1977, RVA 35/77
Brazil, 1976 (Santa Rosa)	EC	0.5			2	76	< 0.02 (oil: < 0.04)	Altenburger, 1977, RVA 36/77
Brazil, 1998 (RS-10)	EC	0.075			2	60	< 0.02, < 0.02	Roncato and Marconi, 1998, IR 054/99
		0.15			2	60	< 0.02, < 0.02	
Brazil, 1998 (Abyara)	EC	0.075		600	2	30	< 0.02, < 0.02	Roncato and Marconi, 1998, IR 053/99
		0.15		600	2	30	< 0.02, < 0.02	
Indonesia, 1985 (Lokon)	EC	0.20, 0.24, 0.28, 0.32, 0.36	0.04	500, 600, 700, 800, 900	10	10	< 0.02 (hull: 0.98)	Kühne-Thu, 1987, 1002/86
Indonesia, 1985 (Lokon)	EC	0.40, 0.48, 0.56, 0.64, 0.72	0.08	500, 600, 700, 800, 900	10	10	< 0.02 (hull: 2.48)	Kühne-Thu, 1988, 1003/86
Indonesia, 1985 (Lokon)	EC	0.80, 0.96, 1.1, 1.3 1.5	0.16	500, 600, 700, 800, 900	10	10	0.02 (hull: 5.7)	Kühne-Thu, 1988, 1004/86
Mexico, 1985 (Davis)	EC	0.75	0.13	571	2	21 28 42	< 0.02 < 0.02 < 0.02	Kühne, 1986, 1007/85
Mexico, 1985 (Davis)	EC	0.75	0.13	571	2	21 28 42	< 0.02 < 0.02 < 0.02	Kühne, 1986, 1008/85
Mexico, 1985 (Davis)	EC	0.75	0.13	571	2	21 28 42	< 0.02 < 0.02 < 0.02	Kühne, 1986, 1009/85
Mexico, 1985 (Davis)	EC	0.75	0.13	571	2	21 28 42	< 0.02 < 0.02 < 0.02	Kühne, 1986, 1010/85
Mexico, 1985 (Davis)	EC	1.5	0.27	571	2	21 28 42	< 0.02 < 0.02 < 0.02	Kühne, 1986, 1011/85
Mexico, 1985 (Davis)	EC	1.5	0.27	571	2	21 28 42	< 0.02 < 0.02 < 0.02	Kühne, 1986, 1012/85

[a] c: sample from control (untreated) plot

Cotton seed

A total of twenty two supervised trials on cotton were conducted using either an EC formulation containing 500 g/L profenofos or an EC formulation containing 370 g/L. A trial in Australia was conducted using a concentrate for ultra-low volume application containing 250 g/L.

Table 35 Profenofos residues in cotton seed from supervised trials in Australia, Brazil and USA

Country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Australia, 1977	EC	1.0		150	7	Pre (-1) 1 3 10 14 28	0.85, 1.2, 1.0, 1.5 2.3, 3.2, 1.8, 1.4 2.0, 2.2, 2.4, 2.0 1.2, 1.6, 1.1, 0.90 1.2, 1.0, 1.6, 0.90 0.65, 0.70, 0.55, 0.70	Crapp and Bull, 1977, 77/7/630
Australia, 1978 (Deltapine)	EC	0.75-1.0[a]		18	9	11 18 25 33 39	0.04, 0.05 0.03, 0.06 0.02, 0.03 0.04, 0.05 0.03, 0.02	Crapp and Bull, 1978, 78/6/673
	EC	0.75-1.0[a]		30	11	Pre (-1) 1 8 14 21 28 36 44	0.17, 0.20 0.77, 0.78 0.75, 0.79 0.74, 0.78 0.21, 0.25 0.05, 0.14 0.06, 0.05 0.03, 0.06	
	ULV	0.75-1.0[a]		Nil	11	Pre (-1) 1 8 14 21 28 36 44	0.21, 0.27 0.21, 0.17 0.97, 0.86 0.08, 0.09 0.08, 0.06 0.04, 0.03 0.04, 0.02 0.05, 0.03	
Australia, 1975	EC	0.75		125	11	Pre (-1) 1 3 9	0.06, 0.04 0.20, 0.30, 0.10 0.30, 0.18 1.1, 0.90, 0.80	Bull and Crapp, 1976, 76/12/604
Brazil, 1981	EC	0.75	1.25	60	3	15	< 0.02	Altenburger, 1982, RVA 1139/81
Brazil, 1973 (IAC-13)	EC	1	0.72		3	27	< 0.03, < 0.03, < 0.03	Blass, 1975 RVA 232/75
Brazil, 1981 (IAC-17)	EC	0.40			1	46	< 0.02	Altenburger, 1981, RVA 1164/81

Country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
Brazil, 1981 (IAC-17)	EC	0.60			2	32	< 0.02	Altenburger, 1981, RVA 1165/81
Brazil, 1981 (IAC-17)	ULV	0.38		2.5	3	15	< 0.02	Altenburger, 1981, RVA 1169/81
USA, 1974	EC	0.56 1.1		47	5	11 11	< 0.05 0.07	Kahrs, 1975, AG-A 3513
USA, 1975 (McNair 511)	EC	1.1		50	6	7 14 21 28	0.09, < 0.10 ≤ 0.10, < 0.10 < 0.10, < 0.10 < 0.10, < 0.10	Kahrs, 1976, AG-A 3908-A
		2.2		50	6	7 14 21 28	0.12, < 0.10 < 0.10, < 0.10 < 0.10, < 0.10 < 0.10, < 0.10	
USA, 1975 (Stone 213)	EC	1.1		133	6	0 7 15 21	0.32, 1.3 0.12, 0.32 0.07 0.25, < 0.05	Kahrs, 1976, AG-A 3928-A
		2.2		133	6	0 7 15 21	0.55, 1.1 0.17, 0.44 0.07 0.06, 0.35	
USA, 1975 (Stone 213)	EC	1.1		133	6	15	0.60	Kahrs, 1976, AG-A 3928 II
		2.2		133	6	15	0.90	
USA, 1975 (Coker 201)	EC	1.1		227	6	15 22 34 74	≤ 0.05, < 0.05 < 0.05, < 0.05 < 0.05, < 0.05 < 0.05, < 0.05	Kahrs, 1976, AG-A 3945
		2.2		227	6	15 22 34 74	< 0.05, < 0.05 < 0.05, < 0.05 < 0.05, < 0.05 < 0.05, < 0.05	
USA, 1975 (A. SJ-2)	EC	1.1		142	1	0 7 14 21 30	1.4, 1.3 0.81, 0.71 0.95, 0.93 0.43, 0.42 0.20, 0.22	Kahrs, 1976, AG-A 3963
		2.2		142	1	0 7 14 21 30	2.8, 3.1 3.3, 2.2 2.8, 1.8 2.4, 1.9 2.0, 1.9	
USA, 1975 (A. SJ-2)	EC	1.1		142	6	14	0.60	Kahrs, 1976, AG-A 3963 II
		2.2		142	6	14	1.1	
USA, 1975 (DPL-61)	EC	1.1		95	6	0 7 14 21	1.2, 1.3 0.81, 1.3 0.68, 0.92 1.0[b], 1.2[b]	Kahrs, 1976, AG-A 3972

Country, year (variety)	Application					PHI Days	Residues, mg/kg	Ref
	Form	kg ai/ha	kg ai/hL	water, L/ha	no.			
USA, 1976 (Stone 213)	EC	1.1		114	6	15	0.35	Kahrs, 1976, AG-A 4421 II
		2.2		114	6	15	0.62	
USA, 1976	EC	1.1		133	10	14	< 0.05	Kahrs, 1977, AG-A 4510
		2.2		133	10	14	1.5 t [c]: 3.4	
USA, 1975 (Stone 7A)	EC	1.1		95 246	6	0 7 14 40	2.6, 1.2 0.83 0.24 0.34	Kahrs, 1976, AG-A 3826
		2.2		95 246	6	0 7 14 40	2.2, 3.4 1.0 0.58 0.52	
USA, 1987 (S-825)	EC	1.1		93	6	14	1.2	Cheung, 1990, AG-A 10735, 01-02
USA, 1974	EC	0.56		64	7	23	0.09, < 0.05	Kahrs, 1975, AG-A 3596 A
USA, 1975	EC	1.1		161	12	38	< 0.05	Kahrs, 1975, AG-A 3876
		2.2		161	12	38	< 0.05	

[a] Used for the last 3 applications.

[b] Residue value is corrected for procedural recoveries.

[c] t: total residues determined as CGA 55960

Table 36 Profenofos residues in feedstuff from cotton seed from supervised trials in Brazil, Colombia, Egypt, Turkey, Paraguay, Spain, South Africa and USA

country, year (country)	Application					PHI Days	Crop part	Residues, mg/kg		Ref.
	Form	kg ai /ha	kg ai /hL	water, L/ha	no.			Profenofos [a] (PF [b])	Total profenofos [c]	
Brazil, 1975 (IAC-RM 3)	EC	0.75			4	54	Crude oil Extracted seeds	0.13 c: 0.04 < 0.02		Altenburger, 1975, RVA 49/77
Colombia, 1995 (HS-46)	EC	0.60	0.64	94	3	36	Hulls Dehulled seed	< 0.05, < 0.05, < 0.05 < 0.05, < 0.05, < 0.05		Kühne-Thu, 1996, 1159/94
Colombia, 1995 (HS-46)	EC	0.60	0.64	94	3	52	Hulls Dehulled seed	< 0.05, < 0.05, < 0.05 < 0.05, < 0.05, < 0.05		Kühne-Thu, 1996, 1158/94
Egypt, 1974	EC	1			5	35	Crude oil Extracted seeds	0.57 c: 0.90 < 0.02		Altenburger, 1976, RVA 43/77
Paraguay, 1995, (Saenz Pena)	EC	0.60	0.39 0.38	155 157	2	14	Hulls Dehulled seed	< 0.05, < 0.05 < 0.05, < 0.05		Kühne-Thu, 1996, 1046/95

country, year (country)	Application					PHI Days	Crop part	Residues, mg/kg		Ref.
	Form	kg ai /ha	kg ai /hL	water, L/ha	no.			Profenofos [a] (PF [b])	Total profenofos[c]	
Spain, 1975 (Coker 310)	EC	0.75			3	73	Crude oil Extracted seeds	0.05 < 0.02		Altenburger, 1977, RVA 50/77
South Africa, 1976 (Delta pine)	EC	0.5			12	29	Crude oil Extracted seeds	0.22 < 0.02		Altenburger, 1977, RVA 48/77
South Africa, 1976 (Delta pine)	EC	1			12	29	Crude oil Extracted seeds	0.26 < 0.02		Altenburger, 1977, RVA 47/77
South Africa, 1976 (Albar)	EC	1			12	38	Crude oil Extracted seeds	0.16 < 0.03		Altenburger, 1978, RVA 116/78
South Africa, 1977 (Delta pine)	EC	0.75	0.38 x 9 0.35 x 4		13	29	Crude oil Extracted cake	0.10 < 0.03		Altenburger, 1978, RVA 45/78
South Africa, 1995 (Sicala)	EC	0.60		150	4	102	Hulls Kernels	< 0.05, < 0.05 < 0.05, < 0.05		Kühne-Thu, 1997, 1141/95
South Africa, 1995 (Sicala)	EC	1.2		150	4	102	Hulls Kernels	< 0.05, < 0.05 < 0.05, < 0.05		Kühne-Thu, 1997, 1142/95
Turkey, 1995 (Cukurova-1518)	EC	0.90	0.75 0.69	120 130	2	21	Hulls Dehulled seed	< 0.05, 0.06, < 0.05, 0.05 < 0.05, < 0.05, < 0.05, < 0.05		Kühne-Thu, 1996, 1070/95
USA, 1975 (Stone 213)	EC	1.1		133	6	15	Gin trash	24		Kahrs, 1976, AG-A 3928-A
		2.2		133	6	15	Gin trash	56		
USA, 1975 (Stone 213)	EC	1.1		133	6	15	Seed Hulls Meal Soapstock	0.60 0.65 (1.1) 0.90 (1.5) < 0.05	1.1 2.0 0.76 4.8	Kahrs, 1976, AG-A 3928 II
		2.2		133	6	15	Seed Hulls Meal Soapstock	0.90 1.9 (2.1) 0.16 (0.18) < 0.05	1.6 3.3 1.3 12	
USA, 1975 (A. SJ-2)	EC	1.1		142	1	14	Gin trash	53		Kahrs, 1976, AG-A 3963
		2.2		142	1	14	Gin trash	176		

country, year (country)	Application					PHI Days	Crop part	Residues, mg/kg		Ref.
	Form	kg ai /ha	kg ai /hL	water, L/ha	no.			Profenofos [a] (PF [b])	Total profenofos[c]	
USA, 1975 (SJ-2)	EC	1.1		142	6	14	Seed	0.60	1.3	Kahrs, 1976, AG-A 3963 II
							Hulls	0.54 (0.90)	1.8	
							Meal	0.07 (0.12)	0.77	
							Soapstock	< 0.05	3.1	
		2.2		142	6	14	Seed	1.1	2.3	
							Hulls	1.7 (1.5)	4.7	
							Meal	0.39 (0.35)	2.6	
							Soapstock	< 0.05	23	
USA, 1987 (S-825)	EC	1.1		93	6	14	Seed	-	4.4, 4.0	Cheung, 1990 AG-A 10735, 01-02
							Seed (Whole)	1.2	3.2	
							Hulls	0.15	2.3	
							Kernels	< 0.05	0.41	
							Meal	< 0.05	0.89	

[a] c: sample from control plots

[b] PF: processing factor in brackets

[c] Total residues of profenofos equivalent determined as CGA55960

FATE OF RESIDUES IN STORAGE AND PROCESSING

In Processing

The Meeting received information on the fate of profenofos residues during the processing of cotton seed for oil. Also information was provided on hydrolysis studies of profenofos to assist the identification of the nature of the residue during processing.

.In two supervised trials on soybeans conducted in Brazil (Altenburger, 1977, RVA 35/77) the residues were also analysed in oil. As the residues in unprocessed seeds as well as oil were below the LOQ, processing factor could not be estimated.

High temperature hydrolysis studies simulating standard food processing conditions have not been conducted on profenofos. The hydrolysis of [U-^{14}C -phenyl]-profenofos over the pH range 5 – 9 at 25°C was studied (Das, 1990; CGA15324/1257). It may be supposed that some hydrolysis will occur during processing at neutral and alkaline pH. Recoveries of total radioactivity ranged from 96% to 104%. Most of the profenofos was hydrolysed to CGA55960 at the pH range of 5 – 9. The hydrolytic stability of profenofos decreased with the increase of pH.

Table 37 Hydrolysis of [^{14}C]profenofos at 25 °C

Profenofos concentration	Hydrolysis conditions	% remaining of profenofos	% concentration of CGA55960
11.6 mg/L	pH 5.0 25 °C 24 hours	97.7	< 0.1
13.1 mg/L	pH 7.0 25 °C 24 hours	95.4	3.0
12.3 mg/L	pH 9.0 25 °C 24 hours	12.7	83.7

In a trial in Australia, cotton were treated with profenofos at 1.0 kg ai/ha (9 applications) and samples harvested 8 weeks after the final application were used for processing to crude cotton seed oil.

In two trials in Australia, Crude oil from untreated cotton seed was fortified to give residue concentrations of 1.8 and 2.0 mg/kg. Processing was designed to simulate the commercial refining

process of alkali treatment, drying, bleaching and deodorizing. Residue data are summarized in Table 38.

In US supervised field trials, cotton seed were treated with profenofos either 4EC or 6E formulation. Six applications were made at rates of either 1.1 or 2.2 kg ai/ha. Cotton was harvested either 14 or 15 days after the final application. Processing was in accordance with commercial practice.

Table 38 Profenofos residues in cotton seed and oil from processing trials

Cotton seed country, year (country)	Application				PHI Days	Commodity	Residues, mg/kg		Ref.
	Form	kg ai /ha	water, L/ha	no.			Profenofos (PF [a])	Total profenofos [b]	
Australia, 1978	EC	1.0		9	56	Seed Crude oil	0.08 0.31 (3.9)		Moore and Bull, 1978, 78/6/674
						Crude oil Alkali refined oil Dried oil Bleached oil Deodorized oil	1.8 (fortified) 0.72 (0.40) 0.71 (0.39) 0.66 (0.37) 0.14 (0.08)		
Australia, 1977						Crude oil Alkali refined oil Bleached oil Deodorized oil	2.0 (fortified) 0.56 (0.28) 0.25 (0.13) 0.15 (0.08)		Moore and Bull, 1978, 77/6/625
USA, 1975 (A. SJ-2)	EC	1.1	142	6	14	Seed Crude oil Refined oil Bleached deodorized oil	0.60 1.6 (2.7) 0.73 (1.2) 0.20 (0.33)	2.3 3.4 1.6 0.4	Senzel, Ross and Clear, 1990, ABR-90024
		2.2	142	6	14	Seed Crude oil Refined oil Bleached deodorized oil	1.1 7.2 (6.5) 4.0 (3.6) 0.82 (0.75)	5.1 14 8.6 1.4	
USA, 1975 (Stone 213)	EC	1.1	133	6	15	Seed Crude oil Refined oil Bleached deodorized oil	0.60 1.1 (1.8) 0.11 (0.18) < 0.05 (< 0.08)	2.0 2.0 0.58 < 0.05	
		2.2	133	6	15	Seed Crude oil Refined oil Bleached deodorized oil	0.90 2.3 (2.6) 0.47 (0.52) < 0.05 (< 0.06)	2.9 4.0 1.3 < 0.05	
USA, 1975 (Stone 213)	EC	1.1	133	6	15	Seed Crude oil Refined oil Bleached deodorized oil	0.60 0.81 (1.4) 0.09 (0.15) < 0.05 (< 0.08)	1.1 1.0 0.24 < 0.05	Kahrs, 1976, AG-A 3928 II

Cotton seed country, year (country)	Application				PHI Days	Commodity	Residues, mg/kg		Ref.
	Form	kg ai /ha	water, L/ha	no.			Profenofos (PF [a])	Total profenofos [b]	
		2.2	133	6	15	Seed Crude oil Refined oil Bleached deodorized oil	0.90 1.7 (1.9) 0.39 ((0.43) < 0.05 (< 0.06)	1.6 2.1 0.60 < 0.05	
USA, 1975 (SJ-2)	EC	1.1	142	6	14	Seed Crude oil Refined oil Bleached deodorized oil	0.60 1.2 (2.0) 0.69 (1.2) 0.14 (0.23)	1.3 1.8 0.73 0.20	Kahrs, 1976, AG-A 3963 II
		2.2	142	6	14	Seed Crude oil Refined oil Bleached deodorized oil	1.1 5.3 (4.8) 3.8 (3.5) 0.72 (0.65)	2.3 7.4 4.0 0.79	
USA, 1976 (Stone 213)	EC	1.1	114	6	15	Seed Crude oil Refined oil	0.35 0.35 (1.0) < 0.05 (< 0.14)	0.94 0.59 < 0.05	Kahrs, 1977, AG-A 4421 II, III, IV
		2.2	114	6	15	Seed Crude oil Refined oil	0.62 0.83 (1.3) < 0.05 (< 0.08)	2.7 2.7 < 0.05	
USA, 1976 (Stone 213)	EC	1.1	133	10	14	Seed Crude oil Refined oil	< 0.05 1.5 0.08	0.05 2.1 0.11	Kahrs, 1977 AG-A 4510 I, II, III
		2.2	133	10	14	Seed Crude oil Refined oil	1.5 3.4 (2.3) 0.20 (0.13)	3.4 5.0 0.42	
USA, 1987 (S-825)	EC	1.1	93	6	14	Seed Seed (whole) Seed (delinted) Crude oil Refined oil	- 1.2 < 0.05 0.09 < 0.05	4.4, 4.0 3.2 0.71 0.23 < 0.05	Cheung, 1990 AG-A 10735, 01-02

PF = Processing factor

Total residues of profenofos equivalent determined as CGA55960

The median processing factors for the above trials are 2.6 for crude oil, 1.2 for refined oil, and 0.28 for refined bleached deodorized oil.

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

The Meeting received a lactating dairy cow feeding study and a laying hen feeding study, which provided information on likely residues resulting in animal commodities, milk and eggs from profenofos residues in the animal diet.

Lactating dairy cow

Groups of 3 lactating dairy cows were dosed once daily via feed with profenofos at 0.25, 0.75 and 2.5 ppm in the diet for 28 consecutive days. One cow was dosed with profenofos at 25 ppm for 28 days (Senzel, Ross and Clear, 1990, ABR-90022). Milk samples for residue analysis were collected from each cow at 0, 3, 5, 7, 10, 21 and 28 days. On days 14, 21 and 28, the animals were slaughtered for tissue collection. Tissues collected for analysis were liver, kidney, perirenal fat, omental fat, round steak, tenderloin and blood. Animals consumed approximately18 kg dry feed per day. Residue data for profenofos and total profenofos determined as CGA55960 are summarised in Table 39. Only samples collected on days 21 and 28 from the higher dose rate feedings were analysed because no residues were found in the shorter-interval and lower-rate samples.

Parent profenofos residues did not occur above LOQ in any tissue samples for any of the test doses (Table 39). Total profenofos residues (determined as CGA55960) did not occur above LOQ in fat tissues, round steak and loin for all dose rates. Total profenofos residues were present in liver, kidney, blood and milk at the 25 ppm feeding level and in the kidney at the 2.5 ppm feeding level.

Table 39 Residue in milk and tissues of lactating dairy cows dosed once daily with profenofos at the equivalent of 0.75, 2.5 and 25 ppm in the diet.

Substrate	Residues, mg/kg					
	Dosing, 0.75 ppm		Dosing, 2.5 ppm		Dosing, 25 ppm	
	profenofos	Total profenofos [a]	profenofos	Total profenofos [a]	profenofos	Total profenofos [a]
Round steak	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Loin	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Liver	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	0.07 (28 days)
Kidney	< 0.05	< 0.05	< 0.05	0.05 (14 days) 0.06 (21 days) < 0.05 (28 days)	< 0.05	0.53 (28 days)
Omental fat	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Perirenal fat	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Blood	< 0.02	< 0.05	< 0.02	< 0.05	< 0.02	0.10 (28 days)
Milk	< 0.01	< 0.02	< 0.01	< 0.02	< 0.01	0.02 (21 days) 0.02 (28 days)

[a] Residue determined as CGA55960 and expressed as profenofos equivalent

Laying hen

Three groups of 15 laying hens were fed rations treated with profenofos at 0.10, 0.30 and 1.0 ppm for 28 consecutive days. Samples of eggs for residue analysis were collected at 0, 1, 3, 7, 10, 14, 21 and 28 days. Tissues collected for analysis were liver, fat, meat of breast and thigh. Birds consumed average 104 g feed per day. Residue data for profenofos and total profenofos determined as CGA55960 are summarized in Table 40. Only samples collected on days 21 and 28 from the higher dose rate feeding were analysed because no residues were found in the shorter-interval and lower-rate samples.

Parent profenofos and total profenofos as CGA55960 residues were not present in the tissues.

Table 40 Residue in eggs and tissue of laying hens fed with profenofos at 0.30 and 1.0 ppm in the diet, for 21 and 28 consecutive days

Substrate	Residues, mg/kg			
	Dosing, 0.30 ppm		Dosing, 1.0 ppm	
	profenofos	Total profenofos [a]	profenofos	Total profenofos [a]
Breast	< 0.05	< 0.05	< 0.05	< 0.05
Thigh	< 0.05	< 0.05	< 0.05	< 0.05
Liver	< 0.05	< 0.05	< 0.05	< 0.05
Fat	< 0.05	< 0.05	< 0.05	< 0.05
Eggs	< 0.02	< 0.05	< 0.02	< 0.05

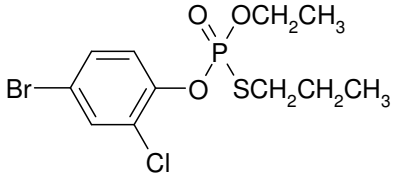
[a] Residue determined as CGA55960 and expressed as profenofos equivalents

APPRAISAL

Profenofos, an organophosphorus insecticide, was first evaluated by the JMPR in 1990 and has been reviewed for residue in 1992, 1994 and 1995. It was listed for periodic re-evaluation for residue evaluation at the 39th Session of the CCPR by the 2008 JMPR. The toxicology of profenofos was re-evaluated by the 2007 JMPR which estimated an ADI of 0–0.03 mg/kg bw and an ARfD of 1 mg/kg bw.

The Meeting received information on physical and chemical properties, animal and plant metabolism, environmental fate, analytical methods, storage stability, use pattern, supervised trials, processing and animal feeding studies.

O-(4-bromo-2-chlorophenyl) O-ethyl S-propyl phosphorothioate



In this appraisal, the following abbreviated names were used for metabolites.

CGA 55960	4-bromo-2-chlorophenol
BCPEE	4-bromo-2-chlorophenyl ethyl ether
BCPME	4-bromo-2-chlorophenyl methyl ether
THPME	2-thioethylenecarboxy-4-hydroxyphenyl methyl ether
MHPME	2-mercapto-4-hydroxyphenyl methyl ether

Animal metabolism

The Meeting received animal metabolism studies with profenofos in lactating goats and laying hens. [U-^{14}C-phenyl]profenofos was used in the metabolism studies.

When two lactating goats were orally dosed with [U-^{14}C-phenyl]-profenofos once daily for 4 consecutive days at 150 mg/animal/day, equivalent to 100 ppm in the feed, most of the administered radioactivity was excreted in the urine (59% and 79%) and faeces (1.7% and 1.2%). None of individual tissues or cumulative milk sample on day 4 contained more than 2% of the administered dose. Residue in milk reached a plateau by days 2–3. Residues of radioactivity were higher in kidney (2.5 mg/kg and 2.3 mg/kg profenofos equivalent) than in other tissues. Metabolite CGA 55960 and its

sulfate and glucuronide constituted 22, 40 and 28% of the TRR, respectively in kidney, with no parent profenofos. Parent profenofos was the major component in fat (44% TRR), and was also present in liver (10% TRR). CGA 55960 sulfate was the major component identified in muscle (56% TRR), kidney (40% TRR) and milk (85% TRR). In addition, the major metabolites in liver and kidney were free CGA 55960 (25%, 22% TRR) and its glucuronide (8%, and 28% TRR), respectively.

When two groups of five laying hens were orally dosed with [U-^{14}C-phenyl]profenofos once daily for 8 consecutive days at a dose equivalent to 1 and 10 ppm in the feed, most of the administered radioactivity was excreted in the excreta (93% and 89%). None of individual tissues or egg samples contained more than 1% of the administered dose. Highest TRR appeared in the kidney (0.12 mg/kg for the 1 ppm dose level and 1.3 mg/kg for 10 ppm dose level). Individual tissue TRR from 10 ppm dose level were approximately 10 times higher than those from 1 ppm dose group. CGA55960 was the major identified component in fat (77% and 89% TRR) and liver (75% and 71% TRR). Parent profenofos accounted for less than 5% TRR in each tissue and egg. For egg, CGA55960 sulfate was the main component of the residue: muscle (85% and 75% TRR), egg yolk (88% and 93% TRR), egg white (98% TRR).

Profenofos was rapidly metabolized following oral administration to animals. Once administered orally, profenofos underwent hydrolysis of the phosphate ester, and then formed either its sulfate or its glucuronide. TRR levels were higher in the kidney than in other tissues. Most of administered dose was rapidly excreted.

The metabolism of profenofos in the lactating goat and the laying hen was qualitatively similar to that described in the toxicology section of the 2007 Report of the JMPR.[7]

Plant metabolism

The Meeting received plant metabolism studies with profenofos in cotton, Brussels sprouts, lettuce and tomatoes. [U-^{14}C-phenyl]profenofos was used in the metabolism studies.

In a greenhouse cotton metabolism study, cotton plants were sprayed at a rate of 1.7 kg ai/ha once to simulate a multiple application of pesticide and maximize metabolites. Immediately after treatment the majority (91%) of the TRR was extractable with organic solvent. The extractable TRR decreased from 19.9mg/kg at day 0 to 1 and 0.55 mg/kg after 6 and 12 weeks. The parent profenofos (89%, 50% TRR) was the major component in the leaves and stems at 0 and 6 weeks after treatment, and then parent profenofos (13% TRR) and CGA 55960 (26% TRR) were identified in the leaves and stems at 12 weeks after treatment.

In a field cotton metabolism study in USA, cotton was sprayed over-the-top at a rate of 2.2 kg ai/ha three times at 2 week intervals. Mature samples of cotton were harvested 7 days after the final application. The TRR of profenofos equivalents was 8.3 mg/kg in the leaves, 0.4 mg/kg in the seeds and 0.2 mg/kg in the cotton fibre. The major compounds identified were parent profenofos (32% TRR) and CGA 55960 glucose conjugate (31% TRR) in mature leaves, and CGA 55960 glucose conjugate (15% TRR) in mature seeds.

In another field cotton metabolism study in USA, cotton was foliar sprayed 6 times at a rate of 2.2 kg ai/ha weekly. Mature samples of stalk, seed and lint were harvested at 61 and 83 days after the final application. Parent profenofos (29% TRR) and CGA 55960 glucosyl sulfate (31% TRR) were major components of the residue (TRR 14 mg/kg) in mature stalk. Mature samples of cotton seed contained parent profenofos (6.5% TRR) and CGA 55960 glucosyl sulfate (17% TRR) as the major part of residue (TRR 0.66 mg/kg).

In a Brussels sprouts metabolism study in Switzerland, Brussels sprouts received 3 foliar sprays at 2 week intervals at a rate equivalent to 1.1 kg ai/ha. In the leaves/stems sampled 21 days after the final treatment, CGA 55960 polysaccharide conjugate (30% TRR) and CGA 55960 monosaccharide conjugate (36% TRR) were major components of the residue (TRR 3.6 mg/kg).

[7] In: Pesticide Residues in Food—2007. Report of the JMPR 2007, FAO Plant Production and Protection Paper, 191, pp 210.

Profenofos was rapidly degraded following application to Brussels sprouts, with TRR of 0.3 mg/kg profenofos equivalents in the sprouts at maturity. Parent profenofos was present at 1.9% TRR in the leaves/stems but not found in the sprouts.

In a lettuce metabolism study, two leaves per lettuce plant were treated by smearing 1 mg of ethanol solution of [U-^{14}C-phenyl]profenofos evenly over each leaf surface. Lettuce leaves were sampled at 0, 7, 14 and 21 days after treatment. Parent profenofos was the major component (68–92% TRR) on lettuce leaves, and no metabolite exceeding 3% of the TRR was identified at 0, 7 and 14 days. Parent profenofos (61% TRR) and CGA 55960 (10%) were the major part of the residue on leaves at 21 days after treatment.

In a tomato metabolism study, tomato plants received three foliar applications at a rate of 0.72, 0.82 and 0.81 kg ai/ha, with a week intervals. Mature tomato fruits as well as tomato leaves were harvested just after the last application, and 4, 7 and 14 days. The TRR was 1.1 mg/kg in tomato fruits and 29 mg/kg in leaves at 14 days. About 42% of the TRR was washed off the tomatoes harvested just after treatment, and 6% TRR at 14 days by rinsing with methanol. The parent was the major component of residue amounting to 0.67 mg/kg (63% TRR) in tomato fruits at 14 days later. Other identified components of the residue in tomato fruits were CGA 55960, CGA 55960 disaccharide conjugate and polysaccharide. Although the parent was the major component of residue (72% TRR) just after application in tomato leaves, it decreased to 6% TRR at 14 days later. Metabolite CGA 55960 was the major residue component in the leaves (20% TRR) at 14 days.

Profenofos is slowly absorbed and metabolized. Profenofos was the major residue when harvested several weeks after the last application, and then profenofos underwent hydrolysis of phosphate ester to form CGA 55960 and its sugar conjugate.

Environmental fate in soil

The Meeting received information on aerobic soil metabolism and rotational crop study.

Aerobic soil metabolism studies were conducted using [U-^{14}C-phenyl]profenofos applied to various soils which were then incubated under aerobic conditions at 21 or 25 °C. Aerobic soil degradation rates were influenced by the nature of the soil, temperature, moisture status of the soil and dose applied. Under aerobic conditions, profenofos applied to soil was rapidly degraded. After 28–30 days, only small amounts (< 0.1–1.6%) of applied profenofos remained as the parent. CGA 55960, BCPEE, THPME, MHPME and BCPME were formed and then degraded during study. Unextracted radioactivity, 1.0% of the applied dose in sandy loam on day 0, increased steadily to 10% of the applied dose on day 270. These results indicate that profenofos is not persistent in soil. Under sterile conditions, CGA 55960 was formed as the only degradate, reaching a maximum level of 93% of the applied dose by 360 days.

In confined rotational crop study, mustard, radish and wheat were planted at 30, 60, 90, 180 and 365 days following the application of maximum seasonal use rate of [U-^{14}C-phenyl]profenofos. Profenofos as an 8E formulation was applied to bare ground at the maximum seasonal rate of 6.7 kg ai/ha. Crops were harvested at maturity, and wheat forage was also harvested at intermediate stage.

TRRs for all crops were 0.026–0.157 mg/kg at 30-day plant back interval. Residue levels were slightly lower at 60, 90 and 180-day intervals. Intact profenofos was positively identified only in the mature root of the 30-day radishes, albeit at very low levels (0.001 mg/kg). For all planting intervals, the majority of the residues in rotational crops were in the post-extraction solids and aqueous-soluble fraction. The aqueous soluble residues were characterized as a mixture of neutral, basic and acidic components. A total hydrolysis method indicated these components were CGA 55960 sugar conjugates.

Profenofos residues are not expected to occur in succeeding crops.

Methods of analysis

The Meeting received description and validation data for analytical methods for residues of parent profenofos in raw agricultural commodities, processed commodities, feed commodities, animal tissues, and milk and eggs. In most of the methods for determination of profenofos, homogenized samples were extracted with methanol or a mixture of methanol and water, and the extract was cleaned up with liquid-liquid partition followed by solid phase column chromatography using silica and florisil singly or in combination. The final residue may be determined by gas chromatography with NPD, FPD or ECD. LOQs were typically in the 0.01–0.05 mg/kg range.

Methods were provided also for residues of parent profenofos, CGA 55960, its sulfate and glucuronide determined as CGA 55960 in raw agricultural commodities and animal tissues, feed commodities, and milk and eggs. Homogenized samples were extracted with methanol or acetonitrile, and the extract was subjected to an acid and a base hydrolysis. The solutions were cleaned up with liquid–liquid partition followed by solid phase column chromatography using silica. The final residue may be determined by gas chromatography with ECD. LOQs were typically in the 0.02–0.05 mg/kg range.

Analytical recovery data were satisfactory for profenofos and total residues determined as CGA 55960 for numerous commodities.

DFG Method S19 (extended version) also demonstrated to be suitable for analysis of profenofos in plant material and foodstuffs of animal origin.

Stability of pesticide residues in stored analytical samples

Information was received on the freezer storage stability of profenofos residues in plant commodities, and of total residues of profenofos determined as CGA 55960 in plant and animal commodities.

Profenofos residues were stable in the following plant commodities for the intervals tested for 1–2 years: cotton seed, cotton seed hulls, cotton seed oil, soap stock and grapes.

Total residues of profenofos determined as CGA 55960 into animal tissues, milk and eggs were stable when stored under freezer storage conditions (approximately –20 °C) for 1 year.

Residue definition

The current residue definition of profenofos is parent profenofos for plant and animal commodities. Parent profenofos is the major component of the TRR in most crops until 2–3 weeks after application. In tomato fruits, profenofos represented 63% of the TRR at 14 days after the last application. Also in lettuce, profenofos (61% TRR) is the major residue component at 21 days after treatment, although CGA 55960 was identified 10% of TRR. No metabolite was found to be more than 10% of the TRR in lettuce leaves and tomato fruits. In Brussels sprouts, however, no parent profenofos was detected in sprouts at 21 days after the last application. Although CGA 55960 and CGA 55960 sugar conjugate were identified in sprouts, concentrations of the metabolites were below the LOQ level.

In cotton seed, parent profenofos and CGA 55960 glucosyl sulfate were identified as the major residue components at 83 days after the final application, although each TRRs were less than 20%. No other metabolites were present higher than 5% of the TRR. Methods of analysis are available for determination of parent profenofos and these metabolites in plants. However, concentrations of these metabolites are expected to be below the LOQ level.

The Meeting decided that parent profenofos is a suitable analyte for enforcement purposes and dietary risk assessment in plant commodities.

Profenofos is rapidly absorbed and eliminated after oral administration in farm animals and is only found in significant amount in goat kidney and hen kidney, liver and eggs. In the lactating goat study, the main components of residue were CGA 55960 sulfate in milk, kidney and muscle, parent profenofos in fat. In the laying hen study, the major residue components were CGA 55960 in fat and liver, CGA 55960 sulfate in muscle and eggs. Methods of analysis are available for determination of parent profenofos and these metabolites in animal tissues, milk and eggs. The metabolites determined

as CGA 55960 by hydrolysis procedure are expected to be detectable as the major compounds in animal tissues, milk and eggs. However, according to farm animal feeding studies, the parent and the metabolites are expected to be present below the LOQ.

The Meeting decided that parent profenofos is suitable analyte for enforcement purposes and dietary risk assessment in animal commodities.

Profenofos may be fat-soluble as it has log P_{ow} of 4.44 at 25 °C. In animal metabolism studies, the TRR in fat (0.07 mg/kg) was much lower than in kidney (2.3 mg/kg), liver (0.51 mg/kg) and milk (0.41 mg/kg). The study results indicated that the parent was rapidly decomposed to water-soluble metabolites, and those metabolites were excreted. Therefore, the Meeting decided the residues would not be fat-soluble.

The Meeting recommended the following as residue definitions for profenofos.

For plants and animals:

Definition of the residue (for compliance with the MRL and for estimation of dietary intake): *profenofos*.

Results of supervised residue trials on crops

The Meeting received supervised trial data for profenofos uses on tropical fruits (mango, mangosteen), cabbages, watermelon, fruiting vegetables (chilli peppers, tomatoes), soya beans and cotton seed. Residue data were also provided on cotton meal and hulls.

Labels (or translation of labels) were available from Australia, Brazil, Chile, Colombia, Indonesia, Malaysia, Philippines, South Africa and USA describing the registered uses of profenofos, and GAP information was also provided from Thailand.

Since no residue data were provided for sweet peppers and potato, the Meeting withdraws its previous recommendations for maximum residue levels for these crops.

Mango

In Thailand, profenofos may be applied to mango trees four times at a spray concentration of 0.075 kg ai/hL, with a 21 days PHI. In six Thai trials conducted in accordance with Thai GAP, profenofos residue in mango whole fruits, were: < 0.01, 0.05, 0.05, 0.06, 0.06 and 0.07 mg/kg. No data were available for residue in edible portion.

The Meeting estimated a maximum residue level, an STMR value and an HR value for profenofos in mango of 0.2, 0.06 and 0.07 mg/kg respectively.

Mangosteen

In Thailand, profenofos may be applied to mangosteen trees three times at a spay concentration of 0.15 kg ai/hL with a 21 days PHI. In four Thai trials conducted in accordance with Thai GAP, profenofos residue in mangosteen whole fruits were 1.9, 1.9, 2.3 and 3.7 mg/kg. No data were available for residue in edible portion.

The Meeting estimated a maximum residue level, an STMR value and an HR value for profenofos in mangosteen of 10, 2.1 and 3.7 mg/kg respectively.

Cabbages

In South Africa, profenofos may be applied to cabbages at a rate of 0.38–0.5 kg ai/ha with a 7 days PHI.

In a South African trial conducted in accordance with South African GAP, profenofos residues were < 0.02, 0.09, 0.13 mg/kg.

The Meeting agreed that insufficient data were available to estimate a maximum residue level for cabbages.

The Meeting withdraws its previous recommendation of 1 mg/kg for cabbages.

Watermelon

In the Philippines, profenofos may be applied to watermelon at a spray concentration of 0.1–0.15 kg ai/hL with a 7 days PHI. Six trials were conducted in the Philippines (0.014–0.075 kg ai/hL with a 13 day PHI) but the spray concentration and PHI did not correspond to Filipino GAP.

The Meeting was unable to estimate residue level as the residue trails conducted do not match the GAP.

Chilli peppers

In Indonesia, profenofos may be applied to chilli pepper crops at a spray concentration of 0.025–0.15 kg ai/hL with no required PHI.

In a Malaysian trial conducted in accordance with Indonesian GAP, profenofos residues on 0 days after the final application was 12 mg/kg. The Meeting agreed that the data matching GAP were insufficient to propose a maximum residue level for chilli peppers.

The Meeting withdraws its previous recommendation of 5 mg/kg for chilli peppers.

Since the Meeting withdraws a maximum residue level in chilli peppers, a maximum residue level in dried chilli peppers, which is estimated using a processing factor of dehydration of chilli peppers, is withdrawn.

Tomatoes

In South Africa, profenofos may be applied to tomato crops at a rate of 0.25–0.75 kg ai/ha with a 4 day PHI. In nine South African trials conducted in accordance with South African GAP, profenofos residues in rank order were 0.18, 0.39, 0.40, 0.81, 1.3, 1.8, 1.9, 4.2 and 4.7 mg/kg. The trials where the samples from control plots contained residues were disregarded.

In Indonesia, profenofos may be applied to tomato crops at a rate of 0.38–1.2 kg ai/ha with no required PHI. In two Indonesian trials conducted in accordance with Indonesian GAP, profenofos residues on 1 day after the final application were 1.3 and 1.6 mg/kg.

Based on the South African trials, the Meeting estimated a maximum residue level, an STMR value and an HR value for profenofos in tomatoes of 10, 1.3 and 4.7 mg/kg respectively.

The Meeting withdraw its previous recommendation of 2 mg/kg for tomato.

Soya beans

In Brazil, profenofos may be applied to soya bean crops at a rate of 0.08–0.1 kg ai/ha with a 21 days PHI. In three Brazilian trials conducted with conditions in line with Brazilian GAP, profenofos residues (ranked order, median underlined) were < 0.02 (3) mg/kg.

In the Philippines, profenofos may be applied at a rate of 0.5–0.75 kg ai/ha with a 7 days PHI. None was conducted in accordance with Filipino GAP.

The Meeting agreed that the data in accordance with GAP were insufficient to propose a maximum residue level for soya beans.

Cotton seed

In Australia, profenofos may be applied to cotton crops at a rate of 0.25–1.0 kg ai/ha, PHI 28 days. In tree Australian trials conducted in accordance with Australian GAP, profenofos residues were 0.04, 0.14 and 0.70 mg/kg.

In a Brazilian trial conducted in accordance with Brazilian GAP (0.25–0.5 kg ai/ha with a 15 day PHI), profenofos residues was < 0.02 mg/kg.

In the USA, profenofos may be applied 2–4 times at a rate of 0.14–0.86 kg ai/ha with a 14 day PHI. In 11 US trials conducted with condition in line with US GAP, profenofos residues in rank order were < 0.05 (2), < 0.10, 0.25, 0.34, 0.35, 0.60, 0.60, 0.92, 0.95 and 1.2 mg/kg.

Based on the US trials, the Meeting estimated a maximum residue level, an STMR value for profenofos in cotton seed of 3 and 0.35 mg/kg respectively.

The Meeting withdraws its previous recommendation of 2 mg/kg for cotton seed.

Animal feedstuffs

Cotton gin trash

In two US trials conducted in accordance with US GAP (0.86 kg ai/ha, PHI of 14 days), profenofos residues in cotton gin trash were 24 and 53 mg/kg respectively.

The Meeting was unable to estimate residue level as the data matching GAP were insufficient to propose a maximum residue level for cotton gin trash.

Fate of residues during processing

The Meeting received information on processing of cotton seed to crude oil and refined oil.

Processing factors were calculated for cotton seed (hulls, meal, crude oil and refined oil) and are shown in the Table below.

Mean processing factors and STMR-P for food and feed

Commodity	Processing factor	Median or best estimate	STMR-P mg/kg
Cotton seed			0.35
Hulls	0.90, 1.1, 1.5, 2.1	1.4	0.49
Meal	0.12, 0.18, 0.35, 1.5	0.54	0.19
Crude oil	1.0, 1.4, 1.5, 1.8, 1.9, 2.0, 2.3, 2.6, 2.7, 3.9, 4.8, 6.5	2.2	0.77
Refined oil	< 0.08, < 0.14, 0.13, 0.15, 0.18, 0.28, 0.40, 0.43, 0.52, 1.2, 1.2, 3.5, 3.6	0.40	0.14
Bleached deodorized oil	< 0.06, < 0.06, < 0.08, < 0.08, 0.08, 0.08, 0.23, 0.33, 0.65, 0.75,	0.08	0.03

Cotton seed oil must be refined to remove a naturally occurring toxin. Therefore the refined oil residues should be used to estimate an STMR for dietary intake.

The Meeting withdraws its previous recommendation of 0.05(*) mg/kg for cotton seed oil, edible.

Farm animal feeding studies

The Meeting received a lactating dairy cow feeding study and a laying hen feeding study, which provided information on likely residues resulting in animal commodities, milk and eggs from profenofos residues in the animal diet.

Lactating dairy cows

Groups of 3 lactating dairy cows were dosed once daily via feed with profenofos at 0.25, 0.75 and 2.5 ppm in the diet for 28 consecutive days. One cow was dosed with profenofos at 25 ppm for 28 days. Milk samples for residue analysis were collected from each cow at 0, 3, 5, 7, 10, 21 and 28 days and samples of liver, kidney, perirenal fat, omental fat, round steak, tenderloin and blood were collected on 14, 21 and 28 days.

Parent profenofos residues did not occur above LOQ in any tissue and milk samples for any of the test doses.

Laying hens

Three groups of 15 laying hens were fed rations treated with profenofos at 0.10, 0.30 and 1.0 ppm for 28 consecutive days. Samples of eggs for residue analysis were collected at 0, 1, 3, 7, 10, 14, 21 and 28 days and samples of liver, fat, breast and thigh were collected.

Parent profenofos residue was not present in the tissues and eggs.

Farm animal dietary burden

The Meeting estimated the dietary burden of profenofos in livestock on the basis of the diets listed in Annex 6 of the 2006 JMPR Report (OECD Feedstuffs Derived from Field Crops). Calculation from highest residue, STMR (some bulk commodities) and STMR-P values provides the levels in feed suitable for estimating MRLs, while calculation from STMR and STMR-P values for feed is suitable for estimating STMR values for animal commodities. The percentage dry matter is taken as 100% when the highest residue levels and STMRs are already expressed as dry weight.

Estimated maximum and mean dietary burdens of farm animals

Dietary burden calculations for beef cattle, dairy cattle, broilers and laying poultry are provided in Annex 6 of the 2006 Report of the JMPR. The calculations were made according to the livestock diets from US-Canada, EU and Australia in the OECD Table (Annex of the 2006 JMPR Report).

	Livestock dietary burden, profenofos, ppm of dry matter diet					
	US-Canada		EU		Australia	
	Max	mean	Max	Mean	Max	mean
Beef cattle	0.11a	0.11b	0.01	0.01	0.11	0.11
Dairy cattle	0.08	0.08c	0.01	0.01	0.05	0.05
Poultry – broiler	0.04d	0.04e	0.01	0.01	0.02	0.02
Poultry – layer	0.04	0.04	0.01	0.01	0.02	0.02

[a] Highest maximum beef or dairy cattle dietary burden suitable for MRL estimates for mammalian meat and milk

[b] Highest mean beef or dairy cattle dietary burden suitable for STMR estimates for mammalian meat

[c] Highest mean dairy cattle dietary burden suitable for STMR estimates for milk

[d] Highest maximum poultry dietary burden suitable for MRL estimates for poultry meat and eggs

[e] Highest mean poultry dietary burden suitable for STMR estimates for poultry meat and eggs

Animal commodity maximum residue levels

For MRL estimation, the residue in the animal commodities is profenofos.

In a feeding study, in which profenofos equivalent to 0.75 ppm in the diet was dosed to lactating cows for 28 consecutive days, no total profenofos residues were detected in tissues (< 0.05 mg/kg) and milk (< 0.01 mg/kg). Therefore no residues are to be expected at the maximum estimated dietary burden of 0.11 ppm feed for beef cattle and dairy cattle.

The Meeting estimated a maximum residue level of 0.05(*) mg/kg in mammalian meat and mammalian edible offal, and 0.01(*) mg/kg in milk. The Meeting confirmed its previous recommendations for mammalian meat and milk.

The mean estimated dietary burden for dairy cattle is 0.08 ppm. No profenofos residues (< 0.01 mg/kg) were found in any samples of milk at the 0.75 ppm feeding level. Therefore the Meeting estimated an STMR of 0 mg/kg in milk.

The mean estimated dietary burden for cattle is 0.11 ppm. In muscle, fat, kidney and liver, no profenofos residues (< 0.05 mg/kg) were detectable at the 0.75 ppm feeding level. The Meeting estimated STMRs and HRs of 0 mg/kg in meat, offal and fat.

In a feeding study, in which profenofos equivalent to 0.30 ppm in the diet was dosed to laying hens for 28 consecutive days, no profenofos were detected in any tissues (< 0.05 mg/kg) and eggs (< 0.02 mg/kg). Therefore no residues are to be expected at the maximum estimated dietary burden of 0.04 ppm feed for poultry.

The Meeting estimated a maximum residue level of 0.05(*) mg/kg in poultry meat and edible offal, and 0.02(*) mg/kg in eggs. The Meeting confirmed its previous recommendation for eggs.

The Meeting estimated STMRs and HRs of 0 mg/kg in poultry meat, offal, fat and eggs.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue levels listed below are suitable for estimating maximum residue limits and for IEDI and IESTI assessment.

Plant and Animal commodities:

Definition of the residue (for compliance with the MRL and for estimation of dietary intake):

Profenofos

Commodity		Recommended MRL mg/kg		STMR or STMR-P, mg/kg	HR or HR-P, mg/kg
CCN	Name	New	Previous		
VB 0041	Cabbage, Head	W	1		
SO 0691	Cotton seed	3	2	0.35	
OR 0691	Cotton seed oil, Edible	W	0.05*	0.14	
MO 0105	Edible offal (mammalian)	0.05*	-	0	0
PE 0112	Eggs	0.02*	0.02*	0	0
FI 0345	Mango	0.2	-	0.06	0.07
FI 0346	Mangosteen	10	-	2.1	3.7
MM 0095	Meat (from mammals other than marine mammals)	0.05*	0.05*	0	0
ML 0106	Milks	0.01*	0.01*	0	
VO 0444	Peppers, Chilli	W	5		
HS 0444	Peppers, Chilli (dry)	W	50		
VO 0445	Peppers, Sweet	W	0.5		
VR 0589	Potato	W	0.05*		
PM 0110	Poultry meat	0.05*	-	0	0
PM 0111	Poultry edible offal of	0.05*	-	0	0
VO 0448	Tomato	10	2	1.3	4.7

* at or about the LOQ.

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Dietary Intakes (IEDIs) of profenofos were calculated for the 13 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting (Annex 3 of the 2008 Report of the JMPR). The ADI is 0–0.03 mg/kg bw and the calculated IEDIs were 1–10% of the maximum ADI (0.03 mg/kg bw). The Meeting concluded that the long-term intakes of residues of

profenofos, resulting from the uses considered by current JMPR, are unlikely to present a public health concern.

Short-term intake

The IESTI of profenofos calculated on the basis of the recommendations made by the current Meeting represented 0–10% of the ARfD (1 mg/kg bw) for children and 0–6% for the general population. The Meeting concluded that the short-term intakes of residues of profenofos resulting from the uses considered by the Meeting are unlikely to present a public health concern.

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