TRIAZOPHOS (143)

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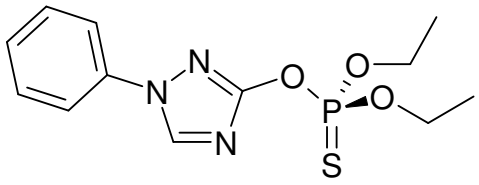
EXPLANATION

Triazophos is an organophosphorus insecticide effective against many insect pests on a wide range of crops. It was originally evaluated for residues by the 1983 JMPR and re-evaluated four times in 1986, 1990, 1992 and 1993, with subsequent revisions in 1984 and 1991.

The manufacturer submitted data on physical and chemical properties, metabolism and environmental fate, methods of residue analysis, use patterns, supervised trials on cotton, residue storage stability and processing. Thailand supplied GAP information and supervised trial data on soybeans (immature seeds).

IDENTITY

ISO Common name:	Triazophos
IUPAC:	*O,O*-diethyl *O*-1-phenyl-1*H*-1,2,4-triazol-3-yl phosphorothioate
CA (index):	Phosphorothioic acid, O,O-diethyl O-(1-phenyl-1H-1,2,4-triazol-3-yl) ester (8CI,9CI)
Manufacturer's code numbers/synonyms:	AE F002960, Hoe 002960
CAS number:	024017-47-8
CIPAC number	353
Molecular formula:	$C_{12}H_{16}N_3O_3PS$
Molecular mass:	313.3 g/mol
Structural formula:	

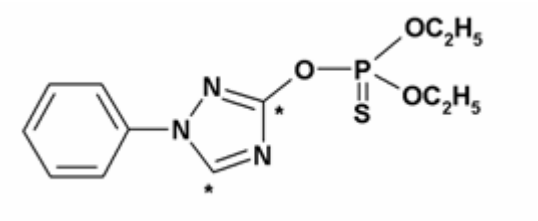
PHYSICAL AND CHEMICAL PROPERTIES

Property	Description or results	References
Physical state and colour	Technical concentrate (784 g/L): Dark brown liquid at RT	Voelkel and Rexer 1998a, b
Odour	Technical concentrate (784 g/L): Typical odour of phosphoric ester	Voelkel and Rexer 1998c
Melting point	Purified active substance (95%):	Mildenberger 1973
Freezing point	Liquid at ambient temperature Freezing point 2 – 5 °C	
Boiling point and Decomposition point	Technical active substance: Boiling point not determined due to Decomposition at Temperatures above 140 °C	Albrecht and Rexer 1983
Relative density	Purified active substance (95%): 1.25 g/cm³ at 20 °C	Mildenberger 1974
	Technical concentrate (784 g/L): 1.12 g/cm³ at 20 °C	Bittner and Rexer 1998a

Property	Description or results	References
Surface tension	Technical concentrate (784 g/L): 32.9 mN/m at 40 °C	Bittner and Rexer 1998b
Vapour pressure	Purified active substance (> 95%): 1.33 10^{-3} Pa at 38 °C 1.33 10^{-2} Pa at 55 °C 1.33 10^{-1} Pa at 74 °C	Heubach 1975
Volatility	Henry's law constant at 20 °C (calculated): $7 \cdot 10^{2}$ Pa·m^{3}·mol^{-1}	Bascou 2006
Solubility in water	Purified active substance: 39 mg/L at 20 °C	Gorlitz 1990
Solubility in organic solvents	Purified active substance (97.4%): all measured at 20 °C n-hexane 11.1 g/L toluene > 500 g/L methanol > 500 g/L 2-propanol > 500 g/L ethyl acetate > 500 g/L polyethylene glycol > 500g/L acetone > 500 g/L dichloromethane > 500g/L dimethylsulfoxide > 500g/L	Gorlitz and Britten 1989
Partition coefficient n-octanol/water	Purified active substance (94.3%): Mean P_{OW} = 2203 log P_{OW} = 3.34 at 22 °C	Schollmeier 1991
Auto flammability	Technical concentrate (784 g/L): 290 °C	Hoffmann 1998a
Explosive properties	Technical concentrate (784 g/L): No danger of explosion	Hoffmann 1998b
Hydrolysis	Purified active substance (95.4%): pH 5: t½ = 296 days at 20 °C; 134 days at 25 °C. pH 7: t½ = 55 days at 20 °C; 30 days at 25 °C. pH 9: t½ = 35 days at 20 °C; 19 days at 25 °C.	Schollmeier and Eyrich 1992
Photochemical degradation	Purified active substance (98-99%): In sterile acetate buffer was irradiated with a xenon arc lamp for 166 hours at 25 °C. Slow photolytic degradation occurred with $t_{½}$ of 392 days.	Stumpf and Jordan 1993

METABOLISM AND ENVIRONMENTAL FATE

Triazophos is an insecticide applied as a foliar spray on several crops e.g. cotton, rice and soybean. In the metabolism and environmental fate studies that were provided by the manufacturer, non-labelled triazophos or 3- and/or 5-triazolyl-[^{14}C]-triazophos was applied.



Animal metabolism

The Meeting received information on the metabolism of triazophos in rats and dogs, but no information on the distribution, excretion or fate of triazophos in livestock and poultry. As the rat and dog studies were reported in the toxicological evaluation of triazophos in 2002, these studies are not reported here.

Plant metabolism

The Meeting received plant metabolism studies for triazophos on cotton and rice.

Cotton

Metabolism in cotton plants was investigated following foliar application under greenhouse and field conditions, in addition to uptake from a hydroponic medium, and from soil (Thier *et al*., 1973a). ^{14}C-labelled triazophos (^{14}C in the 3- and 5- positions of the triazole ring) formulated as a 40% emulsifiable concentrate (EC) was applied to the leaves of plants in the greenhouse (0.5%, single application by syringe ca. 5 weeks prior to bloom at 50 cm height) and to entire plants in the field (0.1%, 3 sprays at 14 and 20 day intervals) using a commercial sprayer. Plants were harvested at 0, 1, 2, 3, 4 and 15 weeks after application (greenhouse trial) and 23 days after the last application (field trial). Plants were separated into leaves (treated, non-treated), stem, roots, bolls, fibre and seeds. Prior to extraction the plants were rinsed to remove any surface residues.

For the hydroponic uptake study, a cotton plant, which had been grown in a greenhouse, was sampled whole with roots and soil intact. The soil was removed with washing and the plant was placed in clean water for 3 days. A hydroponic solution containing 300 mL water with 0.5 mg ^{14}C-triazophos was prepared and the plant placed in this solution for 7 days. Any water that was taken up was replaced daily. At the end of the 7 days, the plant was removed and extracted for radioactivity. The radioactivity in the remaining hydroponic solution was also determined.

In the soil uptake study, a cotton plant that had been grown in a greenhouse was pulled out with roots and soil intact; the soil was washed away as before. The plant was placed in clean soil and left standing until it had taken root again. A ^{14}C-triazophos solution (0.5 mg in 200 mL water) was applied to the surface of the soil, and water was poured onto the soil surface each day for 7 days. At the end of the 7 days, the plant was removed from the treated soil and extracted for radioactivity. Remaining radioactivity at different soil depths in the remaining soil was also determined.

The plant matrices were homogenised and repeatedly extracted with acetone and the acetone extracts were filtered. The solids from the acetone extractions were dissolved in sodium hydroxide (24 h), filtered and washed with water. The resulting extracts were concentrated by evaporation and the radioactivity determined by LSC and TLC. Using silica gel plates, 4 different solvent systems were used to determine the residue profile by chromatography and comparison with reference standards. The results from the greenhouse trial and the field trial are shown in Table 1 and the results from the uptake studies are shown in Table 2.

Triazophos residues persisted longer on cotton plants in the greenhouse trials than in the field trials. Triazophos penetrated quickly into deeper layers of the treated leaves but was not translocated in significant amounts into other parts of the whole plant or roots. In the greenhouse, 27% of the applied radioactivity was still present in the leaves after 4 weeks; the amount of triazophos in these leaves was 25 mg/kg. The P=O analogue of triazophos was only observed after 14 days following application; the maximum radioactivity amounting to 0.2 mg/kg equivalents. The low levels of

radioactive residues found in the cotton fibre and seeds must have reached these parts by translocation as the leaves were treated individually in the greenhouse trials and contamination of the bolls was avoided.

Fifteen weeks (105 days) after initial application, approximately 0.025% of the total radioactivity applied directly to the plants was still present in the cotton fibres. The residues consisted of unchanged triazophos (0.02 mg/kg), 1-phenyl-3-hydroxy-l, 2, 4-triazole (0.01 mg/kg equivalents) and an unidentified compound (0.01 mg/kg equivalents). In the cotton seeds, 0.3 mg/kg triazophos and 0.1 mg/kg equivalents l-phenyl-3-hydroxy-l, 2, 4-triazole was found (Table 3).

Table1. Distribution of radioactivity in cotton plants grown in greenhouse and field conditions

Trial	DALT	Matrix		Triazophos [mg/kg]	Triazole [mg/kg equiv]	O-Triazophos [mg/kg equiv]	Unidentified [mg/kg equiv]
Green house	0		Leaf surface	193	0.5	ND	ND
			Leaf	33	0.1	ND	ND
			Whole plant	ND	ND	ND	ND
			Root	ND	ND	ND	ND
	7		Leaf surface	23	0.1	ND	ND
			Leaf	46	2.9	ND	ND
			Whole plant	0.03	0.01	ND	0.01
			Root	1.5	ND	ND	ND
	14		Leaf surface	15	0.05	0.08	0.07
			Leaf	41	2.4	0.6	ND
			Whole plant	0.3	ND	ND	ND
			Root	4	0.3	ND	0.3
	21		Leaf surface	11	0.05	0.05	ND
			Leaf	49	2.7	0.6	ND
			Whole plant	0.04	ND	ND	ND
			Root	0.4	ND	ND	ND
	28		Leaf surface	4	0.01	0.04	ND
			Leaf	25	3	0.02	ND
			Whole plant	0.003	ND	ND	ND
			Root	0.03	0.01	ND	ND
	105	Bolls	Cotton fibre	0.02	0.01	ND	0.01
			Cotton seeds	0.3	0.1	ND	0.003
Field	23		Leaf surface	0.7	0.03	0.03	ND
			Leaf	3.6	0.4	0.2	ND
			Whole plant	1.1	0.2	0.1	0.01
			Root	0.1	0.01	ND	ND
		Bolls [a]	Cotton fibre	2.2	0.1	ND	ND
			Cotton seeds	1.0	0.1	ND	1.3
		Bolls [b]	Cotton fibre	0.06	ND	ND	ND
			Cotton seeds	0.14	0.06	ND	0.1
		Bolls c	Cotton fibre	0.02	0.01	ND	ND
			Cotton seeds	0.03	0.15	ND	0.4

DALT = Days After Last Treatment

a - already opened at the last treatment, open at harvest

b - closed at the last treatment, open at harvest

c - closed at the last treatment, still closed at harvest

ND= Not Detected

Table 2. Distribution of radioactivity in cotton plants subjected to uptake via soil and hydroponic solution

Trial	Matrix	Triazophos [mg/kg]	Triazole [mg/kg equiv]	O-Triazophos [mg/kg equiv]	Unidentified [mg/kg equiv]
Soil uptake	Roots	0.9	0.02	0.04	ND
	Whole plant	0.04	0.01	ND	0.1
	Soil (0 – 10 cm)	0.05	0.001	0.001	ND
	Soil (10 – 20 cm)	0.05	0.002	0.001	ND
	Soil (20 – 30 cm)	0.02	0.002	ND	ND
Hydroponic uptake	Roots	10	0.005	ND	ND
	Whole plant	4	0.05	ND	ND
	Remaining solution	1.3	0.2	ND	0.4

ND= Not Detected

The results from the uptake study (Table 2) show that a large proportion of the radioactivity is present in the roots compared to the whole plant after either route of uptake, with the hydroponic medium leading to higher levels of radioactivity. Low levels of remaining radioactivity were found in soil and the hydroponic medium, and the radioactivity was primarily composed of parent triazophos. The metabolite l-phenyl-3-hydroxy-l, 2, 4-triazole was also detected in roots and whole plants, however at lower concentrations compared to triazophos. Minor radioactive residues were also detectable in soil.

The results of field applications showed that only low levels of triazophos and its metabolites are likely to be present in cotton fibre and seeds if the last application takes place prior to boll opening. Residues in the cotton fibre and seeds were 0.02 – 0.14 mg/kg triazophos and up to 0.15 mg/kg equivalents l-phenyl-3-hydroxy-l, 2, 4-triazole. Up to 0.4 mg/kg equivalents of an unidentified metabolite was also found.

Rice

Rice plants, grown under both greenhouse and field conditions, were treated with ^{14}C-triazole labelled in the 3- and 5-positions of the triazole ring (Thier *et al.* 1971). The study design was as follows:

- Application to greenhouse plants during stem elongation (BBCH 34)
- Application to greenhouse plants during heading (BBCH 51 – 59)
- Uptake of triazophos by greenhouse plants via roots following application to soil and via water
- Application to field grown plants.

Triazophos was formulated as an EC and applied to rice plants as a spray concentration of 0.5% in greenhouse conditions and as a 0.25% solution in the field. In the greenhouse, the sprays were applied by syringe to the whole leaf surface of the plants. In the field trials, a commercial sprayer was used to apply the radiolabelled material, with up to 4 sprays applied. Samples of plant material were collected from 1 week up to 9 weeks after application in the greenhouse trials and at 13, 10 and 4 weeks after application in the field trials. Whole plants were sampled and separated into grain, husk and remaining whole plant.

In the uptake studies, rice plants from the greenhouse were removed with roots and soil intact and the soil was washed away. One plant was placed in a 300 mL solution containing 0.5 mg ^{14}C-triazophos and left there for 7 days. After 7 days, the plant was removed and the radioactivity was determined in the whole plant as well as in the remaining solution. Similarly, in the soil uptake study, one plant was placed in soil that was covered in water, 10 cm above the soil layer. To that was added 0.5 mg of ^{14}C-triazophos in a spray mixture. Over 7 days, the water in the soil-water set-up was lowered by 2cm each day and refilled with fresh water, to simulate conditions in a rice field. After 7

days the rice plant was removed and radioactivity was determined in the whole plant, the soil and the remaining water.

Surface residues were determined by washing carefully removed leaves and panicles with CH_2Cl_2. These extracts were concentrated prior to LSC analysis. All rice plants and various samples were extracted with acetone and the extracts were filtered. The remaining solids from the acetone extractions were dissolved in 20% NaOH and left to stand overnight. The NaOH extracts were filtered and the radioactivity was determined by LSC. The acetone extracts were combined and evaporated prior to LSC analysis and TLC separation and comparison against reference standards.

Following application to greenhouse plants during stem elongation, surface residues decreased from 76% of the applied radioactivity at 0 weeks to 1.5% at 9 weeks after application. Similarly, surface residues following application at heading decreased from 63% of the applied radioactivity at 0 weeks to 4% at 8 weeks after application. The distribution and characterization of radioactivity in the various fractions is shown in Table 3.

Table 3. Characterisation of radioactivity following a single application to greenhouse rice at heading

Plant fraction	Weeks after application	% of applied radioactivity			
		Triazophos	O-Triazophos	Triazole	Unidentified
Surface wash	0	63	–	–	–
	1	23	–	0.2	–
	2	4.3	0.02	0.12	–
	3	19	0.04	0.24	–
	4	8.4	0.09	0.15	–
	8	4.2	0.06	0.07	–
Panicles	0	3.7	–	–	–
	1	16	–	0.16	–
	2	6.9	0.08	0.58	–
	3	10	0.3	0.5	–
	4	13	0.2	0.6	–
	8	–	–	–	–
Grains	8	0.03	–	0.02	0.65
Husks	8	3.2	0.001	0.09	0.02
Plant remainder	0	19	–	–	–
	1	11	–	1.7	–
	2	20	0.47	5.5	–
	3	27	0.54	8.4	–
	4	16	0.3	7	–
	8	18	0.39	5.8	–
Total	0	86	–	–	–
	1	50	–	2.1	–
	2	31	0.57	6.2	–
	3	56	0.88	9.1	–
	4	37	0.59	7.8	–
	8	25	0.45	6	–

The data in Table 3 show that a large proportion of the applied radioactivity remains on the surface of the rice leaves immediately after application (63%) and slowly declines to < 5% 8 weeks after application. The surface residue is almost all parent triazophos, with some O-triazophos and l-phenyl-3-hydroxy-l, 2, 4-triazole. The radioactivity in rice grains at harvest (8 weeks) is composed of low concentrations of parent triazophos and l-phenyl-3-hydroxy-l, 2, 4-triazole.

In total, the major component of the radioactivity in all plant parts is parent triazophos, comprising 25% to 86% TRR over the 0 week to 8 week interval.

Table 4. Characterisation of radioactivity following a single application to greenhouse rice at stem elongation

Plant fraction	Weeks after application	% of applied radioactivity			
		Triazophos	O-Triazophos	Triazole	Unidentified
Surface wash	9	1.5	–	–	–
Whole plant	9	30		5.4	
Husks	9	0.08		0.02	
Grain	9				0.6

Following a single application of triazophos at stem elongation, the composition of the radioactivity does not differ significantly from the composition following application at heading; the earlier application leaves no detectable residues in rice grain.

Table 5. Distribution of radioactivity from rice uptake studies conducted over 7 days

Plant fraction	Total% TRR	% of applied radioactivity			
		Triazophos	Triazole	O-Triazophos	Unidentified
Uptake from water					
Roots	7.7	6.95	0.41	0.09	0.22
Plant	2.2	1.97	0.17	0.01	0.04
Panicle	0.4	0.37	0.01	0.001	0.06
Remaining solution	83	39	36.6	1.2	6.2
Uptake from water and soil					
Roots	0.06	0.039	0.016	0.005	
Plant	0.15	0.116	0.027		0.007
Panicle	0.06	0.045	0.015		
Water	82	9.9	72		
Soil & water	17.4	8		1.8	3.4

The data from the 7-day uptake studies show that a large proportion of the applied radioactivity remains in either the water, or soil and water following application and very little of the radioactivity is in the rice panicle (< 0.5% of applied radioactivity). The major components of the radioactivity in the water are triazophos and l-phenyl-3-hydroxy-l, 2, 4-triazole.

Table 6. Distribution of radioactivity following four applications of ^{14}C-3-triazophos to rice in field conditions

Plant fraction	Application timing (weeks before harvest)	% of applied radioactivity			
		Triazophos	Triazole	O-Triazophos	Unidentified
Plant surface	13	0.01	0.02	–	–
Whole plant		0.03	0.08	–	–
Roots		0.36	0.55	–	–
Husks		0.21	0.03	–	–
Grain		0.005	0.005	–	0.03
Plant surface	10	0.02	0.01	–	–
Whole plant		0.09	0.29	–	0.07
Roots		0.14	0.18	–	–
Husks		0.1	0.02	–	–
Grain		–		–	0.02
Plant surface	4	0.05	0.005	0.003	–
Whole plant		0.54	1.04	–	0.42
Roots		0.06	0.06	–	–
Husks		5.29	0.22	0.07	0.09
Grain		–	0.02	–	0.62

The results from the field study show that very little of the applied radioactivity is present in harvested rice grain following multiple applications under field conditions. Again, the main components identified are parent triazophos and l-phenyl-3-hydroxy-l, 2, 4-triazole.

Environmental fate in soil and water-sediment systems

The Meeting received information on the uptake of triazophos by leek plants from soil (Thier *et al*. 1973b), and the degradation of triazophos in soil under field conditions (Baedelt and Bürstell, 1993). For products that are applied as foliar sprays, data on degradation in soil (aerobic or anaerobic) are not required as determined by the 2003 meeting of the JMPR[22]. Consequently, the soil degradation study is not reported here.

Uptake from soil by leek

In a field trial, plots of loamy soil and sandy soil were treated with 5-^{14}C-triazophos at application rates equivalent to 0.48 and 0.96 kg ai/ha (Thier *et al*. 1973b). The spray mixtures containing the 40% EC formulation were applied by a sprayer to an area of about 1000 cm^2 (33 × 38 cm) in the centre of which one leek plant was planted. Each type of soil was sprayed at both rates.

Samples were taken from each sprayed plot at 90 days after application. After removal of the leek plants, soil samples at different depths (0 – 10 cm, 10 – 20 cm, 20 – 30 cm) were removed. The soil samples and the leek plants were extracted and the extracts analysed by thin layer chromatography. The leeks contained no detectable radioactivity.

After 90 days, only minor radioactive residues were detected in the extracts of the upper soil layer (0 – 10 cm), while the lower soil layers (10 – 20 cm and 20 – 30 cm) contained low levels of radioactive residues or none at all.

After 90 days, parent triazophos and substances which in thin layer chromatography behaved like l-phenyl-3-hydroxy-l, 2, 4-triazole-5-^{14}C, the P=O analogue (O-triazophos) and like urea-^{14}C were found (Table 9).

Table 7. Radioactive residues in leak plants and soil from field trials 90 days after application of triazophos to the soil

Type of soil	Application rate (kg ai/ha)	Sample	% of applied radioactivity			
			Triazophos	O-Triazophos	Triazole	Urea
Loamy soil	0.48	Leek	ND	ND	ND	ND
		Soil 0-10 cm	0.92	0.50	0.42	ND
		10-20 cm	NA	NA	NA	NA
		20-30 cm	0.14	0.10	0.05	ND
	0.96	Leek	ND	ND	ND	ND
		Soil 0-10 cm	2.2	0.8	1.0	ND
		10-20 cm	0.09	0.08	0.05	0.04
		20-30 cm	0.02	0.02	0.02	ND
Sandy soil	0.48	Leek	ND	ND	ND	ND
		Soil 0-10 cm	0.10	0.02	0.01	ND
		10-20 cm	NA	NA	NA	NA
		20-30 cm	NA	NA	NA	NA

[22] Revised data requirements for studies of environmental fate in FAO Plant Production and Protection Paper 176, Pesticide Residues in Food, Report 2003.

Type of soil	Application rate (kg ai/ha)	Sample	% of applied radioactivity			
			Triazophos	O-Triazophos	Triazole	Urea
	0.96	Leek	ND	ND	ND	ND
		Soil 0-10 cm	1.80	0.4	0.2	ND
		10-20 cm	0.22	0.06	0.04	ND
		20-30 cm	0.01	0.01	0.01	0.01

ND = not detected

NA = chromatographic analysis not possible because of too small amounts of radioactivity in the sample

RESIDUE ANALYSIS

Analytical methods

The Meeting received information on the general method DFG S19 for the determination of triazophos residues in plant materials using GC and modified methods for determination of triazophos in animal commodities, soil and water. Only validated methods with reported recoveries are described here.

A validated method for the analysis of triazophos in cotton seed, leaf, lint (Simplício 1993) and cotton seed oil and press cake (Simplício 1995) was provided to the Meeting. Plant material was extracted with acetone, and water was added so that during extraction the acetone /water ratio remained constant at 2:1 (v/v). The extract was saturated with sodium chloride and diluted with CH_2Cl_2, resulting in separation of excess water. The evaporated organic phase was purified by gel permeation chromatography on Bio Beads S-X3 polystyrene gel, using a mixture of cyclohexane and ethyl acetate as the eluant. The eluate was concentrated and separated by GC using a phosphorus- or nitrogen-selective detector. The LOQ (limit of quantitation) for triazophos was validated at 0.02 mg/kg, with the overall mean recoveries of 83% and RSD of 11 in lint; 82% and 4 in seed; 92% and 8 in leaf; 95% and 13 in seed oil; and 88% and 9 in press cake.

Table 8. Limits of Quantitation and Recoveries in Cotton Matrices, Oranges and Carrots

Matrix		Fortification level (mg/kg)	Recoveries (%)	Mean Recovery (%)	RSD (n)	Reference
Cotton lint		0.02	97, 91	83	11 (6)	Simplício 1993
		0.2	73, 84			
		1	77, 78			
Cotton seed		0.02	88, 79	82	4 (6)	
		0.2	82, 85			
		1	79, 80			
Leaf		0.02	90, 104	92	8 (6)	
		0.2	82, 97			
		1	93, 87			
Cotton press cake		0.02	76, 92	95	13 (6)	Simplício 1995
		0.2	96, 91			
		1	111,104			
Cotton seed oil		0.02	88, 87	88	9 (6)	
		0.2	75, 84			
		1	98, 93			
Cotton fibre		0.8	83-114	100	10 (14)	Tillkes and Weeren 1994a
Cotton seed		0.2	88-102	95	6 (14)	Tillkes and Weeren 1994b
Oranges		0.1	83-115	99	8 (14)	Tillkes and Weeren 1994c
Carrots		0.1	95-116	103	10 (14)	Tillkes and Weeren 1994d

RSD = relative standard deviation

The Meeting received details of Method AL 049/96-0 and its validation (Burstell *et al*. 1996, Brumhard 2006), which was used to determine residues of triazophos in animal commodities such as meat, milk, and eggs. Samples of meat and milk were extracted with acetone and Celite, eggs were extracted with acetone only and centrifuged. The residues were filtered and the filter cakes were washed with acetone or acetone/water. The remaining filtrate was concentrated, partitioned against CH_2Cl_2 followed by drying of the organic phase. The CH_2Cl_2 extracts were combined and evaporated to dryness, and taken up in hexane. The residue in hexane is cleaned up on a silica column and eluted with hexane/toluene. The eluates are evaporated and concentrated to near dryness before being re-dissolved in toluene for GC analysis. The final determination was by GC with a flame photometric detector. The LOQ for milk, meat and eggs was validated at 0.01 mg/kg for the determination of triazophos. Recoveries were conducted by fortification at concentrations of 0.01 – 0.5 mg/kg triazophos in all three matrices. The results are reported below.

Table 9: Validated recoveries of triazophos in milk, meat and eggs

Sample/Matrix	Fortification concentration (mg/kg)	Recovery (%)	Mean Recovery (%)
Milk	0.01	103, 85, 78, 83, 85	87
	0.05	72, 79, 80, 80, 79	78
	0.5	102, 93, 83, 93, 90	92
		Overall	85.7% RSD 8.9%
Meat	0.01	103, 92, 104, 104, 81	97
	0.05	73, 70, 85, 74, 84	77
	0.5	83, 84, 89, 94, 73	84
		Overall	86.2% RSD 11.4
Eggs	0.01	86, 70, 76, 75, 81	77
	0.05	81, 77, 74, 71, 77	76
	0.5	76, 74, 83, 75, 97	81
		Overall	78.2 RSD 6.8

The recoveries of triazophos in milk ranged from 72 – 103%; mean recovery of 86%, with a RSD of 9. The recoveries of triazophos in meat ranged from 70 – 104%, with a mean of 86% and a RSD of 11. The recoveries of triazophos in eggs ranged from 70 – 97%, with a mean of 78% and a RSD of 7.

In Method ADV 4276/05-01 (Manjunatha and Hosmani 2006 ADV 4276/05-01), triazophos was extracted with ethyl acetate from cotton foliage (shoots), cotton lint and seed samples, or with acetonitrile from cotton oil samples. The filtered ethyl acetate extract was concentrated followed by clean-up on an aluminium oxide active neutral column. The acetonitrile extract was partitioned with hexane prior to column clean-up. In both cases the column is washed with cyclohexane and eluted with 30% ethyl acetate in cyclohexane. Quantitation was by gas GC/NPD. The LOQ was 0.02 mg/kg for all matrices.

Table 10. Validation data for method ADV 4276/0501 (Manjunatha and Hosmani 2006 ADV 4276/05-01)

Matrix	Fortification level (mg/kg)	N	Recoveries (%)	Mean recovery (%)	RSD
Cotton lint	0.02	4	105 102	101	3
	0.2		99 99		
Cotton seed	0.02	6	97 97 93	94	3
	0.2		92 91 92		
Cotton seed oil	0.02	4	104 105	102	3
	0.2		99 101		
Cotton foliage	0.02	4	99 97	96	3
	0.2		93 95		

Stability of residues in stored analytical samples

The Meeting received information on the stability of residues in stored analytical samples of cotton fibre, cotton seed, oranges, carrots, and soil at ≤ -18 °C. The soil stability is not reported here.

The stability of triazophos residues was determined in stored samples of cotton fibre (Tillkes and Weeren 1994a), cotton seed (Tillkes and Weeren 1994b), oranges (Tillkes and Weeren 1994c), and carrots (Tillkes and Weeren 1994d). In the cotton study, plant material was extracted with acetone in a Soxhlet apparatus for 6 hours. The extract was concentrated and purified by gel permeation chromatography on Bio Beads S-X3 polystyrene gel. The eluate was concentrated and separated by gas chromatography on a capillary DB-1701 column, and the final determination was performed with a flame photometric detector (P-mode) at 526 nm. Recoveries were spiked at a level of 0.8 mg/kg (fibre) or 0.2 mg/kg (seed) representing 10 × LOQ. Untreated samples were analysed in a single determination. Concurrent recoveries and storage stability samples were analysed in duplicate at each storage period. The LOQ of the analytical method was 0.08 mg/kg (fibre) or 0.02 mg/kg (seed).

The LOQ of the method was 0.01 – 0.08 mg/kg, depending on the matrix analysed. Stored samples of triazophos and concurrent recoveries were spiked at concentrations of 0.2 – 0.8 mg/kg, representing 10 × LOQ. The samples were stored in a deep freezer (≤ -18 °C) for up to 24 months. The samples were thawed and analysed after storage intervals of 1 day, 1, 3, 6, 12, 18 and 24 months. The stored samples were analysed in duplicate at each storage interval and concurrent recoveries were also conducted in duplicate at each interval.

The concurrent recoveries were between 83 and 116% resulting in a mean recovery of 95 – 103% with a RSD (relative standard deviation) of 6 – 10% (Table 11). No significant decrease of triazophos was observed during the storage period of 24 months. Therefore, residues of triazophos on cotton fibre, cotton seed, oranges, carrots, soil are stable during storage in the dark deep freeze for at least 24 months.

Table 11. Stability of triazophos residues in cotton fibre and seed, oranges and carrot up to 24 months of storage at ≤ -18 °C

Matrix	Storage Interval	Fortification concentration (mg/kg)	Residue found (mg/kg)	Concurrent recovery (mg/kg)	Concurrent recovery (%)
Cotton fibre	1 day	0.8 mg/kg	0.731, 0.745	0.715, 0.748	90, 94
	1 month		0.773, 0.765	0.774, 0.818	97, 103
	3 months		0.814, 0.784	0.679, 0.791	85, 99
	6 months		0.709, 0.762	0.660, 0.765	83, 96
	12 months		0.90, 0.80	0.892, 0.916	111, 114
	18 months		0.837, 0.869	0.800, 0.832	100, 104
	24 months		0.833, 0.856	0.919, 0.916	110, 110
Cotton seed	1 day	0.2	0.198, 0.206	0.203, 0.176	102, 88
	1 months		0.182, 0.199	0.195, 0.195	98, 98
	3 months		0.178, 0.187	0.168, 0.176	84, 88
	6 months		0.160, 0.171	0.184, 0.175	92, 88
	12 months		0.191, 0.194	0.202, 0.204	100, 102
	18 months		0.186, 0.184	0.194, 0.188	97, 94
	24 months		0.197, 0.205	0.209, 0.206	100, 99
Oranges	1 day	0.1	0.104, 0.104	0.104, 0.117	101, 115
	1 month		0.105, 0.102	0.106, 0.106	104, 103
	3 months		0.098, 0.102	0.108, 0.099	107, 97
	6 months		0.091, 0.102	0.100, 0.104	100, 105
	12 months		0.102, 0.090	0.096, 0.098	93, 95
	18 months		0.089, 0.088	0.090, 0.086	87, 83
	24 months		0.082, 0.099	0.096, 0.106	93, 102
Carrots	1 day	0.1	0.102, 0.110	0.109, 0.107	110, 107
	1 month		0.106, 0.117	0.107, 0.108	108, 109
	3 months		0.104, 0.103	0.116, 0.111	116, 111
	6 months		0.113, 0.108	0.107, 0.979	107, 98
	12 months		0.086, 0.089	0.094, 0.100	95, 100
	18 months		0.087, 0.088	0.095, 0.074	95, 73
	24 months		0.102, 0.118	0.107, 0.109	107, 109

USE PATTERN

Triazophos is an insecticide and acaricide and shows a broad-spectrum activity against a wide range of sucking pests, mites and some Lepidoptera. Triazophos also exhibits ovicidal action against eggs of mites and Heliothis and possesses nematicidal properties against certain free-living nematodes.

Products containing the active substance triazophos are commercially availability as single active EC formulations or containing combinations of actives as shown in Table 12. The most common combination product is that containing triazophos and deltamethrin.

Table 12. Triazophos formulations either as a combination with other active constituents or alone

Formulation	Content of active ingredients
EC 400	428 g/ Triazophos or 424 g/L Triazophos or 420 g/L Triazophos or 400 g/L Triazophos
EC 200	190 g/L Triazophos
EC 212	200 g/L Triazophos & 12 g/L Deltamethrin
EC 260	250 g/L Triazophos & 10 g/L Deltamethrin
EC 360	350 g/L Triazophos & 10 g/L Deltamethrin
EC 180	150 g/L Triazophos & 30 g/L Cypermethrin
EC 186	150 g/L Triazophos & 36 g/L Cypermethrin
EC 280	250 g/L Triazophos & 30 g/L Cypermethrin
EC 388	300 g/L Triazophos & 72 g/L Cypermethrin & 16 g/L Acetamiprid

The Meeting received information and field trial data for uses of triazophos on cotton and soybean, for which relevant use patterns are summarized in Tables 13 and 14.

Table 13. Use patterns of triazophos containing formulations/products supported by residue data on cotton

Crops	Country	Formulation/ content of active substance (g/L)	Application					PHI (days)
			method	rate (kg a.s./ha)	Water rate (L/ha)	No.	Interval (days)	
Asia								
Cotton	India	EC/triazophos 420	foliar spraying	triazophos: 0.63- 0.84	500 – 1000	1 - n.a. [a]	7-10	field: 21
		deltamethrin + triazophos 10 + 350	foliar spraying	deltamethrin: 0.010-0.013 + triazophos: 0.350-0.438	600 – 1000	1 - n.a. [a]	7-10	field: 21
Cotton	Pakistan	deltamethrin + triazophos 10 + 350	foliar spraying, high-volume	deltamethrin: 0.005- 0.008 + triazophos: 0.175- 0.28	200 – 250	1		field: 14
America								
Cotton	Belice Costa Rica El Salvador Guatemala Honduras Nicaragua Panama Dominican Rep.	deltamethrin + triazophos 12 + 200	foliar spraying	deltamethrin: 0.012-0.018 + triazophos: 0.20-0.30	300 – 600	1-3	5	field: 21
		deltamethrin + triazophos 12 + 200	foliar spraying	deltamethrin: 0.012-0.018 + triazophos: 0.20- 0.30	300 – 600	1-3	5	field: 21

Crops	Country	Formulation/ content of active substance (g/L)	Application					PHI (days)
			method	rate (kg a.s./ha)	Water rate (L/ha)	No.	Interval (days)	
Cotton	Brazil	deltamethrin + triazophos 10 + 350	foliar spraying	deltamethrin: 0.0075-0.01 + triazophos: 0.263-0.350	150 – 300	1-2		field: 28
		(EC formulation)/ triazophos 400	foliar spraying	triazophos: 0.3–0.8	100 – 400	1-3	7-10	field: 28
Cotton	Ecuador	EC/Triazophos 400	foliar spraying	triazophos: 0.60- 0.80	40 - 400	1		field: 21
Cotton	Peru	EC/Triazophos 428	foliar spraying	triazophos: 0.642	300 – 400	1		field: 28
Africa								
Cotton	Sudan	deltamethrin + triazophos 10 + 350	foliar spraying	deltamethrin: 0.012 + triazophos: 0.42	10-50	1		field: 28

a - Maximum up to 5; 5 sprays is the worst case scenario

Table 14. Use patterns of triazophos containing formulations/products supported by residue data on soybean

Crops	country	formulation/ Content of Triazophos (g/L)	Application					PHI (days)
			Method	Rate (kg ai/ha)	Water rate (L/ha)	No.	Interval (days)	
Soybean	Thailand	Triazophos 420	foliar spraying	triazophos: 0.5-0.625	625	1 – 2 [a]	7	field: 14

a - Maximum up to 5; 5 sprays is the worst case scenario

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received information on supervised trials for triazophos uses on cotton and soya bean.

Analytical reports included method validation with procedural recoveries from fortification at concentrations similar to those occurring in samples from supervised field trials. Dates of analyses and/or duration of residue sample storage were provided. Intervals of freezer storage between sampling and analysis were recorded for all trials and were covered by the conditions of the freezer storage stability studies.

All trials included analyses of control samples, however no control data are recorded in the tables except in instances where residues in the control samples exceeded the validated LOQ. Residue data reported in the following tables are not adjusted for analytical recoveries.

In most trials, field samples from an unreplicated plot were taken at each sampling interval and were analysed. For the purposes of the evaluation, where replicate samples were analysed separately, the mean of the two results is reported as the residue from the plot in following tables.

When residues were not detected and were reported as ND in the studies, they are indicated as being 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 trials that reflect the maximum GAP have been used for the estimation of maximum residue levels. Residue trials conducted at exaggerated or higher than label application rates are also included in the estimation of maximum residue levels where the resulting residues are less than the LOQ. The values that are included in the MRL estimation are underlined in the tables below.

Cotton

India

In 2005, field trials on cotton were conducted in northern, southern and western India. Plot sizes were 25 – 2400 m^2. Application was by knapsack sprayers. Growth stage at last application was not reported (e.g., percentage bolls open). Samples were stored frozen for up to seven months prior to analysis. Triazophos residues in/on cotton lint, seeds, seed oil and shoot (foliage) samples were analysed according to method ADV 4276/05-01. Recoveries for samples fortified at 0.02 to 0.2 mg/kg were 91 to 104 %.

Table 15. Results of residue trials conducted with triazophos 40 EC in/on cotton in India in 2006

Region, Country, Year, Variety	Application				Residue			Reference Report No; Trial No.
	FL	No	kg ai/ha	Spray vol L/ha	Portion analysed [a]	PHI (days)	Triazophos (mg/kg)	
Karnataka, India, 2006 NHH-44	40 EC	5	0.87	378-489	shoot	0	40	RES/Anal-4276/05; 1
					lint	21 / 31	0.004	
					seed	21 / 31	<u>0.021</u>	
					oil	21 / 31	0.042	
Andhra Pradesh, India, 2006, 217	40 EC	5	0.87	400-420	shoot	0	42	RES/Anal-4276/05; 2
					lint	21 / 59	0.005	
					seed	21 / 59	<u>0.059</u>	
					oil	21 / 59	0.31	
Tamil Nadu, India, 2006, Surabhi	40 EC	5	0.87	390-480	shoot	0	95	RES/Anal-4276/05; 3
					lint	23 / 33	0.13	
					seed	23 / 33	<u>0.021</u>	
					oil	23 / 33	0.044	
Maharashtra, India, 2006, NCS 145	40 EC	5	0.87	426-478	shoot	0	71	RES/Anal-4276/05; 4
					lint	21 / 31	0.006	
					seed	21 / 31	<u>0.042</u>	
					oil	21 / 31	0.26	
Gujarat, India, 2006, Ankur	40 EC	5	0.87	400-460	shoot	0	62	RES/Anal-4276/05; 5
					lint	21 / 31	0.006	
					seed	21 / 31	<u>0.028</u>	
					oil	21 / 31	0.085	
Rajasthan, India, 2006, RS-2013	40 EC	5	0.87	430-460	shoot	0	35	RES/Anal-4276/05; 6
					lint	21 / 31	0.007	
					seed	21 / 31	<u>0.023</u>	
					oil	21 / 31	0.17	
Haryana, India, 2006, Shakti vardhak	40 EC	5	0.87	400-483	shoot	0	50	RES/Anal-4276/05; 7
					lint	21 / 33	0.004	
					seed	21 / 33	<u>0.060</u>	
					oil	21 / 33	0.78	
Punjab, India, 2006, RIL-110	40 EC	5	0.87	430-460	shoot	0	121	RES/Anal-4276/05; 8
					lint	21 / 31	0.007	
					seed	21 / 31	<u>0.054</u>	
					oil	21 / 31	0.088	

Region, Country, Year, Variety	Application				Residue			Reference Report No; Trial No.
	FL	No	kg ai/ha	Spray vol L/ha	Portion analysed [a]	PHI (days)	Triazophos (mg/kg)	
Madhya Pradesh, India, 2006, Ankur	40 EC	5	0.87	400-460	shoot	0	69	RES/Anal-4276/05; 9
					lint	21 / 31	0.006	
					seed	21 / 31	0.020	
					oil	21 / 31	0.13	

a - Samples collected at the two PHIs were pooled. The oil was extracted from cotton seeds with n-hexane by a 4 hour Soxhlet extraction. The solvent was removed by rotary evaporation and the remaining raw oil was subject to analysis.

Brazil

In Brazil one parallel field trial was run in 2001 (Pimentel Trevizan and Casadei de Baptista 2001). Plot sizes were 50 m^2. Recoveries for samples fortified at 0.03 and 0.3 mg/kg were 126% and 115% with RSDs of 7 and 15, respectively. Samples were analysed within 6 months of harvest.

Table 16. Results of residue trials conducted with triazophos in/on cotton in Brazil 2001

Region, Country, Year, variety	Application				Residue			Reference Report No.; Trial No.
	FL	No	kg ai/ha	kg ai/hL	Portion Analysed	PHI (days)	triazophos (mg/kg)	
Paulinia – SP, Brazil 2001 Deltapine	40 EC	3	0.80	0.27	Seed	*28*	0.03	*ID01BRAZ61FL01* ID01BRAZ61FL01-A
	40 EC	3	1.6	0.53	Seed	*28*	0.2	*ID01BRAZ61FL01* ID01BRAZ61FL01-B

Soybean

Thailand

A total of six field trials on soybean (variety/cultivar: OCB) were conducted in Saraburi province, central Thailand in 1992 – 2006 (Thailand. 2007a, b). The crop was sprayed by knapsack sprayer with 2 – 4 applications of triazophos 40 EC at a rate of 0.6 – 0.75 kg ai/ha, with the last application on 35 – 58 days after planting. Samples of whole fruit were taken randomly 0 – 28 days after last application. Triazophos residues in whole pods including immature seeds of soybean were analysed with GC-FPD by a method reported by Anderson & Palsheden (1991).

The LOQ of 0.02 mg/kg was confirmed for soybean pods including immature seeds, and concurrent recoveries were between 82 and 89 %. No apparent residue of triazophos was observed in any of the control samples and the residues found for triazophos in treated soybean pods including immature seeds are presented in Table 17.

Table 17 Results of residue trials conducted with triazophos 40 EC in soybean in Thailand in 1992-2006

Crop Region, Country Year	Application				Residue			Reference Report No; Trial No.
	FL	No.	kg/ha (ai)	kg/hl (ai)	Portion analysed	PHI (days)	Triazophos (mg/kg)	
Soybean Saraburi Thailand 1992	40 EC	2	0.6	0.1	Whole pods including immature seeds	17	0.05	TRI-VS-01
Soybean	40 EC	3		0.1	Whole pods	7	0.98	TRI-VS-02

Crop Region, Country Year	Application				Residue			Reference Report No; Trial No.
	FL	No.	kg/ha (ai)	kg/hl (ai)	Portion analysed	PHI (days)	Triazophos (mg/kg)	
Saraburi Thailand 1994			0.7		including immature seeds	15	0.60	
						21	0.19	
Soybean Saraburi Thailand 2004	40 EC	4	0.61	0.1	Whole pods including immature seeds	0	3.94	TRI-VS-03
						3	1.78	
						7	1.14	
						10	0.47	
						14	0.43	
						21	ND	
						28	ND	
Soybean Saraburi Thailand 2004	40 EC	4	0.62	0.1	Whole pods including immature seeds	0	3.06	TRI-VS-04
						3	1.69	
						7	1.04	
						10	0.44	
						14	0.31	
						21	ND	
						28	ND	
Soybean Saraburi Thailand 2006	40 EC	3	0.75	0.1	Whole pods including immature seeds	0	7.71	TRI-VS-05
						2	2.87	
						4	1.83	
						7	1.06	
						14	0.52	
						21	0.17	
						28	0.12	
Soybean Saraburi Thailand 2006	40 EC	4	0.66	0.1	Whole pods including immature seeds	0	7.08	TRI-VS-06
						1	1.78	
						4	1.05	
						7	0.67	
						14	0.17	
						21	0.07	
						28	0.06	

FATE OF RESIDUES DURING PROCESSING

The Meeting also received the information on triazophos residues in different processed commodities of cotton. In three parallel field trials in Somerton, Arizona (USA) in 1973 for cotton processed commodities, the plots were treated with one application of triazophos EC corresponding to 0.39, 0.56 and 1.1 kg ai/ha via aircraft (Anon 1974). Samples of cotton seed were processed into hulls, screwpress meal, crude oil, refined oil, refined bleached oil, refined bleached hydrogenated oil and soap stock, and then analysed for residues of triazophos by method AL 70/73.

Recoveries conducted for samples fortified at 0.004 to 0.02 mg/kg were 78 to 94%. No residues could be found in soap stock. Residues were detected in crude oil and processed oils that decreased with increasing processing such that no residues were detected in refined bleached hydrogenated oil. Processing factors could not be determined as residue levels in unprocessed cotton seed were not reported.

Table 18. Results of processing trials conducted with triazophos 40 EC in/on cotton in the USA

Region	Application							Reference
Country, Year	FL	No	kg ai/ha	kg ai/hL	Portion analysed	PHI (days)	triazophos (mg/kg)	*Report No.;* Trial No.
Somerton USA 1973	40 EC	1	0.39	0.82	Hulls	70 - 73	0.30	*USA 1/74* 1A
					Screwpress meal		0.02	
					Crude oil		0.12	
					Refined oil		0.16	
					Ref. bleached oil		< 0.02	
					Ref. bleached hydrogenated oil		< 0.01	
					Soap stock		< 0.02	
			0.56	1.18	Hulls	70 - 73	< 0.02	*USA 1/74* 2B
					Screwpress meal		0.09	
					Crude oil		0.26	
					Refined oil		0.20	
					Ref. bleached oil		0.07	
					Ref. bleached hydrogenated oil		< 0.01	
					Soap stock		< 0.02	
			1.12	2.37	Hulls	70 – 73	< 0.02	*USA 1/74* 3C
					Screwpress meal		< 0.02	
					Crude oil		0.66	
					Refined oil		0.23	
					Ref. bleached oil		0.14	
					Ref. bleached hydrogenated oil		< 0.01	
					Soap stock		< 0.02	

Lactating Cow Feeding Study

A lactating cow feeding study was reported by Krebs *et al*. 1987. The study included four animals (Holstein Friesian), one of which was an untreated control and three of which were exposed to triazophos. Each dosing group consisted of a single animal. Triazophos was mixed with the feed and given to three lactating cows. The doses were mixed into a premix ration which consisted of maize meal; the whole feed consisted of maize silage, hay, sugar beet chips and the premix ration. The total feed intake was 21 kg/day. The premix was provided to the animals once a day each morning after milking and before the rest of the feed was offered. Individual feed consumptions were reported; bodyweights were reported at the start of the pre-conditioning and at the end of the dosing phases.

The dosing regime involved a 2 day pre-conditioning phase for all three cows, one week prior to the actual dosing period, at a dose level of 100 mg triazophos per cow. During the dosing period of 7 days, one cow was given 50 mg triazophos (2.38 ppm in the feed); another cow was given 100 mg triazophos (4.76 ppm in the feed). The third cow received untreated feed in order to evaluate possible residues in milk and tissues originating from the doses during the pre-conditioning phase only. Milk samples from the individual animals were collected prior the dosing period and on each day of the 7 day dosing period. Milk was collected every morning and evening during the dosing period; milk production was reported for each day during the pre-conditioning and dosing phases.

All cows were slaughtered on the day (approximately 24 h) after the final dosing. Samples of muscle, fat, liver and kidney were taken from each cow. Residues of triazophos were determined according to method AL 42/80 (Specht and Tilkes, 1980). The extraction of residues from the various samples involved maceration with acetone three times, filtration of the acetone extracts, evaporation to dryness followed by re-dissolving in water. After clean up on an Extrelut column and elution with hexane/diethyl ether, residues were determined via gas chromatography with flame photometric detection. Milk samples were applied directly to the Extrelut column. The limits of quantitation were 0.01 mg/kg in tissues and 0.05 mg/kg in milk; recovery information is presented in Table 11.

Neither pre-conditioning at 100 mg of the test substance per cow per day nor dosing of 50 and 100 mg per cow per day resulted in any residues above the quantification limit of 0.05 mg/kg in milk. Muscle (diaphragm), fat, blood, kidney and liver did not show any residues above the limit of quantitation of 0.01 mg/kg.

APPRAISAL – RESIDUE AND ANALYTICAL ASPECTS

Triazophos is an organophosphorus insecticide used to control insect pests on a wide range of crops. It was originally evaluated for residues by the 1983 JMPR and re-evaluated four times in 1986, 1990, 1992 and 1993, with subsequent revisions in 1984 and 1991. It was scheduled for periodic re-evaluation by the 2007 JMPR.

The manufacturer submitted data on physical and chemical properties, metabolism and environmental fate, methods of residue analysis, use patterns, residues resulting from supervised trials on cotton and stability of residues during storage and processing. The government of Thailand provided GAP information and supervised trial data for soya bean (immature seeds).

Animal metabolism

The Meeting received information on the metabolism of triazophos in rats and dogs, but no information on the distribution, excretion or fate of triazophos in livestock and poultry. As both the rat and dog studies were described in detail in the toxicological evaluation of the 2002 JMPR, the studies were not reported here.

Plant metabolism

The Meeting received plant metabolism studies for triazophos on cotton and rice. Both studies included foliar application as well as uptake via water and/or soil.

When [^{14}C]triazophos was applied to leaves of cotton plants, the radioactivity was at first present on the leaf surface but quickly penetrated into the leaf itself, with little translocation into other parts of the plant. The predominant component of the radioactivity was parent triazophos. After field application of ^{14}C-triazophos prior to boll opening, only very low residues of triazophos and its metabolites were present in cotton fibre and seeds. In both trials, the residues consisted of unchanged triazophos, 1-phenyl-3-hydroxy-l, 2, 4-triazole and an unidentified compound. Only traces of the P=O analogue of triazophos (O, O-diethyl-O-l-phenyl-lH-l,2,4-triazol-3-yl phosphate) were detectable on the leaves and stem. In general, triazophos penetrated quickly into deeper layers of the treated leaves but was not translocated in significant amounts into other parts of the plant or roots.

In uptake studies via both soil and a hydroponic medium, parent triazophos was the predominant component of the applied radioactivity, with most of the radioactive residues being present in the plant root, compared to the whole plant, or remaining in the soil or hydroponic medium. Again, most of the extracted radioactivity was composed of parent triazophos, although both the P=O analogue and 1-phenyl-3-hydroxy-l, 2, 4-triazole were also found. The results of field applications showed that only low levels of triazophos and its metabolites are likely to be present in cotton fibre and seeds if the last application takes place prior to boll opening.

Greenhouse grown rice plants were treated with [^{14}C]triazole at the growth stages of either stem elongation or heading. Initially the majority of the applied radioactivity was present on the surface of the rice plants, however by 8 weeks after the application, radioactivity was found in the rice panicles. Little radioactivity was found in rice grain. The major component of the extracted radioactivity was parent triazophos in rice panicles, husks, grain and whole plant. The 1-phenyl-3-hydroxy-l, 2, 4-triazole was also present in whole plant (< 10% of applied radioactivity) and very low levels were in rice grain (0.02% of applied radioactivity). The P=O analogue was also present, but in amounts lower than triazophos or the triazole (< 1% of applied radioactivity).

In addition, uptake studies were conducted in rice, with application of ^{14}C-triazophos to water or soil and water. As with the cotton study a large proportion of the applied radioactivity remained in

the soil or soil and water medium, with little radioactivity present in either the whole plant or the panicle. Again parent triazophos was the major component of the radioactivity, although the 1-phenyl-3-hydroxy-l, 2, 4-triazole and the P=O analogue were also detected.

In conclusion, the results from the field study show that very little of the applied radioactivity is present in rice grain following multiple applications under field conditions.

Uptake from soil by leek plants

The Meeting received information on the uptake of triazophos from soil by leek plants.

Plots of loamy soil and sandy soil were treated with [^{14}C]triazophos at application rates equivalent to 0.48 and 0.96 kg ai/ha. Leek plants were present in the treated plots. At 90 days after application, samples of soil, taken at various depths, and leek plants were collected for determination of radioactivity. No detectable radioactivity was found in the leek plants. In the soil samples up to 2.2% of the applied radioactivity was found (0 – 10 cm depth), with lower concentrations (< 0.2% of applied radioactivity) present at 10 – 20 cm and 20 – 30 cm depths. The radioactivity was predominantly composed of parent triazophos and 1-phenyl-3-hydroxy-l, 2, 4-triazole.

Methods of analysis

The Meeting received information on methods capable of determining residues of triazophos in plant materials and animal commodities, using GC with N or P selective detectors. The limits of quantitation were typically 0.02 mg/kg for plant commodities and 0.01 for animal commodities. Reported recoveries were within acceptable limits of 70 – 110%.

Stability of residues in stored analytical samples

Studies were provided to the Meeting demonstrating the stability of residues in stored samples of cotton fibre, cotton seed, oranges, carrots and soil. No significant decrease of triazophos was observed in analytical samples of cotton fibre, cotton seed, oranges and carrots stored at ≤ -18 °C for up to 24 months.

Residue definition in Plants

The results of the plant metabolism studies on cotton and rice, including foliar application and uptake from soil and water, indicate that parent triazophos is the major component of the recovered radioactivity, with the P=O analogue (O, O-diethyl-O-l-phenyl-lH-l, 2, 4-triazol-3-yl phosphate) and 1-phenyl-3-hydroxy-l, 2, 4-triazole also being present.

Analytical methods for plant matrices determine triazophos only.

On the basis of the metabolism in plants and the analytical methodology submitted, the Meeting confirmed the previous residue definition for the purposes of compliance monitoring and for estimation of dietary intake.

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

Results of supervised trials on crops

Cotton

Data were received from ten field trials for triazophos on cotton; nine trials were conducted in nine regions of India and a single trial was conducted in Brazil. In 2005 in India, 5 sprays were applied at 0.87 kg ai/ha (or 0.435 kg ai/hL) and in Brazil in 2001, 3 sprays were applied at 0.80 kg ai/ha (i.e., 0.27 kg ai/hL) or 1.60 kg ai/ha (i.e., 0.53 kg ai/hL). The GAP in India is 0.63 to 0.84 kg ai/ha with a 21 day PHI and 1 – 5 applications. In Brazil, the GAP is 0.3 to 0.8 kg ai/ha with a 28 day PHI and 1 – 3 applications.

The Meeting was informed that in India, as cotton plants do not mature simultaneously, harvest usually occurs over three separate picks, with the majority of cotton collected from first two with an average interval of 10 days between these picks. The raw cotton from, the different picks, is generally pooled prior to sale. Following sale the raw cotton is ginned where the separation of lint and seed occurs. As a consequence the Meeting considered the supervised trials reported from India as representing local practice for the use of triazophos in cotton. In trials from India the cotton samples from two picks (at 21 – 23 days and 31 – 33 days after the last application) were pooled and processed by ginning to separate the lint and seeds. Cotton seed oil was then extracted from cotton seeds using n-hexane in soxhlet extractor, the solvent was removed by rotary evaporation with the resultant oil used for analysis.

Residues of triazophos measured in nine trials conducted according to the GAP in India were 0.020, 0.021 (2), 0.023, 0.028, 0.042, 0.054, 0.059 and 0.060 mg/kg in cotton seed and 0.042, 0.044, 0.085, 0.088, 0.13, 0.17, 0.26, 0.31 and 0.78 mg/kg in cotton seed oil.

From one trial matching GAP in Brazil the residue of triazophos in cotton seed was 0.03 mg/kg; residues in cotton seed oil were not determined. Residues from the trial conducted at double rate reached a maximum of 0.2 mg/kg in cotton seed.

Based on the 10 trials with GAP in India and Brazil, residues were 0.020, 0.021 (2), 0.023, 0.028, 0.03, 0.042, 0.054, 0.059 and 0.060 mg/kg in cotton seed and 0.042, 0.044, 0.085, 0.088, 0.13, 0.17, 0.26, 0.31 and 0.78 mg/kg in cotton seed oil.

The Meeting estimated an STMR of 0.029 mg/kg for triazophos in cotton seed and 0.13 mg/kg in cotton seed oil, and an HR of 0.060 mg/kg for triazophos in cotton seed and 0.78 mg/kg in cotton seed oil. The Meeting recommended a maximum residue level of 0.2 mg/kg in cotton seed and 1 mg/kg in cotton seed oil (crude) for triazophos.

The Meeting also recommended the withdrawal of the current MRL of 0.1 mg/kg for triazophos in cotton seed.

Soya bean

In six field trials conducted in Thailand during 1992 to 2006, 2 to 4 sprays of triazophos were applied at 0.1 kg ai/hL. The GAP in Thailand is 0.1 kg ai/hL with a 14 day PHI. Residues of triazophos in whole pod including immature seeds at 14 – 17 days after the last application were 0.05, 0.17, 0.31, 0.43, 0.52, and 0.60 mg/kg.

The Meeting recommended a maximum residue level of 1 mg/kg for triazophos in soya beans (immature seeds with the pod). The Meeting also estimated an STMR of 0.37 mg/kg and an HR of 0.60 mg/kg.

The Meeting recommended withdrawal of the previous recommendation of 0.05 mg/kg for triazophos in soya bean (dry).

Other commodities

No data on GAP and residues for triazophos was provided on broad bean (shelled), Brussels sprouts, cabbage (head), carrot, cauliflower, cereal grains, coffee beans, common bean, onion (bulb), pea, pome fruit, potato, strawberry and sugar beet. The Meeting recommended withdrawal of the previous recommendations made for these commodities.

Fate of residues during processing

Information regarding the magnitude of triazophos residues in different processed commodities of cotton was provided to the Meeting.

In three field trials conducted in the USA in cotton, residues were found in the processed non oily matrices and in the processed oil. No processing factors could be determined as residue concentrations in unprocessed cotton seed were not reported.

Residues in animal commodities

The Meeting received a feeding study on lactating Holstein cows. The dosing regime involved a 2 day pre-conditioning phase, one week prior to the dosing period, at a dose level of 100 mg triazophos per cow. During the following period of 7 days, one cow was dosed with 50 mg triazophos (2.38 ppm in the feed) and the second cow with 100 mg (4.76 ppm in the feed), the third cow received untreated feed. Neither the pre-conditioning at 100 mg per cow and day nor the dosing of 50 and 100 mg per cow and day resulted in any residues above the LOQ of 0.05 mg/kg for milk and 0.01 mg/kg for muscle, fat, kidney and liver. The Meeting noted that because of the lack of an appropriate livestock metabolism study, a residue definition for animal products could not be determined and therefore the Meeting could not make use of the results of the feeding study.

The Meeting agreed to withdraw the previous recommendations for triazophos of 0.01 mg/kg in cattle meat and cattle milk.

RECOMMENDATIONS

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

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

CCN	Commodity	Recommended MRL, mg/kg		STMR, mg/kg	HR, mg/kg
		New	Previous		
VP 0523	Broad bean, shelled (succulent) (=immature seeds)	W	0.02*		
VB 0402	Brussels sprouts	W	0.1		
VB 0041	Cabbages, Head	W	0.1		
VR 0577	Carrot	W	0.5		
MM 0812	Cattle meat	W	0.01*		
ML 0812	Cattle milk	W	0.01*		
VB 0404	Cauliflower	W	0.1		
GC 0080	Cereal grains	W	0.05*		
SB 0716	Coffee beans	W	0.05*		
VP 0526	Common bean (pods and / or immature seeds)	W	0.2		
SO 0691	Cotton seed	0.2	0.1	0.029	
OC 0691	Cotton seed oil, crude	1		0.13	
VA 0385	Onion, Bulb	W	0.05*		
VP 0063	Peas (pods and succulent = immature seeds)	W	0.1		
FP 0009	Pome fruits	W	0.2		
VR 0589	Potato	W	0.05*		
VD 0541	Soya bean (dry)	W	0.05*		
VP 0541	Soya bean (immature seeds) [a]	1		0.37	0.60
FB 0275	Strawberry	W	0.05*		
VR 0596	Sugar beet	W	0.05*		

a - with the pod

DIETARY RISK ASSESSMENT

Long-term intake

The evaluation of triazophos has resulted in recommendations for MRLs and STMR values for raw and processed commodities. Consumption data were available for 2 food commodities and were used

in the dietary intake calculations. The results of which are shown in Annex 3 of the 2007 Report of the JMPR.

The IEDIs for the 13 GEMS/Food regional diets, based on estimated STMRs were in the range 0 – 20% of the maximum ADI of 0.001 mg/kg bw. The Meeting concluded that the long-term intake of residues of triazophos from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The IESTI for triazophos was calculated for the food commodities for which maximum residue levels and HRs were estimated and for which consumption data were available. The results are shown in Annex 4.

The IESTI calculated for cotton seed oil for the general population and children were 2% and 5% of the ARfD (0.001 mg/kg bw), respectively. The IESTI calculated for soya bean (immature seeds with the pod) for the general population and children were 140% and 230% of the ARfD, respectively (Annex 4 of the 2007 JMPR Report). The Meeting concluded that the short-term intake of residues of triazophos from the consumption of cotton seed oil is unlikely to present a public health concern. The information provided to the JMPR precludes an estimate that the dietary intake would be below the ARfD for consumption for soya bean (immature seeds with pod) by the general population and children.

The Meeting noted that the pod is not normally consumed and that no residue data relating to residues in the edible portion of soya bean pods or alternative GAP were submitted for soya bean (immature seeds with pod).

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