PYRIMETHANIL (226)

First draft was prepared by Mr Stephen Funk, Health Effects Division, US Environmental Protection Agency, Washington, DC, USA

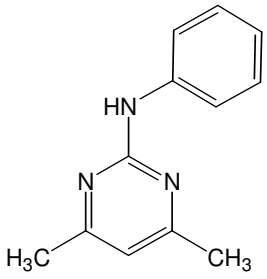
EXPLANATION

Pyrimethanil is an anilinopyrimidine fungicide that inhibits the secretion of hydrolytic enzymes by the fungi that are needed during the infection process. Pyrimethanil blocks the ability of fungi to degrade and digest the plant tissues, thus stopping penetration and development of the disease.

At the 37th session of the CCPR (ALINORM 04/27/24) pyrimethanil was listed as a candidate for evaluation of a new compound by the 2007 JMPR.

The sponsors (Bayer Crop Science and Janssen Pharmaceutical NV) have provided studies on physical and chemical properties, formulations, metabolism in plants (apples, grapes, carrots, tomatoes, lettuce, strawberries), metabolism in livestock (lactating cow), environmental fate (aerobic degradation in soil, anaerobic degradation in soil, hydrolysis in water, photolysis in water, residues in succeeding crops, confined and field), analytical methods, storage stability, supervised trials (citrus, apples, pears, apricots, cherries, peaches, plums, grapes, strawberries, onions, tomato, lettuce, beans, carrot, potatoes, almonds), processing (citrus, apples, plum, grapes, tomatoes, beans, carrots) and livestock feeding (lactating dairy cattle). Additionally, the sponsors provided GAP information and labels. The Government of Australia (Queensland) also supplied information on pyrimethanil.

IDENTITY

ISO common name:	pyrimethanil
IUPAC name:	N-(4,6-dimethylpyrimidin-2-yl) aniline
Chemical Abstract name:	4,6-dimethyl-N-phenyl-2-pyrimidinamine
Others:	2-anilino-4,6-dimethylpyrimidine
CAS No.:	53112-28-0
CIPAC No.:	714
EEC No.:	414-220-3
Manufacturer's experimental name:	AE B100309, SN 100309, ZK 100309
Synonyms used in submission:	R215559, PH066
Molecular Formula:	$C_{12}H_{13}N_3$
Structural Formula:	
Molecular Weight:	199.28 g/mol
Minimum purity	975 g/kg

PHYSICAL AND CHEMICAL PROPERTIES

Pure active ingredient

Physical-Chemical Properties	Results	Reference
Appearance	Almost odourless, off-white to light beige or light yellow crystalline powder	A81564 Steib, 1991
Melting point	96.3 °C	A81566 Lehne, 1991
Relative density at 20 °C	1.15 g/mL	A81612 Steinke, 1993
Vapour pressure	°C — Pa 20 — 1.1×10^{-3} 25 — 2.2×10^{-3} 30 — 3.6×10^{-3}	A81555 Miklautz, 1992
Volatility, Henry's Law Constant at 25 °C	3.6×10^{-3} Pa m^3 mol^{-1}	A81593 Schneider and Miklautz, 1993
Partition coefficient n-octanol/water	Log K_{OW} = 2.84 (shaking flask method)	A81558 Brehm and Miklautz, 1989
Hydrolytic stability (DT50, 22 °C)	pH — DT_{50} 5 — No decay 7 — 2.7 years 9 — 1.9 years	A81864 Tschampel, 1989
Dissociation constant	pKa = 3.52 at 20 °C	A81572 Miklautz, 1991
Solubility in water	pH 4.2 — 0.16 g/L at 20 ºC 6.1 — 0.12 g/L at 25 ºC 9.9 — 0.099 g/L at 20 ºC	A81557 Miklautz, 1989a A81569 Muller, 1991
Solubility in organic solvents	Solvent — g/L at 20 ºC n-hexane — 24. Toluene — 410. DICHLORMETHANE — 1000. Methanol — 180. Acetone — 390. Ethyl acetate — 620.	A81561 Miklautz, 1989b

Technical Material

No information provided. Pyrimethanil has not been considered for a specification by the FAO/WHO JMPS.

FORMULATIONS

Various formulations are available either for pre-harvest foliar application or for post-harvest commodity treatments.

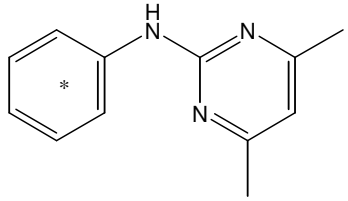
Formulation type	Active substance and content	Trade Name
Suspension concentrate (SC), containing only pyrimethanil as the active ingredient	300 g/L pyrimethanil	Mythos
	400 g/L pyrimethanil	Scala Penbotec 400 SC Sari TF Soleas PH066 [a]
	600 g/L pyrimethanil	Scala Siganex
Suspension concentrate (SC), containing a mixture of pyrimethanil and other active ingredients	200 g/L pyrimethanil + 200 g/L imazalil	Philabuster 400 SC LAg2002258 [a]
	50 g/L fluquinconazol + 200 g/L pyrimethanil	Vision
	50 g/L fluquinconazol + 150 g/L pyrimethanil	Clarinet
	150 g/L chlorothalonil + 375 g/L pyrimethanil	Walabi
	42 g/L prochloraz copper chloride + 16.7 g/L flutriafol + 42 g/L pyrimethanil	Rubin
Emulsifiable concentrate (EC) containing a mixture of pyrimethanil and other active ingredients	100 g/L pyrimethanil + 200 g/L imazalil	LAg2001334 [a]
	150 g/L pyrimethanil + 130 g/L imazalil	LAg2001206 [a]
Formulation for use in thermofogging equipment (HN)	160 g/kg pyrimethanil	Xedathane-A
	120 g/L pyrimethanil + 120 g/L imazalil	Thamsil

a - Company code names used in some studies

METABOLISM AND ENVIRONMENTAL FATE

The fate and behaviour of pyrimethanil in animals, plants, water and soils were investigated using either U-(^{14}C)-anilino labelled or 2-(^{14}C)-pyrimidinyl-labelled pyrimethanil:

[U-^{14}C]-anilino-labelled pyrimethanil or UL-^{14}C-phenyl-pyrimethanil



2-[^{14}C]-pyrimidinyl-labelled pyrimethanil or 2-^{14}C-pyrimidinyl-pyrimethanil

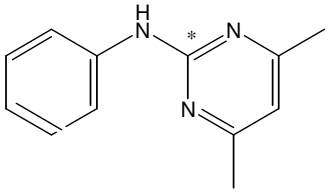
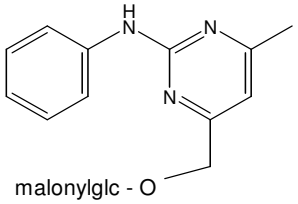
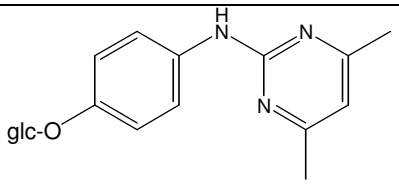
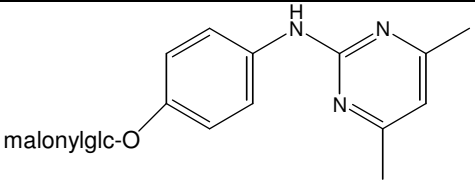
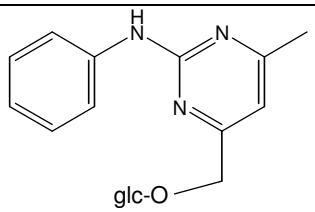
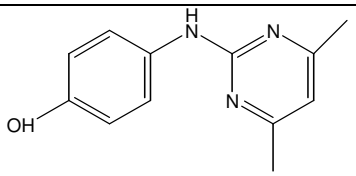
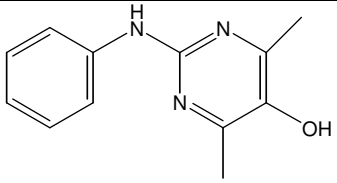
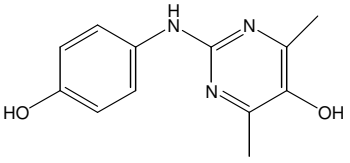
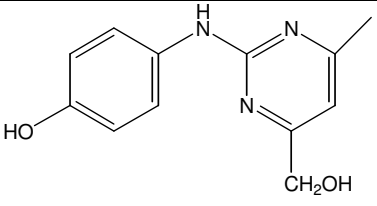
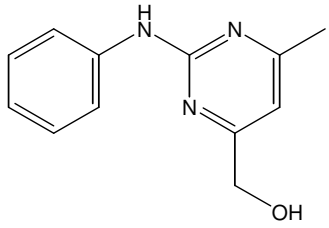
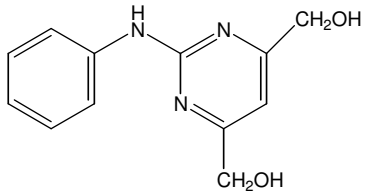
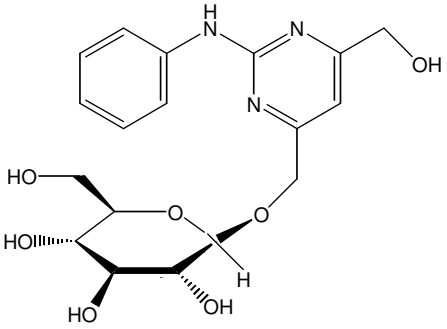
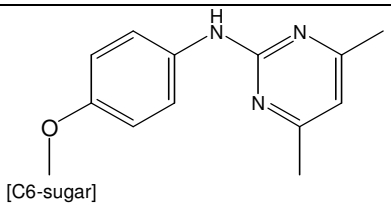
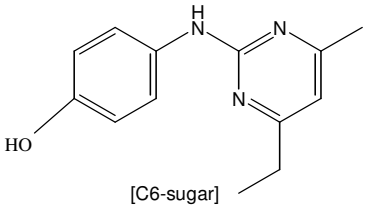
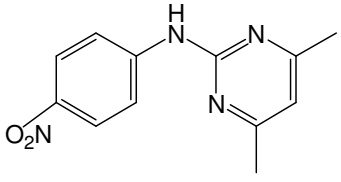
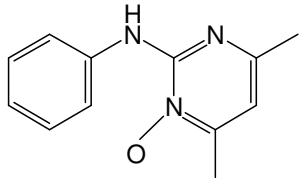
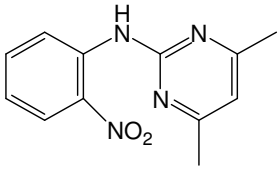
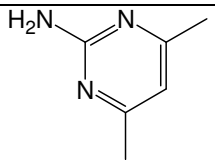
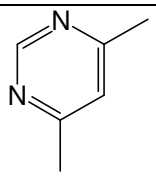
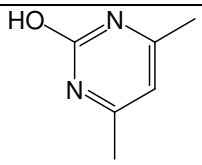


Table 1 summarizes the names, codes and structures of the principle metabolites found in plant, livestock and environmental fate studies.

Table 1. Metabolites/Degradates of Pyrimethanil

Compound Name	Structure	Found in:
Malonyl-beta-O-glucoside of 2-anilino-4-hydroxymethyl-6-methylpyrimidine		Plants
ß-O-glucoside of 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine		Plants
Malonyl- ß-O-glucoside of 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine		Plants
ß-O-glucoside of 2-anilino-4-hydroxymethyl-6-methylppyrimidine (U2/M5)		Plants
2-(4-hydroxyanilino)-4,6-dimethylpyrimidine [AE C614276], [SN 614276] [AN2]		Plants Animals (main metabolite in kidney) Rotational Crops
2-anilino-4,6-dimethylpyrimidin-5-ol [AE C614277], [SN614277], [AN3]		Plants Animals (main metabolite in milk) Rotational crops
2-(4-hydroxyanilino)-6-dimethyl-pyrimidin-5-ol [SN 615224]		Rat
2-(4-hydroxyanilino)-4-hydroxymethyl-6-methylpyrimidine [SN 614800/ AE C614 800] [AN6]		Animals Rotational Crops

Compound Name	Structure	Found in:
2-anilino-6-methylpyrimidine-4-methanol [SN 614278/ AE 614278] [AN4]		Plants Animals Rotational crops
2 anilino-4,6-di(hydroxymethyl)pyrimidine [AE C621312] [AN5]		Soil Plant Main metabolite in rotational crops
ß-O-glucoside of 2-anilino-4-hydroxymethyl-6-hydroxymethylpyrimidine (U1)		Plants
C-6 sugar of 2-(4-hydroxyanilino)-4,6-dimethylpyridine (M1)		Plants
C-6 sugar conjugate of 2-(4-hydroxyanilino)-4-methyl-6-hydrooxymethylpyridine (U3/M4)		Plants Animals
4,6-dimethyl-2-(4-nitroanilino)-pyrimidine (SN 617 916)		Soil
2-anilino-4,6-dimethylprimidine-1-oxide (SN 603 193)		Soil

Compound Name	Structure	Found in:
4,6-dimethyl-2-(2-nitroanilino)-pyrimidine (AZ 196 920)		Soil
2-amino-4,6-dimethylpyrimidine [SN 512 723, AE F132593] [AN7]		Soil, Plant Rotational crops
4,6-dimethylpyrimidine AE 0025462 [AN8]		Soil Plant Rotational crops
2-hydroxy-4,6-dimethyl-pyrimidine (SN 469 626; AE F132512] [AN9]		Soil Plant Rotational Crops

Animal metabolism

Lactating dairy cows

The Meeting received a report on the metabolism, distribution and elimination of [^{14}C]-labelled pyrimethanil in lactating dairy cows (A81627, Reynolds, 1993). A lactating dairy cow was orally dosed for seven consecutive days with (^{14}C)-pyrimethanil at a daily dose rate of 10 ppm in the diet which corresponds to 0.4 mg/kg bw/day for a 600 kg cow, administered as 2 capsules per day with 124 mg active ingredient per capsule. The position of the radiolabel was not specified in the report.

Milk was collected twice daily (approximately 07:30 and 16:00 h). Urine and faeces were collected for each 24 h period; and blood samples were obtained prior to dosing and at various intervals after the first administration and at slaughter.

At sacrifice, within 24 h of the final dosing, liver, kidney, heart, lungs, spleen, muscle and renal fat were sampled. All tissues were minced and stored at -20 °C until analysis. Within 28 days of necropsy an initial analysis of all edible tissues and milk had been completed. Initial analysis involved freeze-dried tissues subjected to soxhlet extraction with organic solvents. All extracts were quantified by HPLC and the profiles of the metabolites were obtained by TLC. Residues in milk and tissues were minimal. In milk, total radioactive residues reached a plateau after about 119 h (5 days) (Table 2) with residues at 0.069 mg/L. In tissues, total radioactive residues ranged from 0.017 mg/kg in muscle to 0.363 mg/kg in liver (Table 3).

Low residue levels precluded identification of residues in muscle and fat. No quantifiable residues of parent were found in any matrix. In kidney, SN 614 276 (AE 614 276) was the primary metabolite identified (46% of the TRR). In milk SN 614 277 was the main metabolite (64% TRR). Minor amounts of SN 614 800 (AE 814 800) and SN 614 277 (SN 614 277) were also identified in the kidney. Results are summarized in Table 4.

Extraction of liver samples with various organic solvents resulted in poor release (26%). Liver was fractionated sequentially with 2% perchloric acid (proteins), ethanol/diethyl ether (lipids), ribonuclease (ribonucleic acid), alkaline hydrolysis (sulphurated glycoaminoglyucans), glucosidase (carbohydrates) and perchloric acid incubation (DNA).

Table 2. Total radioactive residues in whole milk as a function of time from the oral administration of 2-[^{14}C]-pyrimidinyl-labelled pyrimethanil to a lactating dairy cow at a rate of 0.4 mg/kg bw/day (A81627)

Time (hours)	Residue (ug/mL)
Pre-dose	0.0006
0.08	0.0007
7	0.0305
23	0.0494
31	0.0438
47	0.0645
55	0.0615
71	0.0506
79	0.0315
95	0.0518
103	0.0604
119	**0.0688**
127	0.0474
143	0.0094
151	0.0412
167	0.0550

Table 3. Total radioactive residues in tissues, feces and fluids from the oral administration of 2-[^{14}C]-pyrimidinyl-labelled pyrimethanil to a lactating dairy cow at a rate of 0.4 mg/kg bw/day for seven days (A81627)

Matrix	Residue (mg/kg)
Urine (total collected)	136.
Urine at peak (72 hrs)	28.
Faeces	Not reported
Muscle	0.017
Renal fat	0.036
Kidney	0.249
Liver	0.363
Milk (119 hrs)	0.0688
Bile	1.77
Rumen fluid	0.196
Blood	0.034

Table 4. Distribution of parent and metabolites in livestock matrices when dosed with 2-[^{14}C]-pyrimidinyl-labelled pyrimethanil (A81627)

Metabolite fraction	Urine		Muscle (TRR= 0.017 mg/kg)		Fat (TRR= 0.036 mg/kg		Kidney (TRR = 0.249 mg/kg		Liver (TRR = 0.363 mg/kg)		Milk (TRR = 0.062 mg/kg)	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Total extractable	100	-	53.	0.009	77.	0.03	91.	0.23	28.	0.10	92.	0.057
Pyrimethanil	0		0		0		0		0		0	
AE 614 276	69.	-	-	-	-	-	46.	0.12	-	-	?.[a]	-
AE 614 800	6.2	-	-	-	-	-	6.8	0.017	-	-	-	-
AE 614 277	7.1	-	-	-	-	-	5.4	0.013	-	-	64[1]	0.04
Protein	-	-	-	-	-	-	-	-	48.	0.18	-	-
Lipids									9.1	0.033		
Ribonucleic acid									6.7	0.024		
Sulphurated glycoamino-glycans									6.0	0.022		

Metabolite fraction	Urine		Muscle (TRR= 0.017 mg/kg)		Fat (TRR= 0.036 mg/kg		Kidney (TRR = 0.249 mg/kg		Liver (TRR = 0.363 mg/kg)		Milk (TRR = 0.062 mg/kg)	
	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg	%TRR	mg/kg
Highly-polar metabolites	18.	-	-	-	-	-	32.	0.080	-	-	27	0.016
Unidentified	18	-	100	0.017	100	0.036	42	0.104	100	0.363	36	0.040
Unextractable	-	-	47	0.008	24.	0.008	9.3	0.023	7.8	0.03	8.1	0.005

a - The techniques used (primarily TLC) did not separate AE 614 276 from AE 614 277. The livestock feeding study (B0003807) showed that the metabolite was most likely AE 614 277.

The proposed scheme for the metabolism of pyrimethanil in the cow is given in Figure 1.

The Meeting noted that metabolism in the rat (AB81626, Needham and Hemmings, 1993, Report No. TOX/93/223-70) was quite similar to that described above for the cow. In the rat, only small amounts of the administered pyrimethanil were found in faeces, and none was found in urine. The major metabolite in urine and faeces was 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine (SN 614 276) and its sulfate, 13% – 52%. Other metabolites, generally < 10% of total extracted radioactivity in the excreta, were 2-anilino-4,6-dimethylpyrimidin-5-ol, 2-(4-hydroxyanilino)-4-hydroxymethyl-6-methylpyrimidine, 2-(4-hydroxyanilino)-6-dimethyl-pyrimidin-5-ol, and 2-anilino-6-methylpyrimidine-4-methanol. Thus, the metabolites found in rat urine, faeces and blood are consistent with the metabolic pathway of Figure 1.

Metabolism in Poultry

The Meeting did not receive information on the metabolism of pyrimethanil in poultry.

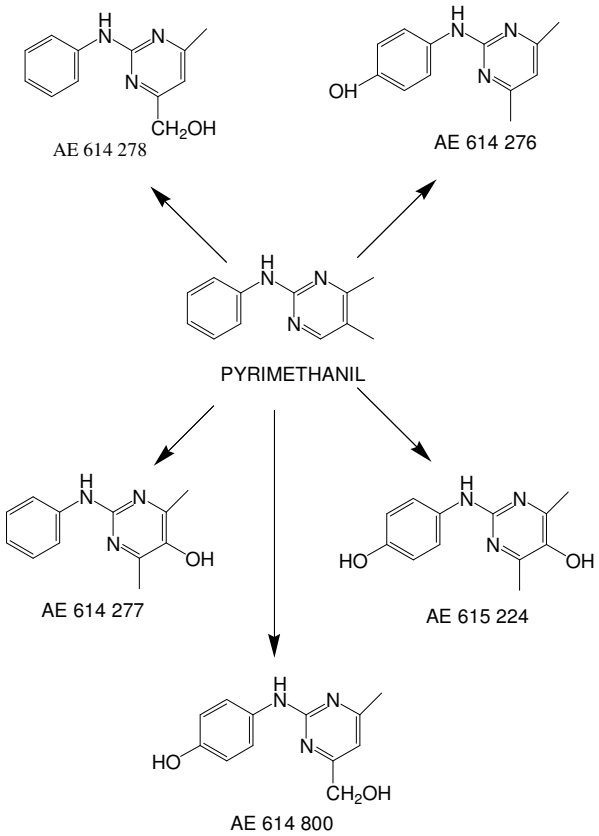


Figure 1. Proposed Scheme for the Metabolism of Pyrimethanil in the Cow[19]

[19] Metabolites AE 614 278 and AE 615 224 are postulated, but not found in the cow metabolism study. These metabolites were found in the rat metabolism study.

Plant Metabolism

The Meeting received a study report on the metabolism of pyrimethanil in/on apples (A81633, Leinhase, *et al*., 1993; Report No. : U/R 09/93). Twelve small apple trees (10 apples and 100 leaves) were each treated 4 times with either 2-(U-^{14}C)-anilino or 2-(^{14}C)-pyrimidinyl labelled pyrimethanil, at an application rate of 33 mg ai per plant as an SC formulation, corresponding to a total application of 82 g ai/ha. Leaves and fruits were treated at the start of ripening (fruit diameter of 20 – 30 mm; start of red pigmentation). Fruits and leaves were harvested at fruit maturity, six weeks after the last treatment.

All samples were washed with dichloromethane and remaining residues extracted with a chloroform/methanol/water mixture. After alkali treatment, the methanol/water layer was further extracted with chloroform. All extracts were analysed using LSC and TLC. For characterisation, the TLC phases of the leaf samples were eluted from the plates and further purified by HPLC. The identification of the metabolites was done by MS and NMR.

Total radioactive residues ranged from 8.8 mg/kg to 14 mg/kg in the fruits and from 54 mg/kg to 63 mg/kg in the leaves. Of the recovered radioactivity, 41 to 45% was present in apple flesh and 48% in the peel. The major part of the residues could be extracted (18 to 19%) in the surface wash from apples and 71 to 74% in the fruit extracts; 41 to 44% in the surface wash of leaves and 51 to 53% in the leaf extracts. The results are summarized in Table 5.

Unchanged pyrimethanil accounted for 70 to 77% of the radioactivity in the fruit and 55 to 61% in the leaves. All metabolites were present in minor amounts (< 10%) in the fruit and leaves, except for one, U1, in the leaves (15 – 16%), which was identified as ß-O-glucoside of 2-anilino-4-hydroxymethyl-6-hydroxymethylpyrimidine. The remainder were *assumed* to be hydroxylated or conjugated derivatives of pyrimethanil. Use of either labelled compound produced similar results, indicating that cleavage of the amine bond between the aromatic rings did not occur.

Table 5. Residue levels, extraction profile and metabolism pattern of pyrimethanil residues in apple fruits and leaves after application of 2-(U-^{14}C)-anilino or pyrimidinyl-2-(^{14}C) labelled pyrimethanil, 6 weeks after the final treatment (averaged over three plants) (A81633)

Label	TRR mg/kg [a]	Extractable residues [c] % mg/kg	Unextractable residue % [b] mg/kg	Parent % [b] mg/kg	U1[d] % [b] mg/kg	U2[e] % [b] mg/kg	U3[e] % [b] mg/kg	U4[e] % [b] mg/kg	Ux[f] % [b] mg/kg
Apple fruits									
2-(U-^{14}C)- anilino	14	93 13	7 0.98	77 11	1.5 0.21	2.4 0.34	1.1 0.15	3.4 0.48	1.5 0.21
Pyrimidinyl-2-(^{14}C)	8.8	89 7.8	11 0.97	70 6.2	1.5 0.13	1.7 0.15	2.2 0.19	3.3 0.29	2.5 0.22
Apple leaves									
2-(U-^{14}C)- anilino	63	93 58	6.7 4.2	61 38	15 9.4	7.5 4.7	2.4 1.5	0.6 0.38	2 1.3
Pyrimidinyl-2-(^{14}C)	54	95 51	4.9 2.6	55 30	16 8.6	6.9 3.7	2.1 1.1	0.6 0.32	2.6 1.4

a - total radioactive pyrimethanil residues, as the sum of various extracts and residuals.

b - percent of *recovered* radioactivity

c - including surface wash

d - ß-O-glucoside of 2-anilino-4-hydroxymethyl-6-hydroxymethylpyrimidine

e - hydroxylated and conjugated derivatives of pyrimethanil (not further specified)

f - unidentified minor components

A study was received by the Meeting on the metabolism of pyrimethanil in/on grape vines (A81628, Feyerabend, 1991; Report No. UPSR 43/91). Two grape vines were each treated twice with 2-U-(^{14}C)-anilino-labelled pyrimethanil at the rate of 200 mg ai/plant/application as a WP formulation. Treatments of leaves and fruits started at the fruit ripening stage. Application was made using automatic pipettes, the formulation being spread as evenly as possible in small droplets over the surface of the plant material by the plastic tip.

Fruits and leaves were collected 21 days after the last treatment. All samples were washed with dichloromethane and residual residues were extracted with chloroform/methanol/water and chloroform/water mixtures. All extracts were combined and separated in order to produce 2 phases: a methanol/water phase and a chloroform phase. All extracts were analysed using LSC, TLC and HPLC. Bound residues were subjected to toluene soxhlet extraction for 24 h and treated with 5N HCl and 0.1 N NaOH.

At harvest, the total radioactive residues were 30 mg/kg in grapes and 23 mg/kg in leaves. The major part of the radioactive residues in grapes were present in the surface wash (56%); 40% of these residues could be extracted and 3.6% remained as bound residues. Identification of radioactivity in the grapes showed 91% TRR to be unchanged parent compound. Unidentified metabolites (M1, M2, M3, and M4) were determined to be < 1% each of recovered radioactivity.

In the leaves, only 23% of the radioactivity could be found in the surface wash; 67% of the radioactivity could be extracted, with 10% remaining as bound residues. Of the total radioactivity recovered in the leaves, 31% were shown to be the parent compound. One metabolite (M1), identified to be the C-6 sugar of 2-(4-hydroxyanilino)-4,6-dimethylpyridine, was present at 17% of the recovered radioactivity. All other metabolites (glucose and other C-6 sugar conjugates of the hydroxylated parent compound) were in the range of 1.9 to 2.8%. The residues in the leaves were further extracted and shown to consist of highly polar compounds and pyrimethanil. The results are summarized in Table 6.

Table 6. Residue levels, extraction profile and characterisation/identification on grapes and vines after application of 2-U-(^{14}C)-anilino- labelled pyrimethanil, 21 days after the final treatment (A81628)

TRR mg/kg [a]	Surface wash %[b]/ mg/kg	Extractable residues %[b]/ mg/kg	Unextracted residues %[b]/ mg/kg	Pyrimethanil %[b]/ mg/kg	M1 [c] %[b]/ mg/kg	M2 [c] %[b]/ mg/kg	M3 [c] %[b]/ mg/kg	M4 [c] %[b]/ mg/kg
Grapes								
29.5	56/ 17	40/ 12	3.6/ 1.1	91/ 27	0.6/ 0.18	0.4/ 0.12	0.3/ 0.09	0.1/ 0.03
Leaves								
23.3	23/ 5.4	67/ 16	18./ 4.2	31/ 7.2	17/ 3.9	2.8/ 0.65	2.8/ 0.65	1.9/ 0.44

a - total radioactive pyrimethanil residue, as the sum of various extracts and residuals

b - percent of *recovered* radioactivity (100%).

c - not identified

The Meeting received a study report on the metabolism of pyrimethanil in/on carrots (C010122, Goodyear, 2000; Report No. CLE 194/206-D2142). The metabolism of (^{14}C)-pyrimethanil was investigated in carrots following soil and foliar treatment with 2-(^{14}C) pyrimidinyl-labelled pyrimethanil. Soil (post emergence) and foliar treatments with a suspension formulation (SC, 400 g ai/L) were made at BBCH 43 and BBCH 47 according to the following scheme:

Treatment	Rate 1st treatment (kg ai/ha)	Rate 2nd treatment (kg ai/ha)	Number of containers treated
Soil	0.77	0.99	2
Foliar	0.77	0.99	2
Foliar	2.44	2.90	2
Control	0	0	1

The plants were grown outside in a caged enclosure. Samples were collected at 1 and 21 days after *each* treatment, with the final sample taken at maturity (BBCH 49). Roots were separated from foliage.

Foliage samples were first rinsed with dichloromethane and both foliage and carrot root samples were homogenized and extracted sequentially with chloroform/methanol and methanol/water. Total radioactivity in extracts was determined by LSC. Efforts at combustion and LSC of the foliage and roots gave variable results and were not reported. The radioactivity recovered in the dichloromethane, chloroform/methanol and methanol/water extracts were analysed by HPLC and TLC. The residues in the foliage and roots remaining after the first extraction steps were further extracted by sequential refluxing with water, hydrochloric acid and sodium hydroxide. The methanol/water fraction from the foliar sample taken 21 days after final treatment was subjected to ß-glucosidase treatment and analysed by HPLC and TLC.

Total radioactive residues immediately after the application ranged from 0.36 – 0.44 mg/kg (roots) and from 26.5 to 52.8 mg/kg (foliage). Twenty-one days after the final foliar application, residues in the roots ranged from 0.44 to 0.83 mg/kg and in the foliage, from 5.14 to 12.2 mg/kg. Soil treatment showed lower values of 0.18 to 0.23 mg/kg in roots and 0.3 to 0.89 mg/kg in foliage, 21 days after treatment.

About 82 to 99% of the recovered radioactivity was found in the extracts (including surface wash of the foliage samples). The parent compound, pyrimethanil, accounted for most of the radioactivity (46 to 98%). The only metabolite present at > 10% of the total radioactivity was "UNK A", which was identified as the malonyl-ß-O-glucoside of 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine (maximum of 167% of recovered radioactivity in foliage samples taken 21 days after treatment). Other metabolites, "UNK B", "UNK C" and "UNK F", which were identified as conjugated compounds of the hydroxylated parent, were only present in minor quantities (< 10% of recovered radioactivity). These identifications were assigned based on HPLC and MS. The results are summarized in Tables 7 and 8.

Table 7. Residue levels, extraction profile and characterisation/identification after foliar treatment of carrots with 2-(^{14}C) pyrimidinyl-labelled pyrimethanil (C010122)

Harvest [Days after treatment]	Total radio-active residues mg/kg [a]	Extractable residues % [b] mg/kg	Unextracted residues % [b] mg/kg	Pyrimethanil % [b] mg/kg	UNK A [d] mg/kg	UNK B [e] mg/kg	UNK C [f] mg/kg	UNK F [g] mg/kg
Roots								
1 day after 1st applic	0.44	93 0.41	6.8 0.030	89 0.39	-	-	-	-
21 days after 1st applic	0.44	87 0.38	13 0.057	78 0.34	-	-	-	-
1 day after 2nd applic	0.36	93 0.33	7.2 0.026	87 0.31	0.8 0.003	-	-	0.3 0.001
21 days after 2nd applic	0.83	90 0.75	10 0.083	86 0.71	-	-	-	-

Harvest [Days after treatment]	Total radio-active residues mg/kg [a]	Extractable residues % [b] mg/kg	Unextracted residues % [b] mg/kg	Pyrimethanil % [b] mg/kg	UNK A [d] mg/kg	UNK B [e] mg/kg	UNK C [f] mg/kg	UNK F [g] mg/kg
Foliage								
1 day after 1st applic	26.	99 [c] 26	0.7 0.18	98 25	0.2 0.052	0.1 0.026	-	0.1 0.026
21 days after 1st applic	5.1	85 [c] 4.3	15 0.76	46 2.3	14 0.71	6.4 0.33	2.0 0.10	7.6 0.39
1 day after 2nd applic	53.	98 [c] 52	1.9 1.0	93 49	2.0 0.11	0.7 0.37	0.2 0.11	0.8 0.42
21 days after 2nd applic	12.	86 [c] 10	14 1.7	48 5.8	16 1.9	5.6 0.67	2.2 0.26	5.7 0.68

a - total radioactive pyrimethanil residues, i.e., sum of extracts and residuals

b - percent of recovered radioactivity

c - extractable residues in foliage samples, including surface wash

d - malonyl-ß-O-glucoside of 2-anilino-hydroxymethyl-6-methylpyrimidine

e - ß-O-glucoside of 2-(4-hydroxyaniline)-4,6-dimethylpyrimidine

f - malonyl- ß-O-glucoside of 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine

g - ß-O-glucoside of 2-anilino-4-hydroxymethyl-6-methylpyrimidine

Table 8. Residue levels, extraction profile and characterisation/identification after soil treatment of carrots with 2-(^{14}C) pyrimidinyl-labelled pyrimethanil (C010122)

Harvest [Days after treatment]	Total radioactive residues mg/kg [a]	Extractable residues % [b] mg/kg	Bound residues % [b] mg/kg	Pyrimethanil % [b] mg/kg	UNK A [c] mg/kg	UNK B [d] mg/kg	UNK C [e] mg/kg	UNK F [f] mg/kg
Roots								
21 days after 1st applic	0.23	95 0.22	4.6 0.010	83 0.19	0.3 < 0.001	0.6 0.001	0.2	0.1
21 days after 2nd applic	0.18	85 0.15	15 0.027	70 0.13	1.3 0.002	1.0 0.002	1.2	0.6
Foliage								
21 days after 1st applic	0.3	87	13	75	3.6 0.011	0.7 0.002	0.7 0.002	1.2 0.004
21 days after 2nd applic	0.89	88	18	53	7.3 0.065	1.9 0.017	1.9 0.017	2.8 0.025

a - total radioactive pyrimethanil residues,, i.e., sum of extracts and residuals.

b - percent of recovered radioactivity

c - malonyl-ß-O-glucoside of 2-anilino-hydroxymethyl-6-methylpyrimidine

d - ß-O-glucoside of 2-(4-hydroxyaniline)-4,6-dimethylpyrimidine

e - malonyl- ß-O-glucoside of 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine

f - ß-O-glucoside of 2-anilino-4-hydroxymethyl-6-methylpyrimidine

The Meeting received a study report on the metabolism of pyrimethanil in/on tomato plants (A91817, Baumecker *et. al.*, 1998; Report No. CM95/038). Tomato plants grown in a climatic chamber were treated 4 times (at 7 day intervals) with U-(^{14}C)-anilino-labelled (six plants) or 2-(^{14}C)-pyrimidinyl-labelled pyrimethanil (six plants), formulated as an SC, at the rate of 8000 mg ai/L or 40 mg ai/plant per application. The plants were grown in hydroponic cultures. The first application took place at the start of fruit ripening. Fruits and leaves were harvested immediately following every treatment, and the final sampling was performed 29 days after the first application, or 8 days after the final application.

Samples of fruits and leaves were first washed with dichloromethane. Tomatoes were then separated into pulp and peel and each part extracted separately with chloroform/methanol/ water or

methanol/water mixture, and the extracts quantified by LSC and TLC. HPLC/MS and NMR were used for isolation and characterisation of metabolites. Extracts were subjected to enzymatic hydrolysis using α-glucosidase, β-glucosidase, α-amylase and amyloglucosidase.

At harvest (8 days after the last treatment), the total radioactive residues in tomato fruits ranged from 59 to 61 mg/kg and in tomato leaves from 790 to 2800 mg/kg. The majority of the radioactivity in the fruit remained in the dichloromethane surface wash (80 to 95% of the recovered radioactivity). The pulp contained 0.88 to 4.6% of the recovered radioactivity. The unextracted residues were in the range of 0.21 to 0.23% (fruits) and 0.66 to 1.0% (leaves). The main residue in the fruit and leaves was found to be unchanged pyrimethanil (97% of the fruit radioactivity; 95 to 96% of the leaf radioactivity). All metabolites were < 1.1% of the recovered radioactivity. Metabolites T1-T3 isolated from the methanol/water phase of leaf samples were shown to be conjugates with C6-sugars and disaccharides. Treatment with β-glucosidase resulted in formation of hydroxylated compounds of pyrimethanil. The 2-(^{14}C) pyrimidinyl-labelled pyrimethanil and the U-(^{14}C)-anilino-labelled pyrimethanil results were the same in terms of extraction profiles and qualitative/quantitative identification of metabolites. The results are summarized in Tables 9 and 10.

Table 9. Residue levels, extraction profile and characterisation/identification after foliar treatment of tomato plants with 2-(^{14}C) pyrimidinyl-labelled pyrimethanil (A91817)

Time of harvest [days after treatment]	Total radioactive residues mg/kg [a]	Surface wash % [b] mg/kg	Extractable residues % [b] mg/kg	Unextracted residues % [b] mg/kg	Pyrimethanil % [b] mg/kg	T1 [c] % mg/kg	T2 [c] % mg/kg	T3 [c] %/ mg/kg	T4 [d] % mg/kg	Tx [e] % mg/kg
Tomato fruits										
Day 0 after applic [f]	700	97	- [g]	3.4	- [h]	- [h]	- [h]	- [h]	- [h]	- [h]
8 days after last applic	61 (37 – 82)	91	7.2 4.4	0.23 0.14	97 59	0.2 0.12	0.36 0.22	0.27 0.16	1.1 0.67	0.16 0.10
Tomato leaves										
Day 0 after applic [f]	11000	97	- [g]	2.6 290	- [h]	- [h]	- [h]	- [h]	- [h]	- [h]
8 days after last applic	790. (700; 890)	67	32 250	1.0 7.9	96 760	0.51 4.0	0.1 0.79	0.08 0.63	0.1 0.79	0.53 4.2

a - total radioactive pyrimethanil residues

b - percent of recovered radioactivity

c - T1-T3 are hydroxylated and conjugated compounds of the active substance

d - Not identified

e - Tx- minor components that have not been isolated or characterised.

f - After 4th treatment

g - Extraction not performed for day 0 samples

h - Characterisation of the residual radioactivity has not been performed on these samples.

Table 10. Residue levels, extraction profile and characterisation/ identification after foliar treatment of tomato plants with U-(^{14}C)-anilino-labelled pyrimethanil (A91817)

Time of harvest [days after treatment]	Total radioactive residues mg/kg [a]	Surface wash % [b] mg/kg	Extractable residues % [b] mg/kg	Unextracted residues % [b] mg/kg	Pyrimethanil % [b] mg/kg	T1 [c] % mg/kg	T2 [c] % mg/kg	T3 [c] % mg/kg	T4 [d] % mg/kg	Tx [e] % mg/kg
Tomato fruits										
Day 0 after applic [f]	960	99 950	- [g]	0.82 7.9	- [h]	- [h]	- [h]	- [h]	- [h]	- [h]
8 days after last applic	59 (41 – 74)	88 52	9.3 5.5	0.21 0.12	97 57	0.27 0.16	0.27 0.16	0.12 0.071	0.08 0.047	0.3 0.18

Time of harvest [days after treatment]	Total radioactive residues mg/kg [a]	Surface wash % [b] mg/kg	Extractable residues % [b] mg/kg	Unextracted residues % [b] mg/kg	Pyrimethanil % [b] mg/kg	T1 [c] % mg/kg	T2 [c] % mg/kg	T3 [c] % mg/kg	T4 [d] % mg/kg	Tx [e] % mg/kg
Tomato leaves										
Day 0 after applic [f]	14000	98 14000	-[g]	2.1 300	-[h]	-[h]	-[h]	-[h]	-[h]	-[h]
8 days after last applic	2800 (960; 4700)	88 2500	12 340	0.66 18	95 2700	0.2 5.6	0.5 14	0.28 7.8	0.06 1.7	0.05 1.4

a - total radioactive pyrimethanil residues

b - percent of recovered radioactivity

c - T1-T3 are hydroxylated and conjugated compounds of the active substance

d - Not identified

e - Tx- minor components that have not been isolated or characterised.

f - After 4th treatment

g - Extraction not performed for day 0 samples

h - Characterisation of the residual radioactivity has not been performed on these samples.

The Meeting received a study report of the metabolism of pyrimethanil on leaf lettuce (A91255, Huang, 1998; Report No. AN97E511). The 2-(^{14}C)-Pyrimidyl-labelled pyrimethanil was applied twice, as an EC formulation, to field grown lettuce plants at the rate of 800 g ai/ha (80 mg ai/m^2). Samples were taken immediately after the first treatment, 7 days after the second application and at harvest (21 days after the second application). Lettuce leaves were washed with dichloromethane and extracted with methanol/chloroform/ water and chloroform/water mixtures.

The non-extractable residues from samples taken at final harvest were further subjected to water soxhlet extraction and hydrolysed by acid and base. Polar extractable residues were hydrolysed using α-glucosidase, β-glucosidase and cellulase, releasing conjugated metabolites. In addition, all extracts containing polar residues were further treated under acidic and basic conditions for further hydrolysis. Residues were quantified using HPLC and TLC.

Lettuce samples contained total radioactive residues of 18 mg/kg 7 days after the second treatment and 4.2 mg/kg at harvest, 21 days after the final treatment. The majority of the radioactivity was present in the surface wash and in the extracts. Bound residues were 8.2% (day 7 samples) to 6.2% (day 21 samples). Most of the radioactivity recovered in the lettuce samples represented unchanged pyrimethanil (80% for day 7 samples and 44% for day 21 samples). Chemical hydrolysis of the extracts released the hydroxylated metabolites identified as AE C614276 (2-(4-hydroxyanilino)-4,5-dimethylpyridine) and AE C614277 (2-anilino-4,6-dimethylpyridin-5-ol) in low quantities (< 8%). The results are summarized in Table11.

Table 11. Residue levels, extraction profile and characterisation/ identification after foliar treatment of leaf lettuce plants (2 × 800 g ai/ha) with 2-(^{14}C)-Pyrimidyl-labelled pyrimethanil (A91255)

Time of harvest [days after treatment]	Total radioactive residues mg/kg [a]	Surface wash % [b] mg/kg	Extractable residues [c] % [b] mg/kg	Un-extracted residues % [b] mg/kg	Pyrimethanil % [b] mg/kg	AE C614276 [d] % [b] mg/kg	AE C614277 [e] % [b] mg/kg
Day 0 of 1st applic	99	93 92	6.1 6.0	0.5 0.50	92 91	-[f]	-[f]
7 days after 2nd applic	18	63 11	29 5.2	8.2 1.5	80 14	1.4 0.25	1.7 0.31

Time of harvest [days after treatment]	Total radioactive residues mg/kg [a]	Surface wash % [b] mg/kg	Extractable residues [c] % [b] mg/kg	Un-extracted residues % [b] mg/kg	Pyrimethanil % [b] mg/kg	AE C614276 [d] % [b] mg/kg	AE C614277 [e] % [b] mg/kg
21 days after 2nd applic	4.2	32 1.3	52 [g] 2.2	6.2 0.26	44 1.8	4.5 0.19	7.9 0.33

a - total radioactive pyrimethanil residues; average of 2 replicates.

b - percent of recovered radioactivity

c - including residues released by water soxhlet extraction (21 days after 2nd treatment only)

d - released after hydrolysis (conjugate of 2-(4-hydroxyanilino)-4,6-dimethylpyridine)

e - released after hydrolysis (conjugate of 2-anilino-4,6-dimethylpyrimidin-5-ol)

f - not detected

g - An additional 9.8 % TRR (0.4 mg/kg) was released by hydrolysis.

The Meeting received a study report on the metabolism of pyrimethanil in/on strawberry plants (A89249, Tarara, 1995; Report No. U/R 73/93). The uptake of 2-(^{14}C) pyrimidine-labelled pyrimethanil was investigated in strawberries under greenhouse conditions, after application of 1000 g ai/ha as a SC to the soil. At each harvest interval (3, 15 and 28 days after treatment) three treated plants and one control plant were selected. Each plant sample was divided into fruits, stems and leaves and roots

The plant material was extracted using chloroform and methanol/water and the methanol/water layer was additionally extracted with chloroform. Total radioactive residues in extracts were determined by LSC.

The amount of total radioactive residues in stems and leaves remained constant for all sampling intervals (0.04 mg/kg for days 3 and 28; 0.03 mg/kg for day 15). For fruits, the maximum level was reached on day 15 at 0.6 mg/kg and decreased to 0.02 mg/kg 28 days after treatment (due to increase of the fresh weight of the fruits). Extractable radioactivity ranged from 2.5 to 33% on days 3 and 28. On day 15, the extractable radioactivity was higher (87%) compared to fruit samples in the other sampling intervals. The radioactivity of the root samples was not reported. Taking into account the radioactivity recovered in the chloroform extract in each of the sampling intervals, a maximum pyrimethanil concentration of 0.52 mg/kg was estimated for day 15, which decreased to < 0.05 mg/kg at day 28. No characterisations or identifications of extract residues were attempted. The results are summarized in Table 12.

Table 12. Residue levels and extraction profile after soil treatment of strawberry plants (1 × 1000g ai/ha) with 2-(^{14}C)-Pyrimidyl-labelled pyrimethanil (A89249)

Harvest [days after soil treatment]	Total radioactive residues mg/kg [a]	Extractable residues % [b]	Unextractable residues % [b]	Chloroform extracts [c] % [b]	Methanol/water extracts [d] % [b]
Strawberry fruit					
3	0.4	2.2	98.	-[e]	2.2
15	0.6	87	13	87	0.2
28	0.02	33	67	24	8.4
Strawberry leaves and stems					
3	0.04	7	93	6.4	1.4
15	0.03	64	36	58	7.8
28	0.04	75	25	72	9.9

a - total radioactive pyrimethanil residues, determined as the sum of extracts and residual solids.

b - percent of recovered radioactivity

c - assumed to be pyrimethanil residues (**not** verified through identification)

d - assumed to be conjugates of hydroxylated pyrimethanil (**not** verified through identification)

e - values accounting for less than two times the background (20 dpm)

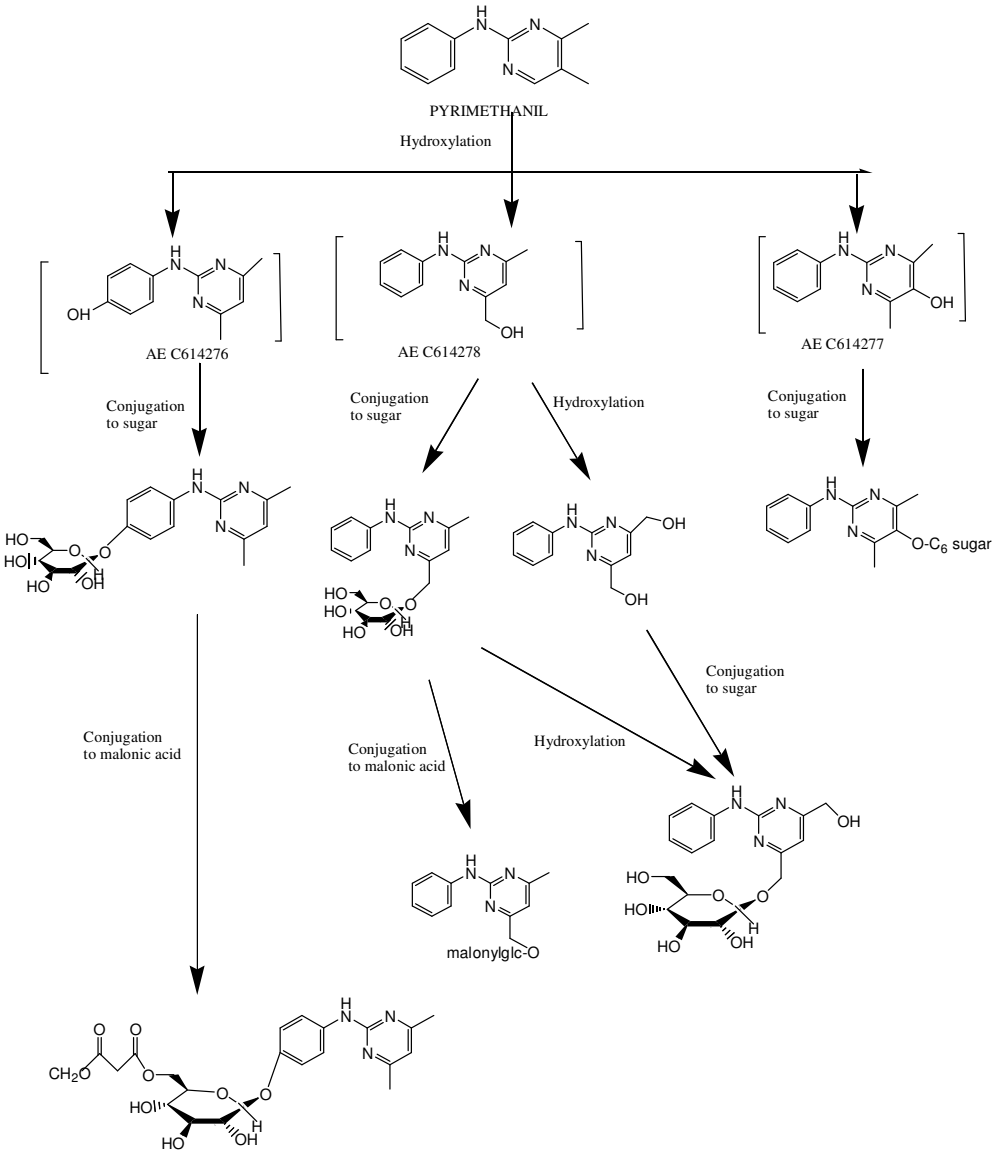


Figure 2. Proposed Metabolic Pathway for Pyrimethanil in/on Plants

Environmental fate

Aerobic Degradation in Soil

The Meeting received reports on the aerobic degradation of pyrimethanil in loamy sand soil (clay 2%, silt 23%, sand 75%) A81889, Feyerabend, 1992; Report No. UPSR 76/91; A81904, Feyerabend, 1993, Report No. U/R 20/93). Metabolites were extracted from German Standard Soil 2.2 after aerobic incubation of pyrimethanil (ZK 100 309) under laboratory conditions at 20 °C. Two different radiolabels were used: UL-^{14}C-phenyl-pyrimethanil and 2-^{14}C-pyrimidinyl-pyrimethanil. In order to obtain sufficient amounts of metabolites, three elevated application concentrations of both radiolabels were used: 100 mg/kg soil, 200 mg/kg and 500 mg/kg. Soil samples in stoppered flasks (1000 g dry soil + 153 g water each) were treated with pyrimethanil in methanol. The samples were maintained at room temperature (20 °C) and samples were aerated 3 times per week. Soils were sampled (about 10 g) after 33, 83, 131, 186, 243, 280 and 321 days. Extraction of soil was done by acetonitrile, soxhlet extraction with acetonitrile/water (9+1) and water. Analyses were carried out with LSC, radio-TLC, HPLC and GC-MS. Volatiles were not monitored.

Dissipation of pyrimethanil at 500 mg/kg was different with both radiolabels used (e.g., parent at day 243: phenyl label, 89.4%; pyrimidinyl label, 1.2%). With the radiolabel in the phenyl-

moiety, *apparent* metabolite formation in the extracts was poor. Ten metabolites were isolated with the maximum amount for a single component found to be 1.7% of the applied radioactivity. With the radiolabel in the pyrimidinyl moiety one major soil metabolite was found, U6 identified as 2-amino-4,6-dimethylpyrimidine (SN 512 723, ZK 512 723) at rates up to 58% of applied radioactivity. All the other 9 degradation products did not exceed 1.2% of applied radioactivity. The formation of this degradation product coincided with the decline of the parent compound, suggesting that this metabolite is a direct conversion product of pyrimethanil. Table 13 provides the details of distribution of radioactivity in treated soil.

Table 13. Distribution of extractable radioactivity in soil (% applied radioactivity) (A81889)

	Phenyl label			Pyrimidinyl label			
DAT	Extractable	Parent	U1-U10 [a]	Extractable	Parent	U1-U10, excl U6	U6 [b]
	100 mg/kg						
33	NA [c]	NA	NA	NA	NA	NA	NA
83	96	94	0.6	95	92	1.1	ND
131	91	88	0.9	89	84	1.1	0.7
186	12.	7.6	1.3	61.	4.8	1.5	52.
243	NA	NA	NA	NA	NA	NA	NA
280	NA	NA	NA	NA	NA	NA	NA
321	NA	NA	NA	NA	NA	NA	NA
	200 mg/kg						
33	101	100	0.3	102	101	0.5	0.1
83	96	95	0.5	98	96	0.9	0.3
131	96	94	0.6	92	88	1.6	0.6
186	40	34	1.2	63	3.1	1.7	56
243	NA	NA	NA	NA	NA	NA	NA
280	NA	NA	NA	NA	NA	NA	NA
321	NA	NA	NA	NA	NA	NA	NA
	500 mg/kg						
33	NA	NA	NA	NA	NA	NA	NA
83	103	101	0.5	102	100	0.5	NA
131	97	95	0.7	99	96	0.9	0.2
186	95	93	0.4	90	74	0.7	13.
243	94	89	2.9	64	1.2	1.7	58.
280	86	80	3.6	NA	NA	NA	NA
321	8.4	2.4	3.7	NA	NA	NA	NA

a - Unknowns. U1 and U2 were common to both labelled compounds.

b - 2-amino-4,6-dimethylpyrimidine (HPLC, GC/MS, NMR).

c - Not analysed.

In a separate study (A81904, Feyerabend, 1993, Report No. U/R 20/93), the aerobic degradation of 2-^{14}C-pyrimidinyl-pyrimethanil at a concentration of 1.3 mg/kg sandy loam soil (German Standard Soil 2.3; 60% sand, clay 7%, silt 33%) at 40% maximum water capacity at 20°C was considered. The study was conducted in glass biometer flasks containing 100 g dry weight of soil to which was added 14.9 g water. Each flask of soil was treated with 0.139 mg of radiolabelled pyrimethanil in 1.0 ml methanol. A total of 20 flasks were incubated at 20 ± 2 °C in the dark. Water was added weekly to maintain the water capacity. The sidearms of the flasks contained 0.1 N KOH to trap volatiles, and the flasks were aerated 3 times a week. Soils were sampled, extracted, and analysed in duplicate on days (DAT) 0, 7, 14, 28, 62, 90, 153, 244 and 364.

Soil samples were extracted sequentially with acetonitrile (ultrasonication); water (ultrasonication); and acetonitrile/water (9:1, v:v, ultrasonication). All extracts were measured by LSC and analysed by radio-TLC. The residual soils were analysed by combustion/LSC. The KOH trap contents were analysed by LSC.

The distribution of the radioactivity with time is summarized in Table 14.

Table 14. Distribution of extractable radioactivity in soil (% applied radioactivity) under aerobic conditions and identification [a] of metabolites (% applied radioactivity) (A81904)

Days after Treatment	Extracted (%)	Unextracted (%)	Total $^{14}CO_2$ (%)	Recovery (total %)	Pyrimethanil (%)	2-amino-4,6-dimethyl pyrimidine (%)	2-hydroxy-4,6-dimethyl pyrimidine (%)
0	95, 96	1.3, 0.5	-	96, 97	92, 94	-	-
7	89, 88	7.8, 7.8	0.2, 0.2	97, 96	85, 84	1.3, 0.7	-
14	82, 84	12, 11	0.4, 0.4	94, 96	78, 79	1.0, 1.2	-
28	57, 61	37, 32	1.7, 1.5	97, 95	45, 51	5.4, 4.1	-
62	32, 29	59, 60	4.4, 5.2	96, 94	8.9, 11	9.8, 6.8	2.7, 1.7
90	26, 27	62, 62	6.5, 6.4	95, 96	12, 14	5.1, 5.3	1.6, 1.1
153	19, 21	66, 65	10, 10	95, 96	5.3, 8.3	3.0, 4.5	1.8, 2.0
244	14, 18	63, 62	14, 13	91, 92	5.6, 5.9	2.0, 2.2	1.3, 1.4
364	11, 11	62, 63	17, 18	90, 92	4.3, 4.7	1.2, 1.0	0.9, 0.9

a - Based on RP-18TLC with co-chromatography. Two unknowns were also found, U1 maximizing at 2.2 % on day 62 and U2 maximizing at 1.1% on day 62.

The half-life of pyrimethanil under first order kinetics was calculated at about 30 days. The DT_{90} was calculated as about 90 days in this particular soil.

Anaerobic Degradation in Soil

The metabolism/degradation of 2-(^{14}C)-pyrimidinyl-labelled pyrimidine was studied in sandy loam German Standard Soil 2.3 at 20 ºC under anaerobic conditions (A89445, Tarara, 1996; Report No. CB94/050).

The test substance was applied at a rate equivalent to 1 kg ai/ha (1.33 mg/kg soil) and aged under aerobic conditions for 30 days. Samples were next converted to anaerobic (reductive) conditions by water logging (water containing 50 mg peptone/L) and put into an atmosphere of nitrogen. Sampling was continued until day 90 after water logging (120 days after application). In addition three batches were incubated at a ten-fold application rate (13.4 mg/kg soil) to allow for the isolation any new metabolite which could be found under anaerobic conditions. Volatiles and $^{14}CO_2$ were not determined. Extraction was carried out with acetonitrile/water (4/1) and with Soxhlet extraction (acetonitrile/water 9/1). Analyses were done by radio-HPLC and TLC.

The applied radioactivity which was all extractable on day 0, decreased by day 30 to 56% while bound residues increased to 44% of applied radioactivity during the same time period. The mineralisation process to CO_2 stopped after water logging; a nearly constant value for $^{14}CO_2$ was measured at all the following sampling intervals until day 120. After 120 days 1.6% of the applied radioactivity (AR) was mineralised. Volatiles other than CO_2 accounted for less than 0.1% AR throughout the study duration.

The parent accounted for the majority of the extractable radioactivity at day 120. The major degradation product was the metabolite, 2-amino-4,6-dimethylpyrimidine (maximum 14% AR, day 30). In addition 2-hydroxy-4,6-dimethylpyrimidine was found in trace amounts (maximum 2.2%, AR day 37). An additional 14 unidentified components were detected, none exceeding 3.8% AR. Analysis of bound residues resulted in a nearly equal distribution of residues in the humic and fulvic acid fractions. The results are summarized in Table 15.

Table 15. Distribution of extractable radioactivity in soil (% applied radioactivity) under anaerobic conditions and identification [a] of metabolites (% applied radioactivity) (A89445)

DAT	Extracted residues	Unextracted residues	Total CO_2	Recovery	Pyrimethanil	2-amino-4,6-dimethyl-pyrimidine	2-hydroxy-4,6-dimethyl-pyrimidine
0	100	1.2	ND	101	99	-	-
7	82	14	0.2	96	79	3.2	-
14	76	22	0.4	99	70	5.8	0.7
30 [a]	56	44	1.1	101	28	14	(11) [b]
37	49	51	1.5	101	24	12	2.2
44	46	52	1.2	99	23	11	1.6
64	44	54	1.3	99	25	11	0.9
90	44	53	1.1	98	25	10	0.8
120	47	51	1.6	100	26	10	1.5

a - End aerobic phase and begin anaerobic phase

b -Includes other components

The proposed metabolic/degradation pathway for pyrimethanil in soil is presented in Figure 3. Note that SN617916, SN603913 and AZ196920 are postulated and were not identified in any study.

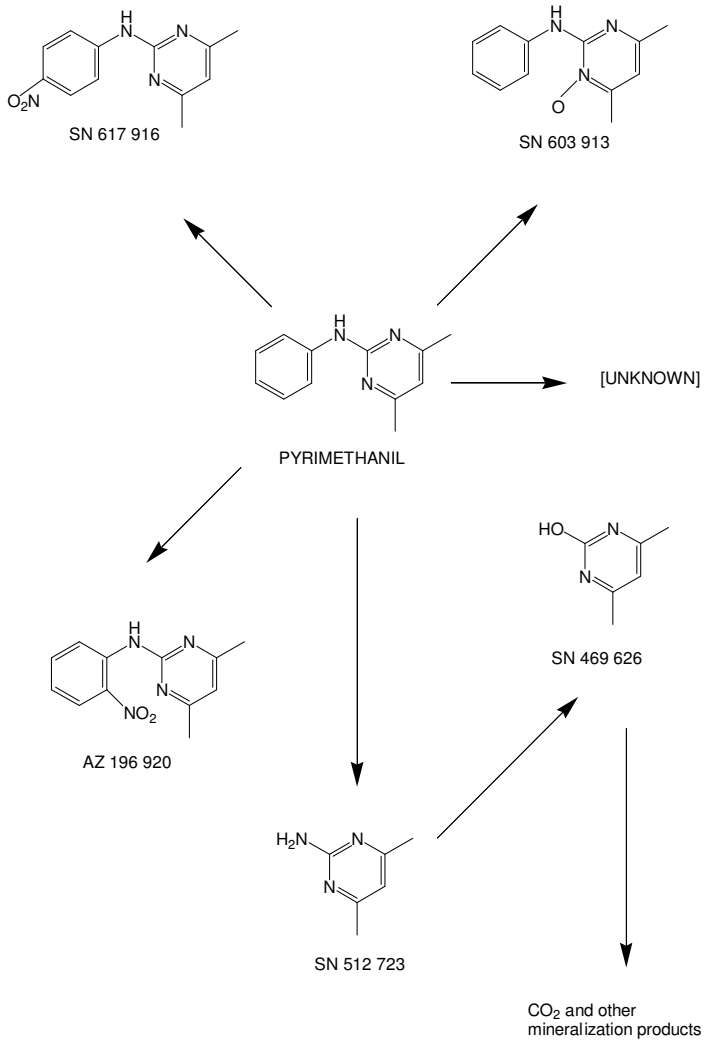


Figure 3. Proposed Pathway for the Metabolism/Degradation of Pyrimethanil in Soil[20]

[20] SN617916, SN603913, and AZ196920 are postulated and were not identified in study.

Rate of Degradation in Soil

Several studies in addition to the above were undertaken in laboratory conditions to estimate the rate of degradation of pyrimethanil in different types of soil samples. In one study, the dissipation of UL-(^{14}C)-phenyl-ZK 100 309 in German Standard Soil 2.2 (loamy sand) was investigated after application at the rate of 1 kg ai/ha and 8 higher concentrations. Soil moisture was adjusted to 39% of its maximum water capacity. Soil was extracted with acetonitrile, acetonitrile/water and for samples with 10% more of bound residues, under agitation with 0.1N NaOH. Residues were analysed by radio-TLC and HPLC. Soil samples of the higher concentration series were incubated for 148 days. The rate constants of $^{14}CO_2$ evolution were averaged for each concentration and plotted against the concentration of pyrimethanil. The half-life of pyrimethanil was estimated by first order multi-compartment models. Based on these models, non-linear regression analysis was made (A81878, Feyerabend, 1991; UPSR 63/91).

The degradation of pyrimethanil in this study was described by 1st order kinetics with a DT_{50} of 82 days (DT_{90} of 272 days), based on single exponential decay. Most of the extractable material was identified as unchanged parent together with one unidentified metabolite (max 6.6% AR) and 8 additional minor metabolites

The dissipation of pyrimethanil (UL-(14C)-phenyl-ZK 100 309) in a clay loam soil (Schering Soil no. 178) under laboratory conditions at an application rate of 1.06 kg ai/ha was investigated in a separate study. Soil moisture was adjusted to 40% of its maximum water capacity. Extraction and analysis were the same as in the previous study (A81879, Feyerabend, 1991; Report No. UPSR 66/91).

The results showed degradation of pyrimethanil by 1st order kinetics with a DT_{50} of 80 days (DT_{90} of 266 days), based on single exponential decay. Most of the extractable material was identified as unchanged parent together with 4 unidentified metabolites (≤ 1% each).

The degradation rate of pyrimethanil in soil was also estimated at 10 ºC (C006902, Terry, 2000; Report No. W-117-1). The rate of soil degradation of pyrimethanil was calculated according to FOCUS recommendations on the basis of the results of the laboratory studies. The Arrhenius equation was applied as a reasonable description of the temperature dependency within realistic environmental temperatures: DT_{50} (10 ºC) = DT_{50} (20 ºC) × Q10. Using this equation, the mean value for the DT_{50} was 112 days and a 95th percentile of 157 days.

Fate and Behaviour in Water

Photolysis

The photodegradation of pyrimethanil (test concentration ≈ 10 mg/L) was studied in two sterile aqueous buffer solutions at pH 4 (citrate buffer) and 7 (phosphate buffer). The test solutions (aliquots of 3 mL in quartz cuvettes) were continuously irradiated with simulated natural sunlight (λ > 290 nm) of a Hg-arc-lamp in a "merry go round" photoreactor up to 4 (pH 4) and 28 days (pH 7) at 29.3 ± 2.6 °C for pH 4 and 30.1 ± 1.6 °C for pH 7. Dark control samples were incubated under conditions equivalent to that of the irradiated test samples. At appropriate time intervals irradiated and non-irradiated test samples were taken and analysed by HPLC (Tschampel, 1992. Document Ref.: A81887). The results are summarized in Table 16.

Table 16. Photolysis of Pyrimethanil in aqueous solution (A81887)

pH 4	Time (h)	0	1.0	2.0	4.0	6.0	8.0	24.	96.
	ai (mg/L)	11	10	10	10	10	9.7	7.3	1.1
	(%)	100	99	99	96	95	92	**70**	**10**
Dark control	ai (mg/L)	11	10	11	11	11	11	11	11
	(%)	100	99	100	100	100	100	101	101
pH 7	Time (h)	0	4.0	21	75	172	341	551	675
	ai (mg/L)	11	11	11.	10	20	9.5	8.8	8.1
	(%)	100	101	100	95	92	**89**	**82**	**76**
Dark control	ai (mg/L)	11	11	11	10	10	10	11	10
	(%)	100	101	101	98	97	98	99	98

The dark control samples (total recovery: 97.4 – 101%) showed no significant degradation of pyrimethanil. Assuming a pseudo first-order reaction kinetic for the photolytic degradation of pyrimethanil, the rate constants (k) and DT_{50} values were estimated for each pH by regression curves (concentration vs. time):

pH	k (h^{-1})	DT_{50} (days)
4	2.39×10^{-2}	1.2
7	3.76×10^{-4}	76.8

In a separate study, when aqueous solutions of pyrimethanil (containing 10 mg/L in distilled water and in sterile nature synthetic water containing soluble humic acids, buffered at pH 7) were continuously irradiated for 4 days with simulated natural sunlight in a "merry go round" photoreactor, photolytic degradation with DT_{50} of 47.5 h was observed. No degradation occurred in dark control samples and distilled water samples (A81930, Tschampel, 1994).

Residues in Succeeding Crops

Accumulation in Confined Rotational Crops

The uptake of ^{14}C-pyrimethanil in soil by rotational crops was investigated under confined conditions in 2002 (B003517, Meyer, 2002; Report No. AN99E512). Tanks were filled with bare sandy loam and treated with 2-(^{14}C)-pyrimidinyl-labelled pyrimethanil at a rate of 2.4 kg ai/ha. Each tank was divided into three parts each allocated to one of the three representative crops (lettuce for leafy, radishes for root crops, and wheat for grains). The rotational crops were planted 30, 130 and 300 days after treatment of the soil. Crops were harvested at an immature (forage) stage in wheat (i.e. 35 – 148 days after planting) and at maturity (i.e. for wheat: 73 – 190 days; for radishes and lettuce: 46 – 79 days).

Samples were extracted with acetonitrile and acidified methanol. Wheat samples were further extracted with acetonitrile/ethanol and water. Aliquots of the non-extractable residues from the 30 day crops were subjected to sequential hydrolysis with mild acid, mild base and strong base (1 M HCI, 1 M NaOH and 6 M NaOH). Total radioactivity in extracts and solutions were determined by LSC; radioactivity in residual solid samples was determined by combustion and the evolved $^{14}CO_2$ by LSC. The initial characterisation of plant extract residues was done by TLC; HPLC was used for quantification and identification of metabolites. Confirmation of identity of metabolites was carried out by LC/MS.

In crops planted in 30 day aged soil, total radioactive residues ranged from 0.23 mg/kg (radish roots) to 8.2 mg/kg (wheat straw). Total radioactive residues declined in crops harvested from soil plots aged 130 days and 365 days. TRRs in those crops were 0.05 mg/kg or less, except for wheat straw, which was 0.15 ppm or less.

Pyrimethanil was extensively metabolized into numerous related components as shown in Table 17. The parent was identified as the major residue in wheat, radish and lettuce. The only metabolite present > 10% of TRR was AN5, AE C621312, 2-anilino-4,6-dihydroxymethyl-pyrimidine in 30 day lettuce and wheat forage.

Table 17. Residue levels, extraction profile, and metabolic pattern of pyrimethanil in rotational crops after application of 2-(^{14}C)-pyrimidinyl-labelled pyrimethanil to bare sandy loam soil at 2.4 kg ai/ha

Crop	Sample/ Days after Planting	TRR (mg/kg) [b]	Extractable (%) [c]	Parent (%) [c]	AN2 [d] (%) [c]	AN3 [e] (%) [c]	AN4 [f] (%) [c]	AN5 [g] (%) [c]	AN6 [h] (%) [c]	AN7 [i] (%) [c]	AN8 [j] (%) [c]
30-day plot [a]											
Wheat	Forage [k] 35	2.4	85	45	0.5	1.1	0.9	**11**	3.1	5.7	6.7
	Grain 73	0.41	88	0.2	0.9	0.4	0.7	1.3	1.2	0.9	1.4
	Straw 73	8.2	62	2.7	0.7	0.5	1.8	2.3	2.2	1.5	1.3
Lettuce	Leaves 46	0.63	75	4.3	7.1	6.6	3.9	**15**	-[l]	7.3	1.4
Radish	Top 46	0.87	83	1.1	4.8	3.1	1.8	2.8	-[l]	5.2	-[l]
	Roots 46	0.23	71	12.7	-[l]	-[l]	-[l]	-[l]	-[l]	-[l]	-[l]
130-day plot [a]											
Wheat	Forage[11] 148	0.027	70	1.2	2.1	-[l]	1.7	7.6	-[l]	2.8	1.2
	Grain 190	0.015	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]
	Straw 190	0.082	58	2.3	-[l]	-[l]	-[l]	6.7	-[l]	4.3	-[l]
Lettuce	Leaves 79	0.03	65	26	-[l]	-[l]	-[l]	5.2	-[l]	-[l]	-[l]
Radish	Top 79	0.04	70	7.4	-[l]	-[l]	-[l]	1.7	2.6	3.8	0.9
	Roots 79	0.012	80	8.3	3.5	-[l]	1.6	2.4	1.8	-[l]	-[l]
300-day plot[l]											
Wheat	Forage[11] 57	0.023	63	0.8	-[l]	-[l]	-[l]	4.9	-[l]	3	-[l]
	Grain 106	0.03	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]
	Straw 106	0.152	52	0.7	-[l]	0.7	-[l]	8.6	-[l]	5.3	-[l]
Lettuce	Leaves 65	0.009	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]
Radish	Top 62	0.029	75	1.7	-[l]	-[l]	-[l]	8.9	-[l]	5.4	-[l]
	Roots 62	0.011	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]	-[m]

a - Days elapsed from treatment to planting.

b - Results expressed as mg/kg pyrimethanil equivalents; determined by combustion/LSC of the crop part.

c - % of total radioactive residues and includes organosoluble and aqueous soluble. Excludes acid and base hydrolysis of non-extractable residues

d - [AE C614276]: 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine

e - [AE C614277]: 2-anilino-4,6-dimethylpyrimidine-5-ol

f - [AE C614278]: 2-anilino-4-hydroxymethyl-6-methylpyrimidine

g - [AE C621312]: 2-anilino-4,6-dihydroxymethyl-pyrimidine

h - [AE C614800]: 2-(4-hydroxyanilino)-4-hydroxymethyl-6-methylpyrimidine

i - [AE F132593]: 2-amino-4,6-dimethylpyrimidine

j - [AE 0025462]: 4,6-dimethylpyrimidine

k - Immature wheat

l - Not detected.

m - Low radioactivity; not investigated. LOQ for combustion/LSC is about 0.002 mg/kg.

Field Accumulation in Rotational Crops

Possible residues of pyrimethanil on rotated wheat planted following treatment of a potato crop were investigated in 1999 (AN99R003, Cole and Benson, 2002; Document Ref.: B003787). Trials were conducted in the USA (North Carolina, Florida and Oregon). A suspension concentrate formulation of pyrimethanil (400 g/L) was applied 3 times on potato plants by foliar spray at a rate of 0.8 kg ai/ha per application and a PHI of 7 days. After the last application, the potato crop was cleared from the plots and winter wheat was planted 29 to 30 days following the last application. Wheat forage was harvested 128 to 232 days after the last application. Hay was harvested 149 to 239 days after the last application. Straw and mature grain was harvested 190 to 316 days after the last application.

Residues of pyrimethanil as determined by GC/MS with an LOQ of 0.05 mg/kg, and residues of AE C621312 (2-anilino-4,6-dihydroxymethyl-pyrimidine) as determined by HPLC/MS with LOQ of 0.05 mg/kg, were non-quantifiable (< 0.05 ppm) in 30 day rotational wheat forage, hay, straw and grain samples collected 150 to 316 days after the last of three applications of pyrimethanil at 0.8 kg ai/ha to potatoes. The pyrimethanil-derived residues found in the individual treated rotated wheat samples are summarized in Table 18.

Table 18. Residues of Pyrimethanil in wheat from rotation with potatoes treated foliarly (3 × 0.8 kg ai/ha) (AN99R003)

Trial Location, year	Application			Timings (days)		Residues (mg/kg)	
	kg ai/ha	Water L/ha	No.	Rotational interval	PHI	Pyrimethanil	AE C621312
Wheat Forage							
Trial R02-01 North Carolina, USA, 1999	0.8 0.8 0.8 0.8	187 187 187 187	3 3 3 3	29 29 29 29	150 150 171 171	< 0.05 < 0.05 ND ND	ND [a] ND ND ND
Trial R03-01 Florida, USA 1999	0.8 0.8 0.8 0.8	187 187 187 187	3 3 3 3	30 30 30 30	128 128 135 135	ND ND ND ND	ND ND ND ND
Trial R12-01 Oregon, USA 1999	0.8 0.8 0.8 0.8	187 187 187 187	3 3 3 3	30 30 30 30	197 197 232 232	ND ND ND ND	ND ND ND ND
Wheat Hay							
Trial R02-01 North Carolina, USA, 1999	0.8 0.8	187 187	3 3	29 29	195 195	ND ND	ND[1] ND
Trial R03-01 Florida, USA 1999	0.8 0.8	187 187	3 3	30 30	149 149	ND ND	ND ND
Trial R12-01 Oregon, USA 1999	0.8 0.8	187 187	3 3	30 30	239 239	ND ND	ND ND
Wheat Straw							
Trial R02-01 North Carolina, USA, 1999	0.8 0.8	187 187	3 3	29 29	226 226	ND ND	ND[1] ND
Trial R03-01 Florida, USA 1999	0.8 0.8	187 187	3 3	30 30	190 190	ND ND	ND ND
Trial R12-01 Oregon, USA 1999	0.8 0.8	187 187	3 3	30 30	316 316	ND ND	ND ND
Wheat Grain							
Trial R02-01 North Carolina, USA, 1999	0.8 0.8	187 187	3 3	29 29	No sample No sample	- -	- -

Trial Location, year	Application			Timings (days)		Residues (mg/kg)	
	kg ai/ha	Water L/ha	No.	Rotational interval	PHI	Pyrimethanil	AE C621312
Trial R03-01 Florida, USA 1999	0.8 0.8	187 187	3 3	30 30	190 190	ND ND	ND[1] ND
Trial R12-01 Oregon, USA 1999	0.8 0.8	187 187	3 3	30 30	316 316	ND ND	ND ND

a - ND = lower than limit of detection (LOD) of 0.012 mg/kg for pyrimethanil and 0.015 mg/kg for AE C621312

RESIDUE ANALYSIS

Analytical Methods

Analytical methods for the determination of residues of pyrimethanil have been developed for a wide range of substrates. After an extraction specific to the matrix, and a cleanup that varies somewhat with the matrix, determination of pyrimethanil is made either by gas chromatography using mass selective detection (GC/MSD) or by HPLC relying on UV detection. The methods have been extensively validated with numerous recoveries on a wide range of substrates. The accuracy of the method has been considered acceptable if the mean recovery over the fortification range for each matrix were in the range of 70% to 120%. The precision has been considered acceptable if the relative standard deviations (% RSD) at the fortification levels (e.g., LOQ and 10 × LOQ) were 20% or less. The methods outlined in this section were used in the residue trials and should be suitable for MRL enforcement purposes.

Plant matrices

Method RESID 95/23; A890006 (HPLC/UV)

A method was presented, including validation data, for the determination of pyrimethanil in *apples, tomatoes, grapes and green beans* by HPLC with UV detection (A89006, Garner and Snowdon, 1995; Report No. RESID 95/23). Residues of pyrimethanil are extracted from representative crop samples by homogenizing with acetone followed by dilution with hydrochloric acid and washing with hexane. The aqueous layer is then neutralized with sodium hydrogen carbonate and partitioned using 25% ethyl acetate in hexane. Clean-up is by solid phase extraction (SPE) using a silica cartridge. Pyrimethanil residues are determined by HPLC with UV detection. The method was validated for use on apples, grapes, tomatoes, and green beans, with a demonstrated limit of quantification of 0.05 mg/kg.

The method was shown to be linear in the range of 0.5 to 10 µg/mL pyrimethanil. The validation data are summarized in Table19.

Table 19. Recovery of pyrimethanil from fortified samples of fruits and vegetables using Method RESID 95/23 (HPLC/UV) (A89006)

Fortification (mg/kg)	Recovery (%)			
	Apples	Grapes	Tomatoes	Green beans
0.05 (mean) (RSD%)	91, 92, 80 (88) (7.6)	74, 79, 66, 78 (74) (8.0)	74, 104, 105 (94) (19)	75, 74, 88 (79) (9.9)
0.25 (mean) (RSD%)	97, 120, 101, 81 (100) (16)	70, 79, 83 (77) (8.6)	103, 94, 103 (100) (5.2)	87, 83, 85 (85) (2.3)
0.50 (mean) (RSD%)		66, 105, 108, 116 (99) **(22)** [a]	91, 106 (98) (-)	
1.0				86, 86, 88

Fortification (mg/kg)	Recovery (%)			
	Apples	Grapes	Tomatoes	Green beans
(mean) (RSD%)				(87) (1.3)
2.0 (mean) (RSD%)			92, 91, 95 (93) (2.2)	
5.0 (mean) (RSD%)		90, 90, 89 (90) (0.64)		
Overall mean RSD n	95 15% 7	85 19% 14	96 9.9% 11	84 6.5% 9

a - Outside acceptable limit

Method U/R 33/93; A83775 (HPLC/UV)

A study report on an alternate HPLC procedure for use with grapes was provided (A83775, Moede, 1993; Report No. U/R 33/93). Residues of pyrimethanil are extracted from grapes with methanol. After clean-up using liquid/liquid partition and a silica gel column, pyrimethanil is quantitatively determined by HPLC with UV detection. The method has been validated in the range of 0.02 to 1.0 mg/kg.

Table 20. Recovery of pyrimethanil from fortified grape samples via HPLC/UV (A83775)

Fortification (mg/kg)	Recovery	
	%	Mean [a] %
0.02	95, 105	100
0.05	90, 98, 94	94
0.1	96, 102, 94	97
1.0	98, 99, 99	99

a - overall mean 97%, RSD 1.6%

This method was subjected to an independent laboratory evaluation (A89691, Dacus, 1996; Report No. A-96R-02). Samples of grapes, grape juice or pomace were extracted with methanol, filtered, diluted with water and partitioned with ethyl acetate. The ethyl acetate fraction was evaporated to dryness and cleaned-up on a silica column. Residues of pyrimethanil were determined by HPLC with UV detection.

Three untreated control samples, one each of grapes, juice and wet pomace were selected from a grape processing study. One control and two samples fortified at 0.02 mg/kg and 1.98 mg/kg were analysed per matrix for a total of nine samples.

Table 21. Independent laboratory validation of A83775 (A89691) (HPLC/UV)

Matrix	Fortification (mg/kg)	Recovery (%)
Grapes	0.02	96
	1.98	98
Grape juice	0.02	87
	1.98	90
Wet grape pomace	0.02	94
	1.98	96

Method UPSR 24/91; A81874 (HPLC/UV)

An HPLC method for the determination of pyrimethanil in water and in wine was presented (A81874, Wrede-Rucker, 1991, Report UPSR 24/91). Pyrimethanil is extracted by solid phase extraction (C18

reversed phase) and eluted with hexane/ethyl acetate (9/1). Clean-up is with florisil. Residues are quantitated with HPLC utilising a UV detector at 268 nm. Acceptable recovery of pyrimethanil from fortified control wine samples was demonstrated in the range of 0.05 – 1.0 mg/kg. Overall mean recovery was 94%, RSD 2.1%, n=10.

Method DGM C05/98-0; C000292 (GC/MS)

The Meeting received a study report on a GC/MS method for the determination of residues of pyrimethanil in fruits and vegetables (C000292, Peatman, 1999; Report DGM C05/98-0). Validation data were included. This method, with minor modifications for some matrices, was the analytical method used in most of the supervised trials on fruits and vegetables for which the Meeting received study reports. The matrix is extracted by maceration with acetone followed by several clean up steps, including washing the acidified extract with hexane, partitioning into solvent from alkali-treated extract and silica solid phase extraction. The eluant from the SPE is taken to dryness and dissolved in ethyl acetate containing 5 mg/kg PCB.

Pyrimethanil is determined by gas chromatography with mass selective detection. The method was validated for use on potatoes, carrots, tomatoes, green beans, lettuce, sweet peppers, strawberries, raspberries, apples, grapes and bananas, with a demonstrated limit of quantification of 0.05 mg/kg.

The method was shown to be linear in the range of 0.25 to 5 µg/mL, using the ratio of pyrimethanil response to PCB response (pentachlorobenzene, 5 ppm). The target ion for pyrimethanil was m/z 198. Confirmatory ions were m/z 199 and m/z 200. The validation data are summarized in Table 22.

Table 22. Recovery of pyrimethanil from fortified samples of fruits and vegetables using Method DGM C05/98-0 (GC/MS) (C000292)

Matrix	Fortification (mg/kg)	Recovery (%)			RSD (%)		n
		Value	Mean (level)	Mean (Overall)	Fortification level	Overall	
Potatoes	0.05	92, 92, 93, 90, 94	92	93	1.6	2.8	5
	0.5	91, 94, 95, 91, 99	94		3.5		5
Carrots	0.05	91, 72, 86, 84, 86	84	83	8.5	10	5
	0.5	100, 78, 72, 80, 80	82		13		5
Tomatoes	0.05	86, 89, 83, 82, 81	84	84	3.9	4.0	5
	0.5	89, 82, 84, 82, 79	83		4.5		5
Green beans	0.05	85, 71, 69, 69, 76	74	80	9.2	9.7	5
	0.5	87, 81, 86, 85, 88	85		3.2		5
Lettuce	0.05	78,81, 75, 76, 75	77	79	3.3	3.6	5
	0.5	81, 81, 82, 82, 78	81		2.0		5
Sweet peppers	0.05	101, 100, 103, 115, 82	100	91	12	14	5
	0.5	82, 82, 84, 80, 82	82		1.7		5
Strawberries	0.05	103, 83, 85, 86, 94	90	92	9.2	7.1	5
	0.5	100, 97, 91, 94, 90	94		4.4		5
Raspberries	0.05	108, 89, 91, 94, 92, 95	95	92	7.2	7.5	6
	0.5	101, 83, 88, 90, 87	90		7.5		5

Matrix	Fortification (mg/kg)	Recovery (%)			RSD (%)		n
		Value	Mean (level)	Mean (Overall)	Fortification level	Overall	
Apples	0.05	82, 80, 95, 100, 70	85	83	14	11	5
	0.5	80, 77, 82, 82, 79	80		2.7		5
Grapes	0.05	73, 83, 80, 80, 81	80	82	2.3	4.3	5
	0.5	80, 80, 83, 84, 81, 80, 87, 87, 78, 90	83		4.7		10
Bananas	0.05	96, 100, 92, 99, 96, 101, 93	97	86	3.6	13	7
	0.5	75, 66, 79, 73, 76, 86, 78, 90, 85, 78	79		8.9		10

The above method DGM C05/98-0 as documented under C000292 was independently validated at Covance Laboratory Ltd. in 1999 (C004680, Croucher, 1999; Report No. 194/199-D2140). Control samples of potato, grapes, lettuce, grain and straw were fortified with known amounts of pyrimethanil at two levels, equivalent to 0.05 and 0.5 mg/kg, in quintuplet and analysed following the analytical method. A small amount of interference was observed for potato, grain and straw. However, the interference was less than 30% of the limit of quantification (0.05 mg/kg) for all matrices. The calibration method was modified by use of a second order polynomial equation to express the relationship of ion abundance (detector response) to pyrimethanil concentration. The ratio of pyrimethanil response to PCB (pentachlorobenzene, 5 ppm) response was used. A summary of the validation data is shown in Table 23.

Table 23. Independent laboratory validation of Method DGM C05/98-0) for determination of pyrimethanil in potatoes, grapes, lettuce, grain, and straw (n = 5) (C004680)

Matrix	Fortification (mg/kg)	Recovery			RSD (%)	
		%	Mean %(level)	Mean %(overall)	At fortification	Overall
Potatoes [a]	0.05	83,81,101, 108,146	93	94	25 [c]	**23** [c]
	0.5	77,76,77,86,102	84		11	
Grapes	0.05	80,82,76,94,90	84	86	8.8	9.3
	0.5	75,86,101, 85,86	87		11	
Lettuce	0.05	86,96,94,80,114	94	99	14	11
	0.5	107,105,107,109,96	105		4.9	
Grain [b]	0.05	62,70,77,80,78 67,68,80,80,78	73	74	10	8.9
	0.5		75		8.7	
Straw b	0.05	79,81,78,68,75	76	72	6.7	9.1
	0.5	74,63,70,62,74	69		8.4	

a -Three attempts necessary

b - Two attempts necessary.

c - Outside acceptable limit (20%)

A modified version of DGM C05/98-0 for the determination of pyrimethanil in peaches and plums was reported (PR 08700.04-CAR17 and PR 08702.04-CAR16, Starner, 2005). Samples were homogenized with acetone and an aliquot of the extract was acidified and washed with hexane before neutralising with a base to enable partition. Final clean-up was by silica SPE and quantification was by gas chromatography with mass selective detection. Ion m/z 199 was used for confirmation, and ion

m/z 198 was used for quantitation. Calibration was by external standards with four points. The PCB internal standard was not used. Recovery data are summarized in Table 24.

Table 24. Recovery of pyrimethanil from fortified peach and plum samples via DGM C05/98-0 (GC/MS) (PR 08700.04-CAR17; PR 08702.04-CAR16)

Matrix	Fortification (mg/kg)	Recovery			
		%	Mean (%)	RSD (%)	n
Peach	0.05 0.5 5.0 Overall	73,81,86 82,82,83 78,83,85	80 82 82 81	4.8	9
Plum	0.05 0.5 5.0 Overall	88,104,115 85,86,93 77,77,79	102 88 78 89	14.	9

The independent laboratory validation of analytical method LOA/SOP/6006.1 for the determination of pyrimethanil in orange and apple samples by GC/MS was reported (AGR 1113, Pigeon, 2006; Report No. 21001). The method is quite similar to DGM C05/98-0, but without the extensive sample preparation.

Residues of pyrimethanil are determined in orange and apple samples by capillary gas chromatography with mass spectrometry detection (GC/MS), after extraction with ethylacetate/isohexane (50/50 v/v). Calibration is by internal standards, using the response ratio of pyrimethanil to PCB (pentachlorobenzene). The ions monitored are m/z 248, 250 and 252 for PCB and m/z 198, 199 and 200 for pyrimethanil. Linearity was demonstrated over the concentration range of 0.02 to 10 µg/mL pyrimethanil by measuring peak area versus concentration for a series of 9 matrix matched standard solutions of known concentrations. Matrix effects necessitated the use of matrix matched solutions.

Table 25. Recovery of pyrimethanil from fortified orange and apple samples by GC/MS (AGR 1113)

Matrix	Fortification (mg/kg)	Recovery			
		%	Mean (%)	RSD (%)	n
Orange	0.05	82, 86, 89, 91, 93	88	4.9	5
	5	99, 102, 102, 102, 104	102	1.8	5
	Overall		95	8.2	10
Apple	0.05	92, 92, 93, 94,94	93	1.1	5
	5	98, 100, 101, 102, 103	101	1.9	5
	Overall		97	4.5	10

A radiovalidation study was reported for method DGM C05/98-0 (B003142, Dacus, 2001; Report No. AN00R002). The extraction efficiency of method DGM C05/98-0 was evaluated using lettuce treated with 14C-pyrimethanil as part of the metabolism study (A91255, Huang, 1998). The sample used was one of three replicates collected on day 18, 7 days after the second application with radiolabelled pyrimethanil. Method DGM C05/98-0 was used for analysis. In addition, total radioactivity of the lettuce samples was determined by combustion and aliquots of the extracts were taken at various steps in the method for LSC counting.

Table 26. Extraction efficiency and overall efficiency of method DGM C05/98-0 for lettuce treated with ^{14}C-labelled pyrimethanil (B003142)

TRR	Total pyrimethanil [b]	Extracted	Extraction efficiency	Final extract/overall efficiency			
				LSC		GC	
(mg/kg)	(mg/kg)	(mg/kg)	(%)	(mg/kg)	% of TRR	(mg/kg)	% of TRR
8.9 [a]	7.1 (80.4% TRR)	6.9 [c]	97 [d]	4.6 [e]	64.	4.8	68.
Corrected for method efficiency [f]				5.5	77.	5.8	81.

a - Combustion/LSC of lettuce 6P from A91255.

b - A91255. Determined in the metabolism study

c - LSC of acetone extract only. Hexane waste and SPE waste totalled 0.45 mg/kg

d - 6.9 / 7.1

e - Final extract for GC/MS analysis.

f - Recoveries from fortification of leaf lettuce with unlabelled pyrimethanil at 0.05 and 20 mg/kg, range 77.7 – 88.5%, mean 83.1%, RSD 5.9%, n = 4.

Animal Tissues and Milk

Method RAM AN/01/02; B003870 (GC/MS)

The Meeting received a study report on Method RAM AN/01/02 for the determination of pyrimethanil and metabolites AE C614276 and AE C614277 in livestock tissues and milk by gas chromatography with ion trap mass selective detection (B003870, Neal, 2002; Report No. AN/01/02). Fat samples are extracted with acetonitrile. Muscle, liver and kidney are extracted with 92:8 acetonitrile:0.6 M HCl. The extracts are acidified by adjusting the solvent composition to 92:8 acetonitrile:0.6 M HCl. The extracts are adjusted to a fixed volume, and an aliquot is partitioned with hexane to remove co-extracted fat/oil. The hexane is discarded and the remaining acetonitrile extracts are evaporated to dryness and reconstituted in methanol. For milk, samples are treated with concentrated HCl (1 mL per 20 g) and acetonitrile and centrifuged. The mixture is filtered and the residual solids are buffered to pH7 and shaken with acetonitrile. The acetonitrile is combined with the original acetonitrile extract and preparation continues as for the other sample types. The methanol extracts of milk and kidney are subjected to enzymatic hydrolysis (37 °C, β-glucuronidase, sulfatase). Extracts of all tissues and milk are methylated with trimethylsilyldiazomethane (TMSD), which yields the methoxy derivatives of the two metabolites.

The GC/MS is calibrated with external standard solutions of pyrimethanil and the methylated metabolites. A least squares regression line of the data (response vs. concentration) was used. The quantitation ions are m/z 182 for pyrimethanil and m/z 214 and/or 213 for AE C588789 and AE C0815072 (methoxy derivatives). Validation is summarized in Table 27.

Table 27. Recovery of pyrimethanil and AE C614276 from fortified samples of bovine tissues and pyrimethanil, AE C614276, and AE C614277 from milk (B003870)

Matrix	Fortification (mg/kg)	Recovery (%) Pyrimethanil	Recovery (%) AE C614276	Recovery (%) AE C614277
Milk	0.01	77, 87	70, 76	95, 90 73, 114 87
	0.10	95	94	100, 70 69, 71 92, 89
Fat	0.05	104	74	
	0.50	122	87	
Muscle	0.05	92, 87	68, 88	
	0.50	100	63	
Liver	0.05	89	88	
	0.50	84	87	
Kidney	0.05	84	87	
	0.50	85	89	

Method RAM AN/01/02 was validated by an independent laboratory (B003871, Algaier, 2002, MRI Laboratory Project 310345.1.001, Aventis Study Number 02AN27440). External calibration was utilised and linear regression equations were calculated relating the area response of each component (same m/z ions as above) to the concentration of the standard solution. The regression coefficients ranged from 0.980 to 0.997 for milk and muscle. The curve was not forced through (0, 0). Recoveries from fortified controls are summarized in Table 28.

Table 28. Independent Laboratory Validation: Recovery of pyrimethanil, AE C614276 and AE C614277 from fortified bovine commodities (n = 5)

Analyte	Matrix	Fortification (mg/kg)	Recovery			RSD	
			%	Mean at fortification (%)	Mean for matrix (%)	For fortification (%)	For matrix (%)
Pyrimethanil	Muscle [c]	0.05	84 85 107 114 162	111	99	**29** [a]	**26** [a]
		0.5	80 76 91 91 102	88		12	
	Milk [b]	0.01	108 120 120 121 133	120	117	7.1	5.8
		0.1	112 112 113 115 117	114		1.9	
AE C614276	Muscle [c]	0.05	106 107 113 130 132	118	102	10	20
	Muscle [d]	0.5	69 76 87 97 105	87		15	
	Milk [b]	0.01	96 122 128 132 134	123	114	13	12.
		0.1	102 103 103 107 109	105		2.8	

Analyte	Matrix	Fortification (mg/kg)	Recovery			RSD	
			%	Mean at fortification (%)	Mean for matrix (%)	For fortification (%)	For matrix (%)
AE C614277	Milk [b]	0.01	58 67 83 84 86	63	80	**54** [a]	12.
		0.1	80 82 85 86 90	85		4.5	

a - Exceeds acceptable limit.

b - Third attempt; difficulty with AE C614277.

c - Third attempt.

d - Third attempt repeated.

Multiresidue methods

The Meeting received a study report on the behaviour of pyrimethanil when analysed by the US FDA multiresidue analytical protocols (04/1989) using protocols C, D, E (A81743, Anson-Moye and Anderson, 1995, Study AN-93R-02). Protocols A and B do not apply, as pyrimethanil is not an N-methyl carbamate, an acid or a phenol. Pyrimethanil was non-detectable with the electron capture detectors at typical residue levels (protocol C). When fortified grapes (non-fat food) were analysed by the method of Protocol D (Luke procedure), recovery was complete (100%) at 0.05 and 5.0 mg/kg. Recovery by Protocol E was poor for both grapes and cottonseed (fatty food) fortified at 0.05 mg/kg.

Multiresidue testing via the US FDA protocols (01/94) was also reported for the metabolites *AE C614276* and *AE C614277* (B003927, Lala and Mollica, 2002, Study 02AN33181). Neither metabolite was recovered from skim milk or meat utilising protocol B. The compounds were tested under protocol C, gas chromatographic screenings with electron capture detection and nitrogen phosphorus detection. Reasonable retention times and responses were demonstrated with several modules (for the methylated derivatives). Both metabolites had inadequate recoveries via Protocol D when spiked into a non-fatty matrix at 0.05 and 0.25 mg/kg. Likewise recovery was poor under Protocols E and F.

Multiresidue method DFG S 19 was tested for pyrimethanil in grapes, wine and apples (A81677, Specht, 1993, Report U/R 29/92). The method was slightly modified by the addition of sodium bicarbonate in the extraction step. This was needed to accommodate the instability of pyrimethanil in acid medium. Solvent extraction was followed by gel permeation chromatography and clean-up with silica. Pyrimethanil was determined by GC with thermo ionic alkali flame ionization detection (FID). Recovery data are summarized in Table 29.

Table 29. Recovery of pyrimethanil from fortified grapes, wine, and apples via DFG S-19 (n = 3) (A81677)

Matrix	Fortification (mg/kg)	Recovery		
		%	Mean (at fortification)	Mean (for matrix), s.d.
Grapes	0.05	93, 98, 99	97	95
	0.5	86, 95, 100	94	5.5
Wine	0.05	77, 77, 82	79	80
	0.5	77, 80, 85	81	4.2
Apples	0.05	92, 92, 95	93	90
	0.5	82, 86, 93	87	5.5

The US FDA multiresidue methods were validated for pyrimethanil in bananas (C002769, Godfrey, 1999, Report RESID/98/35). Pyrimethanil was recoverable via the Luke method. Samples were homogenized with acetone. Extracts were cleaned on florisil and eluted with 50% dichloromethane/1.5% acetonitrile/49.5% hexane (Protocol D). Analysis was by GC/MS, external calibration utilising the total ion chromatogram. Results are summarized in Table 30.

Table 30. Recovery of Pyrimethanil from bananas via US FDA multiresidue Protocol (C002769)

Fortification (mg/kg)	Recovery		
	%	Mean (at fortification)	Mean (for matrix), s.d.
0.05	74 95 101 111 118	100	95 14.
0.50	83 86 88 90 106	91	

Stability of pesticide residues in stored analytical samples

Frozen storage stability studies on a variety of crops were provided to the Meeting. Control samples were fortified with a known concentration of pyrimethanil and placed in frozen storage at about -20 °C. The fortified control samples were removed from storage periodically and analysed by the method(s) used for field trials or processing study samples. Concurrent recoveries were measured by the analysis of controls fortified on the day of analysis of storage stability samples.

Results of the various studies are summarized in Table 31.

Table 31. Stability of Pyrimethanil in various commodities as a function of storage interval (-20 °C)

Commodity	Fortification (mg/kg)	Storage interval	% Recovery (average)	% Concurrent recovery	Reference
Apple	0.5	35 days	99, 92, 94 (95)	96	A81717
		225 days	90, 87, 89 (89)	94	
		351 days	63, 71, 72 (69)	90	
Grapes	2.5	41 days	97, 89, 92 (93)	98	A81649
		122 days	93, 95, 97 (95)	95	
		217 days	91, 89, 87 (89)	96	
		359 days	81, 84, 87 (84)	95	
Tomatoes	0.57 (average incurred residue)	105 days	100, 85. 118 (101)	88	A81714
		184 days	107, 99, 115 (107)	80	
		385 days	76, 83, 113 (94)	81	
Lettuce	0.5	0 day	106, 92, 106 (101)	-	C000967
		32 days	70, 58, 53 (60)	58	
		96 days	92, 96, 94 (94)	101	
		188 days	122, 112, 136 (123)	105	
		280 days	98, 98, 98 (98)	90	
		385 days	106, 108. 108 (107)	99	
Carrots	0.5	0 days	90, 84, 86 (87)	-	C000968
		34 days	64, 64, 66 (65)	73	
		96 days	60, 80, 64 (68)	75	
		188 days	76, 92, 90 (86)	89	
		279 days	82, 78, 82 (81)	84	
		368 days	82, 88, 86 (85)	82	
Peas (dried)	0.5	0	79, 78, 81 (79)	-	C021512
		1 mo	76, 85, 81 (81)	72 (at 0.1 mg/kg)	
		3 mo	98, 69, 88 (85)	86	

Commodity	Fortification (mg/kg)	Storage interval	% Recovery (average)	% Concurrent recovery	Reference
		6 mo	77, 77, 83 (79)	79	
		9 mo	67, 71, 64 (67)	73	
		12 mo	80, 78, 77 (78)	81	
Peach	0.5	435 days	82, 84, 84 (83)	97	PR 08700
Plum	0.5	345 days	81, 78, 85	88	PR 08702

USE PATTERN

Pyrimethanil is applied both pre-harvest and post-harvest for the control of a number of diseases in a wide range of crops. The various national GAPs relevant to the field trial studies and post-harvest treatment studies reported herein are summarized in Table 32 and in Table 33, respectively. These summaries have been assembled from labels (or English translations of labels) supplied by the manufacturers.

Table 32. Summary of pre-harvest GAP uses of pyrimethanil

Crop	Country	Formulation	Application				PHI days
			Method	kg ai/ha	kg ai/hL	No. or max (kg ai/ha/ season)	
Almonds	USA	600 g/L SC	Foliar	0.8	-	no more than 2.4 kg ai/season	30
Pome fruit (apples and pears)	Belgium	400 g/L SC	Foliar	0.45	0.22	5	28
	France	400 g/L SC	Foliar	-	0.02	4	28
	Germany	400 g/L SC	Foliar	-	0.03	5	(c)
	Greece	400 g/L SC	Foliar	-	0.08	2	28
	Italy	400 g/L SC	Foliar	-	0.03- 0.04	5	14
	Netherlands	400 g/L SC	Foliar	-	0.03	5	28
Pome fruit (apples and pears)	UK	400 g/L SC	Foliar	0.45	0.09	5	(a)
	USA	600g/L SC	Foliar	0.45	-	1.8 kg ai/ha/season	72
	Australia	50 g/L fluquinconazole + 200 g/L pyrimethanil SC	Foliar	-	0.015	3	(f)
	Germany	50 g/L fluquinconazole + 200 g/L pyrimethanil SC	Foliar	0.1		5	28
	Greece	50 g/L fluquinconazole + 200 g/L pyrimethanil SC	Foliar	-	0.02	2	56
	Italy	50 g/L fluquinconazole + 200 g/L pyrimethanil SC	Foliar	0.4	0.02	5	21
Banana	Latin America (d)	600 g/L SC	Foliar/ aerial	0.3	-	6 - 8	0(e)
	Brazil	300 g/L SC	Foliar	0.3	-	As needed; aerial	3
Beans	Italy	400 g/L SC	Foliar	0.6	0.08	2	14
	Spain	400 g/L SC	Foliar	0.6- 0.8	-	1	14
	France	400 g/L SC	Foliar	0.6	-	-	14

Crop	Country	Formulation	Application				PHI days
			Method	kg ai/ha	kg ai/hL	No. or max (kg ai/ha/ season)	
	UK	150 g/L pyrimethanil + 375 g/L chlorothalonil	Foliar	0.3	-	2	56
Carrot	Brazil	300 g/L SC	Foliar	0.6	0.06	As needed	14
	France	400 g/L SC	Foliar	0.8	-	2	21
	Italy	400 g/L SC	Foliar	0.8 low volume	0.08	2	7
	France	150 g/L pyrimethanil + 375 g/L chlorothalonil	Foliar	0.3	-	2	21
Cucumber	Spain	400 g/L SC	Foliar	-	0.08		3
Eggplant	Belgium	400 g/L SC	Foliar	0.4	-		1
	Spain	400 g/L SC	Foliar	-	0.08		3
Grapes	Australia	200 g/L SC	Foliar	0.8 Concentrate spray	0.08 Dilute spray	1 - 2	7
	USA	600 g/L SC	Foliar	0.8	-	no more than 1.6 kg ai/season	7
	Brazil	300 g/L SC	Foliar	0.8	0.06	As needed	21
	France	400 g/L SC	Foliar	1	-	1	21
	Germany	400 g/L SC	Foliar	0.2- 0.8 Devel stage 61: 0.4 71: 0.6 75: 0.8	-	3	28
	Greece	400 g/L SC	Foliar		0.06- 0.08	3	35
	Italy	400 g/L SC	Foliar	0.8 Low volume	0.08	3	21
	Spain	400 g/L SC	Foliar	-	0.08	1	21
Leeks	France	400 g/L SC	Foliar	0.8	-	2	14
Lettuce	Spain	400 g/L SC	Foliar (field, closed systems)	-	0.08	1	14
	France	400 g/L SC	Foliar (field, tunnel, glass-house)	0.8	-	2	21
	Italy	400 g/L SC	Foliar (open field)	0.8 low volume	0.08	2	14
Onion and other bulb vegetables: onion (green, dry bulb, welsh), garlic, leek, shallot	USA	600 g/L SC	Foliar	0.8	-	no more than 2.4 kg ai/season	7
Onion	Italy	400 g/L SC	Foliar	0.8 low volume	0.08	2	14
	Brazil	300 g/L SC	Foliar	0.6	0.06	As needed	3
Peas (canning)	France	400 g/L SC	Foliar	0.6	-	1	14

Crop	Country	Formulation	Application				PHI days
			Method	kg ai/ha	kg ai/hL	No. or max (kg ai/ha/ season)	
	France	150 g/L pyrimethanil + 375 g/L chlorothalonil	Foliar	0.3	-	-	14
Peas, field (protein, combining)	France	400 g/L SC	Foliar	0.6	-	-	28
	France	150 g/L pyrimethanil + 375 g/L chlorothalonil	Foliar	0.3	-	2	14
Peas, field (protein, combining)	UK	150 g/L pyrimethanil + 375 g/L chlorothalonil	Foliar	0.3	-	2	42
Pepper	Spain	400 g/L SC	Foliar	-	0.08	1	3
Pistachios	USA	600 g/L SC	Foliar	0.8	-	no more than 2.4 kg ai/season	30
Pome fruit: apple pear, crab-apple, loquat, mayhaw, quince	USA	600 g/L SC	Foliar	0.4	-	no more than 1.8 kg ai/season	72
Potato and other tuberous and corm vegetables: Sweet potato, arracha, arrowroot, artichoke, edible canna, cassava, chayote, chufa, dasheen, ginger, leren, tanier, turmeric, yam bean, true yam	USA	600 g/L SC	Foliar	0.3	-	no more than 1.6 kg ai/season	7
Potato	Australia	375 g/L chlorothalonil + 150 g/L pyrimethanil	Foliar	0.3	-	1 - 3	Do not graze or cut for feed
	Brazil	300 g/L SC	Foliar	0.30- 0.45	0.05	-	3
Stone fruit (except cherries)	USA	600 g/L SC	Foliar	0.8		no more than 2.4 kg ai/season	2
Strawberries	Australia	400 g/L SC	Foliar	0.3	-	1 - 3	1
	USA	600 g/L SC	Foliar	0.8		no more than 2.4 kg ai/season	1
	Belgium	400 g/L SC	Foliar	0.008	-	3	3
	France	400 g/L SC	Foliar	0.8		2	3
	Germany	400 g/L SC	Foliar	1		3	7
	Italy	400 g/L SC	Foliar	0.8 low volume	0.08	-	3
	Netherlands	400 g/L SC	Foliar	0.8	-	-	3
	Spain	400 g/L SC	Foliar	-	0.08	1	3
	UK	400 g/L SC	Foliar	0.8	0.08	2	1

Crop	Country	Formulation	Application				PHI days
			Method	kg ai/ha	kg ai/hL	No. or max (kg ai/ha/ season)	
Tomato	USA	600 g/L SC	Foliar	0.3	-	no more than 1.6 kg ai/season	1
	Belgium	400 g/L SC	Foliar	0.4	-	-	1
	Brazil	300 g/L SC	Foliar	0.75- 0.9	0.08- 0.09	As required	3
	France	400 g/L SC	Foliar (field, tunnel, glass-house)	0.8	-	2	3
	Greece	400 g/L SC	Foliar (outside and glass house)	-	0.06- 0.08	-	3
	Italy	400 g/L SC	Foliar	0.8 low volume	0.08	-	3
	Netherlands	400 g/L SC	Foliar (glass house)	-	0.04	3	1
	Spain	400 g/L SC	Foliar	-	0.08	-	3
	Australia	375 g/L chlorothalonil + 150 g/L pyrimethanil SC	Foliar	0.3	-	1 - 3	1

a - PHI not specified in UK label.

b - EU approved GAP, which is the basis for MRL proposed for pome fruit and grapes in EU.

c - German label states that PHI is not necessary since application is based on growth stages and harvest time and would depend on conditions towards fruit maturity.

d - Latin America – Similar labels for Costa Rica, Colombia, Dominican Republic, Ecuador, Guatemala, and Honduras

e - For bananas, harvesting is continuous as fruits mature and can start even on the day of last treatment

f - Do not apply later than 4 weeks after petal fall.

Table 33. Summary of post-harvest GAP uses of pyrimethanil

Crop	Country	Formulation	Application			
			Method	Time (minutes)	Rate, kg ai/hL	No. (max)/ comment
Cherry	Chile	400 g/L SC	Dipping	1	0.04	1
	Chile	400 g/L SC	Wax line spray	1	0.04	1
Citrus	USA	400 g/L SC	Wax line spray: storage and pack wax	1	0.2	2 or 3 post-harvest treatment combinations
	USA	400 g/L SC	Dip (wash tank)	2	0.1	
	USA	400 g/L SC	Drench	2-4	0.05	
	USA	400 g/L SC	Aqueous line spray	1	0.2	
	USA	204 g/L pyrimethanil + 263 g/L imazalil SC	Dip and wash tank	2	0.08 pyrimethanil	2 total post-harvest mixed treatments
	USA	204 g/L pyrimethanil + 263 g/L imazalil SC	Drench	4	0.08 pyrimethanil	2 total post-harvest mixed treatments

Crop	Country	Formulation	Application			
			Method	Time (minutes)	Rate, kg ai/hL	No. (max)/ comment
	USA	204 g/L pyrimethanil + 263 g/L imazalil SC	Aqueous line spray	-	0.1 pyrimethanil	Use 379 L of solution per 3400 –4540 kg fruit. 2 total post-harvest mixed treatments
	USA	204 g/L pyrimethanil + 263 g/L imazalil SC	Storage wax line	-	0.2 pyrimethanil	Use 379 L of solution per 1720 kg fruit. 2 total post-harvest mixed treatments
	Chile	400 g/L SC	Dipping	2	0.1	1
	Chile	400 g/L SC	Drench	2-4	0.05	1
	Chile	400 g/L SC	Aqueous line spray	-	0.2	1
	Chile	400 g/L SC	Wax line spray	1	0.2	1
	Uruguay	200 g/L pyrimethanil + 200 g/L imazalil SC	Drench		0.1	2
	Uruguay	200 g/L pyrimethanil + 200 g/L imazalil SC	Wax spray		0.2	
	Uruguay	200 g/L pyrimethanil + 200 g/L imazalil SC	Aqueous line spray		0.2	
Lemon	Argentina	200 g/L pyrimethanil + 200 g/L imazalil SC	Drench		0.1	1
	Argentina	200 g/L pyrimethanil + 200 g/L imazalil SC	Aqueous line spray		0.2	1
	Argentina	200 g/L pyrimethanil + 200 g/L imazalil SC	Wax line spray		0.2	1
Pome fruit	Belgium	200 g/L pyrimethanil + 200 g/L imazalil SC	Dip		0.04	1 (1 day PHI)
	Belgium	200 g/L pyrimethanil + 200 g/L imazalil SC	Aqueous line spray		0.04	1 (1 day PHI)
	USA	400 g/L SC	Dipping	1	0.05 – 0.1	Up to maximum combination of 2 methods: (1) drench + dip; (2) drench + wax; (3) drench + aq. Spray; (4) dip + wax; (5) dip + aq. Spray; (6) aq. Spray + wax
	USA	400 g/L SC	Drench	1	0.05 – 0.1	
	USA	400 g/L SC	Aqueous line spray	1	0.1	
	USA	400 g/L SC	Wax line spray	1	0.2	
	Chile	400 g/L SC	Dipping	1	0.05 – 0.1	1
	Chile	400 g/L SC	Drench	1	0.05 – 0.1	1
	Chile	400 g/L SC	Aqueous line spray	1	0.1	1
	Chile	400 g/L SC	Wax line spray	1	0.2	1
	Chile[1]	160 g/kg NH	Thermofogging		8 g ai/ton fruits	1

Crop	Country	Formulation	Application			
			Method	Time (minutes)	Rate, kg ai/hL	No. (max)/ comment
	Uruguay	200 g/L pyrimethanil + 200 g/L imazalil SC	Dip		0.05	2
	Uruguay	200 g/L pyrimethanil + 200 g/L imazalil SC	Drench		0.05	
	Uruguay	200 g/L pyrimethanil + 200 g/L imazalil SC	Aqueous line spray		0.03	
	Uruguay	200 g/L pyrimethanil + 200 g/L imazalil SC	Wax spray		0.03	
Stone fruit (except cherries) [a]	USA	400 g/L SC	Wax or dip	30 sec	0.05 - 0.1	1
			high volume spray	30 sec	0.08- 0.12	1
			low volume spray	30 sec	0.4- 1.2	1
Stone fruit [a]	Uruguay	400 g/L SC	Dipping	45 sec	0.05	1

a - ***Proposed*** GAPs expected to be authorized 2007.

RESIDUES RESULTING FROM SUPERVISED TRIALS

The results of supervised trials are shown in Tables 35 to 62. Where multiple samples were taken from a single plot or multiple analyses conducted on a single sample, the average value is reported. Where results from separate plots were reported, results are listed for each plot. Results have not been corrected for concurrent method recoveries unless indicated. The following table summarizes information on residues resulting from supervised trials.

Table 34. Field Trial Tables

Group	Table No.	Commodity
Citrus	35	Citrus (post-harvest)
Pome fruit	36	Apples (USA)
Pome fruit	37	Apples (Belgium, Italy)
Pome fruit	38	Pears
Pome fruit	39	Apples (post-harvest)
Pome fruit	40	Pears (post-harvest)
Pome fruit	41	Apples/Pears (thermofog)
Stone fruit	42	Apricots
Stone fruit	43	Peach
Stone fruit	44	Plums
Stone fruit	45	Peach/plum (post-harvest)
Stone fruit	46	Cherries (post-harvest)
Berries and other small fruits	47	Grapes (USA)
Berries and other small fruits	48	Grapes (France, Spain)
Berries and other small fruits	49	Strawberries
Assorted tropical fruits with inedible peel	50	Banana
Bulb vegetables	51	Onions (dry bulb; green)
Fruiting vegetables other than cucurbits	52	Tomatoes (USA)
Fruiting vegetables other than cucurbits	53	Tomatoes (glass house: France, Netherlands)
Leafy vegetables	54	Lettuce (head)
Leafy vegetables	55	Lettuce (leaf)

Group	Table No.	Commodity
Leafy vegetables	56	Lettuce (head) (glass house)
Legume vegetables	57	Green beans
Legume vegetables	58	Beans (glass house)
Root and tuber vegetables	59	Carrots
Root and tuber vegetables	60	Potatoes
Legume animal feeds	61	Field peas
Tree nuts	62	Almonds

Citrus

Table 35. Pyrimethanil residues in citrus fruit from post-harvest treatments

CITRUS	Post-harvest Application				Residues	Reference
country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.	mg/kg	
GAP, USA	204 g/L pyrimethanil +263 g/L imazalil SC	Dip, wash tanks	0.08	2 treatment combinations		
		Drench	0.08			
		Aqueous line spray	0.1			
		Wax line spray: storage and pack wax	0.2			
	400 g/L	Dip, wash tanks	0.1	2 or 3 treatment combinations		
		Drench	0.05			
		Aqueous line spray	0.2			
		Wax line spray; storage and pack wax	0.2			
LEMON Florida, USA, 2001 (Eureka)	400 g/L SC	Storage wax line + pack out wax spray	0.2+ 0.2	2	2.8 (3.05 2.54)	AGR 374
		Storage wax line + Aqueous dip	0.2+ 0.1	2	4.3 (4.13 4.52)	
		Aqueous dip	0.1	1	2.8 (2.84 2.79)	
		Aqueous dip + Pack out wax spray	0.1+ 0.2	2	4.6 (4.51 4.62)	
LEMON Florida, USA, 2001 (Eureka)	400 g/L SC	Storage wax line + pack out wax spray	0.2+ 0.2	2	2.8 (3.21 2.48)	AGR 374
		Storage wax line + Aqueous dip	0.2+ 0.1	2	3.6 (3.96 3.20)	
		Aqueous dip	0.1	1	2.5 (2.62 2.47)	
		Aqueous dip + Pack out wax spray	0.1+ 0.2	2	4.1 (4.24 4.05)	
ORANGE California, USA, 2001 (Valencia)	400 g/L SC	Aqueous dip	0.1	1	3.0 (2.91 3.10)	AGR 374
		Aqueous dip + Pack out wax spray	0.1+ 0.2	2	5.5 (5.59 5.47)	
GRAPEFRUIT California, USA, 2001 (Ruby Red	400 g/L SC	Aqueous dip	0.1	1	3.32 3.64	AGR 374
		Aqueous dip + Pack out wax spray	0.1+ 0.2	2	4.2 (4.44 4.00)	

CITRUS	Post-harvest Application				Residues	Reference
country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.	mg/kg	
ORANGE Florida, USA, 2001 (Navel)	400 g/L SC	Aqueous drench + pack out wax spray	0.05+ 0.2	2	2.7 (3.03 2.39)	AGR 374
		Aqueous line spray	0.2	1	1.3 (1.17 1.35)	
		Aq. line spray + pack out wax spray	0.2+ 0.2	2	2.7 (2.85 2.51)	
		Aqueous drench + Aq. line spray	0.05+ 0.2	2	1.2 (1.20 1.30)	
		Aqueous drench + Aq. line spray + Pack out wax spray	0.05+ 0.2+ 0.2	3	2.1 (1.89 2.32)	
ORANGE Florida, USA, 2001 (Hamlin)	400 g/L SC	Aqueous drench + pack out wax spray	0.05+ 0.2	2	2.2 (2.17 2.19)	AGR 374
		Aqueous line spray	0.2	1	1.1 (1.06 1.09)	
		Aq. line spray + pack out wax spray	0.2+ 0.2	2	2.6 (2.97 2.22)	
		Aqueous drench + Aq. line spray	0.05+ 0.2	2	1.2 (1.09 1.23)	
		Aqueous drench + Aq. line spray + Pack out wax spray	0.05+ 0.2+ 0.2	3	2.7 (2.74 2.73)	
TANGELO Florida, USA, 2001 (Orlando)	400 g/L SC	Aqueous drench + Aq. line spray	0.05+ 0.2	2	2.8 (2.66 3.00)	AGR 374
		Aqueous line spray	0.2	1	1.1 (1.04 1.08)	
		Aq. line spray + pack out wax spray	0.2+ 0.2	2	3.1 (3.19 3.09)	
		Aqueous drench + Aq. line spray	0.05+ 0.2	2	1.5 (1.47 1.58)	
		Aqueous drench + Aq. line spray + Pack out wax spray	0.05+ 0.2+ 0.2	3	3.3 (3.07 3.53)	
TANGERINE Florida, USA, 2001 (Sun)	400 g/L SC	Aqueous drench+ pack out wax spray	0.05+ 0.2	2	2.9 (2.90 2.82)	AGR 374
		Aqueous line spray	0.2	1	1.1 (1.09 1.04)	
		Aq. line spray + pack out wax spray	0.2+ 0.2	2	3.4 (3.38 3.35)	
		Aqueous drench + Aq. line spray	0.05+ 0.2	2	1.5 (1.29 1.65)	
		Aqueous drench + Aq. line spray + Pack out wax spray	0.05+ 0.2+ 0.2	3	3.4 (3.43 3.42)	

CITRUS	Post-harvest Application				Residues	Reference
country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.	mg/kg	
GRAPEFRUIT Florida, USA, 2001 (Flame)	400 g/L SC	Aqueous drench+ pack out wax spray	0.05+ 0.2	2	1.7 (1.78 1.64)	AGR 374
		Aqueous line spray	0.2	1	0.93 (0.89 0.97)	
GRAPEFRUIT Florida, USA, 2001 (Flame)	400 g/L SC	Aq. line spray + pack out wax spray	0.2+ 0.2	2	2.3 (2.30 2.26)	AGR 374
		Aqueous drench + Aq. line spray	0.05+ 0.2	2	1.2 (1.15 1.25)	
		Aqueous drench + Aq. line spray + Pack out wax spray	0.05+ 0.2+ 0.2	3	1.9 (1.94 2.01)	
LEMON California, USA, 2002 (Eureka)	400 g/L SC	Aqueous drench + aqueous dip	0.05 + 0.05	2	1.4 (1.52 1.21)	AGR 374
		Aqueous drench + aqueous dip + wax line spray	0.05 + 0.05 + 0.2	3	3.4 (3.22 3.51)	
		Aqueous drench + aqueous dip	0.05 + 0.1	2	4.1 (4.98 3.15)	
		Aqueous drench + aqueous dip + wax line spray	0.05 0.1+ 0.2	3	5.8 (5.30 6.21)	
ORANGE California, USA, 2002 (Olinda)	400 g/L	Aqueous drench + aqueous dip	0.05 + 0.05	2	1.3 (1.35 1.16)	AGR 374
		Aqueous drench + aqueous dip + wax line spray	0.05 + 0.05 + 0.2	3	1.9 (2.07 1.66)	
		Aqueous drench + aqueous dip	0.05 + 0.1	2	1.7 (1.77 1.70)	
		Aqueous drench + aqueous dip + wax line spray	0.05 0.1+ 0.2	3	2.8 (3.07 2.44)	
MANDARIN Spain, 2004 (Fortuna)	200 g/L pyrimethanil + 200 g/L Imazalil SC	Wax application	0.2	1	0.76	AGR 744-AGR 802a
		Wax application	0.3	1	0.93	
		Drench + Wax application	0.05 + 0.3	2	2.02	
MANDARIN Spain, 2004 (Oetanique)	200 g/L pyrimethanil + 200 g/L Imazalil SC	Wax application	0.2	1	0.61	AGR 744-AGR 802a
		Wax application	0.3	1	1.1	
		Drench + Wax application	0.05 + 0.3	2	1.9	
MANDARIN Spain, 2004 (Fortuna)	200 g/L pyrimethanil + 200 g/L Imazalil SC	Drench for 1 minute	0.04	1	0.97	AGR 744-AGR 802a
		Drench for 1 minute	0.05	1	1.3	
MANDARIN Spain, 2004 (Ortanique)	200 g/L pyrimethanil + 200 g/L Imazalil SC	Drench for 1 minute	0.04	1	1.0	AGR 744-AGR 802a
		Drench for 1 minute	0.05	1	1.9	
ORANGE Spain, 2004 (Valencia)	200 g/L pyrimethanil + 200 g/L Imazalil SC	Wax application	0.2	1	0.91	AGR 792
		Wax application	0.3	1	1.4	
		Drench (40 sec) + Wax application	0.05 + 0.3	2	2.2	

CITRUS	Post-harvest Application				Residues	Reference
country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.	mg/kg	
ORANGE Spain, 2004 (Lane late)	200 g/L pyrimethanil + 200 g/L Imazalil SC	Wax application	0.2	1	0.66	AGR 792
		Wax application	0.3	1	1.0	
		Drench (40 sec) + Wax application	0.05 + 0.3	2	1.5	
ORANGE Spain, 2004 (Valencia)	200 g/L pyrimethanil + 200 g/L Imazalil SC	Drench for 1 minute	0.04	1	1.3	AGR 792
		Drench for 1 minute	0.05	1	1.7	
ORANGE Spain, 2004 (Lane late)	200 g/L pyrimethanil + 200 g/L Imazalil SC	Drench for 1 minute	0.04	1	0.90	AGR 793
		Drench for 1 minute	0.05	1	1.4	

Pome fruit

A number of supervised trials on apples and pears conducted in USA, Northern EU (Germany, UK, and Northern France) and Southern EU (France, Italy and Spain) have been provided and are summarized below.

Table 36. Residues of pyrimethanil from supervised trials (pre-harvest) on apples in the USA (B002874)

APPLE	Application					PHI	Residues
country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	mg/kg
GAP, USA	400 g/L SC	0.45			1.8 kg ai/ha/season	72	
Trial R01-01 New York, USA, 1999 (McIntosh)	400 g/L SC	0.45	940	0.05	4	72	0.06
Trial R01-02 New York, USA, 1999 (McIntosh)	400 g/L SC	0.45	945	0.05	4	72	0.15
Trial R01-03 Pennsylvania, USA, 1999 (Red Delicious)	400 g/L SC	0.45	931	0.05	4	71	0.1
Trial R02-01 Virginia, USA, 1999 (Law Roane)	400 g/L SC	0.45	928	0.05	4	72	< 0.05
Trial R5-01 Michigan, USA, 1999 (Red Delicious)	400 g/L SC	0.45	928	0.05	4	72	< 0.05
Trial R5-02 Ohio, USA, 1999 (Liberty)	400 g/L SC	0.45	940	0.05	4	72	< 0.05
Trial R09-01 Colorado, USA, 1999 (Golden)	400 g/L SC	0.45	935	0.05	4	72	0.16

APPLE country, year (variety)	Application					PHI days	Residues mg/kg
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.		
Trial R10-01 California, USA, 1999 (Granny Smith)	400 g/L SC	0.45	940	0.05	4	72	0.12
Trial R11-01 Washington, USA, 1999 (Red Delicious)	400 g/L SC	0.45	938	0.05	4	72	< 0.05
Trial R11-02, Washington, USA, 1999 (Red Delicious)	400 g/L SC	0.45	945	0.05	4	72	< 0.05
Trial R11-03, Washington, USA, 1999 Ace/MM106 Rootstock	400 g/L SC	0.45	931	0.05	4	72	< 0.05
Trial R11-03, Idaho, USA, 1999 Spur Rome	400 g/L SC	0.45	946	0.05	4	73	< 0.05

Table 37. Residues of pyrimethanil from supervised trials (pre-harvest) on apples in the EU

APPLE country, year (variety)	Application					PHI days	Residues mg/kg	Reference
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.			
GAP, Belgium	*400 g/L SC*	*0.45*		*0.22*	*5*	*28*		
Trial 01R685-1, Germany, 2001 (Rubin)	400 g/L SC	0.6	1000	0.06	5	0	2.6	Ref C017379
						14	0.76	
						28	0.35	
						42	0.30	
						56		
Trial 01R685-2, Germany, 2001 (Elstar)	400 g/L SC	0.6	1000	0.06	5	0	1.6	Ref C017379
						14	0.43	
						28	0.39	
						43	0.25	
						57	0.18	
Trial 01R685-3 France, 2001 (Judeline)	400 g/L SC	0.6	1000	0.06	5	0	2.3	Ref C017379
						14	1.5	
						28	0.98	
						42	0.98	
						56	0.60	
Trial 01R685-4 France, 2001 (Golden Delicious)	400 g/L SC	0.6	1000	0.06	5	0	1.9	Ref C017379
						16	0.77	
						29	0.63	
						42	0.31	
						56	0.30	
Trial 01R685-4 UK, 2001 (Golden Delicious)	400 g/L SC	0.6	1000	0.06	5	0	1.7	Ref C017379
						13	1.2	
						26	0.78	
						41	0.57	
						57	0.45	
GAP, Italy	*400 g/L SC*		-	*0.04*	*5*	*14*		
Trial 01R686-1 Spain, 2001 (Starking)	400 g/L	0.6	1000	0.06	5	0	1.4	Ref C017380
						14	0.26	
						28	0.16	
						42	0.07	
						56	< 0.05	

APPLE country, year (variety)	Application					PHI	Residues mg/kg	Reference
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days		
Trial 01R686-2 France, 2001 (Golden Delicious)	400 g/L	0.6	1000	0.06	5	0	1.3	Ref C017380
						14	0.84	
						28	0.66	
						42	0.34	
						56	0.37	
Trial 01R686-3 France, 2001 (Granny Smith)	400 g/L	0.6	775	0.08	5	0	1.2	Ref C017380
						14	0.48	
						28	0.34	
						42	0.26	
						56	0.16	
Trial 01R686-4 Italy, 2001 (Golden Delicious)	400 g/L	0.6	1000	0.06	5	0	2.7	Ref C017380
						14	1.5	
						28	1.0	
						42	0.51	
						56	0.30	
Trial 01R686-5 Italy, 2001 (Golden Delicious)	400 g/L	0.6	1500	0.04	5	0	1.3	Ref C017380
						14	0.56	
						28	0.42	
						42	0.24	
						56	0.15	

A total of six supervised field trials on pears were conducted in major pear growing areas of the USA in 1999, three in Washington, two in California and one in Pennsylvania (B003706, Dacus, 2002a; Report AN99R016).

Table 38. Residues of pyrimethanil from supervised trials (pre-harvest) on pears in the USA (B003706)

PEARS Country, year (variety)	Application					PHI	Residues, mg/kg
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	No.	days	
GAP, USA	600 g/L SC	0.45			total of 1.8 kg ai/ha/season	72	
Trial R01-01 Pennsylvania, USA, 1999 (Bartlett)	400 g/L SC	0.45	946	0.05	4	72	ND
Trial R10-01 California, USA, 1999 (Bartlett)	400 g/L SC	0.45	904	0.05	4	72	ND
Trial R10-02 California, USA, 1999 (Bose)	400 g/L SC	0.45	940	0.05	4	72	ND
Trial R11-01 Washington, USA, 1999 (d'Anjou)	400 g/L SC	0.45	939	0.05	4	65	ND
						72	ND
						79	ND
						86	ND
						93	ND
Trial R11-02 Washington, USA, 1999 (d'Anjou)	400 g/L SC	0.45	905	0.05	4	72	ND
Trial R11-03 Washington, USA, 1999 (Bartlett)	400 g/L SC	0.45	938	0.045	4	72	ND

ND: LOD calculated as 0.012, or 3 × SD of the recovery of the lowest fortification plus the apparent concentration in the controls. LOQ demonstrated to be 0.05 mg/kg.

The Meeting also received studies on the post-harvest treatment of pome fruit, as summarized below.

Table 39. Pyrimethanil residues in apples from post-harvest treatments (dip, drench, wax spray, aqueous spray, or combinations of methods)

APPLE	Post-harvest Application				Residues mg/kg	Reference
country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.		
BELGIUM GAP	200 g/L pyrimethanil + 200 g/L imazalil SC	Spray	0.04	1		
		Dip	0.04			
Spain, 2002 (Starking Delicious)	600 g/L SC	Drench for 30 seconds	0.04	1	0.86 1.16	Serrano, 2003a Report RA-062, [AGR 493]
Spain, 2002 (Esperiaga)	600 g/L SC	Drench for 30 seconds	0.04	1	2.14 1.99	
France, 2001 (Fuji No.6)	100 g/L pyrimethanil + 200 g/L Imazalil EC	Dipping for 30 seconds	0.02	1	0.39	Perny, 2003a Report RA1203, [AGR 407]
			0.03	1	0.57	
France, 2001 (Fuji No. 6)	150 g/L pyrimethanil + 150 g/L Imazalil EC	Dipping for 30 seconds	0.02	1	1.14	Perny, 2003b Report R A1207, [AGR 412]
			0.03	1	1.66	
			0.05	1	1.54	
Spain, 2001 (Golden Delicious)	100 g/L pyrimethanil + 200 g/L Imazalil	Drench for 40 seconds	0.02	1	0.56	Serrano, 2003b Report RA-045 [AGR 403]
Spain, 2001 (Red Chief)	100 g/L pyrimethanil + 200 g/L Imazalil	Drench for 40 seconds	0.02	1	0.54	Serrano, 2003b Report RA-045 [AGR 403]
Spain, 2001 (Golden Delicious)	150 g/L pyrimethanil + 150 g/ Imazalil EC	Drench for 40 seconds	0.05	1	1.15	Serrano, 2003b Report RA-045 [AGR 403]
			0.07	1	1.23	
Spain, 2001 (Red Chief)	150 g/L pyrimethanil + 150 g/ Imazalil	Drench for 40 seconds	0.05	1	1.35	Serrano, 2003b Report RA-045 [AGR 403]
			0.07	1	1.0	
Spain, 2003 (Golden suprema)	200 g/L pyrimethanil + 200 g/L Imazalil	Drench for 40 seconds	0.03	1	0.68	Martos, 2004a; Report RA-080 [AGR 659]
			0.04	1	1.96	
			0.08	1	3.78	
GAP, USA *GAP, CHILE*	400 g/L SC	Dipping up to 1 minute	0.1	USA: Up to max. combination of 2 methods, each applied once Chile: 1		
		Drench up to 1 minute	0.1			
		Aq. line spray	0.1			
		Wax line spray	0.2			
Washington, USA, 2001 (Red Delicious)	400 g/L pyrimethanil SC	Aq. dip/drench	0.05	1	0.59	Carringer and Loriau, 2002 [AGR 386]
		Aq. line spray	0.1	1	0.27	
		Wax line spray	0.2	1	1.10	
		Aqueous dip + aq. line spray	0.05 + 0.1	2	0.44	
		Aqueous dip + wax line spray	0.05 + 0.2	2	1.18	
		Aqueous dip + wax line spray	0.1 + 0.1	2	1.90	
New York, USA, 2001 (Empire)	400 g/L pyrimethanil SC	Aqueous dip + aq. line spray	0.05 + 0.1	2	1.12	Carringer and Loriau, 2002 [AGR 386]
		Aqueous dip + aq. line spray	0.1 + 0.1	2	1.01	
California, USA, 2001	400 g/L pyrimethanil	Aq. dip/drench	0.05	1	0.82	Carringer and Loriau, 2002
		Aq. line spray	0.1	1	0.28	

APPLE	Post-harvest Application				Residues	Reference
country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.	mg/kg	
(Granny Smith)	SC	Wax line spray	0.2	1	0.64	[AGR 386]
		Aqueous dip + aq. line spray	0.05 + 0.1	2	0.51	
		Aqueous dip + wax line spray	0.05 + 0.2	2	0.67	
		Aqueous dip + wax line spray	0.1+ 0.1	2	0.79	
Washington, USA, 2001 (Fuji apple)	400 g/L pyrimethanil SC	Aq. dip/drench	0.05	1	0.63	Carringer and Loriau, 2002 [AGR 386]
		Aq. line spray	0.1	1	0.33	
		Wax line spray	0.2	1	1.22	
		Aqueous dip + aq. line spray	0.05 + 0.1	2	0.55	
		Aqueous dip + wax line spray	0.05 + 0.2	2	1.36	
		Aqueous dip + wax line spray	0.1 + 0.1	2	1.49	
New York, USA, 2001 (Empire)	400 g/L pyrimethanil SC	Aq. dip/drench	0.05	1	0.77	Carringer and Loriau, 2002 [AGR 386]
		Aq. line spray	0.1	1	1.51	
California, USA, 2001 (Fuji apples)	400 g/L pyrimethanil SC	Aq. dip/drench	0.05	1	1.34	Carringer and Loriau, 2002 [AGR 386]
		Aq. line spray	0.1	1	0.39	
		Wax line spray	0.2	1	0.70	
		Aqueous dip + aq. line spray	0.05 + 0.1	2	0.76	
		Aqueous dip + wax line spray	0.05 + 0.2	2	0.86	
		Aqueous dip + wax line spray	0.1+ 0.1	2	1.19	
New York, USA, 2001 (Red Delicious)	400 g/L pyrimethanil SC	Aq.dip/drench	0.05	1	0.71	AGR 386
		Aq. line spray	0.1	1	1.10	
		Aq. dip + aq. line spray	0.05 + 0.1	2	1.51	
		Aqueous dip + aq. line spray	0.1 + 0.1	2	1.18	

Table 40. Pyrimethanil residues in pears from post-harvest treatments (dip, drench, wax spray, aqueous spray, or combinations of methods)

PEAR	Post-harvest Application				Residues	Reference
Country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.	mg/kg	
GAP, USA GAP, CHILE	400 g/L pyrimethanil	Drench/dipping up to 1 minute	0.1	USA: Up to max. combination of 2 methods, each applied once. Chile: 1		
		Drench/dipping up to 1 minute	0.1			
		Aq. line spray	0.1			
		Wax line spray	0.2			
GAP, BELGIUM	200 g/L pyrimethanil + 200 g/L imazalil SC	Spray	0.04	1		
France, 2001 (Doyenné du Comice)	100 g/L pyrimethanil + 200 g/L Imazalil EC	Dipping for 30 seconds	0.01	1	0.27	AGR 404
			0.02	1	0.22	
			0.03	1	0.32	

PEAR	Post-harvest Application				Residues mg/kg	Reference
Country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.		
Spain, 2001 (Decana)	100 g/L pyrimethanil + 200 g/L Imazalil EC	Drench for 40 seconds	0.02	1	0.21	AGR 403
Spain, 2001 (Flor de invierno)	100 g/L pyrimethanil + 200 g/L Imazalil EC	Drench for 40 seconds	0.02	1	0.30	AGR 403
Spain, 2001 (Decana)	150 g/L pyrimethanil + 150 g/ Imazalil	Drench for 40 seconds	0.05	1	0.66	AGR 403
			0.07	1	0.64	
Spain, 2001 (Flor de invierno)	150 g/L pyrimethanil + 150 g/ Imazalil EC	Drench for 40 seconds	0.05	1	0.57	AGR 403
			0.07	1	0.57	
Belgium, 2001 (Conférence)	100 g/L pyrimethanil + 200 g/L Imazalil EC	Dipping for 30 seconds	0.02	1	0.41	AGR 402
			0.03	1	0.55	
Washington, USA, 2001 (D'anjou)	400 g/L pyrimethanil SC	Aqueous dip/drench	0.05	1	0.41	AGR 386
		Aqueous line spray	0.1	1	0.32	
		Wax line spray	0.2	1	0.86	
		Aqueous dip + aq. line spray	0.05 + 0.1	2	0.36	
		Aqueous dip + wax line spray	0.05 + 0.2	2	0.86	
		Aqueous dip + wax line spray	0.1+ 0.1	2	1.07	
Washington, USA, 2001 (Bosc)	400 g/L pyrimethanil SC	Aqueous dip/drench	0.05	1	1.03	AGR 386
		Aqueous line spray	0.1	1	0.45	
		Wax line spray	0.2	1	1.13	
		Aqueous dip + aq. line spray	0.05 + 0.1	2	1.46	
		Aqueous dip + wax line spray	0.05 + 0.2	2	2.53	
Washington, USA, 2001 (Bosc)	400 g/L pyrimethanil SC	Aqueous dip + wax line spray	0.1+ 0.1	2	2.66	AGR 386
California, USA, 2001 (Shinko)	400 g/L pyrimethanil SC	Aqueous dip/drench	0.05	1	1.57	AGR 386
		Aqueous line spray	0.1	1	0.13	
		Wax line spray	0.2	1	1.10	
		Aqueous dip + aq. line spray	0.05 + 0.1	2	0.91	
		Aqueous dip + wax line spray	0.05 + 0.2	2	1.78	
		Aqueous dip + wax line spray	0.1+ 0.1	2	1.91	
California, USA, 2001 (Bosc)	400 g/L pyrimethanil SC	Aqueous dip/drench	0.05	1	1.25	AGR 386
		Aqueous line spray	0.1	1	0.18	
		Wax line spray	0.2	1	0.56	
		Aqueous dip + aq. line spray	0.05 + 0.1	2	0.43	
		Aqueous dip + wax line spray	0.05 + 0.2	2	0.84	
		Aqueous dip + wax line spray	0.1+ 0.1	2	0.56	

Thermofogging is a process of applying post-harvest chemical to fruit in cold storage chambers obviating the need for drenching. Chemicals used for thermofogging are specially formulated for the process. During the fogging process, chemicals are pumped into the thermofogging

machine where they are flash heated to 165 – 170 °C in a fast flow of air, and are dispersed as a fog made up of extremely small particles. The fog is dispersed throughout the room and all fruits are coated with the chemical regardless of the position in the chamber or within the bin.

A total of 8 post-harvest trials were conducted in Italy, 4 on apples and 4 on pears, following the above GAP (ISPaVe-PRM-IML-T-15/02, Pompi, 2004). Apples and pears were placed in plastic cases (50 × 30 × 25 cm), each case with about 9 kg of fruit. Each trial consisted of one control and four treated replicates. During the trials, samples were stored in cold storage. In the treatment cell, four areas were marked; one for every replicate. Every area was further subdivided into 8, where the cases were placed in fully randomized piles. Treatment was made by thermofogging, applying a formulation containing a mixture of 120 g/L pyrimethanil and 120 g/L imazalil at the rate of 60 g of the formulation per ton of fruit, equivalent to 7.2 g ai/ton fruit. Only one treatment was made at the beginning of storage period. After treatment, the temperature of the treatment cell was regulated to + 0.5 °C for 24 h, after which the cases were transferred into refrigerated storage (also at + 0.5 °C) where they were stored up to 72 days. Samples were withdrawn on Days 1, 15, 28, 50l and 72 days after treatment.

One post-harvest trial was conducted in France to determine residues of pyrimethanil on apples (Jonagold variety) treated by thermofogging with the product described as a hot nebulizing product containing 160 g/L pyrimethanil (Xedathane-A HN). One treatment was made at a dose equivalent to 8 g ai/ton of fruits (Loriau, 2007; Report XDA-G002-07).

Fruits were placed in 12 palloxes and were set in a cell (33 m^2). The cell was closed during treatment. Fogging was done for about 5 minutes. Just after application, ventilation into the cell was turned on for 15 seconds to homogenize the fog into the cell. The cell was kept closed for more than 4 h before opening.

Untreated samples (12 fruits or about 1 kg) were collected just before thermofogging. Treated samples were collected after thermofogging application when fog was dissipated and the apple surface was dry. Twelve fruits were manually selected to obtain a minimum of about 1 kg of fruits. Samples were taken from the third top and from the third bottom of the pallox. Samples were kept in separate bags, labelled, and shipped frozen to the laboratory.

Table 41. Pyrimethanil residues in pome fruit resulting from thermofogging

POME	Post-harvest Application					Residues	Reference
Country, year (variety)	Formulation.	Treatment Method	Rate (g ai/ton fruit)	No.	Sampling (day)	mg/kg	
APPLES							
Trial MAT Italy, 2002 (Golden Delicious)	160 g/kg HN	Thermofogging	7.2	1	1	1.1	ISPaVe-PRM-IML-T-15/02
					15	1.3	
					28	1.2	
					50	1.4	
					72	1.2	
Trial MBT Italy, 2002 (Stark Delicious)	160 g/kg HN	Thermofogging	7.2	1	1	1.2	ISPaVe-PRM-IML-T-15/02
					15	1.4	
					28	1.6	
					50	1.4	
					72	1.3	
Trial MCT Italy, 2002 (Pink Lady)	160 g/kg HN	Thermofogging	7.2	1	1	0.73	ISPaVe-PRM-IML-T-15/02
					15	1.2	
					28	1.1	
					50	1.3	
					72	1.4	
Trial MDT Italy, 2002 (Fuji)	160 g/kg HN	Thermofogging	7.2	1	1	1.1	ISPaVe-PRM-IML-T-15/02
					15	0.78	
					28	1.0	
					50	0.86	
					72	0.81	

POME	Post-harvest Application					Residues	Reference
Country, year (variety)	Formulation.	Treatment Method	Rate (g ai/ton fruit)	No.	Sampling (day)	mg/kg	
Trial G002-06F France, 2006 (Jonagold)	160 g/kg HN	Thermofogging	8	1	Top Bottom	1.5 1.1	XDA-G002-07
PEARS							
Trial PAT Italy, 2002 (Decana)	160 g/kg HN	Thermofogging	7.2	1	1	1.6	ISPaVe-PRM-IML-T-15/02
					15	1.0	
					28	1.4	
					50	1.3	
					72	1.1	
Trial PBT Italy, 2002 (Kaiser)	160 g/kg HN	Thermofogging	7.2	1	1	1.3	ISPaVe-PRM-IML-T-15/02
					15	1.2	
					28	1.2	
					50	1.8	
					72	1.4	
Trial PCT Italy, 2002 (Abate Fetel)	160 g/kg HN	Thermofogging	7.2	1	1	3.5	ISPaVe-PRM-IML-T-15/02
					15	1.3	
					28	1.2	
					50	1.6	
					72	1.1	
Trial PDT Italy, 2002 (Conference)	160 g/kg HN	Thermofogging	7.2	1	1	0.96	ISPaVe-PRM-IML-T-15/02
					15	0.92	
					28	0.92	
					50	1.0	
					72	0.99	

Stone Fruits

Supervised field trials on stone fruits (apricots, peaches, and plums) conducted in the USA have been provided and are summarized below.

Table 42. Residues of pyrimethanil on apricots from supervised trials (pre-harvest) in the USA (B003046)

APRICOT	Application					PHI	Residues, mg/kg
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
GAP USA (stone fruit except cherries)	600 g/L SC	0.8			no more than 2.4 kg ai/season	2	
Trial R05-01 Michigan. USA, 1999 (Stanley)	400 g/L SC	0.8	941	0.09	3	2	1.2
Trial R10-01 California, USA, 2000 (French)	400 g/L SC	0.8	939	0.09	3	0	0.96
						2	0.61
						7	0.52
						14	0.48
						21	0.34
Trial R10-03 California, USA, 2000 (Blenheim)	400 g/L SC	0.8	923	0.09	3	2	1.3
Trial R10-04 California, USA, 1999 (Blenheim	400 g/L SC	0.8	938	0.09	3	2	0.64

APRICOT	Application					PHI	Residues, mg/kg
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
Trial R11-01 Washington, USA, 1999 (Tilton)	400 g/L SC	0.8	938	0.08	3	2	1.7

Table 43. Residues of pyrimethanil on peaches from supervised trials (pre-harvest) in the USA (B003037)

PEACH	Application					PHI	Residues, mg/kg
country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
GAP USA (stone fruit except cherries)	600 g/L SC	0.8			no more than 2.4 kg ai/season	2	
Trial R01-01 Pennsylvania, USA, 1999 (Red Haven)	400 g/L SC	0.8	951	0.08	3	2	1.5
Trial R02-01 Virginia, USA, 1999 (Dixie Red)	400 g/L SC	0.8	942	0.08	3	2	1.6
Trial R02-02 Georgia, USA, 1999 (Red Skin)	400 g/L SC	0.8	951	0.08	3	2	0.94
Trial R02-03 South Carolina, USA, 1999 (Contender)	400 g/L SC	0.8	948	0.08	3	2	1.2
Trial R02-04 North Carolina, USA, 1999 (Elberta)	400 g/L SC	0.8	944	0.08	3	2	1.5
Trial R04-01 Arkansas, USA, 1999 (Elberta)	400 g/L SC	0.8	929	0.09	3	2	1.3
Trial R05-01 Michigan, USA, 1999 (Red Haven)	400 g/L SC	0.8	942	0.08	3	2	0.54
Trial R06-01 Oklahoma USA, 1999 (Giohaven)	400 g/L SC	0.8	938	0.09	3	2	1.3
Trial R10-01 California, USA, 1999 (Dr. Davis)	400 g/L SC	0.8	935	0.08	3	0	1.83
						2	2.6
						7	1.1
						14	0.97
						21	0.95
Trial R10-02 California, USA, 1999 (September Sun)	400 g/L SC	0.8	920	0.09	3	2	1.1
Trial R10-03 California, USA, 1999 (Flavorcrest)	400 g/L SC	0.8	932	0.09	3	2	0.38

PEACH	Application					PHI	Residues, mg/kg
country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
Trial R10-04 California USA, 1999 (Andross)	400 g/L SC	0.8	957	0.08	3	2	0.99

Table 44. Residues of pyrimethanil on plums from supervised trials (pre-harvest) in the USA (B003707)

PLUM	Application					PHI	Residues, mg/kg
country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
GAP USA (stone fruit except cherries)	600 g/L SC	0.8			no more than 2.4 kg ai/season	2	
Trial R05-01 Michigan. USA, 1999 (Stanley)	400 g/L SC	0.8	941	0.09	3	2	1.2
Trial R10-01 California, USA, 2000 (French)	400 g/L SC	0.8	939	0.09	3	0	0.96
						2	0.61
						7	0.52
						14	0.48
						21	0.34
Trial R10-02 California, USA, 2000 (French)	400 g/L SC	0.8	930	0.09	3	2	0.05
Trial R10-03 California, USA, 2000	400 g/L SC	0.8	936	0.09	3	2	0.62
Trial R10-04 California, USA, 2000 (Blue Sugar)	400 g/L SC	0.8	931	0.09	3	2	0.58
Trial R10-05 California, USA, 2000 (Blue Sugar)	400 g/L SC	0.8	934	0.09	3	2	0.59
Trial R11-01 Washington, USA, 2000 (Autumn Sweet)	400 g/L SC	0.8	979	0.08	3	2	0.44
Trial R12-01 Oregon, USA, 2000 (Italians)	400 g/L SC	0.8	955	0.08	3	2	0.59

Three post-harvest trials on peaches and three on plums were conducted in the US following a *proposed* GAP for post-harvest use of pyrimethanil on stone fruits (except cherries).

Table 45. Pyrimethanil residues in stone fruit from post-harvest treatments

STONE FRUIT	Post-harvest Application				Residues	Reference
Country, year (variety)	Formulation	Treatment Method	kg ai/hL	no.	mg/kg	
Proposed GAP, USA Stone fruit	400 g/L SC	Wax or dip	0.05 - 0.10	Any combination of 2 methods		
		high volume spray	0.08- 0.12			
		low volume spraying	0.4- 1.2			
PEACHES						
Trial CA170 California, USA, 2005	400 g/L SC	Wax + dip	0.106	1	3.5	PR08700.04 CAR17
		Wax + high volume Spray	0.133 + 0.088	1	1.0	
		Wax + low volume Spray	1.33 + 0.40	1	2.5	
Trial CA142 California, USA, 2004	400 g/L SC	Wax + dip	0.106	1	5.7	PR08700.04 CAR17
		Wax + high volume Spray	0.133 + 0.088	1	2.0	
		Wax + low volume Spray	1.33 + 0.40	1	2.9	
Trial CANJ32 New Jersey USA, 2004	400 g/L SC	Wax + dip	0.106	1	7.4	PR08700.04 CAR17
PLUMS						
Trial CA144 California USA, 2004	400 g/L SC	Wax + dip	0.106	1	0.64	PR08702.04-CAR16
		Wax + high volume Spray	0.133 + 0.088	1	0.20	
		Wax + low volume Spray	1.33 + 0.40	1	0.32	
Trial WA18 Washington USA, 2004	400 g/L SC	Wax + dip	0.106	1	0.91	PR08702.04-CAR16
Trial MI26 Michigan USA, 2004	400 g/L SC	Wax + dip	0.106	1	1.9	PR08702.04-CAR16

Four post-harvest trials were conducted in Germany using *cherries* purchased at the local market (AGR 3393, Balluff, 2006; Report 20064083/G1-FPH). The cherries were produced in Chile or Argentina. Residues of pyrimethanil were determined in cherries using a modified version of a published multiresidue method. The referenced method was not supplied. However, adequate detail was available in the analytical method report section of AGR 3393. Samples were extracted with acetonitrile and an aliquot of the extract was dried over anhydrous sodium sulfate. Clean-up was done by solid-phase extraction on activated carbon (ENVI-Carb) and primary amino phase. Pyrimethanil was eluted with acetonitrile/toluene (3:1, v/v). The solvent was evaporated, the residue taken up in acetonitrile/water (1:1, v/v) and analysed by HPLC with MS/MS detection. Concurrent recoveries for fortified control cherries were 94% ± 4% RSD at 0.01 mg/kg, 92 ± 4% RSD at 0.1 mg/kg, and 92 ± 6% RSD at 2 mg/kg, n = 5 for each concentration.

Table 46. Pyrimethanil residues in cherries from post-harvest treatments (AGR 3393)

CHERRY	Post-harvest Application				Sampling	Residues
country, year (variety)	Formulation.	Treatment Method	kg ai/hL	no.	days	mg/kg
GAP, Chile	400 g/L SC	Dipping	0.04	1		
Trial G06W228R Germany, 2006 (Bing/Chile)	400 g/L SC	Dipping –T1	0.03	1	0	0.68
		Dipping –T1	0.03	1	0	0.61
		Dipping –T1	0.03	1	0	0.64

CHERRY	Post-harvest Application				Sampling	Residues
country, year (variety)	Formulation.	Treatment Method	kg ai/hL	no.	days	mg/kg
		Dipping –T1	0.03	1	0	0.82
		Dipping- T2	0.04	1	0	1.1
		Dipping- T2	0.04	1	0	1.2
		Dipping- T2	0.04	1	0	1.3
		Dipping- T2	0.04	1	0	1.0
		Dipping- T2-CS	0.04	1	3	1.0
		Dipping- T2-AS	0.04	1	3	1.5
		Dipping- T2-CS	0.04	1	7	1.3
		Dipping- T2-AS	0.04	1	7	1.3
		Dipping- T2-AS	0.04	1	14	0.91
		Dipping- T2-CS	0.04	1	28	0.90
		Dipping- T2-CAS	0.04	1	31	0.78
		Dipping- T2-CS	0.04	1	35	0.82
		Dipping- T2-CAS	0.04	1	35	0.54
		Dipping- T2-CS	0.04	1	42	0.74
		Dipping- T2-CAS	0.04	1	42	0.35
Trial G06W229R	400 g/L SC	Dipping –T1	0.03	1	0	0.91
Germany, 2006		Dipping –T1	0.03	1	0	1.0
(Bing/Argentina)		Dipping –T1	0.03	1	0	1.2
		Dipping –T1	0.03	1	0	1.0
		Dipping- T2	0.04	1	0	1.2
		Dipping- T2	0.04	1	0	1.3
		Dipping- T2	0.04	1	0	1.4
		Dipping- T2	0.04	1	0	1.0
		Dipping- T2-CS	0.04	1	3	1.3
		Dipping- T2-AS	0.04	1	3	1.1
		Dipping- T2-CS	0.04	1	7	1.2
		Dipping- T2-AS	0.04	1	7	1.0
		Dipping- T2-AS	0.04	1	14	0.43
		Dipping- T2-CS	0.04	1	28	0.92
		Dipping- T2-CAS	0.04	1	31	0.50
		Dipping- T2-CS	0.04	1	35	0.83
		Dipping- T2-CAS	0.04	1	35	0.30
		Dipping- T2-CS	0.04	1	42	0.71
		Dipping- T2-CAS	0.04	1	42	0.16
Trial G06W230R	400 g/L SC	Dipping –T1	0.03	1	0	1.1
Germany, 2006		Dipping –T1	0.03	1	0	1.1
(Bing/Argentina)		Dipping –T1	0.03	1	0	1.2
		Dipping –T1	0.03	1	0	1.2
		Dipping- T2	0.04	1	0	1.0
		Dipping- T2	0.04	1	0	1.2
		Dipping- T2	0.04	1	0	1.3
		Dipping- T2	0.04	1	0	1.4
		Dipping- T2-CS	0.04	1	3	1.2
		Dipping- T2-AS	0.04	1	3	1.3
		Dipping- T2-CS	0.04	1	7	1.4
		Dipping- T2-AS	0.04	1	7	0.93
		Dipping- T2-AS	0.04	1	14	0.52
		Dipping- T2-CS	0.04	1	28	0.88
		Dipping- T2-CAS	0.04	1	31	0.61
		Dipping- T2-CS	0.04	1	35	1.1
		Dipping- T2-CAS	0.04	1	35	0.62
		Dipping- T2-CS	0.04	1	42	0.91
		Dipping- T2-CAS	0.04	1	42	0.33
Trial G06W231R	400 g/L SC	Dipping –T1	0.03	1	0	0.96
Germany, 2006		Dipping –T1	0.03	1	0	1.1
(Bing/Chile)		Dipping –T1	0.03	1	0	1.0
		Dipping –T1	0.03	1	0	1.2
		Dipping- T2	0.04	1	0	0.93
		Dipping- T2	0.04	1	0	1.1
		Dipping- T2	0.04	1	0	1.2
		Dipping- T2	0.04	1	0	1.4

CHERRY	Post-harvest Application				Sampling	Residues
country, year (variety)	Formulation.	Treatment Method	kg ai/hL	no.	days	mg/kg
		Dipping- T2-CS	0.04	1	3	1.3
		Dipping- T2-AS	0.04	1	3	1.4
		Dipping- T2-CS	0.04	1	7	1.4
		Dipping- T2-AS	0.04	1	7	0.97
		Dipping- T2-AS	0.04	1	14	0.85
		Dipping- T2-CS	0.04	1	28	1.2
		Dipping- T2-CAS	0.04	1	31	0.67
		Dipping- T2-CS	0.04	1	35	0.87
		Dipping- T2-CAS	0.04	1	35	0.42
		Dipping- T2-CS	0.04	1	42	0.98
		Dipping- T2-CAS	0.04	1	42	0.35

T- treatment

CS – cold storage

AS – ambient storage

CAS – cold/ambient storage

Berries and Other Small Fruits

Table 47. Residues of pyrimethanil on grapes from supervised trials in the USA (B002944)

GRAPES Country, year (variety)	Application					PHI days	Residues, mg/kg
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.		
GAP USA	600 g/L SC	0.8			no more than 1.6 kg ai/season	7	
Trial R01-01 New York, USA, 1999 Vidal Blanc 256	400 g/L SC	0.8	945	0.08	2	7	2.0
Trial R01-02 New York, USA, 1999 (Vidal Blanc Ravat)	400 g/L SC	0.8	940	0.09	2	7	1.5
Trial R10-01 California USA, 1999 (Pinot Blanc)	400 g/L SC	0.8	950	0.08	2	1	0.51
						7	0.49
						14	0.40
						21	0.29
						28	0.20
Trial R10-02 California, USA, 1999 (Thompson Seedless)	400 g/L SC	0.8	936	0.09	2	7	0.44
Trial R10-03 California, USA, 1999 (Flame seedless)	400 g/L SC	0.8	926	0.09	2	7	0.64
Trial R10-04 California USA, 1999 (Cabernet Sauvignon)	400 g/L SC	0.8	1127	0.07	2	7	1.6
Trial R10-05 California, USA, 1999 (Thompson seedless)	400 g/L SC	0.8	940	0.09	2	7	2.5
Trial R10-06 California, USA, 1999 (Thompson Seedless)	400 g/L SC	0.8	926	0.09	2	7	0.66

GRAPES Country, year (variety)	Application					PHI days	Residues, mg/kg
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.		
Trial R10-07 California, USA, 1999 (Cabernet Sauvignon)	400 g/L SC	0.8	950	0.08	2	7	1.2
Trial R10-08 California USA, 1999 (Sauvignon Blanc)	400 g/L SC	0.8	945	0.08	2	7	0.12
Trial R11-01 Washington, USA, 1999 (Riesling)	400 g/L SC	0.8	940	0.09	2	7	0.89
Trial R11-02 Washington, USA, 1999 (Meriot)	400 g/L SC	0.8	940	0.09	2	7	0.71

Table 48. Residues of pyrimethanil on grapes from supervised trials in the EU

GRAPES Country, year (variety)	Application					PHI days	Residues, mg/kg	Reference
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.			
GAP, France	*400 g/L SC*	*1*			*1*	*21*		
Trial 01R687-1 Germany, 2001 (Riesling)	400 g/L SC	1.0	500	0.2	1	0	1.6	C017382
						7	1.3	
						14	0.75	
						21	0.97	
Trial 01R687-2 Germany, 2001 (Silvaner)	400 g/L SC	1.0	500	0.2	1	0	2.0	C017382
						7	1.0	
						14	0.98	
						21	0.59	
Trial 01R687-3 France, 2001 (Pinot Meunier)	400 g/L SC	1.0	1000	0.1	1	0	1.0	C017382
						7	0.51	
						14	0.44	
						21	0.44	
Trial 01R687-4 France, 2001 (Pinot Noir)	400 g/L SC	1.0	250	0.4	1	0	1.7	C017382
						7	1.7	
						14	1.4	
						21	1.1	
Trial 01R687-5 France, 2001 (Cabernet Franc)	400 g/L SC	1.0	250	0.4	1	0	1.1	C017382
						7	0.71	
						14	0.49	
						21	0.37	
GAP, Spain	***400 g/L SC***		**-**	***0.08***	***1***	***21***		
Trial 01R688-1 Spain, 2001 (Moscatel)	400 g/L SC	1.0	1000	0.1	1	0	0.72	C017384
						7	0.41	
						14	0.32	
						21	0.28	
Trial 01R688-2 Spain, 2001 (Macabeo)	400 g/L SC	1.0	750	0.14	1	0	1.4	C017384
						7	0.91	
						14	0.86	
						20	0.41	
Trial 01R688-3 France, 2001 (Marlot)	400 g/L SC	1.0	300	0.37	1	0	1.6	C017384
						7	0.96	
						14	0.53	
						21	0.58	

GRAPES Country, year (variety)	Application					PHI days	Residues, mg/kg	Reference
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.			
Trial 01R688-4 Italy, 2001 (Trebbiano)	400 g/L SC	1.0	1000	0.1	1	0	0.91	C017384
						7	0.52	
						14	0.45	
						20	0.48	
Trial 01R688-5 Italy, 2001 (Lambrusco Di Sorbara)	400 g/L SC	1.0	1000	0.1	1	0	2.5	C017384
						7	1.8	
						14	1.4	
						21	1.5	
Trial in Moves, France, 1993 (Cardinal)	400 g/L SC	1.0	100	1.0	1	28	0.36	A81740
Trial in Moves, France, 1993 (Muscat de Hambourg)	400 g/L SC	1.0	100	1.0	1	21	1.0	A81740
Trial in Moves, France, 1993 (Italy)	400 g/L SC	1.0	100	1.0	1	16	0.42	A81740
Trial in Molssac, France, 1993 (Chasselas)	400 g/L SC	1.0	300	0.3	1	89	0.12	A81740
						118	0.06	
Trial in Carpentras, France, 1993 (Alphonse Lavallie)	400 g/L SC	1.0	100	1.0	1	39	0.27	A81740

Eight supervised trials were conducted on strawberries in the USA (3 in California and one each in Pennsylvania, Wisconsin, Oregon, New Jersey, and Florida) (B003038, Brady, 2000; Report AN99R009). Period from sampling to analysis was 393 days. Storage stability study on grapes and tomatoes showed that pyrimethanil is stable for that time period when stored frozen.

Table 49. Residues of pyrimethanil on strawberries from supervised trials in the USA (B003038)

STRAWBERRY country, year (variety)	Application					PHI days	Residues, mg/kg
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.		
GAP USA	600 g/L SC	0.8			no more than 2.4 kg ai/season	1	
Trial R01-01 Pennsylvania, USA, 1999 (Earliglow)	400 g/L SC	0.8	929	0.09	3	1	0.79
						2	0.48
						3	0.36
Trial R02-01 Maryland, USA, 1999 (Alister)	400 g/L SC	0.8	926	0.09	3	1	0.93
						2	0.67
						3	0.58
Trial R03-01 Florida, USA, 1999 (Sweet Charlie)	400 g/L SC	0.8	957	0.09	3	1	0.99

STRAWBERRY country, year (variety)	Application					PHI days	Residues, mg/kg
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.		
Trial R05-01 Wisconsin, USA, 1999 (Raritan)	400 g/L SC	0.8	932	0.09	3	1	2.3
						2	1.19
						3	0.99
Trial R10-01 California, USA USA, 1999 (Lido)	400 g/L SC	0.8	929	0.09	3	0	1.3
						1	1.3
						2	1.1
						3	0.87
						7	0.47
						14	0.35
						21	0.19
Trial R10-02 California, USA, 1999 (Selva)	400 g/L SC	0.8	936	0.09	3	1	1.1
						2	1.11
						3	0.8
Trial R10-03 California, USA, 1999 (Coronado)	400 g/L SC	0.8	932	0.09	3	1	1.0
						2	1.3
						3	0.93
Trial R12-01 Oregon, USA 1999 (Totem)	400 g/L SC	0.8	932	0.09		1	1.1
						2	1.2
						3	1.1

Assorted tropical and sub-tropical fruits - inedible peel

Thirteen field trials were conducted in the following Central and South American countries where bananas are commercially grown: Costa Rica (3); Honduras (2); Colombia (3); Guatemala (2); and Ecuador (3) (C002386, Mickelson, 1999; Report AA980902). The Honduran crops were destroyed by severe weather prior to harvest; therefore, these two trials were discontinued, leaving 11 trials, all conforming to the common GAP in Latin America for bananas. Each test site consisted of two plots, one treated and one control. In each treated plot, a suspension concentrate formulation containing 600 g/L pyrimethanil was applied eight times to bananas plants using commercial aerial application equipment. Each application was made at a nominal rate of 300 g ai/ha. Treatment was made at 8 day (± 1 day) intervals before normal commercial harvest. Application volumes ranged from 17.9 L/ha to 37.5 L/ha.

Fresh green export-quality bagged bananas were collected from the untreated control plot on the day of the last application. *Bagged and unbagged banana samples* from the treated plot were collected immediately after the spray dried on the last application (day 0) and again at 1, 3, 5 and 7 days for the decline trials. Samples were harvested at each collection date from the different horizons of the raceme (top, middle and bottom) and from inner and outer whorls of the hands. Damaged and diseased fruit were avoided. Each sample consisted of about 24 fingers of mature green bananas.

Immediately after collection, the banana fingers were placed on plastic sheets until latex flow had subsided. Samples were bagged, placed in residue bags, labelled and shipped by air courier to the laboratory under ambient temperature conditions. Upon receipt at the laboratory, samples for analysis as whole fruit only were placed in deep freeze storage (< -18 ºC). Samples for determination of residues in whole fruit, pulp, and peel were prepared before freezing. The longest period samples were kept frozen before analysis was about 5 months (from August 1988 to January 1999).

Residues of pyrimethanil were determined in whole fruit, peel, and pulp samples from both bagged and unbagged bananas, using a GC/MS method developed for fruits and vegetables (C000292), with an LOQ of 0.05 mg/kg.

Table 50. Residues of pyrimethanil on bananas from supervised trials in Latin American Countries

BANANA	Application					PHI	Residues, mg/kg			
Country, year (variety)	Form	kg ai/ha	Water L/ha	kg ai/hL	no.	days		Whole	Peel	Pulp
GAP, Latin America [a]	600 g/L SC	0.3			6	0 (cont. harvest)				
Trial CR1 Costa Rica, 1998 (Gran Enano)	600 g/L SC	0.370		1.43	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
						1	Bagged	< 0.05	< 0.05	< 0.05
						1	Unbagged	< 0.05	< 0.05	< 0.05
						3	Bagged	< 0.05	< 0.05	< 0.05
						3	Unbagged	< 0.05	< 0.05	< 0.05
						5	Bagged	< 0.05	< 0.05	< 0.05
						5	Unbagged	< 0.05	< 0.05	< 0.05
						7	Bagged	< 0.05	< 0.05	< 0.05
						7	Unbagged	< 0.05	< 0.05	< 0.05
Trial CR2 Costa Rica, 1998 (Valery)	600 g/L SC	0.301		1.43	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
Trial CR3 Costa Rica, 1998 (Gran Naine)	600 g/L SC	0.292		1.43	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
Trial EC2 Ecuador, 1998 (Giant)	600 g/L SC	0.340		1.31	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
Trial EC3 Ecuador, 1998 (Giant)	600 g/L SC	0.340		1.31	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
Trial EC1 Ecuador, 1998 (Gran Enano)	600 g/L SC	0.305		1.31	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
						1	Bagged	< 0.05	< 0.05	< 0.05
						1	Unbagged	< 0.05	< 0.05	< 0.05
						3	Bagged	< 0.05	< 0.05	< 0.05
						3	Unbagged	0.09	< 0.05	< 0.05
						5	Bagged	< 0.05	< 0.05	< 0.05
						5	Unbagged	< 0.05	< 0.05	< 0.05
						7	Bagged	< 0.05	< 0.05	< 0.05
						7	Unbagged	< 0.05	< 0.05	< 0.05
Trial CO1 Colombia, 1998 (Grand Naine)	600 g/L SC	0.315		1.43	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
Trial CO2 Colombia, 1998 (Valery)	600 g/L SC	0.308		1.43	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	0.06	< 0.05
Trial CO3 Colombia, 1998 (Grand Naine)	600 g/L SC	0.315		1.43	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
Trial GU1 Guatemala, 1998 (Grand Naine)	600 g/L SC	0.306		1.41	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05
Trial GU2 Guatemala, 1998 (Grand Naine)	600 g/L SC	0.304		1.41	8	0	Bagged	< 0.05	< 0.05	< 0.05
						0	Unbagged	< 0.05	< 0.05	< 0.05

a - Ecuador, Colombia, Guatemala, Dominican Republic, Honduras, Costa Rica.

Bulb vegetables

Nine trials were conducted on onions, three in California, two in Texas and one each in New York, Michigan, Colorado and Oregon (B003159, Dacus, 2001; Report AN99R001).

Table 51. Residues of pyrimethanil on onions from supervised trials in the USA (B003159)

ONION	Application					PHI	Residues, mg/kg
country, year (variety)	Form	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
GAP USA	600 g/L SC	0.8			No more than 2.4 kg ai/season	7	
Trial R01-01 New York, USA, 1999 (Crusader)	400 g/L SC	0.8	186	0.4	3	7	0.095
Trial R06-01 Texas, USA, 2000 (101 5Y)	400 g/L SC	0.8	187	0.4	3	7	< 0.05
Trial R08-01 Colorado, USA, 1999 (Teton)	400 g/L SC	0.8	187	0.4	3	7	0.087
Trial R10-01 California, USA, 1999m (Italian Sweet, early burger)	400 g/L SC	0.8	187	0.4	3	7	< 0.05
Trial R10-02 California, USA, 2000 (K99 Bulb onion)	400 g/L SC	0.8	187	0.4	3	0	0.168
						7	0.075
						10	< 0.05
						12	< 0.05
						14	< 0.05
Trial R11-01 Oregon, USA, 1999 (Vision)	400 g/L SC	0.8	182	0.4	3	7	< 0.05
Trial R05-01 Michigan, USA, 1999 (Ishikura improved)	400 g/L SC	0.8	187	0.4	3	7	0.26
Trial R06-02 Texas, USA, 1999 (Texas Early White)	400 g/L SC	0.8	188	0.4	3	7	0.38
Trial R10-03 California, USA, 2000 (Evergreen bunching)	400 g/L SC	0.6	182	0.4	3	6	1.6

Fruiting vegetables, other than Cucurbits

A total of 16 supervised trials were conducted on tomatoes in major tomato growing regions of the US (B003040, Brady, 2000; Report AN99R010).

Table 52. Residues of pyrimethanil on tomatoes from supervised field trials in the USA (B003040)

TOMATO	Application					PHI	Residues, mg/kg
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
GAP USA	600 g/L SC	0.3			No more than 1.6 kg ai/ha per season	1	
Trial R01-01 Pennsylvania, USA, 1999 (Mountain Spring)	400 g/L SC	0.3	187	0.16	5	1	0.07
Trial R02-01 North Carolina, USA, 1999 (Celebrity)	400 g/L SC	0.3	172	0.17	5	0	0.12
						1	0.10
						7	< 0.05
						14	< 0.05
						21	< 0.05
Trial R03-01 Florida, USA, 1999 (Heat Wave)	400 g/L SC	0.3	182	0.16	5	0	0.15
						1	0.13
						7	0.07
						14	< 0.05
						21	< 0.05
Trial R03-02 Florida, USA, 1999 (Celebrity)	400 g/L SC	0.3	191	0.16	5	1	0.07
Trial R05-01 Ohio, USA,1999 (Heinz H94423)	400 g/L SC	0.3	194	0.15	5	1	0.20
Trial R10-01 California, USA, 1999 (Heinz 8892)	400 g/L SC	0.3	187	0.16	5	1	0.16
Trial R10-02 California, USA 1999 (Roma)	400 g/L SC	0.3	187	0.16	5	1	0.14
Trial R10-03 California, USA, 1999 (Rio Grande)	400 g/L SC	0.3	187	0.16	5	1	0.15
Trial R10-04 California, USA, 1999 (UC 82-L)	400 g/L SC	0.3	191	0.16	5	1	0.35
Trial R10-05 California, USA 1999 (Heinz)	400 g/L SC	0.3	187	0.16	5	1	0.07
Trial R10-06 California, USA, 1999 (Rio Grande)	400 g/L SC	0.3	187	0.16	5	1	0.22
Trial R10-07 California, USA, 1999 (Cannery Row)	400 g/L SC	0.3	187	0.16	5	1	0.37
Trial R10-08 California, USA 1999 (CXD 181)	400 g/L SC	0.3	187	0.16	5	1	0.17
Trial R10-09 California, USA 1999 (APT-539)	400 g/L SC	0.3	187	0.16	5	1	0.14

TOMATO Country, year (variety)	Application					PHI days	Residues, mg/kg
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.		
Trial R10-10 California, USA 1999 (8892)	400 g/L SC	0.3	191	0.16	5	1	0.23
Trial R10-11 California, USA, 1999 (Shady Lady)	400 g/L SC	0.3	191	0.16	5	1	0.06

Four glasshouse trials on tomatoes were conducted in Northern France and the Netherlands in 1995 (A89716, Hees, *et. al.*, 1997; Report ER 95 ECN 261). Four additional glasshouse trials on tomatoes were conducted in the Netherlands in 1996 (A81019, Hees and Peatman, 1997; Report ER 96 ECN 261).

Table 53. Residues of pyrimethanil on tomatoes from glasshouse trials in the EU

TOMATO Country, year (variety)	Application					PHI days	Residues, mg/kg	Reference
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.			
GAP, France	400 g/L SC	0.8			2	3		
FRA 00 01 France, 1995 (Recento)	400 g/L SC	0.7	1500	0.05	2	0	0.27	A89716
						1	0.38	
						3	0.36	
						7	0.22	
FRA 00 02 France, 1995 (Recento)	400 g/L SC	0.7	1500	0.05	2	0	0.35	A89716
						1	0.45	
						3	0.26	
						7	0.15	
NLD 00 01 Netherlands, 1995 (Solaizo)	400 g/L SC	0.7	1500	0.05	2	0	0.29	A89716
						1	0.38	
						3	0.33	
						7	0.06	
NLD 00 02, Netherlands, 1995 (Solaizo)	400 g/L SC	0.7	1500	0.05	2	0	0.24	A89716
						1	0.36	
						3	0.31	
						7	0.07	
NLD 00 01 Netherlands, 1996 (Aramato)	400 g/L SC	0.8	1500	0.05	2	0	0.27	A81019
						1	0.38	
						3	0.36	
						7	0.22	
NLD 00 02 Netherlands, 1996 (Aramato)	400 g/L SC	0.8	1500	0.05	2	0	0.35	A81019
						1	0.45	
						3	0.26	
						7	0.15	
NLD 00 03 Netherlands, 1996 (Aramato)	400 g/L SC	0.8	1500	0.05	2	0	0.29	A81019
						1	0.38	
						3	0.33	
						7	0.06	

TOMATO Country, year (variety)	Application					PHI days	Residues, mg/kg	Reference
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.			
NLD 00 04 Netherlands, 1996 (Aramato)	400 g/L SC	0.8	1500	0.05	2	0	0.24	A81019
						1	0.36	
						3	0.31	
						7	0.07	

Leafy vegetables

Several supervised trials were conducted on lettuce in the EU. Fourteen field trials were on head lettuce varieties and four on leaf lettuce. In addition, nine glasshouse trials on lettuce were conducted. All trials used the SC formulation containing 400 g/L pyrimethanil. The trials are summarized below.

Table 54. Residues of pyrimethanil on lettuce (head) from supervised field trials in the EU

LETTUCE, HEAD Country, year (variety)	Application					PHI days	Residues, mg/kg	Reference
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.			
GAP, France	***400 g/L SC***	*0.8*	-	-	*2*	*21*		
Trial 01 England, 1997 (Saladin)	400 g/L SC	0.8	400	0.2	2	0	13	A91280
						3	4.6	
						7	0.47	
						14	0.55	
						21	< 0.05	
Trial 02 Scotland, 1997 (Saladin)	400 g/L SC	0.8	400	0.2	2	0	4.1	A91280
						3	1.5	
						7	0.11	
						14	0.06	
						21	< 0.05	
Trial 03 Northern France, 1997 (Nadine)	400 g/L SC	0.8	400	0.2	2	0	9.3	A91280
						3	2.9	
						7	1.1	
						14	0.34	
						21	0.13	
Trial 04 Northern France, 1997 (Nadine)	400 g/L SC	0.8	400	0.2	2	0	26	A91280
						3	2.2	
						7	2.1	
						14	0.42	
						21	0.28	
Trial 05 Germany, 1997 (Enrica)	400 g/L SC	0.8	400	0.2	2	0	35	A91280
						3	4.5	
						7	1.5	
						14	0.30	
						21	< 0.05	
Trial 02 Scotland, 1998 (Brandon)	400 g/L SC	0.8	400	0.2	2	0	12	C003112
						7	0.80	
						14	< 0.05	
Trial. 03 Germany, 1998 (Nadine)	400 g/L SC	0.8	400	0.2	2	0	22	C003112
						7	1.3	
						14	0.11	
Trial 04 Netherlands, 1998 (Enrica)	400 g/L SC	0.8	400	0.2	2	0	18	C003112
						7	1.2	
						14	0.43	

LETTUCE, HEAD	Application					PHI	Residues, mg/kg	Reference
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days		
Trial 05 England, 1998 (Lincoln)	400 g/L SC	0.8	400	0.2	2	0	2.4	C003112
						7	0.15	
						14	< 0.05	
GAP, Italy	*400 g/L SC*	*0.8*	-	-	*2*	*14*		
Trial 01 Southern France 1997 (Rougette de Montpellier)	400 g/L SC	0.8	400	0.2	2	0	22	A91282
						3	7.8	
						7	4.5	
						14	0.78	
						21	0.77	
Trial 02 Italy, 1997 (Justin)	400 g/L SC	0.8	400	0.2	2	0	13	A91282
						3	1.2	
						7	0.4	
						14	0.31	
						21	< 0.05	
Trial 03 Italy, 1997 (Romana)	400 g/L SC	0.8	400	0.2	2	0	17	A91282
						3	4.6	
						7	0.32	
						14	0.14	
						21	0.11	
Trial 04 Spain, 1997 (Veraniega)	400 g/L SC	0.8	400	0.2	2	0	11	A91282
						3	1.7	
						7	0.45	
						14	0.05	
						21	< 0.05	
Trial 05 Greece, 1997 (Parris island cos)	400 g/L SC	0.8	400	0.2	2	0	4.6	A91282
						3	1.9	
						7	1.9	
						14	1.2	
						21	0.62	

Table 55. Residues of pyrimethanil on lettuce (leaf) from supervised field trials in EU (C003103)

LETTUCE, LEAF	Application					PHI	Residues, mg/kg
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
GAP, Italy	***400 g/L SC***	*0.8*	-	-	*2*	*14*	
Trial FRA 00 01 France, 1998 (Nevada)	400 g/L SC	0.8	250	0.3	2	0	14
						7	2.7
						14	0.62
Trial GRC 00 01 Greece, 1998 (Atraxion)	400 g/L SC	0.8	400	0.2	2	0	33
						7	17
						14	7.5
Trial ITA 00 01 Italy, 1998 (Winter haven)	400 g/L SC	0.8	500	0.2	2	0	20
						7	2.4
						14	0.68
Trial PRT 00 01 Portugal, 1998 (Vanity)	400 g/L SC	0.8	400	0.2	2	0	13
						7	1.7

Table 56. Residues of pyrimethanil from glasshouse trials on lettuce (head) in the EU

LETTUCE, HEAD	Application					PHI	Residues, mg/kg	Reference
Country, year (variety)	Form	kg ai/ha	Water L/ha	kg ai/hL	no.	days		
GAP, Italy	*400 g/L SC*	*0.8 low volume*	-	*0.08*	*2*	*14*		
Trial 01 Scotland, 1997 (Salchin)	400 g/L SC	0.8	1000	0.08	2	0	48	A91279
						3	9.6	
						7	6.9	
						14	1.4	
						21	0.3	
Trial 02 Scotland, 1997 (Impulse)	400 g/L SC	0.8	1000	0.08	2	0	38	A91279
						3	12	
						7	4.1	
						14	0.85	
						21	0.21	
Trial 03 Germany, 1997 (Titan)	400 g/L SC	0.8	1000	0.08	2	0	35	A91279
						3	6.2	
						7	5.0	
						14	0.97	
						21	0.22	
Trial 04 Netherlands, 1997 (Benjamin)	400 g/L SC	0.8	1000	0.08	2	0	26	A91279
						3	5.3	
						7	2.0	
						14	0.49	
						21	0.20	
Trial 04 Netherlands, 1997 (Benjamin)	400 g/L SC	0.8	1000	0.08	2	0	17	A91279
						3	7.1	
						7	2.4	
						14	0.97	
						21	0.30	
Trial 01 England, 1998 (Vegas)	400 g/L SC	0.8	1000	0.08	2	0	42	C003113
						7	2.4	
						14	0.61	
Trial 02 Scotland, 1998 (Impulse)	400 g/L SC	0.8	1000	0.08	2	0	40	C003113
						7	2.2	
						14	0.41	
Trial 03 Germany, 1998 (Flandria)	400 g/L SC	0.8	1000	0.08	2	0	27	C003113
						7	4.5	
						14	1.6	
Trial 04 Netherlands, 1998 (Benjamin)	400 g/L SC	0.8	1000	0.08	2	0	1.9	C003113
						7	0.59	
						14	0.37	

Legume vegetables

Table 57. Residues of pyrimethanil on common (green) beans from supervised trials in the EU

BEANS	Application					PHI	Residues, mg/kg	Reference
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days		
GAP, France	***400 g/L SC***	*0.6*	-	-	*1*	*14*		
Trial FRA 0001 France, 1995	400 g/L SC	0.6	300	0.2	1	7	0.1	A89193
						14	< 0.05	
						22	ND	
Trial FRA 0002 France, 1995	400 g/L SC	0.6	300	0.2	1	7	< 0.05	A89193
						14	< 0.05	
						22	ND	
Trial DEU 05 01 Germany, 1999 (Graffi)	400 g/L SC	0.8	300	0.3	2	0	1.10	C009094
						3	0.70	
						7	0.25	
						14	0.07	
Trial DEU 05 02 Germany, 1999 (Montana)	400 g/L SC	0.8	300	0.3	2	0	2.20	C009094
						3	0.68	
						7	0.24	
						14	0.05	
Trial DEU 06 01 Germany, 1999 (Odeon)	400 g/L SC	0.8	300	0.3	2	0	0.28	C009094
						3	0.67	
						7	0.37	
						14	0.09	
Trial FRA 01 01 France, 1999 (Booster)	400 g/L SC	0.8	250	0.3	2	0	0.63	C009094
						3	0.39	
						7	0.16	
						14	0.08	
Trial FRA 01 02 France, 1999 (Paloma)	400 g/L SC	0.8	250	0.3	2	0	0.64	C009094
						3	0.86	
						7	0.33	
						14	< 0.05	

Table 58. Residues of pyrimethanil from common beans (glasshouse) trials in the EU (A91284)

BEANS	Application					PHI	Residues, mg/kg
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
GAP, France	***400 g/L SC***	*0.6*	-	-	-	*14*	
Trial 1 France, 1997 (Radar)	400 g/L SC	0.6	400	0.15	2	0	1.23
						3	0.61
						7	0.32
						14	0.28
Trial 2 Italy, 1997 (Strike)	400 g/L SC	0.6	400	0.15	2	0	2.7
						3	1.9
						7	2.0
						14	1.9

BEANS	Application					PHI	Residues, mg/kg
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	
Trial 3 Spain, 1997 (Buenos Aires)	400 g/L SC	0.6	1000	0.06	2	0	1.3
						3	0.39
						7	0.10
						14	< 0.05
Trial 4 Greece, 1997 (Tibona)	400 g/L SC	0.6	400	0.15	2	0	1.9
						3	0.61
						7	0.51
						14	0.13
Trial 5 Southern France, 1997 (Primera)	400 g/L SC	0.6	400	0.15	2	0	1.94
						14	0.20
Trial 6 Spain, 1997 (Matabaja Calima)	400 g/L SC	0.6	1000	0.06	2	0	3.7
						14	0.91
Trial 7 Spain, 1997 (Nuria)	400 g/L SC	0.6	1000	0.06	2	0	1.2
						14	0.12
Trial 8 Greece, 1995 (Yellow beans)	400 g/L SC	0.6	400	0.15	2	0	1.9
						14	0.25

Root and tuber vegetables

Supervised trials on carrots and potatoes have been provided and are summarized below. The trials on carrots were conducted in Brazil and the EU while the trials on potatoes were conducted in the USA.

Table 59. Residues of pyrimethanil on carrots from field trials in the EU and Brazil

CARROT	Application					PHI	Residues, mg/kg	Reference
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days		
GAP, France	***400 g/L SC***	***0.8***	**-**	**-**	*2*	*21*		
Trial 1, UK, 1997 (Nairobi)	400 g/L SC	0.8	400	0.2	2	0	0.32	A 91281
						3	0.11	
						7	0.11	
						14	0.11	
						21	0.07	
Trial 2, UK, 1997 (Narman)	400 g/L SC	0.8	400	0.2	2	0	0.21	A 91281
						3	< 0.05	
						7	0.07	
						14	0.08	
						21	< 0.05	
Trial 3, N France, 1997 (ABK)	400 g/L SC	0.8	400	0.2	2	0	1.1	A 91281
						3	0.65	
						7	0.40	
						14	0.47	
						21	0.28	
Trial 4, Germany, 1997 (Hitop)	400 g/L SC	0.8	400	0.2	2	0	< 0.05	A 91281
						3	< 0.05	
						7	< 0.05	
						14	< 0.05	
						21	< 0.05	

CARROT Country, year (variety)	Application Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	PHI days	Residues, mg/kg	Reference
Trial 5, Netherlands (Doukeer)	400 g/L SC	0.8	400	0.2	2	0	0.44	A 91281
						3	0.42	
						7	0.34	
						14	0.34	
						21	0.36	
Trial DEU 05 01 Germany, 1998 (Montana)	400 g/L SC	0.8	300	0.3	2	21	0.07	C003106
Trial DEU 06 01 Germany, 1998 (Rote Reisen)	400 g/L SC	0.8	300	0.3	2	21	0.35	C003106
Trial GBR 00 01 United Kingdom, 1998 (Narman)	400 g/L SC	0.8	400	0.2	2	17	0.13	C003106
Trial GBR 00 02 United Kingdom, 1998 (Nairobi)	400 g/L SC	0.8	400	0.2	2	21	0.24	C003106
GAP, Italy	*400 g/L SC*	*0.8*			*2*	*7*		
ESP 01 01 Spain, 1999 (Mantessa)	400 g/L SC	0.8	300	0.3	2	0	0.09	C008560
						7	0.08	
						14	< 0.05	
						20	0.06	
ESP 02 01 Spain, 1999 (Nantesa 5 "coral")	400 g/L SC	0.8	300	0.3	2	0	< 0.05	C008560
						7	0.05	
						14	< 0.05	
						20	< 0.05	
Trial 7, S France, 1997 (Bolero)	400 g/L SC	0.8	400	0.2	2	0	0.37	A91281
						3	0.25	
						7	0.21	
						14	0.19	
						21	0.15	
Trial 6, S France, 1997 (Bolero)	400 g/L SC	0.8	400	0.2	2	0	0.31	A91281
						3	0.21	
						7	0.20	
						14	0.38	
						21	0.44	
GRC 01 01 Greece, 1999 (Bolero)	400 g/L SC	0.8	300	0.3	2	0	0.42	C008560
						7	0.14	
						13	0.13	
GRC 01 02 Greece, 1999 (Bolero)	400 g/L SC	0.8	300	0.3	2	0	0.10	C008560
						7	0.09	
						14	0.06	
						21	0.05	
ITA 01 01 Italy, 1999 (Bolero)	400 g/L SC	0.8	350	0.2	2	0	0.12	C008560
						7	0.54	
						14	0.32	
						21	0.22	
ITA 02 01 Italy, 1999 (Fedora 'F1"	400 g/L SC	0.8	500	0.2	2	0	< 0.05	C008560
						7	0.33	
						14	0.51	
						21	0.46	

CARROT Country, year (variety)	Application Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	PHI days	Residues, mg/kg	Reference
PRT 01 01 Portugal, 1999 (Nantes)	400 g/L SC	0.8	300	0.3	2	0	0.08	C008560
						7	< 0.05	
						14	0.05	
						21	< 0.05	
GAP, Brazil	*300 g/L SC*	*0.6*	*1000*	*0.06*	*As needed*	*14*		
Cosmopolis/SP Brazil, 1995 (Nantes)	300 g/L SC	1.0	700	0.14	4	0	0.23	A91806
						3	0.42	
						7	0.23	
						14	0.24	
	300 g/L SC	2.0	700	0.28	4	14	0.85	
Itobi/SP Brazil, 1996 (Nantes Royal)	300 g/L SC	1.0	700	0.14	4	0	0.17	A91807
						3	0.11	
						7	0.16	
						14	0.08	
	300 g/L SC	2.0	700	0.28	4	14	0.43	

A total of 16 supervised trials were conducted in major potato growing areas in the USA in 1999. (B003015, Brady, 2000; Report AN99R008).

Table 60. Residues of pyrimethanil on potatoes from supervised trials in the USA (B003015)

POTATO Country, year (variety)	Application Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	PHI days	Residues, mg/kg
GAP USA	***600 g/L SC***	***0.3***			*no more than 1.6 kg ai/season*	*7*	
Trial R01-01 New York, USA, 1999 (Green Mountain)	400 g/L SC	0.3	189	0.16	5	7	< 0.05
Trial R01-02 Pennsylvania, USA, 1999 (Dark Red Norland)	400 g/L SC	0.3	172	0.17	5	7	< 0.05
Trial R02-01 North Carolina USA, 1999 (Red Pontiac)	400 g/L SC	0.3	191	0.16	5	0	< 0.05
						7	< 0.05
						14	< 0.05
						21	< 0.05
						28	< 0.05
Trial R03-01 Florida, USA, 1999 (Red Pontiac)	400 g/L SC	0.3	176	0.17	5	7	< 0.05
Trial R05-01 Wisconsin, USA, 1999 (Russet Norkota)	400 g/L SC	0.3	178	0.17	5	7	< 0.05
Trial R05-02 Ohio, USA, 1999 (Landsglad)	400 g/L SC	0.3	196	0.15	5	7	< 0.05
Trial R05-03 Michigan, USA 1999 (Dark Red Norland)	400 g/L SC	0.3	187	0.16	5	7	< 0.05

POTATO Country, year (variety)	Application					PHI days	Residues, mg/kg
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.		
Trial R05-04 North Dakota USA, 1999 ((Dark Red Norland)	400 g/L SC	0.3	187	0.16	5	7	< 0.05
Trial R09-01 Colorado, USA, 1999 (Norkota)	400 g/L SC	0.3	187	0.16	5	7	< 0.05
Trial R10-01 California, USA,1999 (Red Chieftain)	400 g/L SC	0.3	187	0.16	5	7	< 0.05
Trial R11-01 Idaho, USA, 1999 (Russet Burbank)	400 g/L SC	0.3	185	0.16	5	7	< 0.05
Trial R11-02 Idaho, USA, 1999 (Shepody)	400 g/L SC	0.3	187	0.16	5	7	< 0.05
Trial R11-03 Idaho, USA, 1999 (Russet Burbank)	400 g/L SC	0.3	187	0.16	5	7	< 0.05
Trial R11-04 Idaho, USA, 1999 (Russet Burbank)	400 g/L SC	0.3	105	0.28	5	7	< 0.05
Trial R11-05 Washington USA, 1999 (Russet Burbank)	400 g/L SC	0.3	187	0.16	5	7	< 0.05
Trial R11-06 Washington USA, 1999 (New Leaf + R.B.)	400 g/L SC	0.3	187	0.16	5	7	< 0.05

Legume animal feeds

A total of six supervised field trials were carried out on *field peas* (fodder peas, protein peas, or combining peas) in Northern EU, 2 each in Germany, France, and UK during 2000 (C013330, Sonder, 2001; Report DR00EUN680). An additional 5 trials in Southern France, and one each in Germany and UK were conducted in 2001 (C017378, Sonder, 2002; Report 01R680. Two applications were made at the nominal rate of 0.6 kg ai/ha. Field peas (*Pisum sativum* var. *arvense* (L.) Poir) are cultivars grown for livestock feed only.

Table 61. Residues of pyrimethanil in field peas from supervised trials in the EU

FIELD PEAS Country, year (variety)	Application					PHI days	Residues		Reference
	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.		Portion analysed	mg/kg	
GAP, France (protein peas)	***400 g/L SC***	***0.6***			-	***28***			
Trial DEU0301 Germany, 2000 (Duell)	400 g/L SC	0.6	300	0.2	2	0	Pods	0.72	C013330
						7	Pods	0.11	
						14	Pods	0.07	
						21	Pods	< 0.05	
						28	Seeds, dry	0.09	
						35	Seeds, dry	0.11	

FIELD PEAS	Application					PHI	Residues		Reference
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	Portion analysed	mg/kg	
Trial DEU0601 Germany, 2000 (Eiffel)	400 g/L SC	0.6	300	0.2	2	0	Pods	4.7	C013330
						7	Pods	1.4	
						14	Pods	1.1	
						21	Pods	0.71	
						28	Seeds, dry	< 0.05	
						28	Straw	1.0	
						35	Seeds, dry	< 0.05	
						35	Straw	0.76	
Trial FRA0201 France 2000 (Baccarat)	400 g/L SC	0.6	250	0.2	2	0	Pods	1.1	C013330
						28	Seeds, dry	0.22	
						28	Straw	0.15	
						35	Seeds, dry	0.25	
						35	Straw	0.09	
Trial FRA0202 France 2000 (focus)	400 g/L SC	0.6	250	0.2	2	0	Pods	0.99	C013330
						28	Seeds, dry	0.22	
						28	Straw	0.24	
						35	Seeds, dry	0.21	
						35	Straw	ND	
Trial GBR0101 United Kingdom, 2000 (Maris)	400 g/L SC	0.6	200	0.3	2	0	Pods	1.2	C013330
						7	Pods	0.34	
						14	Pods	0.57	
						20	Pods	0.14	
						29	Seeds, dry	0.12	
						29	Straw	0.28	
Trial GBR0102 United Kingdom, 2000 (Croma)	400 g/L SC	0.6	200	0.3	2	7	Pods	0.18	C013330
						14	Pods	< 0.05	
						20	Pods	< 0.05	
						27	Seeds, dry	< 0.05	
						27	Straw	0.66	
						35	Seeds, dry	< 0.05	
						35	Straw	0.43	
Trial 01R680-1 Germany, 2001 (Metaxa)	400 g/L SC	0.6	300	0.2	2	0	Pods	0.54	C017378
						28	Seeds, dry	0.061	
						28	Straw	< 0.05	
Trial 01R680-2 France, 2001 (Aladin)	400 g/L SC	0.6	250	0.2	2	0	Pods	0.44	C017378
						18	Seeds, dry	0.28	
						18	Straw	3.4	
Trial 01R680-3 France, 2001 (Baccara)	400 g/L SC	0.6	250	0.2	2	0	Pods	1.0	C017378
						17	Seeds, dry	0.42	
						17	Straw	2.7	
Trial 01R680-4 France, 2001 (Athos)	400 g/L SC	0.6	250	0.2	2	0	Pods	1.0	C017378
						32	Seeds, dry	< 0.05	
						32	Straw	< 0.05	
Trial 01R680-5 France, 2001 (Baccara)	400 g/L SC	0.6	250	0.2	2	0	Pods	0.73	C017378
						28	Seeds, dry	< 0.05	
						28	Straw	< 0.05	
Trial 01R680-6 France, 2001 (Athos)	400 g/L SC	0.6	250	0.2	2	0	Pods	0.51	C017378
						27	Seeds, dry	0.30	
						27	Straw	0.64	

FIELD PEAS	Application					PHI	Residues		Reference
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	Portion analysed	mg/kg	
Trial 01R680-7 UK, 2001 (Salome)	400 g/L SC	0.6	250	0.2	2	0	Pods	1.3	C017378
						28	Seeds, dry	0.08	
						28	Straw	0.15	

Tree nuts

Supervised trials on almonds were conducted in six sites in almond growing areas of the US. Each site consisted of one untreated and one treated plot. The interval between applications was 7 days (B003608, Wyatt, 2002; Report 01AN27795).

Table 62. Residues of pyrimethanil on almond hulls and nutmeats from supervised trials in the USA (B003608)

ALMONDS	Application					PHI	Residues, mg/kg	
Country, year (variety)	Formulation	kg ai/ha	Water L/ha	kg ai/hL	no.	days	Almond hulls	Almond Nutmeat
GAP USA	600 g/L SC	0.8			No more than 2.4 kg ai/ha per season	30		
Trial 27795-1001 California, USA, 2001 (Nonpareil)	400 g/L SC	0.8	934	0.08	3	16 23 30 37 44	3.6 7.6 9.2 4.1 3.3	0.19 0.09 0.06 < 0.05 < 0.05
Trial 27795-1002 California, 2001, USA (Monterey, Carmel, Nonpareil)	400 g/L SC	0.8	935	0.08	3	29	2.6	0.10
Trial 27795-1003 California, USA, 2001 (Nonpareil)	400 g/L SC	0.8	935	0.08	3	30	1.9	< 0.05
Trial 27795-1004 California, USA, 2001 (Carmel)	400 g/L SC	0.8	945	0.08	3	30	2.4	< 0.05
Trial 27795-1005 California, USA, 2001 (Nonpareil)	400 g/L SC	0.8	935	0.08	3	30	2.7	< 0.05
Trial 27795-1006 California, USA, 2001 (Nonpareil)	400 g/L SC	0.8	942	0.08	3	30	3.6	< 0.05

FATE OF RESIDUES IN STORAGE AND IN PROCESSING

In storage

No information was provided on the fate of pyrimethanil residues under commercial storage conditions.

In processing

The Meeting received processing studies for oranges, apples, plums, grapes, tomato, beans and carrots.

Oranges grown in Florida USA were treated with a dual application of an in-line post-harvest spray with a 400 g/L SC formulation of pyrimethanil at a nominal rate of 1000 ppm (0.1 kg ai/hL) and 2000 PPM (0.2 kg ai/hL) in shipping wax or in an in-line post-harvest spray at a rate of 2000 ppm (0.2 kg ai/hL) and 4000 ppm (0.4 kg ai/hL). Juice was made using a commercial in-line juice extractor at the University of Florida. Cold pressed oil was extracted from the oil/water/peel frit remaining after juicing. Residues in the fruit and processed fractions were determined by GC/MS using method DGM C05/98-0. Recoveries of pyrimethanil from 0.05 and 5.0 mg/kg fortifications were acceptable for orange (83 – 103%), juice (57 – 88%), dried pulp (61 – 92%), and oil (65 – 98%). (AGR 358, Goodwine, 2002). Results are summarized in Table 63.

Table 63. Residues of pyrimethanil in orange processed commodities (AGR 358)

Treatment rate	Commodity	Residue (mg/kg)	Processing factor
1000 ppm water + 2000 ppm wax line spray	Orange (unwashed)	2.86 (2.70, 2.83, 3.06)	-
	Orange (washed)	0.94	0.33
	Juice	< 0.05 (0.0235, 0.0213, 0.0208)	< 0.017
	Dried pulp	1.47 (1.59, 1.48, 1.33)	0.51
	Citrus oil	65. (65.3, 64.9, 65.4)	22.7
2000 ppm water + 4000 ppm wax line spray	Orange (unwashed)	7.46 (7.84, 7.06, 7.49)	-
	Orange (washed)	1.82	0.24
	Juice	0.05 (0.0406, 0.0588, 0.0526)	0.007
	Dried pulp	2.93 (3.31, 2.94, 2.53)	0.39
	Citrus oil	131 (127, 134, 132)	17.6
Average	Orange (washed) Juice Dried pulp Citrus oil		0.28 0.01 0.45 20.

Apple processing studies were conducted in Germany and in the USA. Two processing studies were conducted on apples in Germany to estimate the levels of residues in processed fractions of apples (peel, pomace, juice and press cake). In the first study, 4 trials on apples involving 12 or 14 applications of the 400 g/L SC formulation of pyrimethanil were conducted in Germany (A81719, Straszewski, 1993; Report U/R 19/93; A81721, Wrede, 1994c – Amendment to A81719). The total application rates were 2.7 – 6.7 kg ai/ha. Samples from two trials (PHI 28 days) were processed in a laboratory into pomace (without peel), peel, juice and press cake. Juice, pomace and press-cake were heated treated (microwaved at 70 – 100 °C for 2 minutes). Residues of pyrimethanil in samples were extracted with ethyl acetate and after clean-up, were quantified by HLPC with UV detection. The LOQ was 0.05 mg/kg. Recoveries at fortification level of 0.05 mg/kg pyrimethanil were 91% for apples and 92% for press-cake. The recovery for juice was 98% at fortification level of 0.5 mg/kg.

In the second study, the two trials conducted in Germany used 14 applications of the 400 SC formulation of pyrimethanil at the rate of 6.3 kg ai/ha. Apples at 28 day PHI were processed into peel, puree, juice, and pressed cake. Puree and juice were microwaved for 2 minutes at temperature between 70 to 100 ºC (A81749, Wrede, 1995; Report R/V 7/94). Residues of pyrimethanil were determined by HPLC with UV detection. LOQ was 0.05 mg/kg. Recoveries of samples fortified with 0.05 mg/kg pyrimethanil were 93% for apples, 83% for puree, 116% for peel, 115% for pressed cake and 101% for juice.

A single trial was conducted in Washington state, USA, where the SC formulation of pyrimethanil was applied four times at the rate of 2.25 kg ai/ha with 7 days between applications (B002853, Brady, 2000h. Report AN99R011). The rate is approximately 5 times the proposed label rate. Samples of mature apples were obtained from the field trial 73 days after the last application and processed by simulation of a commercial process. Samples were analysed using the GC/MS method DGM C05/98-0 (C000292). Recoveries of pyrimethanil from apples fortified at 0.05 and 1.0 mg/kg were 85 – 99% for wet pomace, 92 – 116% for juice, and 97 – 112% for whole apples.

Results of the four processing trials are summarized in Table 64.

Table 64. Residues of pyrimethanil in processed apple commodities (Germany, USA)

Processed Commodity	Residues (mg/kg)	Processing factor	Reference
Fruit	0.83	-	A81719
Peel	2.4	2.9	A81721
Homogenized flesh	0.47	0.57	
Homogenized flesh [a]	0.56	0.67	
Juice	0.36	0.43	
Juice [a]	0.32	0.39	
Press cake- pomace	1.6	2.0	
Press cake- pomace [a]	1.9	2.3	
Fruit	0.99	-	A81719
Peel	1.4	1.4	A81721
Homogenized flesh	0.73	0.73	
Homogenized flesh [a]	0.66	0.67	
Juice	0.60	0.61	
Juice [a]	0.65	0.62	
Press cake- pomace	0.62	0.62	
Press cake- pomace [a]	0.51	0.51	
Fruit	0.78	-	A81749
Peel	0.92	1.2	
Purée	0.28	0.36	
Purée [a]	0.30	0.38	
Juice	0.35	0.45	
Juice [a]	0.39	0.50	
Press cake- pomace	0.69	0.88	
Fruit	0.47	-	A81749
Peel	1.0	2.1	
Purée	0.48	1.0	
Purée [a]	0.46	0.98	
Juice	0.47	1.0	
Juice [a]	0.60	1.4	
Press cake- pomace	0.61	1.3	
Whole apple (RAC)	0.17 (0.11, 0.23)		B002853
Wet pomace	0.70	4.1	
	(0.75, 0.64)		
Apple juice [b]	0.06	0.35	
	(0.06, 0.06)		

Processed Commodity	Residues (mg/kg)	Processing factor	Reference
SUMMARY	Factor	Median	See above
Peel	1.2, 1.4, 2.1, 2.9	1.8	
Juice	0.35, 0.43, 0.45, 0.61, 1.0	0.45	
Puree	0.28, 0.46	0.37	
Wet pomace	4.1	4.1	

a - Heat-treated.

b - Fresh juice stored frozen. No pasteurisation.

Two trials were carried out in the USA (Michigan and Oregon) where the SC formulation of pyrimethanil was applied to *plum* trees three times at the rate of 4 kg ai/ha. The rate is approximately 5 times the label rate. Samples were taken 2 days after final application. The fresh prunes were placed in drying trays and placed in a laboratory tray air dryer at 68 – 79 °C for approximately 18 – 36 h to reduce water content to a desired range of 19 – 29%. The dried prunes were allowed to cool for a minimum 20 minutes prior to packaging (B003598, Dacus, 2002c; Report AN99R017).

Residues of pyrimethanil were determined by GC/MS using method DGM C05/98-0 (C000292). Samples were blended in acetone. Recoveries of pyrimethanil from plums fortified at 0.05, 5.0 and 50 mg/kg were 71 – 90.9% for plums and 69 – 70.0% for prunes.

Pyrimethanil residue levels were 7.0 and 5.7 mg/kg in plums (replicate samples, average 6.33 mg/kg) and 6.07 and 4.21 mg/kg in prunes (average 5.14 mg/kg). The calculated average concentration factor for pyrimethanil residues into prunes is 0.81.

Grape processing trials were reported from Italy and the USA. Grapes from trials on two sites in Italy received three foliar applications of the 40% SC formulation and three applications of a 80% WDG formulation of pyrimethanil. The total application rates were 0.8 – 2.4 kg ai/ha. Mature grapes were harvested 20 – 46 days after final application. Grape samples (10 kg) were used for preparation of white wine (A81697, Moede, 1992; Report UPSR 25/92). No information was supplied on the process. Residues were quantified using HLPC/UV detection with an LOQ of 0.05 mg/kg.

Table 65. Pyrimethanil in white wine from grapes (Italy) (A81697)

Trials site	Residues (mg/kg)		Processing factor
	grapes	wine	
Teriano	2.8	1.4	0.50
Teriano	2.1	1.0	0.48
Teriano	2.7	1.4	0.37
Teriano	0.61	0.58	0.97
Teriano	1.6	0.73	0.46
Teriano	1.5	0.62	0.41
Scorzoletta	2.1	0.72	0.34
Scorzoletta	1.1	0.59	0.54
Scorzoletta	0.35	1.0	2.9
Scorzoletta	2.4	0.18	0.08
Scorzoletta	1.0	0.48	0.48
Scorzoletta	< 0.05	0.28	-
		Average/Median	0.46

In a trial carried out in California, a single application of the SC formulation of pyrimethanil was made to grapes (Thompson Seedless) at a nominal rate of 1 kg ai/ha at each of the following growth stages: flowering, grape closure, colour change and approximately 21 days pre-harvest. Samples of fresh grapes were obtained the same day after the last application and processed into juice and raisin products at the California State University, Fresno (A81725, Brady, 1994, Report AN-92R-01; A89698, Brady, 1996; Report AN-92R-01-Amendment 2). To prepare juice and wet pomace, grapes were crushed in a stemmer-crusher. Raisins were prepared in the field by sun drying for 14 – 21 days on wet strength paper trays. The raisins were then placed in cold storage (13 °C) to

equilibrate the raisin moisture content. Finally, the raisins were placed on a processing line to remove stems and capstems and to grade by size.

Table 66. Pyrimethanil in juice, raisins and pomace from grapes (USA) (A89698)

Processed commodity	Residues (mg/kg)		Processing factors
	HPLC/UV	GC/MS	
Whole fruit	0.49		-
Juice	0.29	0.39	0.7
Wet pomace	1.18		2.4
Dry pomace	2.88	3.77	6.8
Raisins		0.8	1.6
Raisin waste		9.28	19.

A tomato processing study was provided, in which a single trial was conducted in California where the SC formulation of pyrimethanil was applied five times by foliar spray at 1.5 kg ai/ha with 7 days between applications, the last application being made one day prior to harvest. (B003291, Dacus, 2001c; Report AN99R013).

The tomatoes (130 kg) were processed in six steps: washing; grinding/crushing; heating (94 °C); separation into peel/seeds (pomace) and juice by screening; condensation of juice into puree and paste; canning (90 °C). Residues of pyrimethanil were determined using the analytical method DGM C05/98-0, (C000292), validated with an LOQ of 0.05 mg/kg. Recoveries of pyrimethanil from tomatoes fortified at 0.05, 2.0 and 10.0 mg/kg ranged from 67 – 93%.

Table 67. Residues of pyrimethanil in puree and paste from tomato processing (B003291)

Processed Fractions	Residues mg/kg	Processing factor
Tomato (RAC)	1.4 (1.4, 1.3)	-
Tomato puree	0.44 (0.43, 0.46)	0.31
Tomato paste	1.6 (1.5, 1.6)	1.1

Samples of fresh green beans taken from four sites in Europe following treatment with an SC formulation containing 400 g/L pyrimethanil were processed into canned and frozen beans (C012166, Peatman, 2001a; Report RESID 00/21).

Processing was carried out simulating industrial operations. For canned beans, the steps were sorting; washing; snubbing; cutting; blanching (85 °C, 6 minutes); draining and cooling; filling cans, brining (93 °C); seaming cans; heat processing (121 °C, 6 minutes). For frozen beans, the steps were similar through the draining and processing, except blanching was at 97 °C for 1 minute. Next, the beans were frozen in a fluidized bed (-34 °C, 6 minutes) and sealed in plastic bags. Samples of processed fractions were stored until analysis within a year of sampling. Residues of pyrimethanil were determined by GC/MSD, following method DGM C05/98-0 (C000292). The control samples of each processed fraction were fortified with pyrimethanil at levels of 0.01, 0.05, 0.10 and 0.20 mg/kg. Recoveries of pyrimethanil from all samples ranged from 74 to 102% (mean= 84%, RSD= 11%, n=14). The LOQ was 0.01 mg/kg for all matrices analysed.

Table 68. Residues of pyrimethanil in frozen green beans and canned green beans from green bean processing (C012166)

Trial No.	Processed Fractions	Residues [a] mg/kg	Processing factor
FRA 0101	RAC Canned beans Frozen beans	0.16 0.14 0.13	 0.88 0.81
DEU 0601	RAC Canned beans Frozen beans	0.37 0.16 0.21	 0.43 0.56
DEU 0501	RAC Canned beans Frozen beans	0.25 0.08 0.07	 0.32 0.28
DEU 0502	RAC Canned beans Frozen beans	0.24 0.09 0.08	 0.38 0.33
AVERAGE/ MEDIAN	Canned beans Frozen beans		0.50 0.40 0.50 0.44

Samples of fresh carrots taken from four sites in the southern EU following treatment with an SC formulation containing 400 g/L pyrimethanil were processed into canned and frozen carrots (C011650, Peatman, 2001b; Report RESID 00/20).

Processing was carried out simulating industrial operations. The steps for canning were: sort, trim, wash, peel (with steam at 60 – 80 psi for 30 seconds), wash, slice, fill cans, brine (1.2 g sugar and 25.6 g salt per litre of water, 93 °C), seam, and heat process (121 °C, 23 minutes). The process for frozen carrots was identical through slicing. The remaining steps were blanch (93 °C, 3 minutes), drain and cool, freeze in a fluidized bed (-34 °C, 7 minutes), fill plastic bags. The remaining steps or carrot puree after the peel and wash were steam (100 – 106 °C, 30 minutes, in a retort), blend, fill cans and seam, and heat process. Carrots were processed to juice via the same steps as canning through the second wash (after peeling). Samples of processed fractions were stored until analysis within a year of sampling. Residues of pyrimethanil were determined by GC/MSD, following method DGM C05/98-0 (C000292). The control samples of each processed fraction were fortified with pyrimethanil at levels of 0.01, 0.02, 0.10, 0.15, 0.20 and 0.25 mg/kg. Recoveries of pyrimethanil from all samples ranged from 69 to 108% (mean= 89%, RSD= 11%, n=18). The LOQ was 0.01 mg/kg for all matrices analysed.

Table 69. Residues of pyrimethanil in frozen carrots, canned carrots, peeled carrots, steamed carrots and juice from carrot processing (C011650)

Trial No.	Processed Fractions	Residues mg/kg	Processing factor
ESP 0201	RAC (fresh carrot)	0.02	
	Canned carrots	< 0.01	< 0.50
	Frozen carrots	< 0.01	< 0.50
	Carrot juice	< 0.01	< 0.50
	Puree	< 0.01	< 0.50
	Brine (can)	< 0.01	
GRC 0101	RAC (fresh carrot)	0.12	
	Canned carrots	0.07	0.58
	Frozen carrots	0.06	0.50
	Carrot juice	0.01	0.08
	Puree	0.03	0.25
	Brine (can)	< 0.01	

Trial No.	Processed Fractions	Residues mg/kg	Processing factor
GRC 0102	RAC (fresh carrot)	0.05	
	Canned carrots	0.03	0.60
	Frozen carrots	0.02	0.40
	Carrot juice	< 0.01	< 0.20
	Puree	0.03	0.60
	Brine (can)	< 0.01	
ESP 0101	RAC (fresh carrot)	0.05	
	Canned carrots	0.03	0.60
	Frozen carrots	0.02	0.40
	Carrot juice	< 0.01	0.20
	Puree	0.02	0.40
	Brine (can)	< 0.01	
	Peeled carrots	0.02	0.40
	Steamed unpeeled	0.04	0.80
	Wash water	< 0.01	
	Blanc water	< 0.01	
	Trimmings (canning)	0.14	
AVERAGE/ MEDIAN	Canned carrots		0.57 0.59
	Frozen carrots		0.45 0.45
	Carrot juice		0.24 0.20
	Puree		0.44 0.45
	Peeled carrots (for canning)		0.40 [a]
	Steamed (after peeling, for puree)		0.80 [a]

a - One value only

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Pyrimethanil was administered by gelatin capsule to 14 Holstein lactating cattle for 28 days. Dosing was made at the nominal dose rates of 1.0, 3.0, 10 and 50 ppm in the feed (B003807, Tew, 2002; Report 01AN27439). Three cows were assigned to each dose group except the 50 ppm dose group which had 4 cows. One cow was untreated and used as a control (0 ppm).

Milk samples were collected twice daily from study days 0, 1, 4, 8, 11, 15, 18, 22, 25 and 27. From one animal in the highest dose group, milk samples were taken from study days 29, 30, 31 and 35 during depuration. All test animals except for the one depuration cow were sacrificed at the end of a 28-day dose period. The depuration cow was sacrificed on Day 36. Muscle, liver, kidney and fat tissue samples were collected from all cows.

Milk samples were stored frozen (< -20 °C) from the time the AM and PM milk was pooled until analysis. Tissues collected were stored at < -20 °C until analysis. Milk samples were stored up to 169 days from collection to sample extraction. Tissues were stored up to 162 days (kidney), 128 days (fat) and 146 days (liver and muscle) from collection to sample extraction. The Meeting noted a lack of a storage stability study for livestock commodities.

The ruminant metabolism study (A81627, above) showed that pyrimethanil is extensively metabolized in cows and the major residue in kidney and milk was AE C614276 (2-(4-hydroxyanilino)-4,6-dimethylpyrimidine). Another metabolite, AE C614277 (2-anilino-4,6-dimethylpyrimidin-5-ol) was also found in kidney. Therefore, milk and tissue samples were initially analysed for pyrimethanil and AE C614276. During the course of the milk analysis, a second

measurable peak was noted and identified as AE C614277. The major milk metabolite appears to be AE C614 277 and not AE C614 276.

Sample analysis was performed using an analytical method that incorporated gas chromatography with an ion trap mass selective detector for detection and quantification of residues. The method AN/01/01 (B003462, Neal, 2001), was used to analyse milk for pyrimethanil and AE C614276. A revised method AN/01/02(B003870, Neal, 2002; Report AN/01/02 Version 2) was used during the analysis of milk for AE C614 277. Tissues were also analysed using Version 2. This method was detailed in the analytical methods section above.

Milk samples were subsequently analysed for the parent compound, pyrimethanil, and the metabolites AE C614 276 (2-(4-hydroxyanilino-4,6-dimethylpyrimidine) and AE C614 277 (2-anilino-4,6-dimethylpyrimidin-5-ol). Muscle, liver, kidney and fat were analysed for pyrimethanil and AE C614276 only. The LOQ was 0.01 mg/kg in milk, milk fat and skim milk for all compounds investigated. The LOQ in beef tissues was 0.05 mg/kg for analytes investigated. Procedural recovery samples were also run concurrently with every set of study samples analysed. Fortification levels were 0.01 to 0.10 mg/kg in whole milk, skim milk and milk fat and 0.05 to 1.0 mg/kg in bovine tissues. Individual recovery rates were determined to be 92 – 102% (milk), 96 – 137% (muscle), 102% (liver), 83 – 99% (kidney) and 89 – 106% (fat).

The residue levels in milk as a function of time are shown in Table 70 and Figure 4.

Table 70. Mean residues (mg/kg) of pyrimethanil, AE C614276 and AE C614277 in milk after oral administration of pyrimethanil at various dose levels for 28 consecutive days (B003807)

Sampling interval [a]	Dose Level in Feed (ppm)											
	1 ppm			3 ppm			10 ppm			50 ppm		
	Pyr [b]	Met1 [c]	Met2 [d]	Pyr [b]	Met1 [c]	Met2 [d]	Pyr [b]	Met1 [c]	Met2 [d]	Pyr [b]	Met1 [c]	Met2 [d]
0	ND	ND	NA	ND	ND	ND	ND	ND	NA	ND	ND	< LOQ
1	ND	ND	NA	ND	ND	ND	ND	< LOQ	NA	ND	< LOQ	0.029
4	NA	NA	NA	NA	NA	NA	ND	< LOQ	NA	ND	< LOQ	0.019
8	NA	NA	NA	NA	NA	NA	NA	NA	0.013	ND	< LOQ	NA
11	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND	< LOQ	NA
15	NA	NA	NA	NA	NA	< LOQ	NA	NA	0.014	ND	< LOQ	0.058
18	NA	NA	NA	NA	NA	NA	NA	NA	NA	ND	< LOQ	NA
22	NA	NA	NA	NA	NA	< LOQ	NA	NA	0.017	ND	< LOQ	0.063
25	NA	NA	NA	NA	NA	NA	NA	NA	0.016	< LOQ	< LOQ	0.052
27	NA	NA	NA	NA	NA	ND	NA	NA	0.012 [e]	ND	< LOQ	0.069 [f]

a - days after the first dosing

b - Pyrimethanil

c - AE C614276: 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine

d - AE C614277: 2-anilino-4,6-dimethylpyrimidin-5-ol

e - < LOQ, < LOQ, 0.017

f - 0.078, 0.048, 0.060, 0.088 mg/kg.

ND = Not detected (< LOD; LOD=0.0033 mg/kg)

NA = Not analysed

LOQ = 0.01 mg/kg

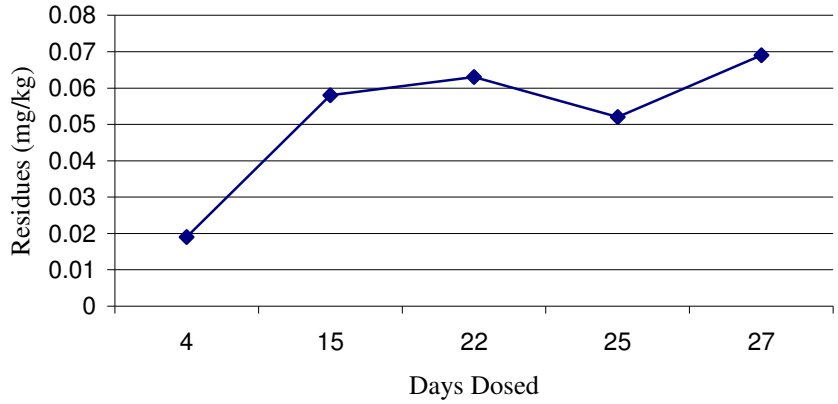


Figure 4. AE C614 277 Residues (Average Values for 50 ppm Dose Group) in Whole Milk as a Function of Time (B003807

Milk fat and skim milk were collected from the control cow and one of the 50 ppm cows on day 27. These samples were analysed to determine if residues would concentrate in milk fat. Residues of AE C614 277 were 0.031 mg/kg in milk fat and 0.064 mg/kg in skim milk. These data are consistent with the whole milk results (day 27 results = 0.078 mg/kg of AE C614 277).

Table 71. Residues in skim milk and milk fat after feeding for 28 consecutive days (B003807)

Dose Level	Matrix	Residues (mg/kg)		
		Pyrimethanil	AE C614 276	AE C614 277
Control	Milk fat	ND	ND	ND
Control	Skim milk	ND	ND	ND
50 ppm	Milk fat	ND	< LOQ	0.031
50 ppm	Skim Milk	ND	0.015	0.064

Tissue samples were analysed for pyrimethanil and AE C614 276 only. Residues of both compounds were < LOQ (0.050 mg/kg) at the 50 ppm dose level in muscle, liver and fat. The only measurable residues were found in kidney. Residues of the parent were either not detected or < LOQ in kidney. Residues of AE C614 276 ranged from < LOQ at the 1.0 ppm level to 0.88 ppm at the 50 ppm level. Table 72 summarizes the data for the tissues.

Table 72. Highest and mean residue levels (mg/kg) of pyrimethanil and AE C614 276 in animal tissues following feeding at various dose levels for 28 consecutive days (B003807)

Matrix	Dose Levels							
	1 ppm		3 ppm		10 ppm		50 ppm	
	Parent	AEC614276	Parent	AEC614276	Parent	AEC614276	Parent	AEC614276
Muscle	NA	NA	NA	NA	NA	NA	< LOQ [a]	< LOQ [a]
(HR)	NA	NA	NA	NA	NA	NA	< LOQ	< LOQ
Liver	NA	NA	NA	NA	NA	NA	< LOQ	< LOQ
(HR)	NA	NA	NA	NA	NA	NA	< LOQ	< LOQ
Kidney	< LOQ [a]	< LOQ [a]	ND	0.066 [c]	ND [b]	0.12 [d]	ND [b]	0.63 [e]
(HR)	< LOQ	< LOQ	ND	0.08	ND	0.13	ND	0.88
Fat	NA	NA	NA	NA	< LOQ [a]	< LOQ	< LOQ	< LOQ
(HR)	NA	NA	NA	NA	< LOQ	< LOQ	< LOQ	< LOQ

a - LOQ = 0.05 mg/kg

b - ND = <LOD = ~0.01 – 0.02 mg/kg

c - 0.06, 0.06, 0.08

d - 0.12, 0.12, 0.11

e - 0.88, 0.23, 0.77

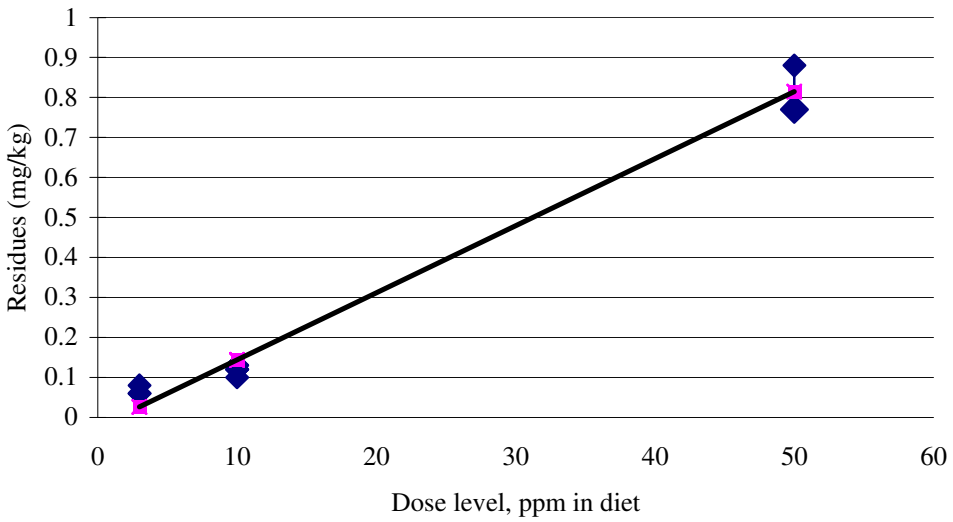


Figure 5. Linear Regression of Residues of AE C614 276 in Kidney (B003807)

One cow from the 50 ppm dose group was kept for 1 week after dosing was terminated in order to measure the decline of residues in milk and tissues. The last dose was administered on the morning of Day 28. By Day 29, AE C614277 residues in milk were below the LOQ; residues of pyrimethanil and AE C614276 residues were not detectable. Day 30 milk samples did not have detectable residue of any analyte. The cow was sacrificed on Day 35 and tissues collected. AE C614276 residues in kidney, the only tissue that had contained measurable residue, were at < LOQ (0.05 mg/kg).

National Residue Definitions

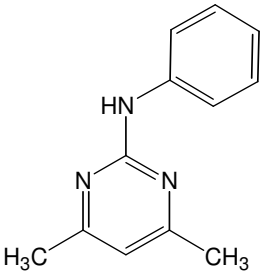
The residue definition for enforcement in the USA is pyrimethanil for plant commodities. For livestock tissues (excluding poultry) the residue definition is pyrimethanil plus 4-(4,6-dimethyl-2-pyrimidinyl) aminophenol, and for milk the residue definition is pyrimethanil plus 4,6-dimethyl-2-(phenylamino)-5-pyrimidinol. The residue definition in the EU for MRLs in plant commodities is pyrimethanil. No residue definition was found necessary for livestock commodities. The residue definition in Canada for plant commodities is pyrimethanil. The residue definition in Australia and New Zealand is pyrimethanil for plant commodities. The residue definition in Japan for plant and animal commodities is pyrimethanil. The residue definition in South Korea for plant commodities is pyrimethanil.

APPRAISAL – RESIDUE AND ANALYTICAL ASPECTS

Pyrimethanil is an anilinopyrimidine fungicide that inhibits the secretion of hydrolytic enzymes by the fungi that are needed during the infection process. Pyrimethanil blocks the ability of fungi to degrade and digest the plant tissues, thus stopping penetration and development of the disease.

At the 37th session of the CCPR (ALINORM 04/27/24), pyrimethanil was listed as a candidate for evaluation of a new compound by the 2007 JMPR.

Chemical name: N-(4,6-dimethylpyrimidin-2-yl) aniline



Animal metabolism

The Meeting received results of an animal metabolism study in lactating dairy cows. A lactating dairy cow was orally dosed for seven consecutive days with [^{14}C]pyrimethanil at a daily dose rate of 10 ppm in the diet, which corresponds to 0.4 mg/kg bw per day for a 600 kg cow. Residues in muscle and fat were too low to isolate and identify (0.02–0.04mg/kg total radioactive residue, TRR). The TRR in milk reached a plateau on about day 5 (0.07 mg/kg). No pyrimethanil was found in the milk from any day of the treatment. The major metabolite present in milk (64% TRR) was identified 2-anilino-4,6-dimethylpyrimidin-5-ol. Also present in milk were metabolites (27% TRR) characterised as highly polar.

Parent pyrimethanil was not found in kidney or liver. The TRR in kidney was identified as 46% 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine, 5% 2-anilino-4,6-dimethylpyrimidin-5-ol, and 7% 2-(4-hydroxyanilino)-4-hydroxymethyl-6-methylpyrimidine. Again, 42% TRR was characterised as highly polar. No metabolite was identified in liver, but the TRR was characterised as 48% protein, 9% lipid, 7% ribonucleic acid and 6% sulfurated glycoamino-glycans.

Metabolism in the rat was quite similar to that of the cow. In the rat, only small amounts of the administered pyrimethanil were found in faeces and none was found in urine. The major metabolite in urine and faeces was 2-(4-hydroxyanalino)-4,6-dimethylpyrimidine and its sulfate, 13–52%. Other metabolites, generally < 10% of total extracted radioactivity in the excreta, were 2-anilino-4,6-dimethylpyrimidin-5-ol, 2-(4-hydroxyanilino)-4-hydroxymethyl-6-methylpyrimidine, 2-(4-hydroxyanilino)-6-dimethyl-pyrimidin-5-ol and 2-anilino-6-methylpyrimidine-4-methanol.

The Meeting concluded that pyrimethanil is very extensively metabolised in cattle, forming monohydroxy and dihydroxy derivatives in milk and kidney, and being incorporated into biological substrates in liver. No accumulation occurs in muscle or fat.

Plant metabolism

The Meeting received plant metabolism studies for the foliar application of [^{14}C]pyrimethanil, radiolabelled either on the aniline ring or at C-2 of the pyrimidine ring, for apples, grapes, carrots, tomato, leaf lettuce and strawberry. Generally the majority of the radioactivity was removed in a dichloromethane surface wash (56% grapes, 90% tomato). In all instances, the major component of the TRR was pyrimethanil (apple fruit, 70 – 77%; carrot root, 70 – 89%; tomato fruit, 95 – 96%; leaf lettuce 44%; strawberry fruits, no identifications made). Minor metabolites identified included hyrdoxylated and conjugated derivatives of pyrimethanil 2% TRR, and the ß-O-glucoside of 2-anilino-4-hydroxymethyl-6-hydroxymethylpyrimidine 3% TRR in apples; malonyl-β-O-glucoside of 2-anilino-hydroxymethyl-6-methylpyrimidine 6% TRR, and the β-glucoside of 2-anilino-4-hydroxymethyl-6-methylpyrimidine 6% TRR on carrot foliage (< 1% TRR each on carrot root); hydroxylated and conjugated compounds of pyrimethanil 6–28% TRR on tomato leaves; conjugate of 2-(4-hydroxyanilino)-4,6-dimethylpyridine 5% TRR and conjugate of 2-anilino-4,6-dimethylpyrimidin-5-ol, 8% TRR on leaf lettuce. Where both radiolabels were tested on the same crop, no significant differences were found in the compositions of the TRRs.

The Meeting concluded that the metabolism of pyrimethanil had been adequately defined via studies on three distinct crop types: fruit, root and leafy. Very little metabolism occurs, and the major

portion of the residue is the parent pyrimethanil. The similarity in metabolic profiles between studies conducted with the radiolabel in either the aniline ring or the pyrimidine ring indicates no cleavage at the ring junction (aniline amino group). Minor metabolites identified are hydroxylated and conjugated derivatives of pyrimethanil, and are generally less than 10% TRR.

Environmental fate

The Meeting received studies on aqueous hydrolysis, aerobic and anaerobic degradation in soil, photolysis in water and residues in succeeding crops. Pyrimethanil is stable to hydrolysis in water at pH 5, 7 and 9 at 20 °C.

Under aerobic conditions, pyrimethanil slowly degraded in soil with about 80% remaining after 130 days. This was followed by a rapid decline in both extractable radioactivity and pyrimethanil levels. At higher soil treatment rates (500 mg/kg) differences were seen in the apparent degradation of the pyrimidine and aniline labels. With the pyrimidinyl label, about 60% of the extractable radioactivity was identified as 2-amino-4,6-dimethylpyrimidine. Cleavage of the aniline linkage is indicated.

Pyrimethanil does undergo photolytic degradation in water (sterile buffer) at pH 4 and pH 7 with estimated half-lives of 1 and 80 days, respectively. In a separate experiment using in sterile water containing humic acids, the half-life was reduced to less than 2 days at pH 7.

The Meeting concluded that pyrimethanil is stable under aqueous hydrolysis at pH 2 – 9 and is relatively stable on soil under aerobic conditions. It was also concluded that pyrimethanil is not stable in water under photolysis.

The uptake of 2-[^{14}C]pyrimidinyl-labelled pyrimethanil in *rotational crops* under confined conditions was reported to the Meeting. The pyrimethanil was applied to soil at a rate of 2.4 kg ai/ha. Substantial residues were found in crops planted 30 days after the treatment, 0.23 to 8.2 mg/kg TRR as pyrimethanil. Pyrimethanil comprised 1% (radish top) to 45% (wheat forage) of the TRR. The major identified metabolite (> 10% TRR) was 2-anilino-4-hydroxymethyl-6-methylprimidine in wheat forage and lettuce. Pyrimethanil was < 0.05 mg/kg in all rotational crops at the 30 day plantback interval, *except* for wheat grain (73 day, 0.41 mg/kg TRR, < 0.001 mg/kg pyrimethanil), forage (35 day immature, 1 mg/kg TRR, 1.1 mg/kg pyrimethanil), and straw (73 day, 8.2 mg/kg TRR, 0.22 mg/kg pyrimethanil). At a 130 day plantback interval, total residues in the crops declined to 0.01 to 0.03 mg/kg, with parent comprising 1 – 26% of the TRR. No extractable metabolite exceeded 10% TRR.

Three field rotational crop studies with a single crop, wheat, were conducted. Using a 30 day plantback interval following harvest of treated potatoes (3 applications at 0.8 kg ai/ha), residues of pyrimethanil and 2-anilino-4-hydroxymethyl-6-methylprimidine were below the limits of detection (< 0.012 mg/kg for pyrimethanil and < 0.015 mg/kg for 2-anilino-4-hydroxymethyl-6-methylprimidine), except for one wheat forage sample (< 0.05 mg/kg LOQ). The intervals from plantback to harvest were 128 – 232 days for forage and 190 – 316 days for straw.

The Meeting concluded that residues of pyrimethanil, in rotational crops planted 30 days or more after the final application of pyrimethanil to the primary crop, will most likely be below the LOQ (< 0.05 mg/kg), with the possible exception of forages and straws.

Methods of Analysis

The Meeting received information for analytical methods on the quantitative determination of pyrimethanil in a variety of crops and for the determination of pyrimethanil and metabolites 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine and 2-anilino-4,6-dimethylpyrimidine in bovine commodities.

The plant commodity methods consist of organic solvent extraction (acetone or methanol), clean-up, and analysis by either gas chromatography, with a mass spectrometer detector (GC/MS, m/z 198), or by high performance liquid chromatography with an ultraviolet detector (HPLC). The HPLC method was validated for apples, tomatoes, grapes, green beans, wine, grape juice, and grape pomace.

The validated limits of quantitation (LOQs) are 0.05, 0.05, 0.02, 0.05, 0.02 and 0.02 mg/kg, respectively. The GC/MS method was validated for potatoes, carrots, tomatoes, green beans, lettuce, sweet peppers, strawberries, raspberries, apples, peaches, plums and oranges. A LOQ of 0.05 mg/kg was demonstrated for all of these commodities.

A radiovalidation study was conducted for the GC/MS procedure. Lettuce from the metabolism study was subjected to the extraction and analysis procedures of the method. Extraction efficiency was 97%.

A GC/MS method was described for the determination of pyrimethanil and metabolites 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine and 2-anilino-4,6-dimethylpyrimidin-5-ol in milk, fat, muscle, liver and kidney. The metabolites are converted to methylated derivatives prior to analysis. The demonstrated LOQs are 0.01 mg/kg for each of the analytes in milk and 0.05 mg/kg in each of the analytes in the various tissues. The independent laboratory validation encountered considerable problems and did not achieve acceptable validation for precision for pyrimethanil in meat at 0.05 mg/kg and overall at levels of 0.05 and 0.5 mg/kg. No radiovalidation of the method was reported.

Multiresidue methods (US FDA and DFG S 19) were reported for pyrimethanil in various plant commodities.

The Meeting concluded that adequate analytical methods exist for both data collection and enforcement purposes for pyrimethanil residues in plant commodities and for pyrimethanil, 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine (SN 614276), and 2-anilino-4,6-dimethylpyrimidin-5-ol (SN 614277) in milk and bovine tissues.

Stability of pesticide residues in stored analytical samples

The Meeting received information on the stability of pyrimethanil in a variety of crop matrices, but no information on stability in livestock commodities. Pyrimethanil is stable (< 30% loss) in apples, grapes, tomatoes, lettuce, carrots, peas (dried), peaches and plums for at least 365 days when the commodities are stored frozen at about -20 °C.

The Meeting concluded that pyrimethanil is stable on frozen plant commodities for at least one year. No conclusions are possible on the stability of pyrimethanil or its metabolites in livestock commodities.

Residue definition

The major component of the residue on numerous plant commodities, from the foliar application of pyrimethanil, is pyrimethanil. Minor amounts of hydroxylated pyrimethanil derivatives are found, generally < 10% each of the total residue. The two analytical methods determine only pyrimethanil.

In livestock (cow) commodities, pyrimethanil is not found following oral administration of the compound. The major metabolites are 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine and 2-anilino-4,6-dimethylpyrimidin-5-ol, in kidney and milk, respectively. The analytical method provided determines the parent and the two named metabolites.

The log of the octanol/water partition coefficient is 2.8. In the cow feeding study, no pyrimethanil (< 0.05 mg/kg) was found in either fat or muscle at a 50 ppm feeding level. In the same study, the milk fat contained 0.031 mg/kg of 2-anilino-4,6-dimethylpyrimidin-5-ol, and the skim milk contained 0.064 mg/kg of 2-anilino-4,6-dimethylpyrimidin-5-ol and 0.015 mg/kg 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine. Thus, the total residue concentrated slightly in the non-fat portion of milk.

The Meeting concluded that the residue definition for both enforcement and dietary exposure considerations for plant commodities is pyrimethanil. The Meeting further concluded that the residue definition for both enforcement and dietary exposure considerations for milk is the sum of pyrimethanil and 2-anilino-4,6-dimethylpyrimidin-5-ol, expressed as pyrimethanil and for livestock

tissues (excluding poultry) is the sum of pyrimethanil and 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine, expressed as pyrimethanil.

The Meeting also decided that pyrimethanil is not fat-soluble.

Results of supervised trials on crops

The Meeting received supervised trials data for the foliar application of pyrimethanil as a suspension concentrate formulation (SC) to a variety of fruit, vegetable, and nut crops. Additionally, supervised trial data reports were received for the post-harvest treatment of citrus, pome fruit and cherries.

Citrus fruits

Various post-harvest treatments of lemon, orange, tangelo, tangerine, and grapefruit were reported for 45 trials from the USA. The USA GAP is: 204 g/L pyrimethanil + 263 g/L imazalil SC, dip or wash at 0.08 kg ai/hL or drench at 0.08 kg ai/hL or aqueous line spray at 0.1 kg ai/hL or wax line spray/storage and pack wax at 0.2 kg ai/hL, with a maximum of two treatments (of all types); 400 g/L pyrimethanil SC, dip or wash at 0.1 kg ai/hL or drench at 0.05 kg ai/hL or aqueous line spray at 0.2 kg ai/hL or wax line spray/storage and pack wax at 0.2 kg ai/hL, with a maximum of 2 or 3 treatments. Additionally, eight trials for the post-harvest treatment of oranges and mandarins in Spain were reported. No GAP was supplied, and the GAP of the USA was utilised. Thirty-three USA trials (9 × lemon, 10 × orange, 5 × grapefruit, 4 × tangelo and 4 × tangerines) were at maximum GAP. No Spanish trials matched the USA GAP.

Residues in the 32 trials in ranked order (median underlined) were: 1.2 (3), 1.4, 1.5 (2), 1.7 (2), 1.9, 2.1, 2.2, 2.3, 2.6, 2.7 (3), 2.8 (3), 2.9, 3.1, 3.3, 3.4 (2), 3.6, 4.1 (2), 4.2, 4.3, 4.6, 5.5, 5.8 mg/kg. No data were provided on the analysis of the edible portion (pulp). The Meeting estimated a maximum residue level of 7 mg/kg (Po) and an STMR of 2.8 mg/kg.

Pome fruits

Pre-harvest apple trials were reported from Europe and the USA. Pear trials were reported from the USA.

Two apple trials were conducted in Germany, two in northern France, and one in the UK. None of the trials matched the GAP of Belgium, 400 g/L SC, 0.45 kg ai/ha, 0.22 kg ai/hL, 5 applications, 28 day PHI. Two apple trials were conducted in southern France, two in Italy and one in Spain. One trial matched the GAP of Italy, 400 g/L SC, 0.04 kg ai/hL, 5 applications, 14 day PHI. The residue (Italy) was 0.56 mg/kg.

Twelve apple trials were conducted in the USA at the GAP, 400 g/L SC, 0.45 kg ai/ha, 1.8 kg ai/ha per season, 72 day PHI. The residues in ranked order are: < 0.05 (7), 0.06, 0.10, 0.12, 0.15, 0.16 mg/kg.

Six pear trials were conducted in the USA under the same USA GAP as apples. The residues found were: < 0.05 (6) mg/kg.

Post-harvest treatment of apples was reported from Spain and France and the USA. The GAP of Belgium is 200 g/L pyrimethanil + 200 g/L imazalil SC, spray or dip at 0.04 kg ai/hL, one treatment. Two of nine European trials were at the maximum GAP, and residues are 0.57 and 1.7 mg/kg. An additional trial matched the GAP of Chile, 3.78 mg/kg.

The GAP of the USA is dipping, drenching or aqueous line spray at 0.1 kg ai/hL or wax line spray at 0.2 kg ai/hL. Up to 2 treatments (of any combination) may be used. The GAP of Chile is identical, but only one treatment is permitted. Using the GAP of the USA, no trials are at GAP. Using the GAP of Chile, 10 of 32 trials were at maximum GAP. The residues in ranked order on apples were: 0.27, 0.28, 0.33, 0.39, 0.64, 0.70, 1.1 (2), 1.2, 1.5 mg/kg. Studies on the post-harvest treatment of pears in the USA were also reported. The GAPs of Chile and the USA are the same as for apples. Using the GAP of the USA, the residues of two trials are at GAP 1.01 and 1.18 mg/kg. Using the GAP of Chile, an additional eight of 35 trials were at the maximum GAP. Residues of pyrimethanil in

ranked order were: 0.13, 0.18, 0.32, 0.45, 0.56, 0.86, 1.1 (2) mg/kg. Six post-harvest treatment trials on pears were reported from France, Spain and Belgium. No trials matched the GAPs of Chile or the USA. Two trials (BE, FR) matched the GAP of Belgium (200 g/L pyrimethanil + 200 g/L imazalil SC, spray or dip at 0.04 kg ai/hL, one treatment), and the residue values are 0.32 and 0.55 mg/kg.

Studies on the thermofogging post-harvest treatment of apples and pears in Europe was reported. However, the only GAP supplied (Chile) has yet to be approved by the national government. The Meeting noted that the maximum residue under the proposed GAP was 3.5 mg/kg on pears in Italy.

The residue values for post-harvest treatment of apples and pears in the USA and Europe at the GAPs of Chile or the USA are from the same population and may be combined. Residues in the 21 trials in ranked order (median underlined) were: 0.13, 0.18, 0.27, 0.28, 0.32, 0.33, 0.39, 0.45, 0.56, 0.64, 0.70, 0.86, 1.0, 1.1 (4), 1.2 (2), 1.5, 3.8 mg/kg. Based on the post-harvest treatments, the Meeting estimated an STMR of 0.70 mg/kg and a maximum residue level of 7 mg/kg for pome fruit (Po).

Stone fruits

Apricot, peach and plum trials were reported from the USA. The GAP is identical for all: 600 g/L SC, 0.8 kg ai/ha, 2.4 kg ai/ha/season, 2 day PHI. Five apricot trials were at maximum GAP: 0.61, 0.64, 1.2, 1.3, 1.7 mg/kg. Twelve peach trials were at maximum GAP: 0.38, 0.54, 0.94, 1.1, 1.2, 1.3 (3), 1.5, 1.6, 2.6 mg/kg. Eight plum trials were at maximum GAP: 0.05, 0.44, 0.58, 0.59 (2), 0.61, 0.62, 1.2 mg/kg.

The Meeting considered the apricot, peach and plum trials not to be from the same population. The Meeting estimated an STMR of 1.2 mg/kg and a maximum residue level of 3 mg/kg for apricots. The meeting estimated an STMR of 1.3 mg/kg and a maximum residue level of 4 mg/kg for peaches and for nectarines. The Meeting estimated an STMR of 0.59 mg/kg and a maximum residue level of 2 mg/kg for plums.

Reports on the post-harvest treatment of peaches and plums in the USA were reported, but no GAP was provided.

Reports on the post-harvest treatment of cherries in Germany were reported. A GAP was supplied for Chile (400 g/L SC, dipping, 0.04 kg ai/hL, 1 application. Eight trials were at maximum GAP, and the values in ranked order were: 0.82, 1.0, 1.1, 1.2, 1.4(3), 2.5 mg/kg. The Meeting estimated an STMR of 1.3 mg/kg and a maximum residue level of 4 mg/kg (Po) for cherries.

Berries and other small fruits

Supervised trials for the foliar application of pyrimethanil to grapes were reported from the EU and the USA. Five trials in northern Europe (two from Germany and three from France) were evaluated against the GAP of France (400 g/L SC, 1 kg ai/ha, 1 application, 21 days PHI: 0.37, 0.44, 0.59, 0.97, 1.1 mg/kg); and 10 trials in southern Europe (2 Spain, 6 France, 2 Italy: 0.28, 0.48, 1.0, 1.5 mg/kg) were evaluated against the GAP of Spain (400 g/L SC, 0.08 kg ai/hL, one application, 21 day PHI). Nine trials were at maximum GAP, and the residues in ranked order were: 0.28, 0.37, 0.44, 0.48, 0.59, 0.92, 1.0, 1.1, 1.5 mg/kg.

Twelve trials were reported from the USA (USA GAP: 600 g/L SC, 0.8 ka ai/ha, 1.6 kg ai/ha/season, 7 day PHI). All trials were at maximum GAP, and the residues found were: 0.12, 0.44, 0.49, 0.64, 0.66, 0.71, 0.89, 1.2, 1.5, 1.6, 2.0, 2.5 mg/kg.

The Meeting considered the EU and USA trials to be from the same population and combined the results. Residues in the 21 trials in ranked order (median underlined) were: 0.12, 0.28, 0.37, 0.44(2), 0.48, 0.49, 0.59, 0.64, 0.66, 0.71, 0.89, 0.92, 1.0, 1.1, 1.2, 1.5 (2), 1.6, 2.0, 2.5 mg/kg. The Meeting estimated an STMR of 0.71 mg/kg and a maximum residue level of 4 mg/kg for grapes.

Eight trial were conducted on the foliar application of pyrimethanil to strawberries in the USA, where the GAP is 600 g/L SC, 0.8 kg ai/ha, 2.4 kg ai/ha/season, 1 day PHI. All trials were at

maximum GAP, and the residues in ranked order (median underlined) were: 0.79, 0.93, 0.99, 1.1, 1.2, 1.3(2), and 2.3 mg/kg. The Meeting estimated an STMR of 1.2 mg/kg and a maximum residue level of 3 mg/kg for strawberries.

Bananas

Eleven trials each on the foliar treatment of bagged and unbagged bananas with pyrimethanil were reported from Costa Rica (3), Ecuador (3), Colombia (3) and Guatemala (2). The GAP is identical in all these countries: 600 g/L SC, 0.3 kg ai/ha, 6 applications, 0 day PHI (constant harvesting). All residues were below the LOQ except one bagged banana sample in Ecuador. The residues in ranked order were: < 0.05 (21), 0.09 mg/kg. All pulp samples were < 0.05 mg/kg. The Meeting estimated an STMR of 0.05 mg/kg and a maximum residue level of 0.1 mg/kg for bananas.

Bulb vegetables

Nine trials were conducted on the foliar application of pyrimethanil to dry bulb onions and spring onions in the USA, where the GAP is: 600 g/L SC, 0.8 kg ai/ha, 2.4 kg ai/ha/season, 7 days PHI. All trials were conducted at maximum GAP, and the residues in ranked order on bulb onions were: < 0.05 (3), 0.075, 0.087, 0.095 mg/kg. Residues on green onions in ranked order are: 0.26, 0.38, 1.6 mg/kg. The Meeting estimated an STMR of 0.062 mg/kg and a maximum residue level of 0.2 mg/kg for bulb onions (dry). The Meeting estimated an STMR of 0.38 mg/kg and a maximum residue level of 3 mg/kg for spring onions.

Fruiting vegetables, other than Cucurbits

Sixteen trials were conducted on the foliar application of pyrimethanil to tomatoes in the USA, where the GAP is: 600 g/L SC, 0.3 kg ai/ha, 1.6 kg ai/ha/season, 1 day PHI. All trials were at maximum GAP, and the residues in ranked order were: 0.06, 0.07 (3), 0.10, 0.13, 0.14 (2), 0.15, 0.16, 0.17, 0.20, 0.22, 0.23, 0.35, 0.37 mg/kg.

Eight glasshouse trials were conducted in Europe, 2 in France and 6 in the Netherlands. The GAP of France is 400 g/L SC, 0.8 kg ai/ha, 2 applications, 3 day PHI. All trials were at maximum GAP, and the residues in ranked order (median underlined) were: 0.26 (2), 0.31 (2), 0.33 (2), 0.36 (2) mg/kg.

The USA and EU trials were not considered to be from the same population, and the Meeting used the EU trials to estimate an STMR of 0.32 mg/kg and a maximum residue level of 0.7 mg/kg for tomatoes.

Leafy vegetables

Trials were conducted on both head lettuce and leaf lettuce in Europe. The GAP of France (400 g/L SC, 0.8 kg ai/ha, 2 applications, 21 day PHI) was applied to field trials in the UK (4), the Netherlands (1), France (North, 2), and Germany (2): < 0.05 (5), 0.11, 0.13, 0.28, 0.43 mg/kg. The GAP of Italy (400 g/L SC, 0.8 kg ai/ha, 2 applications, 14 day PHI) were applied to trials in Italy (2), Greece (1), France (South, 1), and Spain (1): 0.05, 0.14, 0.31, 0.77, 1.2 mg/kg. The residues in ranked order for head lettuce were: < 0.05 (5), 0.05, 0.11, 0.13, 0.14, 0.28, 0.31, 0.43, 0.77, 1.2 mg/kg.

Glasshouse trials were also reported from Europe (UK, Netherlands and Germany) for head lettuce. The GAP of Italy is 400 g/L SC, 0.8 kg ai/ha low volume, 0.08 kg ai/hL high volume, 2 applications, 14 day PHI. All trials were at maximum GAP, using high volume, and the residues in ranked order were: 0.37, 0.41, 0.49, 0.61, 0.85, 0.97 (2), 1.4, 1.6 mg/kg.

The Meeting considered the field and glasshouse trials in Europe not to be from the same population and used the glasshouse trials to estimate an STMR of 0.85 mg/kg and a maximum residue level of 3 mg/kg for head lettuce.

Field trials were also conducted in France, Greece, Italy and Portugal for leaf lettuce. Using the GAP of Italy (400 g/L SC, 0.8 kg ai/ha, 2 applications, with a 14 day PHI), three of the four trials were at maximum GAP. The residues in ranked order are 0.62, 0.68, 7.5 mg/kg. The Meeting

considered three trials an insufficient number for the estimation of an STMR and a maximum residue level for leaf lettuce.

Legume vegetables

Trials for the application of pyrimethanil to common beans (green beans) were reported from France (4) and Germany (3). The GAP in France is 400 g/L SC, 0.6 kg ai/ha, 1 application, 14 day PHI. Residues in ranked order were: < 0.05 (3), 0.05, 0.07, 0.08, 0.09.

Trials were also reported for the treatment of green beans in glasshouses in France (2), Italy (1), Spain (3), and Greece (2). The GAP of France is 400 g/L SC, 0.6 kg ai/ha, 14 day PHI. The residues in ranked order (median underlined) were: < 0.05, 0.12, 0.13, 0.20, 0.25, 0.28, 0.91, 1.9 mg/kg.

The Meeting considered the field and glasshouse trials on green beans not to be from the same population and used the glasshouse trials to estimate an STMR of 0.22 mg/kg and a maximum residue level of 3 mg/kg for common beans.

Root and tuber vegetables

Trials were reported on the foliar application of pyrimethanil to carrots in Brazil and Europe. Two trials in Brazil did not match the GAP of Brazil (300 g/L SC, 0.6 kg ai/ha, with a 14 day PHI). Nine trials, conducted in Northern Europe were received from the UK, France, Germany and the Netherlands. Eight trials were at the maximum GAP of France, i.e., 400 g/L SC, 0.8 kg ai/ha × 2 applications, with a 21 day PHI. Residues in rank order were: < 0.05 (2), 0.07 (2), 0.24, 0.28, 0.35, 0.36 mg/kg. Nine trials were conducted in Southern Europe in Spain, France, Greece, Italy and Portugal, and all were conducted at the maximum GAP of Italy (400 g/L SC, 0.8 kg ai/ha × 2 applications, with a 7 day PHI), residues in rank order were: < 0.05, 0.05, 0.08, 0.09, 0.14, 0.21, 0.33, 0.44, 0.54 mg/kg. Residues in the two areas were comparable, and the combined residue values in ranked order (median underlined) were: < 0.05 (3), 0.07 (3), 0.08, 0.09, 0.13, 0.14, 0.21, 0.24, 0.28, 0.33, 0.35, 0.36, 0.44, 0.54 mg/kg. The Meeting estimated an STMR of 0.14 mg/kg and a maximum residue level of 1 mg/kg for carrots.

Supervised trials for the foliar application of pyrimethanil to potatoes were reported from the USA where the GAP is 0.3 kg ai/ha (600 g/L SC), with a maximum of 1.6 kg ai/ha/season, with a 7 day PHI. The ranked order of residue values for 16 trials at maximum GAP was: < 0.05(16). The Meeting estimated an STMR of 0.05 mg/kg and a maximum residue level of 0.05* mg/kg for potatoes.

Tree nuts

The Meeting received a report on supervised field trials on almonds in the USA, where the GAP is 0.8 kg ai/ha (600 g/L SC), with a maximum of 2.4 kg ai/ha/season, and a 30 day PHI. Six trials were at the maximum GAP and the ranked order of residue values on almond hulls were: 1.9, 2.4, 2.6, 2.7, 3.6, 9.2 mg/kg. The ranked order of values on almond nutmeat was: < 0.05(4), 0.06, 0.10 mg/kg. The Meeting estimated an STMR of 2.6 mg/kg and a maximum residue level of 12 mg/kg for almond hulls. The Meeting also estimated an STMR of 0.05 and a maximum residue level of 0.2 mg/kg for almond nutmeats.

Legume animal feeds

Thirteen supervised trials were carried out in Europe (France, Germany and the UK) for the foliar application of pyrimethanil to fodder peas (field peas, combining peas, protein peas). The GAP in France is 400 g/L SC, 0.6 kg ai/ha, with a 28 day PHI. Eleven trials were conducted at this maximum GAP, and the values in ranked order for dry seeds were: < 0.05 (4), 0.08, 0.09, 0.11, 0.12, 0.22, 0.25, 0.30 mg/kg. The highest residue was 0.30 mg/kg. The values in ranked order for straw were: < 0.05 (3), 0.15(2), 0.24, 0.28, 0.64, 0.66, 1.0 mg/kg. The highest residue was 1.0 mg/kg. The Meeting

estimated an STMR of 0.09 mg/kg and a maximum residue level of 0.5 mg/kg for fodder pea seed (dry) and an STMR of 0.20 mg/kg and a maximum residue level of 3 mg/kg for fodder pea straw.

Fate of residues during processing

The Meeting received processing studies for oranges, apples, grapes, tomatoes, green beans and carrots. No information was supplied on the fate of radiolabelled pyrimethanil under general processing conditions.

Oranges with incurred residues of pyrimethanil from post-harvest treatment (2.9 mg/kg; 7.5 mg/kg) were processed by a commercial process into juice, dried pulp and citrus oil. The average processing factors were 0.01 for juice, 0.45 for pulp (dried), and 20 for citrus oil. Applying these factors to the STMR for citrus (2.8 mg/kg), the Meeting estimated the following STMR-Ps for citrus juice, citrus pulp (dried) and citrus oil, respectively: 0.028 mg/kg; 1.3 mg/kg; 56 mg/kg.

Apple processing studies were conducted in Germany (four trials) and the USA (one trial). The median processing factor for juice was 0.45 (n=5), the average factor for puree (n=2) was 0.37, and the factor for wet pomace (n=1) was 4.1. Applying these factors to the STMR, the Meeting estimated: STMR-P of 0.32 mg/kg for juice; a STMR-P of 2.9 mg/kg for wet apple pomace, and a STMR-P of 0.26 mg/kg for apple puree. The STMR-P and maximum residue limit estimates for dry apple pomace are 7.2 mg/kg (0.7 mg/kg × 4.1/0.40) and 40 mg/kg (3.8 mg/kg × 4.1/0.4), respectively, assuming that wet apple pomace contains 40% dry matter (*Table of OECD Feedstuffs Derived from Field Crop).*

A plum to prune processing study was conducted in the USA. The processing factor of 0.81 applied to the STMR of fresh plums (0.59 mg/kg) yields an STMR-P of 0.48 mg/kg for (dried) prunes.

Processing studies for the conversion of grapes to white wine was reported from Italy. The median processing factor (n=11, one value > 1 with all others < 1) was 0.48. Applying this factor to the STMR for grapes of 0.71 mg/kg yields a STMR-P of 0.34 mg/kg for wine.

A processing study for the conversion of grapes to juice and raisin (USA) was reported to the Meeting. The processing factors for juice, wet pomace and raisins are 0.7, 2.4 and 1.6, respectively (n=1). Applying these factors to the appropriate STMRs or HR levels the Meeting estimated the following: STMR-P for juice 0.50 mg/kg; STMR-P for wet grape pomace 1.7 mg/kg; STMR-P for grape raisins 1.1. The Meeting also estimated a maximum residue level of 5 mg/kg for grape raisins.

A tomato processing study was conducted in the USA in which tomatoes with incurred residues were processed by a commercial-type method into puree and paste, with processing factors (n=1) of 0.31 and 1.1, respectively. Applying these factors to the STMR for tomatoes (0.32 mg/kg) yields STMR-Ps of 0.10 mg/kg and 0.35 mg/kg for tomato puree and tomato paste, respectively.

Samples of green beans with incurred pyrimethanil residues (Europe) were processed utilising commercial canning and freezing techniques (n=4). The median processing factor was 0.40 for canning and the median factor for freezing was 0.50. Using the freezing factor, the STMR-P for processed green (common) beans was estimated as 0.11 mg/kg (0.50 × 0.22).

Samples of carrot from four locations in Southern Europe with incurred residues of pyrimethanil were processed by commercial-type procedures into canned carrots, frozen carrots, carrot juice and carrot puree. The median processing factors (n=4) for canned carrots and frozen carrots were 0.59 and 0.45, respectively. The median processing factors (n=4) for juice and puree were 0.20 and 0.45, respectively. Using these factors, STMRs were derived for canned carrots, 0.083 mg/kg, and frozen carrots, 0.063 mg/kg; the average STMR for canned/frozen carrots, 0.073 mg/kg; carrot juice 0.028 mg/kg; and carrot puree 0.063 mg/kg. The HR for canned/frozen carrots is 0.28 mg/kg (the average of 0.59 × 0.54 mg/kg and 0.45 × 0.54 mg/kg).

Livestock dietary burden

Based on the *Table of OECD Feedstuffs Derived from Field Crops*, Annex 4, ENV/JM/MONO (2006) 32, also published as Annex 6 of the 2006 JMPR *Report*, the following feed items are potentially available: pea hay (straw), carrot culls, potato culls, pea seed, almond hulls, apple pomace (wet), citrus (dried pulp), potato (processed waste), grape pomace (wet). 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 livestock dietary burdens

Dietary burden calculations for beef cattle and dairy cattle are provided below. The calculations were made according to the animal diets from US-Canada, EU and Australia in the *Table of OECD Feedstuffs Derived from Field Crop* (Annex 6 of the 2006 JMPR Report).

Poultry metabolism, poultry analytical methods and poultry feeding studies were not provided. The manufacturers noted a lack of poultry feed items. However, the *Table of OECD Feedstuffs Derived from Field Crop* indicates several poultry feeding items that potentially contain pyrimethanil residues: carrot culls (10% Australia); pea seed (20% US, EU), pea hay (straw) (10% Europe) and potato culls (10% Europe).

Animal dietary burden, pyrimethanil, ppm of dry matter diet

	US-Canada		EU		Australia	
	max	mean	max	mean	max	mean
Beef cattle	2.42	1.90	2.49	1.70	3.52 [a]	2.76
Dairy cattle	1.69	1.18	1.76	0.93	3.52 [a]	2.86 [b]

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 and milk.

Animal commodity maximum residue levels

The Meeting received a report on the feeding of Holstein lactating cattle for 28 days with pyrimethanil. Dosing was made on a daily basis at the nominal dose rates of 1, 3, 10 and 50 ppm in the diet. The total residue (pyrimethanil + 2-(4-hydroxyanilino-4,6-dimethylpyrimidine + 2-anilino-4,6-dimethylpyrimidin-5-ol) reached a plateau in milk between day 15 and day 22 at the 50 ppm dosing level.

Residues in milk (final day 27) were below the LOQ (0.01 mg/kg per compound) at the 50 ppm dosing level for each of pyrimethanil and 2-(4-hydroxyanilino-4,6-dimethylpyrimidine. The metabolite 2-anilino-4,6-dimethylpyrimidin-5-ol had a maximum concentration of 0.088 mg/kg and an average concentration of 0.069 mg/kg in final milk from the 50 ppm dosing regimen. The same metabolite was found at a maximum concentration of 0.017 mg/kg in milk at the 10 ppm feeding level and was absent (< 0.01 mg/kg) at the 3 ppm dosing level.

A milk sample from day 27 was separated into skim milk and milk fat. The residue in skim milk consisted of 0.015 mg/kg 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine and 0.064 mg/kg 2-anilino-4,6-dimethylpyrimidin-5-ol. Milk fat contained 0.031 mg/kg 2-anilino-4,6-dimethylpyrimidin-5-ol. Thus, the residue is not fat soluble.

At the 50 ppm level, each of the parent and metabolite 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine was absent at the LOQ (0.05 mg/kg) in all tissues except kidney. Pyrimethanil was absent in kidney (at the 50 ppm feeding level). The average concentration of 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine in kidney was 0.63 mg/kg and the maximum residue was

0.88 mg/kg. At the 3 ppm feeding level, the average concentration of 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine in kidney was 0.066 mg/kg and the maximum was 0.08 mg/kg. At the 10 ppm feeding level, the average concentration of 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine in kidney was 0.12 mg/kg and the maximum was 0.13 mg/kg.

In the table below, dietary burdens are shown in round brackets (), feeding levels and residue concentrations from the feeding study are shown in square brackets [] and estimated concentrations related to the dietary burdens are shown without brackets.

Pyrimethanil total residues, [a] mg/kg

Dietary burden (ppm) Feeding level [ppm]	Milk	Muscle	Liver	Kidney	Fat
MRL					
	Mean	Highest	Highest	Highest	Highest
MRL, beef cattle (3.52) [3.0]		(< 0.1) [< 0.1]	(< 0.1) [< 0.1]	(0.09 [b] + < 0.05 [c]) [0.08 [b] + < 0.05 [c]]	(< 0.1) [< 0.1]
MRL, dairy cattle (3.52) [3.0]	(< 0.03) [< 0.03 [d]]	(< 0.1) [< 0.1]	(< 0.1) [< 0.1]	(0.09 [b] + < 0.05 [c]) [0.08 [b] + < 0.05}	(< 0.1) [< 0.1]
STMR					
	Mean	Mean	Mean	Mean	Mean
STMR beet cattle (2.76) [3.0]		(< 0.1) [< 0.1]	(< 0.1) [< 0.1]	(0.058 [b] + < 0.05 [c]) [0.066 [b] + < 0.05 [c]]	(< 0.1) [< 0.1]
STMR dairy cattle (2.86) [3.0]	(< 0.02) [< 0.02]	(< 0.1) [< 0.1]	(< 0.1) [< 0.1]	(0.060 + < 0.05 [c]) [0.066 [b] + < 0.05 [c]]	(< 0.1) [< 0.1]

a - The LOQ is 0.05 for each of pyrimethanil and 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine, in animal tissues. The LOQ is 0.01 mg/kg for each of pyrimethanil, 2-anilino-4,6-dimethylpyrimidin-5-ol, 2-anilino-4,6-dimethylpyrimidin-5-ol in milk.

b - 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine.

c - Pyrimethanil. At a 50 ppm pyrimethanil feeding level, pyrimethanil was < 0.05 mg/kg. By extrapolation, at the 3 ppm feeding level, the pyrimethanil concentration would be < 0.005 mg/kg.

d - pyrimethanil + 2-(4-hydroxyanilino-4,6-dimethylpyrimidine + 2-anilino-4,6-dimethylpyrimidin-5-ol. At a 50 ppm feeding level only 2-anilino-4,6-dimethylpyrimidin-5-ol had quantifiable residues.

The Meeting estimated an STMR of 0.01 mg/kg for milk and estimated a maximum residue level of 0.01 mg/kg for milk. The Meeting estimated STMRs of 0.0 mg/kg for each of meat and fat and maximum residue levels of 0.05 (*) mg/kg for meat. The Meeting estimated an STMR of 0.065 mg/kg for edible offal based on the STMR value for dairy cow kidney. The Meeting estimated a maximum residue level of 0.1 mg/kg for edible offal (mammalian) based on the value of kidney.

RECOMMENDATIONS

The Meeting estimated the maximum residue levels and STMR values shown below. The maximum residue levels are recommended for use as MRLs.

Definition of the residue

Plants and Animals

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

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

For milk is the sum of pyrimethanil and 2-anilino-4,6-dimethylpyrimidin-5-ol, expressed as pyrimethanil; for livestock tissues (excluding poultry) is the sum of pyrimethanil and 2-(4-hydroxyanilino)-4,6-dimethylpyrimidine, expressed as pyrimethanil.

Commodity		MRL, mg/kg		STMR or
CCN	Name	New	Previous	STMR-P, mg/kg
TN 0660	Almonds	0.2		0.05
AM 0660	Almond hulls	12		2.6
JF 0226	Apple juice	-		0.32
AB 0226	Apple pomace (dry)	40		7.2 (2.9 wet)
FS 0240	Apricot	3		1.2
FI 0327	Banana	0.1		0.05
VR 0577	Carrot	1		0.14
FS 0013	Cherries	4 Po		1.3
FC 000 1	Citrus fruits	7 Po		2.8
JF 0001	Citrus juice	-		0.028
-	Citrus oil			56
VP 0526	Common bean (pods and/or immature seeds)	3		0.22
DF 0269	Dried grapes (= currants, raisins, and sultanas)	5		1.1
MO 0105	Edible offal (mammalian)	0.1		0.065
VD 0561	Field pea (dry)	0.5		0.09
FB 0269	Grapes	4		0.71
JF 0269	Grape juice	-		0.50
VL 0482	Lettuce, Head	3		0.85
MF 0100	Mammalian fats (except milk fat)	-		0
MM 0095	Meat (from mammals other than marine mammals)	0.05 (*)		0
ML 0106	Milks	0.01		0.01
FS 0245	Nectarine	4		1.3
VA 0385	Onion, bulb	0.2		0.062
VA 0389	Onion, Spring (green)	3		0.38
AL 0072	Pea hay or pea fodder dry	3		0.20
FS 00247	Peach	4		1.3
FS 0014	Plums (including Prunes)	2		0.59
FP 000 9	Pome fruits	7 Po		0.70
VR 0589	Potato	0.05 (*)		0.05
DF 0014	Prunes	-		0.48
FB 0275	Strawberry	3		1.2
VO 0448	Tomato	0.7		0.32
-	Apple puree	-		0.26
-	Carrot, frozen/canned	-		0.073
-	Carrot juice	-		0.028
-	Carrot puree	-		0.063
-	Common beans, frozen/canned	-		0.11
-	Tomato puree	-		0.10
-	Tomato paste	-		0.35
-	Wine	-		0.34

DIETARY RISK ASSESSMENT

Long-term intake

The International Estimated Daily Intakes (IEDI) of pyrimethanil based on the STMRs estimated for 32 commodities for the thirteen GEMS/Food cluster diets were in the range of 0% to 5% of the

maximum ADI (0.2 mg/kg bw). The Meeting concluded that the long-term intake of residues of pyrimethanil resulting from its uses that have been considered by JMPR is unlikely to present a public health concern.

Short-term intake

The 2007 JMPR decided that an ARfD is unnecessary. The Meeting therefore concluded that the short-term intake of pyrimethanil residues is unlikely to present a public health concern.

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