CYROMAZINE (169)

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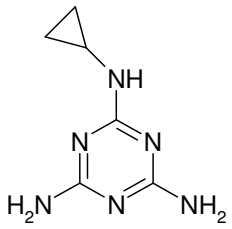
EXPLANATION

Cyromazine is a selective insecticide used on a broad range of vegetable crops. It acts by inhibiting the moulting processes, particularly in Dipteran insects. The compound was evaluated by JMPR 90 TR, 91 R, 92 R and 06T. An ADI of 0-0.06 mg/kg bw (increased from 0-0.02 mg/kg bw in 1990) and an ARfD of 0.1 mg/kg bw were established by JMPR in 2006. The compound was listed at the 38th Session of the CCPR for periodic review for residues by 2007 JMPR. The manufacturer submitted data of physical and chemical properties, metabolism in animals, plants and soils, residues in succeeding crops, analytical methods and storage stability, supervised trials on mangoes, bulb vegetables, brassica leafy vegetables, cucurbits, fruiting vegetables, mushrooms, beans, potatoes, artichokes, celery and animal commodities, and processing studies on tomatoes and potatoes. The Australian Government provided information on GAP, monitoring data on sheep kidney samples and residue definitions.

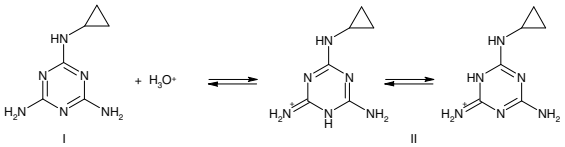
IDENTITY

ISO Common Name	cyromazine
Chemical Name	
IUPAC	N-cyclopropyl-[1,3,5]triazine-2,4,6-triamine
CAS	N-cyclopropyl-1,3,5-triazine-2,4,6-triamine
Structural Formula	
Molecular Formula	$C_6H_{10}N_6$
Molecular Weight	166.2

PHYSICAL AND CHEMICAL PROPERTIES

Property	Results	Method; test substance	References
Melting point	223.2 °C with decomposition	EEC A.1; pure 99.2%	Das (1998)
Boiling point	Oxidative decomposition starts immediately after melting	EEC A.2; pure 99.2%	Das (1999a)
Temperature of decomposition or sublimation	Decomposition starts immediately after melting	EEC A.2; pure 99.2%	Das (1999a)
	No decomposition between room temperature and 150 °C	OECD 113; technical, 97.5%	Schürch (1994)
Relative density	1.34×10^3 kg/m^3 corresponding to a relative density of 1.34	OECD 109; pure 99.2%	Füldner (1998)

Property	Results	Method; test substance	References
Vapour pressure	4.48×10^{-7} Pa, at 25 °C	EEC A.4; pure 99.3%	Rordorf (1988)
Volatility	Henry's law constant: 5.8×10^{-9} Pa · m³/mol, at 25 °C	calculation	Burkhard (1998)
Physical state and colour	pure active substance: white fine powder technical grade active substance: off-white fine powder	pure, 99.2% technical, 97.5%	Das (1999b) Das (1999c)
Odour	odourless	Pure, 99.2% Technical, 97.5%	Das (1999b) Das (1999c)
Spectra active substance	IR (KBr pellet); mass spectra (EI); ^{13}C-NMR (75 MHz, DMSO); ^{1}H-NMR (300 MHz, DMSO) UV absorption characteristics : Solution in methanol / Wavelength [nm] / molar extinction coefficient [L/mol · cm] neutral: 208.0 / 39070 acidic: 215.5 / 29540; 241.3 / 17340 basic: 230.0 / 11540 No absorption maximum between 340 nm and 750 nm	Pure, 99.2%	Käser (1998) Kettner (1999)
Solubility in water at 25 °C	Pure water: 13 g/L at pH 7.9 Buffered solutions: 8.0 g/L at pH 5.3 13 g/L at pH 7.1 13 g/L at pH 9.0	EEC A.6; pure, 99.2%	Das (2001) Rodler (1992a)
Solubility in organic solvents at 25 °C	Acetone: 1.4 g/L Hexane: < 1 mg/L ethyl acetate: 660 mg/L octanol: 1.5 g/L toluene: 11 mg/L dichloromethane: 210 mg/L methanol: 17 g/L	SOP 209/5/ CIPAC MT 157.3; technical, 97.5%	Stulz (1998)
Partition coefficient n-octanol / water (Kow) at 25 °C	0.4411 ± (0.0125) at pH 5.4 0.8536 ± (0.0180) at pH 7.0 0.9134 ± (0.0182) at pH 9.0	OECD 107/ EEC A.8 ; pure, 99.6%	Rodler (1992b)
Hydrolysis rate	Hydrolytically stable at pH 5, 7 and 9 respectively, during 28 days at 30, 50 and 70 °C	Pure, 97.2%	Burkhard (1979)
Photochemical degradation	Photolitically stable under aqueous solution at 25 °C and pH 7	EPA 540/9-82-021; pure, 96.2%	Reischmann (2000)

Property	Results	Method; test substance	References
Dissociation constant	pK_a = 5.22 at 20 °C 	OECD 112; pure, 99.6%	Burkhard (1999) Jäkel (1992)
Atmospheric oxidation by hydroxyl radical	Half life = 102 h (1.5×10^6 OH-radicals/cm³ and a 12 h day)	Atkinson R., Environ. Toxicol. Chem., 7, 435 (1988)	Stamm (1996)
Flammability	not considered highly flammable	EEC A.10; technical, 97.5%	Schürch (1992a)
Auto-flammability	no self-ignition	EEC A.16; technical, 97.5%	Schürch (1992b)
Explosive properties	not considered an explosive	EEC A.14; technical, 97.5%	Schürch (1992c)
Oxidizing properties	not considered an oxidizing substance	EEC A.17; technical, 97.5%	Schürch (1992d)
Surface tension, at 20 °C	σ = 59.0 mN/m (1.0 g/L aqueous solution) Cyromazine is a surface active substance	OECD 115/ EEC A.5 technical, 97.5%	Martin (2000)

METABOLISM AND ENVIRONMENTAL FATE

The structure of the labelled cyromazine used in the metabolism and environmental studies is shown in Figure 1. The metabolites and degradation products found are shown in Table 1.

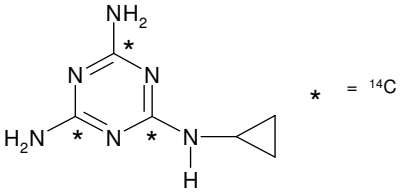
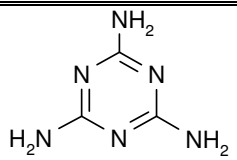
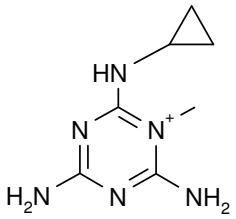
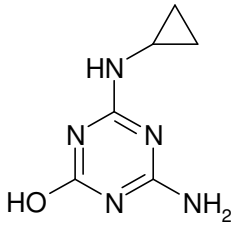
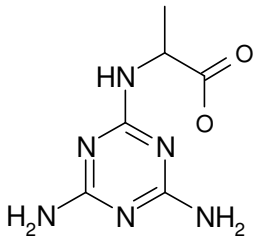


Figure 1. [triazine- U-^{14}C] cyromazine

Table 1. Cyromazine metabolites and degradation products

Name	Isolated from	Structure
[1,3,5]triazin-2,4,6-triamine (melamine)	Celery, lettuce, tomato, mushrooms, carrot, rat, sheep, goat, hen, eggs, cow, milk, liver, rotational celery, soil laboratory, soil field, aquatic	
1-Methyl-cyromazine (2,4-diamino-6-cyclopropylamino-1-methyl-[1,3,5]triazin-1-ium salt)	Rat, goat	
4-amino-6-cyclopropylamino-[1,3,5]triazin-2-ol (Hydroxy-cyromazine)	Rat, goat	
N-(4,6-diamino-[1,3,5]triazin-2-yl)-alanine (NOA 435343)	Soil laboratory	

METABOLISM

Animal metabolism

Metabolism studies in rats were submitted and evaluated by the 2006 JMPR within the Periodic Review programme for toxicology. Administered [^{14}C]-cyromazine as single and repeated oral doses showed that the compound is rapidly and almost completely absorbed from the gastrointestinal tract and distributed to all organs and tissues. More than 97% of the administered dose was excreted within 24 h, almost exclusively in the urine. Cyromazine was incompletely metabolized, essentially by methylation, hydroxylation or N-dealkylation. Cyromazine accounted for 71.5% of the radiolabel in urine; a further 7% was attributable to melamine and 8 – 11% to hydroxy-cyromazine and methylcyromazine.

The metabolic fate of cyromazine was investigated in laying hens using [^{14}C]-cyromazine (Simoneaux and Cassidy, 1979). After a three day acclimation period, two laying hens received doses of the test material via gelatine capsules at 5.0 ppm in the feed (equivalent to 0.5 mg/kg body weight/day) for seven consecutive days. Eggs and excreta were collected daily, while volatiles and CO_2 were trapped over 24 h periods. The birds were sacrificed 24 h after the last dose and samples collected included those of lean meat (breasts, thighs), fat, kidneys, hearts and livers. Tissues and excreta were homogenized in the presence of dry ice and aliquots combusted to $^{14}CO_2$ followed by LSC to determine the total radioactive residue (TRR). Sub-samples of egg white and egg yolk were extracted with methanol/water and excreta were extracted with acetonitrile/water, both under reflux for two hours. Extracts were cleaned-up by TLC, Sephadex A-25 GPC and cation exchange chromatography. Determination of radioactivity was by TLC with zone scraping followed by LSC.

On average, 99.8% of the applied radioactivity was recovered in the experiment, mostly in the excreta (99.1%). Egg whites and egg yolks accounted for 0.4% and 0.2% of the total applied dose, respectively, tissue residues for 0.1% and expired CO_2 and other volatiles for < 0.1%. Table 2 shows the radioactivity found in eggs during the experiment period.

Table 2. Recovery of radioactivity in eggs from laying hens dosed with [triazine-U-14C]-cyromazine at 5.0 ppm in the diet for seven consecutive days

	Hen 1				Hen 2			
	Egg white		Egg yolk		Egg white		Egg yolk	
Day	% daily dose	mg/kg [a]	% daily dose	mg/kg [a]	% daily dose	mg/kg [a]	% daily dose	mg/kg [a]
1	0.76	0.19	0.17	0.08	No egg		No egg	
2	No egg		No egg		0.51	0.14	0.16	0.09
3	0.48	0.12	0.21	0.11	0.86	0.22	0.28	0.15
4	0.71	0.15	0.27	0.12	0.68	0.16	0.31	0.15
5	0.35	0.09	0.22	0.11	No egg		No egg	
6	0.41	0.10	0.23	0.11	0.59	0.15	0.29	0.14
7	0.63	0.16	0.30	0.15	0.56	0.13	0.32	0.15

a - As cyromazine equivalents

Residues in tissues for both hens are given in Table 3, with the highest levels found in livers (mean of 0.32 mg/kg cyromazine eq). The radioactivity in tissues was not characterized.

Table 3. Residues in tissues of laying hens dosed with [^{14}C]-cyromazine

Tissue	Residues in mg/kg of cyromazine eq	
	Hen 1	Hen 2
Liver	0.026	0.037
Kidney	0.017	0.021
Heart	0.011	0.009
Breast Muscle	0.010	0.010
Thigh Muscle	0.008	0.009
Thigh Skin	< 0.007	0.010
Breast Skin	< 0.007	0.008
Internal Fat	< 0.002	< 0.002

The balance and characterization of radioactivity in eggs from day seven is presented in Table 4. Cyromazine represented about 64% TRR and an uncharacterized metabolite (peak A) had the same retention volume after ion-exchange column of a melamine standard, but no further characterization was carried out in this study.

Table 4. Balance and characterization of egg residues (from day 7)

	Egg White (mean)		Egg Yolk (mean)	
	% TRR	mg/kg [a]	% TRR	mg/kg [a]
Extracted	108.6	-	100.2	-
Cyromazine	58.1	0.09	69.6	0.08
Peak A	24.5	0.04	6.7	0.01
Minor peaks	26.0	-	23.9	-
Non-extracted	1.1	-	1.8	-
Total	109.7	-	102.0	-

a - As cyromazine equivalents

Two metabolism studies conducted in lactating goats fed with [^{14}C]-cyromazine were submitted. In the first study (Simoneaux and Marco 1984), two animals were conditioned to cages for four days and fed with radioactive cyromazine in gelatine capsules for ten consecutive days at levels of 4.6 ppm (low rate) and 48.4 ppm (high rate) in the feed. Expired gases/volatiles from the high dose

animal were trapped and collected daily, together with urine and faeces. The animals were sacrificed 24 h after the last dosing and tissue samples were taken. Faeces and tissue samples were homogenised and combusted, with the resulting $^{14}CO_2$ determined by LSC. Radioactivity in urine and volatiles was assayed directly by LSC. Faeces (day seven) and liver samples were extracted by reflux with acetonitrile/water (4 h). Milk was fractionated into fat, casein and whey; the whey being partially cleaned-up on a cation exchange resin, as were the extracts of faeces and livers. The partially purified liver extract samples were analysed by TLC and radioactivity determined by zone scraping and LSC. The partially purified milk whey was analysed by cation exchange chromatography.

Total recovery of radioactivity was 102 and 90.4% of the applied dose from the low and higher dosed animals, respectively, with most of the administered dose excreted in the urine (90.4 and 82.1%) and faeces (7.5 and 5.7%). Residues in tissues represented < 2% of the applied doses, mostly found in livers and kidneys (Table 5).

Table 5. Radioactivity in tissues from goats fed cyromazine in the diet (Simoneaux and Marco 1984)

Tissue	4.6 ppm in the feed		48.4 ppm in the feed	
	% applied dose	mg/kg cyromazine eq.	% applied dose	mg/kg cyromazine eq.
Liver	1.09	0.791	0.15	1.522
Kidney	0.01	0.043	0.01	0.437
Tenderloin	0.01	0.009	0.01	0.104
Leg muscle	0.01	0.010	0.01	0.142
Omental fat	< 0.01	0.004	< 0.01	0.029
Skeletal fat	< 0.01	0.005	< 0.01	0.062
Brain	< 0.01	0.008	< 0.01	0.118
Heart	< 0.01	0.010	< 0.01	0.129

Radioactivity in milk accounted for < 0.4% of the applied doses and plateaued rapidly at both dose levels (Table 6).

Table 6. Radioactivity in milk of goats fed cyromazine in the diet (Simoneaux and Marco, 1984)

Day	4.5 ppm in the feed		48.4 ppm in the feed	
	% applied dose	mg/kg cyromazine eq	% applied dose	mg/kg cyromazine eq
1	0.03	0.020	0.03	0.330
2	0.03	0.017	0.03	0.321
3	0.03	0.019	0.04	0.354
4	0.03	0.017	0.04	0.374
5	0.03	0.016	0.04	0.338
6	0.03	0.016	0.04	0.247
7	0.03	0.017	0.04	0.296
8	0.03	0.017	0.04	0.310
9	0.03	0.017	0.04	0.306
10	0.03	0.017	0.04	0.306
Mean		0.017		0.318

The radioactivity in the day-7 milk was mostly associated with the whey fraction. No radioactive residues were detected in casein and fat/milk fractions. Most of radioactive residues were extracted from livers and milk whey (Table 7). In milk whey, cyromazine was the major radioactive component while in livers cyromazine corresponded to only 0.2% TRR with zone-4 being the predominant metabolite (not identified).

Table 7. Characterization residues in liver and milk from goats fed cyromazine in % TRR (Simoneaux and Marco, 1984)

	4.5 ppm in the feed		48.4 ppm in the feed	
Fraction	Liver	Milk whey	Liver	Milk whey
Extracted	94.0	74.4	92.0	71.1
Cyromazine	0.2	32.5	1.9	41.0
Melamine	1.7	9.2	5.6	4.5
Zone-4	92.7	1.0	71.1	0.2
Non-extracted	6.8	25.6	10.6	28.9

Tortora (1991) studied the metabolism of cyromazine in goats (Alpine and Nubian) dosed daily with [^{14}C]-cyromazine by gelatine capsules at 150 mg/capsule/day for four consecutive days, following a six day acclimation period, corresponding to approximately 100 mg/kg in the diet, respectively. Urine and faeces were sampled daily and milk collected twice daily, including during the acclimation period. The animals were sacrificed approximately 6 h after the last dose and tissue samples were taken for analysis. The total radioactive residue (TRR) was determined by combustion, followed by trapping of $^{14}CO_2$ and LSC. Milk and urine were assayed by direct counting. Samples were diluted or extracted with acetonitrile or acetonitrile/water. Fat was dissolved in hexane, partitioned with acetonitrile and cleaned-up on a C-18 SPE cartridge. Determination of radioactivity was by HPLC/LSC. Metabolite identification was achieved through co-chromatography with authentic standards using 2-dimensional TLC and HPLC. GC/MSD or HPLC/MS confirmed the structures.

Overall average recovery of radioactivity was 73.9% of the applied dose, with 2.7 and 59% being eliminated via faeces and urine, respectively; 7.4% of the total dose was found in the gastrointestinal tract. On average, 5.9% of the applied dose was recovered in the tissues, mostly in muscle (Table 8).

Table 8. Recovery of radioactivity from goats dosed with [^{14}C]-cyromazine

Tissue	% applied dose (goat 1/goat 2)	Mean, mg/kg cyromazine eq
Tenderloin	3.14 / 2.18	0.866
Leg muscle	2.91 / 2.40	0.879
Heart	0.03 / 0.03	0.893
Liver	0.43 / 0.37	2.703
Kidney	0.11 / 0.08	4.588
Omental fat	0.02 / 0.04	0.102
Perirenal fat	0.01 / 0.004	0.164

On average, a total of 0.76% of the applied dose was recovered in the milk, with an average of 0.656 mg/kg cyromazine eq/day. The daily milking values indicated that a plateau residue level was reached during the first day of dosing and remained constant during the dosing period (Table 9).

Table 9. Radioactivity in milk from dosed goat

	PM milk, goat 1 / goat 2		AM milk, goat 1 / goat 2		AM and PM
Day	% applied dose	Mean, mg/kg cyromazine eq	% applied dose	Mean, mg/kg cyromazine eq	Mean, mg/kg cyromazine eq
1	0.14 / 0.08	1.07	0.04 / 0.03	0.225	0.650
2	0.16 / 0.08	1.15	0.03 / 0.02	0.164	0.654
3	0.16 /0.08	1.14	0.03 / 0.023	0.170	0.665
4	0.12 / 0.07	1.28	No sample	No sample	-
Total	0.58 / 0.32	-	0.11 / 0.08	-	-

From 85 to 118% TRR was recovered in the acetonitrile extract of milk and tissues—the identities of the compounds are given in Table 10. Cyromazine was the major identified compound in milk, followed by the metabolite, melamine. In livers, the metabolite 1-methylcyromazine was the major compound found, followed by the parent cyromazine. Only cyromazine was detected in fat. The non-extracted radioactivity in the livers (14.4% TRR) was re-extracted with aqueous acetonitrile, acetone, water and methanol. This combined extract yielded three chromatographic peaks, one being identified as 1-methylcyromazine and a second hydroxy-cyromazine (up to 0.167 mg/kg).

Table 10. Characterization of the extractable radioactivity in milk, and tissues of dosed goats

Tissue / Substrate	Characterization of Radioactivity					
	Cyromazine		Melamine		1-methylcyromazine	
	% TRR	mg/kg [c]	% TRR	mg/kg [c]	% TRR	mg/kg [c]
Milk [a, b]	67.7	0.66	2.9	0.028	< 0.02	< 0.02
Liver [b]	33.6	0.93	5.9	0.16	41.9	1.16
Kidney	70.2	3.78	23.0	1.24	5.86	0.31
Tenderloin	79.8	0.79	3.2	0.03	3.6	0.03
Omental fat	50.4	0.11	< 0.02	< 0.02	ND	ND

a - Averaged values for day 1 and day 4 samples

b - Averaged values for goat 1 and goat 2

c - Expressed as cyromazine eq ; ND = Not detected

In one study conducted by Simoneaux and Cassidy (1981), a mature female sheep was fed with [^{14}C]-cyromazine in gelatine capsules for nine consecutive days at a level of 5 ppm in the diet (equivalent to 0.15 mg/kg bw/day). Urine, faeces, volatiles and CO_2 were collected daily and tissue samples were taken at the time of sacrifice. Total radioactivity in tissues and faeces was determined after combustion; urine and washings of the volatile trap were radioassayed directly. Samples of livers and faeces were extracted by refluxing for 2 h with acetonitrile/water (9:1) and filtered extracts spotted on a silica gel TLC plate; urine aliquots were applied directly on the TLC plate. After visualisation under UV light, radioactivity that co-chromatographed with standards was determine after plate scraping and direct scintillation counting. Liver extracts were further cleaned-up on Sephadex columns. All extracts and urine samples were then submitted to ion-exchange chromatography.

Recovery of administered radioactivity was 93.4%, with urine containing 89.3% and faeces 3.6% of the applied doses. Small amounts were found in tissues (0.09%) and the gastro-intestinal tracts (0.28%). In tissues higher radioactivity levels were found in the livers and the kidneys (Table 11).

Table 11. Radioactivity in tissues from a sheep fed cyromazine in the diet for nine days

Tissue	% of applied dose	mg/kg cyromazine eq
Liver	0.06	0.174
Kidney	0.01	0.048
Leg muscle	0.01	0.013
Tenderloin	0.01	0.012
Brain	< 0.005	0.012
Heart	< 0.005	0.013
Omental fat	< 0.005	< 0.003
Back fat	< 0.005	< 0.010
Total	0.09	0.272

The characterization of the urine, faeces and liver acetonitrile extracts are shown in Table 12. Urine and faeces contained mostly cyromazine while the main metabolite found in liver extracts was melamine, characterized by TLC and co-chromatography with authentic standards, cation exchange chromatography and inverse isotope dilution analysis with non-radiolabelled melamine. Metabolites

a, b and c were not identified (Table 12). Over 40% of the residues in faeces and livers were not extracted.

Table 12. Extraction and characterization of liver and Day nine urine and faeces from a sheep fed [^{14}C]-cyromazine in the diet for nine consecutive days

	Proportion of Total Radioactivity (%)		
Fraction	urine	faeces	liver
Extracted	100	53.8	67.0
Cyromazine	84.0	44.0	11.6
Melamine	0.1	0.3	43.0
Metabolite a	2.2	1.0	1.3
Metabolite b	6.8	1.3	1.5
Metabolite c	3.6	6.4	2.9
Remainder	3.3	0.8	6.7
Non-extracted	-	44.4	32.3

The proposed metabolic pathway of cyromazine in rats, hens, goats and sheep is presented in Figure 2.

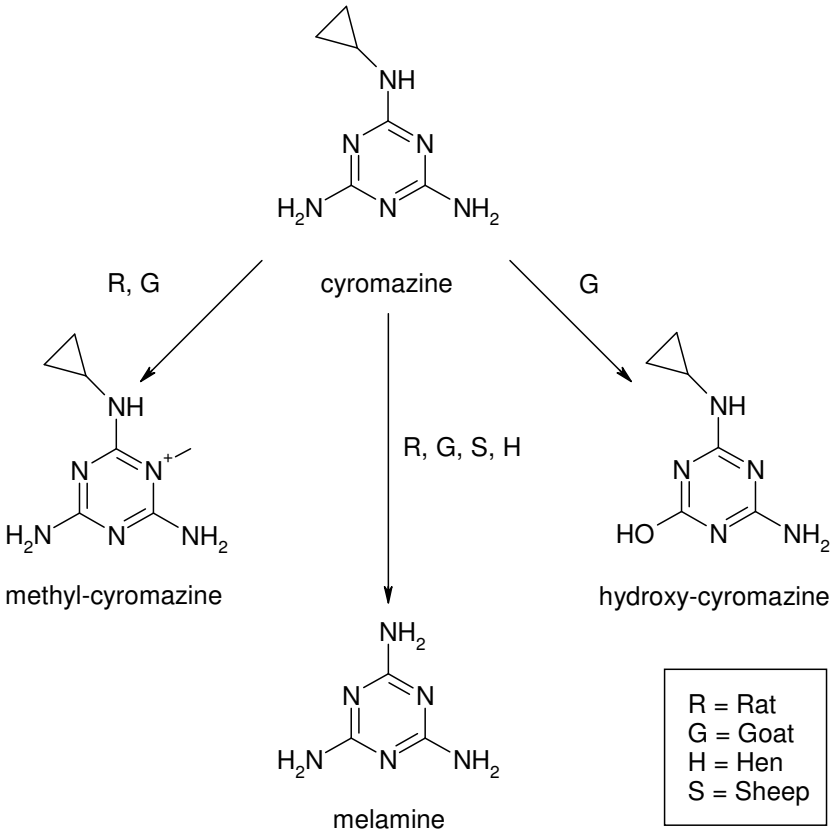


Figure 2. Proposed pathway of cyromazine metabolism in animals

Plant metabolism

Plant uptake, distribution and metabolism of cyromazine were studied in greenhouse-grown celery and lettuce plants and reported by Simoneaux and Marco (1983a). In the first phase of this study, half-mature plants received two foliar applications of [^{14}C]-cyromazine, with a seven day interval (total 0.42 kg ai/ha). The crops were sampled at maturity seven days after the second application. In the second phase of the study, celery plants received six foliar applications (0.28 kg ai/ha/application) at

two weekly intervals starting shortly after transplanting and plants were sampled as immature stalks seven days after the third application and as mature stalks 14 days after the sixth application. Lettuce received four foliar applications at 0.28 kg ai/ha/application at two weekly intervals and immature heads were sampled seven days after the second application and seven days after the fourth application (crop maturity). The total radioactive residue (TRR) was determined by combustion to CO_2 followed by liquid scintillation counting (LSC). Samples were extracted using a biphasic (Bligh-Dyer) extraction technique that generated an organic fraction, an aqueous fraction, and a non-extracted residue. The crop extracts were analysed by TLC with characterization by co-chromatography against authentic standards and quantification by zone scraping and LSC.

Radioactivity in the plants was mostly extracted in the aqueous phase (Table 13). Characterization of the aqueous extracts showed cyromazine as the major residues (representing 48 to 74% TRR in both plants) and melamine was the only metabolite found. Non-extracted radioactivity represented 4.5 – 6.5% TRR for the immature and mature harvested crops. No further work was carried out on these residues.

Table 13. Radioactivity in celery and lettuce following foliar applications of [triazine-U-^{14}C]-cyromazine (Simoneaux and Marco 1983a)

Days [a]	cyromazine mg/kg eq.	Balance, % TRR				Characterization (aqueous phase),% TRR		
		Un-extracted	Organic phase	Aqueous Phase	Total	Cyromazine	Melamine	Total
Celery: 2 applications, total 420 g ai/ha (Phase 1)								
71	1.46	6.1	10.5	90.5	107.1	56.0	32.9	88.9
Celery: 3 applications, total 840 g ai/ha (Phase 2)								
36	5.84	6.0	8.6	79.7	94.3	63.9	15.7	79.6
Celery: 6 applications, total 1680 g ai/ha (Phase 2)								
85	1.55	6.5	5.9	74.5	86.9	48.2	25.4	73.6
Lettuce: 2 foliar applications, total 420 g ai/ha (Phase 1)								
48	2.55	6.2	9.3	77.6	93.1	56.0	16.4	70.4
Lettuce: 2 foliar applications, total 560 g ai/ha (Phase 2)								
22	4.05	5.5	9.5	88.9	103.9	73.5	12.3	85.8
Lettuce: 2 foliar applications, total 1120 g ai/ha (Phase 2)								
50	3.69	4.5	11.3	88.9	104.7	74.0	10.9	84.9

a - Days after transplanting

The metabolism of [^{14}C]-cyromazine in tomatoes grown in a field plot in California was investigated (Simoneaux, 1984). Six foliar applications at 280 g ai/ha were made to the crop at two week intervals and tomato samples taken at 0, 7, and 14 days after the fourth or sixth applications. Tomato stalks (aerial portion of the plant after removal of the tomato fruits) were also sampled 14 days after the sixth application. The total radioactive residue (TRR) was determined by combustion to $^{14}CO_2$ followed by liquid scintillation counting (LSC). Tomato fruit and stalk samples were extracted using a biphasic (Bligh-Dyer) extraction technique. Characterization of extractable radioactivity in plant samples was by ion-exchange chromatography of a methanol/water extract.

TRR was low in samples harvested after the fourth application and ranged from 64 – 89% of the applied radioactivity, but was > 90% in tomato and stalk samples harvested after 7 to 14 days of the sixth application (Table 14). Tomato stalks taken 14 days after the sixth application contained significantly higher residues. The radioactivity in tomato fruit and stalks was mostly extracted, with 56 to 87% TRR found in the aqueous phase. Cyromazine and melamine represented on average 38 and 35% TRR in the fruit 14 days after the 4th or the 6th application.

Table 14. Uptake, balance, and characterization of radioactivity in tomatoes following four or six foliar applications of [^{14}C]-cyromazine at 280 g ai/ha/application (Simoneaux 1984)

Days [a]	cyromazine mg/kg eq	Balance,% TRR				Characterization,% TRR		
		Un-extracted	Organic phase	Aqueous Phase	Total	Cyromazine	Melamine	Total
4 applications								
0	0.19	2.9	19.8	66.1	88.8	76.4	10.9	82.1
7	0.08	4.0	4.1	55.9	64.0	41.2	21.6	79.3
14	0.12	2.0	<14.2	76.4	78.4	38.9	25.8	79.9
6 applications								
0	0.15	4.9	13.8	73.5	92.2	-	-	-
7	0.44	4.9	8.9	80.5	94.2	-	-	-
14	0.37	3.8	7.0	87.4	98.3	37.1	43.5	90.5
14 (stalks)	36.61	8.4	9.2	74.7	92.3	29.3	33.7	74.7

a - after the last application

Five trials were conducted on mushrooms in the United States in which a 5% SC formulation of cyromazine was applied to wet compost at 5 mg ai/kg or 10 mg ai/kg. The treated compost was then inoculated and a casing layered over it (Ballantine, 1984b). Mushrooms were harvested from the resulting 1 – 6 flushes for analysis. Cyromazine residues were < 0.05 mg/kg in mushrooms in all trials/flushes with the exception of one trial at a target cyromazine concentration in the compost of 10 mg/kg for which the sample from the third flush contained a residue of 0.08 mg/kg. Residues of melamine, identified as the major metabolite of cyromazine in plants, were in the range of 1.5 – 6.6 mg/kg at a target cyromazine concentration in the compost of 5 mg ai/kg, and in the range of 3.1 – 17.4 mg/kg at a target cyromazine concentration in the compost of 10 mg ai/kg. Residues of cyromazine in the compost were in the range of 2.2 – 20 mg/kg at a PHI of 0 – 1 days, 0.70 mg/kg at a PHI of 17 days and 0.48 – 1.0 at a PHI of 67 days when applied at a target of 5 mg ai/kg. At the 10 mg ai/kg rate they were in the range of 7.4 – 42 mg/kg at 0 – 1 day PHI, 1.2 mg/kg at 17 day PHI and 0.6 mg/kg at 61 days PHI. Melamine residues increased to maxima at 17 days PHI of 8 mg/kg and 14 mg/kg at 5 mg ai/kg and 10 mg ai/kg rates respectively and then declined to levels of 1.4 – 2.3 mg/kg at 67 days PHI.

Environmental fate in soil

The aerobic degradation of [^{14}C]-cyromazine was investigated by Cargile (1986) in a Californian sandy loam and Florida sand soils incubated at laboratory ambient temperatures (17 – 25 °C) in the dark. The test substance was applied to the soil at a concentration of 11 mg ai/kg soil corresponding to an exaggerated field rate of 9.5 kg ai/ha, assuming an average bulk density of the soil of 1.3 g/mL and a distribution in the top five cm of the soil. The fortified soil was incubated in Erlenmeyer flasks connected to a metabolism apparatus allowing collection of volatiles with a continuous airflow. Sterile samples were incubated under the same conditions, but without air flushing. Duplicate 10 g soil aliquots were sampled (nine sampling days) up to 12 months for analysis by combustion, extraction with acetonitrile/water (90/20 v/v) and acetone, and TLC-analysis. Details of the soil parameters are given in Table 15.

Table 15. Soil characteristics for Cargile (1986) study

Origin of soil:	Florida, sand	California, sandy loam
pH (KCl)	6.5	6.7
Organic matter (OM) (%)	31.0	0.7
Cation exchange capacity (CEC) (meq/100 g soil)	24.1	7.3
Field capacity (%)	86.36	17.74
Particle size (%): Clay / Silt / Sand	2.6 / 7.4 / 90.0	14.4 / 29.6 / 56.0

The overall balance of the applied radioactivity comprising the soil extracts, non-extracted residues and volatile products decreased in both soils with incubation time from about 90% at day 0 to 61 and 73% at the end of the study for the sandy and sandy loam soil, respectively. The major degradation product was melamine, which represented a maximum of 4.1% of the applied radioactivity on the sand soil after three months of incubation and 31.1% of on day 31 of Californian sandy loam soil. Non-extracted residues reached a maximum of 62.6% of the applied radioactivity on day 31 in Florida sandy soil. Calculated DT_{50}s were 107 days for the sand soil and 142 for the sandy loam soil. Sterile incubations showed < 3% of the applied radioactivity as melamine.

A series of degradation studies with [U-^{14}C] triazine labelled cyromazine were performed by Plücken (1986a and b), Plücken (1988a and b) in two BBA standard soils (Neuhofen 2.2, Hatzenbühl 2.3, stored under artificial conditions for at least four years) and in two Swiss soils (Mosimann, Pappelacker, sampled immediately before starting the experiment). The characteristics of the soils used are described in Table 16. Volatiles were not trapped in this study. The soils (100 g aliquots) were dosed with 2.07 mg ai/kg and incubated at 25 °C in the dark at 40% MWC. Sampling was performed at days 0, 14, 19, 28, 49, 84, 126 and 203.

Table 16. Soil characteristics (Plücken 1986a and b)

Origin of soil:	Mosimann Switzerland	Pappelacker Switzerland	BBA 2.2 Neuhofen	BBA 2.3 Hatzenbühl
Classification (USDA)	Sandy loam	Silt loam	Loamy sand	Loamy sand
Organic carbon content,%	1.9	1.5	2.6	1.1
pH	7.3	7.4	6.0	7.0
Microbial biomass, mg/100 g dry soil	101.4	112.4	24.1	35.7

The data are presented in Table 17 and demonstrate melamine to be the primary degradation product. The amount of non-extractable radioactivity increased in all four soils with incubation time to between 35% and 58% of applied radioactivity. The total recovery decreased to 71% in the Swiss soils and to 88% in the BBA standard soils during the incubation period of 203 days. The unrecovered radioactivity was postulated to be losses of $^{14}CO_2$ reflecting the greater microbial activity of the freshly collected Swiss soils.

Table 17 Dissipation of ^{14}C- cyromazine in BBA soil experiments (Plücken 1986a and b)

Days after application	Soil extraction (%)	Cyromazine (%)	Melamine (%)	Unknown (%)	Non-extractable (%)	Total (%)
Mosimann soil						
0	95.2	89.4	5.8	1.2	2.3	98.7
14	73.2	2.3	68.5	0	20.4	93.6
19	71.2	0	71.2	2.4	25.3	98.9
28	67.7	0	67.7	0	26.1	93.8
49	58.9	0	58.9	0	29.8	88.7
84	49.9	0	49.9	0	35.1	85
126	43.8	0	43.8	0	34.9	78.7
203	35.3	0	35.5	0	36.3	71.6
Pappelacker soil						
0	95.4	89.5	4.5	1.2	2.1	98.7
14	75.2	9.2	66	1.4	20.3	96.9
19	72.6	1.6	71	0	23.3	95.9
28	66.9	0	66.9	0	24.5	91.4
49	64.1	0	64.1	0	27.3	91.4
84	56	0	56	0	30.8	86.8
126	48.5	0	48.5	0	35.5	84
203	36.1	0	36.1	0	35.3	71.4

Days after application	Soil extraction (%)	Cyromazine (%)	Melamine (%)	Unknown (%)	Non-extractable (%)	Total (%)
BBA-2.2 soil, Neuhofen						
0	93.4	89.5	7	1	6.7	101.1
14	71.6	66.3	5.3	0	26.3	97.9
28	57.8	52.6	5.2	0	37.9	95.7
49	50.8	41.1	9.7	0	43.3	94.1
84	43.4	29.4	21.9	0	51.5	94.9
126	41.4	24.6	16.8	0	53.9	95.3
203	30.7	11.4	19.3	0	57.8	88.5
BBA-2.3 soil, Hatzenbühl						
0	93.7	91.9	3.5	1.2	2.5	97.4
14	83.6	74.5	9.1	1.2	14.1	98.9
28	76.2	61.6	14.6	0	20.2	96.4
49	72.2	46.8	25.4	0	23.4	95.6
84	60	25.2	34.8	0	30.5	90.5
126	57.4	15.3	42.1	0	32.1	89.5
203	49.7	5.4	44.3	0	37.9	87.6

Esser (1995) investigated the degradation of ^{14}C cyromazine in a Californian sandy loam soil (pH = 6.7, OM = 0.32%, CEC= 4.8 meq/100 g). The soil (50 g aliquots) was dosed with 9.0 mg ai/kg and incubated in Erlenmeyer flasks in the dark at 25 °C, at 75% field moisture capacity at 1/3 bar. The flasks were periodically flushed with air to remove and trap volatiles. Duplicate samples were analysed at time points up to 12 months by combustion and extraction with acetonitrile/water 9:1 and subsequent harsh extraction with dimethylformamide/0.1 N oxalic acid 1/1 (v/v), followed by TLC and HPLC analysis. The soil was shown to be microbially viable throughout the study.

Cyromazine was degraded almost completely over the course of the study (1.9% remaining after one year). The major degradate was melamine that reached 60.2% of the dose by the end of the study. An additional metabolite was formed in an amount of up to 14.7% of the applied dose after one year, being identified by HPLC/MS as the carboxylic acid of cyromazine formed through oxidative opening of the cyclopropane ring. The calculated pseudo-first order half life for cyromazine was 61.8 days. The material balance was 97.4 ± 6.2% of the applied dose through the study and soil bound radioactivity did not exceed 10% of applied radioactivity. Volatiles accounted for up to 14.6% of the applied radioactivity after one year, with 14.3% characterized as carbon dioxide.

The rate of degradation of [^{14}C]-cyromazine was determined by Glänzel (2000) in a Gartenacker loam/silt loam soil (2.11% OM; 14.29 meq/100 g CEC), at both 20 °C and 10 °C. Samples of 75 g of soil (dry weight) at 40% soil moisture were treated at a rate of 0.25 mg ai/kg (0.33 kg ai/ha field rate) and incubated in the dark for up to 182 days in a glass flow-through incubation system. Solvent traps for volatile components were included in the exhaust gas stream. Soil samples were extracted with acetonitrile/water 8/2 (v/v) at room temperature and with acetonitrile under reflux, followed by harsh extraction with both hot (80 °C) acetonitrile/water 4/1 (v/v) and acetonitrile/hydrochloric acid 9/1. Cyromazine and its metabolites were determined in extracts by TLC and HPLC. Residual radioactivity in the soils after extraction was quantified by combustion and LSC. After the harsh extraction, day 120 samples were mixed with 0.5 N NaOH and shaken for 17 h at room temperature to fractionate the soil organic matter into insoluble humin, humic acid and fulvic acid.

Total radioactive recoveries were between 92 and 101% of total applied radioactivity at both temperatures. Melamine was the only major degradation product extracted, which reached maximum amounts of 73% and 81% of the applied dose after 15 days incubation at 20 °C and 10 °C, respectively. About 32% was evolved as CO_2 by day 120 at 20 °C and 13.7% by day 182 at 10 °C. The non-extracted radioactivity amounted to < 20% of the applied radioactivity, with humic acid

being the main fraction at 20 °C (11.4%) and fulvic acid the main fraction at 10 °C (7.8%). DT_{50} and DT_{90} for cyromazine and melamine at both experiments are shown in Table 18.

Table 18. Rates of decay of cyromazine and melamine in Gartenacker soil (Glänzel, 2000)

	Cyromazine		Melamine	
Incubation conditions	20 °C	10 °C	20 °C	10 °C
DT_{50} (days)	2.9	5.6	125	> 1 year [a]
DT_{90} (days)	9.6	18.5	> 1 year [a]	-

a - Extrapolated values

The degradation of cyromazine and melamine was investigated in the laboratory in three soils (Table 19) at 0.5 mg ai/kg soil (Deener *et al.* 2003). Duplicate 50 g (dry weight) soil samples were incubated at 20 °C in the dark and at a moisture content of 28.2% or 23% (Westmaas soil). Samples (duplicate) were collected immediately after application and at 11 additional sampling dates during the incubation period of 121 days for cyromazine and 178 days (for Westmaas soil 206 days) for melamine. Samples were extracted with acetonitrile/0.05 M ammonium carbonate (9:1). The extracts were cleaned up in a cation exchange resin and analysed by HPLC/UV at 214 nm.

Table 19. Properties of the soils used in the degradation study for cyromazine and melamine (Deener *et al.* 2003)

Soil	Horst loamy sand	Westmaas loam	Naaldwijk loamy sand
Clay / silt (%)	3.8 / 16.6	22.5 / 41.1	7.7 / 10.8
$CaCO_3$	< 0.1	5.1	0.4
Organic matter / organic carbon (%)	1.4 / 1.9	2.5 / 1.5	2.7 / 1.8
pH ($CaCl_2$)	5.7	7.5	6.8
Cation exchange capacity (meq/kg)	98	178	134
Respiration (mg CO_2/day/kg) at day 0 after 4 months after 6 months	 5.5 3.8 - 2.4[a]	 20.1 18.6 13.7	 7.2 6.6 6.2

a - unreliable value when compared to blank

DT_{50} and DT_{90} were calculated using regression of the remaining mass of the test substance versus time (Table 20). While the degradation of cyromazine in Horst and Naaldwijk soils are comparable, it occurs much faster in Westmass soil, with a DT_{50} of 15 days. Degradation of melamine in soil from Horst was faster than in the other two soils.

Table 20 Calculated DT_{50} and DT_{90} for cyromazine and melanine in soil (Deener *et al*. 2003)

Parameter	Horst		Westmaas		Naaldwijk	
	Cyromazine	Melamine[b]	Cyromazine	Melamine[b]	Cyromazine	Melamine[b]
Coefic. determ.	0.969	0.852	0.992	0.825	0.990	0.812
DT_{50} (days)	46	135	15	194	56	217
DT_{90} (days)	153[a]	449[a]	50	645[a]	186[a]	721[a]

a - extrapolated values;

b - based on the first 80 days after application as no further transformation of melamine was observed

The degradation of [^{14}C]-cyromazine at 20 ± 2 °C was studied in two soils (Adam 2003), with the properties shown in Table 18. Each soil was treated at a rate of 0.44 mg/kg soil corresponding to a field rate of 0.33 kg ai/ha. The treated soil samples were incubated for 120 days under aerobic conditions in the dark at a soil moisture content of 40% of the maximum water holding capacity (Marsillargues) or at a water potential of the soil of pF 2 (18 Acres).

Duplicate soil samples were harvested at 0, 2, 7, 15, 28, 56, 86 and 120 days after treatment and submitted to extraction with acetonitrile and acetonitrile/water (80/20 v/v), followed by extraction with acetonitrile under reflux. The combined extracts were analysed by TLC and HPLC. In addition, a harsh extraction with all soil samples was performed and the radioactivity in the extracts determined. Residual radioactivity remaining in the soil after the last extraction step was determined by combustion and LSC.

A total radioactivity balance and a ^{14}C distribution were established for each time point. The overall radioactivity recovery comprising the soil extracts, non-extracted residues and volatile products, was between 100.6% and 109.2% of the total applied radioactivity. Carbon dioxide increased as the study progressed and amounted to about 7.3% at day 120. Negligible amounts (< 0.1%) of organic volatiles were generated. Non-extracted or bound residues reached up to 7.3% in the Mairsillarques soil and 25.0% in soil 18 Acres at the end of the study. Subsequent fractionation of the bound residues by the soil organic matter fractionation method (day 120) showed that 2.7 – 6.0% of the applied radioactivity was associated with fulvic acid and up to 1% with humic acid. From 3.8 to 18.9% of the radioactivity was associated with the insoluble humic fraction.

Melamine was the major degradation product found in the soils, in addition to two minor degradates detected at amounts ≤ 1.4% of the applied radioactivity. The calculated half lives (DT_{50}) and DT_{90} are also shown in Table 21.

Table 21. Properties of the soils used in the degradation study for cyromazine

Soil	Marsillargues	18 Acres
Classification (origin)	silty clay loam (France)	sandy clay loam (UK)
pH (KCl) / pH (H_2O)	-- / 8.1	5.6 / -
$CaCO_3$ (%)	45.5	0.2
Organic carbon (%)	0.5	3.3
CEC (meq / 100 g soil)	9.9	not given
Microbial biomass (mg C / 100 g soil), at start / at study end	25.1 / 18.9	117.4 / 78.6
DT_{50}, days	38.2	49.6
DT_{90}, days	126.9	164.8

The photolytic degradation of [^{14}C]-cyromazine was evaluated on a soil surface (Gartenacker loam/silt loam; 2.6% OM; pH = 7.1) at 20°C under an artificial light source (Morgenroth, 2000). Test samples were prepared by coating glass slides with a soil slurry and allowing the moisture to evaporate until either the soil was at 75% field capacity (moist soil) or almost dry (dry soil). The thickness of the final soil layers was approximately 2 mm. ^{14}C-cyromazine was applied to the soil surfaces at a rate of 3.2 µg/cm^2 corresponding to a field rate of 0.32 kg ai/ha. Samples were irradiated for 240 h, equivalent to 30 days natural summer sunlight at latitude 30-50°N. Dark control samples (moist soil) were also included. Duplicate samples were taken after about 17, 24, 41, 65 and 137 h of irradiation and extracted with acetonitrile and acetonitrile/water. At sampling times the soil moisture was adjusted and trapping solutions were removed and replaced by fresh solutions. Due to the low radioactivity levels (by LSC) these trapping solutions were not further analysed. The extracts were analysed for cyromazine and its degradation products by HPLC/UV and 2D-TLC. Non-extracted radioactivity was determined by combustion and LSC.

The total recovery of radioactivity from samples of both the irradiated and non-irradiated treatments was between 94 and 99% of total applied. Volatile radioactivity was < 0.1% of applied doses for all treatments. In moist irradiated soil, cyromazine degraded rapidly with a calculated DT_{50} of 3.5 days by simple first order kinetics (DT_{90} 12 days). In dry irradiated soil the level of cyromazine fell rapidly from 97% to 74% of applied radiochemical during the first 24 h. Thereafter, degradation was slow with 62% of the parent remaining after 240 h irradiation (DT_{50} > 1 year). In both moist and dry soils, melamine was the major degradation product observed (53 and 15% of applied radioactivity in the moist and dry soils, respectively).

In four field studies carried out on bare soil plots in the USA in 1982/83 (Swidensky, 1987), the dissipation of cyromazine and melamine was followed in soil located in Fresno CA (sandy loam), York NE (silty clay), Vero Beach FL (sand) and in South Bay FL (peat). The soil characteristics are detailed in Table 22. Plots were treated at an exaggerated rate of 5.6 kg ai/ha as a single application and a double application (superimposed at year 2) at all four sites. Samples were taken for up to two years after the first application.

Table 22. Soil characteristics for the USA bare soil dissipation studies

	Fresno, CA	York, NE	Vero Beach, FL	South Bay, FL
Classification (USDA)	sandy loam	silty clay	sand	peat
Organic matter content (%)	1.3	2.7	22.3	72.0
pH	6.5	6.5	6.4	-
Cation exchange capacity (meq/100 g)	4.9	17.9	4.5	-

The residue data for parent cyromazine and its major soil degradate melamine in the 0 – 45 cm soil layers are presented in Table 23. Cyromazine residues were found in the 30 – 45 cm soil layer only at the Florida trials. Melamine was detected in the day 0 sample times at three of the four sites following a single application. Parent DT_{50} calculations assuming pseudo first order reaction kinetics gave half-lives shown in Table 23.

Table 23. US bare ground soil degradation studies

	First year [a]						Second year [a]						
	Cyromazine, (mg/kg)			Melamine, (mg/kg) [c]			Cyromazine, (mg/kg)				Melamine, (mg/kg) [c]		
Day[a]	0 - 15 cm	15 - 30 cm	30 - 45 cm	0 - 15 cm	15 - 30 cm	30 - 45 cm	Day[b]	0 - 15 cm	15 - 30 cm	30 - 45 cm	0 - 15 cm	15 - 30 cm	30 - 45 cm
Fresno, California – Cyromazine DT_{50} = 180 days (1st year) and 75 days (2nd year)													
0	0.39	n.a.	n.a.	< 0.05	n.a.	n.a.	0	1.7	n.a.	n.a.	0.91	n.a.	n.a.
15	0.46	n.a.	n.a.	0.19	n.a.	n.a.	16	0.98	n.a.	n.a.	0.86	n.a.	n.a.
34	0.26	n.a.	n.a.	0.15	n.a.	n.a.	32	0.78	n.a.	n.a.	0.48	n.a.	n.a.
60	0.93	< 0.05	n.a.	0.36	0.09	n.a.	60	0.76	< 0.05	n.a.	0.85	0.13	n.a.
120	0.35	< 0.05	n.a.	0.29	0.10	n.a.	120	1.0	< 0.05	< 0.05	1.4	0.37	0.10
181	0.62	< 0.05	< 0.05	0.74	0.17	0.08	179	0.56	< 0.05	< 0.05	2.7	0.94	0.12
271	0.07	< 0.05	< 0.05	0.44	0.20	< 0.05	269	0.07	< 0.05	< 0.05	1.5	0.51	0.07
366	< 0.05	< 0.05	< 0.05	0.59	0.16	< 0.05	366	< 0.05	< 0.05	< 0.05	0.47	0.10	0.07
545	< 0.05	< 0.05	< 0.05	0.78	0.48	< 0.05							
732	< 0.05	< 0.05	< 0.05	0.35	0.09	0.18							
York, Nebraska - Cyromazine DT_{50}= 244 days (1st year) and 204 days (2nd year)													
0	1.06	n.a.	n.a.	0.23	n.a.	n.a.	0	1.8	n.a.	n.a.	1.16	n.a.	n.a.
18	2.2	n.a.	n.a.	0.49	n.a.	n.a.	15	2.2	n.a.	n.a.	2.2	n.a.	n.a.
24	1.5	n.a.	n.a.	0.55	n.a.	n.a.	29	1.3	n.a.	n.a.	2.35	n.a.	n.a.
62	1.6	n.a.	n.a.	1.1	n.a.	n.a.	62	1.4	< 0.05	n.a.	3.35	0.14	n.a.
125	0.52	< 0.05	n.a.	0.96	0.07	n.a.	125	0.77	< 0.05	< 0.05	2.9	0.20	0.15
187	0.63	< 0.05	< 0.07	1.0	0.05	0.12	181	0.74	< 0.05	< 0.05	3.1	0.16	0.12
309	0.72	< 0.05	< 0.05	1.2	0.11	0.10	289	0.54	< 0.05	0.07	2.0	0.10	0.12
368	0.55	< 0.05	< 0.05	1.3	< 0.05	< 0.05	362	0.46	0.08	0.06	2.6	0.24	0.08
550	0.25	< 0.05	< 0.05	1.5	0.15	0.10							
731	0.20	< 0.05	< 0.05	1.1	0.14	0.12							
Vero Beach, Florida – cyromazine DT_{50}= 284 days (1st year) and 185 days (2nd year)													
0	1.95	n.a.	n.a.	0.11	n.a.	n.a.	0	1.5	n.a.	n.a.	0.65	n.a.	n.a.
14	2.4	n.a.	n.a.	0.96	n.a.	n.a.	14	0.87	n.a.	n.a.	1.1	n.a.	n.a.
30	1.0	n.a.	n.a.	0.67	n.a.	n.a.	29	0.97	n.a.	n.a.	1.0	n.a.	n.a.
59	0.70	n.a.	n.a.	0.73	n.a.	n.a.	60	0.30	< 0.05	n.a.	0.51	0.13	n.a.
122	0.25	0.20	n.a.	0.34	0.30	n.a.	120	1.0	0.14	< 0.05	1.6	0.24	< 0.05

	First year [a]						Second year [a]						
	Cyromazine, (mg/kg)			Melamine, (mg/kg) [c]			Cyromazine, (mg/kg)				Melamine, (mg/kg) [c]		
Day[a]	0 - 15 cm	15 - 30 cm	30 - 45 cm	0 - 15 cm	15 - 30 cm	30 - 45 cm	Day[b]	0 - 15 cm	15 - 30 cm	30 - 45 cm	0 - 15 cm	15 - 30 cm	30 - 45 cm
183	0.24	0.11	0.11	0.33	0.08	0.04	183	0.37	0.13	< 0.05	1.0	0.26	0.10
274	0.13	0.14	0.10	0.37	0.11	0.10	272	0.24	0.12	0.07	0.99	0.33	0.21
365	0.20	0.17	0.09	0.62	0.47	0.11	365	0.26	< 0.05	< 0.05	1.0	0.10	0.18
548	0.07	< 0.05	< 0.05	0.24	0.12	0.07							
730	< 0.05	< 0.05	< 0.05	0.46	0.12	0.09							
South Bay, Florida - cyromazine DT_{50}= 146 days (1st year) and 203 days (2nd year)													
0	4.3	n.a.	n.a.	0.07	n.a.	n.a.	0	6.55	n.a.	n.a.	3.0	n.a.	n.a.
15	2.8	n.a.	n.a.	< 0.05	n.a.	n.a.	16	6.5	n.a.	n.a.	2.0	n.a.	n.a.
30	2.9	n.a.	n.a.	0.59	n.a.	n.a.	31	6.0	n.a.	n.a.	1.3	n.a.	n.a.
61	1.9	0.11	n.a.	0.19	< 0.05	n.a.	63	4.3	0.95	n.a.	1.9	0.42	n.a.
120	4.3	0.13	n.a.	1.8	< 0.05	n.a.	122	2.2	0.18	0.48	1.5	0.25	0.39
150	4.8	0.09	0.37	4.1	0.07	0.15	180	5.6	0.14	0.38	3.6	0.24	0.56
272	1.1	0.43	< 0.05	2.8	1.0	0.88	273	0.87	< 0.12	< 0.15	2.7	0.36	0.44
365	1.0	< 0.05	< 0.05	3.2	0.34	0.20	364	1.8	0.35	0.63	4.1	1.1	1.7
487	1.4	< 0.09	0.14	5.0	0.42	0.84							
729	0.58	< 0.08	< 0.13	2.7	0.46	0.39							

a, b - Treatment at day 0 of first and second year.

c - Cyromazine equivalents

n.a. - Not analysed

In four field studies carried out on bare soil plots in the potato growing areas in Canada (Purdy 1995), cyromazine was applied at 0.280 kg ai/ha in mid-late June and a second time 25–31 days later, at the Truro site, a rate of 0.476 kg ai/ha was used in the first application. Soil sampling was performed at a series of time points after the first and second applications. The soil and site characteristics are shown in Table 24.

Table 24. Soil characteristics for cyromazine for the Canadian bare ground soil degradation studies

Origin of soil:	Plattsville Ontario	Cambridge Ontario	Truro [a] Nova Scotia	Kentville Nova Scotia
Classification (USDA):	Silt loam	Loam	Sandy loam	Sandy loam
Organic matter content (%)	3.0	1.8	2.7	2.4
pH:	6.1	6.3	6.5	6.3
Cation exchange capacity (meq/100g)	7.1	7.4	6.7	6.4
Approx. depth to water table (m)	12	15	>5	>5

a - First application was 476 g ai/ha, all others at 280 g ai/ha.

Cyromazine residues declined rapidly after the second application and continued to decline over the winter period at a slower rate. The majority of the parent residue was retained in the upper soil layer (0 – 15 cm) (Table 23). Melamine was present from background sources in the three topsoil layers in the range of 5–11 µg/kg. Most of the melamine residues remained in the top 30 cm. The data was evaluated by Dorn (2004) in order to generate first order exponential decay rates for cyromazine and for melamine, taking formation and decline of the metabolite into account. The data obtained after the winter season were not used because degradation occurred at a reduced rate due to low in Table 25.

Table 25. Canadian bare ground soil degradation studies analytical results

	Residues (mg/kg) [b] Cyromazine					Residues (mg/kg) [b] Melamine				
Plattsville, Ontario:										
Day	0 - 15 cm	15 - 30 cm	30 - 60 cm	60 - 90 cm	90 - 120 cm	0 - 15 cm	15 - 30 cm	30 - 60 cm	60 - 90 cm	90 - 120 cm
0	0.178	n.a.	n.a.	n.a.	n.a.	0.065	n.a.	n.a.	n.a.	n.a.
26	0.111	< 0.0025	< 0.0025	< 0.0025	0.0013	0.022	0.0025	< 0.005	< 0.005	< 0.005
27[a]	0.289	n.a.	n.a.	n.a.	n.a.	21	n.a.	n.a.	n.a.	n.a.
41	0.193	0.0056	< 0.0025	0.0014	0.0015	0.065	0.0054	< 0.005	< 0.005	< 0.005
85	0.053	0.0052	0.0031	0.0015	< 0.0025	0.042	0.0066	< 0.005	< 0.005	< 0.005
126	0.048	0.0013	< 0.0025	< 0.0025	< 0.0025	0.068	0.013	0.005	< 0.005	< 0.005
306	0.026	< 0.025	0.0013	< 0.025	0.0013	0.048	0.0077	0.0033	< 0.005	< 0.005
	DT_{50}= 31 days					DT_{50}= 24 days				
Cambridge, Ontario:										
0	0.142	n.a.	n.a.	n.a.	n.a.	4.6	n.a.	n.a.	n.a.	n.a.
27	0.048	0.0013	< 0.0025	< 0.0025	< 0.0025	0.026	< 0.005	< 0.005	< 0.005	< 0.005
28[a]	0.174	n.a.	n.a.	n.a.	n.a.	0.022	n.a.	n.a.	n.a.	n.a.
41	0.068	0.008	0.0018	20.0029	< 0.0025	0.075	0.013	< 0.005	0.0025	< 0.005
84	0.041	0.007	0.010	< 0.0025	< 0.0025	0.066	0.0031	< 0.0025	< 0.005	< 0.005
121	0.007	< 0.0025	< 0.0025	< 0.0025	< 0.0025	0.018	0.0056	< 0.005	< 0.005	< 0.005
300	0.032	< 0.0025	< 0.0025	< 0.0025	< 0.0025	0.132	0.0096	0.0025	< 0.005	< 0.005
	DT_{50}= 29 days					DT_{50}= 28 days				
Truro, Nova Scotia:										
0	0.298[c]	n.a.	n.a.	n.a.	n.a.	0.0078	n.a.	n.a.	n.a.	n.a.
26	0.221	0.0017	0.0025	0.0026	< 0.0025	0.062	0.0034	< 0.005	< 0.005	< 0.005
27[a]	0.311	n.a.	n.a.	n.a.	n.a.	0.067	n.a.	n.a.	n.a.	n.a.
37	0.182	< 0.0025	< 0.0025	0.0041	< 0.0025	0.055	< 0.005	< 0.005	< 0.005	< 0.005
86	0.102	< 0.0025	< 0.0025	< 0.0025	< 0.0025	0.109	0.005	< 0.005	< 0.005	< 0.005
142	0.062	0.0027	< 0.0025	< 0.0025	< 0.0025	0.114	0.0058	< 0.005	< 0.005	< 0.005
321	0.055	< 0.0025	< 0.0025	< 0.0025	< 0.0025	0.048	0.0084	< 0.005	< 0.005	< 0.005
	DT_{50}= 42 days					DT_{50}= 54 days				
Kentville, Nova Scotia:										
0	0.182	n.a.	n.a.	n.a.	n.a.	0.0043	n.a.	n.a.	n.a.	n.a.
26	0.156	0.017	0.0035	< 0.0025	< 0.0025	0.052	0.010	< 0.005	< 0.005	< 0.005
27[a]	0.254	n.a.	n.a.	n.a.	n.a.	0.035	n.a.	< 0.005	n.a.	n.a.
45	0.154	< 0.0025	< 0.0025	< 0.0025	0.0015	0.076	0.0058	< 0.005	0.0025	< 0.005
89	0.045	0.011	0.0028	< 0.0025	< 0.0025	0.041	0.015	0.0031	< 0.005	< 0.0025
145	0.055	< 0.0025	< 0.0025	< 0.0025	< 0.0025	0.050	< 0.005	< 0.005	< 0.005	< 0.005
312	0.045	0.0077	< 0.0025	< 0.0025	< 0.0025	0.059	0.005	< 0.005	< 0.005	< 0.005
	DT_{50}= 35 days					DT_{50}= 26 days				

a - Day of second treatment;

b - Averages of two replicates from two plots;

c - First treatment with double rate (476 g ai/ha).

Two field studies were conducted in Switzerland and France by Evans (2004 a, b) to investigate the dissipation of cyromazine in soil following a broadcast spray treatment at 0.30 kg ai/ha to the soil surface. Soil samples from the treated plot were taken immediately after the application and on 3, 7, 14, 30 and 59 days after application (DAA) in France, in addition to 120, 244 and 360 DAA in Switzerland. Samples were taken from the 0 – 10 cm, 10 – 20 cm and 20 – 30 cm soil layer, apart from on two occasions (120 and 360 DAA), when samples were taken from the 0 – 90 cm soil layer.

Residues of cyromazine and melamine in the soil layers 0 – 20 cm in 2002/2003 in France and Switzerland are given in Table 26. In France, no residues were detected at the 20 – 30 cm soil layer. For cyromazine, the DT_{50} and DT_{90} calculated from the 0 – 10 cm layer residue data were 17 days and 56 days, respectively. A DT_{50} of 40 days was estimated for melamine (Dorn 2004). In Switzerland, no cyromazine residues were found at the 20 cm layer or deeper and a DT_{50} of 2.3 days and a DT_{90} of 7.5 days were calculated based on the 0 – 10 cm layer data. Residues of melamine were only detected in the 10 – 20 cm soil core depth after 59, 120 and 244 days after application. A single melamine residue of 0.008 mg kg^{-1} dry soil was found in the 20 – 30 cm soil core depth at 120 days after application. The melamine residue data were evaluated by Dorn (2004), who estimated a DT_{50} of 51 days.

Table 26. Cyromazine and melamine soil residues in South France

	France				Switzerland			
	Cyromazine, mg/ kg		Melamine, mg /kg		Cyromazine, mg/ kg		Melamine, mg /kg	
DAA	0 – 10 cm	10 - 20 cm	0 – 10 cm	10 - 20 cm	0 – 10 cm	10 - 20 cm	0 – 10 cm	10 - 20 cm
0	0.21	0.0068	0.007	< 0.005	0.28	**< 0.0025**	0.039	< 0.005
3	0.18	< 0.0025	< 0.005	< 0.005	0.11	< 0.0025	0.10	< 0.005
7	0.14	< 0.0025	0.038	< 0.005	0.033	< 0.0025	0.12	< 0.005
14	0.096	< 0.0025	0.049	< 0.005	0.0068	< 0.0025	0.075	< 0.005
30	0.071	< 0.0025	0.074	< 0.005	< 0.0025	< 0.0025	0.15	< 0.005
59	0.035	0.0040	0.063	0.018	< 0.0025	< 0.0025	0.051	0.024
120	-	-	-	-	< 0.0025	< 0.0025	0.013	0.023
244	-	-	-	-	< 0.0025	< 0.0025	0.011	0.015
360	-	-	-	-	< 0.0025	< 0.0025	0.008	< 0.005

In another study, Evans (2004c) evaluated the dissipation and accumulation behaviour of cyromazine at one site in Greece. Cyromazine was applied annually four times at approximately seven day intervals to bare ground (sandy loam soil type) from 2001 to 2003 at a rate of 0.30 kg ai/ha in 400 L/ha water (nominal). The plot was cultivated with mustard one to two days before the first application of each year. Samples were taken from different depths to a maximum of 90 cm below the surface. Residues in soil up to the 30 cm soil layer are shown in Table 27. The 50 – 70 cm layer contained residues on two occasions, at a maximum of 0.011 mg/kg cyromazine and 0.009 mg/kg melamine. No residues were found in the 70 – 90 cm soil layer. In the first six months of the first year, the total rainfall was only 103 mm and this drought was not compensated by few irrigation events, so the biological activity of this site during the first season was very low. In the second year, rainfall was higher, resulting in a faster decline of residue levels. Melamine levels in soil increased during the trial following the degradation of cyromazine.

Table 27. Cyromazine and melamine soil residues in Greece

	Cyromazine, mg/kg (dry matter)			Melamine, mg/kg (dry matter)		
DALA	0-10 cm	10-20 cm	20-30 cm	0-10 cm	10-20 cm	20-30 cm
0 (08/2001)	1.2086	0.0369	< 0.0025	0.0447	0.0104	< 0.0050
3	1.3187	0.0326	< 0.0025	0.0587	0.0124	< 0.0050
7	1.0057	0.0088	< 0.0025	0.0586	0.0081	< 0.0050
14	1.1686	< 0.0025	< 0.0025	0.0775	0.0092	< 0.0050
33	0.5913	0.0227	< 0.0025	0.0868	0.0078	< 0.0050
66	0.7117	0.0206	0.0037	0.0907	0.0076	< 0.0050
97	0.8538	0.0221	0.0048	0.1021	< 0.0050	< 0.0050
184	0.6483	< 0.0025	< 0.0025	0.111	< 0.0050	< 0.0050
276	0.7987	0.0078	< 0.0025	0.0991	< 0.0050	< 0.0050
0 (05/2002)	0.8311	0.0062	< 0.0025	0.1248	< 0.0050	< 0.0050
31	1.2127	0.0182	0.003	0.6755	0.0141	< 0.0050
184	0.5278	0.0066	0.0057	0.6713	0.0115	0.0067
339	0.3265	0.003	< 0.0025	0.9707	0.0069	< 0.0050

	Cyromazine, mg/kg (dry matter)			Melamine, mg/kg (dry matter)		
DALA	0-10 cm	10-20 cm	20-30 cm	0-10 cm	10-20 cm	20-30 cm
0 (06/2003)	1.0145	0.0497	0.003	1.1498	0.0475	0.0071
30 (07/2003)	2.2053	0.0324	0.0414	1.3764	0.0523	0.0571

DALA: days after the last application (date of application)

In the winter season the dissipation of cyromazine slowed due to low temperatures and remained on a similar level until May 2002. Therefore for the estimation of the DT_{50} for cyromazine and melamine in the first season only the values until October (66 DALA) were taken into account. The evaluation resulted in a DT_{50} of 61 days and a DT_{90} of 202 days for cyromazine and a DT50 of 31 days and a DT_{90} of 111 days for melamine.

In a study conducted in Spain (Balluf, 2004), cyromazine was applied yearly to a loamy silt (pH= 7.7, OM= 2.0%) in four subsequent early summer applications at 0.30 kg ai/ha per treatment in 400 L water to a 408 m^2 plot. Tomato was planted starting in the second year of the experiment just before the application. The residues levels found in the 0 – 30 cm deep soil layers are presented in Table 28. Cyromazine residues in deeper than 30 cm soil layers were < 0.005 mg/kg. In the consecutive years residues of melamine were found in soil layers up to 50 cm depth, with two findings in deeper layers up to 100 cm in the second and in the third year of the study, respectively. In this period (November 2001 – May 2002), the rainfall of 620 mm far exceeded the long term average of 297 mm. The values for the dissipation of cyromazine were calculated from the first year data using a function of 1st order, resulting in a DT_{50} of 51 days and a DT_{90} of 169 days. After three years of treatment, no increase of residue levels was observed .

Table 28. Cyromazine soil residues in Spain

	Cyromazine, mg/kg (dry matter)			Melamine, mg/kg (dry matter)		
(DALA)	0-10 cm	10-20 cm	20-30 cm	0-10 cm	10-20 cm	20-30 cm
0 (07/2000)	0.55	0.005	0.011	0.12	< 0.01	< 0.01
7	0.56	0.005	0.009	0.16	0.01	< 0.01
14	0.63	< 0.005	0.018	0.19	< 0.01	0.01
34	0.25	0.006	< 0.005	0.13	< 0.01	< 0.01
92	0.21	< 0.005	< 0.005	0.16	0.01	0.01
126	0.22	0.022	0.013	0.19	0.02	0.01
364	0.13	0.014	< 0.005	0.27	0.04	0.01
0 (08/2001)	0.5020	0.0162	< 0.003	0.3174	0.0203	< 0.006
28	0.5368	0.0047	< 0.003	0.4421	0.0170	< 0.006
180	0.1206	0.0314	< 0.006	0.6356	0.1835	0.0229
297	0.1104	0.0244	< 0.006	0.4536	0.2177	0.0531
0 (06/2002)	0.6234	0.0078	< 0.006	0.6685	0.1995	0.0566
29	0.3721	0.0072	< 0.006	0.6755	0.2261	0.0975
187	0.0966	< 0.006	< 0.003	0.4062	0.0505	0.0239
337	0.0394	0.0030	< 0.003	0.3429	0.2226	0.0330
0 (05/2003)	0.8545	0.0309	0.0030	0.4278	0.2573	0.0320
32	0.1972	0.0269	0.0056	0.3670	0.2564	0.0527
180	0.1129	0.0123	< 0.003	0.2761	0.2116	0.0183

Figure 3 shows the proposed metabolic pathway of cyromazine in soil. The dealkylation of cyromazine i.e. the cleavage of the cyclopropyl ring moiety yielding melamine and the formation of non-extractable soil material proceeds most favourably under aerobic conditions.

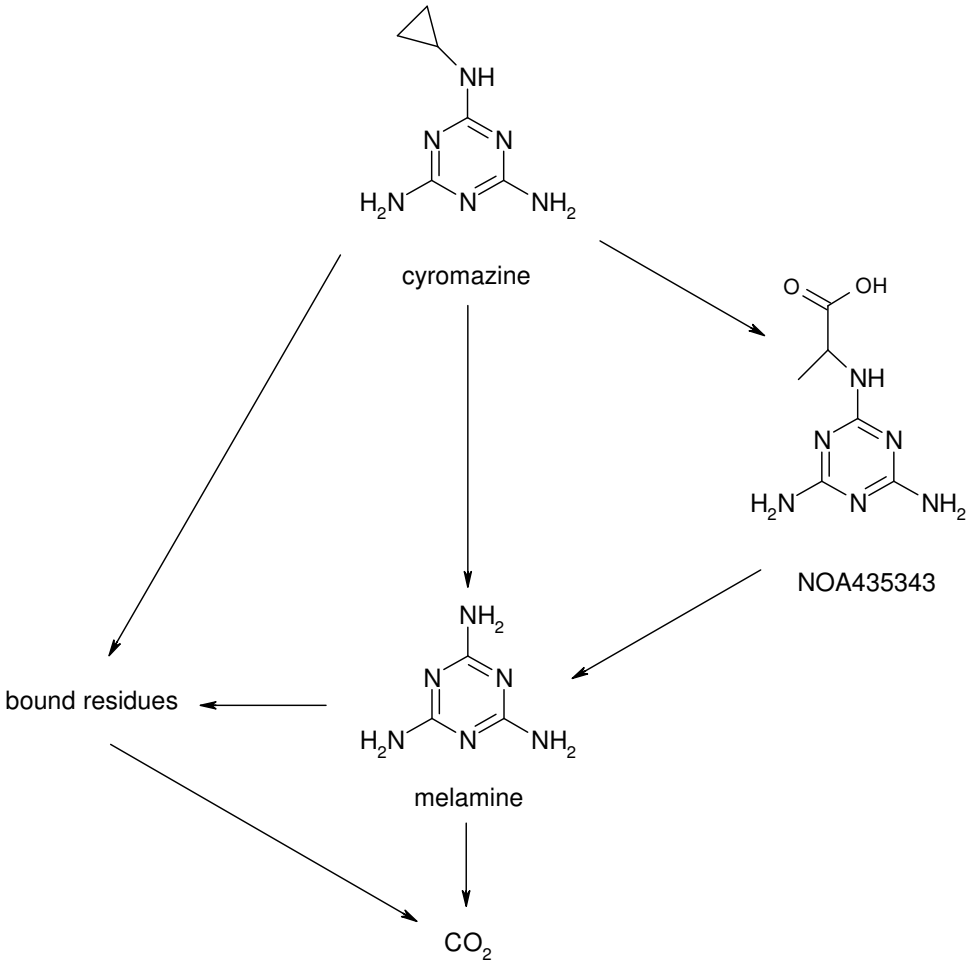


Figure 3 Proposed metabolic pathway of cyromazine in soil

Residues in succeeding crops

In a greenhouse study carried out by Simoneaux and Marco (1983b), celery seedlings were transplanted into a Florida muck soil (pH 7.6, CEC 146.3 meq/100 g, OM 60.3%, density 0.2 g/cm3) and [^{14}C]-cyromazine uniformly mixed with a sample of muck soil (3 kg) was applied as a top dressing to the crop at 1 kg ai/ha). Celery samples were taken 42 days and 84 days post treatment after which the containers were then immediately re-planted with radishes (variety Sparkler) or sweet corn (variety Funk G-80). Mature radish samples were taken 130 days post-treatment. The sweet corn was thinned to one plant per container 29 days after planting and mature samples (stalk, cobs and grain) taken 159 days post-treatment.

Soil samples were taken at 0 days post-treatment, 42 days (50% maturity of celery), 84 days (harvest of celery, planting of rotational crops), 130 days (harvest of radish) and 159 days (harvest of sweet corn) at depths of 0 – 7.7 cm, 7.7 – 15.3 cm, and 15.3 – 20 cm. Because of the low bulk density of the soil, the coring technique was considered to have led to some contamination of lower horizons and a soil sectioning technique was used at 217 days post-treatment to determine distribution of radioactivity. Total radioactive residue (TRR) in soil (air-dried) and crop samples were determined by combustion to $^{14}CO_2$ followed by trapping in solution and LSC. Soil samples and homogenised crop samples were cold-extracted by shaking with methanol/water and the extract filtered. Unextracted radioactivity in the dried residue was determined as $^{14}CO_2$ by combustion and LSC; the extract was partitioned between water and chloroform and radioactivity in each fraction determined by LSC. Characterization of radioactivity was by co-chromatography against authentic standards using TLC with quantification achieved by removal of radioactive zones followed by LSC.

Most of the radioactivity in celery stalks was found in the aqueous extract, with cyromazine being the main residue found at both sampling times (Table 29). Levels of [^{14}C]-cyromazine in radish and mature sweet corn planted 84 days following foliar application of the compound to celery were too low for additional balance and characterization work.

Table 29. Distribution of radioactivity and residual cyromazine in celery and following crops of radish and sweet corn following application of [^{14}C]-cyromazine in soil to celery at 1 kg ai/ha

Days after treatment	Plant part/soil layer	Total residues mg/kg [a]	Balance (as% TRR)				Characterization	
			Organic-soluble	Aqueous-soluble	Unextracted radioactivity	Total	Cyromazine mg/kg [b]	Melamine mg/kg [b]
Celery (target)								
42	Stalks	0.75	10.5	80.5	4.9	95.9	0.45	0.08
84	Stalks	0.34	8.7	75.9	4.7	89.3	0.15	0.10
Radish (rotational)								
130	Tops	0.02	-	-	-	-	-	-
130	Root	0.01	-	-	-	-	-	-
Sweet corn (rotational)								
159	Stalks	0.02	-	-	-	-	-	-
159	Cob	0.02	-	-	-	-	-	-
159	Grain	0.02	-	-	-	-	-	-

a - As cyromazine equivalents

b - Expressed as % TRR in report, recalculated as cyromazine equivalents

Results from soil analyses are summarized in Table 30. The erratic results obtained for distribution of radioactivity in the soil layers were thought to be the result of using a 2.5 cm core to sample the low bulk density soil with consequent compression and contamination of the lower layers. The radioactivity in the 0 – 7.7 cm layer was in the range 1.93 – 3.80 mg/kg (as cyromazine equivalents), with rapid conversion to non-extracted radioactivity within six weeks of application. Since non-extracted residues are presumed to be unavailable for leaching, this also suggests that the soil concentrations of 0.56 mg/kg, 0.21 mg/kg and 0.11 mg/kg in the 15.3 – 20 cm layers at 12, 23 and 31 weeks were artefacts of the sampling method. The samples taken at 217 days after treatment by carefully sectioning the soil layers rather than by coring indicate no significant movement of radioactivity to the > 7.7 cm layers.

Table 30. Distribution of radioactivity and residual cyromazine in soil following application of [^{14}C]-cyromazine to celery at 1 k g ai/ha

Days after treatment	Soil layer, cm	Total residues mg/kg [a]	Balance (as% TRR)			
			Organic-soluble	Aqueous-soluble	Unextracted radioactivity	Total
42[b]	0-7.7	2.40	<2.0	23.7	187.2	210.9
	7.7-15.3	1.10	<2.0	9.2	97.5	106.7
	15.3-20	0.02	-	-	-	-
84[b]	0-7.7	3.8	< 0.4	11.3	125.4	136.7
	7.7-15.3	0.45	<3.0	10.2	132.5	142.7
	15.3-20	0.21	<6.0	11.8	132.0	143.8
161[b]	0-7.7	1.93	-	-	-	-
	7.7-15.3	1.74	-	-	-	-
	15.3-20	0.56	-	-	-	-
217[c]	0-7.7	1.24	-	-	-	-
	3-15.3	0.07	-	-	-	-
	15.3-20	0.11	-	-	-	-

a - As cyromazine equivalents

b - Obtained by sectioning a 1× 20 soil core

c - Obtained by hand-scraping each soil layer to the appropriate depth

In a second rotational crop study, field grown tomatoes were treated with [^{14}C]-cyromazine at 6 × 0.28 kg ai/ha (Simoneaux 1985). Following harvest (14 days after last application) the plot was roto-tilled to a depth of 10.2 cm to obtain a homogeneous distribution of the soil residues. Winter wheat was planted immediately after harvest of the target tomato crop, and the remaining rotational crops (lettuce, carrot, soya, sugar beet) were planted the following spring. Two harvests (immature and mature crop) were taken from each crop for analysis. Soil samples at 0 – 23 cm horizons were taken 223 days after last treatment (planting of lettuce crop) and 484 days after last treatment (harvest of mature soybean).

Total radioactive residue (TRR) in soil (air-dried) and crop samples were determined by combustion to $^{14}CO_2$ followed by trapping in solution and LSC. Soil samples and homogenised crop samples were extracted with methanol/water and the extract partitioned with chloroform; unextracted radioactivity was determined as $^{14}CO_2$. Characterization of radioactivity was by co-chromatography against authentic standards using TLC, with quantification achieved by removal of radioactive zones followed by LSC. The soil samples taken 223 days after last application were refluxed in acetic acid/2M sodium acetate/methanol and cleaned-up by cation exchange chromatography.

Residues in succeeding crops were ≤ 0.05 mg/kg for all crop parts other than half-mature carrot tops (0.19 mg/kg). This latter residue was extractable and polar (93% partitioned into the aqueous layer). It was characterized as 14% cyromazine and 79% melamine. Other crop samples contained levels of radioactivity too low for characterization. Results are shown in Table 31. The distribution of radioactivity in the soil over time is shown in Table 32.

Table 31. Distribution of radioactivity in succeeding crops following application of [^{14}C]-cyromazine as a foliar spray to a target tomato crop at 1.68 kg ai/ha

Plant	Growing stage	Plant part	Residues, mg/kg cyromazine eq
Lettuce	¾ mature	leaves	0.03
	mature	leaves	< 0.01
Sugar beet	¾ mature	tops	0.02
		beets	< 0.01
	mature	tops	0.01
		beets	< 0.01
Winter wheat	¾ mature	stalks	0.01
	mature	stalks	0.04
		grain	< 0.01
		hulls	0.01
Soybeans	60 days	stalks	0.02
	mature	stalks	0.04
		pods	0.05
		beans	0.03
Carrots	½ mature	tops	0.19
		roots	0.03
	mature	tops	0.05
		roots	< 0.02

Table 32. Distribution of radioactivity in soil following application of [^{14}C]-cyromazine as a foliar spray to a target tomato crop at 1680 g ai/ha

DALT	Soil layer, cm	Total residue, mg/kg [a]	Balance,% TRR				Characterization,% TRR	
			Organic	Aqueous	Non-extracted	Total	Cyromazine	Melamine
223	0 - 7.7	0.32	< 0.1	6.5	94.2	100.7	21.0	45.1
			-	70.1[b]	25.8[b]	95.9[b]		
	7.7 - 15.3	0.14	< 0.1	6.2	78.9	85.1	17.6	82.4
			-	112.2[b]	21.8[b]	134.0[b]		
	15.3 - 23	< 0.05	-	-	-	-	-	-
484	0 - 7.7	0.34	< 0.1	5.5	90.6	96.1		
	7.7 - 15.3	0.15	< 0.1	5.1	96.7	101.8		
	15.3 - 23	< 0.05	-	-	-	-		

a - As cyromazine equivalents

b - Extraction with acetic acid : 2M sodium acetate : methanol (8 + 1 + 1 v/v) for 1 h ;

DALT= Days after last treatment

The third study performed by Simoneaux (1986) was undertaken to determine the fate of cyromazine in chicken manure amended soil and to determine the uptake and metabolism of cyromazine residues by rotational cops. Spring wheat, lettuce and sugar beets were planted in buckets containing a 7.6 cm top layer of cyromazine-treated soil and grown to maturity in the greenhouse. The cyromazine-treated Georgian sandy loam soil was prepared by incorporating chicken manure equivalent to a concentration of 11.2 t manure/ha and aged for 30 days prior to planting. Chicken manure was previously fortified with [^{14}C]-cyromazine to give a concentration of 5 mg cyromazine/kg manure. Crops were sampled at 75% maturity and at maturity. Soil samples were taken directly after treatment (day zero), at planting (30 days post treatment) and after the last harvest (131 days post treatment) and represented cores of 0 – 7.7, 7.7 – 15.3, and 15.3 – 20.0 cm. TRR in soil and crop samples was determined by combustion to $^{14}CO_2$ followed by trapping in solution and LSC. Soil and crop extracts were purified prior to TLC on a Sephadex A-25 anion exchange column, lyophilised and taken up in methanol. Characterization of radioactivity was done by TLC, with quantification achieved by removal of radioactive zones followed by LSC.

At maturity, TRR in lettuce, sugar beets and spring wheat grain were < 0.01 mg/kg (Table 33). Mature spring wheat straw and hulls contained residues greater than the initial soil concentration of 0.064 mg/kg. Cyromazine and melamine accounted for 42.5% and 27.8% of the TRR wheat straw, respectively.

Table 33. Distribution of radioactivity in succeeding crops following application of [^{14}C]-cyromazine amended chicken manure to soil

Plant	Growing stage	Plant part	Residues, mg/kg cyromazine eq
Lettuce	¾ mature	leaves	< 0.009
	mature	leaves	< 0.009
Sugar beet	¾ mature	tops	0.011
		beets	< 0.009
	mature	tops	< 0.009
		beets	< 0.009
Spring wheat	¾ mature	stalks	0.022
	mature	straw	0.112
		hulls	0.078
		grain	< 0.009

The distribution of radioactivity in the soil over time is shown in Table 34. Residue concentration in soil horizons deeper than 7.7 cm were too low to characterise.

Table 34. Distribution of radioactivity in soil following application of [^{14}C]-cyromazine amended chicken manure

DALT	Soil layer, cm	Total residue mg/kg [a]	Balance,% TRR			Characterization,% TRR	
			Extracted	Non-extracted	Total	Cyromazine	Melamine
30	0-7.7	0.064	81.0	14.8	95.8	72.3	6.5
	7.7-15.3	0.020	-	-	-	-	-
	15.3-20.0	0.002	-	-	-	-	-
	0-20.0	0.032 [b]	-	-	-	-	-
131	0-7.7	0.044	82.8	20.6	103.4	65.4	17.8
	7.7-15.3	0.016	-	-	-	-	-
	15.3-20.0	0.009	-	-	-	-	-
	0-20.0	0.025 [b]	-	-	-	-	-

a - As cyromazine equivalents

b - Calculated average value for a 0-20 cm soil horizon;

DALT=Days after last treatment

Supervised field trials on rotational crops were carried out in Mississippi (USA) in which tomato crops were treated with 12 foliar applications of cyromazine at 0.14 or 0.28 kg ai/ha (Cheung and Edmonds 1985). The tomatoes were grown to maturity and harvested at 0 – 14 days after last application. Following a post-harvest interval of 10 weeks the plot was re-planted with wheat. Cyromazine residues in samples of forage, straw and grain harvested nine weeks after planting ranged from < 0.05 to 0.08 mg/kg, with the higher levels found in fall forage.

Field trials were carried out in California and Florida in which celery was treated with 11–15 foliar applications of cyromazine at rates of 0.11 to 0.28 kg ai/ha (Ballantine 1985a). The celery was grown to maturity and harvested at 0 – 14 days after the last application. Following a post-harvest interval of one to six weeks (Florida) or eight weeks (California) the field plots were re-planted with lettuce, sweet corn or radishes. Residues of cyromazine in rotational crops ranged from < 0.05 to 0.22 mg/kg (Table 35).

Table 35. Cyromazine residues in rotational sweet corn, radish and lettuce following foliar applications to celery

Succeeding crop (Trial ref)	Location/year	No. applications and rate (kg ai/ha)	Plant-back interval (weeks)	Harvest to planting (weeks)	Crop part	Cyromazine (mg/kg)
Sweet corn (7364 I FL)	Florida, 1982/3	12 x 0.14	1-3	1	Forage Ears	0.08[a] 0.06[a]
Sweet corn (7427 I FL)	Florida 1982/3	12 x 0.14	6-8	6	Forage Ears	0.19[a] < 0.05[a]
Sweet corn (7446-01A FL)	Florida 1982/3	13 x 0.14	2-4	2	Forage Ears	< 0.05[a] < 0.05[a]
Sweet corn (7454-01/02 FL)	Florida 1982/3	12 x 0.14	3-5	3	Forage Fodder Ears	< 0.05[a] < 0.05[a] < 0.05[a]
		12 x 0.28	3-5	3	Forage Fodder Ears	< 0.05 < 0.05 < 0.05
Sweet corn (7533-01/02 CA)	California 1982/3	15 x 0.14	8-10	8	Forage Ears	0.08[a] < 0.05[a]
		15 x 0.28	8-10	8	Forage Ears	0.15 < 0.05
Sweet corn (8228-01 FL)	Florida 1982	12 x 0.14	61-63	61	Forage Ears	< 0.05[a] < 0.05[a]
Radish (7024 FL)	Florida 1982	11 x 0.28	Not stated	Not stated	Roots	< 0.05[a]
		11 x 0.56	Not stated	Not stated	Roots	< 0.05[a]
		11 x 1120	Not stated	Not stated	Roots	0.09[a]

Succeeding crop (Trial ref)	Location/year	No. applications and rate (kg ai/ha)	Plant-back interval (weeks)	Harvest to planting (weeks)	Crop part	Cyromazine (mg/kg)
Radish (7362 FL)	Florida 1982/3	12 x 0.14	6-8	6	Roots Tops	< 0.05[a] < 0.05[a]
Radish (7372 I FL)	Florida 1982/3	13 x 0.14	2-4	2	Roots Tops	0.15[a] 0.11[a]
Radish (7373 I FL)	Florida 1982/3	12 x 0.14	1-3	1	Roots Tops	< 0.05[a] < 0.05[a]
Radish (7485 I CA)	CA 1982/3	15 x 0.14	8-10	8	Roots Tops	< 0.05[a] 0.11[a]
		15 x 0.28	8-10	8	Roots Tops	< 0.05 0.22
Lettuce (7361 I-II FL)	Florida 1982/3	12 x 0.14	6-8	6	Leaf	0.05[a]
Lettuce (7425 I FL)	Florida 1982/3	13 x 0.14	2-4	2	Leaf	< 0.05[a]
Lettuce (7426 I FL)	Florida 1982/3	12 x 0.14	1-3	1	Leaf	0.06[a]
Lettuce (7484 I-II FL)	Florida 1982/3	15 x 0.14	6-8	6	Leaf	< 0.05[a]
		15 x 0.28	6-8	6	Leaf	0.08

a - mean of replicate samples

METHODS OF RESIDUE ANALYSIS

Residues in plants

In method AG-402 (Buettle 1983), residues of cyromazine and melamine are extracted from plants by refluxing chopped samples in water/methanol, an aliquot of the extract is evaporated to leave the aqueous phase, acidified with 0.1M hydrochloric acid and cleaned up by partition with dichloromethane and hexane, followed by cation exchange chromatography. An additional clean-up by automated gel permeation chromatography (GPC) might be used to remove interferences. Residues are analysed by HPLC on a LiChrosorb-NH2 column using acetonitrile/water as mobile phase, with determination by UV absorption at 214 nm. The method was validated for celery, lettuce (head and leaf), tomatoes and mushrooms (Table 36). Accountability of the method was investigated by analysis of three celery samples with incurred residues of [^{14}C]-cyromazine at levels from 0.25 to 1.21 mg/kg cyromazine equivalents. The sum of cyromazine and melamine residues determined by method AG-402 accounts for an average of 122% of the cyromazine plus melamine residues determined radiochemically.

Table 36. Recovery of cyromazine from fortified crop samples using Analytical Method AG-402

Matrix	Fortification (mg/kg)	Recovery (%)	
		Cyromazine	Melamine
Leaf lettuce	0.05	75	76
	0.40	106	133
	1.0	93	93
	2.0	100	102
Head lettuce	0.05	148	65
	0.40	113	107
	1.0	97	93
	2.0	111	102
	4.0	95	-
Celery	0.05	90	97
	0.20	77	98

Matrix	Fortification (mg/kg)	Recovery (%)	
		Cyromazine	Melamine
	0.40	94	94
	1.0	89	92
	2.0	93	87
Tomatoes	0.05	87	90
	0.5	98	91
	1.0	97	97
Mushrooms	0.05	106	85
	2.0	80	86

Method AG-408 is a modification of Method AG-402 in which the clean-up by GPC is replaced by anion exchange chromatography. Validation data for this method was not provided. The analysis of celery stalks containing incurred residues of [^{14}C]-cyromazine from 0.34 to 1.46 mg/kg cyromazine eq (Buettle, 1983) by both methods showed accountability from 74 to 105% TRR.

Method AG-621 is identical to method AG-402 in the critical extraction and primary cleanup steps, but the quantification is performed after gas chromatographic separation on a CP-Wax 52 CB column an NPD (Vincent, 1995). Validation studies were performed on variety of crops and are shown in Table 38.

Table 38. Recovery data from fortified crop substrates using Analytical Method AG-621 (Reported in Selman 1995)

Crop	Fortification (mg/kg)	Cyromazine			Melamine		
		Recovery,%	CV,%	n	Recovery,%	CV,%	n
Lettuce	0.05	99	25	3	93		2
	0.20	115	-	1	-	-	-
	1.00	107	21	3	98	16	3
	2.00	95.5	-	2	100	-	2
	5.00	91	-	1	97	-	1
	10.00	77	-	1	77	-	1
Alfalfa forage	0.05	80	-	1	120	-	1
	0.10	86	20	5	86	15	5
	0.30	92	-	2	89	-	2
	0.50	-	-	-	125	-	1
	1.00	86	-	1	110	-	1
	2.00	80	-	1	60	-	1
	3.00	74	-	1	-	-	-
Alfalfa hay	0.05	126	-	1	119	-	1
	0.10	81	4	3			1
	0.20	77	-	1	53	-	1
	0.50	85	-	1	63	-	1
	1.00	101	-	2	107	-	2
	2.00	83	-	1	89	-	1
	5.00	61	-	1	88	-	1
Alfalfa meal	0.05	90	-	1	88	-	1
	0.20	72	-	1	-	-	-
	4.00	-	-	-	56	-	1
Alfalfa pellets	0.04	-	-	-	84	-	1
	0.05	121	-	1	-	-	-
	0.10	110	-	1	115	-	1
	0.20	126	-	1	107	-	1
	3.00	107	-	1	93	-	1
Alfalfa seed	0.05	97	-	1	70	-	1
	0.50	105	-	1	76	-	1

Crop	Fortification (mg/kg)	Cyromazine			Melamine		
		Recovery,%	CV,%	n	Recovery,%	CV,%	n
Cotton hulls	0.04	-	-	-	72	-	1
	0.05	77	-	1	-	-	-
	0.50	92	-	1	79	-	1
Cotton meal	0.04	-	-	-	119	-	1
	0.05	77	-	1	-	-	-
	0.30	92	-	1	122	-	1
Cotton oil	0.04	-	-	-	87	-	1
	0.05	92	-	2	71	-	1
	0.20	78	-	1	58	-	1
Cotton seed	0.04	-	-	-	105	16	3
	0.05	83.0	10	5	104	-	2
	0.10	80	-	2	91	-	1
	0.20	79	-	2	87	-	2
	0.30	80	-	2	81	-	2
Cotton soapstock	0.04	-	-	-	67	-	1
	0.05	50	-	1	-	-	-
	0.50	78	-	1	77	-	1
Sudangrass forage	0.04	-	-	-	95	-	1
	0.05	100.5	-	2	73	-	1
	0.10	68	-	1	83	-	1
	0.30	103	21	4	97	20	3
Sudangrass hay	0.04	-	-	-	70	-	1
	0.05	86	-	2	92	-	2
	0.10	97	-	2	81	-	2
	0.20	127	-	1	125	-	1
	0.30	134	-	2	87	-	2

In the Method REM 4/86 (Altenburger, 1986), cyromazine and melamine are extracted from crop material with methanol by blending: an aliquot of the extract is evaporated to dryness and the residue dissolved in 0.1M hydrochloric acid. Clean-up is by consecutive passage through SPE columns (C18 reversed phase, weak cation exchange and strong cation exchange), the analytes eluted with methanol/25% ammonia; further cleaned-up in an amino-SPE cartridge and elution with acetonitrile/water. Analysis and quantitation is by HPLC on a Nucleosil-NH_2 column with UV detection at 215 nm. Recovery data are shown in Table 39.

Table 39. Recovery of cyromazine from fortified crop samples using Analytical Method AG-4/86

Matrix	Fortification (mg/kg)	Cyromazine			Melamine		
		Recovery,%	CV,%	n	Recovery,%	CV,%	n
Cucumber	0.1	104	16.6	4	96	9.1	3
	0.5	99	4.8	4	99	8.3	4
Zucchini	0.1	100	16.2	5	109	29.3	5
	0.5	96	7.0	5	99	9.9	5
Green peppers	0.1	87	14.7	3	72	6.5	3
	0.5	99	-	2	84	-	2
	1.0	65	-	1	-	-	-
Tomatoes	0.1	105	-	2	80	-	2
	0.5	86	-	2	81	-	2
Sugar beet	0.1	94	-	2	110	-	2
	0.5	93	-	2	91	-	2
Onions	0.1	92	-	2	106	-	2
	0.5	91	-	2	94	-	2
Lettuce	0.1	101	-	1	127	-	1
	0.5	109	-	1	109	-	1
Celery	0.1	104	-	1	106	-	1
	0.5	100	-	1	91	-	1

Matrix	Fortification (mg/kg)	Cyromazine			Melamine		
		Recovery,%	CV,%	n	Recovery,%	CV,%	n
Grapes	0.1	85	-	2	117	-	2
	1.0	78	-	2	98	-	2
Melons	0.1	85	-	1	115	-	1
	1.0	83	-	1	93	-	1
Chilli peppers	0.1	71	-	1	99	-	1
	1.0	77	-	1	79	-	1
Peas	0.1	89	-	1	71	-	1
	0.5	100	-	1	99	-	1
Mushrooms	0.1	100	10.9	4	91	-	2
	0.5	87	17.2	3	64	-	1
	1.0	102	-	2	90	15.2	3
Grass	0.1	83	33.0	3	111	10.9	3
	0.5	99	6.1	3	90	5.0	3
Barley grain	0.1	94	-	1	85	-	1
	0.5	79	-	1	76	-	1
Barley straw	0.2	80	-	1	81	-	1
	0.5	92	-	1	95	-	1

In Method REM 174.02 (Lütolf 2001a, b), two extraction methods are used depending on crop type. For crops with high water content and fruits with high acid content, a sample is macerated with an acidic solution of potassium dihydrogen phosphate and extracted with methanol. Crops with high fat content, cereals and other dry crops are extracted first with water by maceration and then, after addition of methanol, by mechanical shaking. Celite is added, the mixture centrifuged, filtered, and the filtrate acidified. Analysis is by column switching HPLC using two cation exchange columns. On the first column (Zorbax 300 SCX), cationic compounds are separated from other co-extractives using a mobile phase of 0.025 M aqueous potassium dihydrogen phosphate adjusted to pH 3. On the second column (Spherisorb S5 SCX) melamine and cyromazine are separated using a mobile phase of 0.04 M aqueous ammonium sulphate adjusted to pH 3. Detection is by UV within the range 215-245 nm depending on the crop type and co-extractives. The results are shown in Table 40. This method was independently validated by Kühne (2004) for tomato and sunflower seeds.

Table 40. Recovery of cyromazine from fortified crop samples using analytical method REM 174.02

Crop	Fortification (mg/kg)	Cyromazine			Melamine		
		Recovery,%	CV,%	n	Recovery,%	CV,%	n
Sunflower seeds	0.05	108	3.79	5	107	2.52	5
	0.5	104	1.45	5	103	1.37	5
	0.05	80	1.4	5	80	4.5	5
	0.5	83	1.1	5	76	1.4	5
Tomatoes	0.05	105	0.67	5	99	5.25	5
	0.5	102	0.87	5	96	0.88	5
	0.05	97	1.7	5	105	2.1	5
	0.5	96	0.9	5	95	1.6	5
Oranges	0.05	101	1.57	5	98	2.21	5
	0.5	102	0.54	5	96	0.57	5
Beans	0.05	87	1.74	5	83	2.31	5
	0.5	90	0.61	5	85	0.00	5
Potatoes	0.05	110	3.19	5	99	2.62	5
	0.5	107	0.78	5	99	0.85	5

Residues in food of animal origin

In method AG-341 (Balasubramanian 1979), cyromazine in eggs or tissues of chickens are homogenised with methanol, an aliquot of the extract is cleaned-up on a cation exchange resin column, followed by silica gel column and finally on a celite column. Cyromazine is eluted from the celite column with diethyl ether saturated with pH 7 buffer. After the organic phase is evaporated to dryness and taken up in methanol, cyromazine is determined by GC-NPD using a glass column packed with 2% Carbowax 20M and 0.5% KOH on Chromosorb W. Alternatively, quantitation is

carried out using MS detection monitoring the M+1 ion (*m/z* 223) of the methyl derivative after on-column methylation with trimethylanilinium hydroxide. Validation data are discussed by Ross (1979) and shown in Table 41.

Table 41. Recovery data for cyromazine obtained during validation of AG-341

Matrix	Fortification level (mg/kg)	Recoveries (%)
Egg white	0.05 0.1 0.2 0.5	85 58, 86 62, 76 71, 73
Egg yolk	0.1 0.2 0.32 0.4 0.5 0.64	53 70, 75 70 91 61 56
Chicken fat	0.1 0.2	70 56
Chicken liver	0.1 0.2	63 55
Chicken meat (breast and thigh)	0.1 0.2	72, 74 76, 82
Chicken skin	0.1 0.2	88 94

In Method REM 22/80 (Schnabel, 1980), cyromazine residues are extracted from animal tissue by maceration with ethanol, filtered, an aliquot concentrated and diluted with sodium chloride solution and re-extracted with ethyl acetate. Fat samples are extracted with n-hexane/acetonitrile. The organic phase is evaporated to dryness and taken up in acetone/methanol for further clean-up on a silica gel column. Liver samples are submitted to an additional clean-up with a florisil column. Cyromazine is determined by GC-NPD using a glass column packed with 3% OV 101 and 3% Carbowax 20 M. Recoveries values are displayed in Table 42.

Table 42. Recovery data for cyromazine obtained during validation of REM 22/80

Matrix	Fortification level (mg/kg)	Recoveries (%)		CV (%)	n
		Mean	Range		
Cattle muscle	0.025 0.05 0.20 1.00	126 119 91 88	108, 144 103, 135 80 - 98 85 - 91	- - 9 3	2 2 4 4
Cattle kidney	0.025 0.20 1.00	99 93 86	94, 104 75 - 112 86	- 22 -	2 4 1
Cattle liver	0.025 0.20 1.00	130 88 90	120, 140 849 - 92 90	- 4 -	2 4 2
Cattle fat	0.025 0.20 1.00	96 93 102	84, 108 77 - 102 98, 105	- 13 -	2 4 2

The Method AG-364 was validated for cyromazine and melamine in poultry muscle, liver, fat skin and eggs (Williams and Balasubramanian, 1982). Samples are blended with dry ice and residues are extracted in methanol/water. An aliquot is cleaned up on Dowex 50W-X4 cation exchange resins, eluting the analytes with concentrated ammonium hydroxide/methanol, followed by a celite column, eluting the analytes with ethyl acetate/methanol. The compounds are analysed by GC/MS in the chemical ionisation mode, cyromazine on the *m/z* 167 ± 0.2 ion and melamine on the *m/z* 127 ± 0.2 ion. Validation data is given by Ross (1982) and are shown in Table 43.

Table 43. Recovery of cyromazine and melamine from poultry tissue and eggs using Method AG-364

Matrix	Fortification (mg/kg)	Cyromazine Recovery,%	Melamine Recovery,%
Eggs	0.04	-	98
	0.05	-	93
	0.10	77	-
	0.15	-	79
	0.20	71, 71	78
	0.40	-	72
Chicken fat	0.04	-	71, 81
	0.10	58	-
	0.15	-	73
	0.20	68	-
Chicken skin	0.04	-	73
	0.10	94, 100	-
	0.15	-	94, 94
	0.20	101, 98	-
Chicken lean meat	0.04	-	108, 66
	0.10	87	-
	0.15	-	64, 66
	0.20	65	-
Chicken liver	0.04	-	74, 65
	0.10	74	-
	0.15	-	86, 97
	0.20	51	-

In the analytical method "R&D. 204" (Anonymous, 1984a) cyromazine is extracted from chicken eggs by maceration of the samples with acetone. The extract is cleaned up by partitioning with acetonitrile, acidified with hydrochloric acid, salinated with sodium chloride and washed with ethyl acetate. The aqueous phase is basified with 10N sodium hydroxide solution and cyromazine is re-extracted into ethyl acetate. Cyromazine residues are determined by GC-NPD using a glass column packed 1:1 with 2% NPGS and 2% FFAP on Gas Chrom Q as a stationary phase and a nitrogen/hydrogen/air mixture as a mobile phase. Recoveries at 0.1 and 1 mg/kg were 70 and 72%, respectively.

In the analytical method "R&D. No. 205" (Anonymous, 1984b), cyromazine is extracted from chicken muscle by maceration of the samples with methanol/water. The suspension is acidified with hydrochloric acid, salinated with sodium chloride and evaporated to a volume of 10 mL. The concentrate is neutralised by adding 10N sodium hydroxide solution followed by a clean-up step on an Extrelut cartridge. Cyromazine is eluted from the cartridge with ethyl acetate and partitioned back into hydrochloric acid saturated with sodium chloride. The acidic extract is made alkaline and re-extracted into ethyl acetate. Cyromazine residues are determined by GC-NPD using a glass column packed 1:1 with 2% NPGS and 2% FFAP on Chromosorb G as a stationary phase and a nitrogen/hydrogen/air mixture as a mobile phase. Recoveries of cyromazine from fortified muscle samples at the 0.1 mg/kg or 1.0 mg/kg level are 81% or 70%, respectively.

In the analytical method "R&D. No. 213" (Anonymous, 1985), cyromazine and melamine residues are extracted from chicken muscle and eggs by maceration of the samples with methanol. The filtrate is partitioned with hexane; the methanol extract was then concentrated, basified and cleaned up on an Extrelut column. Residues are eluted with ethyl acetate and further cleaned-up on a silica Sep Pak followed by ion-exchange chromatography using anionic and cationic resins. Quantitation is performed by HPLC on a C_{18} column with an acetonitrile/water mobile phase and UV detection at 208 nm. Recoveries are shown in Table 44.

Table 44. Recovery data for cyromazine in chicken muscle and eggs using method R&D 213

Matrix	Fortification level (mg/kg)	Recoveries (%)	
		Cyromazine	Melamine
Chicken muscle	0.1	89	88
	1.0	75	77
Chicken eggs	0.1	90	87
	1.0	100	99

In Method AG-403 (Williams and Burge, 1983), residues of cyromazine and melamine are extracted with 90% methanol water (eggs) or 90% acetonitrile/water (meat and milk), cleaned up on a C18 column, cation exchange column and automatic GPC. A second cation exchange column is used to rapidly concentrate the aqueous eluate of the gel permeation step. The compounds are determined by HPLC on a NH_2 column using acetonitrile/water as mobile phase and UV detection at 214 nm. Validation data are given in Ross (1983) (Table 45).

Table 45. Recovery of cyromazine from animal tissues and products using Method AG-403

Matrix	Fortification (mg/kg)	Cyromazine Recovery,%	Melamine Recovery,%
Eggs	0.05	75	74
	0.10	64	92
	0.50	74	85
	0.56	82	-
	67	-	85
	1.0	81	92
Lean meat (chicken)	0.05	61, 87	76, 83
	0.50	77, 94	77, 84
	0.67	84	-
	0.70	73	-
	0.80	-	73, 80
	1.0	89	79
Beef liver	0.05	98	81
	0.10	83	80
	0.20	121	68
	0.69	97	-
	0.80	-	50
Milk (cow)	0.01	95	105
	0.10	81	90
	0.70	71	-
	0.80	-	78
Milk (goat)	0.01	78	57
	0.20	79	73

Methods AG-417 and AG-417A (Smith et al. 1983) differs from the previous method only in the clean-up step. Cyromazine and melamine residues are extracted in methanol/water (eggs) or acetonitrile/water (meat), cleaned up in a C-18 column, a cation exchange column and an anion exchange column. A second cation exchange column is used to concentrate the aqueous eluate from the anion exchange column, and residues are determined by HPLC/UV detection at 214 nm using an amino column. Validation data were reported by Cheung (1984a) and are shown in Table 46.

Table 46. Recovery from animal tissues and products using Method AG-417

Matrix	Fortification (mg/kg)	Cyromazine			Melamine		
		Recoveries,%	CV,%	n	Recoveries,%	CV,%	n
Chicken muscle	0.05	69 - 113	21.3	4	82 – 112	15.9	4
	0.20	74, 97	-	2	89, 89	-	2
	0.25	93	-	1	78	-	1
	0.30	99 – 109	5.0	4	98 – 110	5.5	4
	0.40	95	-	1	88	-	1
	0.50	92, 103	-	2	78, 94	-	2
Chicken Liver	0.05	88	-	1	102	-	-
	0.20	72	-	1	82	-	-
Chicken eggs	0.05	76 – 115	18.9	8	70 – 119	14.5	8
	0.10	84	-	1	113	-	1
	0.20	71 – 109	15.6	4	87 – 113	12.2	4
	0.30	77	-	1	84	-	1
	0.40	80 – 93	7.0	3	74 – 101	14.8	3
	0.50	90	-	1	96	-	1
	1.00	93	-	1	97	-	1

In Method REM 7/83 (Giannone and Formica, 1983)**,** cyromazine residues are extracted from sheep muscle, kidney, or liver by maceration with 90% methanol/water, the extract is concentrated and partitioned with dichloromethane. The aqueous solution is transferred to a partition column and cyromazine eluted with ethyl acetate and methanol. After evaporation of the organic phase in the presence of ethylene glycol, the residue is cleaned up on a silica gel column. Samples of sheep fat are extracted with hexane followed by partitioning with acetonitrile. An aliquot of the acetonitrile phase is evaporated and the residue dissolved in an alkaline solution of sodium chloride, which is then transferred to a partition column as described before. Quantitation is performed by HPLC using a LiChrosorb-NH_2 column with an acetonitrile/water mobile phase and UV detection at 215 nm. Recoveries values are displayed in Table 47.

Table 47. Recovery data for cyromazine obtained during validation of REM 7/83

Matrix	Fortification level (mg/kg)	Recoveries (%)		CV (%)	n
		Mean	Range		
Sheep muscle	0.04	84	72 - 93	12	4
	0.40	89	85 - 92	5	4
Sheep kidney	0.04	71	62 - 79	11	4
	0.40	76	62 - 86	14	4
Sheep liver	0.04	83	70 - 100	19	4
	0.40	77	72 - 85	7	4
Sheep fat	0.04	105	84 - 150	29	4
	0.40	81	76 - 90	8	4

Method REM 4/86 reported by Altenburger (1986) for vegetal crops was modified to quantify cyromazine and melamine residues in muscle and liver. The modifications included omitting the clean-up on the weak cation and weak anion exchange and replacing the SCX-Bondelut cartridges was replaced by PRS-Bondelut columns (both strong cation exchange columns). Determination was by HPLC with an amino column and UV detection at 215 nm. Validation data are presented by Altenburger (1987) (Table 48).

Table 48. Recovery data for cyromazine in chicken tissues using method REM 4/86

Matrix	Fortification level (mg/kg)	Recoveries (%)	
		Cyromazine	Melamine
Chicken liver	0.5	81	116
	2.5	75	77
Chicken muscle	0.1	90	72
	0.5	82	70

Method 99-146 was summarized by Inoue (2000) and describes analysis of cyromazine in chicken eggs, plasma, small intestine, skin, muscle, fat, liver, and kidneys. Samples are homogenised and extracted with ethanol, centrifuged and the supernatant evaporated to dryness. The residue is re-dissolved in acetonitrile-saturated n-hexane followed by consecutive clean-up on C_{18} and silica cartridges. Residues are eluted with methanol, evaporated to dryness and taken up in 4 ml mobile phase for HPLC separation on an amino column followed by UV detection at 215 nm. Recovery data are shown in Table 49.

Table 49. Recovery data for cyromazine using analytical method 99-146 fortified at 0.33 mg/kg (n=3)

Matrix	Recovey, mean (%)	Recoveries Range (%)	CV (%)
Chicken muscle	85	83-87	2.4
Chicken liver	90	89-91	1.3
Chicken kidney	84	82-85	1.8
Chicken fat	95	95-96	0.6
Chicken skin	84	82-87	3.0
Egg yolk	81	80-81	0.7
Egg white	89	87-91	2.3

In Method RAM 394/01 (Kwiatkowski, 2003) cyromazine and melamine residues are extracted from liver, kidney, muscle tissue and milk by homogenisation with acetonitrile/water and from eggs with methanol/water. After centrifugation, aliquots are adjusted to pH 4 with glacial acetic acid and subjected to SPE cation exchange clean-up. The compounds are eluted in 5% ammonia in acetonitrile, the eluate evaporated and the residue taken up in acetonitrile/water. Final determination is by HPLC-MS/MS using matrix-matched standards. Validation data is shown in Table 50.

Table 50. Recovery data for cyromazine obtained during validation of RAM 394/01

Matrix	Fortification level (mg/kg)	Cyromazine			Melamine		
		Recoveries,%	$CV_{,\%}$	n	Recoveries,%	$CV_{,\%}$	n
Bovine milk	0.01	94-98	2.1	5	86-98	5.9	5
	0.1	84-95	4.7	5	84-90	2.6	5
Bovine muscle	0.01	87-98	4.6	5	96-137	16.1	5
Chicken eggs	0.01	89-98	4.5	5	90-103	5.5	5
	0.1	83-91	3.3	5	90-94	2.0	5
Chicken kidney	0.01	85-93	3.8	5	87-102	7.0	5
	0.1	82-90	3.8	5	93-99	3.2	5
Chicken liver	0.01	90-107	5.5	5	83-108	10.2	5
	0.1	82-96	6.4	5	87-97	4.3	5
	0.1	85-93	4.3	5	98-106	3.4	5

Method RAM394/01 was independently validated by Kang (2004) on bovine muscle tissue, bovine milk and chicken eggs (Table 51).

Table 51. Recovery data during independent validation of Method 394/01

Matrix	Fortification (mg/kg)	n	Cyromazine		Melamine	
			Recoveries,%	CV,%	Recoveries,%	CV,%
Bovine milk	0.01	5	97-108	4.0	98-105	2.7
	0.10	5	86-94	3.7	77-80	1.7
Chicken eggs	0.01	5	96-101	1.9	92-96	2.0
	0.10	5	88-94	2.7	72-76	2.0
Bovine muscle	0.01	5	70-75	2.7	93-103	4.4
	0.10	5	74-77	1.7	78-82	2.0

Stability of residues in stored analytical samples

The storage stability of field-incurred residues of cyromazine and its metabolite melamine in head lettuce, leaf lettuce, celery, mushrooms and tomatoes was tested by Cheung (1995). The crops were treated with multiple applications of cyromazine and samples taken at a 0 day PHI, except for the mushrooms where the sampling was made 75 days after the soil (compost) was amended with the treatment. Samples were analysed at 0 day and then after storage at ≤ 18 °C for 9.5 to 24 months depending upon the crop. Residues of cyromazine in the mushroom samples were < LOQ. The results are shown in Table 52. In all cases, mean procedural analytical recovery were in the range of 84 to 98%.

Table 52. Stability of field-incurred residues in under freezer storage conditions

Crop, storage interval (months)	Cyromazine		Melamine	
	Mean residue before storage (mg/kg)	% remaining after storage	Mean residue before storage (mg/kg)	% remaining after storage
Head lettuce, 23	6.0	148	0.62	177
	15.5	97	1.3	108
	21.5	112	1.5	107
Leaf lettuce, 23	8.5	153	0.44	220
	12	117	0.75	160
	16	144	0.61	180
Celery, 24	0.96	53	0.67	61
	2.0	125	1.1	136
	3.5	157	1.1	145
Tomato, 9.5	0.07	142	0.06	167
	0.49	120	0.41	107
Mushroom, 11	< LOQ	-	3.6	97
			9.3	85

The stability of cyromazine and melamine in freezer-stored samples of mangoes fortified at 0.5 mg/kg level was tested by Hofherr (1995). Samples were analysed at 0, 1, 3, 6, 12 and 24 months. With each analysis date one untreated control specimen and two freshly prepared fortified specimens were analysed in parallel. Results are summarized in Table 53.

Table 53. Stability of residues in fortified mangoes fortified at 0.5 mg/kg under freezer storage conditions

Storage period (Months)	Cyromazine residue		Melamine residue	
	Mean procedural recovery (%) [a]	Mean% remaining [b,c]	Mean procedural recovery (%) [a]	Mean% remaining [b,c]
0	88	107	104	115
3	80	97	95	83
7	75	93	79	88
12	89	94	78	95
19	74	93	73	103
24	103	112	125	108

a - Mean recovery from 2 freshly fortified samples;

b - Recoveries are corrected for mean procedural recoveries;

c - Mean of 3 replicates

The stability of cyromazine and melamine residues was studied by Giannone (2003) in freezer-stored samples of tomatoes, potatoes, haricot beans and sunflower seeds fortified at 1 mg/kg. Samples were stored in polythene containers at ≤ -18°C for up to two years. Results for cyromazine and melamine are presented in Table 54. The mean procedural analytical recovery for cyromazine or melamine in all sampling periods ranged from 90 – 111%.

Table 54. Stability of residues in fortified at 1 mg/kg under freezer storage conditions

Matrix Months	Cyromazine		Melamine	
	Mean % remaining	CV,% (n=3)	Mean % remaining	CV,% (n=3)
Tomato 0	103	5.7	104	5.2
3	114	8.2	110	8.6
7	80	14.7	94.2	4.4
12	90.3	15.9	101	6.9
19	89.1	2.0	96.4	3.0
24	102	0.9	106	0.5
Potato 0	106	3.2	105	2.3
3	106	11.2	104	13.1
7	94.2	4.0	96.6	4.7
12	103	0.1	101	0.5
19	89.8	1.0	90.3	0.7
24	110	1.3	116	1.2
Bean 0	99.8	3.0	95.2	0.8
3	95.1	5.1	87.6	9.0
7	89.3	2.9	74.3	8.0
12	89.5	9.0	89.3	12.4
19	77.9	6.3	76.5	8.4
24	117	4.2	111	5.1
Sunflower 0	99.7	3.2	99.9	3.8
3	96.5	7.3	82	2.6
7	88.7	4.6	84.7	6.6
12	91.7	1.3	85.9	2.2
19	85.2	2.4	86.2	4.2
24	83.9	3.0	99.1	2.1

The storage stability of cyromazine and its animal metabolites, melamine and 1-methylcyromazine, was studied in meat, milk and eggs under freezer storage conditions by Eudy (1994). Homogenised samples of beef muscle, beef liver and poultry eggs were spiked with cyromazine and melamine at 0.50 mg/kg; milk was fortified at 0.10 mg/kg. Likewise, beef muscle and liver were fortified with 1-methylcyromazine at 0.50 mg/kg; milk was fortified at 0.10 mg/kg. Samples were analysed at 0-day and 2, 13, 18, and 24-month intervals. Results for cyromazine, melamine and 1-methylcyromazine are presented in Table 55. Mean procedural analytical recovery were in the ranged of 80 – 94%, except for melamine in beef liver (55.6%).

Table 55. Storage stability of residues in samples fortified at 0.5 mg/kg or 0.10 mg/kg (milk)

Matrix	Days	Mean% remaining[a]		
		Cyromazine	Melamine	1-methylcyromazine
Beef muscle	0	66.4	72.2	81.6
	76	84	84	89.6
	400	128.2	103.2	86.8
	554	84.4	75.4	87.6
	723	81	72.8	97
Beef liver	0	76.4	63	78.2
	76	69.4	40.2	82.6
	392	80.4	53.4	79.2
	543	83.4	49.8	83.6
	720	69	51.4	79
Eggs	0	82.8	83.6	-
	78	99.4	87.8	-
	406	72.4	69.8	-
	546	93	89	-
	727	94.8	88.4	-
Milk	1	72	78	90
	76	104	86	83
	409	113	98	89
	549	94	108	87
	729	97	86	93

a - two replicate samples

USE PATTERNS

A summary of the critical use patterns of cyromazine in plants are presented in Table 56. A WP 75% ai formulation is registered in all cases, except for mushroom in the USA, where a 5% ai formulation is registered. The products can be used in the field (F) or protected (P).

Table 56. Use patterns of cyromazine in crops using a WP formulation

Crop	Country	Application				PHI , days
		Method	kg ai/hL	kg ai/ha	Maximum number (interval, days)	
Mango	Mexico	F, foliar		0.07-0.10	NS (14)	0
Bulb vegetables	USA	F, foliar	-	0.140	6 (7)	7
		F, seed treatment	-	5 g ai/100g seed	1	-
Brassica leafy	USA	F, foliar	-	0.140	6 (7)	7
Cucumber	France	F or P, foliar	-	0.3	NS (10-15)	3
	Greece	F, foliar	0.015-0.022	-	3 (7)	14
		P, foliar	0.015-0.022	-	3 (7)	7
	Italy	F, foliar	0.02-0.03	0.2-0.3	NS (7-14)	14

Crop	Country	Application				PHI , days
		Method	kg ai/hL	kg ai/ha	Maximum number (interval, days)	
		P, foliar	0.02-0.03	-	NS (7-14)	14
	Switzerland	P, foliar	0.015	0.15-0.3	NS (7)	3
Cucurbits	USA	F, foliar		0.14	6 (7)	0
Summer squash	France	F or P, foliar	-	0.3	NS (10-15)	3
	Italy	F, foliar	0.02-0.03	0.2-0.3	NS (7-14)	14
		P, foliar	0.02-0.03	-	NS (7-14)	14
	Spain	F, foliar	0.015-0.03	-	NS (14-30)	3
Melon	France	F or P, foliar	-	0.3	NS (10-15)	7
	Greece	F, foliar	0.015-0.022	-	3 (7)	14
		P, foliar	0.015-0.022	-	3 (7)	7
		F, Soil irrigation		0.98	1	14
		P, Soil irrigation		0.98	1	7
	Italy	F, foliar	0.02-0.03	0.2-0.3	NS (7-14)	14
		P, foliar	0.02-0.03	-	NS (7-14)	14
		Soil irrigation		0.75	1	14
	Spain	F, foliar	0.015-0.03	-	NS (14-30)	3
Tomato	France	F or P, foliar	-	0.3	NS (10-15)	3
	Greece	F, foliar	0.015-0.022	-	3 (7)	14
		P, foliar	0.015-0.022	-	3 (7)	7
		F, Soil irrigation		0.98	1	14
		P, Soil irrigation		0.98	1	7
	Italy	F, foliar	0.02-0.03	0.2-0.3	NS (7-14)	14
		P, foliar	0.02-0.03	-	NS (7-14)	14
		Soil irrigation		0.75	1	14
	Spain	F, foliar	0.015-0.03	-	NS (14-30)	3
	Switzerland	P, foliar	0.015	0.15-0.3	NS (7)	3
	USA	F, foliar	-	0.14	6 (7)	0
Eggplant	France	F or P, foliar	-	0.3	NS (10-15)	3
	Greece	F, foliar	0.015-0.022	-	3 (7)	14
		P, foliar	0.015-0.022	-	3 (7)	7
	Italy	F, foliar	0.02-0.03	0.2-0.3	NS (7-14)	14
		P, foliar	0.02-0.03	-	NS (7-14)	14
Peppers	USA	F, foliar	-	0.14	6 (7)	0
Mushrooms	Spain	Spray to casing soil/compost	-	0.38-0.75g/m^2	NS (-)	15
	France	Soil/compost		0.4 g g/m^2	NS (-)	14
	Switzerland	Spray to casing soil/compost	-	0.5-1 g/m^2 0.5-1 g/kg compost	NS (-)	14
	USA	Coarse drenching spray	-	0.57g/ m^2 (5 mg/kg)[a]	-	-
Lettuce	France	F, foliar	-	0.3	NS (15)	21
	Italy	F, foliar	0.02-0.03	0.2-0.3	NS (7-14)	14
	Spain	F, foliar	0.015-0.03	-	NS (14-30)	7
	Switzerland	F, foliar	0.02	0.2	NS (7)	14
		P, foliar	0.02	0.2-0.4	NS (7)	7
Leafy vegetables, including brassicas	USA	F, foliar	-	0.14	5 or 6[a] (7) [a] celery and head lettuce)	7
Lima bean	USA	F, foliar	-	0.14 Total= 0.74	6 (7)	7

Crop	Country	Application				PHI , days
		Method	kg ai/hL	kg ai/ha	Maximum number (interval, days)	
Dry bean	USA	F, foliar	-	0.14 Total= 0.84	6 (7)	7
Potato	Spain	F, foliar	0.02	0.12-0.24	NS (14-30)	21
	Italy	F, foliar	0.02-0.03	0.2-0.3	NS (7-14)	35
		P, foliar	0.02-0.03	-	NS (7-14)	35
	USA			0.14-0.28 Total= 0.84	NS (7)	7
Artichoke	Spain	F, foliar	0.015-0.03		NS (14-30)	7
	Greece	F, foliar	0.015-0.022	-	3 (7)	14
		P, foliar	0.015-0.022	-	3 (7)	7
Celery	France	F or P, foliar	-	0.3	NS (10-15)	14
	Greece	F, foliar	0.015-0.022	-	3 (7)	14
		P, foliar	0.015-0.022	-	3 (7)	7

NS= not specified;

a - 95% SC formulation; rate will depending on the wet compost bed

Cyromazine is registered in Australia and the USA for the feed-through use in poultry as an insect growth regulator to control larvae of nuisance flies in manure and for protection of sheep against blowfly strike when applied as a topical treatment. Cyromazine is effective against the house fly (*Musca domestica*), the lesser house fly (*Fannia canicularis*), the false stable fly (*Muscina stabulans*), and the American soldier fly (*Hermetia illucens*). The use patterns are shown in Table 57.

Table 57. Use patterns of cyromazine in animal in Australia and the USA

Target species	Use	Formulation	Maximum application	Application period	Withholding period [days]
Australia					
Poultry (broilers, breeders, layers)	Feed-through	FAP 01 or TK 10	5 mg ai/ kg feed	feed continuously for 4-6 weeks, to be repeated when flies re-establish	Meat: 3 Eggs: 0
Sheep	Spray-on	AL	1-5 g ai/animal	Do not use in sheep which are producing or may produce milk	Meat = 7 Milk = not for consumption
	Dip or spray	SC	0.1 kg ai/hL	Do not use in sheep which are producing or may produce milk	Meat = 7 Milk = not for consumption
USA					
Poultry	Blending and feeding		5 mg ai/ kg feed	feed continuously for 4-6 weeks, to be repeated when flies re-establish	Meat: 3

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

Supervised residue trials conducted with cyromazine in Europe, the USA and Mexico in mango, melon and a variety of vegetable crops were submitted to the Meeting. Table 58 summarizes the data. All studies were conducted according to GLP and most of the reports submitted included detailed information on trial conditions and analytical method validation. The trials were conducted in the field (F) or protected in a glasshouse (P). Unless specified, in all trials concurrent determinations of residues in untreated crops gave residues < LOQ. All trials were conducted using a 75 WP formulation and unless specified, using foliar application.

In most of the trials both cyromazine and melamine were determined in the crops, but with the exception of the trials on mushrooms, only residues of cyromazine are reported. Residues within 30% GAP are underlined and were considered for recommendations on STMR, HR and MRL. When

residues in samples harvested at a latter stage were higher than what was found at the critical PHI, they were selected for the recommendations.

Table 58. Summary of supervised trial conducted with cyromazine submitted

Crop	No. of trials	Country or Region	Table
Mango	6	Mexico	59
Bulb onion	20	USA	60
Spring onion	4	USA	61
Broccoli	6	USA	62
Cabbage	6	USA	62
Mustard green	5	USA	62
Cucumber	26	Europe and USA	63
Summer squash	20	Europe and USA	64
Melon	25	Europe and USA	65
Watermelon	2	USA	65
Tomato	36	Europe and USA	66
Eggplant	4	Europe	67
Peppers	12	USA	68
Mushroom	9	Europe	69
Lettuce	66	Europe and USA	70
Spinach	16	USA	71
Mustard greens	5	USA	72
Beans, succulent	14	USA	73
Beans, dry	9	USA	74
Potato	8	Spain	75
Artichokes	4	Spain	76
Celery	23	Europe and USA	77

Mango

Six field trials were carried out in Mexico. Residues in mango were analysed using method AG-408 (HPLC-UV, LOQ=0.05 mg/kg). Full validation of this method was not provided to the Meeting. The reports for mango did not include an analytical validation. A summary of the residue data is given in Table 59.

Table 59. Summary of cyromazine residues in mango from trials conducted in Mexico

Variety, year	Application				Residue	Reference
	No.	kg ai/hL	kg ai/ha	PHI (days)	[mg/kg]	
Kent, 1984	5	0.02	0.095-0.096	0	0.07	Hofherr, 1994a; Report: 1069/93[a]
				7	0.04	
				14	0.10	
				21	0.09	
				28	0.08	
Petacon Kent, 1993	5	0.02	0.095-0.098	0	0.25	Hofherr, 1994b; Report: 1065/93[a]
				7	0.07	
				17	< 0.05	
				21	< 0.05	
Petacon Kent, 1993	5	0.02	0.098	0	0.14	Hofherr, 1994c Report: 1066/93[a]
				7	0.08	
				14	< 0.05	
				21	0.11	
				28	0.07	
Kent, 1993	5	0.02	0.094-0.097	0	0.10	Hofherr, 1994d

Variety, year	Application				Residue	Reference
	No.	kg ai/hL	kg ai/ha	PHI (days)	[mg/kg]	
				7	0.10	Report: 1068/93[a]
				14	0.11	
				21	0.09	
				28	0.10	
Petacon Kent, 1993	5	0.02	0.093-0.095	0	0.14	Hofherr, 1994e
				7	0.05	Report: 1067/93
				14	0.06	
				21	0.10	
				28	0.06	
Kent, 1993	5	0.02	0.094-0.095	0	< 0.05	Hofherr, 1994f
				7	0.06	Report: 1070/93
				14	< 0.05	
				21	< 0.05	
				28	< 0.05	

a - Only a summary of the report was submitted.

Bulb onion

Twenty residue trials on bulb onions were performed during in the US in 1993 and 1999 using foliar or seed application of cyromazine. Samples from foliar treatment were analysed using either an HPLC-UV (foliar) or GC-NPD (seed) method. A summary of the residue data is given in Table 60.

Table 60. Cyromazine residues in bulb onion from trials conducted in the USA

State, variety, year	Application			PHI, days	Portion analysed	Residues, mg/kg [a]	Reference (Report No.)
	Treatment	No.	kg ai/ha or g/100g seed				
NY, Buffalo F-1, 1993	Seed	1	5	61	Whole Plant	0.44 [a]	Moore, 1995 (ABR-94077)
				98	Bulbs	< 0.05[a]	
				98	Dried bulb	< 0.05 [a]	
			10	61	Whole Plant	0.76	
				98	Bulbs	< 0.05	
				98	Dried bulb	< 0.05	
MI, Extra, 1993	Seed	1	5	60	Whole Plant	0.34 [a]	
				104	Bulbs	< 0.05[a]	
				104	Dried bulb	< 0.05 [a]	
TX, Collosal, 1993	Seed	1	5	60	Whole Plant	0.35 [a]	
				165	Bulbs	< 0.05[a]	
				165	Dried bulb	< 0.05[a]	
GA, Collosal, 1993	Seed	1	5	61	Whole Plant	0.09 [a]	
				207	Bulbs	< 0.05[a]	
				207	Dried bulb	< 0.05 [a]	
CA, Rogers Foods, 1993	Seed	1	5	60	Whole Plant	0.83 [a]	
				157	Bulbs	0.06 [a]	
				157	Dried bulb	0.06 [a]	
				157	Flakes, dehydrated	0.07 [a]	
			10	60	Whole Plant	1.9	
				157	Bulbs	0.13	
				157	Dried bulb	< 0.05	
				157	Flakes, dehydrated	0.09	
CO, Ferry-Morse,	Seed	1	5	60	Whole Plant	1.7[a]	Moore, 1995

State, variety, year 1993	Application			PHI, days	Portion analysed	Residues, mg/kg [a]	Reference (Report No.)
	Treatment	No.	kg ai/ha or g/100g seed				(ABR-94077)
				138	Bulbs	< 0.05[a]	
				138	Dried bulb	< 0.05 [a]	
OR, Sweet perfection 1993	Seed	1	5	60	Whole Plant	0.16 [a]	
				147	Bulbs	< 0.05[a]	
				147	Dried bulb	< 0.05 [a]	
	Seed		10	60	Whole Plant	0.39	
				147	Bulbs	< 0.05	
				147	Dried bulb	< 0.05	
Sweet perfection 1993	Seed	1	5	75	Whole Plant	0.27 [a]	
				146	Bulbs	< 0.05[a]	
				146	Dried bulb	< 0.05 [a]	
NJ, Festival, 1999	Foliar	6	0.144	7	Bulbs	< 0.05 [a]	Markle, 2002a;
TX, 1015, 1999	Foliar	6	0.14 - 0.15	7	Bulbs	< 0.05 [a]	(IR4-07239)
OH, Burgos, 1999	Foliar	6	0.13 – 0.14	7	Bulbs	< 0.05 [a]	
IN, Yellow Sweet Spanish, 1999	Foliar	6	0.13 -0.14	7	Bulbs	< 0.05 [a]	
WA, Salem, 1999	Foliar	6	0.14	6	Bulbs	< 0.05 [a]	
OR, Santos F1, 1999	Foliar	6	0.14	6	Bulbs	< 0.05 [a]	
CO, Candy, 1999	Foliar	6	0.13- 0.15	6	Bulbs	< 0.05 [a]	Markle, 2002a;
CA, Hybrid T-406, 1999	Foliar	6	0.14	7	Bulbs	0.07 [a]	(IR4-07239)
CA, Early Red Burger, 1999	Foliar	6	0.14	7	Bulbs	0.07 [a]	

a - Mean of duplicate samples

Spring or green onions

Four residue trials on spring onions were performed in the United Sates during 1999–2000. Samples were analysed using method AG-621 (GC-NPD). A summary of the residue data for spring onion is given in Table 61.

Table 61 Cyromazine residues in spring onion (whole plant) from foliar trials conducted in the USA (Markle, 2002; Report No. IRA4-07238)

State, year, variety	Application		PHI, days	Residue, mg/kg a
	No.	kg ai/ha		
NJ, 1999, Bunching	6	0.17 - 0.14	8	0.30
FL, 1999, White Spear Bunching,	6	0.14	7	0.75
CA, 1999, White Spear Bunching,	6	0.14	8	0.26
HI, 2000, Koba	6	0.14	7	0.78

a - Mean of duplicate samples

Broccoli and cabbage

Seventeen supervised residue trials were carried out on brassicas in the USA, six in broccoli and six in cabbage. A summary of the residue data is given in Table 62.

Table 62. Cyromazine residues in brassica from trials conducted in the USA in 1997 using six applications at a 0.142 kg ai/ha rate (Eudy, 1999; Report No. 42-97)

Crop, state, variety	Portion analysed	PHI, days	Residue, mg/kg[a]
Broccoli, CA, Decicco	flower head and stem	0 7	1.96 0.21

Crop, state, variety	Portion analysed	PHI, days	Residue, mg/kg[a]
Broccoli, TX, So. Comet Veg	flower head and stem	0 7	0.91 0.26
Broccoli, CA, Arcadia	flower head and stem	0 7	0.36 0.09
Broccoli, AZ, HMX 7144	flower head and stem	0 7	0.66 0.51
Broccoli, OR, Gem. Hybrid	flower head and stem	0 7	0.18 0.05
Broccoli, CA, Marathon	flower head and stem	0 1 3 5 7 9	0.26 0.28 < 0.05 < 0.05 < 0.05 < 0.05
Cabbage, CA	head with wrapper leaves	0 7	5.8 6.1
Cabbage, FL	head with wrapper leaves	0 7	2.6 0.28
Cabbage, TX	head with wrapper leaves	0 7	0.56 0.10
Cabbage, NC	head with wrapper leaves	0 7	1.2 0.06
Cabbage, WI	head with wrapper leaves	0 7	1.7 0.24
Cabbage, CA	head with wrapper leaves	0 1 3 5 7	1.1 0.7 1.0 0.30 0.50

a - Mean of duplicate samples

Cucumbers

A total of 26 supervised residue trials were carried out in cucumbers in Europe and in the USA. Cyromazine was applied in the field or in the glasshouse at rates varying from 0.14 to 0.34 kg ai/ha. The results are shown in Table 63.

Table 63. Cyromazine residues in cucumbers from trials conducted in Europe and the USA

Country, year, variety	Application				PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha			
France, 1999, Avalon	Protected	3	0.04	0.3	0 3 7	0.67[a] 0.50 [a] 0.48 [a]	Lütolf, 2000a (1107/98)
France, 1999, Avalon	Protected	3	0.03	0.31	0 3 7 14	0.33 0.46 [a] 0.30 0.25	Lütolf, 2000b (1012/99)
France, 2000, Serenade	Protected	3	0.03	0.3-0.34	0 3 7	0.36 0.30 [a] 0.25	Lütolf, 2002a (1059/00)
France, 2000, Serenade	Protected	3	0.03	0.31-0.34	0 3	0.48 0.52 [a]	Lütolf, 2002b (1060/00)

Country, year, variety	Application				PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha			
					7	0.24	
France, 2000, Prestige	Protected	3	0.03	0.32-0.33	0 3 7 14	0.46 <u>0.52</u> 0.50 0.26	Pointurier, 2001a (33002)
France, 2000, Prestige	Protected	3	0.03	0.33-0.34	0 3 7 14	0.35 0.22 [a] <u>0.32</u>[a] 0.07	Pointurier, 2001b (33001)
Greece, 2001, Deltastar	Protected	3	0.03	0.33	0 1 3 7 14	0.90 0.86 <u>1.30</u> [a] 1.10 <u>0.57</u>	Bourry, 2002a (1058/01)
Greece, 2001, Aris	Protected	3	0.03	0.33	0 1 3 7 14	1.40 0.89 <u>0.88</u> [a] 0.72 0.53	Lütolf, 2002c (1057/01)
Italy, 1999, Colorado	Field	3	0.03	0.33	0 1 3 7 14	1.15 1.07 <u>0.74</u> 0.67 0.35 [a]	Lütolf, 2000c (1046/99)
Italy, 2000, Early Set	Field	4	0.03	0.33	0 1 3 7 14	0.41 0.36 0.30 [a] <u>0.33</u> 0.19	Lütolf, 2001c (1064/00)
Italy, 2000, Ashley	Field	4	0.03	0.33-0.34	0 1 3 7 14	0.71 0.59 <u>0.71</u> [a] 0.70 0.40	Lütolf, 2001d (1065/00)
Italy, 2000, Darina	Field	4	0.03	0.33-0.34	0 3	0.36 <u>0.62</u> [a]	Lütolf, 2002d (1066/00)
Italy, 2000, Lorac	Protected	4	0.03	0.33	0 1 3 7 14	0.88 0.84 <u>0.79</u> [a] 0.74 0.42	Lütolf, 2001d (1063/00)
Spain, 2000, Slice Master	Protected	4	0.03	0.32-0.34	0 1 3 7 14	0.36 0.44 <u>0.40</u> [a] 0.27 0.19	Lütolf, 2002e (1062/00)
Switzerland, 1999, Thyria	Protected	3	0.03	0.33	0 3 7 14	0.30 0.38 [a] <u>0.43</u> 0.23	Lütolf, 2000d (,1063/99)

Country, year, variety	Application				PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha			
USA (CA), 1986, Marketmore	Field	8		0.140	0 6 14 21	0.56 [a] 0.17[a] 0.08 < 0.05	Hackett, 1991 (ABR-91042)
				0.280	0 6 14 21	0.22 0.17 0.11 0.07	
USA (NE), 1986, Marketmore 70	Field	8		0.140	0 7 14 21	0.20 [a] 0.24 [a] 0.21 [a] 0.12 [a]	
				0.280	0 7 14 21	0.29 0.21 0.26 0.17	
USA (NY), 1986, Marketmore 80	Field	8		0.140	0 7 14 21	0.16 [a] 0.07 [a] 0.10 [a] 0.07 [a]	Hackett, 1991 (ABR-91042)
				0.280	0 7 14 21	0.09 0.24 < 0.05 0.19	
USA (NC), 1990, Burpee	Field	6		0.140	0 7 14	0.16 [a] 0.07 [a] < 0.05 [a]	
USA (MI), 1990, Marketmore 76	Field	6		0.140	0 7 14	0.22 0.22 0.23	
				0.280	0 7 14	0.44 0.36 0.27	

a - Mean of replicate analysis or samples

Summer squash

A total of 20 trials were conducted on summer squash (courgette) in Europe (from 200 to 2003) and in the USA (from 1986 – 1990). Cyromazine was applied three to eight times to the crops at a rate ranging from 0.14 to 0.35 kg ai/ha. The trials results are summarized in Table 64.

Table 64. Summary of cyromazine residues in field grown summer squash in Europe and the USA

Country, year, variety	Application			PHI (days)	Residues [mg/kg]	Reference (Report No.)
	No.	g ai/hL	g ai/ha			
France, 2000, Sandra	3	0.03	0.3-035	0 3 7 10	0.48 0.27 [a] 0.26 0.21	Lütolf, 2002f (1037/00)
France, 2000, Sandra	3	0.03	0.31-0.35	0 3 7 9	0.56 0.27 [a] 0.15 0.08	Lütolf, 2002g (1036/00)
France, 2000, Sandra	3	0.03	0.3-0.33	0 1 3 7	0.12 0.21 0.18 < 0.05	Pointurier, 2002a (132001)

Country, year, variety	Application			PHI (days)	Residues [mg/kg]	Reference (Report No.)
	No.	g ai/hL	g ai/ha			
France, 2001, Radian	3	0.03	0.31-0.32	0 1 3 7	0.41 0.27 <u>0.16</u> 0.11	Pointurier, 2002b (0132002)
Spain, 2003, Jedida	3	0.03	0.30	0 1 3	0.28 0.28 <u>0.11</u>	Kwiatkowski and Boxwell, 2004a (03-5014)
Spain, 2003, Eto	3	0.03	0.30	0 1 3 7 10 14	0.51 0.43 <u>0.21</u> 0.14 0.10 0.06	Kwiatkowski and Boxwell, 2004b (03-5015)
Italy, 2003 Karisma	3	0.03	0.30	0 1 3 7 10 14	0.39 0.31 <u>0.15</u> 0.13 0.10 0.08	Kwiatkowski and Boxwell 2004c (03-5017)
Italy, 2003 Monitor	3	0.03	0.30	0 1 3	0.29 0.16 <u>0.14</u>	Kwiatkowski and Boxwell, 2004d (03-5018)
USA (TX), 1987, Early Prolific Straightneck	8		0.140	0 7 14 21	<u>1.0</u> [a] 0.14 [a] 0.25 [a] < 0.05 [a]	
			0.280	0 7 14 21	2.00 < 0.05[a] 0.38 0.52	
USA (GE), 1990 , Dixie	6		0.140	0 7 14	<u>0.11</u> [a] < 0.05 [a] < 0.05 [a]	
USA (MI), 1990, Black Beauty	6		0.140	0 7 14	<u>0.07</u> [a] < 0.05 [a] < 0.05 [a]	
USA (NE), 1986, Black Eagle	8		0.140	0 7 14 21	<u>0.11</u> [a] < 0.05 [a] < 0.05 [a] < 0.05 [a]	
			0.280	0 7 14 21	0.10 < 0.05 < 0.05 < 0.05	
USA (NY), 1986, Yellow Crook Neck	8		0.140	0 7 14 21	<u>0.07</u> [a] 0.06[a] 0.07 [a] < 0.05 [a]	
USA (FL), 1986, Lemondrop	8		0.280	0 7 14 21	0.15 [a] < 0.05 [a] 0.15 [a] < 0.05 [a]	
			0.140	0 7 14 21	<u>0.18</u> [a] 0.06 [a] 0.10 [a] 0.10	

Country, year, variety	Application			PHI (days)	Residues [mg/kg]	Reference (Report No.)
	No.	g ai/hL	g ai/ha			
			0.280	0 7 14 21	0.25 0.13 0.13 0.10	
USA (CA), 1986 ,Ambassador/ Champion	6		0.140	0 7 14	0.22 [a] 0.08 [a] 0.05 [a]	Hackett, 1991 (ABR-91042)
			0.280	0 7 14	0.28 0.07 0.05	

a - Mean of replicate samples

Melons

Seven supervised residue trials were carried out in the US. Residues in melons were analysed using methods AG-408 and AG-417A (both HPLC-UV, LOQ=0.05 mg/kg). With exception of trial SW-IR-301-86 from Texas, concurrent determinations of residues in untreated crops gave residues < LOQ. A summary of the residue data is given in Table 65.

Table 65. Summary of cyromazine residues in melons (including cantaloupe) and watermelon

Country, year, variety	Application				Portion analysed	PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha				
France, 1999, Heliobel	Field	3	0.075	0.30	fruit fruit fruit[b] peel pulp	0 3 7 7 7	0.10 0.12 0.08 [a] 0.12 [a] 0.07 [a]	Lütolf, 2000e (1141/99)
France, 2000 Sunset	Field	3	0.03	0.30-0.32	fruit fruit[b] peel pulp fruit fruit fruit	0 3 3 3 7 10 14	0.16 0.08 [a] 0.15 [a] < 0.05 [a] 0.06 0.06 0.06	Lütolf, 2001f (1035/00)
France, 2000 Pancha	Field	3	0.06	0.29	fruit	0 3 7 10 14	0.38 0.24 [a] 0.16 0.25 0.21	Lütolf, 2001c (1033/00)
France, 2000 Buffalo	Field	3	0.03	0.29-0.30	fruit fruit[b] peel pulp fruit fruit fruit	0 3 3 3 7 10 14	0.38 0.12 [a] 0.24 [a] < 0.05 [a] 0.08 0.07 0.09	Lütolf, 2001d (1034/00)
France, 2000 Cezame	Protected	3	0.06	0.30	fruit	0 3 7 10 14	0.12 0.12 0.14 [a] 0.09 0.18	Pointurier, 2001c (31504)
France, 2000 Figaro	Protected	3	0.05	0.3	fruit	0 3 7 10 14	< 0.05 < 0.05 0.09 [a] 0.09 0.05	Pointurier, 2001d (31503)

Country, year, variety	Application				Portion analysed	PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha				
France, 2000 Sesame	Protected	3	0.038	0.30	fruit	0 3 7 9 14	0.09 0.09 0.08 [a] 0.08 <u>0.12</u>	Pointurier, 2001e (31502)
France, 2000 Lunastar	Protected	3	0.038	0.30	fruit	0 3 7 10 13	0.09 0.08 0.05 [a] <u>0.09</u> 0.09	Pointurier, 2001f (31501)
France, 2001 Lunastar	Protected	3	0.03	0.30	fruit fruit [b] peel pulp	0 7 7 7	0.08 <u>0.13</u> 0.16 [a] <u>< 0.05</u> [a]	Pointurier, 2002c (131601)
France, 2001 Lunastar	Protected	3	0.03	0.29-0.31	fruit fruit [b] peel pulp	0 7 7 7	< 0.05 <u>0.06</u> 0.06 [a] <u>< 0.05</u> [a]	Pointurier, 2002d (131602)
Italy, 2000 Lunastar	Protected	3	30	0.30	fruit	0 1 3 7 14	0.09 0.10 0.12 0.12 [a] <u>0.16</u>	Lütolf, 2001g (1104/00)
Spain, 1998, Sancho	Field	3	33	0.328-0.333	fruit	0 3 7 14 21	0.05 < 0.05 <u>< 0.05</u> < 0.05 < 0.05	Sack, 1999a (1013/98)
Spain, 2002, Balboa	Field	3	30	0.298-0.307	fruit fruit fruit [b] pulp peel fruit fruit	0 1 3 3 3 7 13	0.21 0.19 0.15 [a] < 0.05 [a] 0.25 <u>0.07</u> < 0.05	Kühne-Thu, 2003a (02-1082)
Spain, 2002, Aitana	Protected	3	0.03	0.284-0.300	fruit fruit fruit [b] pulp peel fruit fruit	0 1 3 3 3 7 13	0.15 0.09 0.16 [a] < 0.05 [a] 0.26 [a] <u>0.11</u> 0.11	Kühne-Thu 2003b (02-1075)
USA (FL), 1986, Cantaloupe	Field	8		0.140	fruit	0 7 14 21	<u>0.09</u> [a] 0.13 [a] 0.09 [a] 0.07 [a]	Hackett, 1991 (ABR-91042)
				0.280	fruit	0 7 14 21	0.20 0.11 0.09 < 0.05	
USA (AZ), 1986, Cantaloupe	Field	8		0.140	fruit	0 7 14 21	<u>0.08</u> [a] < 0.05 [a] 0.06 [a] < 0.05 [a]	

Country, year, variety	Application				Portion analysed	PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha				
USA (CO), 1986, Cantaloupe	Field	8		0.140	fruit	0 7 14 21	0.45 [a] 0.07 [a] 0.15 [a] 0.17 [a]	Hackett, 1991 (ABR-91042)
				0.280	fruit	0 7 14 21	0.53 0.15 0.20 0.21	
USA (NC), 1986 Cantaloupe	Field	8		0.140	fruit	0 7 14 21	0.11 [a] 0.10 [a] 0.10 [a] < 0.05 [a]	
USA (TX), 1986 Cantaloupe	Field	8		140	fruit	0 7 14	0.11 [a] 0.13 [a] 0.36 [a]	
				0.280	fruit	0 7 14	0.37 0.53 0.43	
USA (CA), 1986, Honeydew Melon	Field	8		0.140	fruit	0 7 14 21	0.13 [a] 0.07 [a] 0.08 [a] 0.05 [a]	
				0.280	fruit	0 7 14 21	0.19 0.06 0.12 0.17	
USA (AZ), 1986, Honeydew melon	Field	8		0.140	fruit	0 7 14 21	< 0.05 0.06 [a] 0.06 [a] < 0.05 [a]	
Watermelon, USA (CA), 1986	Field	8		0.140	fruit	0 7 14 21	0.13[a] 0.09[a] 0.12[a] 0.13[a]	
	Field	8		0.280	fruit	0 7 14 21	0.40[a] 0.14 0.13 0.10	

a - Mean of replicate analysis or samples;

b - The residue in whole fruit was calculated from the corresponding pulp and peel sub-specimens.

Tomato

A total of 36 supervised residue trials were carried out with tomato in Europe (from 1998 to 2001) and in the USA (in 1999). Cyromazine was applied in the field or in glasshouses using foliar, drip irrigation or soil drench applications. A summary of the residue data is given in Table 66.

Table 66. Residues of cyromazine residues in tomatoes from trials conducted in Europe and in the USA

Country, year, variety	Application				PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha			
France, 1998, Roma	Field	4	0.083	0.33	0 3	0.34 [a] 0.11 [a]	Lütolf, 2000f (1109/98)
France, 1998, Ondina	Field	4	0.04	0.31-0.34	0 3	0.25 [a] 0.21 [a]	Lütolf, 2000g (1108/98)

Country, year, variety	Application				PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha			
France, 1999, FMx115	Field	4	0.083	0.33	0 3	0. 29 [a] 0.11 [a]	Giannone, 2000a (1011/99)
France, 1999, Felicia	Protected	4	0.033	0.33	0 3	0.06 [a] 0.05 [a]	Giannone, 2000b (1010/99)
France, 2000 Felicia	Protected	4	0.033	0.33	0 3 7 10 14	0.08 0.09 [a] 0.11 0.06 0.09	Pointurier, 2001g (31701)
France, 2000, Servanne	Protected	4	0.033	0.33	0 3 7 10 14	0.26 0.30 [a] 0.34 0.26 0.13	Pointurier, 2001h (31702)
Greece, 2000, Carina	Field	4	0.033	0.32-0.33	0 3 7	0.75 0.58 [a] 0.57	Lütolf, 2002h (1052/00)
Greece, 2001 Senna	Field	4	0.033	0.327	0 1 3 7 14	0.28 0.27 0.13 [a] 0.13 0.13	Lütolf, 2002i (1061/01)
	Protected Drip irrigation		0.016	-	0 1 3 7 14	0.11 0.24 0.06 < 0.05[a] < 0.05	
Greece, 2001, 9036 hybrid	Field	4	0.033	0.33	0 1 3 7 14	0.33 0.42 0.16 [a] 0.11 0.15	Lütolf, 2002j (1062/01)
Italy, 2000, PS1296	Field	4	0.044	0.319-0.341	0 1 3 7 14	0.61 0.45 0.42 [a] 0.24 0.05	Bourry, 2002b (1099/00)
	Field Soil drench	-	0.018	0.77	3 14	< 0.05 < 0.05	
Spain,1998, Batlle	Field	4	0.033	0.297-0.330	0 3 7 14	0.50 0.23 0.22 0.10	Sack, 1999b (1016/98)
Spain, 1999, Batlle	Field	4	0.033-0.068	0.328	0 3	0.17 [a] 0.09 [a]	Giannone, 2000c (1132/99)
Spain, 2000, Durinta	Protected	4	0.033	0.32-0.34	0 3 7 14	0.27 0.13 [a] 0.13 0.12	Lütolf and Clarke, 2002 (1106/00)
	Field Drip irrigation	-	-	0.188	0 3 7 14	< 0.05 < 0.05[a] < 0.05 < 0.05	
Spain, 2000, Belmon	Protected	4	0.033	0.30-0.37	0 3 7	0.22 0.20 [a] 0.26	Lütolf, 2002k (1025/00)

Country, year, variety	Application				PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha			
					10 14	0.29 0.21	
Spain, 2000, Rollesta	Protected	4	0.033	0.32-0.33	0 3 7 14	0.12 0.16 [a] 0.22 0.10	Lütolf, 2002l (1061/00)
	Field Drip irrigation	-		0.188	0 3 7 14	< 0.05 < 0.05[a] < 0.05 < 0.05	
Switzerland, 1998, Paola	Field	4	0.017	0.33	0 3	0.25 [a] 0.15 [a]	Lütolf, 1999 (1023/98)
Switzerland, 1999, Calibra	Protected	4	0.033	0.330	0 3	0.22 0.14 [a]	Lütolf, 2000h (1066/99)
Switzerland, 2000, Petula	Protected	4	0.033	0.33	0 3	0.19 0.18 [a]	Lütolf, 2002m (1005/00)
USA (CA), 1990, Ace	Field	6	-	0.140	0 7 14	0.11 [a] 0.10 [a] 0.07 [a]	Grunenwald, 1992 (ABR-91045)
				0.280	0 7 14	0.08 0.13 0.08	
USA (CA), 1990, UC82	Field	6		0.140	0 6 14	0.26 [a] 0.09 [a] 0.09 [a]	
	Field	6		0.280	0 6 6 14	0.45 0.27 0.15 0.28	
USA (CA), 1990, Ace	Field	6		0.140	0 7 14	0.12 [a] 0.06 [a] < 0.05 [a]	
USA (FL), 1990, Sunny	Field	6		0.140	0 7 14	0.22 [a] 0.13 [a] 0.12 [a]	
	Field	6		0.280	0 7 14	0.39 0.40 0.32	
USA (FL), 1990, Floradade	Field	6		0.140	0 7 14	0.21 [a] 0.14 [a] 0.16 [a]	
USA (TE), 1990, Better Boy	Field	6		0.140	0 7 14	0.30 [a] 0.11 [a] 0.08 [a]	
USA (TX), 1990, Theresa/ Alamo	Field	6		0.140	0 7 14	0.10 [a] 0.10 [a] 0.07 [a]	Grunenwald, 1992 (ABR-91045)
USA (SC), 1990, Celebrity/ Park	Field	6		0.140	0 7 14	0.09 [a] 0.06 [a] 0.06 [a]	
	Field	6		0.280	0 7 14	0.16 0.11 0.12	
USA (CA), 1990, Heinz 2710	Field	6		0.140	0 7 15	0.28 [a] 0.12 [a] 0.08 [a]	

Country, year, variety	Application				PHI, days	Residues, mg/kg	Reference (Report No.)
	Treatment	No.	kg ai/hL	kg ai/ha			
	Field	6		0.280	0 7 15	0.74 0.49 [a] 0.16	
USA (MI), 1990, Peto 197	Field	6		0.140	0 7 14	0.14 [a] 0.06 [a] 0.07 [a]	
USA (OH), 1990, Heinz 8245	Field	6		0.140	0 7 14	0.21 [a] 0.08 [a] 0.07 [a]	
USA (NJ), 1990, Ole	Field	6		0.140	0 7 14	0.18 [a] 0.17 [a] 0.14 [a]	
USA (PE), 1990, Floradade	Field	6		0.140	0 7 14	0.13 [a] 0.11 [a] 0.07 [a]	

a - Mean of duplicate samples

Eggplants

Four trials were conducted in eggplants using foliar applications in glasshouses in France and Switzerland in 2000-2001. The results are shown in Table 67.

Table 67. Residues of cyromazine in protected eggplants in Europe

Country, year, variety	Application			PHI days	Residues mg/kg	Reference (Report No.)
	No.	kg ai/hL	kg ai/ha			
France, 2000, Abrivado	3	0.03	0.34-0.36	0 3 7 10 15	0.25 0.23 [a] 0.13 0.09 < 0.05	Lütolf, 2002n (1038/00)
France, 2000, Orion	3	0.03	0.30-0.34	0 3	0.43 0.26 [a]	Lütolf, 2002o (1039/00)
France, 2001, Telar	3	0.03	0.32-0.34	0 3 7 14	0.16 0.14 [a] 0.10 < 0.05	Pointurier, 2002e (0132101)
Switzerland, 2001 Berinda	3	0.03	0.33	0 3	0.08 0.05 [a]	Lütolf, 2003a (1010/01)

a - Mean of duplicate samples

Peppers

Twelve supervised trials were conducted on peppers (chilli and bell) in the USA in 1984 and 1985 using 12 applications of cyromazine at 0.14 or 0.28 kg ai/ha. The results are shown in Table 68.

Table 68. Residues of cyromazine in peppers in the USA (Ballantine, 1985a; Report No. ABR-85096)

State, year, Variety,	Application		PHI, days	Residues, mg/kg
	No.	kg ai/ha		
CA, 1985, Bell	12	0.140	0 7 14 21	0.14 [a] 0.12 [a] 0.07 [a] < 0.05 [a]
		0.280	0 7 14 21	0.29 0.19 < 0.05 0.09

State, year, Variety,	Application		PHI, days	Residues, mg/kg
	No.	kg ai/ha		
PA, 1984, Bell	12	0.140	0 7 14 22	0.10 [a] 0.12 [a] 0.10 [a] 0.07 [a]
		0.280	0 7 14 22	0.11 0.22 0.07 0.10
MI, 1984, Bell	12	0.140	0 7 14	0.11 [a] 0.19 [a] 0.27 [a]
NC, 1984, Chilli,	12	0.140	1 7 14	0.17 [a] 0.11 [a] < 0.05 [a]
NC, 1984, Bell	12	0.140	1 7 14	0.10 [a] < 0.05 [a] < 0.05 [a]
MS, 1984, Tabasco	12	0.140	0 7 14	0.96 [a] 0.84 [a] 0.79 [a]
CA, 1985, Chilli,	12	0.140	0 7 14 21	0.24 [a] 0.20 [a] 0.13 [a] 0.08 [a]
		0.280	0 7 14 21	0.57 0.46 0.20 0.07
FL, 1984, Bell,	12	0.140	0 8 15	0.59 [a] 0.36 [a] 0.38 [a]
		0.280	0 8 15	0.95 0.62 0.24

a - Mean of replicate samples or analysis

Mushrooms

Nine supervised trials were conducted with mushrooms in France, Italy and Switzerland from 1986 to 2001 using 2.0 to 5.0 kg ai/ha of cyromazine in the field and in the glasshouse. The results are shown in Table 69.

Table 69. Summary of cyromazine residues in mushrooms in protected trials

Country, year, variety	Application		PHI days)	Residues, mg/kg		Reference (Report No.)
	No.	g ai/m^2		Cyromazine	Melamine	
France, 1988, Champignon	1	0.4	23 30 37 44 50 57	2.2 2.1 2.0 1.6 1.5 1.1		Tournayre, 1989 (65/88)
	1	0.8	23 30 37 44 50 57	2.6 3.0 2.7 2.3 1.8 1.7		

Country, year, variety	Application		PHI days)	Residues, mg/kg		Reference (Report No.)
	No.	g ai/m^2		Cyromazine	Melamine	
France, 2001, Petit Blanc	1	0.4	21 29	2.1 [a] 2.8	0.36[a] 0.36	Solé, 2002a (133002)
France, 2001, Petit Blanc	1	0.4	13 20 28	2.7 4.2 [a] 3.8	0.14 0.23 [a] 0.22	Solé, 2002b (0133001)
Italy, 1988, Champignon	1	0.4	25 33 43	0.71 0.66 0.68	0.16 0.20 0.17	Mostert, 1990a (1147/88)
Italy, 1988, Champignon	2	0.2-0.4	10 18 28	0.92 0.85 0.84	1.1 1.3 0.97	Mostert, 1990b (1148/88)
Italy, 2001, Agraricgus bisporus	1	0.4	14 21 28	0.19 [a] 0.36 [a] 0.37 [a]	2.1[a] 2.1[a] 2.7 [a]	Lütolf, 2003b (1041/01)
Italy, 2001, Champignon	1	0.4	14 21 28	1.2 [a] 0.51[a] 1.3 [a]	0.14 [a] 0.17 [a] 0.19 [a]	Lütolf, 2003c (1040/01)
Switzerland, 1986, Champignon	1	0.5	15 23 26	1.1 1.7 2.4	1.9 1.3 1.7	Altenburger,1988 (1029/86)

a - Mean of duplicate samples

Lettuce

Forty trials were conducted on lettuce from 1993 to 2004 in Europe and 26 from 1982 to 1987 in the USA. Cyromazine was applied in the field or in glasshouses on head and cos lettuce using foliar, drench or drip applications. The results are shown in Table 70.

Table 70. Residues of cyromazine in lettuce (Head and Cos)

Crop (variety) Country,Year	Application				Portion part	PHI, days	Residues, mg/kg	Reference (Report No.)
	Protected/Field	No.	kg ai/hL	kg ai/ha				
Head (Rougette) France, 1993	Field	3	0.075	0.30	head	7 14 22	1.2 1.7 0.56	Maffezzoni, 1995a (OI93310)
Head (Carlane) France-S, 1993	Field	3	0.075	0.30	head	7 14 21	0.94 0.34 0.06	
Head (Ultra) France-N, 1993	Field	3	0.075	0.30	head	7 14 21	0.17 < 0.03 < 0.03	
Head (Carmen) France, 1998 (protected)	Protected	3	0.075	0.30	head	0 7 14 21	11 8.5 3.0 2.0	Lütolf, 2000i (1130/98)
Head (Flandria) France, 1998	Protected	3	0.075	0.30-0.32	head	0 7 14 21	9.5 5.2 2.9 2.7	Lütolf, 2000j (1004/98)
Head (Audrey) France, 1998	Protected	3	0.075	0.30-0.32	head	0 6 13 20	9.0 3.2 2.2 1.6	Lütolf, 2000k (1005/98)

Crop (variety) Country,Year	Application				Portion part	PHI, days	Residues, mg/kg	Reference (Report No.)
	Protected/Field	No.	kg ai/hL	kg ai/ha				
Head (Sansai) France, 1999	Protected	3	0.10	0.30	head	0 7 14 21	15 10 <u>4.9</u> 2.7	Lütolf, 2000l (1129/98)
Cos (Entrada) France-N 2000	Field	3	0.03	0.30	whole plant	0 3 7	3.4 0.39 <u>0.18</u> [a]	Pointurier, 2001i (0030901)
		3	0.03	0.29-0.30	whole plant	0 10 14	5.0 0.15 0.15[a]	
Cos (Entrada) France-N 2000	Field	3	0.03	0.28	whole plant	0 3 7	6.1 4.7 <u>1.8</u> [a]	Pointurier, 2001j (0030902)
	Field	3	0.03	0.28-0.30	whole plant	0 9 14	5.1 1.8 <u>2.0</u>[a]	
Cos (Terlana) France, 2001	Field	3	0.03	0.29-0.30	whole plant	0 1 3 7	4.2 3.1 1.4 <u>1.3</u> [a]	Pointurier, 2002d (0131901)
	Field	3	0.03	0.28-0.33	whole plant	0 7 15 22	7.4 <u>0.45</u> 0.08 < 0.05 [a]	
Cos (Amadeus) France, 2001	Protected	3	0.03	0.31	whole plant	0 10 14	6.1 <u>1.5</u> 1.2 [a]	Pointurier, 2001k (0130202)
	Protected	3	0.03	0.31-0.32	whole plant	0 3 7	6.5 3.6 <u>2.8</u>[a]	
Cos (Amadeus) France, 2000	Protected	3	0.03	0.31-0.33	whole plant	0 10 14	8.0 <u>5.2</u> 5.2 [a]	Pointurier, 2001l (0130201)
		3	0.03	0.31-0.33	whole plant	0 3 7	9.0 7.8 <u>6.2</u>[a]	
Cos (Rosario) France, 2002	Protected	3	0.1	0.30-0.31	whole plant	0 3 7 14	11 3.2 2.5 <u>2.4</u>	Solé, 2003a (02-1170)
Cos (Rosario) France, 2002	Protected	3	0.1	0.30	whole plant	14 21 28	<u>6.5</u> 4.7 4.0	
Cos (Rosario) France, 2002	Protected	3	0.07	0.32-0.38	whole plant	0 3 7	2.8 [a] 2.0 5.2	Solé, 2003b (02-1171)
Cos (Rosario) France, 2002	Protected	3	0.07	0.31-0.32	whole plant	14 21 28	<u>0.28</u> [a] 0.55 [a] 0.22	
Cos (Focea) France, 2002	Protected	3	0.07	0.29-0.31	whole plant	0 3 7 14	12 11 8.3 <u>5.8</u>	Solé, 2003c (02-1172)
Cos (Focea) France, 2002	Protected	3	0.07	0.30-0.32	whole plant	14 21 28	<u>4.7</u> 4.2 3.6	

Crop (variety) Country,Year	Application				Portion part	PHI, days	Residues, mg/kg	Reference (Report No.)
	Protected/Field	No.	kg ai/hL	kg ai/ha				
Cos (Amadeus) France, 2004	Protected	3	0.1	0.30-0.31	whole plant whole plant head head head	0 3 7 10 14	24 24 14 17 <u>14</u>	Kwiatkowski, 2006 (04-5040)
	Protected	3	0.1	0.28-0.30	whole plant whole plant head head head	0 3 7 10 14	12 9.4 0.20 0.23 <u>0.13</u>	
Cos (Melissa) Italy, 2000	Protected	3	0.03	0.30	whole plant	0 10 14	18 <u>18</u> 3.8 [a]	Lütolf, 2002p (1102/00)
	Protected	3	0.03	0.30	whole plant	0 3 7	21 16 <u>11</u>	
Cos (Sofia) Italy, 2000	Field	3	0.03	0.30	whole plant	0 3 7	3.9 1.2 <u>1.5</u> [a]	Lütolf, 2002q (1100/00)
	Field	3	0.03	0.30	whole plant	0 10 14	4.0 <u>0.22</u> 0.12[a]	
Cos (Funly) Italy, 2000	Field	3	0.03	0.30	whole plant	0 3 7	10 0.74 <u>0.28</u>	Lütolf, 2002r (1101/00)
	Field	3	0.03	0.30	whole plant	0 10 14	10 <u>0.19</u> 0.07[a]	
Cos (Soave) Italy, 2000	Protected	3	0.03	0.30	whole plant	0 10 14	16 <u>7.9</u> 6.1 [a]	Lütolf, 2002s (1103/00)
	Protected	3	0.03	0.30	whole plant	0 3 7	16 15 <u>15</u>[a]	
Cos (Freccia) Italy, 2002	Protected	3	0.03	0.30	whole plant	0 1 3 7 14	15 9.9 5.2 <u>3.3</u> 0.89	Solé, 2003d (02-1142)
	Protected Drench application	2		0.188	whole plant	0 1 3 7 14	0.17 0.15 0.16 0.14 0.10	
Cos (Mula) Spain, 2000	Field	3	0.03	0.30	whole plant	0 3 7	8.0 1.6 <u>0.24</u> [a]	Lütolf, 2002t (1028/00)
	Field	3	0.014	0.187	whole plant	0 3 7	0.45 0.17 0.10	

Crop (variety) Country,Year	Application				Portion part	PHI, days	Residues, mg/kg	Reference (Report No.)
	Protected/Field	No.	kg ai/hL	kg ai/ha				
Cos (Monterrey) Spain, 2000	Field	3	0.03	0.30	whole plant	0 7 10 14	9.7 0.27 0.14 0.14 [a]	Lütolf, 2002u (1026/00)
Head (Levis) Switzerland 1999	Protected	3	0.038	0.300	whole plant	0 7 14 21	10 5.8[a] 3.4 1.8	Lütolf, 2000m (1064/99)
Cos (Remus) Switzerland 2001	Field	3	0.03	0.30	whole plant	0 3 7 14 21	12 3.7 1.8 0.37 0.12 [a]	Lütolf, 2002v (1007/01)
Cos (Terlana) Switzerland 2001	Field	3	0.03	0.30	whole plant	0 3 7 14 21	5.4 1.1 0.34 0.10 < 0.05 [a]	Lütolf, 2002x (1008/01)
Head USA (FL), 1982	Field	9		0.140	head	0 7 14	2.0 [a] 1.2 [a] 0.08	Ballantine, 1984a (ABR-84076)
Head USA (FL, 1982	Field	8		0.140	head	0 7 14	5.8 [a] 0.12 [a] 0.06 [a]	
	Field	8		0.280	head	0 7 14	16 0.06 0.07	
Head USA (CA), 1983	Field	8		0.140	head	0 7 14	0.19 [a] 0.05 [a] < 0.05 [a]	
	Field	8		0.280	head	0 7 14	0.57 < 0.05 < 0.05	
Head USA (WI), 1983	Field	8		0.140	head	0 7 14	5.4 [a] 1.9 [a] 0.93 [a]	
Head USA (TX), 1983	Field	8		0.140	head	0 8 15	1.2 [a] 0.38 [a] 0.53 [a]	
Head USA (FL), 1983	Field	8		0.140	head	0 7 14	7.7 [a] 1.4 [a] 0.08 [a]	
	Field	8		0.280	head	0 7 14	9.3 [a] 3.8 [a] 1.70 [a]	
Leaf lettuce (Prize Head) USA (AZ), 1986	Field	5		0.140	whole plant	0 7 14 22	7.1 [a] 2.0 [a] 0.87 [a] 0.35 [a]	Cheung, 1991 ABR-89103
	Field	5		0.280	whole plant	0 7 14 22	11 4.8 3.0 0.27	

Crop (variety) Country,Year	Application				Portion part	PHI, days	Residues, mg/kg	Reference (Report No.)
	Protected/Field	No.	kg ai/hL	kg ai/ha				
Leaf lettuce (Waldmanns green) USA (CA), 1986	Field	5		0.140	whole plant	0 7 14 21	7.8 [a] 3.9 [a] 0.79 [a] 0.15 [a]	
	Field	5		0.280	whole plant	0 7 14 21	14 3.40 2.30 0.56	
Leaf lettuce (Black Seeded Simpson) USA (NY), 1986	Field	5		0.140	whole plant	0 7 14 21	5.8 [a] 1.6 [a] 0.50 [a] 0.15 [a]	
	Field	5		0.280	whole plant	0 7 14 21	12 [a] 1.60 0.88 0.05	
Leaf lettuce (Ruby Red) USA (CO), 1986	Field	5		0.140	whole plant	0 7 14 21	12 [a] 5.2 [a] 2.0 [a] 0.99 [a]	
Leaf lettuce (Prize Head) USA (AZ), 1986	Field	5		0.140	whole plant	0 7 14 21	9.4 [a] 2.8 [a] 0.64 [a] 0.53 [a]	
	Field	5		0.280	whole plant	0 7 14 21	14 8.3 0.53 0.62	
Leaf lettuce (Waldmanns green) USA (CA), 1986	Field	5		0.140	whole plant	0 7 14 21	7.7 [a] 1.5 [a] 0.59 [a] 0.56 [a]	
	Field	5		0.280	whole plant	0 7 14 21	23 4.1 3.8 2.4	
Leaf lettuce (Oak) USA (FL), 1986	Field	5		0.140	whole plant	0 7 14 21	7.0 0.58 [a] 0.13 [a] < 0.05 [a]	
	Field	5		0.280	whole plant	0 7 14 21	14 0.87 0.27 0.10	
Leaf lettuce (Black Seeded Simpson) USA (TX), 1987	Field	5		0.140	whole plant	0 7 14 21	6.3 [a] 4.4 [a] 1.7 [a] 0.19 [a]	
	Field	5		0.280	whole plant	0 7 14 21	19 4.0 [a] 4.2 0.10	

Crop (variety) Country,Year	Application				Portion part	PHI, days	Residues, mg/kg	Reference (Report No.)
	Protected/Field	No.	kg ai/hL	kg ai/ha				
Leaf lettuce (Royal Oak) USA (FL), 1987	Field	5		0.140	whole plant	0 7 14 21	16 2.8 [a] 0.62 0.14	
	Field	5		0.280	whole plant	0 7 14 21	26 1.70 0.26 0.11	

a - Mean of replicate samples or analysis

Spinach

Sixteen trials were conducted with spinach in the USA in 1986/1987 using five to eight applications of cyromazine at 0.14 or 0.28 kg ai/ha. The results are shown in Table 71.

Table 71. Residues of cyromazine in spinach from trials conducted in the USA (Cheung, 1991; Report No. ABR-89103

State, year	Application		PHI, days	Residue, mg/kg
	No.	kg ai/ha		
MS, 1996	5	0.140	0 7 14 21	6.1[a] 1.1[a] 0.52[a] 0.05[a]
	5	0.280	0 7 14 21	14 2.7 2.0 0.18
CA, 1986 [b]	5	0.140	0 7 14	10[a] 1.8[a] 0.82[a]
	5	0.280	0 7 14	16 6.5 1.4
NE, 1986	5	0.140	0 7 14 21	1.4[a] 0.40[a] 0.10[a] < 0.05[a]
	5	0.280	0 7 14 21	0.29 0.26 0.11 < 0.05
CA NY, 1986	5	0.140	0 7 14 21	4.7[a] 2.3[a] 0.60[a] 0.10[a]
	5	0.280	0 7 14 21	6.6 2.4 1.7 0.38
NC, 1986	5	0.140	0 7 14 22	15[a] 6.1[a] 2.5[a] 0.49[a]

State, year	Application		PHI, days	Residue, mg/kg
	No.	kg ai/ha		
NY, 1986	5	0.140	0 7 14 21	4.2[a] 4.2[a] 0.14[a] < 0.05[a]
	5	0.280	0 7 14 21	0.33 0.18 0.23 0.08
CA, 1986	5	0.140	0 7 14 21	18[a] 5.4[a] 2.7 2.1[a]
	5	0.280	0 7 14 21	36 9.8 9.2 4.0
TX, 1987	5	0.140	0 7 14 21	6.0[a] 1.2[a] 0.85[a] 0.55[a]
FL, 1987	8	0.140	0 7 14 21	12[a] 3.7[a] 1.9[a] 0.64[a]
	8	0.280	0 7 14 21	3.4 1.9 1.2 0.83

a - Mean of replicate samples or analysis; control = 0.41 mg/kg

Mustard greens

Five trials were conducted in the USA in 1997 with mustard greens. The residues found in the leaves are shown in Table 72.

Table 72. Cyromazine residues in mustard greens from trials conducted in the USA using six applications at a 0.142 kg ai/ha rate (Eudy, 1999; Report No. 42-97)

State	Portion analysed	PHI, days	Residue, mg/kg [a]
California	Leaves	0 7	15.6 6.5
Georgia	Leaves	0 7	7.5 1.6
Louisiana	Leaves	0 7	8.4 2.7
Illinois	Leaves	0 7	9.0 7.4
Texas	Leaves	0 1 3 5 7 9	6.2 4.0 0.97 0.23 1.1 0.58

a - Mean of replicate samples

Beans

Fourteen trials were conducted in the USA with lima beans in pods from 1991 to 2000 using six to eight applications of cyromazine at 0.14 or 0.28 kg ai/ha. The results are shown in Table 73.

Table 73. Summary of cyromazine residues in Lima beans in pods (VP 0534) in the USA

State, year, variety	Application		PHI (days)	Residue, mg/kg [a]	Reference (Report No.)
	No.	kg ai/ha			
GA, 1990, Improved Kingstons	8	0.140	7	0.38	Dorschner, 1997 (3908)
		0.280	7	1.0	
WI, 1990, Improved Kingstons	8	0.140	7	0.56	
		0.280	7	1.3	
CA, 1991, Maria	8	0.140	7	0.23	
		0.280	7	0.42	
WA, 1991, Henderson Bush	6	0.140	7	0.19	
		0.280	7	0.93	
NJ, 1992, 8-78 [b]	6	0.140	7	0.17	
		0.280	7	0.36	
WI, 1993, Improved Kingston	6	0.140	7	0.32	
MD, 2000, Fordhook 242	6	0.140	7	0.11	Starner, 2004 (A3908)
NJ, 2000, Fordhook 242	6	0.140	8	< 0.05	
CA, 2000, Henderson	6	0.140	7	0.23	

a - Mean of replicate samples or analysis;

b - Mean analytical recoveries at 1 and 2 mg/kg of 130% and 160%

Dry beans

Nine field trials in or on beans were carried out in the USA in 1998 using six applications at 0.14 kg ai/ha. The results are shown in Table 74.

Table 74. Summary of cyromazine residues in dry beans in the USA (Markle, 2000; Report IR4-06744)

State, year, variety	Application		PHI, days	Residue, mg/kg [a]
	No.	kg ai/ha		
CA, 1998, Blackeyed pea	6	0.140	7	1.1
CO, 1998, Pinto	6	0.140	7	1.8
GA, 1998 Navy	6	0.140	8	0.68
ID, 1998, Pinto	6	0.140	8	1.1
NE, 1998, Kidney	6	0.140	7	0.97
CA1998, Pinto	6	0.140	7	0.23
WI, 1998, Great Northern	6	0.140	7	0.84
WI, 1998, Great Northern	6	0.140	6	1.0
WI, 1998, Great Northern	6	0.140	8	1.2

a - Mean of replicate samples

Potato

Eight trials were conducted in Spain in 1998 – 1999 using three applications of cyromazine at 0.17 to 0.34 kg ai/ha. The results are shown in Table 75.

Table 75. Summary of cyromazine residues in potatoes in Spain and the USA

Country, year, variety	Application			PHI, days	Residues, mg/kg	Reference (Report No.)
	No.	kg ai/hL	kg ai/ha			
Spain, 1998, Agros	3	0.027-0.031	0.180	0 3 7 14 21	0.25 0.14 0.21 0.18 0.11	Sack, 1999c (1002/98)
Spain, 1998, Cara	3	0.026	0.17-0.18	21	0.06 [a]	Sack, 1999d (1003/98)
Spain, 1998, Red Pontiac	3	0.046	0.34-0.37	0 7 14 21 28 35	< 0.05 < 0.05 0.05 < 0.05 < 0.05 < 0.05	Sack, 1999e (1015/98)
Spain, 1998, Red Pontiac	3	0.046	0.28-0.33	0 14 21 35	< 0.05 0.09 0.06 0.12	Sack, 1999f (1014/98)
Spain, 1999, Red Pontiac	3	0.042	0.32-0.34	0 21	0.27 [a] 0.48 [a]	Lütolf, 2000n (1128/99)
Spain, 1999, Sirius	3	0.042	0.33	0 21	0.08 [a] 0.15 [a]	Lütolf, 2000o (1129/99)
Spain, 1999, Sandy	3	0.041	0.33	0 3 7 14 21	0.29 0.37 0.56 0.85 0.80 [a]	Lütolf, 2000p (1130/99)
Spain, 1999, Kenbec	3	0.041	0.33	0 3 7 14 21	0.23 0.29 0.35 0.30 0.42 [a]	Lütolf, 2000q (1131/99)

a - Mean of duplicate samples

Artichoke

Four trials were conducted with artichokes in Spain using three applications of cyromazine at 0.25 to 0.30 kg ai/ha. The results are shown in Table 76.

Table 76. Summary of cyromazine residues in globe artichokes (flower heads) in Spain

Year, variety	Application			PHI, days	Residues mg/kg	Reference (Report No.)
	No.	kg ai/hL	kg ai/ha			
2001, Blanca de Tudela	3	0.03	0.32	3 8	1.6 [a] 0.95	Lütolf, 2003d (1019/01)
2001, Tudela	3	0.03	0.31	3 7	1.3 [a] 1.3	Lütolf, 2003e (1020/01)
2002, Blanca de Tudela	3	0.03	0.25-0.29	0 3 7 14	1.0 1.3 0.85 0.38	Kühne-Thu, 2003c (02-1000)
2002, Blanca de Tudela	3	0.03	0.28-0.30	0 3 7 14	1.8 1.4 1.1 0.73	Kühne-Thu, 2003d (02-1001)

a - Mean of duplicate samples

Celery

Twelve trials were conducted in celery in Europe from 1993 to 2003 and 11 trials were conducted in the USA in 1982/1983. Cyromazine was applied up to 12 times and at rates from 0.14 to 0.36 kg ai/ha. The results are shown in Table 77.

Table 77. Summary of cyromazine residues in celery

Country, year, variety	Application			Part analysed	PHI, days	Residues, mg/kg	Reference (Report No.)
	No.	kg ai /hl	kg ai/ha				
France (St. J. de Conceles), 1993, Golden	4	0.03	0.30	Stems	7 14 21	0.42 0.39 <u>0.57</u>	Maffezzoni, 1995b (OI93308)
France (Fourges), 1993, Elne	4	0.03	0.30	Stems	7 14 20	2.6 <u>1.6</u> 0.47	
France (Le Cailar), 1993, Elne	4	0.03	0.30	Stems	7 14 21	0.68 <u>0.27</u> 0.22	
France, 1998, Lino	4	0.083	0.30-0.35	Whole plant	0 7 14	4.9 0.73 <u>0.60</u>	Giannone, 1999a (1042/98)
France, 1998, Lino	4	0.083	0.30-0.34	Whole plant	0 7 14	3.7 0.42 <u>0.27</u>	Giannone, 1999b (1043/98)
France, 1999, Lino	4	0.083	0.309-0.360	Whole plant	0 3 7 14	3.2 1.4 0.95 [a] <u>0.68</u> [a]	Lütolf, 2000r (1069/99)
France, 1999, Golden Spartang	4	0.083	0.336-0.358	Whole plant	0 3 7 14	3.1 1.4 1.0 [a] <u>0.58</u> [a]	Lütolf, 2000s (1070/99)
Spain, 1999, Utha	3	0.033	0.33	Whole plant	0 3 7 14	0.93 1.3 0.30 [a] <u>0.36</u>	Sack, 1999g (1000/99)
Spain, 1998, Golden Spartang	3	0.041	0.33	Whole plant	0 3 7 14	1.8 0.53 0.48 <u>0.27</u>	Sack, 1999h (1001/98)
Spain, 2001, Florida	3	0.033	0.33	Stems	0 3 7 14	6.0 2.3 1.9 [a] <u>1.8</u>	Solé, 2002c (1129/01)
Spain, 2001, Imperial	3	0.033	0.33	Stems	0 3 7 14	7.9 5.2 2.4 [a] <u>2.3</u>	Solé, 2002d (1128/01)
Spain, 2003, Sistel	3	0.05	0.30	Whole plant	0 3 7	4.5 2.9 3.0	Kwiatkowski, 2004e (03-5040)
USA (FL), 1982	12	-	0.140	Stems	7	0.09[a]	Ballantine, 1984a (ABR-84076)
USA (FL), 1982	12	-	0.140	Stems	0 7 14	17[a] 5.2[a] 1.3[a]	
	12	-	0.280	Stems	0 7 14	26 13 3.6	

Country, year, variety	Application			Part analysed	PHI, days	Residues, mg/kg	Reference (Report No.)
	No.	kg ai /hl	kg ai/ha				
USA (FL), 1982	12	-	0.140	Stems	0 7 14	0.75[a] 0.12[a] 0.24[a]	
USA (CA), 1982	12	-	0.140	Stems	7	2.6[a]	
	12	-	0.280	Stems	7	2.4	
USA (CA), 1982	12	-	0.140	Stems	0 7 14	0.32[a] 0.16[a] 0.17[a]	
USA (FL), 1983	12	-	0.140	Stems	7	0.34[a]	
USA (FL), 1983	12	-	0.140	Stems	0 7 14	0.17[a] 0.09[a] < 0.05[a]	
USA (MI), 1983	12	-	0.140	Stems	0 7 14	4.7[a] 4.8[a] 3.0[a]	
USA (NY), 1983	12	-	0.140	Stems	0 7 14	3.6[a] 0.76[a] 0.42[a]	
USA (CA), 1983	12	-	0.140	Stems	0 7 14	0.17[a] 0.13[a] 0.05[a]	
	12	-	0.280	Stems	0 7 14	0.06 0.05 0.07	
USA (TX), 1983	12	-	0.140	Stems	0 7 14	0.25[a] 0.20[a] 0.17[a]	

a - Mean results from replicate samples

PROCESSING STUDIES

A hydrolysis study *(*Adam, 2000*)* was carried out to assess whether breakdown or reaction products could be formed from cyromazine residues during processing of treated crops. [^{14}C]-cyromazine was dissolved in 5 mL of the sterile aqueous buffer solutions at pH 4, pH 5 and pH 6 (ionic strength ≤ 0.01M) at a mean concentration of 4.99 mg/l. Duplicate test solutions were heated to the appropriate temperature and time, and then cooled and neutralised to pH 7. Sub-samples of the neutralised test solutions were directly analysed by HPLC and/or 2D-TLC. The radioactivity in the neutralised aqueous buffer solutions after incubation consisted entirely of unchanged cyromazine under each set of test conditions. Recovery efficiency of radioactivity ranged from 98.4 – 101.1% (Table 78).

Table 78. Recovery of unchanged cyromazine following hydrolysis under three conditions

Incubation			Sample	Recovery,% applied	Cyromazine, % applied	Process represented
pH	Temperature (°C)	Time (min)				
4	90	20	1 2	99.0 100.8	99.0 100.8	Pasteurisation
5	100	60	1 2	99.8 98.4	99.8 98.4	Baking, Boiling, brewing
6	120	20	1 2	101.1 100.1	101.1 100.1	Sterilisation

Residues in processed commodities

In a study conducted in California (USA) in 1990 using tomato, (a variety normally used for processing), plants were treated with six foliar applications of a 75 WP formulation at 0.140 or 0.250 kg ai/ha and samples harvested at PHI of 7 days (Grunenwald, 1992). Treated samples were pooled and processed according to the flowchart shown in Figure 4, simulating normal commercial processing conditions. The processes include preparation of tomato juice and puree and canning of tomato fruit. Processed fractions were stored at -20 °C until prepared, then re-frozen and stored until analysed (7 – 13 months). Residues of cyromazine and melamine were determined by methods AG-408 and AG-417A. Mean residues of cyromazine in unwashed fruit and tomato processed fractions and associated processing factors (PF) are shown in Table 79.

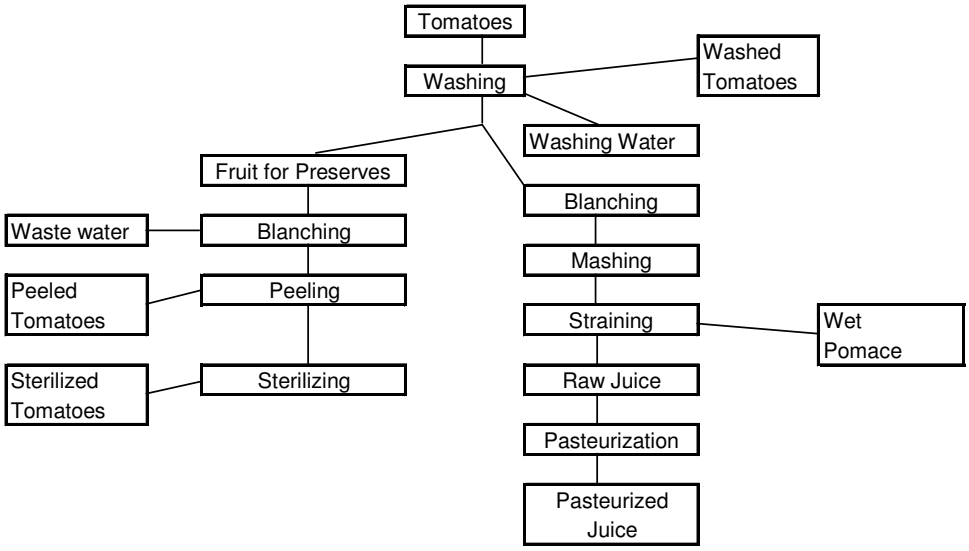


Figure 4. Flow diagram describing the processing of tomatoes

Table 79. Residues and processing factors for cyromazine in tomatoes and processed fractions

Processed fraction	Cyromazine residue (mg /kg)	Processing factor (PF)	Mean PF
Fruit, unwashed	0.08[a] , 0.19[a]	-	-
Fruit, washed	< 0.05, 0.15	< 0.63, 0.79	0.71
Fruit, canned	< 0.05, 0.08	< 0.63, 0.42	0.53
Pomace, wet	0.09[a], 0.19[a]	1.06, 0.97	1.00
Pomace, dry	0.22[a], 0.55[a]	2.69, 2.89	2.80
Juice, canned	0.06, 0.14	0.75, 0.74	0.75
Puree	0.11, 0.19	1.38, 1.0	1.20
Paste	0.16, 0.40	2.0, 0.2.1	2.10
Catsup (ketchup)	0.10, 0.08	1.25, 0.42	0.84

a - mean of duplicate samples

In a study conducted in the United States (Ohio and North Dakota), six trials were conducted with potato plants that received three to five applications of cyromazine with a total application of 0.56, 1.68 and 2.80 kg ai/ha (Selman, 1995). The samples were harvested at 48 days PHI and processed according to the flowchart shown in Figure 5. Residues of cyromazine in potatoes and processed fractions were analysed using method AG-621 and are shown in Table 80.

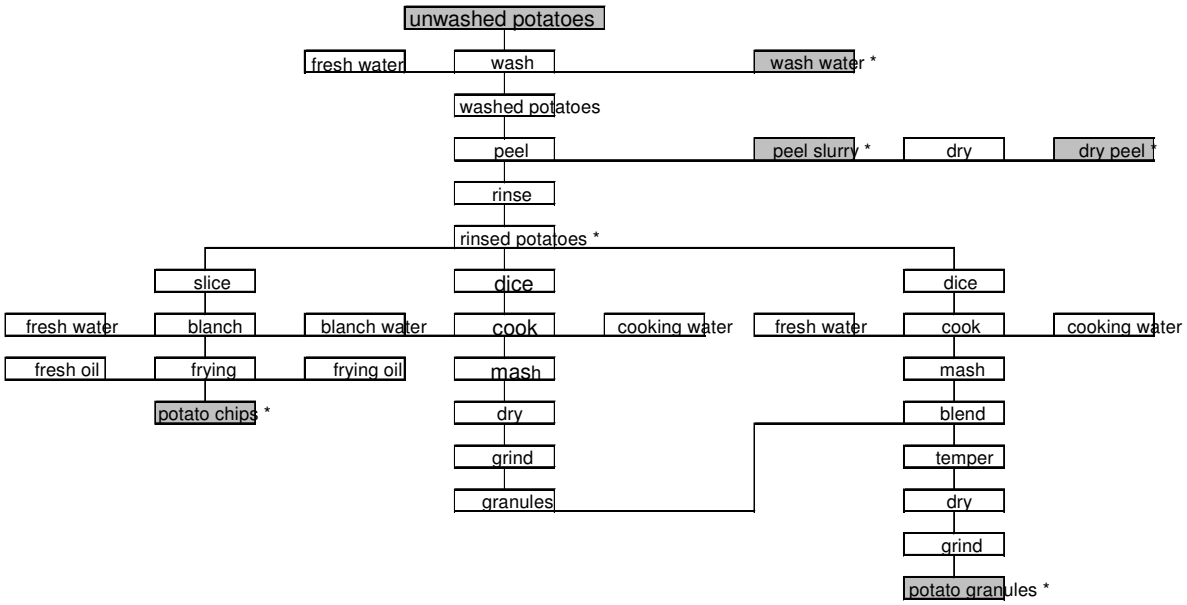


Figure 5. Processing flowchart for potatoes

Table 80. Residues and processing factors for cyromazine in potatoes and processed fractions

Processed fraction	Cyromazine residues, mg/kg			Processing factor (PF)			Mean PF
Tubers	0.20 0.40	0.60 0.85	1.15 1.12	- -	- -	- -	-
Peeled and rinsed potatoes	0.16 0.26	0.57 0.87	1.08 1.17	0.8 0.7	1.0 1.0	0.9 1.0	0.9
Wet peel and trimmings	0.09 0.14	0.22 0.46	0.42 0.44	0.5 0.4	0.4 0.5	0.4 0.4	0.4
Dry peel and trimmings	0.40 0.64	0.85 1.84	1.88 1.87	2.0 1.6	1.4 2.2	1.6 1.7	1.8
Potato chips	0.07 0.70	0.69 0.47	1.71 3.11	0.4 1.8	1.2 0.6	1.5 2.8	1.3
Potato granules	0.57 0.77	1.73 2.76	3.51 3.25	2.9 1.9	2.9 3.2	3.1 2.9	2.8

RESIDUES IN ANIMAL COMMODITIES

Farm animal feeding studies

Eighteen feeding studies in poultry were carried out in Australia, France, Israel, Japan and the US during the period 1979 and 2000. In the Australian study (Bull, 1985; Report No. 85/4/1019), hens were fed *ad libitum* at rates of 1.5 to 5 mg cyromazine/kg in their diet with treatment periods ranging from 3 to 35 days. Feed was pre-mixed with cyromazine as a 0.3% formulation. The residues found in tissues and eggs from the various treatments are shown in Table 81.

Table 81. Cyromazine residues following supervised feeding to poultry in Australia

mg ai/kg feed	Feeding / withholding	Residues, mg/kg		Feeding / withholding, days	Residues, mg/kg	
		muscle	liver		eggs	Observations
1.5	28 days / 0 days 35 days / 4 days	< 0.02 < 0.02		6 / 0 13 / 0 20-24 / 0 25-31 / 0 32-35 / 0 35 / 1 35 / 3 35 / 5	< 0.04 < 0.04 < 0.04 < 0.04 < 0.04 < 0.04 < 0.04 < 0.04	422 laying pullets were fed daily
3	1 days / 0 days 2 days / 0 days 3 days / 0 days 4 days / 0 days				< 0.04 < 0.04 < 0.04 < 0.04	
5	7 days / 0 h 7 days / 2h 7 days / 4h 7 days / 8h 7 days / 1 day 7 days / 2 days	0.03 0.04 0.04 < 0.02 < 0.02 < 0.02	0.12 0.12 0.12 0.09 0.05 < 0.05			12 hens were treated; 2 slaughtered at each withholding period
9	1 days / 0 days 2 days / 0 days 3 days / 0 days 4 days / 0 days				0.04 0.08 0.12 0.09	

In two studies conducted in France in 1985/1986, laying hens were fed for 35 days or 38 days with a feed containing 5 mg/kg cyromazine (10% formulation). Only the residue report was provided in both cases. The residues found in eggs from the treated hens are shown in Table 82.

Table 82. Cyromazine residues following supervised feeding to poultry in France at 5 mg/kg feed

Feeding / withholding, days	Residues in eggs, mg/g White / yolk	Reference (Report No.)
1 / 0	< 0.02 / < 0.02	Tournayre, 1986a (02/86)
4 / 0	0.06 / 0.04	
5 / 0	0.03 / 0.04	
15 / 0	0.15 / 0.16	
25 / 0	0.08 / 0.11	
35 / 0	0.12 / 0.05	
35 / 1	0.04 / 0.08	
35 / 3	< 0.02 / 0.04	
35 / 5	< 0.02 / 0.03	
35 / 7	< 0.02 / < 0.02	
35 / 9	< 0.02 / < 0.02	
35 / 11	< 0.02 / < 0.02	
35 / 22	< 0.02 / < 0.02	

Feeding / withholding, days	Residues in eggs, mg/g White / yolk	Reference (Report No.)
1 / 0	0.06 / < 0.02	Tournayre, 1986b (03/86)
3 / 0	0.15 / < 0.02	
6 / 0	0.12 / 0.07	
15 / 0	0.12 / 0.05	
24 / 0	0.15 / 0.11	
35 [a] / 0	< 0.02 / 0.03	
38 / 0	0.08 / 0.04	
38 / 1	0.08 / 0.06	
38 / 3	< 0.02 / 0.03	
38 / 5	< 0.02 / < 0.02	
38 / 7	< 0.02 / < 0.02	
38 / 9	< 0.02 / < 0.02	
38 / 11	< 0.02 / < 0.02	
38 / 20	< 0.02 / < 0.02	

a - Treatment interrupted from day 28 to day 34

A trial with broiler chickens was conducted in Israel in 1987 (Altenburger, 1987; Report No. 4010/87). Two groups of eight animals (four males and four females) were fed with a diet mixed with cyromazine formulation at the 5 and 50 mg/kg levels for 31 days. The animals were sacrificed four hours after removal of the feed and tissues collected for analysis. At the 5 mg/kg treatment level, residues of cyromazine in each animal ranged from 0.04 to 0.09 mg/kg (mean/median of 0.06 mg/kg) in breast muscle and from 0.05 to 0.10 mg/kg (mean/median of 0.07 mg/kg) in liver At the higher dose level, residues in muscle ranged from 0.52 to 0.71 mg/kg (mean/median = 0.52/0.54 mg/kg) and in liver from 0.36 to 0.76 mg/kg (mean/median = 0.56/0.59 mg/kg).

One study was conducted in the Philippines in 1982 with laying hens fed for 60 days with a 1% cyromazine formulation/feed mixture at a final concentration of 1.6 mg/kg (Gianonne, 1982; Analytical Report No. RVA4021/82 and Gianonne, 1983; Analytical Report RVA4021/82A). The residues of cyromazine found in tissues and the white of the eggs are shown in Table 83. The yolk samples were lost before analysis.

Table 83. Cyromazine residues following feeding to poultry in the Philippines at 1.6 mg/kg feed

Feeding / withholding, days	Residues, mg/kg			
	Thigh muscle	liver	fat	Eggs (white)
1 / 0	0.06	0.05	< 0.025	< 0.02
3 / 0	< 0.025	< 0.04	< 0.025	< 0.02
6 / 0	0.06	< 0.04	< 0.025	< 0.04
29 / 0	0.03	< 0.04	< 0.025	0.02
60 / 2	< 0.025	< 0.04	< 0.025	< 0.02
60 / 5	< 0.025	< 0.04	< 0.025	< 0.04
60 / 9	< 0.025	< 0.04	< 0.025	-

In two studies conducted in 1979 and 1981 in the USA (Seim, 1979 a, b; Seim 1981a, b), laying hens were fed cyromazine daily at levels of 2.5, 5.0 and 25 mg/kg in the feed. One or two birds were sacrificed during the studies and tissues collected for analysis. Eggs were collected daily and analysed as such, or separated into yolks and whites. In another study (Boone and Cheung 1985), three groups of white leghorn hens, each consisting of 30 birds of approximately equal egg productions were treated feed with 5 mg/kg cyromazine prepared using a 0.3% formulation. On days 14, 28, 42, 56 and 1, 3, 7 and 14 days after the treatment were withdrawn, three birds from each group were sacrificed and tissues removed for analysis. The results of these three studies are shown in Table 84.

Table 84. Cyromazine residues following feeding to poultry in the USA

mg ai/kg feed	Feeding / withholding, days	Residues, mg/kg					Reference, (Report No.)
		Muscle	liver	skin	fat	Egg White / yolk	
2.5	1/ 0					< 0.05 / < 0.05	Unknown, 1979a (5447) Unknown, 1979b (5447-2) (only analytical report)
	4/ 0					< 0.05 / < 0.05	
	7/ 0					0.06 / < 0.05	
	10/ 0					0.08 / < 0.05	
	13-14/ 0	< 0.05 [a]	0.08	< 0.05	< 0.05	< 0.05 / 0.07	
	21/ 0					< 0.05 / 0.06	
	27/ 0	0.05[a]	< 0.05	< 0.05	< 0.05	< 0.05 / < 0.05	
5	1/ 0					< 0.05 / < 0.05	
	4/ 0					0.10 / 0.06	
	7/ 0					0.13 / 0.06	
	10/ 0					0.07 / < 0.05	
	13-14/ 0	0.07[a]	0.10	< 0.05	< 0.05	0.09 / 0.08	
	21/ 0					0.11 / 0.06	
	27/ 0	0.06[a]	0.09	< 0.05	< 0.05	0.11 / 0.07	
25	1/ 0					< 0.05 / < 0.05	
	4/ 0					0.38 / 0.33	
	7/ 0					0.60 / 0.20	
	10/ 0					0.28 / 0.23	
	13-14/ 0	0.41[a]	0.41	0.15	< 0.05	0.44 / 0.51	
	21/ 0					0.52 / 0.37	
	27/ 0	0.29[a]	0.41	0.09	< 0.05	0.37 / 0.15	
5	0/ 0					< 0.05	Cheung, 1981a (6511) Cheung, 1981b (6512) (only analytical report
	1/ 0					< 0.05	
	3/ 0					0.08	
	7/ 0	0.07	0.10	0.06	< 0.05	0.16	
	10/ 0					0.11	
	14/ 0	0.08	0.08	< 0.05	< 0.05	0.16	
	17/ 0					0.11	
	21/ 0	0.08	0.13	< 0.05	< 0.05	0.10	
	24/ 0					0.09	
	28/ 0	0.05	0.09	< 0.05	< 0.05	0.12	
25	0/ 0					< 0.05	
	1/ 0					< 0.05	
	3/ 0					0.33	
	7/ 0	0.42	0.75	0.30	< 0.05	0.80	
	10/ 0					0.49	
	14/ 0	0.43	0.67	0.22	< 0.05	0.73	
	17/ 0					0.32	
	21/ 0	0.35	0.30	0.11	< 0.05	0.65	
	24/ 0					0.49	
	28/ 0	0.24	0.50	0.18	< 0.05	0.50	
50	0/ 0					< 0.05	
	1/ 0					< 0.05	
	3/ 0					1.6	
	7/ 0	1.1	1.1	0.31	< 0.05	1.1	
	10/ 0					2.2	
	14/ 0	0.93	0.92	0.40	< 0.05	1.3	
	17/ 0					2.4	
	21/ 0	0.86	0.85	0.16	< 0.05	2.1	
	24/ 0					1.8	
	28/ 0	0.57	0.63	0.13	< 0.05	2.0	

mg ai/kg feed	Feeding / withholding, days	Residues, mg/kg					Reference, (Report No.)
		Muscle	liver	skin	fat	Egg White / yolk	
5	0/ 0					< 0.05	Boone and Cheung 1985 (ABR-84082) [b]
	1/ 0					< 0.05	
	3/ 0					0.10	
	7/ 0					0.09	
	14/ 0	0.06	0.09	< 0.05	< 0.05	0.10	
	28/ 0	0.06	0.08	< 0.05	< 0.05	0.09	
	42/ 0	0.07	0.10	< 0.05	< 0.05	0.09	
	56/ 0	0.06	0.09	< 0.05	< 0.05	0.10	
	56 / 1	< 0.05	< 0.05	< 0.05	< 0.05	0.10	
	56 / 2					< 0.05	
	56 / 3	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	
	56 / 4					< 0.05	
	56 / 7	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	

a - mean residues found in breast and thigh muscle;

b - residues represented the mean of duplicate samples

Two studies were conducted in the USA in 1981 and 1986 with hens fed cyromazine in the feed at 5 mg/kg level. In the study reported by Cheung (1981), two birds were fed for 37 days and eggs collected three days after the treatment was terminated. Cheng and Oakes (1986) reported the result of an 84 day study conducted in California using a feeding level of 5 mg/kg cyromazine in the poultry diet. The birds were fed with cyromazine either on a continuous daily basis or on an alternative schedule, with seven days cyromazine feed followed by seven days without cyromazine in their diet. Eggs were taken at weekly intervals during the study in both treatment regimes. The number of birds used in the study was not reported. The levels of cyromazine found in eggs in both studies are shown in Table 85.

Table 85. Cyromazine residues following feeding to poultry in the USA at 5 mg/kg level

Feeding regime	Feeding / withholding, days	Residues in eggs, mg/kg	Reference (Report No.)
Continuous feeding	37 / 3	0.07	Cheung, 1981c (6288)
Continuous feeding	7/ 0	0.09	Cheung and Oakes, 1986 (ABR-86104)
	14/ 0	0.10	
	21/ 0	0.10	
	28/ 0	0.12	
	42/ 0	0.11	
	56/ 0	0.10	
	70/ 0	0.12	
	84/ 0	0.12	
Alternate feeding, i.e., fed with cyromazine in diet for 7 days and off for 7 days)	7/ 0	0.12	
	14/ 0	< 0.05	
	21/ 0	0.12	
	28/ 0	< 0.05	
	35/ 0	0.11	
	42/ 0	< 0.05	
	49/ 0	0.09	
	56/ 0	< 0.05	
	63/ 0	0.13	
	70/ 0	< 0.05	
	77/ 0	0.08	
	84/ 0	< 0.05	

In one study conducted in Japan in 2000, 36 laying hen were fed cyromazine for 28 days at a 5 mg/kg level (Inoue, 2000; Report No. 99-146-II). At each sampling day (2 h, 1, 2 and 3 days after the treatment was terminated), nine birds were sacrificied and tissues samples taken for analysis. Three eggs were collected from the birds at each sampling day and separated into white and yolk for analysis. The results are shown in Table 86. This study was conducted according to GLP.

Table 86. Cyromazine residues following feeding to hens in the Japan at 5 mg/kg level

Feeding / withholding, days	Residues, mg/kg [a]					
	Muscle	liver	Kidney	skin	fat	White / yolk
28 / 2h	0.05	0.07	0.09	0.02	< 0.02	0.04 / 0.07
28 / 1	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02	< 0.02 / 0.05
28 / 2	< 0.02	< 0.02	< 0.02	< 0.02	-	< 0.02 / 0.03
28 / 3	-	-	-	-	-	- / 0.02

a - Residues in tissues in a mean found in duplicate samples of nine birds in each sampling time

Cattle feeding studies

Poultry manure can be used as a fertilizer in cow pasture and might be used as a cattle feed supplement. Two feeding studies were conducted in the USA using dairy Holstein cows (Clayton, 1983 and Hacket, 1992). In the first study, three cows were dosed at 5 mg ai/kg diet, two cows at 25 mg ai/kg diet, and three cows at 50 mg ai/kg diet. Animals were sacrificed on test days 14, 21, and 28. The treated cows sacrificed on test day 14 had a 5 to 6.5 h post-treatment interval prior to sacrifice. In the second study, cows received diets containing 0, 10, 50, and 100 mg ai/kg diet (three cows per group) for 28 days. Tissue samples were taken at sacrifice on test days 14, 21, and 28 after a 20 – 24 h post-dosage interval. In both studies, milk samples consisted of pooled aliquots from the evenings and the following morning's milk. The results of the two studies are shown in Table 87.

Table 87. Summary of cyromazine residues following supervised feeding to dairy cattle in the USA

Application		Residues [mg/kg]					Reference (Report No.)
mg ai/kg feed	Sample, days	Round / tenderloin meat [a]	liver	kidney	fat [b]	milk	
5	0					< 0.01 [c]	Clayton, 1983 (ABR-83067)
	1					0.01 [c]	
	7					0.02 [c]	
	14	0.12 / < 0.05	0.09	0.13	< 0.05	0.03 [c]	
	20-21	< 0.05 / < 0.05	< 0.05	< 0.05	< 0.05	0.03 [c]	
	27-28	< 0.05 / < 0.05	< 0.05	0.06	< 0.05	0.03 [c]	
25	0					< 0.01 [c]	
	1					0.08 [c]	
	7					0.08 [c]	
	14	0.37 / 0.20	0.28	0.88	< 0.05	0.12 [c]	
	20-21	0.09 / < 0.05	< 0.05	0.11	< 0.05	0.11 [c]	
	27-28	0.13 / 0.08	< 0.05	0.17	< 0.05	0.07	
50	0					< 0.01 [c]	
	1					0.19 [c]	
	7					0.19 [c]	
	14	0.59 / 0.31	0.63	1.9	< 0.05	0.26 [c]	
	20	0.26 / 0.14	0.20	0.44	< 0.05	0.24 [c]	
	27-28	0.16 / 0.11	0.16	0.46	< 0.05	0.08	
10	0					< 0.01 [c]	Darnow, 1992 (ABR-91080)
	1					0.04 [c]	
	4					0.04 [c]	
	7					0.04 [c]	
	12-14	< 0.05 / < 0.05	< 0.05	< 0.05	< 0.05	0.04 [c]	
	19-21	< 0.05 / 0.05	< 0.05	< 0.05	< 0.05	0.05 [c]	
	26-28	< 0.05 / 0.05	< 0.05	0.09	< 0.05	0.06	

Application		Residues [mg/kg]					Reference (Report No.)
mg ai/kg feed	Sample, days	Round / tenderloin meat [a]	liver	kidney	fat [b]	milk	
50	0					< 0.01 [c]	
	1					0.19 [c]	
	4					0.22 [c]	
	7					0.20 [c]	
	12-14	0.16 / 0.17	0.18	0.42	< 0.05	0.22 [c]	
	19-21	0.16 / 0.13	0.16	0.49	< 0.05	0.17 [c]	
	26-28	0.11 / 0.16	0.12	0.66	< 0.05	0.22	
100	0					< 0.01 [c]	
	1					0.33 [c]	
	4					0.45 [c]	
	7					0.51 [c]	
	12-14	0.13 / 0.17	0.16	0.30	< 0.05	0.40 [c]	
	19-21	0.30 / 0.32	0.27	0.63	< 0.05	0.41 [c]	
	26-28	0.20 / 0.25	0.21	0.80	< 0.05	0.39	

a - Highest residue in round or tenderloin muscle

b - Omental or peri-renal fat;

c - Mean of 2-3 composite samples (evening and morning).

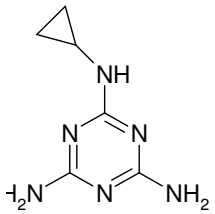
RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

The Australian Government provided a summary of the results of the 2004–2005 and the 2005–2006 National Residue Survey program of monitoring sheep. No residues of cyromazine or melamine were found in the 599 sheep kidney samples analysed in the program. The residue definition of cyromazine in Australia is cyromazine.

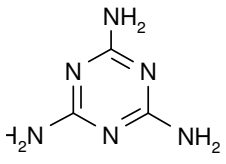
APPRAISAL – RESIDUE AND ANALYTICAL ASPECTS

Cyromazine was last evaluated by the JMPR in 2006 for toxicology within the Periodic Review program, where an ADI of 0-0.06 mg/kg bw and an ARfD of 0.1 mg/kg bw were established. The compound was listed at the 38th Session of the CCPR for periodic review for residues by the 2007 JMPR. Data submitted by the manufacturer include physical and chemical properties, metabolism in animals and plants, environmental fate in soils, residues in succeeding crops, analytical methods, storage stability, supervised trial on mangoes, vegetables and animal commodities and processing studies. Residue and information on good agricultural practices (GAP) was also submitted by the Netherlands.

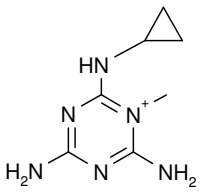
Cyromazine is a selective insecticide that acts by inhibiting the moulting process in insects, particularly in members of the Dipteran family. The figure below shows the compound structure and its main metabolites or degradation products found in animals, plants and/or soils. Metabolism and environmental fate studies submitted to the Meeting were conducted with [triazine- U-^{14}C]-cyromazine.



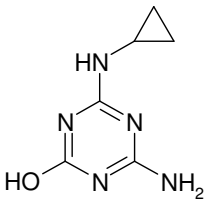
Cyromazine



Melamine



1-methyl cyromazine



Hydroxy cyromazine

Animal metabolism

Metabolism studies in rats evaluated by the 2006 JMPR showed that more than 97% of the administered [^{14}C]-cyromazine dose was excreted within 24 h, almost exclusively in the urine. Cyromazine was the major compound found in urine (71.5% of the applied radioactivity), with a further 7% attributed to melamine and 8 – 11% to hydroxy-cyromazine and 1-methyl-cyromazine.

Laying hens that received cyromazine at 5.0 ppm in the feed (equivalent to 0.5 mg/kg body weight/day) for seven consecutive days had > 99% of the applied radioactivity recovered in the excreta. Egg white and egg yolk had 0.4% and 0.2% of the total applied radioactive dose, respectively; for egg white and egg yolk, an average of 0.15 and 0.12 mg/kg cyromazine equivalents were found in the daily collected eggs of two animals, respectively. Cyromazine represented about 64% TRR in eggs; a metabolite (15.6% TRR) had the same retention volume in an ion exchange column as melamine, but no confirmation of the identity of this compound was performed. Hen tissue residues accounted for 0.1% of the total applied dose, with the highest radioactive levels found in liver, kidney, heart and muscle (0.032, 0.019, 0.10 and 0.09 mg/kg cyromazine equivalents, respectively). The residues in tissues were not characterized. Expired CO_2 and other volatiles accounted for < 0.1% of the applied dose.

Lactating goats dosed with [^{14}C]-cyromazine for ten consecutive days at levels of 4.6 ppm (low rate) and 48.4 ppm (high rate) in the feed had most of the administered dose excreted in the urine (86%) and faeces (6.6%). Residues in tissues represented < 2% of the applied dose, mostly found in liver (0.79 and 1.5 mg/kg cyromazine eq. for the low and higher dose, respectively) and kidney (0.043 and 0.44 mg/kg cyromazine eq). About 93% TRR was extracted from the liver, the majority (> 70%) as a non-identified metabolite with cyromazine and melamine representing 2 and 5.6% TRR, respectively. Radioactivity in milk accounted for < 0.4% of the applied dose and plateaued rapidly at both dose levels, with a mean of 0.017 and 0.32 mg/kg cyromazine eq found in the samples collected daily at the low and high dose levels, respectively. The radioactivity in the day 7 milk was mostly associated with the whey fraction, with cyromazine representing about 37% TRR and melamine 9.2 and 4.5% TRR for the low and higher dose, respectively.

In a second study, goats were dosed with cyromazine at 100 ppm in the feed for four consecutive days. The TRR was 74% of the applied dose, with 2.7 and 59% eliminated via faeces and urine, respectively; 7.4% of the total dose was found in the gastrointestinal tract. About 6% of the applied dose was recovered in the tissues, mostly in muscle (mean of 2.6%). Highest levels were found in kidney and liver (4.6 and 2.7mg/kg cyromazine eq, respectively). In liver, the metabolite 1-methylcyromazine was the major compound found (42% TRR), followed by the parent cyromazine (34% TRR). Kidney and muscle had mostly cyromazine (> 70% TRR) and only cyromazine was detected in fat. On average, a total of 0.76% of the applied dose was recovered in the milk, with an average of 0.66mg/kg cyromazine eq/day. Cyromazine was the major identified compound in milk (68% TRR) followed by melamine (2.9% TRR).

In one study conducted with a mature female sheep fed with [^{14}C]-cyromazine for nine consecutive days at a level of 5ppm in the diet (equivalent to 0.15mg/kg bw/day), 94% of the applied radioactivity was recovered, mostly in urine (89%). Tissues represented 0.09% of the applied radioactivity, mostly found in liver (0.17mg/kg cyromazine eq), kidney (0.048mg/kg eq) and muscle (0.13mg/kg eq). While the excreta extracts contained mostly cyromazine, the main compound found in liver extract was melamine (44% total radioactivity), with cyromazine corresponding to 12%.

In summary, animals dosed daily with [^{14}C]-cyromazine excreted over 90% of the radioactivity, mainly in urine. Eggs and milk represented < 0.5% and tissues < 2% of the applied or recovered radioactivity. Metabolism of cyromazine involves mainly dealkylation to melamine. Alkylation to 1-methylcyromazine was specific to ruminants; hydroxylation was identified in goats fed at 100 ppm. Cyromazine was the major compound found in hen eggs and milk of goats and sheep. The major compounds found in liver of goat and sheep were the metabolites 1-methylcyromazine and melamine, respectively.

Plant metabolism

In one greenhouse study conducted at a 0.28 kg ai/ha foliar rate in two phases, celery plants received two (1st phase) or six applications (2nd phase) and were sampled seven days after the second application (mature) in the 1st phase or third application (immature) and 14 days after the sixth application (mature) in the 2nd phase. Lettuce received two or four applications and heads were sampled seven days after the second application (mature or immature) and seven days after the fourth application (mature). Radioactivity in the plants was mostly extracted in the aqueous phase (> 74% TRR), with cyromazine representing the major residues (48 to 74% TRR in both plants) and melamine was the only metabolite found (11 to 33% TRR).

In a study conducted with tomatoes in a California field, six foliar applications at 0.28 kg ai/ha were made to the crop at two week intervals. Tomato samples were taken at 0, 7, and 14 days after the 4th or 6th applications and a stalk sample after the last application. TRR in the samples ranged from 64–98% of the applied radioactivity, mostly found in the aqueous extract (56 to 87% TRR) and being characterized as cyromazine (29 to 76% TRR) and melamine (11 to 44% TRR). While the residues ranged from 0.08 to 0.44 mg/kg cyromazine eq. in tomato samples, they reached 37 mg/kg eq in the stalk sample (aerial portion of the plant after removal of the tomato fruits).

Five trials conducted with non-radioactive cyromazine demonstrated that residues did not accumulate significantly in mushrooms under normal use conditions when the crop was grown in amended compost. Cyromazine residues were < 0.05 mg/kg in mushrooms in all trials/flushes with the exception of one trial at a target cyromazine concentration in the compost of 10 mg/kg for which the sample from the third flush contained a residue of 0.08 mg/kg. Residues of melamine were in the range 1.5 – 6.6 mg/kg at a compost of 5 mg ai/kg, and in the range 3.1 – 17 mg/kg at a compost of 10 mg/kg.

The results indicated that the major pathway of cyromazine in lettuce, celery and tomato was by dealkylation to form melamine, which can represent about 40% TRR. Melamine was the major compound found in cold treated mushroom.

Environmental fate in soil

Fourteen cyromazine aerobic degradation laboratory studies conducted with a variety of soils were submitted to the Meeting. Soil samples containing from 0.25 to 10.7 mg ai/kg (0.33 to 9.5 kg ai/ha) cyromazine were incubated for up to 12 months in the dark at temperatures ranging from 10 to 25 ˚C. In most studies, TRR was over 90% of the applied radioactivity. Melamine was the only or major degradation product found in the soils. In a 12 months/25 ˚C study conducted with a sandy loam soil (9 mg/kg cyromazine) 15% of the applied dose, at the end of the study, was identified as the carboxylic acid of cyromazine, formed through oxidative opening of the cyclopropane ring. Cyromazine half lives (DT_{50}) varied widely, ranging from 2.9 to 107 days, being lesser at lower temperatures, organic matter content and microbial biomass. In general, the degradation rate of melamine was slower than that of cyromazine.

The photolytic degradation of cyromazine was studied at 20 ˚C under artificial light on soil at a rate of 3.2 µg ai/cm^2 (corresponding to a field rate of 0.32 kg ai/ha). Samples were irradiated for 240 h, equivalent to 30 days natural summer sunlight. In moist irradiated soil, cyromazine degraded rapidly with a calculated DT_{50} of 3.5 days; in dry irradiated soil the level fell rapidly during the first 24 h and the degradation was slow thereafter (DT_{50} > 1 year). In both moist and dry soils, melamine was the major degradation product observed (53 and 15% of applied radioactivity in the moist and dry soil, respectively).

Eleven field soil studies conducted with cyromazine were submitted to the Meeting. In one study cyromazine was applied to bare soil at four different sites in various states in the USA during 1982/83. Plots were treated at an exaggerated rate of 5.6 kg ai/ha as a single application with a second application in Year 2. Residues in the soil at day 0 in both years ranged from 0.39 to 6.6 mg/kg in the 0 – 15 cm layer, with the highest value occurring from one site in Florida. Cyromazine dissipated at a slow rate at all four sites, and could remain unchanged for over 100 days. At the sites in California

and Nebraska, essentially no movement of cyromazine was observed below a depth of 15 cm; at the two sites in Florida, cyromazine residues were found to a depth of 30 – 45 cm in both years. Melamine was detected at all sites down to a depth of 30 – 45 cm towards the end of each year of the trial.

In four field studies carried out on bare soil plots in potato growing areas of Canada during 1993 and 1994, cyromazine was applied twice at 0.28 – 0.48 kg ai/ha. Cyromazine residues declined rapidly after the second application and continued to decline over the winter period at a slower rate, with half-lives ranging from 29 to 42 days. The majority of the parent residue was retained in the upper soil layer (0 – 15 cm) and most of the melamine residues remained in the top 30 cm. Melamine was present from background sources in the three topsoil layers up to 0.011 mg/kg.

In two field studies conducted in Switzerland and France in 2002/2003, soil samples (0 – 30 cm deep) from plots treated at 0.30 kg ai/ha were taken up to 360 days. In France, no residues were detected in soil below a depth of 20 cm and in Switzerland no cyromazine residues were found in soil below a depth of below a depth of 10 cm. DT_{50} for cyromazine calculated from the 0 – 10 cm layer residue data was 17 days and 2.3 days for France and Switzerland, respectively.

In one study conducted in Greece from 2001 to 2003, cyromazine was applied annually four times at a rate of 0.30 kg ai/ha at approximately seven day intervals to bare ground (sandy loam soil type). The plot was cultivated with mustard one to two days before the first application in each year. Cyromazine and melamine were found in some occasions at depths of up to 50 – 70 cm. The degradation of cyromazine was slower during a drought season of the first year (low biological activity) and during the winter.

In a study conducted in Spain, cyromazine was applied yearly to a loamy silt soil in four subsequent early summer applications at 0.30 kg ai/ha (2000 – 2003). Tomatoes were planted in the second year of the experiment just prior to the cyromazine application. Cyromazine residues were not found in soil layers deeper than 30 cm; melamine could be detected in soil layers up to 100 cm deep in the second and third year of the study, a possible result of higher than average rainfall during this period of the study. DT_{50} for cyromazine, calculated from the first year data, was 51 days.

The proposed metabolic pathway of cyromazine in soil involves the initial cleavage of the cyclopropyl ring moiety to form melamine, with a possible involvement of the carboxylic acid of cyromazine as intermediate. The rate of degradation of cyromazine under laboratory or field conditions vary widely, with estimated half lives ranging from a few days to over 100 days, depending on the soil type and environmental conditions.

Succeeding Crops

Five studies on succeeding crops conducted in the USA were submitted to the Meeting. In a greenhouse study conducted in Florida in 1982, celery seedlings were transplanted into a muck soil and [^{14}C]-cyromazine mixed with a sample of the soil applied as a top dressing to the crop at 1 kg ai/ha. Celery plants were harvested after 84 days and radishes or sweet corn immediately planted in the same container. About 78% TRR in celery stalks was found the aqueous extract, with cyromazine being the main residue found after 42 and 82 days after treatment (up to 0.45 mg/kg). Radioactivity in radish and mature sweet corn planted 84 days following foliar application of the compound to celery and harvested after 130 or 159 days after planting ranged from 0.01 to 0.02 mg/kg cyromazine eq and were too low for characterization.

In a field study conducted in California, tomatoes were treated in the fall with [^{14}C]-cyromazine at 6 × 0.28 kg ai/ha. Tomato plants were harvested 14 days after the last application and winter wheat planted immediately after that. Lettuce, carrot, soy bean and sugar beet were planted the following spring. Two samplings (immature and mature harvest) were taken from each crop for analysis. Radioactive residues in succeeding crops were ≤ 0.05 mg/kg for all crop parts other than half-mature carrot tops (0.19 mg/kg cyromazine eq). Most of the residues in this sample (93%) was partitioned into the aqueous layer and was characterized as 14% cyromazine and 79% melamine.

A study was performed in Georgia to determine the fate of cyromazine in chicken manure-amended soil and the uptake and metabolism of cyromazine residues by rotational crops. The soil was prepared by incorporating manure fortified with [^{14}C]-cyromazine at 5 mg/kg at a rate of 11.2 t/ manure/ha and aged for 30 days. Spring wheat, lettuce and sugar beets were planted in buckets containing a 7.6 cm top layer of cyromazine-treated soil and grown to maturity in the greenhouse. TRR ranged from < 0.01 to 0.011 mg/kg cyromazine eq in mature and immature lettuce leaves, sugar beet tops and beets and wheat grain. Immature stalks and mature wheat straw and hulls contained residues between 0.022 to 0.11 (straw) mg/kg eq, greater than the initial soil concentration of 0.064 mg/kg. Cyromazine and melamine accounted for 43% and 28% TRR in wheat straw, respectively.

In a study carried out in Mississippi, tomato crops were treated with 12 applications of cyromazine at 0.14 or 0.28 kg ai/ha, the tomatoes harvested at 14 days after last application and wheat planted 10 weeks after the last application. Cyromazine residues in samples of forage, straw and grain harvested 23 to 43 weeks after planting ranged from < 0.05 to 0.08 mg/kg with the highest value in forage. In California and Florida, 21 field trials were carried with celery treated with 11 – 15 foliar applications of cyromazine at rates from 0.14 to 0.28 kg ai/ha. Celery was harvested at 0 – 14 days after the last application and following a post-harvest interval of 1 – 6 weeks (Florida) or 8 weeks (California) the field plots were re-planted with sweet corn (eight trials), radishes (eight trials) and lettuce (five trials). Residues of cyromazine were detected in sweet corn forage (0.08 to 0.19 mg/kg), radish roots and tops (0.09 to 0.22 mg/kg) and lettuce leaf (0.05 to 0.08 mg/kg).

In summary, detectable residues of cyromazine and melamine can be found in crops cultivated after cyromazine treated plants have been harvested. Crops planted in cyromazine fortified manure-amended aged soil also showed detectable residues.

Analytical methods

Four analytical methods for the analysis of cyromazine and melamine in vegetable crops were provided. In methods developed in the 1980´s, residues can be extracted with water/methanol or pure methanol, cleaned up and partitioned with dichloromethane and hexane, followed by C18 and/or cation exchange columns and additional clean-up with amino column or gel permeation chromatography. The analytes are quantified by HPLC with an amino column and UV detection at 214/215 nm or by GC/NPD. Samples of lettuce, celery, tomato, mushrooms, cucurbits, peppers, grapes, peas and alfalfa, cotton, Sudan grass and/or barley products were fortified at levels that varied from 0.04 to 10 mg/kg level of cyromazine and melamine. In most cases, recoveries were within 70 – 120% range, however in only a few cases were replicate samples analysed.

In a method reported in 2001 (REM 174.02), samples of crops with high water content and fruits with high acid content, are macerated with an acidic solution of potassium dihydrogen phosphate and extracted with methanol. Crops with high fat content, cereals and other dry crops are macerated with water and then extracted with methanol by mechanical shaking. Celite is added, the mixture centrifuged, filtered, and the filtrate acidified. Analysis is by column switching HPLC using two cation exchange columns and detection within the range 215−245 nm depending on crop type and co-extractives. This method was fully validated for sunflower seeds, tomatoes, oranges, beans and potatoes fortified at 0.05 and 0.5 mg/kg (n=5 in each case). Recoveries of cyromazine and melamine ranged from 80 to 110%, with a coefficient of variation (CV) up to 5.2%.

Twelve methods for the analysis of cyromazine and melamine in food of animal origin were provided. The analytes could be extracted with methanol, methanol/water, ethanol or acetone, and the extracts cleaned-up on cation exchange, silica and/or celite column. In some methods, a solvent partitioning step was included before the clean-up, or replaced the clean-up. Quantification was by GC/NPD or MS in most cases, but also by HPLC/UV 214 nm. The methods were validated for eggs, milk and tissues fortified at 0.04 to 1 mg/kg. Recoveries were satisfactory in most cases, and whenever replicate samples were analysed, CV were < 20%. In one method reported in 2003 (RAM 394/01), cyromazine and melamine residues are extracted from liver, kidney, muscle tissue and milk with acetonitrile/water and from eggs with methanol/water. After centrifugation, aliquots are adjusted

to pH 4 with glacial acetic acid, subjected to cation exchange clean-up and the compounds quantified by HPLC-MS/MS. This method was fully validated at 0.01 and 0.1 mg/kg levels, with recoveries from 70 to 107% and CV up to 16% (n=5).

Stability of pesticide residues in stored analytical samples

Residues of cyromazine and melamine in crop samples fortified at 0.5 or 1.0 mg/kg levels were stable after being frozen for up to two years, with the amount remaining in the range of 80 to 110%. Residues of cyromazine, melamine and 1-methylcyromazine in beef tissues, eggs and milk fortified at 0.5 or 1.0 mg/kg levels were also stable for over three years under freezer conditions. Field-incurred residues of cyromazine and melamine in lettuce, celery and tomatoes samples at levels from 0.07 to 21.5 mg/kg increased more than 150% of the initial concentration after being stored for up to 23 months under freezer conditions.

Definition of the residue

In 1990, the JMPR defined the residue for cyromazine in food as cyromazine. At the 1992 JMPR, the possibility of including melamine in the definition was discussed, but the Meeting decided to maintain the previous definition as melamine was considered to be less toxic than cyromazine, and that melamine may have originated from sources other than from the use of cyromazine. Nevertheless the Meeting recognized that the monitoring of good agricultural practice in growing mushrooms under certain conditions was not possible when melamine was omitted from the residue definition.

Data submitted to the present Meeting have shown that melamine is the main metabolite found in all crops and most animal products. Cyromazine is the major compound found in all crops, with the exception of mushroom, where melamine can be present at levels higher than cyromazine. Most analytical methods analyse cyromazine and melamine and most of the supervised trials submitted to the Meeting contained data for both compounds. It is known that cyromazine is not the only source of melamine in agriculture and in the environment and that melamine can be a component in fertilizers and is used in a variety of manufacturing processes, including plastics. Data provided by the manufacturer have shown that, with the exception of Switzerland, the residue definition in most countries in all foods is cyromazine.

Based on the present knowledge and for practical purposes, the Meeting agreed that the residue definition for cyromazine for enforcement purposes for food of plant and animal origin should continue to be cyromazine.

Toxicological data evaluated by the 2006 JMPR confirmed that melamine is less toxic than the parent compound. The Meeting agreed that the definition for cyromazine in food of plant and animal origin, for dietary intake purposes, is cyromazine

The octanol-water partition coefficient of cyromazine is < 1 and the compound does not accumulate in fat of animals dosed with cyromazine. The Meeting concluded that cyromazine is not fat soluble.

Definition of residues (for compliance with MRL and for estimation of dietary intake) for plants and animal commodities: *cyromazine.*

Results of supervised trials on crops

Metabolism studies conducted in plants and plant residue data for melamine (not included in this evaluation) indicate that cyromazine is always present at a higher concentration than melamine in the treated crops considered in this evaluation. Cyromazine might be absent or present at lower concentration than melamine in treated mushrooms.

Mango

Cyromazine is registered in Mexico to be used as a foliar application in mango at rates of 0.07 to 0.1 kg ai/ha. The maximum number of applications is not specified on the label and the PHI is 0 day. Six trials were conducted with cyromazine in mango in Mexico from 1984 to 1993. The compound

was applied five times at 0.09 kg ai/ha and samples harvested from day 0 to day 28 after the last application. Residues of cyromazine in the whole fruit at 0 day PHI were: 0.06, 0.10, 0.11, 0.14(2) and 0.25 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg, an STMR of 0.125 mg/kg and an HR of 0.25 mg/kg for cyromazine in mango.

Onions

The registered use of cyromazine in bulb vegetables in the USA allows up to six foliar applications at 0.14 kg ai/ha rate with a 7 day PHI. A seed treatment at 5 g ai/100 g seed is also recommended, but with no PHI specified. Eight trials were conducted in 1993 with bulb onions using the seed treatment at the GAP rate and resulted in residues in the onion bulbs at 98 to 207 days PHI of < 0.05 (7) and 0.06 mg/kg. Residues in the whole plant at 60 – 75 days PHI were 0.09, 0.16, 0.27, 0.34, 0.35, 0.44, 0.83 and 1.7 mg/kg. Dried onion bulb samples (fresh bulbs dried in the field) from all trials gave residues of < 0.05 and 0.06 mg/kg. In nine trials conducted in 1999, using foliar application within US GAP, residues found in onion bulbs were: < 0.05 (7) and 0.07 (2) mg/kg.

The Meeting agreed that trials conducted at GAP using foliar and seed application gave residues in onion bulbs at the same level and could be combined. Residues in ranked order (median underlined) were: < 0.05 (14), 0.06 and 0.07 (2) mg/kg. The Meeting estimated a maximum residue level of 0.1 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.07 mg/kg for cyromazine in bulb onions.

Four trials were conducted with cyromazine in spring (green) onions in the USA in 1999 using six foliar applications at 0.14 to 0.17 kg ai/ha. Residues in the whole plant within 7 days PHI were 0.26, 0.30, 0.75 and 0.78 mg/kg. Data from immature plant from the bulb onion trials can be considered for the estimation for spring onion and the results from the trials can be combined. Residues in ranked order (median underlined) were: 0.09, 0.16, 0.26, 0.27, 0.30, 0.34, 0.35, 0.44, 0.75, 0.78, 0.83 and 1.7 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg, a STMR of 0.35 mg/kg and a HR of 1.7 mg/kg for cyromazine in spring onion.

Broccoli and cabbage

In the USA, cyromazine is registered for the brassica leafy vegetable group to be applied six times as a foliar application in the field at 0.41 kg ai/ha and 7 days PHI. In six trials conducted in broccoli at GAP, residues found in flower head and stem at 7 days PHI were: < 0.05, 0.05, 0.09, 0.21, 0.26 and 0.51 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg, an STMR of 0.15 mg/kg and an HR of 0.51 mg/kg for cyromazine in broccoli.

In six trials conducted at GAP rate in USA, residues in cabbage head (with wrapper leaves) were: 0.06, 0.10, 0.24, 0.28, 0.50 and 6.1 mg/kg;

The Meeting estimated a maximum residue level of 10 mg/kg, an STMR of 0.26 mg/kg and an HR of 6.1 mg/kg for cyromazine in cabbage head.

The IESTI calculation indicates that the consumption of cabbage at the HR level of 6.1 mg/kg will lead to an exceedance of the ARfD, but no residue data was available from an alternative GAP to estimate a lower maximum residue level.

Fruiting vegetables, Cucurbits

Cyromazine is registered for use in cucumber, melon and summer squash in many European countries at similar rates, with the PHI ranging from 3 to 14 days. In France, the maximum GAP rate is 0.3 kg ai/ha (field and protected), with a PHI of three days for cucumber and summer squash. Fifteen protected cropping or field trials were conducted in cucumber from 1999 to 2001 in France (6), Greece (2), Italy (5), Spain (1) and Switzerland (1) using three to four applications at 0.30 –

0.34 kg ai/ha rate. Residues found at 3 days PHI, in ranked order were: 0.30, 0.32, 0.33, 0.40, 0.43, 0.46, 0.50, 0.52 (2), 0.62, 0.71, 0.74, 0.79, 0.88 and 1.3 mg/kg.

In the USA, cyromazine is registered for the Cucurbits crop group with a recommendation of a maximum of six foliar applications at 0.14 kg ai/ha at 7 day intervals with a 0 day PHI. Due to the rapid rate of growth exhibited by cucurbits, it was considered unlikely that early applications would contribute significantly to the final residues. As a result, trials conducted with a higher number of applications were considered to comply with the US GAP. In five trials conducted in the USA on cucumbers in 1986 and 1990 where six or eight applications, at the GAP rate were made, residues at 0 days PHI were: 0.16 (2), 0.20, 0.22 and 0.56 mg/kg. In four trials conducted at the double rate, residues found were within the same range.

The Meeting considered that the data from the 20 trials conducted in cucumbers according to the GAPs of Europe and the USA belonged to the same population and could be combined. Residues in ranked order (median underlined) were: 0.16 (2), 0.20, 0.22, 0.30, 0.32, 0.33, 0.40, 0.43, 0.46, 0.50, 0.52 (2), 0.56, 0.62, 0.71, 0.74, 0.79, 0.88 and 1.3 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg, an STMR of 0.048 mg/kg and an HR of 1.3 mg/kg for cyromazine in cucumbers.

The Meeting recommended withdrawing the previous recommendation of 0.2 mg/kg for cyromazine in cucumbers.

In eight field trials conducted in summer squash in France, Italy and Spain, between 2000 and 2003, using three applications of 0.3 to 0.35 kg ai/ha (0.03 kg ai/hL) with a 3 day PHI, residues found in ranked order were: 0.11, 0.14, 0.15, 0.16, 0.18, 0.21 and 0.27 (2) mg/kg.

In seven trials conducted in the USA from 1986 to 1990 based on the cucurbits GAP rate (six or eight applications), residues at 0 days PHI were 0.07 (2), 0.11(2), 0.18, 0.22 and 1.0 mg/kg. In five trials conducted at double rate, residues were within the same range.

The Meeting considered that the residues from 15 trials conducted in summer squash according to European GAP at 3 days or 14 days PHI and in USA according to US GAP belonged to the same population and could be combined. Residues in ranked order (median underlined) were: 0.07 (2), 0.11 (3), 0.14, 0.15, 0.16, 0.18 (2), 0.21, 0.22, 0.27 (2) and 1.0 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg, an STMR of 0.16 mg/kg and an HR of 1 mg/kg for cyromazine in summer squash.

GAP for melons in Spain is 0.3 kg ai/ha (14–30 days interval) with a PHI of 3 days. In France, the rate is also 0.3 kg ai/ha (10 – 15 days interval) and a PHI of 7 days. A total of fourteen trials (field and glasshouse) matching French GAP were conducted in melons in France, Italy and Spain from 1998 to 2001 using three applications of 0.28 to 0.33 kg ai/ha (7 days interval). Residues in the whole fruit were: < 0.05, 0.06 (3), 0.07, 0.08, 0.09 (2), 0.11, 0.12, 0.13, 0.16, 0.18 and 0.25 mg/kg.

In seven trials conducted in the USA in 1986 in melons and cantaloupe using eight applications at the GAP rate, residues at 0 days PHI were: < 0.05, 0.08, 0.09, 0.11 (2), 0.13 and 0.45 mg/kg. In one trial conducted in watermelon at the same rate, residues found were 0.13 mg/kg.

The Meeting considered that residues from trials conducted in melons according to GAP in Europe and the USA appeared to be from similar populations and could be combined. Residues in melons from the 21 trials, in ranked order (median underlined), were: < 0.05 (2), 0.06 (3), 0.07, 0.08 (2), 0.09 (3), 0.11 (3), 0.12, 0.13 (2), 0.16, 0.18, 0.25 and 0.45 mg/kg.

In some of 3 day PHI trials conducted in melons in Europe, residues in fruit were calculated from the levels found in the pulp and in the peel; residues in the pulp were < 0.05 (4) mg/kg. The residue ratio fruit/pulp calculated from these and other trials was 1.1, > 1.6, > 2.4, > 2.6, > 3 and > 3.2, with an estimated mean of > 2.3. A fruit/pulp ratio of 2.3 was applied to the median and highest residues for melons (0.09 and 0.45 mg/kg), estimating the median and the highest residue in melon pulp as 0.04 and 0.19 mg/kg.

The Meeting estimated a maximum residue level of 0.5 mg/kg for cyromazine in melons, except watermelons, an STMR of 0.04 mg/kg and an HR of 0.19 mg/kg for cyromazine in melons (pulp).

The Meeting recommends withdrawing of the previous recommendation of 0.2 mg/kg for cyromazine in melons, except watermelons.

Fruiting vegetables, other than Cucurbits

Cyromazine is registered in tomato and eggplant in many European countries for either in the field or under protected cropping. In France (F&P) and Italy (F) the maximum application rate is 0.3 mg/kg ai/ha. The recommended PHI is 3 days in France and 14 days in Italy. Eighteen foliar trials complying with French GAP were conducted in France, Greece, Italy and Spain under field or glasshouse conditions with four foliar applications at 0.3 to 0.34 kg ai/ha. Residues at 3 days PHI were: 0.05, 0.09, 0.11 (3), 0.13 (2), 0.14, 0.15, 0.16, 0.18, 0.21, 0.22, 0.23, 0.29, 0.34, 0.42 and 0.58 mg/kg.

Application through irrigation is also recommended in Italy and Greece, at rates of 0.75 and 0.98 kg ai/ha with a PHI of 14 days. Only one of the four trials conducted in Greece, Italy and Spain using either drip or soil drench irrigation matched the GAP of Italian and Greece, giving residues of cyromazine at 14 days PHI of < 0.05 mg/kg.

In the USA, cyromazine can be applied to tomatoes up to six times at a rate of 0.14 kg ai/ha with a 0 day PHI. In 13 trials conducted according to GAP, residues at day 0 were: 0.09, 0.10, 0.11, 0.12, 0.13, 0.14, 0.18, 0.21 (2), 0.22, 0.26, 0.28 and 0.30 mg/kg. Five trials conducted at double rate gave residues in the same range.

The Meeting considered that residues in tomatoes from 31 foliar trials conducted in Europe and the USA matching GAP belonged to the same population and could be combined. Residues found, in ranked order (median underlined) were: 0.05, 0.09 (2), 0.10, 0.11 (4), 0.12, 0.13 (3), 0.14 (2), 0.15, 0.16, 0.18 (2), 0.21 (3), 0.22 (2), 0.23, 0.26, 0.28, 0.29, 0.30, 0.34, 0.42 and 0.58 mg/kg.

In four field trials conducted with eggplant in France and Switzerland three applications were made at 0.30 to 0.36 kg ai/ha, residues found at a 3 day PHI were: 0.05, 0.14, 0.23 and 0.26 mg/kg.

The Meeting considered that the residues of cyromazine found in tomatoes and eggplant belonged to the same population and could be combined. Residues from the trials, in ranked order (median underlined) were: 0.05 (2), 0.09 (2), 0.10, 0.11 (4), 0.12, 0.13 (3), 0.14 (3), 0.15, 0.16, 0.18 (2), 0.21 (3), 0.22 (2), 0.23 (2), 0.26 (2), 0.28, 0.29, 0.30, 0.34, 0.42 and 0.58 mg/kg.

In the USA cyromazine may be applied to peppers at up to six applications at a rate of 0.14 kg ai/ha with a PHI of 0 days. Data from 12 US trials on chilli and bell pepper in 1984/1985 did not comply with GAP as 12 applications were made at 0.14 or 0.28 kg ai/ha, residues found at 0 day ranged from 0.10 to 0.95 mg/kg. Although these trials were conducted with a higher number of applications, residues found at 0 days PHI were within the same range as residues found in tomato and eggplants. The Meeting decided that data from trials on tomato and eggplants, conducted according to GAP, supported the residues found in peppers.

Based on the residues found in tomato and eggplant, the Meeting estimated a maximum residue level of 1 mg/kg, an STMR of 0.16 mg/kg and an HR of 0.58 mg/kg for cyromazine in fruiting vegetables, other than cucurbits, except sweet corn (on-the-cob) and mushrooms.

The Meeting recommends withdrawing the previous recommendations of 0.5 mg/kg for cyromazine in tomato and of 1 mg/kg for cyromazine in peppers.

Mushrooms

Spanish GAP allows cyromazine application to mushrooms at rates up to 0.75 g ai/m^2; Swiss GAP allows treatment at 1 g/m^2 to the casing layer or compost with a PHI of 14/15 days. In France, the application rate is 0.4 g/m^2 with a 14 day PHI. In the USA, cyromazine can be used as a coarse drenching spray at a maximum rate of 0.57 g ai/ m^2, with no specified PHI. Nine mushroom-house trials were conducted in France, Italy and Switzerland from 1986 to 2001 using one or two

applications of cyromazine at 0.4 to 0.8 g ai/m^2. In four trials conducted according to French GAP, residues of cyromazine were 0.37, 1.3, 2.4 and 4.2 mg/kg. Samples collected at a higher PHI in three other trials giving residues of 0.75, 2.2 and 2.8 mg/kg were also considered for MRL estimation. Two trials conducted at double rate gave residues in the same range. Residues considered for the estimation of an MRL and STMR level were: 0.37, 0.75, 1.3, 2.2, 2.4, 2.8 and 4.2 mg/kg.

The Meeting estimated a maximum residue level of 7 mg/kg, an STMR of 2.2 mg/kg and an HR of 4.2 mg/kg for cyromazine in mushrooms.

The Meeting recommends withdrawing of the previous recommendations of 5 mg/kg for cyromazine in mushroom.

Lettuce

Cyromazine is approved for use on lettuce in a number of European countries. French and Italian GAP for field grown lettuce allows a maximum rate of 0.3 kg ai/ha with a PHI of 21 days and 14 days, respectively. In Spain and Switzerland, GAP comprises a maximum rate of 0.03 kg ai/hL or 0.3 kg ai/ha with a PHI of 7 days in the production of either field grown or protected lettuce. Between 1993 and 2002, 40 trials were conducted in Europe in head and leaf lettuce in both field and greenhouse situations using three applications at 0.19 to 0.30 kg ai/ha (0.03 to 0.1 kg ai/hL). These trials were evaluated against either the GAP of Italy (PHI of 14 days) or of Spain/Switzerland (PHI of 7 days).

In three field trials conducted in France in head lettuce complying with the Spanish GAP rate, residues at 14 days PHI were: < 0.03, 0.34 and 1.7 mg/kg. In four protected cropping trials (plastic tunnels), residues were: 2.2, 2.9, 3.0 and 4.9 mg/kg.

In 14 field trials conducted in France, Italy, Spain and Switzerland in Cos lettuce (leaf) complying with the Italian GAP rate, residues at 7 days PHI were: 0.15, 0.18, 0.19, 0.22, 0.24, 0.27, 0.28, 0.34, 0.45, 1.3, 1.5, 1.8 (2) and 2.0 mg/kg. Residues in trials according to Spanish or Italian GAP with PHIs of 7 or 14 days in 17 protected trials were: 0.13, 0.55, 1.5, 2.4, 2.8, 3.3, 4.7, 5.2 (2), 5.8 (2), 6.2, 6.5, 7.9, 11, 14, 15 and 18 mg/kg.

On the basis of the European trials, the Meeting concluded that residues conducted with head and Cos lettuce using the same rate gave residues in the same range. Residues from 17 field trials conducted in lettuce in Europe in ranked order (median underlined), were: < 0.03, 0.15, 0.18, 0.19, 0.22, 0.24, 0.27, 0.28, 0.34 (2), 0.45, 1.3, 1.5, 1.7, 1.8 (2) and 2.0 mg/kg. Residues from 22 protected trials were: 0.13, 0.55, 1.5, 2.2, 2.4, 2.8, 2.9, 3.0, 3.3, 4.7, 4.9, 5.2 (2), 5.8 (2), 6.2, 6.5, 7.9, 11, 14, 15 and 18 mg/kg.

In the USA, GAP for leafy vegetables, including brassica leafy vegetables, is a maximum of five or six applications (six for head lettuce, five for all other leafy vegetables) at 0.14 kg ai/ha with a 7 days PHI. In nine trials conducted with head lettuce using eight applications at 0.14 or 0.28 kg ai/ha rate, residues seven days after the final application ranged from < 0.05 to 3.8 mg/kg. In nine field trials conducted with leaf lettuce using five applications at 0.14 kg ai/ha, residues at seven days after the final application were: 0.58, 1.5, 1.6, 2.0, 2.8 (2), 3.9, 4.4, 5.2 mg/kg.

The Meeting considered the field and protected cropping trials conducted in Europe and the field trials conducted in the USA were from different populations and could not be combined. The Meeting considered that the 22 protected trials, conducted in Europe, reflected the most critical use in lettuce.

The Meeting estimated a maximum residue level of 25 mg/kg, an STMR of 2.8 mg/kg and an HR of 18 mg/kg for cyromazine in head lettuce and leaf lettuce.

The IESTI calculation indicates that the consumption of lettuce, at the HR level of 18 mg/kg coming from protected trials, would lead to an exceedance of the ARfD. Consequently, the Meeting used the prospective alternative GAP approach and selected the USA residue data for the maximum residue level estimation.

The Meeting estimated a maximum residue level of 10 mg/kg, an STMR of 2.8 mg/kg and an HR of 5.2 mg/kg for cyromazine in head lettuce and leaf lettuce

The IESTI calculation indicates that the consumption of lettuce at the HR level of 5.2 mg/kg, coming from the USA field trials, would lead to an exceedance of the ARfD for children. The Meeting once again used the prospective alternative GAP approach and selected the EU residue data population, coming from field trials, for the recommendations.

The Meeting estimated a maximum residue level of 4 mg/kg, an STMR of 0.34 mg/kg and an HR of 2 mg/kg for cyromazine in head lettuce and leaf lettuce.

Mustard greens

Five trials were conducted in the USA according to the GAP for brassicas leafy vegetables (maximum of five applications at 0.14 kg ai/ha with a 7 days PHI). Residues in mustard greens (leaves) were: 1.1, 1.6, 2.7, 6.5 and 7.4 mg/kg.

The Meeting estimated a maximum residue level, an STMR value and an HR value for cyromazine in mustard greens of 10, 2.7 and 7.4 mg/kg.

Spinach

From 16 trials conducted with spinach in the USA, eight were conducted according to GAP for leafy vegetables. Residues at 7 days PHI were: 0.4, 1.1, 1.2, 1.8, 2.3, 4.2, 5.4 and 6.1 mg/kg. The Meeting estimated a maximum residue level of 10 mg/kg, an STMR value of 2.0 mg/kg and an HR value of 6.1 mg/kg for cyromazine in spinach.

The IEST calculation indicates that the consumption of spinach at the HR level of 6.1 mg/kg will lead to an exceedence of the ARfD, however no data was available for an alternative GAP review to estimate a lower maximum residue level.

Beans

US GAP allow six applications of cyromazine in lima bean and dry beans at a rate of 0.14 kg ai/ha with a PHI of 7 days. In nine trials conducted in lima beans complying with GAP from the USA, residues found in ranked order (median underlined), were: < 0.05, 0.11, 0.17, 0.19, 0.23 (2), 0.32, 0.38 and 0.58 mg/kg. Trials conducted with eight applications at 0.14 kg ai/ha or 6 applications at 0.28 kg ai/ha gave residues in the range of 0.23 to 1.0 mg/kg.

The Meeting estimated a maximum residue level of 1 mg/kg, an STMR of 0.23 mg/kg and an HR of 0.58 mg/kg for cyromazine in lima beans.

In nine trials in dry beans from the USA complying with that countries GAP, residues found in ranked order (median underlined), were: 0.23, 0.68, 0.84, 0.97, 1.0, 1.1 (2), 1.2 and 1.8 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg and an STMR of 1 mg/kg for cyromazine in beans (dry).

Potato

In Spain cyromazine may be applied to potatoes at a maximum rate of 0.24 kg ai/ha (0.02 kg ai/hL) with a PHI of 21 days. In Italy, field and greenhouse GAP consists of an application rate of 0.3 kg ai/ha with a PHI of 35 days. In eight potato trials conducted in Spain using three applications at 0.17 to 0.33 kg ai/ha with samples harvested at 21 or 35 days post application. Four trials could be evaluated against Spanish or Italian GAP, with residues of < 0.05, 0.06, 0.11, and 0.12 mg/kg.

The Meeting decided that there were insufficient trials submitted, complying with GAP, and did not estimate a maximum residue level.

Stalk and stem vegetables

In Spain, cyromazine can be used in artichoke at 0.03 kg ai/hL with a PHI of 7 days. In four Spanish trials conducted complying with that countries GAP, residues were: 0.85, 0.95, 1.1 and 1.3 mg/kg.

The Meeting estimated a maximum residue level of 3 mg/kg, an STMR of 1 mg/kg and an HR of 1.3 mg/kg for cyromazine in artichoke.

In France, cyromazine can be applied to celery in the field or in greenhouses at an application rate of 0.3 kg ai/ha with a PHI of 14 days. In 11 trials conducted in France and Spain from 1998 to 2003 using three or four applications at 0.3–0.36 kg ai/ha, residues, in ranked order (median underlined), were: 0.27 (3), 0.36, 0.57, 0.58, 0.60, 0.68, 1.6, 1.8 and 2.3 mg/kg. The commodity description in the trials specified whole plant or stems and was interpreted by the Meeting as matching the Codex description for celery (whole commodity).

Fourteen trials were conducted in the USA in celery at a greater than GAP application frequency (maximum of six applications at 0.14 kg ai/ha with a 7 days PHI). Residues seven days after the last application ranged from 0.05 to 13 mg/kg.

Based on the European trials, the Meeting estimated a maximum residue level of 4 mg/kg, an STMR value of 0.58 mg/kg and an HR value of 2.3 mg/kg for cyromazine in celery.

Fate of residues in processing

Hydrolysis studies representing food processing procedures of pasteurization, baking, boiling, brewing and sterilization were conducted with [^{14}C]-cyromazine in buffer solution at pH 4, 5 and 6, incubated for 20 or 60 minutes at 90 – 120 ˚C, showed that cyromazine was the only compound found at the end of the incubation period.

In a tomato processing study in the USA, tomatoes were harvested seven days after the last of six applications at 0.140 or 0.250 kg ai/ha. Treated samples were pooled and subjected to treatments simulating normal commercial processing. Residues decreased in washed fruit, canned tomato, tomato juice and in ketchup, with mean processing factors (PF) of 0.71, 0.53, 0.75 and 0.84. Residues in wet pomace remained unchanged, but increased in dry pomace (PF=2.8), puree (PF=1.2) and paste (PF=2.1).

Based on the estimated PFs and STMR for tomato of 0.16 mg/kg, the Meeting estimated an STMR-P of 0.11 mg/kg for washed tomato, 0.09 mg/kg for canned tomato, 0.12 for tomato juice, 0.13 mg/kg for ketchup, 0.19 mg/kg for puree and 0.34 mg/kg for tomato paste.

In one study conducted with potatoes, samples from six trials conducted in USA in 1996 were processed according to commercial practices. Mean cyromazine residues in the raw potato was 0.71 mg/kg, which decreased in peeled/rinsed potatoes with a mean PF of 0.9, but increased in potato chips (PF=1.3) and potato granules (PF=2.8). As no recommendation was made for the raw commodity potato, the Meeting did not make a recommendation for processed potato products.

Residues in animal commodities

Direct treatment of poultry (in-feed use)

In four feeding trials, conducted in Australia, hens were fed at rates of 1.5 mg ai/kg for 35 days, 3 mg ai/kg for four days, 5 mg ai/kg for seven days and 9 mg ai/kg for four days, samples were taken of muscle, liver and/or eggs. Muscle and liver samples from hens, treated at the GAP rate of 5 mg ai/kg feed, were taken immediately after treatment and up to two days post-treatment. Cyromazine residues in muscle were found to decrease from 0.03 to < 0.02 mg/kg in muscle and from 0.12 to < 0.05 mg/kg in liver.

In two trials conducted in France in 1985/1986, hens were fed cyromazine at 5 mg ai/kg feed for up to 38 days. Residues in eggs (egg white/yolk) sampled from 0 to 22 days, following 15 to 38 days of feeding were: 0.15/0.16, 0.15/0.11, 0.12/0.05, 0.08/0.11, 0.12/0.05, 0.04/0.08, 0.08/0.06, 0.08/0.04, < 0.02/0.04, < 0.02/0.03 (3) and < 0.02/0.02 (9) mg/kg. As the egg white comprises

approximately 66% and the yolk 33% of a typical poultry egg, the levels found in egg white/yolk can be expressed on a whole egg basis, in ranked order, as: < 0.02 (12), 0.04, 0.05, 0.07 (2), 0.09, 0.10 (2), 0.14 and 0.15 mg/kg.

In a trial conducted in Israel in 1987, hens were fed cyromazine at 5 and 50 mg/kg feed for 31 days with the animals sacrificed four hours after removal of the feed. Residues of cyromazine in muscle and liver ranged from 0.04 to 0.10 mg/kg at the 5 mg/kg treatment level and from 0.36 to 0.76 mg/kg at the higher feeding level. Eggs were not analysed.

In a study conducted in the Philippines in 1982 hens were fed for 60 days, at a rate of 1.6 mg ai/kg of feed; no residues were detected in muscle (< 0.025 mg/kg) and liver (< 0.04 mg/kg) in animals slaughtered two to nine days after cessation of feeding. Residues in eggs collected from animals fed cyromazine from 29 to 60 days, zero to nine days after feeding ceased were 0.02, < 0.02 and 0.04 mg/kg.

In ten studies conducted in the USA from 1979 to 1986, hens were fed cyromazine for up to 56 days at levels of 2.5, 5.0, 25 and 50 mg ai/kg of feed. In one trial where hens were fed cyromazine at 5 mg/kg from 14 to 27 days, residues in egg white/yolk at day 0 were: 0.09/0.08, 0.11/0.06 and 0.11/0.07 mg/kg, or 0.09 (2) and 0.10 mg/kg in the whole egg. In three trials conducted at 5.0 mg ai/kg feed, egg samples were collected zero to seven days after feeding from animals fed 14 to 56 days. Residues in eggs in ranked order, were: < 0.05 (4), 0.11 (2), 0.07, 0.09 (3), 0.10 (7), 0.12 (4) and 0.16 mg/kg. In one feeding study conducted at a 2.5 mg/kg feeding level for 14 days, residues at 0 days were < 0.05 mg/kg in muscle, skin and fat and 0.08 mg/kg in liver. The levels found after the animals had been fed for 27 days were 0.05 mg/kg in meat and < 0.05 mg/kg in the other tissues. In all cases, residues in fat were < 0.05 mg/kg.

In one study conducted in Japan in 2000, hens were fed cyromazine for 28 days at the level of 5 mg ai/kg. Residues in egg white/yolk collected from 0 to 2 days after treatment were < 0.02/0.03, < 0.02/0.05 and 0.04/0.07 mg/kg, or < 0.02, 0.04 and 0.05 mg/kg expressed on a whole egg basis. Residues at 0 day were 0.05 mg/kg in muscle, 0.07 mg/kg in liver and 0.09 mg/kg in kidney. Cyromazine levels in all tissues after one to three days were < 0.02 mg/kg.

In summary, residues of cyromazine in eggs from trials conducted according to GAP were: < 0.02 (13), 0.02, 0.04 (3), < 0.05 (4), 0.05 (2), 0.07 (3), 0.09 (4), 0.10 (9), 0.11 (2), 0.12 (4), 0.14, 0.15 and 0.16 mg/kg. Residues at 0 day at 2.5 mg/kg level were < 0.05 and 0.05 mg/kg in muscle, < 0.05 and 0.08 mg/kg in liver and < 0.05 mg/kg in fat.

Lactating dairy cows

In two feeding studies conducted in the USA in 1983 and 1992, Holstein dairy cows were dosed at 5.0, 10, 25, 50 or 100 mg ai/kg diet for up to 28 days. Animals were sacrificed on test days 14, 21 and 28 with tissue samples taken. Milk samples consisted of pooled aliquots from the evening and the following morning's milk. Residues in milk plateaued rapidly on the commencement of dosing with the mean levels, after 28 days treatment, increasing proportionally with the dose, ranging from 0.02 mg/kg at the 5 mg/kg to 0.42 mg/kg at the 100 mg/kg level. In one study, the highest residues in tissues, at the lower dose, were found in the animals sacrificed following 14 days of feeding with 0.13 mg/kg in kidney and 0.12 mg/kg in meat; these levels increased to 1.9 and 0.59 mg/kg, respectively in the animals dosed at 50 mg/kg. In the second study, no residues (< 0.05 mg/kg) were found in tissues from animals dosed at 10 mg/kg level. No residues were found in fat samples from any animal at any dosing level in either study.

Livestock Dietary burden

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

Animal (feed items)	Livestock dietary burden, cyromazine, ppm of dry matter diet					
	US-Canada		EU		Australia	
	max	mean	max	mean	max	mean
Beef cattle	0.31	0.17	8.54	0.57	1.02	0.57
Dairy cattle	0.31	0.17	8.54	0.57	0.31	0.17
Poultry - broiler and layer	0.41	0.06	2.4	0.14	1.4	0.21

Residues in animal commodities

The high and the mean estimated dietary burden for cattle were 8.5 and 0.57 ppm. The estimations were done by interpolation of residues at the 5 ppm feeding level.

Dietary burden (mg/kg)[a] Feeding level [ppm][b]		Cyromazine residues, mg/kg[3c]				
		Milk Mean	Muscle High	Liver High	Kidney High	Fat High
MRL cattle beef and dairy	(8.5) [5] high		*(0.204)* 0.12	*(0.153)* 0.09	*(0.221)* 0.13	*(< 0.05)* 0
STMR cattle beef and dairy	(0.57) [5] av		*(0.01)* 0.06	*(0.01)* 0.06	*(0.01)* 0.08	*(< 0.05) 0*
STMR cattle beef and dairy	0.57) [5] av	*(0.005)* 0.045				

a - Values in parentheses are the estimated dietary burdens

b - Values in square brackets are the actual feeding levels in the transfer study

c - Residue values in parentheses in italics are interpolated from the dietary burden, feeding levels in the transfer study and the residues found in the transfer study. High is the highest individual animal tissue residue in the relevant feeding group. Mean is mean animal tissue (or milk) residue in the relevant feeding group.

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.01 and an HR of 0.20 mg/kg for cyromazine in meat (from mammals other than marine mammals).

The Meeting estimated a maximum residue level of 0.3 mg/kg, an STMR of 0.01 and an HR of 0.187 mg/kg for cyromazine in edible offal (mammalian).

The Meeting estimated a maximum residue level of 0.01 mg/kg and an STMR of 0.005 mg/kg for cyromazine in milks. The Meeting also estimated a median and a highest residue level of 0 mg/kg in mammalian fat.

The high and the mean estimated dietary burden for poultry were 2.4 and 0.14 ppm. The direct treatment for poultry (in-feed use) conducted at 2.5 mg/kg feeding level can be used for estimating residues in poultry tissues.

The Meeting estimated a maximum residue level of 0.1 mg/kg, an STMR of 0.05 mg/kg and an HR of 0.05 mg/kg for cyromazine in poultry meat.

The Meeting estimated a maximum residue level of 0.2 mg/kg, an STMR of 0.065 mg/kg and an HR of 0.08 mg/kg for cyromazine in poultry edible offal. The Meeting also estimated a median and a highest residue level of 0 mg/kg in poultry fat.

The Meeting agreed that for the residue estimation in eggs, the residues coming from the direct use of cyromazine in the feed at 5 mg/kg level, according to GAP (0 day withholding period) represents a better estimation of the likely residues.

The Meeting estimated a maximum residue level of 0.2 mg/kg and an STMR of 0.07 mg/kg and an HR of 0.16 mg/kg for cyromazine in eggs.

The Meeting recommends the withdrawal of the current recommendation of 0.2* mg/kg for cyromazine in eggs, of 0.01* mg/kg in milks, and of 0.05* mg/kg in poultry meat and sheep meat.

RECOMMENDATIONS

On the basis of the data from supervised trials, the Meeting concluded that the residue concentrations listed below are suitable for establishing MRLs and for assessing IEDIs and IESTIs.

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

CCN	Commodity name	Recommended MRL (mg/kg)		STMR (P) mg/kg	HR (P) mg/kg
		New	Previous		
VS 0620	Artichoke, globe	3		1.0	1.3
VD 0071	Beans (dry)	3		1.0	
VB 0400	Broccoli	1		0.15	0.51
VB 0041	Cabbages, head	10		0.26	6.1
VS 0624	Celery	4	5	0.58	2.3
VC 0424	Cucumber	2	0.2	0.48	1.3
MO 105	Edible offal (Mammalian)	0.3		0.01	0.19
PE 0112	Eggs	0.3	0.2 * [a]	0.07	0.16
VO 0050	Fruiting vegetables, other than cucurbits, except mushrooms and sweet corn-on-the-cob	1		0.16	0.58
VL 0482	Lettuce, head	4	5	0.34	2
VL 0483	Lettuce, leaf	4		0.34	2
VP 0534	Lima beans young pods and/or immature beans	1		0.23	0.58
FI 0345	Mango	0.5		0.125	0.25
MM 0095	Meat (from mammals other than marine mammals)	0.3		0.01	0.20
VC 0046	Melons, except watermelon	0.5	0.2	0.04	0.19
ML 0106	Milks	0.01	0.01* [a]	0.005	
VO 0450	Mushroom	7	5	2.2	4.2
VL 0485	Mustard greens	10		2.7	7.4
VA 0385	Onion, bulb	0.1		0.05	0.07
VO 0051	Peppers	W	1		
PO0111	Poultry, edible offal	0.2		0.065	0.08
PM 0110	Poultry meat	0.1	0.05* [a]	0.05	0.05
MM 0822	Sheep meat	W	0.05* [a]		
VL 0502	Spinach	10		2.0	6.1
VA 0389	Spring onion	3		0.345	1.7
VC 0431	Summer squash	2		0.16	1
VO 0448	Tomato	W	0.5		
JF 0048	Tomato juice			0.12	
	Tomato, washed			0.11	
	Tomato, canned			0.09	
	Ketchup			0.13	
	Tomato, puree			0.19	
	Tomato, paste			0.34	

* at or about the LOQ.

a - MRL accommodates external animal treatment

DIETARY RISK ASSESSMENT

Long-term intake

The ADI for cyromazine is 0-0.06 mg/kg bw. The International Estimated Daily Intakes (IEDI) for cyromazine was estimated for the 13 GEMS/Food cluster diets using the STMR or STMR-P values estimated by the current Meeting for 35 commodities. The results are shown in Annex 3 of the 2007 Report of the JMPR. The IEDI ranged from 0 – 2% of the maximum ADI. The Meeting concluded that the long-term intake of residues of cyromazine from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The ARfD for cyromazine is 0.1 mg/kg bw. The International Estimated Short Term Intake (IESTI) for cyromazine was calculated for the plant commodities for which STMRs and HRs were estimated and for which consumption data were available. The results are shown in Annex 4 of the 2007 Report of the JMPR. For the general population, the IESTI was higher than the ARfD for cabbage (120%) and spinach (130% ARfD). For children, the IESTI was higher than the ARfD for cabbage (280%) and spinach (390%). For all the other commodities, the intakes ranged from 0 – 40%.

The Meeting concluded that the short-term intake of residues of cyromazine from uses other than cabbage and spinach that had been considered by the JMPR is unlikely to present a public concern. The information provided to the JMPR precludes an estimate that the short-term intake of residues of cyromazine from the consumption of cabbage and spinach will be below the ARfD.

The ARfD established by the Meeting in 2006 was based on body-weight loss and decrease in food consumption in dams in developmental toxicity studies. The reason for these effects was unknown and there is a rapid recovery on cessation of administration. The Meeting noted that this ARfD may be conservative. Furthermore, it is possible that the short-term risk assessment conducted by the Meeting may also be conservative.

The Meeting noted that no residue data was available from an alternative GAP to estimate a lower maximum residue level for cyromazine in cabbage and spinach.

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1059/00	Lütolf, W	2002a	Residue Study with Cyromazine (CGA 72662) in or on Cucumbers in France (North). Syngenta Crop Protection AG, Basel, Switzerland, Report No 1059/00. GLP, not published
1060/00	Lütolf, W	2002b	Residue Study with Cyromazine (CGA 72662) in or on Cucumbers in France (North). Syngenta Crop Protection AG, Basel, Switzerland, Report No 1060/00; GLP, not published
1057/01	Lütolf, W	2002c	Residue Study with Cyromazine (CGA 72662) in or on Cucumbers in Greece. Syngenta Crop Protection AG, Basel, Switzerland, Report No 1057/01. GLP, not published
1066/00	Lütolf, W	2002d	Residue Study with Cyromazine (CGA 72662) in or on Cucumbers in Italy. Syngenta Crop Protection AG, Basel, Switzerland, Report No 1066/00; GLP, not published
1062/00	Lütolf, W	2002e	Residue Study with Cyromazine (CGA 72662) in or on Cucumbers in Spain. Syngenta Crop Protection AG, Basel, Switzerland, Report No 1062/00; GLP, not published
1037/00	Lütolf, W	2002f	Residue Study with Cyromazine (CGA 72662) in or on Summer Squash in France (North). Syngenta Crop Protection AG, Basel, Switzerland, Report No 1037/00; GLP, not published
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1025/00	Lutolf, W	2002k	Residue Study with Cyromazine (CGA 72662) in or on Tomatoes in Spain. Syngenta Crop Protection AG, Basel, Switzerland, Report No 1025/00, GLP, not published
1061/00	Lütolf, W	2002l	Residue Study with Cyromazine (CGA 72662) in or on Tomatoes in Spain. Syngenta Crop Protection AG, Basel, Switzerland, Report No 1061/00. GLP, not published

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1038/00	Lütolf, W	2002n	Residue Study with Cyromazine (CGA 72662) in or on Eggplant in France (South). Syngenta Crop Protection AG, Basel, Switzerland, Report No 1038/00, GLP, not published
1039/00	Lütolf, W	2002o	Residue Study with Cyromazine (CGA 72662) in or on Eggplant in France (North). Syngenta Crop Protection AG, Basel, Switzerland, Report No 1039/00. ADME - Bioanalyses, Vergèze, France, Report No 0132101. GLP, not published
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1100/00	Lütolf, W	2002q	Residue Study with Cyromazine (CGA 72662) in or on Cos Lettuce in Italy. Syngenta Crop Protection AG, Basel, Switzerland, Report No 1100/00, GLP, not published
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0030902	Pointurier, R	2001j	Residue Study with Cyromazine (CGA 72662) in or on Cos Lettuce in France (North). Syngenta Crop Protection AG, Basel, Switzerland. ADME - Bioanalyses, Vergèze, France, Report No 0030902, GLP, not published
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0131601	Pointurier, R	2002c	Residue Study with Cyromazine (CGA 72662) in or on Melons in France (North). Syngenta Crop Protection AG, Basel, Switzerland. ADME - Bioanalyses, Vergèze, France, Report No 0131601. GLP, not published
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