CHLORMEQUAT (015)

EXPLANATION

Chlormequat was evaluated in the CCPR Periodic Review Programme in 1994. The Meeting estimated maximum residue levels for a number of commodities, but they were recorded as only Guideline Levels because the ADI was withdrawn. As an ADI was allocated by the 1997 JMPR, the 1994 estimates were then recommended for use as MRLs. The 1994 JMPR had requested further information on feeding studies with cows and poultry, analytical methods for animal products, processing studies on cotton seed and residue studies on mushrooms grown on straw with a residue level of 15 to 20 mg/kg.

At the 30th Session of the CCPR, it was noted that animal transfer studies on poultry and cattle would be available in 1998.

The compound was reviewed again for toxicology in 1999, when an acute reference dose was allocated and the Meeting recommended that an acute risk assessment should be carried out.

A dairy cattle and a poultry feeding study, analytical methods for the determination of chlormequat residues in water, cereals, pears and animal products as well as the results of trials on pears and cereals were reported to the present Meeting by the manufacturers (CCC Task Force). The government of the Netherlands reported the official method of analysis for chlormequat in pears. Information on national MRLs and GAP was provided by the governments of Germany, Poland and The Netherlands.

METHODS OF RESIDUE ANALYSIS

Analytical methods

The methods used in the past were based on semi-quantitative thin-layer chromatographic or photometric determination (JMPR, 1994). These involved a lengthy clean-up process, with poor reproducibility and high values in samples from untreated control plots. Recent methods are based on head-space gas chromatography after pyrolysis in an alkaline medium, ion-pair HPLC with conductivity detection, or LC-MS.

Cereals. Caddy and Carroll (1982) adapted the colorimetric method of Mooney and Pasarela (1967) for the determination of chlormequat in barley grain and straw. After methanolic extraction, clean-up was by chromatography on a column of alumina with an acetone/methanol mixture. The residue was determined colorimetrically as a dipicrylamine-chlorocholine chloride complex. The LOD was 0.1 mg/kg.

Chlormequat residues in grain and straw samples were determined by Byast and Tolhurst (1990, 1992), with extraction and clean-up procedures based on those of Mooney and Pasarela (1967). After extraction with methanol and clean-up on an alumina column, the sample was derivatized with sodium thiophenolate to form a volatile ether for gas chromatography with a flame photometric detector in the sulfur mode. The LOD was 0.1 mg/kg but the lowest fortification level was 1 mg/kg for wheat and barley (recoveries: grain 95%, straw 103%) and 2 mg/kg for rye and triticale (grain 73-80%, straw 73-84%).

An analytical method for the determination of chlormequat in cereal forage, straw, grain, bran, flour and oat flakes was validated by Schneider (1992, 1993). The compound was extracted with methanol, and an aliquot of the methanol extract concentrated, transferred with water to a C-18 cartridge, and the eluate cleaned up further by ion exchange. Identification was by GLC with an FID after pyrolytic decomposition of the chlormequat to acetylene by heating an alkaline medium in a closed tube for 15 min at 200°C. The recoveries are shown in Table 1. The LOD for all samples was 1 mg/kg. High values were found in the untreated control samples.

Table 1. Recoveries of chlormequat chloride from forage, grain, straw and processed cereal products (Schneider 1993).

Sample	Fortification level, mg/kg	Recovery, mg/kg	Mean, mg/kg	SD, mg/kg	CV, %
Forage (oats)	0	1.9, 1.8, 2, 1.9	1.9	0.056	2.9
	1	2.9, 2.8, 2.5, 2.8	2.7 (83%[1])	0.18	6.7
	10	8.5, 9, 11, 12	10 (83%[1])	1.6	16
	50	48, 46, 44, 42	45 (87%[1])	2.6	5.8
Straw (wheat)	0	0.38, 0.48, 0.53, 0.52	0.48	0.068	14
	1	1.4, 1.3, 1.3, 1.3	1.3 (82%[1])	0.042	3.2
	10	6.8, 8, 9.7, 8.8	8.3 (78%[1])	1.2	14
	50	41, 41, 44, 40	42 (82%[1])	1.3	3.1
Grains (oats)	0	0.25, 0.26, 0.29	0.27	0.021	7.8
	0.5	0.75, 0.74, 0.66	0.72 (90%[1])	0.049	6.8
	1	1.4, 1.3, 1.3, 1.2	1.29 (103%[1])	0.053	4.1
	3	2.5, 2.4, 2.9, 2.8	2.62 (79%[1])	0.23	8.8
Bran (rye)	0	1.1, 0.98, 0.87, 0.94	0.96	0.075	7.8
	1	2.1, 1.8, 2	1.95 (99%[1])	0.12	6.1
	3	3.1, 3.8, 3.5, 3.2	3.43 (82%[1])	0.31	9.0
Flour (rye)	0	0.54, 0.47, 0.64, 0.62	0.57	0.078	14
	1	1.36, 1.33, 1.42, 1.42	1.38 (81%[1])	0.042	3.0
	3	2.6, 2.9, 3.3, 3.2	2.98 (80%[1])	0.31	10
Flakes (oats)	0	0.36, 0.33, 0.34, 0.34	0.34	0.013	3.8
	1	1.1, 1.1, 0.96, 1.1	1.07 (72%[1])	0.076	7.1
	3	2.6, 2.8 2.3, 2.4	2.51 (72%[1])	0.21	8.4

SD: standard deviation
CV: coefficient of variation
[1]after substraction of mean blank value

Fegert (1996) validated the BASF method 314/1 for wheat and barley (forage, grain and straw). The compound was extracted with water/acetone (1:2). After liquid-liquid partition with dichlormethane/water the active ingredient was isolated as an ion-pair with sodium tetraphenyl borate as complexing reagent, extracted with hydrochloric acid and further purified by alumina column chromatography. Determination was based on ion-pair HPLC with column switching. The compound was paired with hexanesulfonic acid and chromatography was on a neutral, hydrophobic column with an aqueous/acetonitrile mobile phase and suppressed conductivity detection. (Dionex ion chromatograph; pre-column PRP-1, 150 x 4.1 mm, 10 µm; analytical column PRP-1, 250 x 4.1 mm, 10 µm). The LOD for chlormequat chloride residues was 0.5 mg/kg in straw and 0.05 mg/kg in all other samples. Control samples fortified with chlormequat chloride at 0.05, 0.5 and 5 mg/kg showed mean recoveries ranging from 71.4% ± 2.7% to 94.6% ± 3.4% (Table 2).

Table 2. Recoveries of chlormequat chloride from fortified forage, grain and straw of cereals (Fegert, 1996).

Sample	Fortification level, mg/kg	Recovery, %	Mean, %	SD, %	CV, %
Wheat forage	0.05	80, 82, 78, 75, 77	78	2.6	3.3
	5	77, 75, 75, 76, 74	76	0.9	1.2
Wheat grain	0.05	81, 79, 95, 78, 81	83	7.0	8.5
	5	74, 73, 73, 70, 67	71	2.7	3.8
Wheat straw	0.5	81, 83, 82, 84, 83	83	1.4	1.7
	5	79, 79, 79, 81, 81	80	1.3	1.6

Sample	Fortification level, mg/kg	Recovery, %	Mean, %	SD, %	CV, %
Barley forage	0.05	74, 75, 76, 76, 75	75	0.7	1.0
	5	87, 87, 88, 85, 82	86	2.2	2.6
Barley grain	0.05	85, 83, 96, 87, 85	87	4.8	5.5
	5	93, 89, 86, 89, 93	90	2.7	3.0
Barley straw	0.5	97, 97, 93, 98, 89	95	3.4	3.6
	5	93, 90, 93, 92, 93	92	1.4	1.6

BASF Method 314/1 was also validated for wheat grain by Schulz (1996a) and Kuhlmann (1977) with samples fortified at 0.05 mg/kg and 5 mg/kg. Schulz obtained a mean recovery and coefficient of variation of 81% and 6.5% respectively from 5 determinations at each level, with only small peaks in the control samples in the relevant retention time range, corresponding to <0.05 mg/kg as chlormequat chloride. Kuhlmann (1997) reported a mean recovery and coefficient of variation of 89.8% and 7.14% respectively from two analyses at each level.

Schneider (1997a) validated the GC-MS "Dr G. Krebs Analytik Method DrK120" in which chlormequat chloride is determined in wheat grain from a decomposition fragment formed in the injector of the gas chromatograph by comparison with an internal standard. Validation was at fortification levels of 0.05 and 5 mg/kg. The mean recovery was 92% ± 7.6%, with values around 0.05 mg/kg in the untreated control samples.

A study by Sasturain (1997) was designed to demonstrate that contract and official laboratories in Germany are capable of analysing residues of chlormequat in wheat grain. An inter-laboratory evaluation (ring test) using spiked samples (level 1 = 0.08 mg/kg, level 2 = 2.8 mg/kg) was carried out at four laboratories using method DrK120 and BASF method 314/1. The mean recovery and the coefficient of variation were 106.3% and 24.5% for level 1, and 98.2% and 2% for level 2, showing a correlation between the levels of chlormequat chloride and the precision of the results. There were no false positive results. The results indicate that the laboratories' findings do not depend on the analytical method but on the expertise and experience of each laboratory with the individual methods.

<u>Pears</u>. The official analytical method of The Netherlands for the determination of chlormequat in plant material (Anon., 1996) has been applied to pears. A methanol extract is cleaned by ion-exchange and alumina chromatography, then evaporated to dryness and heated in an alkaline medium in a closed tube at 215°C to convert chlormequat to acetylene, which is determined in the head-space by gas chromatography with flame-ionisation detection. The LOD of chlormequat in pears was 0.01 mg/kg.

A new analytical method relying on quantification by tandem liquid chromatography with mass spectrometric detection (LC-MS-MS) was validated for pears (Quirijns and van Dam, 1999; Quirijns, 1999). Chlormequat chloride was extracted from the plant material with water after homogenization. After filtration, 20 µl of the extract was injected on to a 100 x 3 mm I.D. 5 µm Spherisorb CN column with a 10 x 3 mm I.D. R2 guard column. Isocratic elution was with a mobile phase of methanol/water/1 M aqueous ammonium acetate (50:49:1) at a flow rate of 0.3 ml/min. MS/MS data were acquired by selecting the ion at m/z 122 as the precursor ion and the ion at m/z 58 as the product ion. The multiplier was run at 1200 V.

The recovery and repeatability are shown in Table 3. The limit of detection was 0.007 mg/kg. The LOD claimed by the authors was 0.1 mg/kg, but as the lowest fortification level was 0.29 mg/kg the validated LOD is 0.3 mg/kg.

Table 3. Recovery and repeatability of chlormequat determination in pears (Quirijns and van Dam, 1999).

Chlormequat chloride added, mg/kg (N = 6)	Mean recovery, %	SD, %
0.29	106	2.7
2.9	105	1.6
9.8	89	2.6

Animal products. Weidenauer (1999a,b) validated an ion-pair HPLC method to determine chlormequat in fortified poultry and dairy cow products. Samples of hen eggs, meat, liver and fat, and cow milk, meat, liver, kidney and fat were homogenized, extracted with a mixture of acetone and water (2:1), and passed through a cation exchange column. Chlormequat was eluted with diluted HCl and the eluant evaporated. The dry residue was re-dissolved in water and washed with dichloromethane, and the aqueous phase was evaporated to dryness. The residue was then transferred to an alumina column, eluted with a mixture of acetonitrile and methanol, and the eluate evaporated to dryness. The residue was transferred via methanol to water for analysis by ion-pair chromatography with column switching. The mobile phase was hexanesulfonic acid (2mmol/l)/CH_3CN (97:3) in a gradient system. A Hamilton PRP-1, 15 cm x 4.1 mm column was used as pre-column and the analyte was transferred to the separation column by means of a motor driven switch valve during a period of about 1-1.5 min. A Hamilton PRP-1, 25 x 4.1 mm column was used for analyte determination. A Dionex CDM-2 conductivity detector with background conductivity suppression was used for detection. Under these conditions the retention time for chlormequat was about 22 min.

In the hen trial, no residues of chlormequat were found at or above the LOD of 0.05 mg/kg in any control samples of meat, liver, fat or eggs. At spike levels of 0.05-0.5 mg/kg the average recovery from tissues was 80% with a relative standard deviation of 9.5%, and from eggs 83.5% with a relative standard deviation of 16% (Table 4).

Table 4. Recoveries of chlormequat chloride from fortified hen tissues and eggs (Weidenauer, 1999a).

Sample	Fortification level, mg/kg	Measured value, mg/kg	Recovery, %
Hen meat	0	n.d. [1]	
	0.05	0.042	84
	0.5	0.38	75.5
Hen liver	0	0.035	
	0.05	0.08	92[2]
	0.5	0.38	69[2]
Hen fat	0	n.d.	
	0.05	0.04	80
	0.5	0.4	80
Eggs	0	n.d.	
	0.05	0.057	114
		0.039	78
		0.058	116
		0.044	88
		0.041	82
		0.051	102
		0.046	92
		0.045	90
		0.035	70
		0.041	82
		0.037	74
		0.044	88
		0.053	106
		0.036	72
		0.043	86
		0.036	72
	0.5	0.44	88
		0.43	86

Sample	Fortification level, mg/kg	Measured value, mg/kg	Recovery, %
		0.49	98
		0.35	70
		0.39	78
		0.40	80
		0.50	100
		0.40	80
		0.38	76
		0.35	70
		0.34	68
		0.35	70
		0.32	64
		0.41	82
		0.41	82
		0.43	86
		0.37	74
		0.38	76
		n (eggs)	34
		Average recovery % (eggs)	83.5
		SD	13
		rel. SD, %	16

[1] not detected
[2] corrected for control

No residues of chlormequat were found at or above the LOD of 0.05 mg/kg in any control sample of cow meat, liver, kidney or fat. 0.037 mg/kg was detected in one kidney control sample. The average recovery from the tissues was 86% at spike levels of 0.05 and 0.5 mg/kg with a relative standard deviation of 18.9% (Table 5).

Table 5. Recoveries of chlormequat chloride from fortified cow tissues (Weidenauer, 1999b).

Sample	Fortification level, mg/kg	Measured value, mg/kg	Recovery, %
Meat	0	n.d. [1]	
	0.05	0.042	84
	0.5	0.4	80
Liver	0	n.d.	
	0.05	0.052	104
	0.5	0.41	82
Kidney	0	n.d., 0.037	
	0.05	0.061, 0.085	122, 170 (96)[2]
	0.1	0.078	78
Fat	0	n.d.	
	0.05	0.031, 0.039	62, 78
	0.5	0.4, 0.355	80, 71

[1]not detected
[2]corrected for control

Table 6 shows the recoveries of chlormequat chloride from spiked milk, skimmed milk and cream. Only two milk and one cream control samples showed interference peaks at the retention time of chlormequat. At spike levels of 0.01-0.2 mg/kg, the average recovery of chlormequat chloride was 84.7% from milk and 88.8% from skimmed milk and cream with relative standard deviations of 14.6% and 21% respectively.

Table 6. Recoveries of chlormequat chloride from fortified cow milk, skimmed milk and cream (Weidenauer, 1999b).

Sample	Fortification level, mg/kg	Measured value, mg/kg	Recovery, %
Milk	0.01	0.013 (corresponding control sample: 0.0036)	95[1]
		0.0089	89
		0.0069	69
		0.008	80
		0.0092	92
		0.0077	77
		0.0074	74
		0.012	120
		0.0069	69
		0.0079	79
		0.0099	99
		0.0082	82
	0.1	0.078	78
		0.079 (corresponding control sample: 0.0036)	76[1]
		0.071	71
		0.1	100
		0.097	97
		0.097	97
		0.08	80
		0.099	99
		0.078	78
		0.091 (corresponding control sample: 0.0032)	88[1]
		0.089	89
		0.094	94
		0.065	65
		0.06	65
	0.2	0.17	85
Skimmed milk	0.01	0.0098	98
		0.0087	87
		0.0076	76
		0.011	110
	0.1	0.071	71
		0.12	117
		0.063	63
		0.068	68
	0.2	0.23	115
Cream	0.01	0.01	100
		0.009	90
		0.0071	71
		0.01	100
	0.1	0.089	89
		0.088 (corresponding control sample: 0.019)	69[1]

[1]corrected for control

Water. Mackenroth and Sasturain (1995) validated BASF method 370 for the determination of chlormequat in tap water, leachate (lysimeter) water, and water from a small stream and the German Rhine river (surface waters). A 1000 ml sample of water was extracted with dichloromethane to remove non-polar components. The ion-pairing reagent, sodium tetraphenyl borate, was then added and chlormequat was partitioned into dichloromethane, then re-extracted from the dichloromethane phase with 2 M hydrochloric acid. The HCl phase was taken to dryness, and the residue re-dissolved in acetonitrile/methanol (95:5 v/v) and cleaned up on an acid alumina column. The eluate was concentrated to dryness, dissolved in ultra-pure water and quantified by ion chromatography (Dionex ion chromatograph with suppressed conductivity detection; pre-column PRP-1, 150 x 4.1 mm, 10 µm; analytical column PRP-1, 250 x 4.1 mm, 10 µm). The mobile phase for the pre-column and analytical column consisted of a 2 mM hexanesulfonic acid solution and acetonitrile (95:5 v/v). The LOD was

0.05 µg/l, with mean recoveries at that level of 106% from tap water, 82% from lysimeter water, 77% from Rhine river water and 76% from stream water (Table 7).

Table 7. Recoveries of chlormequat chloride from water (Mackenroth and Sasturain, 1995).

Water	Fortification level, µg/l	Recovery, %	Mean, %	SD, %	CV, %
Limburgerhof tap water	0.05	106, 110, 103, 103, 107	106	2.7	2.6
	0.1	107, 94, 90, 80, 82	91	11	12
	5	90, 94, 97, 98, 97	95	3.1	3.3
Lysimeter water	0.05	89, 78, 78, 78, 89	82	5.8	7.0
	0.1	71, 68, 58, 73, 74	69	6.6	9.5
	5	95, 93, 95, 91, 91	93	2.1	2.3
Rhine river water	0.05	76, 84, 65, 81, 82	77	7.6	9.8
	0.1	83, 79, 67, 88, 76	79	7.9	10
	5	98, 96, 97, 97, 98	97	0.7	0.7
Stream water	0.05	87, 89, 87, 90, 26	76	28	37
	0.1	80, 75, 87, 85, 74	80	5.6	6.9
	5	108, 105, 105, 106, 115	108	4.0	3.7

SD: standard deviation
CV: coefficient of variation

BASF Method 370 was also validated by Schulz (1996b, 1997) for tap water and by Kuhlmann (1997) for drinking water. Five samples of tap water and 2 samples of drinking water were each fortified at 0.05 µg/l and 5 µg/l. The mean recovery and the coefficient of variation were 90.5% and 10.9% respectively for tap water and 102.7% and 7.4% respectively for drinking water. No peak was observed in the control tap water.

Schneider (1997b) validated Dr G. Krebs Analytik Methods DrK086 and DrK199 for drinking water. In DrK086 disodium ethylenediaminetetraacetate (ETDA) was added to complex the calcium ions in the water. The sample was made alkaline with sodium hydroxide beads and chlormequat was extracted with a solution of dipicrylamine in dichloromethane, then back-extracted with 1 N hydrochloric acid and taken to dryness. The residue was transferred with methanol to a thick-walled screw-top centrifuge tube, taken to dryness and 5% potassium hydroxide added. The centrifuge tube was closed and heated for 15 min at 200°C to pyrolyse the chlormequat in the alkaline solution. After cooling, 250 µl of the gas phase was injected into the GC. The acetylene produced was determined by GLC with an FID. The LOD of the method was 0.05 µg/l (Table 8).

Table 8. Recoveries of chlormequat chloride from water by method DrK086 (Schneider, 1997b).

Addition, µg/l	Measured value, µg/l	Recovery, %
0	0.0074	
0.05	0.054	107
0.05	0.053	106
5	4.9	99

In DrK119 determination was by GC-MS, the chlormequat being decomposed in the injector of the gas chromatograph as in DrK120 for wheat grain. An internal standard is used. Fortification levels were 0.05, 0.1 and 0.3 µg/l. Because the control samples contained apparent residues of about 0.025 µg/l, the results at 0.05 µg/l could not be relied upon and the LOD was 0.1 µg/l (Table 9).

Table 9. Recoveries of chlormequat chloride from water by method DrK119 (Schneider, 1997b).
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Addition, µg/l	Measured value, µg/l	Uncorrected recovery, %	Corrected anal. value, µg/l	Corrected recovery, %	Mean µg/l	Mean corr. recovery, %	SD, µg/l	CV, %
0	0.028				0.025		0.0023	9.3
0	0.026							

Addition, µg/l	Measured value, µg/l	Uncorrected recovery, %	Corrected anal. value, µg/l	Corrected recovery, %	Mean µg/l	Mean corr. recovery, %	SD, µg/l	CV, %
0	0.024							
0	0.022							
0.1	0.098	98	0.073	72	0.072	71	0.0022	3.1
0.1	0.096	96	0.071	70				
0.1	0.1	100	0.075	74				
0.1	0.096	96	0.07	70				
0.3	0.27	89	0.25	81	0.23	77	0.012	5.2
0.3	0.26	87	0.24	79				
0.3	0.25	81	0.22	73				
0.3	0.25	81	0.22	73				

The inter-laboratory ring test on wheat grain described above (Sasturain, 1997) was also carried out with drinking water spiked at 0.11 and 0.25 µg/l by five laboratories using methods DrK086, DrK119 and BASF 370. The mean recoveries and the coefficients of variation were 85.5% and 5.8% at 0.11 µg/l, and 96% and 9% at 0.25 µg/l, showing satisfactory accuracy and reproducibility. There were no false positive results.

Stability of pesticide residues in stored analytical samples

The stability of chlormequat residues in animal products stored in freezers (milk, eggs and edible tissues) was investigated by Weidenauer (2000). Control and treated samples from the animal feeding studies A-49-97-05 (Weidenauer, 1999a) and A-49-97-06 (Weidenauer, 1999b) were used for the trial. The eggs were broken and homogenized without shells with a spatula, and the fat and liver were homogenized using a Tecator homogenizer. Aliquots of about 500 g were placed in plastic boxes and stored at ≤-18°C. Whole milk was not homogenized. The HPLC method of Weidenauer (1999a,b) described above was used. Table 10 shows the individual results and the corresponding recoveries.

Table 10. Effect of freezer storage on incurred chlormequat chloride residues in milk, eggs and edible tissues (Weidenauer, 2000).

Com-modity	Sample no.	Sampling date	Initial analysis		Analysis after freezer storage			% remaining
			Date	Residue, mg/kg	Date	Interval, months	Residue, mg/kg	
Milk	LA 43	2-Oct-97	7-Jan-98	<0.01	14-Aug-00	31	<0.01	
Milk	LA 829	5-Oct-97	20-Nov-97	0.35	14-Aug –00	33	0.48	137
Milk	LA 902	5-Oct-97	20-Nov-97	0.33	14-Aug –00	33	0.25	76
Eggs	LA 1232	17-Mar-98	17-Sep-98	<0.05	14-Aug –00	23	<0.05	
Eggs	LA 1170	16-Mar-98	21-Apr-98	<0.05	21-Aug-00	28	<0.05	
Eggs	LA 1475	16-Mar-98	21-Apr-98	0.19	14-Aug –00	28	0.085	45
Eggs	LA 1445	17-Mar-98	21-Apr-98	0.12	21-Aug –00	28	0.12	100
Liver	LA1104	14-Oct-97	9-Jul-98	<0.05	14-Aug –00	25	<0.05	
Liver	LA1136	14-Oct-97	9-Jul-98	0.4	14-Aug –00	25	0.33	82.5
Liver	LA1144	14-Oct-97	9-Jul-98	0.5	14-Aug –00	25	0.3	60
Fat	LA1106	14-Oct-97	4-Feb-98	<0.05	14-Aug –00	30	<0.05	
Fat	LA1102	13-Oct-97	4-Feb-98	<0.05	21-Aug –00	31	<0.05	
Fat	LA 1138	14-Oct-97	4-Feb-98	0.12	14-Aug –00	30	0.085	71
Fat	LA 1146	14-Oct-97	4-Feb-98	0.09	21-Aug –00	31	0.081	90

USE PATTERN

The main use of the plant growth regulator chlormequat is to consolidate the stems of cereals to prevent lodging. In pears, common uses are to inhibit vegetative growth and promote flowering in the following season. The Meeting was provided with information on currently registered uses by the governments of The Netherlands, Germany and Poland as well as by the Task Force members BASF, Ciba Speciality, Nufarm and UCB (Table 11).

Table 11. Registered uses of chlormequat in Europe at June 2000. All foliar spraying and field uses, ai expressed as chlormequat chloride.

Crop	Country	Product, % ai	Application					PHI, days
			Rate, kg ai/ha	Spray conc., kg ai/hl	Water, l/ha	Growth stage	No.	
Almonds	Spain	SL 40	0.72	0.08	900	1 week before flowering	1	
Barley	Italy	SL 30.5	0.76					
		SL 46	1.8					
	Spain	SL 23.6	0.59					
Barley, summer	Belgium	SL 23	0.46		200		1	F[2]
	Germany	SL 30.5	0.6		200-400	BBCH[1] 32-49	1	42
	Ireland	SL 75	1.5		220		1	F
	Netherlands	SL 30.5	0.61				1	F
		SL 36	0.32-0.63		200-600	Beginning of shooting until stage 6-8 of Feekes scale (BBCH 30-37)	1	
		SL 36	0.62-0.90				1	
	UK	SC 23	0.35					
		SL 64.5	1.6				1-2	F
Barley, winter	Belgium	SL 23	0.69		200		1	F
	Germany	SL 30.5	0.76		200-400	BBCH 32-49	1	42
	Ireland	SL 75	0.56-1.5		220		1	F
	Netherlands	SL 30.5	0.92				1	F
	UK	SC 23	0.46					
		SC 34.5	0.69					
		SL 64.5	1.6				1-2	F
		SL 72	1.6				1	F
		SL 75	1.7				1	F
Cereals	Germany	SL 30.5	0.61-0.76		200		1	42
	Spain	SL 40	1.4		200-300	From BBCH 30 and at formation of each node	1	30
Cereals, summer (barley, rye, triticale, wheat)	Poland	SL 46	0.46-0.92		150-300	Beginning of shooting (BBCH 30)	1	42
Cereals, winter (barley, rye, triticale, wheat)	Poland	SL 46	0.69-1.2		150-300	Beginning of shooting (BBCH 30)	1	42
	Luxembourg	SL 23	0.92					
Cotton	Brazil	SL 10	0.1			70 days after emergence	1	
Grapes	Italy	SL 46	1.6					
	Spain	SL 40	1.3	0.14	900	2-3 weeks before flowering	1	
Linseed and fibrous flax	Netherlands	SL 30.5	1.4				1	F
		SL 36	0.90-1.4		200-600	At plant height of 30-45 cm	1	
	UK	SL 64.5	1.6				1	F
Maize	Belgium	SL 23	0.46		200		1	F
Oats	Austria	SL 40	1.1-1.6		200-600	BBCH 31-39	1	42
	Belgium	SL 72	1.4		200-600	At plant height of 40 cm	1	F
		SL 75	1.4					F
		SL 75	1.4		200-600	At plant height of 40 cm	1	F
	Denmark	SL 46	1.4		200-400	BBCH 30-37	1	90
		SL 46	1.8					
		SL 75	1.1		200-400	BBCH 30-37	1	90
		SL 75	1.1					
	Finland	SL 46	1.5					F
		SL 75	1.5					F
	Germany	SL 72	1.4		200-600	BBCH 32-49	1	42
		SL 72	1.4		200-400	BBCH 32-49	1	42
	Italy	SL 46	1.4					
	Luxembourg	SL 72	1.4		200			F
		SL 75	1.4				4-5	
	Netherlands	SL 45.7	1.4		200-600	Beginning of shooting until stage 6-7 of Feekes scale (BBCH 30-37)	1	F
		SL 40	1.2				1-2	F
		SL 75	1.4				1	F
	UK	SL 64.5	1.6				1	F
		SL 72	1.6				1	F

Crop	Country	Product, % ai	Application					PHI, days
			Rate, kg ai/ha	Spray conc., kg ai/hl	Water, l/ha	Growth stage	No.	
		SL 75	1.7				1	F
Peach	Spain	SL 40	0.9	0.1	900 900 900 900	Flowering May June Post-harvest	2-3 1 1 1	
Pear	Belgium	SL 72	1.4	0.24	600		4-5	F
		SL 75	1.4	0.24	600		4-5	F
	Denmark	SL 46	1.8	0.18	1000	2 and 4 weeks after flowering	2	42
		SL 75	0.75-1.5	0.075-0.15	1000	3 and 6 weeks after flowering	2	42
	Netherlands[3]	SL 40	1.3	0.16	800		1-2	90
		SL 46	1.5	0.19	800		1-2	90
		SL 45.7	0.93-1.8	0.094-0.15	1000-1200	1st treatment at end blossoming, 2nd 2-3 weeks later	1-2	90
		SL 75	1.1-2.3	0.11-0.19	1000-1200		1-2	90
		SL 75	0.75-2.3	0.094-0.15	800-1500		2	90
	Spain	SL 40	0.9	0.1	900 900 900 900	Flowering May June Post-harvest	2-3 1 1 1	
Rape	UK	SL 64.5	1.9				1	F
Rape, winter	Belgium	SL 23	0.69		200		1	F
Rye	Austria	SL 40	1.5-2.4		200-600	BBCH 31-32	1	63
	Belgium	SL 23	0.81		200		1	F
	Denmark	SL 46	1.8					
		SL 46	1.2		200-400	BBCH 30-31	1	90
		SL 75	0.94		200-400	BBCH 30-31	1	90
		SL 75	1.1					
	Finland	SL 46	1.4					F
		SL 75	2				1	F
	Italy	SL 46	1.4					
	Spain	SL 23.6	0.59					
	UK	SL 64.5	1.6				1	F
		SL 72	1.6				1	F
		SL 75	1.7				1	F
Rye, winter	Germany	SL 30.5	0.61		200-400	BBCH 32-49	1	42
		SL 72	1.4		200-600	BBCH 30-37	1	63
	Ireland	SL 75	1.5-2.3		220		1	F
	Netherlands	SL 36	0.32-0.63		200-600	Beginning of shooting until stage 6-7 of Feekes scale (BBCH 30-32)	1	
		SL 30.5	0.61				1	F
	Sweden	SL 14	0.42					F
Spelt wheat	Belgium	SL 72	0.72		200-600	BBCH 30-32	1	F
		SL 72	0.9		200		1	F
		SL 75	0.9					F
		SL 75	0.9		200-600	BBCH 30-32	1	F
	Luxembourg	SL 72	0.9		200			F
		SL 75	0.9				4-5	
Tomato	Italy	SL 46	0.69					
Triticale	Belgium	SL 23	0.69		200		1	F
		SL 72	0.9		200		1	F
		SL 72	0.72		200-600	BBCH 30-32	1	F
		SL 75	0.9					F
		SL 75	0.75		200-600	BBCH 30-32	1	
	Ireland	SL 75	1.9		220		1	F
	Luxembourg	SL 72	0.9		200			F
		SL 75	0.9				4-5	
	UK	SL 64.5	1.6				1	F
		SL 72	1.6				1	F
		SL 75	1.7				1	F
Wheat, winter	Austria	SL 40	0.19-1.6		200-600	BBCH 21-30	1	63
	Belgium	SL 23	0.69		200		1	F
		SL 72	0.72		200-600	BBCH 30-32	1	F
		SL 72	0.9		200		1	F
		SL 75	0.9					F
		SL 75	0.75		200-600	BBCH 30-32	1	F
	Denmark	SL 46	1.8					
		SL 46	0.92		200-400	BBCH 30-31	1	90

Crop	Country	Product, % ai	Application					PHI, days
			Rate, kg ai/ha	Spray conc., kg ai/hl	Water, l/ha	Growth stage	No.	
		SL 75	0.75		200-400	BBCH 30-32	1	90
		SL 75	1.1					
	Finland	SL 46	1.8				1	F
		SL 75	2				1	
	France	SL 34.5	0.69					
		SL 40	0.9		200-300		1	F
		SL 46	0.92		200-300	BBCH 21-30	1	F
		SL 75	0.9					
	Germany	SL 30.5	0.76		200-400	BBCH 32-49	1	42
		SL 72	1.5		200-600	BBCH 21-31	1	63
	Ireland	SL 75	1.5		220		1	F
		SL 75	1.1		220		2	F
	Luxembourg	SL 72	0.9		200			F
		SL 75	0.9				4-5	
	Netherlands	SL 36	0.63		200-600	Beginning of shooting until stage 6-7 of Feekes scale (BBCH 30-32)	1	
		SL 30.5	0.61				1	F
		SL 40	0.8				1	F
		SL 45.7	0.38-0.92		200-600	Stage 5 of Feekes scale (BBCH 30)	1	F
		SL 75	0.38-0.92		200-600		1	F
		SL 75	0.38-0.75		200-600		1	F
	Poland	SL 46	0.69-1.2		200-300	Beginning of shooting (BBCH 30)	1	42
		SL 46	0.92-1.6		200-300		1	42
		SL 67.5	1.2-2		200-300		1	
		SL 72	0.86-1.6		200-300		1	
		SL 75	0.90-1.7		200-300		1	
	UK	SC 23	0.46					
		SC 34.5	0.69					
		SL 64.5	1.6				1	F
		SL 72	1.6				1	F
		SL 75	1.7				1	F
Wheat, summer	Austria	SL 40	0.19-1.6		200-600	BBCH 21-30	1	63
	Belgium	SL 23	0.69		200		1	F
		SL 72	0.72		200		1	F
		SL 72	0.47-0.72		200-600	BBCH 29-30	1	F
		SL 75	0.75					F
		SL 75	0.45-0.75		200-600	BBCH 29-30	1	F
	Denmark	SL 46	1.8					
		SL 46	0.69		200-400	BBCH 30-31	1	90
		SL 75	0.56		200-400	BBCH 30-31	1	90
		SL 75	1.1					
	Finland	SL 46	1.5				1	F
		SL 75	1.5				1	F
	France	SL 40	0.9		200-300	BBCH 21-30	1	F
		SL 46	0.92		200-300	BBCH 21-30	1	F
		SL 75	0.9					
	Germany	SL 30.5	0.61		200-400	BBCH 32-49	1	42
		SL 72	0.93		200-600	BBCH 21-29	1	63
	Ireland	SL 75	0.75		220		1	F
	Luxembourg	SL 72	0.72		200			F
		SL 75	0.75				4-5	
	Poland	SL 46	0.69		200-300	Phase of 5 leaves (BBCH 15)	1	42
		SL 46	0.69-0.92		200-300	Beginning of shooting (BBCH 30)	1	
		SL 67.5	1.2-1.6		200-300		1	
		SL 72	0.65-0.86		200-300		1	
		SL 75	0.68-0.9		200-300		1	
	Netherlands	SL 30.5	0.61				1	F
		SL 36	0.63		200-600	Beginning of shooting until stage 6-7 of Feekes scale (BBCH 30-32)	1	
		SL 40	0.4				1	F
		SL 45.7	0.38-0.46		200-600	Stage 5 of Feekes scale (BBCH 30)	1	F
		SL 75	0.38-0.46		200-600		1	F
		SL 75	0.38		200-600		1	F
	UK	SL 64.5	0.81-1.6				1	F
		SL 72	0.79				1	F

Crop	Country	Product, % ai	Application					PHI, days
			Rate, kg ai/ha	Spray conc., kg ai/hl	Water, l/ha	Growth stage	No.	
		SL 75	0.83				1	F
Wheat, hard	France	SL 75	1.6					
	Italy	SL 46	1.6					
Wheat, soft	Ireland	SL 46	1.9					
	Italy	SL 30.5	0.76					
		SL 46	1.4					
	Spain	SL 23.6	0.59					
		SL 46	1.8					28

[1]BBCH scale (Bleiholder *et al*., 1997)
[2]F: PHI fixed by approved use (growth stage at treatment)
[3]Information by the government of The Netherlands (Olthof, 2000): GAP for pears will be changed in the near future

RESIDUES RESULTING FROM SUPERVISED TRIALS

The Meeting received new information on supervised residue trials on pears and cereals as well as on animal feeding studies, and the trials reported in 1994 on which the current MRL recommendations are based were re-evaluated for the estimation of STMRs and HRs. Residue data on pears, cereals (grains, forage and fodder) and rape are summarized in Tables 12-26.

Table 12. Residue trials on pears reported to the 1994 and 2000 JMPRs.
Table 13. Residue trials on cereals in Austria 1965-1970, to 2000 JMPR.
Table 14. Residue trials on summer barley in the UK 1983/84, reported to the 2000 JMPR.
Table 15. Residue trials on summer barley, reported to the 1994 JMPR.
Table 16. Residue trials on winter barley, reported to the 1994 JMPR.
Table 17. Residue trials on oats, reported to the 2000 JMPR.
Table 18. Residue trials on oats, reported to the 1994 JMPR.
Table 19. Residue trials on triticale in the UK 1989, reported to the 2000 JMPR.
Table 20. Residue trials on rye, reported to the 2000 JMPR.
Table 21. Residue trials on rye, reported to the 1994 JMPR.
Table 22. Residue trials on wheat, reported to the 2000 JMPR.
Table 23. Residue trials on summer wheat, reported to the 1994 JMPR.
Table 24. Residue trials on winter wheat, reported to the 1994 JMPR.
Table 25. Residue trials on maize, reported to the 1994 JMPR.
Table 26. Residue trials on rape seed, reported to the 1994 JMPR.

Residue levels and application rates were reported as chlormequat chloride, but the residues are generally recalculated as cation in the Appraisal. When residues were not detected they are shown as below the LOD (e.g. <0.1 mg/kg). Residues, application rates and spray concentrations have generally been rounded to two significant figures but for residues approximating the LOD to one significant figure. HRs and STMRs from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels. These results are double underlined.

Pears (Table 12). The trials from the Netherlands on which the recommended MRL was based were not correctly evaluated by the 1994 JMPR: each trial included 4 field replicates, so only one figure from each trial should have been selected for the estimation of a maximum residue level. Further supervised trials on pears were carried out in 1998/99 in France.

Table 12. Residues of chlormequat chloride in pears.

Reference, report no., year, country, location	Crop variety	Application rate per treatment			Dates of treatment	Growth stage	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Water l/ha	kg ai/hl					
Wit (1969) CvF/PD 4-6-01, (Tox 16),1968, The Netherlands	Beurre Hardy	1.5			6/10/1968		91	1.6 1.9 2.0 1.5	JMPR 1994, 4 field replicates, colorimetric method (Mooney and Pasarela, 1967)
Wit (1969) CvF/PD 4-0-01 (Tox 16), 1968, The Netherlands	Beurre Hardy	0.74 0.74			6/10/1968 7/01/1968		70	1.5 0.9 2.8 0.5	JMPR 1994, 4 field replicates, colorimetric method (Mooney and Pasarela, 1967)
Wit (1969) CvF/PD 4-6-01 (Tox 16), 1968, The Netherlands	Beurre Hardy	1.5			5/16/1968		116	0.3 0.4 0.4 0.3	JMPR 1994, 4 field replicates, colorimetric method (Mooney and Pasarela, 1967)
Wit (1969) CvF/PD 4-6-01 (Tox 16), 1968, The Netherlands	Beurre Hardy	0.74 0.74			5/16/1968 6/10/1968		91	0.4 0.8 0.3 0.6	JMPR 1994, 4 field replicates, colorimetric method (Mooney and Pasarela, 1967)
Wit (1969) CvF/PD 4-6-01 (Tox 16), 1968, The Netherlands	Doyenne du Comice	1.2			5/08/1968		142	<0.1 <0.1 <0.1 0.2	JMPR 1994, 4 field replicates, colorimetric method (Mooney and Pasarela, 1967)
Wit (1969) CvF/PD 4-6-01 (Tox 16), 1968, The Netherlands	Doyenne du Comice	1.2			5/08/1968		142	0.3 <0.1 <0.1 0.6	JMPR 1994, 4 field replicates, colorimetric method (Mooney and Pasarela, 1967)
Greve and Hagedoorn (1983) RIVM63760 1109A 1980, The Netherlands, Marknesse	Doyenne du Comice	1.6 1.2 1.2 1.2	1500 1500 1500 1500	0.11 0.08 0.08 0.08	last treatment 7/02/1980		90	0.94 <u>1.6</u> 1.3 1.5	JMPR 1994, 4 field replicates, head-space GLC (Greve and Hagedoorn, 1983)
Greve and Hagedoorn (1983) RIVM63760 1109A 1980 The Netherlands, Huissen	Doyenne du Comice	1.6 1.2 1.2 1.2	150 150 150 150	1.1 0.8 0.8 0.8	last treatment 6/24.1980		101	<u>8.1</u> 4.2 7.4 5.1	JMPR 1994, 4 field replicates, head-space GLC (Greve and Hagedoorn, 1983)
Greve and Hagedoorn (1983) RIVM2104000311B 1983 The Netherlands, Kapelle	Doyenne du Comice	1.8 1.8	1000 1000	0.18 0.18	last treatment 5/24/1983		124	3.5 5.3 3.1 2.4	JMPR 1994, 4 field replicates, head-space GLC (Greve and Hagedoorn, 1983)

Reference, report no., year, country, location	Crop variety	Application rate per treatment			Dates of treatment	Growth stage	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Water l/ha	kg ai/hl					
Greve and Hagedoorn (1983) RIVM2104000311B 1983 The Netherlands, Geldermalsen	Doyenne du Comice	1.8 1.1	1000 1000	0.18 0.11	last treatment 02.06.83		113	6.5 5.5 5.4 6.9	JMPR 1994, 4 field replicates, head-space GLC (Greve and Hagedoorn, 1983)
Perny (1999) R 8090 AN1, 1998 Northern France 67330-Riedheim	Williams	1.5 1.5 1.4 1.5 1.4	614 613 605 620 606	0.24 0.24 0.24 0.24 0.24	6/04/1998 6/18/1998 7/02/1998 7/18/1998 7/31/1998	71-72 73 75 81	 1 13 25 44	 17 14 9 5.6	LC-MS (Quirijns and van Dam, 1999; Quirijns, 1999)
Perny (1999) R 8090 BM1, 1998 Northern France 72800-Thoree-les-Pins	Conference	1.5 1.4 1.4 1.5 1.4	610 595 601 612 603	0.24 0.24 0.24 0.24 0.24	6/12/1998 6/22/1998 7/02/1998 7/13/1998 7/29/1998	73 73-75 75 77 77	45	4.6	LC-MS (Quirijns and van Dam, 1999; Quirijns, 1999)
Perny (1999) R 8090 BM2, 1998 Northern France 72800-Thoree-les-Pins	Beurre-Hardy	1.4 1.4 1.4 1.4 1.5	606 596 604 605 611	0.24 0.24 0.24 0.24 0.24	6/12/1998 6/22/1998 7/02/1998 7/13/1998 7/29/1998	73 73-75 75 77 77	45	4.0	LC-MS (Quirijns and van Dam, 1999; Quirijns, 1999)
Perny (1999) R 8090 BG1, 1998 Belgium 4280-Hannut-Bertree	Conference	1.4 1.4 1.4 1.4 1.5	590 589 578 604 622	0.24 0.24 0.24 0.24 0.24	9/17/1998 6/29/1998 7/13/1998 7/27/1998 8/08/1998	74 74 77 79 79	44	7.5	LC-MS (Quirijns and van Dam, 1999; Quirijns, 1999)
Perny (2000) R 9067 AN1, 1999 Northern France 67310-Traenheim	Conference	1.65	658	0.25	6/10/1999	72	63	3.1	LC-MS (Quirijns and van Dam, 1999; Quirijns, 1999)
Perny (2000) R 9067 AN2, 1999 Northern France 67330-Riedheim	Williams	1.5	583	0.25	6/18/1999	73-74	55	<0.5	LC-MS (Quirijns and van Dam, 1999; Quirijns, 1999)
Perny (2000) R 9067 BM1, 1999 Northern France 72800-Thoree les Pins	Doyenne du Comice	1.3	513	0.25	6/24/1999	75	63	0.57	LC-MS (Quirijns and van Dam, 1999; Quirijns, 1999)
Perny (2000) R 9067 BM2, 1999 Northern France 72800-Thoree les Pins	Conference	1.4	563	0.25	6/24/1999	75	63	<0.5	LC-MS (Quirijns and van Dam, 1999; Quirijns, 1999)

Cereals (Table 13). In 1965 and 1967-1970 numerous residue trials were carried out in Austria to determine chlormequat in cereals (Bayzer, 1966, 1968, 1984). Analysis was by semi-quantitative thin-layer chromatography (Dragendorff reagent) after extraction with ethanol, separation from other quartenary ammonium compounds by ion-exchange chromatography and preparative TLC. The LOD was reported as 0.1 mg/kg, but no validation was carried out. Control samples were not included. The trials were reported to the present Meeting.

Table 13. Residues of chlormequat chloride in cereals in Austria 1965-1970.

Reference	Year, appl. rate, kg ai/ha	Commodity	No. of samples, residues in mg/kg										
			Total	<0.1	0.1	0.25	0.5	1	2	3-4	5-6	7-8	>10
Bayzer, 1966	1965, 1-5	Wheat grain	127	2	4	16	30	38	36		1		
		Wheat flour	38	22	8	8							
		Wheat straw	69	13		4	10	12	16	10	1		3
Bayzer, 1966	1965, 6	Barley grain	10	2		2	3	3					
Bayzer, 1966	1965, 4-6	Rye grain	24	3		3	6	5	7				
Bayzer, 1968	1967, 0.75-4	Wheat grain	287	79	23	69	65	34	15	2			
		Wheat flour	10	4	3	2	1						
Bayzer, 1968	1967, 4-5	Barley grain	17			3	4	7	3				
		Barley straw	17	13					1	1		2	
Bayzer, 1968	1967, 2-5	Oat grain	16			2	3	5	6				
Bayzer, 1968	1967, 2-5	Rye grain	80	8		5	7	14	19	18	7	2	
Bayzer, 1984	1968, 1-3	Wheat grain	131	47	13	30	26	15					
Bayzer, 1984	1968, 2-4	Barley grain	32				11	14	7				
Bayzer, 1984	1968, 2-4	Oat grain	9			1	1			7			
Bayzer, 1984	1968, 2-5	Rye grain	96	14	2	11	13	46	7	3			
Bayzer, 1984	1969/70, appl. rate: no information	Oat grain	92			5	13	31	28	15			

Barley. Six supervised trials carried out in 1983/84 in the UK were reported to the Meeting. No information on PHIs or analytical methods was reported. The grain and straw samples were harvested at ripening. A further trial was reported from Latvia but no information was included on application rates or analytical methods (Table 14).

The trials reported to the 1994 JMPR which complied with current GAP, and from which the present Meeting estimated a maximum residue level, are shown for summer barley in Table 15 and for winter barley in Table 16.

Table 14. Residues of chlormequat chloride in summer barley reported to the 2000 JMPR.

Reference, report no., year, country, location	Crop variety	Application		Sample	PHI, days	Residues, mg/kg	Analytical method
		kg ai/ha	Growth stage, BBCH				
Summer barley							
Ipatova *et al*. (1998), V/15, 1998 Latvia-Riga		No information ("0.6 l/ha Stabilan")		grain straw	71 71	<0.05 <0.05	No information
Denes (1991) Hungary		No information ("3 l /ha Stabilan")		grain straw	61 61	<0.2 <0.3	Semi-quantitative TLC. No reference, no validation
Lyttle and Baughan (1984), 1167106 UK-Tickencote	Triumph	0.52 1.6	13 31-32	grain *control* straw *control*	no information	3.9 *0.72* 4.3 *4.2*	No information
		0.52 1.6 +surfactant	13 31-32	grain *control*	no information	2.6 *0.72*	No information
Lyttle and Baughan (1984), 1167106 UK-Cranwell	Triumph	0.52 1.6	13 31-32	grain *control*	no information	2.0 *1.4*	No information
		0.52 1.6 +surfactant	13 31-32	grain *control*	no information	2.3 *1.4*	No information

Reference, report no., year, country, location	Crop variety	Application		Sample	PHI, days	Residues, mg/kg	Analytical method
		kg ai/ha	Growth stage, BBCH				
Winter barley							
Lyttle and Baughan (1983), 1167106 UK-Casterton	Maris Otter	0.52 0.52 1.6	13 29 31-32	grain *control*	no information	1.5 *0.68*	No information
		0.52 0.52 1.6 +surfactant	13 29 31-32	grain *control*	no information	1.4 *0.63*	No information
Lyttle and Baughan (1983), 1167106 1983 UK-Harringworth	Igri	0.52 0.52 1.6	13 29 31-32	grain *control* straw *control*	no information	3.8 *1.6* 2.5 *1.7*	No information
		0.52 0.52 1.6 +surfactant	13 29 31-32	grain *control*	no information	2.2 *1.6*	No information
Lyttle and Baughan (1983), 1167106 1983 UK-Epingham	Sonja	0.52 0.52 1.6	13 29 31-32	grain *control*	no information	1.4 *2.0*	No information
		0.52 0.52 1.6 +surfactant	13 29 31-32	grain *control*	no information	2.2 *2.0*	No information
Lyttle and Baughan (1983), 1167106 1983 UK-Yaxley	Igri	0.52 0.52 1.6	13 29 31-32	grain *control* straw *control*	no information	1.9 *1.3* 4.3 *2.2*	No information
		0.52 0.52 1.6 +surfactant	13 29 31-32	grain *control*	no information	1.8 *1.3*	No information

Table 15. Residues of chlormequat chloride in summer barley reported to the 1994 JMPR.

Report no., Year, Country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
83/10206 1983 Denmark	0.46	forage grain straw	30 59 59	2.5 0.05 2.7	78/10213 1978 Sweden	0.23 0.46 0.92	grain	72 72 72	<0.05 0.1 0.1
83/10207 1983 Denmark	0.46	forage grain straw	29 70 70	0.85 0.3 1.3	78/10214 1978 Sweden	0.23 0.46 0.92	grain	86 86 86	<0.05 <0.05 <0.05
82/10190 1982 Denmark	0.61	grain straw	61 61	<0.05 4.3	78/10215 1978 Sweden	0.23 0.46 0.92	grain	112 112 112	<0.05 <0.05 0.08
82/10191 1982 Denmark	0.61	grain straw	77 77	<0.05 4.4					
82/10207 1982 Germany	0.61	forage grain straw	0 21 35 41 48 69 69	10 2.1 0.96 0.55 0.36 0.17 4	78/10216 1978 Sweden	0.23 0.46 0.92	grain	75 75 75	0.23 0.5 0.73

Report no., Year, Country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
82/10208	0.61	forage	0	7.6	80/10237	1.6	grain	97	0.37
1982			20	1.5	1980		straw	97	4.9
Germany		grain	34	0.46	UK				
			41	0.5					
			48	0.62					
		straw	34	4.4					
			41	3.9					
			48	4					
78/10210	0.23	grain	82	0.06	80/10238	1.6	straw	104	1.6
1978	0.46		82	0.1	1980				
Sweden	0.92		82	0.19	UK				
78/10211	0.23	grain	111	<0.05	82/10186	0.81	grain	135	0.18
1978	0.46		111	<0.05	1982				
Sweden	0.92		111	<0.05	UK				
78/10212	0.23	grain	107	<0.05	82/10187	1.6	grain	110	0.24
1978	0.46		107	<0.05	1982		straw	110	1.6
Sweden	0.92		107	<0.05	UK				

Table 16. Residues of chlormequat chloride in winter barley reported to the 1994 JMPR.

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
82/10213	0.76	ear	61	0.1	82/10198	0.76	grain	75	0.24
1982		stalk	61	0.1	1982		straw	75	4.7
Denmark		grain	69	0.05	France				
		straw	69	0.9		0.76	grain	63	0.35
							straw	63	5.4
82/10195	0.76	grain	69	0.18	83/10210	0.76	grain	56	0.3
1982		straw	69	1.8	1983		straw	56	4.4
France					France				
	0.76	grain	56	0.16					
		straw	56	11					
82/10196	0.76	grain	70	<0.05	83/10211	0.76	grain	68	0.29
1982		straw	70	3.1	1983		straw	68	5.5
France					France				
	0.76	grain	57	<0.05					
		straw	57	8.5					
82/10197	0.76	grain	77	<0.05	83/10212	0.76	grain	67	0.3
1982		straw	77	0.36	1983		straw	67	2.8
France					France				
	0.76	grain	62	0.21					
		straw	62	2.4					
82/10205	0.76	forage	0	8.3	82/10206	0.76	forage	0	9.9
1982			21	4.3	1982			21	3
Germany		grain	35	1.1	Germany		grain	35	1.6
			42	1.5				42	1.5
			49	1.6				49	1.6
		straw	35	7.8			straw	35	4.1
			42	6.4				42	3.5
			49	5.8				49	5.8
83/10201	0.76	forage	0	9	83/10202	0.76	forage	0	6.4
1983		ear	21	7.3	1983			20	1.3
Germany			35	6.5	Germany			33	0.89
		stalk	21	7.7				53	1.9
			35	8.8			grain	68	0.18
			42	12				76	0.2
		grain	49	2.3			straw	86	6.2
		straw	49	12				76	3

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
83/10203 1983 Germany	0.76	forage grain straw	0 20 35 43 53 43	0.76 3.3 1 1.3 7.3 8.7	83/10204 1983 Germany	0.76	forage ear stalk grain straw	0 21 35 43 49 43 49 76 76	7.3 2 2.2 0.54 0.78 2.9 2.6 0.17 5.8
83/10205 1983 Germany	0.76	forage ear	0 21 35 42	10 7.7 4.7 2.5	84/10231 1984 Switzerland	0.61	grain straw	72 72	0.23 4.5
		stalk grain straw	21 35 42 49 49	4.9 7.8 11 2.1 9	84/10232 1984 Switzerland	0.61	grain straw	70 70	0.29 4.2
83/10195 1983 Sweden	0.61	grain	68	0.07 0.13 0.32 0.42	82/10188 1982 UK	1.6	forage grain straw	40 96 96	0.97 0.07 1.1
80/10236 1980 UK	1.6	grain straw	80 80	0.15 1	87/10378- 10380 1987 UK	1.9	forage grain straw	0 128 128	17,19,24 0.16,0.15, 0.36 1.7,2.1,2.4
82/10189 1982 UK	1.6	forage grain straw	51 115 115	0.41 <0.05 2.2	83/10186 1983 UK	0.48+1.6	grain straw	31 31	0.24 0.98
83/10185 1983 UK	0.48+1.6	grain straw	98 98	0.05 8.9	87/10366 1987 UK	0.46	forage grain straw	5 82 82	2, 3.3, 4.8 0.45, 0.5, 0.58 10,11,12
84/10226 1984 UK	0.48+1.6	grain straw	113 113	<0.05 2.4	87/10366 1987 UK	0.46	forage grain straw	0 75 75	9.2 0.43 16

Oats. The trials reported to the 2000 JMPR are shown in Table 17. High values were found in untreated control plots in four trials in Austria (1992). Semi-quantitative thin-layer chromatographic or colorimetric methods were used to analyse the samples from one trial in Germany and one in the UK which could be evaluated.

The results of trials reported to the 1994 JMPR which were used by the present Meeting for the estimation of maximum residue levels for grain, straw and forage are shown in Table 18.

Table 17. Residues of chlormequat chloride in oats reported to the 2000 JMPR.

Reference, report no., year, country, location	Crop Variety	Application		Sample	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Growth stage, BBCH				
Pfarl (1993a), R92-14 / 1162, 1992 Austria-Seitenstetten	Lord	1.4	39	whole plant *control*	0	13.9 *1.6*	product: Stabilan 460 head-space GLC (Schneider, 1993)
				plant without ear *control*	17	2.9 *2.0*	
				ear *control*	17	3.8 *4.6*	

Reference, report no., year, country, location	Crop Variety	Application		Sample	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Growth stage, BBCH				
				grain *control*	51	0.53 *0.85*	LOD 1 mg/kg
				straw *control*	51	1.3 *0.54*	
Pfarl (1993a), R92-14 / 1162, 1992 Austria – Leonding	Lorenz	1.4	39	whole plant *control*	0	11.5 *3.4*	product: Stabilan 460
				plant without ear *control*	13	3.8 *1.1*	head-space GLC (Schneider, 1993)
				ear *control*	13	3.9 *5.4*	
				grain *control*	49	1.0 *0.23*	LOD 1 mg/kg
				straw *control*	49	1.5 *0.70*	
Pfarl (1993b) R92-15 / 1163, 1992 Austria- Seitenstetten	Lord	1.4	39	whole plant *control*	0	10 *2.3*	product: Stabilan 720
				plant without ear *control*	17	3.0 *1.9*	head-space GLC
				ear *control*	17	5.6 *3.9*	(Schneider, 1993) LOD 1 mg/kg
				grain *control*	51	0.80 *0.77*	
				straw *control*	51	1.5 *0.61*	
Pfarl (1993b), R92-15 /1163, 1992 Austria – Leonding	Lorenz	1.4	39	whole plant *control*	0	13 *1.0*	product: Stabilan 720
				plant without ear *control*	13	4.1 *1.6*	head-space GLC
				ear *control*	13	4.95 *4.1*	(Schneider, 1993)
				grain *control*	49	1.4 *0.25*	LOD 1 mg/kg
				straw *control*	49	1.5 *0.46*	
Brüggemann and Ocker (1988), D 87/88-912, 1986 Germany- München (Puch)	Fabian	1.4	37	whole plant	0 30 42 50	226 28 22 18	semi-quantitative TLC (Brüggemann and Ocker, 1986)
				grain *control*	74	3.0 0.3	
				straw	74	0.7	
Bayzer (1979a) AE/Ni/Kl 1979 01 11, 1978, UK- Brant Broughton, Nottinghamshire	Maris Quest	1.6	32	grain	65	0.8 0.8 0.5	3 replicates, semi-quantitative colorimetric or TLC (no detailed information)
				straw	65	2.0 3.0 3.0	

Table 18. Residues of chlormequat chloride in oats reported to the 1994 JMPR.

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
74/10197 1974 Germany	1.2	forage grain straw	0 21 43 63 81 81	116 15 9.2 4.8 2.4 8.2	74/10198 1974 Germany	1.2	forage grain straw	0 21 49 74 74	100 17 1.8 1.5 4.0
73/10129 1973 Germany	1.4	forage grain straw	0 23 44 49 49	84 8.1 6.8 3.7 5.2	73/10130 1973 Germany	1.4	forage grain straw	24 48 59 70 70	15 4.0 3.9 3.3 1.2
75/10184 1975 Germany	1.2	forage grain straw	0 21 42 63 63	17 3.7 2.5 0.14 0.9	75/10185 1975 Germany	1.4	forage grain straw	0 21 32 51 51	17 7.6 3.3 1.6 2.2
75/10186 1975 Germany	1.4	forage grain straw	0 21 42 55 55	17 6.4 5.1 1.9 1.9	76/10144 1976 Germany	1.4	grain straw	59 59	1.8 1.2
78/10209 1978 Germany	1.4	forage grain straw	0 21 42 54 75 75	9.9 3.5 2.3 3.2 2.4 1.9	76/10155 1976 Germany	1.4	forage grain straw	0 21 42 57 63 42 57 63	11 1.5 0.05 1.0 1.1 1.6 1.6 1.6
76/10156 1976 Germany	1.4	forage grain straw	0 21 42 73 60 73	20 1.8 0.36 0.45 1.3 0.78	76/10157 1976 Germany	1.4	forage grain straw	0 22 42 44 42 44 62	14 6.9 1.2 1.5 12 9.6 5.3
76/10158 1976 Germany	1.4	forage grain straw	0 19 82 89 82 89	19 4.3 2.0 1.9 4.8 <0.1	80/10244 1980 Germany	1.4	forage grain straw	0 32 82 91 82 91	12 2.5 0.86 1.2 9.9 3.0
80/10245 1980 Germany	1.4	forage grain straw	0 21 30 91 91 91	6.3 0.69 1.1 0.09 0.79 3.0	80/10246 1980 Germany	1.4	forage grain straw	0 21 50 57 57	3.8 2.9 2.0 0.51 9.9
80/10247 1980 Germany	1.4	forage grain straw	0 20 42 62 62	6.7 3.1 2.1 0.9 6.3	80/10248 1980 Germany	1.4	forage grain straw	0 21 70 73 70 73	3.8 1.3 1.7 1.2 9.9 8.1
74/10199 1974 UK	1.7	grain straw	51 51	9.2 25	76/10159 1976 UK	1.7	grain straw	34 34	0.63 0.48

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
77/10248 1977 UK	1.7	forage grain straw	27 58 94 94	4.7 1.6 0.1 3.3					

Rye and triticale (Tables 19, 20). Four trials on triticale and two on winter rye were carried out by Byast and Tolhurst (1990). No chlormequat residues were found in any of the untreated control samples or in the treated grain. Two trials on rye were carried out in Austria in 1992 by Pfarl (1993a,b) and analysed by Schneider (1993) as described above.

The residue data reported to the 1994 JMPR which the present Meeting re-evaluated for the estimation of maximum residue levels are shown in Table 21.

Table 19. Residues of chlormequat chloride in triticale reported to the 2000 JMPR.

Reference, report no., year, country, location	Crop variety	Application		Sample	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Growth stage, BBCH				
Byast and Tolhurst (1990), 52287, 1989 UK-Brighton	Salvo	2.5	31-32	grain straw	95 95	<0.1(3) <0.1(3)	GLC after thiophenolate derivatization (Byast and Tolhurst, 1990)
Byast and Tolhurst (1990), 52287, 1989 UK-Brighton	Salvo	5.0	31-32	grain straw	95 95	<0.1(3) <0.1(3)	GLC after thiophenolate derivatization (Byast and Tolhurst, 1990)
Byast and Tolhurst (1990), 52287, 1989 UK –Stockbridge	Lasko	2.5	31-32	grain straw	76 76	<0.1(3) 0.24 0.74 0.59	GLC after thiophenolate derivatization (Byast and Tolhurst, 1990)
Byast and Tolhurst (1990), 52287, 1989 UK –Stockbridge	Lasko	5.0	31-32	grain straw	76 76	<0.1(3) 0.66 0.13 0.23	GLC after thiophenolate derivatization (Byast and Tolhurst, 1990)

Table 20. Residues of chlormequat chloride in rye reported to the 2000 JMPR.

Reference, report no., year, country, location	Crop variety	Application		Sample	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Growth stage, BBCH				
Pfarl (1993a), R92-14 / 1162, 1992 Austria-Linz	Eho-Kurz	1.15	32	whole plant *control*	0	11 *2.2*	product: Stabilan 460
				plant without ear *control*	14 29 *14* *29*	2.4 2.1 *1.4* *2.6*	head-space GLC (Schneider, 1993)
				ear *control*	14 29 *14* *29*	6.1 5.8 *6.0* *3.9*	
				grain *control*	79	0.81 *0.3*	LOD 1 mg/kg
				straw *control*	79	1.6 *0.64*	

Reference, report no., year, country, location	Crop variety	Application		Sample	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Growth stage, BBCH				
Pfarl (1993b), R92-15 /1163, 1992 Austria-Linz	Eho-Kurz	2.16	32	whole plant *control*	0	11.5 *3.4*	product: Stabilan 720
				plant without ear *control*	14 29 *14* *29*	4.3 2.1 *1.3* *1.7*	head-space GLC (Schneider, 1993)
				ear *control*	14 29 *14* *29*	14.7 6.5 *7.5* *6.2*	
				grain *control*	79	1.7 *0.43*	LOD 1 mg/kg
				straw *control*	79	2.1 *0.73*	
Byast and Tolhurst (1990) 52287, 1989 UK-Elvedon		2.3	30	grain straw	107 107	<0.1 <0.1	GLC after thiophenolate derivatization (Byast and Tolhurst, 1990)
Byast and Tolhurst (1990) 52287, 1989 UK-Elvedon		4.6	30	grain straw	107 107	<0.1 <0.1	GLC after thiophenolate derivatization (Byast and Tolhurst, 1990)
Byast and Tolhurst (1990) 52287, 1989 UK-Isle of Wight		2.3	30	grain straw	107 107	<0.1 <0.1	GLC after thiophenolate derivatization (Byast and Tolhurst, 1990)
Byast and Tolhurst (1990) 52287, 1989 UK-Isle of Wight		4.6	30	grain straw	107 107	<0.1 <0.1	GLC after thiophenolate derivatization (Byast and Tolhurst, 1990)

Table 21. Residues of chlormequat chloride in rye reported to the 1994 JMPR.

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
Summer rye									
77/10249 1977 Germany	1.1	forage grain straw	0 19 29 48 70 70	14 12 0.5 0.1 0.06 0.3	77/10250 1977 Germany	1.1	forage grain straw	0 17 38 69 46 59 69	24 13 8.8 2.1 18 <0.1 0.2
77/10251 1977 Germany	1.1	forage grain straw	0 21 42 63 63	11 12 5.6 2.6 9	77/10252 1977 Germany	1.1	forage grain straw	0 22 43 64 92 85 92	13 9.7 11 9.4 1.5 3.1 4.7

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
Winter rye									
74/10195 1974 Germany	1.4	forage grain straw	0 55 83 122 122	468 20 9.7 0.24 4.8	74/10194 1974 Germany	1.4	forage grain straw	0 27 53 81 123 123	193 18 9 4.4 0.22 2.8
74/10196 1974 Germany	1.4	forage grain straw	0 28 57 84 117 117	264 9 3 1 0.3 3.1	75/10188 1975 Germany	1.4	forage grain straw	0 29 56 84 99 99	25 2.1 1.5 1.3 0.3 4.3
75/10187 1975 Germany	1.4	forage grain straw	0 28 58 83 98 105 105	52 2.2 1.1 1.4 1.3 0.34 2.2	75/10189 1975 Germany	1.4	forage grain straw	0 28 56 85 92 92	13 4.1 1.2 3.5 0.33 5.7
75/10190 1975 Germany	1.4	forage grain straw	0 28 56 85 92 92	39 1.9 0.73 2.3 <0.05 2.7	75/10197 1975 Germany	1.4	forage grain straw	0 28 56 84 96 93 96	26 4.9 3.4 1.5 0.62 6.9 5.2
75/10198 1975 Germany	1.4	forage grain straw	0 28 56 85 84 85	19 5.9 0.13 1.2 6.6 9.6	76/10152 1976 Germany	1.4	forage grain straw	3 32 59 66 84 91 66 91	24 4.3 1.2 0.26 0.36 0.45 2.9 4.5
76/10153 1976 Germany	1.4	forage grain straw	0 28 52 58 77 77 85	3.1 28 12 0.92 1.9 16 9.6	76/10154 1976 Germany	1.4	forage grain straw	0 29 56 67 56 67	24 17 2.0 1.4 18 12
82/10203 1982 Germany	0.61	forage grain straw	0 21 35 42 49 75 75	7.3 3.6 2.9 2.8 1.8 0.43 5.5	82/10204 1982 Germany	0.61	forage grain straw	0 20 34 41 48 34 41 48	8.4 4.2 1.8 1.1 1.1 7.5 4.5 2.8
82/10193 1982 Sweden	0.46	grain	77	0.09	82/10192 1982 Sweden	0.61	grain	85	<0.05
83/10191 1983 Sweden	0.61	grain	80	0.09	83/10197 1983 Sweden	0.61	grain	77	0.07
83/10193 1983 Sweden	0.61	grain	86	0.08	83/10194 1983 Sweden	0.61	grain	97	0.05
76/10149 1976 UK	1.6	grain straw	92 92	0.88 12	76/10150 1976 UK	1.6	grain straw	113 113	0.45 0.48

Wheat. Data from trials in Austria (2), Germany (3) and the UK (8) were reported to the Meeting (Table 22). High chlormequat values in the untreated control plots were found in the two trials in Austria (1992). Semi-quantitative thin-layer chromatographic or colorimetric methods were used for analysis in the trials in Germany and the UK. A further trial was reported in Latvia but no information on application rates or analytical methods was included.

The residue data reported to the 1994 JMPR which complied with current GAP and which the present Meeting used to estimate maximum residue levels are shown in Tables 23 and 24.

Table 22. Residues of chlormequat chloride in wheat reported to the 2000 JMPR.

Reference, report no., year, country, location	Crop variety	Application		Sample	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Growth stage, BBCH				
Winter wheat							
Pfarl (1993a), R92-14 / 1162, 1992 Austria-Ansfelden	Ikarus	1.4	32	whole plant *control*	0 14 *0* *14*	23 3.4 *2.9* *1.6*	product: Stabilan 460 head-space GLC (Schneider, 1993)
				plant without ear *control*	29	3.5 *1.4*	
				ear *control*	29	3.4 *3.9*	
				grain *control*	79	0.34 *0.27*	LOD 1 mg/kg
				straw *control*	79	1.2 *0.67*	
Pfarl (1993b), R92-15 /1163, 1992 Austria-Ansfelden	Ikarus	1.4	32	whole plant *control*	0 14 *0* *14*	24 4.9 *3.5* *2.6*	product: Stabilan 720 head-space GLC (Schneider, 1993)
				plant without ear *control*	29	2.3 *1.1*	
				ear *control*	29	6.3 *5.0*	
				grain *control*	79	0.41 *0.33*	LOD 1 mg/kg
				straw *control*	79	1.2 *0.34*	
Brüggemann and Ocker (1988), UCB/D87/88-116/3, 1985 Germany-Bonn (Kessenich)	Kanzler	1.4	25-29	plant	0 29 49 63	100 1.0 0.8 0.3	semi-quantitative TLC (Brüggemann and Ocker, 1986)
				grain *control*	94	0.24 *0.07*	
				straw	94	0.9	
Brüggemann and Ocker (1988), D 87/88-03775, 1986 Germany-Hannover (Pattensen)	Kanzler	1.4	22-25	plant	0 31 52 65	134 8.6 1.7 1.4	semi-quantitative TLC (Brüggemann and Ocker, 1986)
				grain *control*	129	0.2 *0.06*	
				straw	129	0.5	

Reference, report no., year, country, location	Crop variety	Application		Sample	PHI, days	Residues, mg/kg	Remarks, method
		kg ai/ha	Growth stage, BBCH				
Brüggemann and Ocker (1988), D 87/88-03788, 1986 Germany-Kiel (Rabendorf)	Kanzler	1.4		grain *control*	110	0.2 *0.2*	semi-quantitative TLC (Brüggemann and Ocker, 1986)
Bayzer (1979a) AE/Ni/Kl 1979 01 11, 1978, UK-Farcett-Fen Huntingdonshire	Flanders	1.6	6-7 leaves (BBCH 17)	grain	112	<0.1 <0.1 <0.1	3 replicates, semi-quantitative colorimetric or TLC (no detailed information)
				straw	112	1.0 1.0 1.5	
Bayzer (1979a) AE/Ni/Kl 1979 01 11, 1978, UK-Winkburn Nottinghamshire	Maris Huntsman	1.6	7-8 leaves (BBCH 18)	grain	105	0.5 0.3 0.5	3 replicates, semi-quantitative colorimetric or TLC (no detailed information)
				straw	105	2.0 2.0 2.0	
Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Newark Nottinghamshire	M. Huntsman	1.7		grain straw	100 100	<0.1 1.0	semi-quantitative colorimetric or TLC (no detailed information)
Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Newark Nottinghamshire	M. Huntsman	3.4		grain straw	100 100	<0.1 2.0	semi-quantitative colorimetric or TLC (no detailed information)
Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Barton Nottinghamshire	Sports-man	3.6		grain straw	91 91	<0.1 2	semi-quantitative colorimetric or TLC (no detailed information)
Summer wheat							
Ipatova et al. (1998), V/15, 1998 Latvia-Riga		no information (“0.5 l/ha Stabilan”)		grain straw	99 99	<0.05 <0.05	No information
Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Isleham Cambridgeshire	M. Dove	0.84		grain straw	87 87	0.1 0.5	semi-quantitative colorimetric or TLC (no detailed information)
Bayzer (1979b) AE/Ni/Kl 1979 01 11, 1978, UK-Isleham Cambridgeshire	M. Dove	1.7		grain straw	87 87	0.5 1.0	semi-quantitative colorimetric or TLC (no detailed information)
Bayzer (1979a) AE/Ni/Kl 1979 01 11, 1978, UK-Barnby Nottinghamshire	Sappo	0.8	7-8 leaves (BBCH 18)	grain	95	<0.1 <0.1 <0.1	3 replicates, semi-quantitative colorimetric or TLC (no detailed information)
				straw	95	1.5 1.5 1.5	

Table 23. Residues of chlormequat chloride in summer wheat reported to the 1994 JMPR.

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
79/10190 1979 Germany	1.4	forage grain straw	0 21 35 48 62 70 62 70	15 10 8.9 5 1.3 1.3 29 9.4	79/10192 1979 Germany	1.4	forage grain straw	0 20 36 55 86 86	6.1 3.9 5.4 3.3 0.32 13
79/10194 1979 Germany	1.4	forage grain straw	0 21 42 63 70 77 70 77	16 0.18 1.6 1.2 0.34 0.59 10 4.4	79/10198 1979 Germany	1.4	forage grain straw	0 21 42 63 71 71	7.5 7.3 5.3 6.7 1.2 17
79/10200 1979 Germany	1.4	forage grain straw	0 21 35 48 62 70 62 70	11 6.7 4 8.2 1.1 1.5 21 13	79/10202 1979 Germany	1.4	forage grain straw	0 20 36 55 86 86	11 5.5 3.2 5.3 0.09 17
79/10204 1979 Germany	1.4	forage grain straw	0 21 42 63 70 77 70 77	9.7 1.3 1.7 1.1 0.62 0.68 13 1.6	79/10208 1979 Germany	1.4	forage grain straw	0 21 42 63 71 71	8.9 9.8 5.1 6.5 1.3 18
80/10220 1980 Germany	1.6	forage grain straw	0 22 43 64 64 71 85 64 71 85	1.2 6 7.8 0.31 0.31 0.31 0.31 15 16 18	80/10222 1980 Germany	1.6	forage grain straw	0 21 57 71 71	1.4 0.95 0.54 0.52 14
80/10224 1980 Germany	1.6	forage grain straw	0 20 42 74 92 74 92	9 1.6 0.85 0.41 0.40 5.2 7	80/10226 1980 Germany	1.6	forage grain straw	0 21 83 87 83 87	10 2.9 0.25 0.33 7 4.6
80/10228 1980 Germany	1.6	forage grain straw	0 21 83 87 83 87	8.2 4.2 0.30 0.48 15 13	80/10239 1980 Germany	1.7	forage grain straw	0 22 43 64 71 85 64 71 85	7.3 8.5 6.3 0.31 0.33 0.39 20 13 18

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
80/10240 1980 Germany	1.7	forage grain straw	0 20 42 74 92 74 92	9.7 3.6 1.1 0.56 0.59 11 7.3	80/10241 1980 Germany	1.7	forage grain straw	0 21 83 87 83 87	12 3.6 0.44 0.39 5.8 4.5
80/10243 1980 Germany	1.7	forage grain straw	0 21 83 87 83 87	7.5 6.6 0.42 0.44 6 12	82/10201 1982 Germany	0.61	forage grain straw	0 21 35 42 48 69 48 69	8.3 3.2 2.4 1.7 0.81 0.77 6.2 4.3
82/10202 1982 Germany	0.61	forage grain straw	0 20 34 42 34 42	12 8.2 1.5 1.4 13 12					

Table 24. Residues of chlormequat chloride in winter wheat reported to the 1994 JMPR.

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
82/10214 1982 Denmark	0.61	ear stalk grain straw	47 47 99 99	<0.1 0.11 0.15 1.5	83/10197 1983 France	0.61	grain straw	96 96	<0.05 2.3
83/10198 1983 France	0.61	grain straw	82 82	<0.05 4.8	83/10199 1983 France	0.61	grain straw	84 84	<0.05 2.6
80/10230 1980 Germany	1.6	grain straw	94 98 94 96	0.15 0.17 6.1 6	80/10232 1980 Germany	1.6	grain straw	94 98 94 96	0.28 0.17 3.8 5.1
80/10234 1980 Germany	1.6	grain straw	94 98 94 98	0.34 0.29 2.8 3.9	80/10249 1980 Germany	1.7	grain straw	94 98 94 98	0.22 0.23 7.4 8
80/10251 1980 Germany	1.7	grain straw	94 98 94 98	0.25 0.31 5.7 6.6	80/10253 1980 Germany	1.7	grain straw	94 98 94 98	0.33 0.37 4.4 4.8
82/10199 1982 Germany	0.76	forage[a] ear stalk grain straw	0 21 35 42 49 42 49 56 56	10 4.4 2 0.29 0.84 2.9 4 0.28 7.2	82/10200 1982 Germany	0.76	forage[1] ear stalk grain straw	0 21 35 35 42 49 42 49	8.8 3.3 2.7 8.3 0.62 0.53 15 15
76/10147 1976 UK	1.6	grain straw	93 93	0.05 5.4	77/10247 1977 UK	1.6	grain straw	51 131 131	1.4 0.3 0.5

Maize. The residue data reported to the 1994 JMPR were re-evaluated for the estimation of maximum residue levels. They are shown in Table 25.

Table 25. Residues of chlormequat chloride in maize reported to the 1994 JMPR.

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
84/10237	0.61	forage	0	4.4	84/10238	0.61	forage	0	22
1984			26	2.7	1984			17	6.2
Germany			34	4.8	Germany		cob	35	0.88
		cob	98	0.34				86	1.6
		rem[1]	98	2.7				113	1.7
			111	4.1			rem	35	8.3
		grain	111	0.14				86	6
								113	4.3
84/10239	0.61	forage	0	9.1	84/10240	0.61	forage	0	26
1984			21	2.4	1984			22	1.6
Germany			32	1.6	Germany			34	0.69
		cob	68	0.82			cob	83	<0.05
			109	1.2				106	<0.05
		rem	68	1.2			rem	83	0.79
			109	2.5				106	0.68
84/10241	0.61	forage	0	20	85/10309	0.61	forage	0	4.8
1984			20	0.92	1985			13	5.0
Germany			30	0.89	Germany			36	1.2
		cob	62	0.34			cob	71	1.2
		rem	62	<0.5			rem	71	3.7
			92	0.8				90	2.4
		grain	92	0.5			grain	90	0.68
85/10310	0.61	forage	0	6.3	85/10311	0.61	forage	0	3.1
1985			20	0.32	1985			35	3.4
Germany			33	0.39	Germany		cob	64	0.4
		cob	71	0.20				77	0.35
			93	0.23				107	0.44
		rem	71	<0.05			rem	64	2.7
			93	0.36				77	3.9
								107	5.1
85/10312	0.61	forage	0	5.3					
1985			13	4.3					
Germany			27	3.6					
		cob	61	2.9					
		rem	61	2.7					
			78	4.5					
		grain	78	2.4					

Rape seed. The residue data reported to the 1994 JMPR on which that Meeting estimated a maximum residue level are shown in Table 26. The present Meeting re-evaluated the results to estimate an STMR>

Table 26. Residues of chlormequat chloride in rape seed reported to the 1994 JMPR.

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
85/10313	0.92	forage	0	4.2	85/10314	0.92	forage	0	2.1
1985			14	1.4	1985			14	6.1
Germany		seed	75	2.3	Germany		seed	70	1.4
								87	4.3
85/10315	0.92	forage	0	6.0	85/10316	0.92	forage	0	4.1
1985			15	4.8	1985			14	1.8
Germany		seed	88	2.2	Germany		seed	77	2.6

Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg	Report no., year, country	Application, kg ai/ha	Sample	PHI, days	Residues, mg/kg
85/10317 1985 Germany	0.92	forage seed	0 14 77	8.9 6.5 <u>5.8</u>	86/10378 1986 Germany	0.92	forage seed	0 14 80	8.3 1.7 <u>2.9</u>
86/10379 1986 Germany	0.92	forage seed	0 15 86	15 1.4 <u>2.1</u>	86/10380 1986 Germany	0.92	forage seed	0 14 90	2.7 0.96 <u>1.7</u>
86/10381 1986 Germany	0.92	forage seed	0 14 77	9.9 3.0 <u>2.7</u>	83/10190 1983 UK	1.9	seed	93	<u>3.7</u>

<u>Livestock feeding trials</u>

<u>Hens</u>. Four groups each of four laying Lohmann brown hens were dosed with 0, 0.72, 2.16 or 7.2 mg chlormequat chloride bird/day for 28 days, equivalent to 0, 6, 18 or 60 ppm in the feed (Weidenauer, 1999a). The hens in each group were killed after the last dose and tissue samples collected. Two additional groups of 12 hens were dosed at the highest level for 28 days and allowed to recover after the last dose for 2 or 7 days. The tissues from the birds in each group were then analysed. Eggs from the hens in each group were analysed as indicated in Table 29.

The eggs and tissues were analysed for chlormequat as described in “Analytical methods” (Weidenauer, 1999a). The LOD was 0.05 mg/kg. Table 27 shows sampling, freezing, shipping, homogenization, extraction, and analysis dates. The information on storage stability (Weidenauer, 2000) was inadequate.

Table 27. Hen feeding study dates (Weidenauer, 1999a).

Procedure	Eggs		Tissues	
	First	Last	First	Last
Sampling	March 2, 1998	April 6, 1998	March 30, 1998	April 6, 1998
Sample freezing	March 2, 1998[1]	April 6, 1998[1]	March 30, 1998[1]	April 6, 1998[1]
Sample receipt at lab	March 10, 1998	April 6, 1998	March 30, 1998	April 6, 1998
Homogenization	March 2, 1998[2]	April 6, 1998[2]	April 1, 1998	April 7, 1998
Extraction	March 13, 1998	December 4, 1998	May 7, 1998	June 4, 1998
Analysis	March 13, 1998	December 7, 1998	May 13, 1998	June 26, 1998

[1]All egg and tissue samples were frozen on the day of sampling.
[2]All egg samples were homogenized on the day of sampling.

The residues of chlormequat chloride in the hen meat, liver, fat and eggs are shown in Tables 28 and 29.

Table 28. Residues of chlormequat chloride in hen tissues (Weidenauer, 1999a).

Group no.[1]	Residues, mg/kg			
	Feeding level	Meat	Liver	Fat
4	6 ppm	<0.05	0.09	<0.05
5		<0.05	<0.05[2]	<0.05
6		<0.05	<0.05[2]	<0.05
Mean		<0.05	0.05	<0.05
7	18 ppm	<0.05	<0.05[2]	<0.05
8		<0.05	0.1	<0.05
9		<0.05	0.09	<0.05
Mean		<0.05	0.07	<0.05
10	60 ppm	<0.05	0.12	<0.05
11		<0.05	0.1	<0.05
12		<0.05	0.33	<0.05

Group no.[1]	Residues, mg/kg			
	Feeding level	Meat	Liver	Fat
Mean		<0.05	0.18	<0.05
13	60 ppm	<0.05	0.12	<0.05
13		<0.05	<0.05[2]	<0.05
13		<0.05	<0.05[2]	<0.05
Mean		<0.05	0.06	<0.05
14	60 ppm	<0.05	<0.05[2]	<0.05
14		<0.05	0.08	<0.05
14		<0.05	<0.05[2]	<0.05
Mean		<0.05	<0.05[2]	<0.05

[1] Groups 4-12 were each of 4 hens. Groups 13 and 14 were each of 12 hens and the 3 samples were each composites of 4 hens.
[2] Half of the LOD (0.025 mg/kg) used for mean calculation

Table 29. Residues of chlormequat chloride in hen eggs (Weidenauer, 1999a).

Group no.[1]	Feeding level	Day 0/1, mg/kg	Day 1/2, mg/kg	Day 3/4, mg/kg	Day 5/6, mg/kg	Day 7/8, mg/kg	Day 10/11, mg/kg	Day 12/13, mg/kg	Day 14/15, mg/kg	Day 17/18, mg/kg	Day 20/21, mg/kg	Day 23/24, mg/kg	Day 25/26, mg/kg
4	6 ppm	<0.05	<0.05	<0.05	<0.05	0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
5		<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	0.05	<0.05	<0.05	<0.05
6		<0.05	<0.05	0.06	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
Mean		<0.05	<0.05	0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05	<0.05
7	18 ppm	<0.05	<0.05	0.06	<0.05	0.09	0.06	<0.05	<0.05[2]	<0.05	<0.05	<0.05	<0.05
8		<0.05	<0.05	<0.05	<0.05	0.12	0.1	0.07	0.09	<0.05	<0.05	<0.05	<0.05
9		<0.05	<0.05	<0.05	<0.05	0.1	0.07	<0.05	0.06	0.05	<0.05	<0.05	0.06
Mean		<0.05	<0.05	<0.05	<0.05	0.1	0.08	<0.05	0.06	<0.05	<0.05	<0.05	<0.05
10	60 ppm	<0.05	<0.05	0.1	<0.05[2]	0.13	0.08	0.08	0.12	0.07	0.07	0.09	0.06
11		<0.05	<0.05	<0.05[2]	0.08	0.08	0.07	0.08	0.19	0.14	<0.05[2]	0.08	0.15
12		<0.05	<0.05	0.07	0.16	0.08	0.11	0.1	0.16	<0.05[2]	<0.05[2]	0.05	0.07
13		<0.05	<0.05	0.06	0.18	0.17	0.09	0.07	<0.05[2]	0.08	0.05	0.06	0.08
14		<0.05	<0.05	<0.05[2]	0.11	0.08	0.13	<0.05[2]	0.07	0.06	0.06	0.06	0.07
Mean		<0.05	<0.05	0.06	0.11	0.11	0.1	0.07	0.11	0.08	0.05	0.07	0.09

NA: not applicable

[1] Groups 4-12 were each of 4 hens. Groups 13 and 14 were each of 12 hens and the 3 samples were each composites of 4 hens.

[2] half of the LOD (0.025 mg/kg) used for calculation of mean

Cows. A feeding study was carried out on lactating cows by Weidenauer (1999b). Groups of three Holstein dairy cows were dosed with chlormequat chloride for 28 consecutive days at 0, 240, 720 or 2400 mg/animal/day, or 0, 0.4, 1.3 or 4 mg/kg bw/day, equivalent to 0, 12, 36 or 120 ppm in the diet on a dry weight basis. Two extra cows were treated at the high dose level for 28 days and slaughtered 2 or 7 days after their last dose. The doses were equivalent to 0, 0.31, 1.01 and 3.1 mg/kg bw/day calculated as chlormequat cation.

Milk was collected from each cow throughout the study. After the final dose, three cows in each group were slaughtered and tissue samples collected. Milk and tissue samples were analysed for chlormequat chloride as described above ("Analytical methods", Weidenauer, 1999b). The LOD of the ion-pair chromatography method was 0.01 mg/kg for milk and 0.05 mg/kg for tissues.

Table 30 shows the sampling, freezing, shipping, sample preparation, extraction, and analysis dates. The information on storage stability (Weidenauer, 2000) was inadequate.

Table 30. Cow feeding study dates (Weidenauer, 1999b).

Procedure	Milk		Tissues	
	First	Last	First	Last
Sampling	September 8, 1997	October 20, 1997	October 13, 1997	October 20, 1997
Sample freezing	September 8, 1997[1]	October 20, 1997[1]	October 13, 1997[1]	October 20, 1997[1]
Sample receipt at lab	September 17, 1997	October 20, 1997	October 14, 1997	October 20, 1997
Sample preparation	September 17, 1997[2]	October 16, 1997[2]	October 17, 1997	October 24, 1997
Extraction	November 14, 1997	October 9, 1998	February 2, 1998	October 7, 1998
Analysis	November 18, 1997	October 15, 1998	February 4, 1998	October 15, 1998

[1] All milk and tissue samples frozen on day of sampling except samples for preparation of skimmed milk and cream, which were stored at +4°C and frozen after separation (1 or 2 days after sampling).
[2] Preparation of skimmed milk and cream; no preparation of whole milk samples required.

The residues of chlormequat chloride in cow meat, liver, kidney, fat, milk, skimmed milk and cream are shown in Tables 31, 32 and 33. The cream and fat content of the milk samples is given in Table 34.

Table 31. Residues of chlormequat chloride in cow tissues (Weidenauer, 1999b).

Cow no.	Dose	Residue, mg/kg			
		Meat	Liver	Kidney	Fat
4	12 ppm	<0.05	0.08	0.30	<0.05
5		<0.05	0.10	0.07	<0.05
6		<0.05	0.06	0.12	<0.05
Mean		<0.05	0.08	0.16	<0.05
7	36 ppm	<0.05	0.09	0.46	0.05
8		0.11	0.09	0.44	<0.05
9		<0.05	0.05	0.31	<0.05
Mean		<0.05	0.08	0.40	<0.05
10	120 ppm	<0.05	0.04	0.95	0.10
11		<0.05	0.24	0.27	0.05
12		0.07	0.50	1.06	0.10
Mean		<0.05	0.38	0.76	0.08
13	120 ppm recovery	<0.05	<0.05	0.16	<0.05
14		<0.05	<0.05	0.09	<0.05

Table 32. Residues of chlormequat chloride in skimmed milk and cream samples (Weidenauer, 1999b).

Cow no.	Dose	Residue, mg/kg					
		Skimmed milk Day 1	Skimmed milk Day 14	Skimmed milk Day 28	Cream Day 1	Cream Day 14	Cream Day 28
4	12 ppm	0.04	0.10	0.02	<0.01	0.02	0.02
5		0.02	0.04	0.03	<0.01	0.03	0.02
6		0.02	0.01	0.02	<0.01	0.03	0.03
Mean		0.03	0.05	0.02	<0.01	0.03	0.02
7	36 ppm	0.04	0.14	0.22	0.02	0.04	0.04
8		0.03	0.02	0.15	<0.01	0.04	0.07
9		0.02	0.10	0.04	0.01	0.05	0.04
Mean		0.03	0.09	0.14	0.01	0.04	0.05
10	120 ppm	0.09	0.06	0.11	0.07	0.07	0.02
11		0.09	0.38	0.16	0.07	0.11	0.09
12		0.05	0.31	0.07	0.09	0.05	0.06
13		0.03	0.02	0.13	0.09	0.10	0.04
14		0.06	0.36	0.11	0.11	0.09	0.10
Mean		0.06	0.23	0.12	0.09	0.08	0.06

Table 33. Residues of chlormequat chloride in cow milk (Weidenauer, 1999b).

Animal No.	Dose	Day 0/1, mg/kg	Day 1/2, mg/kg	Day 3/4, mg/kg	Day 5/6, mg/kg	Day 7/8, mg/kg	Day 10/11, mg/kg	Day 12/13, mg/kg	Day 14/15, mg/kg	Day 17/18, mg/kg	Day 20/21, mg/kg	Day 23/24, mg/kg	Day 25/26, mg/kg
4	12 ppm	0.02	0.02	0.02	0.01	0.01	0.01	0.05	0.04	0.02	0.03	0.05	0.02
5		<0.01	<0.01	0.05	0.05	0.02	0.05	0.02	0.08	<0.01	0.03	<0.01	0.04
6		<0.01	0.01	0.01	0.05	<0.01	0.05	<0.01	0.04	<0.01	0.03	<0.01	0.05
Mean		<0.01	0.01	0.03	0.04	0.01	0.04	0.02	0.05	0.01	0.03	0.02	0.04
7	32 ppm	<0.01	0.04	0.14	0.17	0.11	0.17	0.10	0.26	0.07	0.09	<0.01	0.09
8		<0.01	0.06	0.03	0.10	0.09	0.19	0.07	0.21	0.07	0.08	0.24	0.12
9		<0.01	0.01	0.05	0.07	0.08	0.13	0.06	0.09	0.02	0.06	0.13	0.12
Mean		<0.01	0.04	0.07	0.11	0.09	0.16	0.08	0.19	0.05	0.08	0.12	0.11
10	120 ppm	<0.01	0.07	0.47	0.06	0.28	0.23	0.29	0.13	0.09	0.26	0.16	0.30
11		<0.01	0.07	0.21	0.40	0.23	0.14	0.31	0.65	0.13	0.23	0.29	0.21
12		<0.01	0.20	0.16	0.10	0.25	0.21	0.11	0.07	0.32	0.35	0.33	0.13
13		<0.01	0.14	0.32	0.35	0.18	0.11	0.19	0.20	0.20	0.33	0.16	0.16
14		<0.01	0.07	0.56	0.33	0.2	0.29	0.35	0.07	0.21	0.05	0.19	0.21
Mean		<0.01	0.11	0.34	0.25	0.23	0.20	0.25	0.22	0.19	0.24	0.23	0.20

[1]NA: not applicable

Table 34. Cream and fat content of the milk samples (Weidenauer, 1999b).

Cow no.	Dose	Fat, %			Cream, %		
		Day 1	Day 14	Day 28	Day 1	Day 14	Day 28
4	12 ppm	4.95	4.40	4.35	3.88	4.30	5.03
5		5.45	5.05	5.00	4.58	4.48	4.57
6		4.85	4.50	4.70	4.03	5.23	3.70
7	33 ppm	3.85	3.85	3.90	3.09	2.37	2.48
8		5.30	4.75	4.95	4.74	5.63	5.84
9		3.80	4.95	5.20	2.10	4.10	5.44
10	120 ppm	4.50	4.20	3.40	5.14	4.95	3.65
11		3.75	3.95	3.55	1.92	3.81	1.34
12		3.83	4.35	4.25	3.41	3.87	3.03
13		4.30	4.05	3.80	3.11	3.34	3.18
14		5.13	4.85	4.10	4.45	4.83	5.99

FATE OF RESIDUES IN STORAGE AND PROCESSING

In storage

No information.

In processing

Rye and oat grain from supervised trials (Pfarl, 1993b) were processed to rye bran and flour and oat flakes by Schneider (1993) but no details of the processing were reported. Brüggemann and Ocker (1988) prepared oat flakes and wheat flour according to commercial practice.

Table 35. Residues of chlormequat chloride in cereal grains and their processed products.

Reference, report no., year, country, location	Application rate, kg ai/ha	PHI, days	Sample	Residues, mg/kg	Processing factor
Schneider (1993),	1 x 2.2	79	Rye grain	1.7 *(control 0.43)*	
Pfarl (1993b),			Rye bran	1.9 *(control 0.97)*	1.1
R 92-15 A /1163, 1992			Rye flour	1.3 *(control 0.57)*	0.76
Austria-Linz					
Schneider (1993),	1 x 1.4	49	Oat grain	1.4 *(control 0.25)*	
Pfarl (1993b),			Oat flakes	1.8 *(control 0.35)*	1.3
R 92-15 /1163,1992,					
Austria-Leonding					
Brüggemann and Ocker (1988), D 87/88-912,	1 x 1.4	74	Oat grain	3.0	
1986, Germany-München (Puch)			Chaff	4.2	1.4
			Unchaffed grain		
			Oat flakes	1.9	0.63
				0.8	0.27
Brüggemann and Ocker (1988), UCB/D 87/88-116/3, 1985,	1 x 1.4	94	Wheat grain	0.24	
			Wholemeal	0.24	1
			Wheat bran	0.61	2.5
Germany-Bonn (Kessenich)			Wholemeal bread	0.15	0.63

RESIDUES IN FOOD IN COMMERCE OR AT CONSUMPTION

Olthof (2000) reported results of the national food monitoring programme in The Netherlands for pears from 1994 to 1996. They are shown in Table 36.

Table 36. Residues of chlormequat in commercial pears, The Netherlands 1994-1996 (Olthof, 2000).

Samples analysed	Samples without residues (<LOQ of 0.05 mg/kg)	Samples with residues < MRL	Samples with residues >MRL	Mean[1], mg/kg	MRL, mg/kg
478	455	23	-	0.1	3

[1]For samples with residues <LOD a residue of 0.025 mg/kg was taken to calculate the mean

NATIONAL MAXIMUM RESIDUE LIMITS

The national MRLs shown below were reported.

Country	Residue definition	Commodity	MRL, mg/kg
Germany	Chlormequat, expressed as chlormequat cation	Rape seed	10
		Cultivated mushrooms	10
		Oats	5
		Maize	5
		Apples	3
		Pears	3
		Barley, rye, triticale, wheat	2
		Grapes	1
		Hops	0.1
		Olives	0.1
		Other oil seeds	0.1
		Tree nuts	0.1
		Tea	0.1
		Other commodities of plant origin	0.05
The Netherlands	Chlormequat, expressed as chlormequat cation	Nuts	0.1*
		Pears	3
		Table and wine grapes	1
		Olives	0.1*
		Beans (with pods)	0.05
		Beans (without pods)	0.05
		Peas (with pods)	0.05
		Peas (without pods)	0.05
		Oil seeds	0.1*
		Tea	0.1*
		Hops	5
		Oats	2
		Wheat, rye, triticale, barley	0.05*
		Other food commodities	0.05*
Poland	Chlormequat	Cereal grains	3
		Pear	3
		Tomato	0.2
		Other products of plant origin	0.05

* At or about the LOQ

APPRAISAL

Chlormequat was evaluated within the CCPR periodic review programme in 1994. The Meeting estimated maximum residue levels for a number of commodities, which were recorded as guideline levels only, since the ADI was withdrawn. The 1994 JMPR noted that feeding studies in farm animals and analytical methods for residues in animal products would be desirable. As an ADI was allocated by the 1997 JMPR, the estimates made in 1994 were recommended for use as MRLs in 1997.

The CCPR at its thirtieth session noted that animal transfer studies in poultry and cattle would be available in 1998.

The compound was reviewed toxicologically again in 1999, when an acute RfD was allocated. The 1999 JMPR recommended that an evaluation of residues should be scheduled shortly so that an acute risk assessment could be concluded.

Analytical methods for the determination of residues of chlormequat in water, cereals, pears, and animal products, data on stability in storage of animal products, data on residues in pears and cereals, and the results of a feeding study in dairy cattle and poultry were made available to the Meeting by the manufacturers. The Netherlands submitted its official method of analysis for chlormequat in pears. Information on national MRLs and GAP was provided by the governments of Germany, The Netherlands, and Poland .

Methods of analysis

Chlormequat is difficult to analyse because of its chemical nature and because the residue must be separated from native quarternary ammonium compounds in plant material. Older methods involve lengthy clean-up, liquid-liquid partition or column chromatography (ion-exchange, alumina), and semi-quantitative thin-layer chromatographic or photometric detection, but these methods allow only poor reproducibility. More recent methods are based on head-space gas chromatography after pyrolysis of chlormequat to acetylene in an alkaline medium, HPLC by ion-pair chromatography with conductivity detection, or liquid chromatography with mass spectrometric detection.

For cereal grains, the LOQ was 0.05 mg/kg with ion-pair chromatography and 1 mg/kg with head-space gas chromatography with flame ionization detection. The latter method resulted in high values in samples from untreated control plots. The liquid chromatography-mass spectrometric method was used to determine chlormequat residues in pears (LOQ, 0.3 mg/kg).

The ion chromatographic method was validated for animal products, resulting in LOQs of 0.05 mg/kg for eggs and tissues and 0.01 mg/kg for milk. The ion chromatographic technique was also used to analyse chlormequat in water, with an LOQ of 0.05 mg/l.

Stability of residues in stored analytical samples

Two samples each of milk, eggs, liver, and fat from farm animals fed chlormequat were stored for 25-33 months at -18 °C. The remaining compound represented 76-140% of the initial concentration in milk, 45-100% in eggs, 60-82% in liver, and 71-90% in fat, with great variation. The Meeting was not able to decide whether chlormequat is stable in enzyme-containing matrices and noted that the study was inadequate.

Results of supervised trials

The present Meeting received the results of new supervised trials on pears and cereals. These data and those reported by the 1994 JMPR on which the recommended MRLs for numerous commodities are based were re-evaluated in the view of current GAP and to estimate STMR and HR values.

Chlormequat is registered for use on *pear* in Belgium (at four to five applications of 1.4 kg ai/ha, 0.24 kg ai/hl, 600 l water/ha), Denmark (at two applications of 0.75-1.8 kg ai/ha, 0.075-0.18 kg ai/hl, 1000 l water/ha), The Netherlands (at one or two applications of 0.75-2.3 kg ai/ha, 0.094-0.15 kg ai/hl, 800-1500 l water/ha), and Spain (at five to six applications of 0.9 kg ai/ha, 0.1 kg ai/hl, 900 l water/ha). The PHIs range from 42 days in Denmark to 90 days in The Netherlands, or treatment is fixed at a certain growth stage (Belgium, Spain).

Six trials carried out in The Netherlands in 1968 were not included in the assessment as no information on the spray concentration was received, but the application rates used in two trials conducted in 1983 (two applications of 1.1-1.8 kg ai/ha, 0.11-0.18 kg ai/hl, 1000 l water/ha) were acceptable in respect of GAP in The Netherlands. The concentrations of residues were 5.3 and 6.9 mg/kg (calculated as chlormequat chloride) 124 and 113 days after the last treatment, respectively. Two further trials (four applications of 1.2-1.6 kg ai/ha, 0.2 kg ai/hl, 600 l water/ha; PHI, 90 and 101 days) complied with the Belgian GAP. The concentrations of residues were 1.6 and 8.1 mg/kg, calculated as chlormequat chloride.

Eight supervised trials were conducted in France in 1998 and 1999. Those carried out in 1998 were in accordance with the Belgian GAP (five applications of 1.4-1.5 kg ai/ha, 0.24 kg ai/hl, 600 l water/ha). The concentrations 44 or 45 days after the last treatment were 4, 4.6, 5.6, and 7.5 mg/kg, calculated as chlormequat chloride. In 1999, only one application was given. The rates used in these trials were not compatible with a currently registered GAP.

The concentrations of residues found in the trials conducted according to GAP were, in rank order (median in italics), 1.6, 4, 4.6, ***5.3***, ***5.6***, 6.9, 7.5, and 8.1 mg/kg calculated as chlormequat chloride or 1.2, 3.1, 3.6, ***4.1***, ***4.3***, 5.3, 5.8, 6.3 mg/kg calculated as chlormequat cation. The Meeting estimated a maximum residue level of 10 mg/kg, confirming the previous recommendation, an STMR value of 4.2 mg/kg, and a HR value of 6.3 mg/kg for pears, calculated as chlormequat cation.

The Meeting received the results of numerous supervised trials on *barley* carried out in the UK, but these data could not be evaluated as high values were determined in samples from untreated plots and no information was submitted about the analytical method used. Trial carried out in Latvia in 1998 and in Hungary in 1991 provided no information on application rates or the analytical method used.

The supervised trials reported by the 1994 JMPR that are in accordance with current GAP were re-evaluated:

Country	No. of trials	Concentration of residues, calculated as chlormequat chloride, mg/kg	In accordance with GAP of
Summer barley			
Denmark	4	<0.05 (2 trials), 0.05, 0.3	Netherlands, Belgium
Germany	2	0.17, 0.62	Netherlands, Belgium
Sweden	7	<0.05 (4 trials), 0.1, 0.19, 0.73	Netherlands
UK	3	0.18, 0.24, 0.37	UK
Winter barley			
Denmark	1	0.05	Netherlands
France	11	<0.05 (3 trials), 0.16, 0.18, 0.21, 0.24, 0.29, 0.3 (2 trials), 0.35	Netherlands, Belgium
Germany	5	1.3, 1.6, 1.6, 2.1, 2.3	Germany
Germany	2	0.17, 0.18	Netherlands, Belgium
Sweden	1	0.42	Netherlands, Belgium
Switzerland	2	0.23, 0.29	Netherlands, Belgium
UK	9	<0.05 (2 trials), 0.05, 0.07, 0.15, 0.24, 0.36, 0.43, 0.58	UK

The 47 values for residues, in rank order, were <0.05 (11 trials), 0.05 (3 trials), 0.07, 0.1, 0.15, 0.16, 0.17 (2 trials), 0.18 (3 trials), ***0.19***, 0.21, 0.23, 0.24 (3 trials), 0.29 (2 trials), 0.3 (3 trials), 0.35, 0.36, 0.37, 0.42, 0.43, 0.58, 0.62, 0.73, 1.3, 1.6 (2 trials), 2.1, and 2.3 mg/kg calculated as chlormequat chloride, or <0.04 (11 trials), 0.04 (3 trials), 0.05, 0.08, 0.12 (2 trials), 0.13 (2 trials), 0.14 (3 trials), ***0.15***, 0.16, 0.18, 0.19 (3 trials), 0.22 (2 trials), 0.23 (3 trials), 0.27, 0.28, 0.29, 0.33 (2 trials), 0.45, 0.48, 0.57, 1.0, 1.2 (2 trials), 1.6, and 1.8 mg/kg calculated as chlormequat cation.

The Meeting estimated a maximum residue level of 2 mg/kg to replace the previous recommendation of the 1994 JMPR (0.5 mg/kg), an STMR value of 0.15 mg/kg, and a HR value of 1.8 mg/kg for barley, calculated as chlormequat cation.

The Meeting received the results of four supervised trials on *oats* carried out in 1993 in Austria, but the data could not be evaluated as high values were determined in samples from untreated plots. One trial in Germany and one in the UK were conducted in accordance with Belgian and British GAP, respectively. The concentrations were 3 and 0.8 mg/kg in oat grains, calculated as chlormequat cation.

The trials carried out in accordance with current GAP and summarized by the 1994 JMPR were re-evaluated:

Country	No. of trials	Concentration of residues, calculated as chlormequat chloride, mg/kg	In accordance with GAP of
Germany	16	0.09, 0.14, 0.45, 0.51, 0.9, 1.1, 1.2,	Belgium, Finland
		1.5, 1.6, 1.7, 1.8, 1.9, 2.0, 2.4, 2.4, 3.3	Netherlands
Germany	2	1.5, 3.7	Germany
UK	3	0.1, 0.63, 9.2	UK

The 23 values (21 from submissions in 1994, two from submissions in 2000), in rank order, were 0.09, 0.1, 0.14, 0.45, 0.51, 0.63, 0.8, 0.9, 1.1, 1.2, ***1.5*** (2 trials), 1.6, 1.7, 1.8, 1.9, 2.0, 2.4, 2.4, 3, 3.3, 3.7, and 9.2 mg/kg calculated as chlormequat chloride, or 0.07, 0.08, 0.11, 0.35, 0.39, 0.49, 0.62, 0.7, 0.85, 0.93, ***1.2*** (3 trials), 1.3, 1.4, 1.5, 1.6, 1.9 (2 trials), 2.3, 2.6, 2.9, and 7.1 mg/kg calculated as chlormequat cation.

The Meeting estimated a maximum residue level of 10 mg/kg, confirming the previous recommendation, an STMR value of 1.2 mg/kg, and a HR value of 7.1 mg/kg for oats, calculated as chlormequat cation.

Triticale, rye and wheat have comparable use patterns in GAP of Belgium, Denmark, Germany, and the UK. The Meeting received the results of four trials each on triticale and on rye (2.5-5 kg ai/ha) in the UK, which were not in accordance with GAP (maximum, 1.7 kg ai/ha). Two Austrian trials each on rye and wheat could not be evaluated as high values were determined in samples from untreated plots. Eight trials on wheat in the UK could not be evaluated as information on the analytical method used was not submitted. One trial of wheat was carried out in Latvia in 1998, but no information on the application rate or the analytical method used was received. In two trials conducted in Germany in 1988, the application rates were acceptable with regard to British, Italian, and Finnish GAP (1.5-1.7 kg ai/ha); the concentrations were 0.2 and 0.24 mg/kg, calculated as chlormequat chloride.

The supervised trials in accordance with current GAP and summarized by the 1994 JMPR were re-evaluated:

Country	No. of trials	Concentration of residues, calculated as chlormequat chloride, mg/kg	In accordance with GAP of
Summer rye			
Germany	4	0.06, 1.5, 2.1, 2.6	Belgium
Winter rye			
Germany	13	<0.05, 0.22, 0.24, 0.3 (2 trials), 0.33, 0.34,	UK
		0.45, 0.62, 0.88, 1.2, 1.4, 1.9	
Germany	2	0.45, 1.8	Germany
Germany	7	<0.05, 0.05, 0.07, 0.08, 0.09 (2 trials), 0.43	Netherlands
Summer wheat			
Germany	9	0.31, 0.39, 0.52, 0.59, 0.68, 1.2, 1.3 (2 trials), 1.5	Austria, Germany, Finland, UK
Germany	8	0.09, 0.32, 0.33, 0.41, 0.44 (2 trials), 0.48, 0.59	Finland, UK
Germany	2	0.81, 1.5	Netherlands
Winter wheat			
Denmark	1	0.15	Belgium
France	3	<0.05 (3 trials)	Belgium
Germany	6	0.17, 0.23, 0.28, 0.31, 0.34, 0.37	Denmark, UK

Country	No. of trials	Concentration of residues, calculated as chlormequat chloride, mg/kg	In accordance with GAP of
Germany	2	0.28, 0.62	Belgium, Germany, Netherlands
UK	2	0.05, 1.4	UK

The concentrations of residues in 26 trials in *rye grain*, in rank order, were <0.05 (2 trials), 0.05, 0.06, 0.07, 0.08, 0.09 (2 trials), 0.22, 0.24, 0.3 (2 trials), ***0.33***, ***0.34***, 0.43, 0.45 (2 trials), 0.62, 0.88, 1.2, 1.4, 1.5, 1.8, 1.9, 2.1, and 2.6 mg/kg calculated as chlormequat chloride, or <0.04 (2 trials), 0.04, 0.05 (2 trials), 0.06, 0.07 (2 trials), 0.17, 0.19, 0.23 (2 trials), ***0.26*** (2 trials), 0.33, 0.35 (2 trials), 0.48, 0.68, 0.93, 1.1, 1.2, 1.4, 1.5, 1.6, and 2 mg/kg calculated as chlormequat cation.

The concentrations of residue in 35 trials in *wheat grain* (33 from 1994, 2 from 2000) were <0.05 (3 trials), 0.05, 0.09, 0.15, 0.17, 0.2, 0.23, 0.24, 0.28 (2 trials), 0.31 (2 trials), 0.32, 0.33, 0.34, ***0.37***, 0.39, 0.41, 0.44 (2 trials), 0.48, 0.52, 0.59 (2 trials), 0.62, 0.68, 0.81, 1.2, 1.3 (2 trials), 1.4, and 1.5 (2 trials) mg/kg calculated as chlormequat chloride, or <0.04 (3 trials), 0.04, 0.07, 0.12, 0.13, 0.16, 0.18, 0.19, 0.22 (2 trials), 0.24 (2 trials), 0.25, 0.26 (2 trials), ***0.29***, 0.3, 0.32, 0.34 (2 trials), 0.37, 0.4, 0.46 (2 trials), 0.48, 0.53, 0.63, 0.93, 1 (2 trials), 1.1, and 1.2 (2 trials) mg/kg calculated as chlormequat cation.

As the use patterns are comparable and the STMR values are close, the two data sets were combined: <0.04 (5 trials), 0.04 (2 trials), 0.05 (2 trials), 0.06, 0.07 (3 trials), 0.12, 0.13, 0.16, 0.17, 0.18, 0.19 (2 trials), 0.22 (2 trials), 0.23 (2 trials), 0.24 (2 trials), 0.25, ***0.26*** (4 trials), 0.29, 0.3, 0.32, 0.33, 0.34 (2 trials), 0.35 (2 trials), 0.37, 0.4, 0.46 (2 trials), 0.48 (2 trials), 0.53, 0.63, 0.68, 0.93 (2 trials), 1 (2 trials), 1.1 (2 trials), 1.2 (3 trials), 1.4, 1.5, 1.6, and 2 mg/kg calculated as chlormequat cation.

The Meeting estimated a maximum residue level of 3 mg/kg, an STMR value of 0.26 mg/kg, and a HR value of 2 mg/kg, calculated as chlormequat cation, for rye and wheat, and recommended that these values be extrapolated to triticale. The previous MRL recommended by the 1994 JMPR for rye (3 mg/kg) was confirmed, whereas that for wheat (2 mg/kg) was replaced.

Chlormequat is registered for use on *rape seed* in Belgium (at 0.69 kg ai/ha) and in the UK (at 1.9 kg ai/ha). No new GAP and no data on residues were submitted.

The 1994 JMPR estimated a maximum residue level of 5 mg/kg for rape seed on the basis of one British and nine German trials. The concentrations of residues, in rank order, were 1.7, 2.1, 2.2, 2.3, ***2.6***, *2.7*, 2.9, 3,7, 4.3, and 5.8 mg/kg calculated as chlormequat chloride or 1.3, 1.6, 1.7, 1.8, *2*, ***2.1***, 2.2, 2.8, 3.3, 4.5 mg/kg calculated as chlormequat cation.

The Meeting estimated an STMR value of 2.05 mg/kg for rape seed.

The 1994 JMPR estimated a maximum residue level of 20 mg/kg (fresh weight) for *dry straw and fodder of barley, oats, rye, and wheat*. The 2000 JMPR considered the results of all the available supervised trials conducted according to current GAP:

Country	No. of trials	Concentration of residues, calculated as chlormequat chloride, mg/kg	In accordance with GAP of
Summer barley straw			
Denmark	4	1.3, 2.7, 4.3, 4.4	Netherlands, Belgium
Germany	2	4, 4.4	Netherlands, Belgium
UK	3	1.6, 1.6, 4.9	UK
Winter barley straw			
Denmark	1	0.9	Netherlands
France	11	0.36, 1.8, 2.4, 2.8, 3.1, 4.4, 4.7, 5.4, 5.5, 8.5, 11	Netherlands, Spain
Germany	5	5.8, 6.2, 6.4, 8.7, 12	Germany

Country	No. of trials	Concentration of residues, calculated as chlormequat chloride, mg/kg	In accordance with GAP of
Germany	2	5.8, 9	Netherlands, Belgium
Switzerland	2	4.2, 4.5	Netherlands, Belgium
UK	9	0.98, 1, 1.1, 2.2, 2.4 (2 trials), 8.9, 12, 16	UK
Oat straw			
Germany	16	0.9, 1.2 (2 trials), 1.3, 1.6, 1.9 (2 trials), 2.2,	Belgium, Finland,
		3.0, 4.0, 4.8, 6.3, 8.2, 9.9 (3 trials)	Netherlands
Germany	2	5.2, 12	Germany
UK	3	0.48, 3.3, 25	UK
Summer rye straw			
Germany	4	0.2, 0.3, 4.7, 9	Belgium
Winter rye			
Germany	12	2.2, 2.7, 2.8, 3.1, 4.3, 4.5, 4.8, 5.7, 6.9, 9.6 (2 trials), 12	UK
Germany	1	7.5	Germany
Germany	1	5.5	Netherlands
UK	2	0.48, 12	UK
Summer wheat straw			
Germany	9	10, 13, 14, 17, 18 (2 trials), 20, 21, 29	Austria, Germany,
			Finland, UK
Germany	8	5.8, 7, 7, 11, 12, 13, 15, 17	Finland, UK
Germany	2	6.2, 13	Netherlands
Winter wheat straw			
Denmark	1	1.5	Belgium
France	3	2.3, 2.6, 4.8	Belgium
Germany	6	3.9, 4.8, 5.1, 6.1, 6.6, 8.0	Denmark, UK
Germany	2	7.2, 15	Belgium, Germany, Netherlands
UK	2	0.5, 5.4	UK

Two further trials each on oats and wheat were submitted to the current JMPR. The residues of chlormequat chloride were 0.7 and 3 mg/kg in oat straw and 0.5 and 0.9 mg/kg in wheat straw (fresh weight).

The concentrations of residues (fresh weight) in 39 trials with *barley straw*, in rank order, were 0.36, 0.9, 0.98, 1, 1.1, 1.3, 1.6 (2 trials), 1.8, 2.2, 2.4 (3 trials), 2.7, 2.8, 3.1, 4, 4.2, 4.3, ***4.4*** (3 trials), 4.5, 4.7, 4.9, 5.4, 5.5, 5.8 (2 trials), 6.2, 6.4, 8.5, 8.7, 8.9, 9, 11, 12 (2 trials), and 16 mg/kg calculated as chlormequat chloride or 0.28, 0.7, 0.76, 0.78, 0.85, 1, 1.2 (2 trials), 1.4, 1.7, 1.9 (3 trials), 2.1, 2.2, 2.4, 3.1, 3.3 (2 trials), ***3.4*** (3 trials), 3.5, 3.7, 3.8, 4.2, 4.3, 4.5 (2 trials), 4.8, 5, 6.6, 6.8, 6.9, 7, 8.5, 9.3, 9.3, and 12 mg/kg calculated as chlormequat cation.

The concentrations in 23 trials with *oat straw*, were 0.48, 0.7, 0.9, 1.2 (2 trials), 1.3, 1.6, 1.9 (2 trials), 2.2, *3* (2 trials), 3.3, 4, 4.8, 5.2, 6.3, 8.2, 9.9 (3 trials), 12, and 25 mg/kg calculated as chlormequat chloride or 0.37, 0.54, 0.7, 0.93 (2 trials), 1, 1.2, 1.5 (2 trials), 1.7, ***2.3*** (2 trials), 2.6, 3.1, 3.7, 4, 4.9, 6.4, 7.7 (3 trials), 9.3, and 19 mg/kg calculated as chlormequat cation.

The values in 20 trials with *rye straw* were 0.2, 0.3, 0.48, 2.2, 2.7, 2.8, 3.1, 4.3, 4.5, ***4.7***, ***4.8***, 5.5, 5.7, 6.9, 7.5, 9, 9.6 (2 trials), and 12 (2 trials) mg/kg calculated as chlormequat chloride or 0.16, 0.23, 0.37, 1.7, 2.1, 2.2, 2.4, 3.3, 3.5, ***3.7*** (2 trials), 4.3, 4.4, 5.4, 5.8, 7, 7.4 (2 trials), and 9.3 (2 trials) mg/kg calculated as chlormequat cation.

The concentrations in 35 trials with *wheat straw* were 0.5 (2 trials), 0.9, 1.5, 2.3, 2.6, 3.9, 4.8 (2 trials), 5.1, 5.4, 5.8, 6.1, 6.2, 6.6, 7 (2 trials), ***7.2***, 8, 10, 11, 12, 13 (3 trials), 14, 15 (2 trials), 17 (2 trials), 18 (2 trials), 20, 21, and 29 mg/kg calculated as chlormequat chloride, or 0.39 (2 trials), 0.7, 1.2, 1.8, 2, 3, 3.7 (2 trials), 4, 4.2, 4.5, 4.7, 4.8, 5.1, 5.4 (2 trials), ***5.6***, 6.2, 7.8, 8.5, 9.3, 10 (3 trials), 11, 12 (2 trials), 13 (2 trials), 14 (2 trials), 16 (2 trials), and 22 mg/kg calculated as chlormequat cation.

The 117 values available for *straw* (fresh weight), in rank order, are: 0.16, 0.23, 0.28, 0.37 (2 trials), 0.39 (2 trials), 0.54, 0.7 (3 trials), 0.76, 0.78, 0.85, 0.93 (2 trials), 1, 1, 1.2 (4 trials), 1.4, 1.5 (2 trials), 1.7 (3 trials), 1.8, 1.9 (3 trials), 2, 2.1 (2 trials), 2.2 (2 trials), 2.3 (2 trials), 2.4 (2 trials), 2.6, 3, 3.1 (2 trials), 3.3 (3 trials), 3.4 (3 trials), 3.5 (2 trials), ***3.7*** (6 trials), 3.8, 4, 4.2 (2 trials), 4.3 (2 trials), 4.4, 4.5 (3 trials), 4.7, 4.8 (2 trials), 4.9, 5, 5.1, 5.4 (3 trials), 5.6, 5.8, 6.2, 6.4, 6.6, 6.8, 6.9, 7 (2 trials), 7.4 (2 trials), 7.7 (3 trials), 7.8, 8.5 (2 trials), 9.3 (6 trials), 10 (3 trials), 12 (3 trials), 13 (2 trials), 14 (2 trials), 16 (2 trials), 19, and 22 mg/kg calculated as chlormequat cation.

Allowing for the standard 89% of dry matter (FAO, 1997) in cereal straw (barley, 89%; oats, 90%; rye, 88%; wheat, 88%), the Meeting estimated a maximum residue level and an STMR value for dry straw and fodder of cereal grains of 30 mg/kg and 4.2 mg/kg (3.7/0.89), respectively, calculated as chlormequat cation. The previously recommended MRL of 20 mg/kg (fresh weight) for dry straw and fodder of barley, oats, rye, and wheat is withdrawn.

The 1994 JMPR estimated a maximum residue level of 20 mg/kg for *oat and rye forage* (*green*) on a fresh weight basis. The current Meeting considered the supervised trials that had been conducted according to current use patterns:

Country	No. of trials	Concentration of residues, calculated as chlormequat chloride, mg/kg	In accordance with GAP of
Oats			
Germany	17	1.1, 1.3, 1.5, 1.8, 2.5, 2.9, 3.1, 3.5, 3.7, 4.3, 6.4, 6.9, 7.6, 8.1, 15, 15, 17	Belgium, Germany, Finland, Netherlands
UK	1	4.7	UK
Summer rye			
Germany	4	11, 12, 12, 13	
Belgium			
Winter rye			
Germany	14	1.9, 2.1, 2.2, 3.6, 4.1, 4.2, 4.3, 4.9, 5.9, 9, 17, 18, 20, 28	Germany, UK, Netherlands

A further trial on oats was submitted for consideration by the 2000 JMPR, in which the concentration of residue was 28 mg/kg, expressed as chlormequat chloride, in the whole green plant 30 days after treatment.

The concentrations found in all 37 trials on oat and rye forage (fresh weight) were, in rank order, 1.1, 1.3, 1.5, 1.8, 1.9, 2.1, 2.2, 2.5, 2.9, 3.1, 3.5, 3.6, 3.7, 4.1, 4.2, 4.3 (2 trials), 4.7, ***4.9***, 5.9, 6.4, 6.9, 7.6, 8.1, 9, 11, 12 (2 trials), 13, 15 (2 trials), 17 (2 trials), 18, 20, and 28 (2 trials) mg/kg calculated as chlormequat chloride, or 0.85, 1, 1.2, 1.4, 1.5, 1.6, 1.7, 1.9, 2.2, 2.4, 2.7, 2.8, 2.9, 3.2, 3.3 (3 trials), 3.6, ***3.8***, 4.6, 5, 5.3, 5.9, 6.3, 7, 8.5, 9.3 (2 trials), 10, 12 (2 trials), 13 (2 trials), 14, 16, and 22 (2 trials) mg/kg calculated as chlormequat cation.

Allowing for the standard 30% of dry matter in cereal forage (FAO, 1997), the Meeting estimated a maximum residue level of 100 mg/kg and an STMR value of 13 mg/kg (3.8/0.3), calculated as chlormequat cation (dry weight), for oat and rye forage. The previous MRL recommendation (20 mg/kg, fresh weight) is withdrawn.

No new data on residues or GAP for maize were submitted. The 1994 JMPR had received the results of nine supervised trials conducted in Germany at rates within the range of Belgium GAP. For green maize plants, including cobs, the following concentrations (fresh weight) were reported (PHI, 13-35 days): 0.39, 0.92, 1.6, 2.4, ***3.4***, 4.3, 4.8, 5.0, and 6.2 mg/kg calculated as chlormequat chloride or 0.3, 0.71, 1.2, 1.9, ***2.6***, 3.3, 3.7, 3.9, and 4.8 mg/kg calculated as chlormequat cation.

Allowing for the standard 40% of dry matter in maize forage (FAO, 1997), the Meeting estimated a maximum residue level of 15 mg/kg and an STMR value of 6.5 mg/kg (2.6/0.4), calculated as chlormequat cation (dry weight).

For maize fodder, the following concentrations (fresh weight) were reported (PHI, 78-113 days): 0.36, 0.68, 0.8, 2.4, *2.5*, 4.1, 4.3, 4.5, 5.1 mg/kg calculated as chlormequat chloride, or 0.28, 0.53, 0.62, ***1.9*** (2 trials), 3.2, 3.3, 3.5, and 4 mg/kg calculated as chlormequat cation.

Allowing for the standard 83% of dry matter in maize stover (FAO, 1997), the Meeting estimated a maximum residue level of 7 mg/kg and an STMR value of 2.3 mg/kg (1.9/0.83), calculated as chlormequat cation (dry weight).

Residues in animal and poultry commodities

The Meeting estimated the dietary burden of chlormequat residues in farm animals on the basis of the diets listed in Appendix IX of the FAO Manual. Calculation from the MRLs yields maximum concentrations of residues in feed suitable for estimating MRLs for animal commodities. Calculation from the STMR values for feed allows estimation of STMR values for animal commodities.

Commodity	MRL, mg/kg	Group	% dry matter	MRL/ dry matter	% of diet			Concentration of residue, mg/kg		
					Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Barley grain	2	GC	88	2.3						
Barley straw	30	AS	100	30						
Oat grain	10	GC	89	11	35	40	80	3.9	4.5	9.0
Oat forage	100	AF	100	100	25	60		25	60	
Oat straw	30	AS	100	30						
Maize forage	15	AF	100	15	40			6		
Maize fodder	7	AS	100	7						
Rye grain	3	GC	88	3.4						
Rye forage	100	AF	100	100						
Rye straw	30	AS	100	30						
Wheat grain	3	GC	89	3.4			20			0.67
Wheat straw	30	AS	100	30						
Sum					100	100	100	35	65	9.6

Commodity	STMR, mg/kg	Group	% dry matter	STMR/ dry matter	% of diet			Concentration of residue, mg/kg		
					Beef cattle	Dairy cows	Poultry	Beef cattle	Dairy cows	Poultry
Barley grain	0.15	GC	88	0.17						
Barley straw	4.2	AS	100	4.2						
Oat grain	1.2	GC	89	1.4	35	40	80	0.47	0.54	1.1
Oat forage	12.7	AF	100	13	25	60		3.2	7.6	
Oat straw	4.2	AS	100	4.2						
Maize forage	6.5	AF	100	6.5	40			2.6		
Maize fodder	2.3	AS	100	2.3						
Rye grain	0.26	GC	88	0.30			20			0.06
Rye forage	12.7	AF	100	13						
Rye straw	4.2	AS	100	4.2						
Wheat grain	0.26	GC	89	0.29						
Wheat straw	4.2	AS	100	4.2						
Sum					100	100	100	6.3	8.1	1.1

Cows

Groups of three lactating cows were given chlormequat chloride in the diet twice daily at a dose of 240, 720, or 2400 mg/animal per day, equivalent to 0.4, 1.3, and 4 mg/kg bw per day or 12, 36, and 120 ppm on a dry weight basis, for 28 consecutive days. Two additional animals were treated at the high dose for 28 days and slaughtered 2 or 7 days after the last dose. The doses were equivalent to 0.31, 1, and 3.1 mg/kg bw per day (or 9.3, 28, and 93 ppm), calculated as chlormequat cation. At the lowest dose, the average concentrations of chlormequat chloride residues were 0.029 mg/kg in milk, 0.1 mg/kg in liver, and 0.2 mg/kg in kidney. No residues were found in meat or fat. At the medium and high doses, the plateau concentrations of chlormequat chloride residue in milk were 0.1 and 0.2 mg/kg. Concentrations up to 0.11 mg/kg were determined in some meat and fat samples. The concentrations were 0.1 and 0.4 mg/kg in liver and 0.4 and 0.8 mg/kg in kidney at the two doses, respectively, indicating that the values in kidney were at least twice as high as in liver. The concentrations of chlormequat chloride in skimmed milk were similar to those in whole milk.

The concentration of chlormequat residues in milk reached a plateau 10-11 days after the first treatment with the medium dose, but after 3-4 days with the low and high doses. The residues were cleared rapidly from meat, fat, and liver, and none could be determined in these tissues 2 days after the end of dosing. The concentrations in milk and kidney fell to about 20% of their plateau values. After 7 days, the values for milk were below the LOQ of 0.01 mg/kg, but 0.09 mg/kg remained in kidney. Although milk and tissue samples were frozen on the day of sampling, they were analysed in part 1 year later, and no adequate information on stability was received.

According to the recommendation of the 1997 JMPR, the maximum residue level and the STMR value for milk were calculated on the basis of dietary burdens of 65 and 8.1 mg/kg, respectively, for dairy cattle. The maximum residue levels and the STMR values for meat, liver, and kidney were derived from dietary burdens of 35 or 6.3 mg/kg, respectively, for beef cattle. The following table shows the highest and the mean actual and extrapolated concentrations of residues for estimation of MRLs and STMR values for chlormequat.

Dose, ppm	Concentration of residues, mg/kg, calculated as chlormequat cation									
	Milk		Liver		Kidney		Muscle		Fat	
	High	Mean[1]	High	Mean	High	Mean	High	Mean	High	Mean
MRL for beef cattle										
Extrapolated: 35			0.088	0.078	0.35	0.3	0.11	<0.04	0.05	<0.04
Actual: 28			0.07	0.062	0.28	0.24	0.085	0.04		
MRL for dairy cows										
Extrapolated: 65	0.35	0.13								
Actual: 93	0.5	0.18								
STMR for beef cattle										
Extrapolated: 6.3			0.053	0.042	0.16	0.084	<0.04	<0.04	<0.04	<0.04
Actual: 9.3			0.078	0.062		0.23	0.124			
STMR for dairy cows										
Extrapolated: 8.1	0.05	0.018								
Actual: 9.3	0.06	0.021								

[1] The mean concentration in milk was calculated from samples taken on days 3-28.

The Meeting estimated maximum residue levels of 0.5 mg/kg for milk, 0.1 mg/kg for liver, 0.5 mg/kg for kidney, and 0.2 mg/kg for meat and recommended that the HR values be 0.35 mg/kg for milk, 0.088 mg/kg for liver, 0.35 mg/kg for kidney, and 0.11 mg/kg for meat. The estimated STMR values are 0.018 mg/kg for milk, 0.042 mg/kg for liver, 0.084 for kidney, and 0.04 mg/kg for meat. No maximum residue level was recommended for fat.

Chickens

Three groups of four hens were given capsules containing chlormequat chloride at a dose of 0.72, 2.1, or 7.2 mg/bird per day, equal to 6, 18, and 60 ppm on a dry weight basis, for 28 consecutive days. Two additional groups of 12 hens were treated with the high dose for 28 days and slaughtered 2 or 7 days after the last dose. The doses were equivalent to 4.6, 14, and 46 ppm when calculated as chlormequat cation.

The lowest dose resulted in concentrations of chlormequat chloride residues in eggs at or above the LOQ of 0.05 mg/kg, while 0.05 mg/kg was found in liver and none in meat or fat. Plateau concentrations of 0.06 and 0.1 mg/kg were found in eggs of hens treated with the two higher doses after 1 week of dosing. The concentrations in meat and fat samples were below the LOQ of 0.05 mg/kg, while those in liver were 0.07 mg/kg at the medium dose and 0.18 mg/kg at the high dose.

The residues were cleared rapidly from meat, fat, and liver. No chlormequat chloride was determined in meat or fat. The concentrations in liver had fallen to 0.05 mg/kg 2 days after the end of dosing and to below the LOQ after 7 days. After 2 and 7 days, the residues in eggs had fallen to values below the LOQ of 0.05 mg/kg.

Egg and tissue samples were frozen on the day of sampling but were analysed in part 3 months (tissues) or 10 months (eggs) later. No adequate information on stability was received.

According to the recommendation of the 1997 JMPR, the maximum residue level and the STMR values for eggs and poultry tissues were calculated on the basis of dietary burdens of 9.6 and 1.1 mg/kg, respectively. The following table shows the highest and the mean actual and extrapolated concentrations of residues for estimation of MRLs and STMR values for chlormequat.

Dose, ppm	Concentrations of residues, mg/kg, calculated as chlormequat cation							
	Eggs		Meat		Liver		Fat	
	Highest	Mean[1]	Highest	Mean	Highest	Mean	Highest	Mean
MRL								
Extrapolated: 9.6	0.064	0.032	<0.04	<0.04	0.053	0.037	<0.04	<0.04
Actual: 14	0.093	0.046			0.077	0.054		
STMR								
Extrapolated: 1.1	0.011	<0.04	<0.04	<0.04	0.017	0.0096	<0.04	<0.04
Actual: 4.6	0.047				0.07	0.04		

[1] The mean concentration in eggs was calculated from samples taken on days 3-28.

The Meeting recommended an MRL of 0.1 mg/kg for eggs and offal, and 0.04* mg/kg for meat; no MRL was recommended for fat. The estimated STMR values were 0.04 for eggs, 0.0096 for liver, and 0 for meat. HR values of 0.064 mg/kg for eggs, 0.053 mg/kg for offal, and 0 for meat were estimated.

Fate of residues during processing

In three studies of the processing of *rape seed* reported by the 1994 JMPR, the mean processing factor for crude rape seed oil was <0.018. On the basis of the STMR value of 2.0 mg/kg for rape seed, an STMR-P value of 0.037 mg/kg was estimated for crude rape seed oil.

One study on the processing of *barley* to barley pearls submitted to the 1994 JMPR indicates a processing factor of 0.06. On the basis of the STMR value of 0.15 mg/kg for barley grain, an STMR-P value of 0.009 mg/kg was estimated for barley pearl. Another study indicated processing factors of 0.69 for malt and 0.015 for beer. On the basis of the STMR value of 0.15 mg/kg for barley, STMR-P values of 0.1 mg/kg and 0.0023 mg/kg were estimated for malt and beer, respectively.

Two studies on the processing of *oats* to oat flakes were submitted to the 2000 JMPR, but only one could be used for evaluation (processing factor, 0.27) because high values were found in samples from untreated plots in the second study. Two further studies were reported by the 1994

JMPR (processing factors, 0.1 and 0.25). On the basis of an STMR value of 1.2 mg/kg for oat grains and a mean processing factor of 0.21, an STMR-P value of 0.25 mg/kg was estimated for oat flakes.

One study on the processing of *rye* was submitted to the 2000 JMPR but could not be used for evaluation because high values were found in samples from untreated plots. In a study reported by the 1994 JMPR, the processing factors were 3.2 for bran, 0.99 for flour, 1.3 for wholemeal, and 0.95 for wholemeal bread. On the basis of the MRL of 3 mg/kg for rye, the following maximum residue levels were estimated: 10 mg/kg for rye bran, 3 mg/kg for rye flour, and 4 mg/kg for rye wholemeal. On the basis of the STMR value of 0.26 mg/kg, STMR-P values were estimated as 0.83 mg/kg for rye bran, 0.26 mg/kg for rye flour, 0.34 mg/kg for rye wholemeal, and 0.25 mg/kg for rye wholemeal bread.

One study on the processing of *wheat* submitted to the 2000 JMPR showed processing factors of 2.5 for wheat bran, 1 for wholemeal, and 0.63 for wholemeal bread. In a study reported by the 1994 JMPR, processing factors of 4.6 for bran, 0.41 for flour, 1.4 for wholemeal, and 0.79 for wholemeal bread were estimated. The following processing factors were estimated: bran, 3.6; flour, 0.41; wholemeal, 1.2; and wholemeal bread, 0.71. On the basis of the MRL of 3 mg/kg for wheat grain, the following maximum residue levels were estimated: 10 mg/kg for wheat bran, 2 mg/kg for wheat flour, and 5 mg/kg for wheat wholemeal. On the basis of the STMR value of 0.26 mg/kg for wheat grain, STMR-P values of 0.94 mg/kg for wheat bran, 0.11 mg/kg for wheat flour, 0.31 mg/kg for wheat wholemeal, and 0.18 mg/kg for wheat wholemeal bread were estimated.

RECOMMENDATIONS

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

Definition of the residue for compliance with MRLs and for estimation of dietary intake: chlormequat cation.

Commodity		MRL, mg/kg		STMR, mg/kg	HR, mg/kg
CCN	Name	New	Previous		
GC 0640	Barley	2	0.5	0.15	1.8
	Barley beer			0.0023	
	Barley malt			0.1	
	Barley pearl			0.009	
AS 0640	Barley straw and fodder, dry	W[1]	20		
PE 0112	Eggs	0.1	-	0.04	0.064
MM 0814	Goat meat	0.2	-	0.04	0.11
MO 0098	Kidney of cattle, goats, pigs and sheep	0.5	-	0.084	0.35
MO 0099	Liver of cattle, goats, pigs and sheep	0.1	-	0.042	0.88
AS 0645	Maize fodder	7 (dry wt.)	-	2.3 (dry wt.)	
AF 0645	Maize forage	15 (dry wt.)	-	6.5 (dry wt.)	
MM 0097	Meat of cattle, pigs and sheep	0.2	-	0.04	0.11
ML 0107	Milk of cattle, goats and sheep	0.5	-	0.018	0.35
GC 0647	Oats	10	10	1.2	7.1
	Oat flakes			0.25	
AF 0647	Oat forage (green)	100 (dry wt.)	20	12.7 (dry wt.)	
AS 0647	Oat straw and fodder, dry[1]	W	20		
FP 0230	Pear[2]	10	10	4.2	6.3
PM 0110	Poultry meat	0.04*	-	0	0
PO 0111	Poultry, Edible offal of	0.1	-	0.0096	0.053

Commodity		MRL, mg/kg		STMR, mg/kg	HR, mg/kg
CCN	Name	New	Previous		
SO 0495	Rape seed			2.05	
OC 0495	Rape seed oil, crude			0.037	
GC 0650	Rye	3	3	0.26	2
CM 0650	Rye bran, unprocessed	10	10	0.83	
CF 1250	Rye flour	3	-	0.26	
AF 0650	Rye forage (green)	100 (dry wt.)	20	12.7 (dry wt.)	
AF 0650	Rye straw and fodder, dry	W[1]	20		
CF 1251	Rye wholemeal	4	3	0.34	
	Rye wholemeal bread			0.25	
AS 0081	Straw and fodder (dry) of cereal grains[3]	30 (dry wt.)	-	4.2 (dry wt.)	
GC 0653	Triticale	3	-	0.26	2
GC 0654	Wheat	3	2	0.26	2
CM 0654	Wheat bran, unprocessed	10	5	0.94	
CF 1211	Wheat flour	2	0.5	0.11	
AS 0654	Wheat straw and fodder, dry	W[1]	20		
CF 1212	Wheat wholemeal	5	2	0.31	
	Wheat wholemeal bread			0.18	

[1] Now included in recommendation for Straw and fodder (dry) of cereal grains
[2] The information provided to the JMPR precludes an estimate that the dietary intake would be below the acute RfD of 0.05 mg/kg bw
[3] Except Maize fodder

Further work or information

Desirable

Analytical study of stability in frozen storage of samples of animal products fortified with chlormequat.

Dietary risk assessment

Chronic intake

STMR or STMR-P values were estimated by the present Meeting for 27 raw and processed food commodities. When data on consumption were available, these values were used to estimate dietary intake. The results are shown in Annex 3.

The IEDIs for the five GEMS/Food regional diets, based on the estimated STMR values, represented 0-3 % of the ADI. The Meeting concluded that long-term intake of residues of chlormequat from uses that have been considered by the JMPR is unlikely to present a public health concern.

Short-term intake

The IESTI for chlormequat was calculated for the food commodities (and their processing fractions) for which maximum residue levels and STMR values were estimated and for which data on consumption were available. The results are shown in Annex 4. Pears were the only commodity for which the IESTI exceeded the acute RfD, with values of 240% for the general population and 700% for children.

The Meeting concluded that short-term intake of residues of chlormequat when used, other than on pears, in ways that have been considered by the JMPR is unlikely to present a public health concern.

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