CHLORMEQUAT (015)

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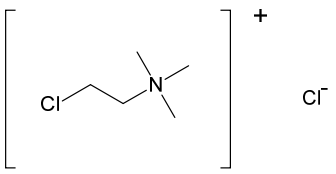
EXPLANATION

Chlormequat (usually manufactured and formulated as the chloride salt) is a plant growth regulator which acts primarily by reducing cell elongation, but also by lowering the rate of cell division. It inhibits the synthesis of gibberellins. It was scheduled for periodic review evaluation by the 2017 JMPR at the 48th session of the CCPR (2016). Chlormequat was previously evaluated by the JMPR in 1970, 1972, 1994 (periodic review), 1997, 1999 and 2000. It was evaluated for toxicology in 1997 and 1999 (in 1999 an acute reference dose was established).

Chlormequat has been considered by the Joint Meeting on Pesticides Specifications (JMPS), and specifications were established for chlormequat technical concentrates and soluble concentrates in 2005.

The manufacturer supplied information on identity, physicochemical properties metabolism (plant, confined rotational crops, and anima), environmental fate, methods of residue analysis, freezer storage stability, registered use patterns, supervised residue trials in grapes and cereals, fate of residues in processing, and animal transfer studies.

IDENTITY

ISO common name:	Chlormequat-chloride
IUPAC name:	2-chloroethyl-trimethylammonium chloride
Chemical Abstract name:	2-chloro-*N*, *N*, *N* -trimethylethanaminium chloride
CAS No.:	999-81-5
CIPAC No.:	143
Manufacturer's experimental name:	BAS 062W
Molecular Formula:	$C_5H_{13}Cl_2N$
Structural Formula:	
	Chlormequat-chloride
Molecular Weight:	158.1 g/mol (chlormequat-chloride) 122.6 g/mol (chlormequat cation)

PHYSICAL AND CHEMICAL PROPERTIES

The following data on the physicochemical properties of chlormequat chloride was received by the Meeting. The results are determined using pure active ingredients (typically > 99%), except where noted otherwise.

Property	Results	Test material purity and specification	Reference
Chlormequat-chloride			
Melting point	236 °C.	39-161-1, purity 99.5% w/w	Daum 2001a, 2001/1001821
	225 °C.	FW18414, purity 96.6% w/w (technical grade)	Kästel, R 2002a, 2002/1014123
Boiling point	The test substance decomposes immediately afer melting.	FW18414, purity 96.6% w/w (technical grade)	Daum 2001a, 2001/1001821
Relative Density	Active substance, pure: 1.241 g/mL at 20 °C	01743-257, purity 99.9% w/w	Kästel, R 2011a, PA09/006
Vapour pressure	Extrapolated: $< 1 \times 10^{-8}$ hPa at 20 °C	01743-257, purity 99.9% w/w	Kästel, R 2001a, 2001/1006102
	$< 1 \times 10^{-7}$ Pa at 20 °C $< 1 \times 10^{-7}$ Pa at 25 °C	CH 68 90 90 purity 100%	Guckel 1988 1988/10476
Henry's law constant	Henry's law constant at 20 °C: $<3.16 \times 10^{-13}$ kPa × m^3 × mol^{-1}; A vapour pressure of $<1 \times 10^{-9}$ kPa and a water solubility of $>50 \times 10^4$ g/L (pH 4 and 7) at 20 °C were used to calculate the Henry's law constant.	Not given	Ohnsorge 2001, 2001/1009199
Description of the physical state and colour, purity of the ai. and of technical grade	White solid	FW 18414, purity 96.6% w/w	Kästel, R 2001b, 2001/1009127
	Colourless solid	PFV107N004, purity 97.6% w/w	Daum 2001a, 2001/1001821
Solubility of purified active substance in water	pH 4 >500 g/L at 20 °C pH 7 >500 g/L at 20 °C pH 9 >500 g/L at 20 °C	39-161-1, purity 99.5% w/w	Daum 2000a, 2000/1012282
	>886 g/L at room temeprature	AC12042-69, purity 97.2% w/w	Weissenfeld M 2006a, 2006/1049813
Solubility in organic solvents	[g/100mL at 20 °C] acetone < 1.0 acetonitrile < 1.0 dichloromethane < 1.0 N, N-dimethylformamide < 1.0 ethyl acetate < 1.0 n-heptane < 1.0 methanol >25 1-octanol < 1.0 olive oil < 1.0 2-propanol 2.0-2.5 toluene < 1.0	39-161-1, purity 99.5% w/w	Daum 2000c, 2000/1003764
	[g/100mL at 20 °C] acetone 0.013 acetonitrile 0.297 dichloromethane 0.007 ethyl acetate < 0.001 n-heptane < 0.001 methanol 50.6 1-octanol 0.982 toluene < 0.001	01743-257, purity 99.9% w/w	Daum 2001, 2001/1009850
n-Octanol/ water partition coefficient	log P_{ow} at 25 °C deionised water log Pow -3.39 pH 4 log P_{ow} -3.08 pH 7 log P_{ow} -3.47 pH 9 log P_{ow} -3.07	39-161-1, purity 99.5% w/w	Daum 2000c, 2000/1013492
Hydrolysis rate at pH 4, 7 and 9 under sterile and dark conditions	Chlormequat is hydrolytically stable in aqueous solution at pH 4 to pH 9 (50 °C) for 5 days ($t_{1/2}$ > 1 year)	^{14}C-chlormequat-chloride, batch 94238 radiochemical purity 99%	Zohner 1995, 1998/10588

Property	Results	Test material purity and specification	Reference
Direct phototransformation in sterile water using artificial light	The first order half-life for photolytic degradation of chlormequat in double distilled water was 4844.9 experimental hours.	[Furanone-4-^{14}C] BYI 02960, vial no. C-1116A radiochemical purity 99.3%	Offizorz 1993, 1998/10585
Quantum yield of direct transformation	A mean quantum yield of $\Phi=4.74 \times 10^{-7}$ in aqueous solution of pH 5.4	30830, purity 95% w/w and ^{14}C-chlormequat-chloride, batch 93211 radiochemical purity ≥98%	Offizorz 1993, 1998/10585
Dissociation in water of purified active substance	Chlormequat-chloride is fully dissociated in aqueous solutions and has therefore no dissociation constant	Not given	Ohnsorge 2001c, 2001/1006083
pH	pH 4.7 (1% suspension in CIPAC water D at room temperature) pH 4.5 (1% suspension in pure water at room temperature)	FW 18414, purity 96.6% w/w	Kästel, R 2001b, 2001/1009127

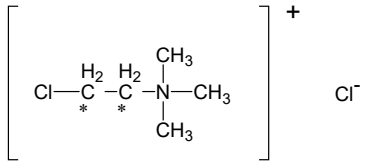
Formulations

BASF Code	Formulation type	Chlormequat-chloride content	Other active substances
BAS 062 00 W	SL	460g /L	320 g/L choline chloride
BAS 062 01 W	SL	460g /L	-
BAS 062 03 W	SL	750g /L	-
BAS 062 05 W	SL	500g /L	-
BAS 062 18 W	SL	120g /L	-
BAS 062 20 W	SL	400g /L	-
BAS 062 23 W	SL	460g /L	-
BAS 062 25 W	SL	77g /L	-
BAS 107 01 W	SL	230g /L	155 g/L ethephon, 75 g/L mepiquat-chloride
BAS 114 02 W	SL	368g /L	28 g/L choline chloride, 0.8 g/L imazaquin
BAS 120 00 W	SL	345g /L	115 g/L mepiquat-chloride

METABOLISM AND ENVIRONMENTAL FATE

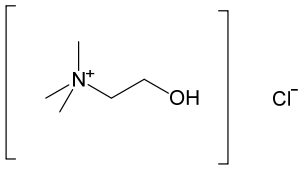
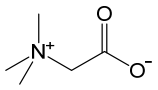
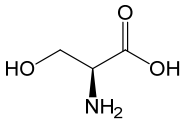
General

The studies for plant metabolism, animal metabolism and confined rotational crops were conducted with the test material shown below, with the label positions indicated in the following structural formula:



^{14}C-Chlormequat-chloride

Table 1 Structures of metabolites of chlormequat chloride

Name	Structure
Choline (chloride salt shown)	 Choline chloride
Betaine	 Betaine
Serine	 Serine

Plant metabolism

The metabolism of chlormequat-chloride has been investigated in grapes and wheat. A confined rotational crop study has also been conducted on spring wheat, lettuce and radish grown after three plant back intervals. Another study was conducted to investigate the metabolism of chlormequat-chloride in spring wheat, green beans, carrots, and head lettuce sown 30 days after soil application.

Grapes

Radiolabelled chlormequat-chloride was applied to grapevines (variety: *Müller-Thurgau*) as three consecutive foliar applications at growth stages BBCH 13–15, 15–17 and 57 using the BAS 062 05 W 05 formulation (soluble concentrate, de-ionised water as solvent) (Thianer and Deppermann 2013, 2012/1071012). The application rates were 180, 360 and 90 g ai/ha (total 630 g ai/ha).

Leaves were sampled from the immature plants immediately before and 22 days after the last application. The mature grapes were harvested at BBCH 89 (90 DALA) and the remaining plant material was separated into leaves, branches and stalks.

All collected plant samples were homogenised and the Total Radioactive Residues (TRR) were determined by combustion analysis followed by LSC. Additionally, the TRR was calculated as the sum of the Extracted Radioactive Residues (ERR) and Residual Radioactive Residues (RRR). The two methods for determining the TRRs, resulted in similar values for both grapes and leaves.

Table 2 Total Radioactive Residue (TRR) levels in grapevine samples treated with ^{14}C-Chlormequat-chloride

Matrix	DALA	TRR Measured (mg/kg)	TRR Calculated (mg/kg)
Grapes	90	0.155	0.182
Leaves	90	1.863	1.97

With subsequent methanol (×3) and water (×2) extractions, 99.3% of the radioactive residues in grapes and 94.7% of the radioactive residues of leaves were extracted.

Table 3 Extractability of radioactive residues in grapevine samples

Matrix	Methanol Extract		Aqueous Extract		ERR		RRR		TRR[a] Calculated (mg eq/kg)
	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	
Grapes	0.176	97.0	0.004	2.3	0.181	99.3	0.001	0.7	0.182
Leaves	1.70	86.2	0.167	8.5	1.86	94.7	0.103	5.2	1.97

[a]=calculated as the sum of ERR + RRR

In total, 0.178 mg eq/kg (97.9% TRR) of the Extracted Radioactive Residues of grapes and 1.65 mg eq/kg (83.8% TRR) of the Extracted Radioactive Residues of leaves were identified as the active substance chlormequat-chloride. A further 0.004 mg eq/kg in grapes was characterised by their chromatographic properties. In total 0.182 mg eq/kg or 100.1% TRR was identified or characterised in grapes. A further 0.100 mg eq/kg in leaves were characterised by their chromatographic properties. In total 1.75 mg eq/kg or 88.9% TRR was identified or characterised in leaves. The post-extraction solids (PES) in grapes after solvent extraction contained 0.001 mg eq/kg (0.7% TRR) and in leaves 0.103 mg eq/kg (5.2% TRR).

No metabolites of chlormequat were identified in the extracts of grapes and leaves, although some were characterised (see previous paragraph).

Table 4 Summary of identified components in grapes and leaves samples at 90 DALA

Designation	Methanol Extract		Aqueous Extract		Sum of Extracts	
	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)	(mg eq/kg)	(% TRR)
Grapes						
TRR Calculated					*0.182*	*100.0*
Identified						
Chlormequat-chloride	0.177	97.1	0.001	0.7	0.178	97.9
Total Identified					0.178	97.9
Characterised						
One peak characterised	0.002	0.8	0.003	1.4	0.004	2.2
Total Characterised					0.004	2.2
Total Identified and Characterised					0.182	100.1
Post-extraction solids (PES)					0.001	0.7
Sum of Total Identified and Characterised and PES					0.183	100.8
Leaves						
TRR Calculated					*1.97*	*100.0*
Identified						
Chlormequat-chloride	1.60	81.4	0.047	2.4	1.65	83.8
Total Identified					1.65	83.8
Characterised						
One peak characterised	0.005	0.3	0.095	4.9	0.100	5.1
Total Characterised					0.100	5.1
Total Identified and Characterised					1.75	88.9
Post-extraction solids (PES)					0.103	5.2
Sum of Total Identified and Characterised and PES					1.85	94.1

Using BASF Method 530/0 (extraction with water, methanol and 2N HCl), 97.8% of the radioactive residues were extracted from grapes and 91.0% from leaves. These results are comparable with the ERR calculated after extraction with the method used in the grape metabolism study (grapes 99.3% and leaves 94.7%). The concentration of the active substance chlormequat-chloride in grapes (0.170 mg/kg) was in accordance with the concentration determined using the method used in the metabolism study (0.178 mg/kg). The concentration of the active substance chlormequat-chloride in leaves (2.06 mg/kg) however was slightly higher than the concentration determined using the method used in the metabolism study (1.65 mg/kg).

Wheat

A metabolism study for spring wheat (Star variety) grown in a phytotron was provided to the Meeting (Keller, 1990). This study was also reviewed by the 1994 JMPR. Radiolabelled compound (2-chloro-[1,2-^{14}C]-ethyl-trimethylammonium chloride) was applied by foliar application to wheat plants at a target rate of 1380 g ai/ha.

Forage was collected at 0, 28, and 84 days after application, while grain and straw were collected at harvest maturity 118 days after application. Samples were homogenised, with the forage samples additionally being lyophilised.

Forage and straw samples were extracted at least 4× with methanol, with the extracted and unextracted radioactivity being determined by LSC. The extracted radioactivity was characterised by partitions into different solvents in turn (cyclohexane, dichloromethane, then ethyl acetate). Further harsher extractions were conducted on the post-extraction solids (refluxing with 1:1 v/v methanol/water for 2 hours, and for straw samples, an additional reflux step with water for 1.5 hours was carried out).

The grain sample were extracted using the same techniques as straw, with additional work-up procedures being required for the aqueous phase of the intial solvent extract after liquid-liquid partition, and the methanol/water reflux extract on the PES both containing significant amounts of starch. This involved first digesting the starch by incubation with α-amylase, then breaking down the resultant sugars to water and carbon dioxide by incubation with baker's yeast. After digestion of the starch, the liquid phases were cleaned up using a cation exchange column before analysis.

The extracts and post-extraction solids were analysed using LSC, TLC, ion chromatography, and HPLC with UV, radio and MS detection, with reference standard of parent, betaine, choline and lecithine being used to aid in identification of residue components.

Further characterisation of the unextracted straw residues was carried out. Radioactivity incorporated into protein was extracted from the PES by stirring with dilute NaOH, with the extracted protein being precipitated by acidification, separated by centrifuging, and redissolution of the precipitate in dilute NaOH. An extraction of a separate fraction of PES for characterisation of radioactivity incorporated as lignin was carried out, by soaking with concentrated sulfuric acid, which was then diluted with water, and the precipitate filtered and washed. Radioactive residues incorporated as cellulose were characterised by extraction with Schweizers reagent ($Cu(OH)_2$ and NH_3), with the precipitated residue being separated and analysed by combustion and LSC.

Unextracted residues in grain were characterised as starch by extraction with DMSO/water, followed by precipitated and washing of the starch with ethanol. The starch was further characterised by amylase and yeast treatment, and by acid hydrolysis and osazone formation, with recrystallization. Radioactivity incorporated into grain as protein, lignin and cellulose was extracted and characterised in a similar manner to that in straw.

Table 5 Total radioactive residues of ^{14}C-chlormequat chloride in spring wheat matrices

Matrix	TRR (mg eq/kg)
0-day forage	49.24
28-day forage	41.98
84-day forage	14.36
Straw	45.84

Matrix	TRR (mg eq/kg)
Grain	1.32

Table 6 Extraction and characterization of the radioactive residues in spring wheat

Component	Residues									
	0-day forage		28-day forage		84-day forage		Straw		Grain	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Methanol-extracted residues	89.6	44.16	84.8	35.6	79.0	11.33	81.0	37.12	36.9	0.49
PES	4.4	2.18	8.7	3.65	9.9	1.42	11.6	5.32	-	-
Methanol/ water reflux	0.8	0.41	3.2	1.34	6.0	0.86	5.0	2.28	16.8	0.22
Water reflux	-	-	-	-	-	-	2.8	1.28	-	-
Starch	-	-	-	-	-	-	-	-	15.8	0.21
Protein	-	-	-	-	-	-	0.0	0.004	0.2	< 0.01
Lignin	-	-	-	-	-	-	5.1	2.34	35.6	0.47
Cellulose	-	-	-	-	-	-	0.1	0.03	1.2	0.02
Unextracted	0.1	0.04	0.1	0.05	0.5	0.07	nd	nd	nd	nd
Mass accountability	90.5	44.6	88.1	37.0	85.5	12.4	94.0	43.1	106.5	1.41

'-' indicates extraction not performed

Residues were readily extractable from forage and straw using methanol (79.0-89.6% TRR extracted from forage and 81.0% TRR from straw). Extractability from grain was lower, with 36.9% TRR extracted using methanol, together with a further 16.8% released using a methanol/water reflux. A significant proportion of the radioactivity in grain had been incorporated into biomolecules, with 15.8% TRR present as starch, and 35.6% TRR present as lignin. A smaller proportion of the residue in straw (5.1% TRR) had been incorporated into lignin. Incorporation into protein or cellulose was not significant in either straw or grain.

Table **7** Identification of residues in spring wheat [a]

Component	Residues									
	0-day forage		28-day forage		84 day forage		Straw		Grain	
	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg	%TRR	mg eq/kg
Chlormequat chloride	80.9-86.0	39.9-42.4	76.1-79.5	31.9-33.4	67.2-73.3	9.66-10.5	77.7-81.4	35.6-37.3	27.9-30.1	0.37-0.41
Betaine	nd	nd	nd	nd	nd	nd	0.1	0.06	2.9-4.7	0.037-0.054
Unidentified components (sum)[b]	< 0.1-4.8	0.01-2.44	< 0.01-3.4	0.014-1.47	0.1-6.2	0.02-0.88	< 0.1-3.8	0.002-1.77	0.5-1.5	0.005-0.026

[a] Slightly different results for each component were obtained from different HPLC methods.

[b] 3 components, characterised only by retention times.

Parent compound was the largest individual identified component in all matrices, at 9.7–42.4 mg eq/kg (67–86% TRR) in forage, 35.6–37.3 mg eq/kg in straw (78–81% TRR), and 0.37–0.41 mg eq/kg (27.9–30.1% TRR) in grain. Small amounts of betaine were identified in grain (up to 0.054 mg eq/kg, 4.7% TRR), and straw (0.06 mg eq/kg, 0.1% TRR), with other unidentified components at up to 2.4 mg eq/kg (6.2% TRR) in forage, up to 1.8 mg eq/kg (3.8% TRR) in straw, and up to 0.026 mg eq/kg (1.5% TRR) in grain.

Chlormequat chloride is not metabolised to a significant extent in wheat. The major metabolic fate of chlormequat chloride is incorporation into biomolecules, principally lignin and starch, with a small amount of betaine being found in grain and straw.

Further plant metabolism information

A number of other plant metabolism studies submitted to the 1994 JMPR were re-submitted to the current Meeting where the studies were available. This information was mostly non-contemporary data (some dating back to the 1960s) and largely from published papers rather than supervised trials. This information is considered below.

In experiments on potted wheat and barley plants to study the uptake, decomposition and translocation of [^{14}C]methyl- or [^{14}C]ethyl-labelled chlormequat in wheat Schilling and Bergmann (1971) found that within four weeks after leaf-application, only 10% of the chlormequat absorbed was metabolized. In the wheat plants acropetal transport of chlormequat was predominant, while in barley chlormequat was transported in a basipetal direction.

Studies of the metabolism of chlormequat in higher plants have produced varying results. Bohring (1972), Blinn (1967), Birecka (1967), Bier and Dedek (1970) and Bettner (1974) found negligible amounts of labelled metabolites in studies with ^{14}C- or ^{15}N-labelled chlormequat. The formation of choline in particular is ruled out by some of the above authors.

The capacity of vegetable plants to metabolize chlormequat was also found to be insignificant by Müller and Schuphan (1975), with the conversion rates being 1–6% in kohlrabi, 1–4% in cauliflower,and 1–2% in tomatoes.

The metabolism of [*methyl*-^{14}C]chlormequat during the reproductive stage was studied by Bohring (1982) in pot experiments with spring wheat. The persistence of ^{14}C-labelled chlormequat in wheat kernels was also examined during a period of one year. The following results were found after spray treatment at late growth stages (tillering, ear emergence). The mobility of chlormequat in the plant was very low. Even when it was applied at the beginning of ear emergence, 98% of the applied ^{14}C remained in the shoots and only 1–2% was translocated towards the ears. Chlormequat was very stable in the plants. By far the main proportion of the applied ^{14}C was recovered as chlormequat and only 2–5% was found in the choline fraction. The radioactivity in the other chemical fractions was extremely low or zero. In the kernels the ^{14}C activity in the choline fraction amounted to 12% of the total ^{14}C and thus was twice as high as in the straw. This relatively high level of ^{14}C in the choline fraction may be related to metabolic processes typical of grain growth. It is also possible that choline synthesized in the leaves is more easily translocated than chlormequat towards the kernels. Mature kernels stored at room temperature did not show any metabolism of chlormequat during a period of one year. Neither the total ^{14}C activity nor the content of chlormequat changed significantly during this time.

Other authors showed that metabolism was extensive. Jung and El Fouly (1966) showed that the active ingredient was quickly converted to choline in aqueous extracts of many plants. It is noted that this data relates to plant extracts, not whole plants.

Stephan and Schütte (1970) studied the metabolism of methyl-labelled chlormequat chloride in barley, wheat, tobacco and maize. Ten to 20% of the applied radioactivity was located in the choline fraction, and a small proportion was found in the betaine fraction. Degradation to $^{14}CO_2$ was observed to only a small extent.

Dekhuijzen and Vonk (1974) determined the distribution and degradation of chlormequat as 2- chloro[1,2-^{14}C]ethyltrimethylammonium chloride after uptake by the roots of summer wheat seedlings. The compound was completely translocated from the roots to the parts above and converted into choline. Choline was further metabolized to betaine which upon demethylation yielded finally glycine and serine. Both amino acids were incorporated into a protein fraction (see Figure 1). The occurrence of radio-labelled glycine and serine in the amino acid pool and the evolution of $^{14}CO_2$ from chlormequattreated plants indicated that serine was formed from glycine with the release of $^{14}CO_2$ during photorespiration. One week after the uptake period 82% of the [^{14}C]chlormequat taken up by the roots was recovered as the parent compound or as breakdown products in the wheat plants, and a further 5% was released as $^{14}CO_2$ by the leaves. Fifty percent of the chlormequat originally present in the wheat plant was metabolized after 7½ days.

Confined rotational crop studies

The Meeting received two confined crop rotation metabolism studies that had not previously been considered by JMPR.

A study was conducted to investigate the metabolism of chlormequat-chloride in the representative rotational crops spring wheat (variety: *Star*), lettuce (varieties: *Sprinter* and *Nadine*) and white radish (variety: *April Cross*) from three different plant-back intervals, after spraying ^{14}C-chlormequat-chloride formulated as an SL 300 onto bare soil in plastic containers (0.20 m^2) (Veit 2003, 2003/1004686). The actual application rate corresponded to 2000 g ai/ha. The crops were each sown at 30, 120, and 365 days after the soil application, representing the first, second and third rotation. A second application was made for the plant back interval of 30 days and with lettuce as the rotational crop (treament group 2), as the 30 day lettuce yield from the first test was significantly reduced due to a fungal disease. Raw agricultural commodities (RAC) sampled included the immature samples of forage from wheat, while all other samples (wheat straw, chaff and grain, lettuce, white radish leaves and roots) were harvested in each rotation at maturity. In addition, soil samples were taken after application, after ploughing and after harvest of mature crops.

An aliquot of each homogenised RAC was extracted with methanol and then water. The methanol extractable radioactive residues and in some cases the water extractable radioactive residues were analysed by HPLC. The residual radioactive residues after extraction were characterised by sequential solubilisation procedures with alkaline solutions and/or incubations with various glycoside-cleaving enzymes. Some of the solubilisates obtained by treatment with aqueous ammonia or by enzymatic digestion were also analysed by HPLC.

The TRRs in the crops from the 3 different rotations were determined by direct combustion and by calculation of the extractable and non-extractable residues and are summarised below (Table 8). Residue levels in lettuce leaf were low for all plant back intervals (0.011–0.021 mg eq/kg). For white radish root and radish leaf, the highest residue levels were detected after a plant back interval of 30 days (≤ 0.046 mg eq/kg). Residues in both matrices had decreased to ≤ 0.005 mg eq/kg after a plant back interval of 365 days. In the wheat matrices forage, straw and chaff the highest residue levels were detected after 30 DAT (0.153, 0.336 and 0.229 mg eq/kg respectively). The residue levels in wheat grain were 0.170 mg eq/kg after 30 DAT and 0.197 mg eq/kg after 120 DAT. Residues in all wheat matrices decreased to ≤ 0.027 mg eq/kg after a plant back interval of 365 days.

Table 8 Total Radioactive Residue (TRR) levels in rotational crops after ^{14}C-chlormequat-chloride treatment and plant back intervals of 30, 120 and 365 days

Crop Parts Days After Sowing /Planting (DAP)	TRR Determined by Direct Combustion (mg eq/kg)	TRR Calculated[a] (mg eq/kg)	Recovery[b] (% TRR)
Plant back interval: 30 DAT			
Lettuce leaf	0.011	0.012	109.1
White radish root 76	0.046	0.046	100
White radish leaf 76	0.043	0.037	86.0
Wheat forage 55	0.164	0.153	93.3
Wheat straw 157	0.359	0.336	93.6
Wheat chaff 157	0.242	0.229	94.6
Wheat grain 157	0.171	0.170	99.4
Plant back interval: 120 DAT			
Lettuce leaf 55	0.018	0.021	116.7
White radish root 86	0.015	0.015	100.0
White radish leaf 86	0.017	0.021	123.5
Wheat forage 83	0.036	0.041	113.9
Wheat straw 169	0.136	0.135	99.3
Wheat chaff 169	0.176	0.172	97.7
Wheat grain 169	0.186	0.197	105.9
Plant back interval: 365 DAT			
Lettuce leaf 52	0.008	0.011	137.5
White radish root 77	0.003	0.004	133.3
White radish leaf 77	0.004	0.005	125.0

Crop Parts Days After Sowing /Planting (DAP)	TRR Determined by Direct Combustion (mg eq/kg)	TRR Calculated[a] (mg eq/kg)	Recovery[b] (% TRR)
Wheat forage 62	0.011	0.010	90.9
Wheat straw 127	0.024	0.025	104.2
Wheat chaff 127	0.028	0.027	96.4
Wheat grain 127	0.022	0.020	90.9

DAT Days After Treatment

[a] TRR was calculated as the sum of ERR + RRR

[b] Recovery=TRR calculated × 100% / TRR combustion

Soil samples were combusted for determination of the radioactive residues. The initial values of 24.0 and 19.9 mg eq/kg (treatment groups 1 and 2) decreased after aging and ploughing to values of 0.508/0.294 mg eq/kg after 30 DAT, to a level of 0.307 mg eq/kg after 120 DAT and to a level of 0.257 mg eq/kg after 365 DAT. The residue levels in the soil were only slightly lower after harvest of the ripe crops (30 DAT: 0.195–0.444 mg eq/kg; 120 DAT: 0.195–0.372 mg eq/kg; 365 DAT 0.160–0.257 mg eq/kg).

Table 9 Total radioactive residues in soil samples after ^{14}C-chlormequat-chloride treatment and plant back intervals of 30, 120 and 365 days

Soil Samples	TRR Determined by Direct Combustion (mg/kg)
After Application	
0 DAT (treatment group 1)	24.0
0 DAT (treatment group 2)	19.9
Plant back interval: 30 DAT	
After ploughing	
30 DAT (treatment group 1)	0.508
30 DAT (treatment group 2)	0.294
After harvest of ripe crops	
Lettuce	0.195
White Radish	0.266
Wheat	0.444
Plant back interval: 120 DAT	
After ploughing	
120 DAT	0.307
After harvest of ripe crops	
Lettuce	0.243
White Radish	0.372
Wheat	0.195
Plant back interval: 365 DAT	
After ploughing	
365 DAT	0.257
After harvest of ripe crops	
Lettuce	0.164
White radish	0.160
Wheat	0.257

DAT Days After Treatment

Extractability of radioactive residues from all commodities of all rotations of lettuce leaf, radish root and leaf and wheat forage ranged from 37.9–68.1% TRR. The extractability was lower in the dry matrices wheat straw and chaff (21.7–35.5% TRR) and in wheat grain (11.6–20.1% TRR).

Table 10 Extractability of radioactivity in rotational crops after ^{14}C-chlormequat-chloride treatment and plant back intervals of 30, 120 and 365 days

Crop Parts Days After Sowing /Planting (DAP)	TRR[a] (mg eq/kg)	MeOH		H_2O		ERR[b]		RRR[c]	
		mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR	mg eq/kg	% TRR
Plant back interval: 30 DAT									
Lettuce leaf	0.012	0.006	46.5	0.002	12.8	0.008	59.3	0.005	40.7
White radish root	0.046	0.017	37.5	0.004	8.4	0.021	45.9	0.025	54.0
White radish leaf	0.037	0.012	32.2	0.006	17.2	0.018	49.4	0.019	50.6
Wheat forage	0.153	0.056	36.4	0.006	3.9	0.062	40.3	0.092	59.7
Wheat straw	0.336	0.066	19.6	0.014	4.3	0.080	23.9	0.256	76.1
Wheat chaff	0.229	0.052	22.8	0.012	5.3	0.064	28.1	0.165	71.9
Wheat grain	0.170	0.024	13.8	0.011	6.3	0.035	20.1	0.136	79.8
Plant back interval: 120 DAT									
Lettuce leaf	0.021	0.008	36.2	0.003	12.9	0.011	49.1	0.011	50.9
White radish root	0.015	0.009	57.0	0.002	11.1	0.011	68.1	0.005	31.9
White radish leaf	0.021	0.007	35.4	0.005	24.2	0.012	59.6	0.009	40.4
Wheat forage	0.041	0.011	28.1	0.004	9.8	0.015	37.9	0.025	62.2
Wheat straw	0.135	0.019	14.3	0.011	8.1	0.030	22.4	0.105	77.7
Wheat chaff	0.172	0.022	12.7	0.015	9.0	0.037	21.7	0.135	78.3
Wheat grain	0.197	0.012	6.0	0.011	5.6	0.023	11.6	0.174	88.4
	Plant back interval: 365 DAT								
Lettuce leaf	0.011	0.003	30.7	0.001	14.0	0.004	44.7	0.006	55.2
White radish root	0.004	0.002	47.1	0.001	19.0	0.003	66.1	0.001	33.9
White radish leaf	0.005	0.001	22.6	0.001	23.2	0.002	45.8	0.003	54.1
Wheat forage	0.010	0.003	25.7	0.001	13.8	0.004	39.5	0.006	60.5
Wheat straw	0.025	0.005	21.6	0.004	13.9	0.009	35.5	0.016	64.5
Wheat chaff	0.027	0.006	22.0	0.002	8.7	0.008	30.7	0.019	69.2
Wheat grain	0.020	0.002	9.6	0.001	3.8	0.003	13.4	0.017	86.6

DAT Days After Treatment

[a] TRR was calculated as the sum of ERR + RRR

[b] ERR=Extractable Radioactive Residues

[c] RRR=Residual Radioactive Residues

Chlormequat-chloride was converted to mainly polar degradation products and at longer plant back intervals was no longer detected or only in minor portions. Considerable amounts of residual radioactive residues of radish root and leaf and wheat straw, chaff and grain were detected, after plant back intervals of 30 and 120 days. These residues were shown to contain essentially the same components as were detected in the extracted radioactive residues. The residual radioactive residues therefore mainly consisted of polar degradation products and parent compound in association with insoluble plant polymers. The distribution of parent and fractions in the RACs for each rotation are summarised below in Table 11.

Table 11 Summary of major components in rotational crops after ^{14}C-chlormequat-chloride treatment and plant back intervals of 30, 120 and 365 days

Crop Parts	TRR (mg/kg)	MeOH/H_2O mg eq/kg (%TRR)	RRR mg eq/kg (%TRR)	Parent mg/kg (%TRR)	Degradation products mg eq/kg (%TRR)
Plant back interval: 30 DAT					
Lettuce leaf	0.012	0.006 (46.5)	0.005 (40.7)	n.d.	Polar fraction[b]: (1 peak) 0.006 (46.5)
Radish root	0.046	0.017 (37.5)	0.025 (54.0)	0.009 (18.8)	Polar fraction: (1 peak) 0.008 (18.7)
Radish leaf	0.037	0.012 (32.2)	0.019 (50.6)	0.008 (20.4)	Polar fraction: (1 peak) 0.004 (11.8)
Wheat forage	0.153	0.056 (36.4)	0.092 (59.7)	0.049 (31.7)	Polar fraction: (1 peak) 0.007 (4.7)

Crop Parts	TRR (mg/kg)	MeOH/H_2O mg eq/kg (%TRR)	RRR mg eq/kg (%TRR)	Parent mg/kg (%TRR)	Degradation products mg eq/kg (%TRR)
Wheat straw	0.336	0.066 (19.6)	0.256 (76.1)	0.060 (17.7)	Polar fraction: (1 peak) 0.006 (1.9)
		H_2O 0.014 (4.3)		0.012 (3.8)	Polar fraction: (1 peak) 0.002 (0.5)
Wheat chaff	0.229	0.052 (22.8)	0.165 (71.9)	0.048 (21.1)	Polar fraction: (1 peak) 0.004 (1.7)
		H_2O 0.012 (5.3)		0.010 (4.3)	Polar fraction: (1 peak) 0.002 (1.0)
Wheat grain	0.170	0.024 (13.8)	0.136 (79.8)	0.015 (8.8)	Polar fraction: (1 peak) 0.009 (5.0)
Plant back interval: 120 DAT					
Lettuce leaf[1]	0.021	0.006[a] (28.5)	0.011 (50.9)	n.d.	Polar fraction: (1 peak) 0.006 (28.5)
Radish root	0.015	0.007[a] (46.3)	0.005 (31.9)	n.d.	Polar fraction: (1 peak) 0.007 (46.3)
Radish leaf	0.021	0.007 (35.4)	0.009 (40.4)	n.d.	Polar fraction: (1 peak) 0.007 (35.4)
Wheat forage	0.041	0.011 (28.1)	0.025 (62.2)	0.004 (9.2)	Polar fraction: (1 peak) 0.007 (18.9)
Wheat straw	0.135	0.019 (14.3)	0.105 (77.7)	n.d.	Polar fraction: (2 peaks) ≤0.011 (≤8.3)
Wheat chaff	0.172	0.022 (12.7)	0.135 (78.3)	n.d.	Polar fraction: (1 peak) 0.022 (12.7)
Wheat grain	0.197	0.012 (6.0)	0.174 (88.4)	0.005 (2.3)	Polar fraction: (1 peak) 0.007 (3.7)
		H_2O 0.011 (5.6)		0.004 (1.8)	Polar fraction: (2 peaks) ≤0.005 (≤2.5)
Plant back interval: 365 DAT					
Lettuce leaf	0.011	0.003 (30.7)	0.006 (55.2)	n.d.	Polar fraction: (1 peak) 0.003 (30.7)
Wheat grain	0.020	0.002 (9.6)	0.017 (86.6)	n.d.	Polar fraction: (1 peak) 0.002 (9.6)

[a] Value for MeOH conc.

[b] Polar fraction (t_R=approximately 4 min)

n.d. not determined

Storage stability investigations with the stored methanol extract and rework up of lettuce leaf and radish root 120 DAT samples demonstrated that, within a storage time of approximately 2.6 years, no changes in the metabolic pattern could be observed between the first analysis of the extract, the re-analysis of the extract after storage and the new extract after rework up. The polar fraction was the only detectable component. The radioactive residues in lettuce leaf and radish root were stable under the chosen conditions.

In another study (Hofmann 1992, 92/10223) the metabolism of chlormequat-chloride was investigated in the representative rotational crops spring wheat (variety: *Star*), green beans (variety: *Marona*), carrots (variety: *Tip-Top*) and head lettuce (variety: *Debby*) using ^{14}C-chlormequat-chloride dissolved in water and added to 10 kg of loamy sand soil, giving an actual application rate corresponding to 1.51 kg ai/ha. The soil was homogenised and then stored in a 30 litre drum with a loosely attached lid. After 30 days the soil was diluted using untreated soil (ratio 1:9) to simulate ploughing. The crops were each planted/sown at 30 days after the soil application. Beans, carrots and lettuce were cultivated in a greenhouse while spring wheat was grown in a phytotron with fluorescent lamps. Samples of soil and/or plant were taken at different stages of the study (day of treatment, planting of rotational crop, as soon as sufficient plant material was available, earliest possible

utilisation, normal harvesting of the rotational crop) and total radioactive residues were determined by LSC.

Concentrations of total residue in the edible parts of the four crops ranged from 0.003 mg eq/kg in beans at 92 DAT to 0.052 mg eq/kg in wheat grain at 210 DAT, but were < 0.01 mg eq/kg for lettuce heads and carrot roots. The concentrations of test compound in the soil immediately after treatment was 5.27 mg/kg. At the time of planting/seeding, the concentration of ^{14}C-chlormequat-chloride in soil accounted for 0.22 mg eq/kg. After harvest of beans, carrot and lettuce, the residues in soil decreased to levels of about 0.067–0.131 mg eq/kg whereas after harvest of wheat grain, only 0.030 mg eq/kg were observed, consistent with the higher residues observed in wheat grain compared to other crops.

The distribution of radioactivity in wheat, beans, lettuce and carrots grown on soil treated with ^{14}C-chlormequat-chloride is summarised in Table 12.

Table 12 Summary of the distribution of residues in rotational crops planted/sowed on soil 30 days after treatment with^{14}C-chlormequat chloride

Crop		Growing Period (days) (start 30 DAT)	TRR (mg eq/kg)
Wheat	Plant	75	0.041
	Grain	210	0.052
	Spelt	210	0.061
	Straw	210	0.066
	Root	210	0.051
	Soil	210	0.030
Beans	Plant	49	0.021
	(Green matter)	110	0.019
	Beans	92	0.003
		110	0.010
	Root	110	0.114
	Soil	110	0.090
Carrots	Plant	42	0.039
	Leaves	92	0.016
		131	0.018
	Root	92	0.005
		131	0.006
	Soil	131	0.067
Lettuce	Plant	41	0.022
	Leaves	92	0.009
	Root	92	0.070
	Soil	92	0.131

Animal metabolism

Metabolism in the rat

Evaluation of the metabolism studies in rodents was carried out by the WHO Core Assessment Group.

In data for rats given chlormequat chloride via the oral or intravenous route, chlormequat chloride was not metabolised to any significant extent. Other than parent compound, only a single unidentified polar component was found after intravenous administration, while for oral administration, only parent and two other components tentatively identified as salts of chlorocholine (chlormequat) were found (choline itself was not identified). Chlormequat is not metabolised to a significant extent in rats.

Lactating goats

A contemporary study on the metabolism of chlormequat-chloride in lactating goats was conducted with the test compound labelled in both positions of the chloroethyl group. Two lactating goats (breed not specified, 3 years, weight 59.5–68.0 kg on arrival) were orally dosed twice daily for seven

consecutive days at a dose level of 25 ppm in the diet (equivalent to 62.5 mg ai/day and 0.96 mg ai/kg bw/days) (Phillips, McCombe and Gedik 2003a, 2003/1012830 and the amendment study Phillips, McCombe and Gedik 2004, 2004/1020717).

Urine and faeces were collected prior to the first dose and urine, faeces and cagewashes at intervals of 24 hours thereafter until the final dosing. The goats were milked twice daily immediately prior to the morning and afternoon dosing. Each animal was sacrified approximately 23 hours after the last administration. Samples of kidney, liver, omental fat, renal fat, muscle (hind and forequarter), gastronintestinal tract and content were harvested and analysed. Total radioactive residues (TRR) were measured in all samples of excreta, cage wash, milk and edible tissues. TRR values determined in various fractions of milk and edible tissues are shown below (Table 13).

Table 13 Total Radioactive Residue (TRR) levels and their distribution in lactating goats after administration of [^{14}C]-chlormequat-chloride

Matrix	25 ppm in the diet	
	Mean TRR (mg eq/kg)	Mean recovery of TRR (% of administered dose)
Milk	n.a.	0.56[a]
Urine	n.a.	49[1]
Faeces	n.a.	30[1]
Kidney	1.5	0.05
Liver	0.36	0.08
Skeletal muscle	0.23	n.i.
Renal fat	0.022	n.i.
Omental fat	0.008	n.i.
Gastrointestinal tract content	n.i.	1.6

n.i.=not indicated

n.a.=not applicable

[a] Cumulative excretion

Urinary excretion was the major route of elimination, accounting for 49% of the administered dose, followed by faecal excretion (30%). In milk, the radioactivity accounted for a mean of 0.56% of the total administered dose. The plateau level of the total radioactive concentration in milk was reached 104h after administration of the first dose, resulting in a concentration of 0.26 mg eq/kg, which declined to 0.12 mg eq/kg after the final dosing. The highest tissue concentration was measured in kidney (1.5 mg eq/kg, recovery 0.05%).

Radioactivity was extracted from composite samples of faeces (48h and 175h), liver, kidney, renal fat, skeletal muscle and milk (56h and 144h) with solvent, and where appropriate, additional enzymatic and hydrolytic methods. The extraction, characterisation and identification of residues in milk and edible tissues of two lactating goats following oral administration of [^{14}C]-chlormequat-chloride twice daily for 7 consecutive days are summarised in Table 14.

Table 14 Extraction, characterization and identification of the residues in milk and edible tissues of two lactating goats following oral administration of [^{14}C]-chlormequat-chloride twice daily for 7 consecutive days (mean values)

	Kidney		Liver		Muscle		Fat (renal)		Milk (56h)		Milk (144h)	
TRR [mg eq/kg]	1.5		0.36		0.23		0.022		0.24		0.20	
	%	mg eq/kg	%	mg eq/kg	%	mg eq/kg	%	mg eq/kg	%	mg eq/kg	%	mg eq/kg
Water soluble (initial extract)[a]	92	1.3	77	0.27	90	0.21	67	0.015	17	0.041	20	0.039
Water soluble (processed)[b]	90	1.3	52	0.19	90	0.21	62	0.014	16	0.038	17	0.034
Post-extraction solids (PES)	8.0	0.12	23	0.084	10	0.024	34	0.007	83	0.20	80	0.16
Pepsin extract	7.3	0.11	15	0.054	7.7	0.018	-	-	63	0.15	80	0.16
Protease extract	-	-	1.5	0.005	-	-	-	-	16	0.039	-	-

Strong acid hydrolysis (6M HCl)	-	-	6.9	0.025	-	-	-	-	-	-	-	-
Identified[c] Chlormequat-chloride	83	1.2	42	0.15	76	0.18	-	-	4.4	0.011	1.1	0.002[d]
Characterised Total amount per fraction (mg eq/kg) [number of fractions]	< 0.01 [2] 0.01-0.05 [2] >0.01 [2]		< 0.01 [2] 0.01-0.05 [4]		< 0.01 [4] 0.01-0.05 [2]		< 0.05 [1]		< 0.01 [1] 0.01-0.05 [4] >0.05 [1]		< 0.01 [4] 0.01-0.05 [3] >0.05 [1]	

[a] Tissues were extracted with methanol, milk was extracted with acetonitrile

[b] Initial tissue extracts were partitioned with hexane, milk extracts with diethyl ether/hexane (1:1)

[c] Chromatographic analysis performed on the water soluble fractions (water soluble fractions after enzyme hydrolysis are included as well)

[d] Assigned as chlormequat-chloride because of the similar retention time

For analysis of parent compound and any metabolites, milk was initially extracted with acetonitrile recovering 17 and 20% from the 56h and 144h samples respectively. Following further purification and processing of the combined extracts, the final overall extraction efficiencies were 16% and 17% TRR respectively. Pepsin hydrolysis of the post-extracted solid (PES) released 63% TRR and 80% TRR from the 56h and 144h PES samples, respectively. Protease released a further 16% TRR from the 56h sample.

Initial extraction of kidney, liver, muscle and renal fat with methanol recovered 92, 77, 90 and 67% TRR respectively. Subsequent processing led to some losses of radioactivity such that the processed extracts accounted for 90, 52 and 62% TRR for kidney, liver and fat. Pepsin hydrolysis of the PES released 7.3, 15 and 7.7%% TRR for kidney, liver and muscle respectively. Protease released a further 1.5% TRR for liver, while the processed 6 N HCl hydrolysate released another 6.9% of the TRR.

The only compound identified in milk and edible tissues was chlormequat-chloride. The percentages of total radioactivity present as chlormequat-chloride in kidney, liver and muscle extracts were 83, 42 and 76% TRR respectively (1.2, 0.15 and 0.18 mg/kg). Chlormequat-chloride accounted for <5% TRR in the 56h and 144h milk samples (0.011 and 0.002 mg/kg respectively). The substantial portions of the radioactivity extracted by protease and pepsin digestions (0.25 mg eq/kg for kidneys, 0.21 mg eq/kg for liver and 0.056 mg eq/kg for muscle) indicate that a proportion of the residue is present as macromolecules, formed by incorporation of chlormequat-chloride *via* biosynthetic pathways.

Laying hens

A study on the metabolism of chlormequat-chloride in laying hens was conducted with the test compound labelled in both positions of the chloroethyl group (Phillips, McCombe and Gedik 2003b, 2003/1012836).

Ten hens (breed and age not specified, 1.74–2.12 kg mean body weight throughout the study) were dosed orally once daily in the morning for 14 consecutive days with gelatin capsules at 12 ppm in the diet (equivalent to 3 mg ai/day and 1.6 mg ai/kg bw /day). Eggs were collected pre-dose and then twice daily and were separated into egg yolk and white. Excreta was collected prior to the first dose and at 24h intervals thereafter until day 14 of dosing. The hens were sacrificed at approximately 23 hours after the last dose and liver, kidney, muscle (composite breast and thigh) and abdominal fat pad were retained. A composite sample was prepared from the ten hens. Partially formed eggs were retained for each animal and a composite sample prepared.

Total radioactive residues (TRR) were determined daily in the egg yolks and whites and excreta, and at sacrifice in the dissected organs and tissues (kidney, liver, abdominal fat pad, and muscle (composite of breast and thigh), partially formed eggs). TRR values determined in various fractions of eggs and edible tissues are shown below (Table 15).

Table 15 Total Radioactive Residue (TRR) levels and their distribution in laying hens after administration of [^{14}C]-chlormequat-chloride for 14 days at 12 ppm in the diet

Matrix	12 ppm in the diet	
	TRR (mg eq/kg) [plateau level, where available]	Recovery of TRR (% of administered dose)
Egg white	0.042[a] [no plateau level observed]	0.05[b]
Egg yolk	0.59[a] [0.97]	0.34[b]
Kidney	0.35	0.01
Liver	0.36	0.03
Muscle (breast and thigh)	0.12	n.i.
Abdominal fat	0.062	n.i.
Pre-laid eggs	0.89	0.13
Excreta	n.i.	93
Total recovered	-	93.56

n.i.=not indicated

n.a.=not applicable

[a] Average value of all samples taken

[b] Cumulative excretion

Chlormequat–chloride is rapidly and almost quantitatively excreted, with 93% of the administered radioactivity being present in excreta. Egg yolk and egg white accounted for 0.34 and 0.05% of the total administered dose respectively and pre-laid eggs accounted for 0.13% of the total administered dose. The concentration of the composite egg yolk samples increased steadily from < 0.001 mg eq/kg at 24h after the first dosing, with a plateau level of 0.97 mg eq/kg reached at 264 h after the first dosing. Concentrations in egg white were lower with 0.001 mg eq/kg at 24 h after the first dosing, increasing steadily without reaching a plateau level. The maximum concentration (0.057 mg eq/kg) was observed in the last sample (335h after first dosing). In cage washes 1.4% of the total administered radioactivity was found. In the tissue samples the highest concentrations were measured in liver and kidney (0.36 and 0.35 mg eq/kg respectively), while 0.12 and 0.062 mg eq/kg were detected in muscle and fat respectively.

The extraction, characterisation and identification of residues in eggs and edible tissues of laying hens following oral administration of [^{14}C]-chlormequat-chloride twice daily for 14 consecutive days are summarised in Table 16.

Table 16 Extraction, characterization and identification of the residues in hen matrices following oral administration of [^{14}C]-chlormequat-chloride

	Liver		Kidney		Muscle		Fat	
TRR [mg eq/kg]	0.36		0.35		0.12		0.062	
	%	mg eq/ kg	%	mg eq/ kg	%	mg eq/ kg	%	mg eq/ kg
Water soluble (initial extract)[a] Water soluble (processed)[b]	66 53	0.23 0.19	65 55	0.23 0.19	75 71	0.093 0.087	15 13	0.009 0.008
Post extraction solids (PES)[c]	34	0.12	35	0.12	25	0.03	85	0.053
Pepsin extract	23	0.082	26	0.092	11	0.013	5.3	0.003
Protease extract	2.9	0.01	2.5	0.008	4.1	0.005	0.3	< 0.001
Acid reflux	2.1	0.007	0.9	0.003	1.3	0.002	0.2	< 0.001
Unextracted residue (after exhaustive extraction)	6.3	0.022	5.7	0.020	8.4	0.010	65	0.04
Identified[e] Chlormequat-chloride	1.8	0.007	6.5	0.023	-	-	-	-
Characterised[e] Total amount per fraction	< 0.01 [5] 0.01-0.05 [3]		< 0.01 [2] 0.01-0.05 [4]		< 0.01 [1] 0.01-0.05 [2]		< 0.01 [3]	

	Liver		Kidney		Muscle		Fat	
(mg eq/kg) [number of fractions]	>0.01 [1]		>0.05 [2]		>0.05 [1]			
	Egg White				Egg yolk			
	96h		264h		96h		264h	
TRR [mg eq/kg]	0.037		0.051		0.30		0.97	
	%	mg eq/ kg	%	mg eq/ kg	%	mg eq/ kg	%	mg eq/ kg
Water soluble (initial extract)[a] Water soluble (processed)[b]	5.7 -	0.002 -	5.5 -	0.003 -	69 57	0.20 0.17	62 50	0.60 0.48
Post extraction solids (PES)[c]	94	0.035	95	0.048	31	0.093	38	0.37
Pepsin extract	85	0.032	87	0.044	9.7	0.029	11	0.103
Protease extract	-	-	-	-	1.6	0.005	2.3	0.022
Acid reflux	-	-	-	-	3.8	0.011	3.6	0.035
Unextracted residue (after exhaustive extraction)	8.9	0.003	7.8	0.004	16	0.048	22	0.21
Identified[e] Chlormequat-chloride	-	-	-	-	-	-	48	0.47
Characterized [e] Total amount per fraction (mg eq/kg) [number of fractions]	< 0.01 [7]		< 0.01 [3] 0.01-0.05 [2]					

[a] Tissues were extracted with methanol

[b] Initial tissue extracts were partitioned with hexane and the resulting layer was concentrated under nitrogen

[c] Remained after initial extraction

[d] After further extraction with hexane, additional 14% TRR (0.009 mg eq/kg) released

water soluble

[e] Sum of amounts found in the solvent extract and the exhaustive extractions

Radioactivity was extracted from pooled samples of excreta (day 1 and day 14), liver, kidney, composite breast and thigh muscle, abdominal fat, egg yolk (day 4 and day 11) and egg white (day 4 and day 11) with solvent. Where appropriate, addditional enzymatic (liver, kidney, muscle, abdominal fat, egg yolk and white) and acid reflux methods (all except egg white) were also employed.

In liver, kidney, muscle and egg yolk the majority of the radioactive residue was recovered in the methanol extract (water-soluble fraction) (66, 65, 75, and 62% respectively) while proteolytic enzyme hydrolysis released a further substantial part of the radioactivity.

In egg white, pepsin enzyme hydrolysis significantly released radioactive residue of the not extracted fraction (85 and 87% TRR for the day 4 and 11 fractions respectively). In fat most of the radioactive residues remained not extracted after solvent extraction, enzyme hydrolysis and acid relux. From the additional processing of sub-samples (lipase treatment, Soxhlet extraction) it was concluded that the not extracted radioactive residues are covalently incorporated into the matrix, most likely in fatty acids, glycerol or similar endogenous components of fat.

Parent chlormequat-chloride was the only compound identified. It was found as a major fraction in kidney, liver and egg yolk (in the samples taken after 264 h but not in the 96 h sample). In the water-soluble extracts of liver, kidney muscle and egg yolk (96 h sample), regions of radioactive residue, accounting for >0.05 mg eq/kg were not identified. The substantial portions of the radioactivity extracted by protease and pepsin digestions (0.33 mg eq/kg for kidneys, 0.35 mg eq/kg for liver and 0.12 mg eq/kg for muscle) indicate that a substantial proportion of the residue was

present as macromolecules, formed by incorporation of chlormequat-chloride *via* biosynthetic pathways.

Environmental fate in soil

The Meeting received information on aerobic soil metabolism, field dissipation, metabolism in aquatic systems and phototransformation in water. Only the aerobic soil metabolism study and the field dissipation study (which was also considered by the 1994 JMPR), which are relevant to the current evaluation, are reported here.

Aerobic Soil Metabolism

The route and rate of degradation of ^{14}C-chlormequat-chloride was studied under laboratory aerobic conditions in three soils (sandy loam, clay loam and loamy sand) from Europe at temperatures of 20 ± 2 °C over a 120-day period (Adam 2006, 2006/1044907). Application rates of radiolabelled chlormequat-chloride to soils were 6.46 mg ai/kg dry soil (equivalent to 1.615 kg ai/ha). Test systems consisted of all glass metabolism flasks maintained in the dark and equipped with 2N sodium hydroxide traps for the collection of CO_2 and ethylene glycol traps for the collection of volatile organic compounds. Duplicate soil samples were taken for extraction and analysis immediately after treatment (day 0) and after 5 hours and 1, 7, 14, 27, 57, and 120 days of incubation.

Soil samples were submitted to solvent extraction carried out in the following sequence: methanol/water (1:1; v/v, once) and water acidified to pH 2 using hydrochloric acid (up to six times) at room temperature followed by Soxhlet extraction using acetonitrile/water (1:1; v/v, except for time 0). The concentrated extracts were quantified by LSC and then analysed by HPLC and 1D-TLC to determine the amounts of the test item and any metabolites. A total balance of radioactivity and the amount of the test item and degradates were established for each sampling interval. In order to investigate the non-extracted residues, the samples from the end of incubation (day 120) were submitted to an additional harsh extraction procedure comprising aqueous acidic extraction under reflux conditions followed by organic matter fractionation.

Total mean recoveries were 101.5 ± 4.8%, 96.8 ± 2.9% and 97.7 ± 2.9% of the applied radioactivity (AR) for the soils Speyer 5M, Itingen III and Speyer 2.2, respectively.

Immediately after treatment (day 0), virtually all of the applied radioactvity (99.9-101.0% AR) was extracted from the soils Speyer 5M, Itingen III and Speyer 2.2, respectively, while at the end of the study (day 120), only 2.7%, 6.9%, and 16.7% were extracted from these soils. In the soils Speyer 5M and Itingen III, the non-extractable radioactvity reached levels of 14.5–29.3% AR and 31.6–49.5% AR between 5 hours and 27 days of incubation. In soil Speyer 2.2, the non-extracted residues were significantly lower, steadily increasing with time to reach a peak value of 25.7% AR (day 120).

The mineralisation rate reached maximum levels of 81.3% AR in Speyer 5M soil, 52.6% AR in Itingen III soil and 50.4% AR in Speyer 2.2 soil on day 120. Volatile products other than $^{14}CO_2$ were below 0.1% AR.

The parent compound ^{14}C-chlormequat-chloride, represented the only major radioactive fraction detected in the soil extracts. ^{14}C-chlormequat-chloride rapidly degraded in all three soils tested, decreasing from 99.9%, 100.3% and 101.0% AR immediately after treatment in the soils Speyer 5M, Itingen III and Speyer 2.2, respectively, to 7.1%, 19.5%, and 53.0% AR in the corresponding soils after just 27 days of incubation and only 0.9%, 4.4%, and 12.2% AR at the end of incubation (day 120). As described above, mineralisation to CO_2 was the major route of degradation besides formation of bound residues. Apart from the parent compound, four very minor metabolites (M1 to M4, $\leq$ 2.6% AR) were detected in the soil extracts. None of the metabolites co-chromatographed with the reference items choline chloride and acetylcholine chloride.

Table 17 Composition of radioactivity from three European soils after treatment with ^{14}C-chlormequat-chloride and incubation under aerobic conditions

Soil Pattern (Mean % AR)	Incubation time in days							
	0	0.21	1	7	14	27	57	120
Soil I, Speyer 5M, Germany								
Parent	99.9	71.5	77.7	79.2	43.5	7.1	2.3	0.9
M1	-	-	-	-	2.0	2.1	0.8	0.5
M2	-	-	-	-	1.3	1.7	1.3	0.4
M3	-	-	-	-	2.5	2.5	0.5	0.4
M4	-	-	-	-	-	-	0.5	0.5
Non-extracted	0.4	26.1	17.9	14.5	21.0	29.3	21.8	18.8
$^{14}CO_2$	n.p.	< 0.1	0.7	3.3	40.7	60.4	76.3	81.3
Soil II, Itingen III, Switzerland								
Parent	100.3	67.7	45.6	52.1	43.7	19.5	8.6	4.4
M1	-	-	0.7	-	0.7	1.0	1.0	0.7
M2	-	-	-	-	1.0	1.0	0.7	0.6
M3	-	-	-	-	0.8	1.1	0.8	0.7
M4	-	-	-	-	-	-	0.6	0.5
Non-extracted	0.4	31.6	49.5	41.1	39.5	47.2	36.4	35.1
$^{14}CO2$	n.p.	< 0.1	0.5	1.1	14.4	26.3	45.2	52.6
Soil III, Speyer 2.2, Germany								
Parent	101.0	89.8	91.8	91.9	84.7	53.0	34.6	12.2
M1	-	-	0.7	-	0.6	2.0	1.8	1.6
M2	-	-	-	-	-	-	-	0.3
M3	-	-	-	-	0.5	1.9	1.8	1.7
M4	-	-	-	-	-	-	-	0.8
Non-extracted	0.4	7.7	5.3	5.9	6.9	17.2	21.0	25.7
$^{14}CO_2$	n.p.	< 0.1	0.3	2.3	7.8	22.3	35.9	50.4

n.p.: Not performed

'-': Not detected or below limit of quantification

Best fit DT_{50}, DT_{75} and DT_{90} values for various soils are shown in Table 18. Chlormequat-chloride is rapidly degraded in aerobic soils with DT_{50} values in the range of 10.2–36.5 days.

Table 18 Best fit DT_{50} and DT_{90} values for chlormequat-chloride in aerobic soils (20 °C)

SOIL	DT_{50} [days]	DT_{75} [days]	DT_{90} [days]	r^2	Model
Soil I, Speyer 5M, Germany [sandy loam, pH 7.1, OC 1.34%]	11.1	22.2	36.8	0.867	SFO
Soil II, Itingen III, Switzerland [clay loam, pH 7.24, OC 2.50%]	10.2	20.3	33.8	0.889	SFO
Soil III, Speyer 2.2, Germany [loamy sand, pH 6.5, OC 2.33%]	36.5	73.0	121	0.980	SFO

OC=Organic Carbon, SFO=Single Phase First Order

Field dissipation

The degradation of chlormequat in soil was investigated by Keller (1993) with [^{14}C]chlormequat (2-chloro[1,2-^{14}C]ethyltrimethylammonium chloride) in a field experiment with a sandy loam soil and in a greenhouse with a clay soil. The rates of treatment corresponded to 3.4 and 2.7 kg ai/ha respectively. Rapid microbiological degradation occurred in both cases. The applied radioactivity decreased to 88% of the original in loam and 33% in clay after three weeks and to 22% in loam and 33% in clay after six weeks. In both soils 70–98% of the activity was in the top 5 cm layer. Chlormequat was extensively mineralized and CO_2 was the ultimate product of degradation. Other degradation products could not be identified. The DT_{50} depends on several factors including temperature, and is in the range of < 1 to 28 days. DT_{90} periods are less than 100 days.

RESIDUE ANALYSIS

Methods of analysis

Details of analytical methods including validation data were supplied for the determination of chlormequat-chloride in plant and animal matrices, soil and water and are considered satisfactory. A summary of all submitted analytical methods for plants and animals is given in Table 19.

Table 19 Summary of analytical methods developed for plant and animal matrices

Matrix	Analyte	Method No.	Detection system	LOQ	Reference
Plant	Chlormequat-chloride	530/0	HPLC-MS/MS	Cereal forage, cereal grain, cereal straw, apple fruit and maize seed LOQ=0.1 mg/kg for chlormequat-chloride in cereal straw and 0.05 mg/kg in all other matrices Lettuce, lemon, oilseed rape seed, grain and cereal plant LOQ=0.5 mg/kg for chlormequat-chloride in cereal straw and 0.05 mg/kg in all other matrices	Kerl and Mackenroth 2006, 2006/1009664 Richter 2006, 2006/1011404 (ILV of Method 530/0)
Plant	Chlormequat-chloride	146	Gas chromatography	Wheat grain, wheat straw, oat and rye LOQ=0.05 mg/kg for chlormequat chloride in cereal grains and 0.5 mg/kg in wheat straw	Elzner 1979, 1979/10136
Animal	Chlormequat-chloride	397	Ion chromatography	Cow meat, liver, kidney, fat and milk, hen meat, liver, fat and eggs LOQ=0.01 mg/kg for chlormequat-chloride in milk and 0.05 mg/kg for chlormequat-chloride in all other matrices	Weidenauer 1998, 1998/11454
Animal	Chlormequat-chloride	397/0	HPLC-MS/MS	Cow muscle, liver, kidney and fat, milk, eggs, hen muscle and liver LOQ=0.01 mg/kg for chlormequat-chloride in milk and 0.05 mg/kg for chlormequat-chloride in all other matrices Cow muscle and liver, milk and eggs LOQ=0.05 mg/kg for chlormequat-chloride in liver and 0.01 mg/kg for chlormequat-chloride in all other matrices Bovine meat, bovine liver, pork kidney, milk, fat and egg LOQ=0.05 mg/kg for chlormequat-chloride for liver and 0.01 mg/kg for chlormequat-chloride in all other matrices Meat, liver, kidney, milk, fat and egg LOQ=0.05 mg/kg for chlormequat-chloride for liver and 0.01 mg/kg for chlormequat-chloride in all other matrices	Tilting 1999, 1999/10026 Tilting 2004, 2004/1006522 Schulz and Meyer 2007, 2007/1043394 (ILV of Method 397/0) Weber 2010, 2011/1036855 (ILV of Method 397/0)

Plant commodities

BASF Method No. 530/0

Method 530/0 for the determination of chlormequat-chloride in plant matrices by means of high performance liquid chromatography with tandem mass spectrometry (HPLC-MS/MS) was reported by Kerl and Mackenroth in 2006 (2006/1009664).

Chlormequat-chloride is extracted from plant material with water/methanol/hydrochloric acid (65/30/5, v/v/v). After homogenisation, water is added and the extract is macerated. A portion is centrifuged, then a fraction of the supernatant is evaporated to dryness. A SPE cartridge filled with Al_2O_3 is used for clean-up, with elution (three times) using methanol/acetonitrile (10/90, v/v). After the extracts are evaporated to dryness the residue is dissolved in water/formic acid (100/0.1, v/v). The final solution is analysed by HPLC-MS/MS. Two MRM transitions for quantitation of chlormequat-chloride are possible (*m/z* 122/58 or *m/z* 122/63).

The accuracy of the method was assessed on the basis of the determined recovery rates. The materials tested included cereal forage, grain and straw, apple fruit and maize seed. Samples were fortified with chlormequat-chloride at concentrations of 0.1 and 1.0 mg/kg (cereal straw) or 0.05 and 0.5 mg/kg (other matrices).

Mean recoveries per fortification level obtained with the two transitions were all between 70 and 110% and relative standard deviations were less than 20%, except for the mean recovery at 0.05 mg/kg for cereal grain for the 122/63 transition (114%) and the relative standard deviation (RSD) for cereal straw at 1.0 mg/kg (36.3% due to one recovery value of 138%).

The limit of quantitation (LOQ) for chlormequat-chloride, defined as the lowest validated fortification level, was 0.05 mg/kg in all matrices tested except straw (LOQ=0.1 mg/kg).

Method linearity was validated over the range 0.1 to 1.0 mg/L for chlormequat-chloride (r^2 > 0.999).

Table 20 Method recoveries for method 530/0: Chlormequat-chloride in plants

Matrix	Analyte	Transition [m/z]	Fortification Level	No. of Tests	Recoveries		
					Range [%]	Mean ± SD [%]	RSD [%]
Cereal forage	Chlormequat-chloride	122→63	0.05 0.5	5 5	93-114 90-105	107±8.9 98±6.1	8.3 6.2
		Overall		10		103±8.6	8.4
		122→58	0.05 0.5	5 5	96-110 103-110	101±5.9 106±3.3	5.9 3.1
		Overall		10		104±5.1	4.9
Cereal grain	Chlormequat-chloride	122→63	0.05 0.5	5 5	104-122 89-122	114±6.7 103±11.8	5.8 11.4
		Overall		10		109±10.8	9.9
		122→58	0.05 0.5	5 5	93-109 103-108	101±7.0 105±2.3	6.9 2.2
		Overall		10		103±5.3	5.1
Cereal straw	Chlormequat-chloride	122→63	0.1 1.0	5 5	79-101 47-138	88±9.2 90±32.7	10.5 36.3
		Overall		10		89±22.7	25.4
		122→58	0.1 1.0	5 5	98-106 88-109	102±3.6 96±8.7	3.6 9.1
		Overall		10		99±7.0	7.1
Apple fruit	Chlormequat-chloride	122→63	0.05 0.5	5 5	91-109 72-113	103±7.2 90±16.2	7.0 17.9
		Overall		10		97±13.7	14.1
		122→58	0.05 0.5	5 5	101-109 94-105	106±3.3 99±4.2	3.1 4.3
		Overall		10		102±5.2	5.1

Matrix	Analyte	Transition [m/z]	Fortification Level	No. of Tests	Recoveries		
					Range [%]	Mean ± SD [%]	RSD [%]
Maize seed	Chlormequat-chloride	122→63	0.05	5	79-123	99±17.5	17.6
			0.5	5	83-103	90±8.3	9.3
		Overall		10		94±13.8	14.6
		122→58	0.05	5	92-107	101±6.5	6.4
			0.5	5	100-113	109±5.4	4.9
		Overall		10		105±7.0	6.6

An independent laboratory validation was conducted for method 530/0. Samples of lettuce, lemon, oilseed rape seed, grain and cereal plant were fortified with chlormequat-chloride at LOQ fortification levels of 0.05 mg/kg for all matrices except straw (0.50 mg/kg) and at 10×LOQ (Richter 2006, 2006/1011404).

The average recovery rates for all matrices, for both fortification levels, and for both MRM transitions monitored were between 70–110%, with RSD values < 20%. A summary of the independent laboratory validation results is given in Table 21.

Table 21 Method recoveries for method 530/0: Chlormequat-chloride in plants

Matrix	Analyte	Transition [m/z]	Fortification Level	No. of Tests	Recoveries		
					Range [%]	Mean ± SD [%]	RSD [%]
Lettuce	Chlormequat-chloride	122→63	0.05	5	100-114	106±5.3	5.0
			0.5	5	92-99	96±3.0	3.1
		Overall		10		101±6.9	6.8
		122→58	0.05	5	98-105	102±3.1	3.1
			0.5	5	99-104	101±1.9	1.9
		Overall		10		101±2.5	2.5
Lemon	Chlormequat-chloride	122→63	0.05	5	100-109	104±3.5	3.4
			0.5	5	98-106	102±3.3	3.2
		Overall		10		103±3.5	3.4
		122→58	0.05	5	97-105	101±2.9	2.8
			0.5	5	99-107	101±3.2	3.2
		Overall		10		101±2.9	2.8
Oilseed rape seed	Chlormequat-chloride	122→63	0.05	5	106-121	111±5.9	5.3
			0.5	5	94-110	101±5.8	5.7
		Overall		10		106±7.5	7.1
		122→58	0.05	5	102-108	105±2.4	2.3
			0.5	5	106-111	108±2.1	1.9
		Overall		10		107±2.8	2.6
Grain	Chlormequat-chloride	122→63	0.05	5	86-95	90±3.7	4.1
			0.5	5	94-101	98±3.0	3.1
		Overall		10		94±5.1	5.5
		122→58	0.05	5	91-100	97±3.7	3.8
			0.5	5	90-99	95±3.3	3.5
		Overall		10		96±3.5	3.6
Cereal plant	Chlormequat-chloride	122→63	0.05	5	90-106	98±7.3	7.5
			0.5	5	86-110	99±11.7	11.8
		Overall		10		98±9.2	9.3
		122→58	0.05	5	98-106	101±3.3	3.3
			0.5	5	84-107	97±11.9	12.3
		Overall		10		99±8.5	8.6
Straw	Chlormequat-chloride	122→63	0.5	5	89-102	97±4.7	4.9
			5.0	5	85-105	94±8.4	9.0
		Overall		10		95±6.6	6.9
		122→58	0.5	5	89-108	98±7.5	7.6
			5.0	5	86-107	95±8.3	8.7
		Overall		10		97±7.6	7.8

BASF Method No. 146

Method 146 for the determination of chlormequat-chloride in cereal matrices by means of gas chromatography was reported (Elsner, 1979/10136).

Chlormequat-chloride is extracted from plant material with methanol. It is isolated with a cation exchanger, interfering substances are precipitated and the compound is purified chromatographically. It is then converted to N, N-dimethyl-2-(thiophenyl)ethylamine using sodium thiophenolate. This is then determined by gas chromatography.

The accuracy of the method was assessed on the basis of the determined recovery rates. The materials tested were wheat grain and straw, oat and rye. Samples were fortified with chlormequat-chloride at concentrations of 0.05-10.0 mg/kg (wheat grain); 0.5, 0.66 and 1.0 mg/kg (wheat straw); 0.1, 0.5, 1.0 and 2.0 mg/kg (oat) and 0.1, 0.5 and 2.0 mg/kg (rye).

Mean recoveries per fortification level for chlormequat-chloride for all matrices were in a range of 69–106%, with RSD values < 20%.

The LOQ for chlormequat-chloride was 0.05 mg/kg for cereal grain and 0.5 mg/kg for straw.

Table 22 Method recoveries for method 146: Chlormequat-chloride in plants

Matrix	Analyte	Fortification Level	No. of Tests	Recoveries		
				Range [%]	Mean ± SD [%]	RSD [%]
Wheat grain	Chlormequat-chloride	0.05, 0.1, 0.2	10	76-93	85±5.6	6.6
		0.25	4	77-95	86±7.5	8.8
		0.5	25	67-107	83±11.1	13.4
		2.0	3	86-88	87±1.4	1.6
		10	4	74-86	80±5.1	6.4
		Overall	46		84±9.0	10.7
Wheat straw	Chlormequat-chloride	0.5	3	67-72	69±2.1	3.0
		0.66	3	80-83	81±1.4	1.7
		1.0	13	74-106	89±10.0	11.2
		Overall	19		85±11.1	13.1
Oat	Chlormequat-chloride	0.1	3	83-100	93±9.4	10.1
		0.5	13	71-110	86±11.9	13.8
		1.0	6	72-98	80±10.1	12.5
		2.0	3	79-80	80±0.9	11.6
		Overall	25		85±10.8	12.8
Rye	Chlormequat-chloride	0.1	3	81-96	86±8.7	10.0
		0.5	7	81-96	91±5.8	6.4
		2.0	3	90-114	106±13.9	13.1
		Overall	13		93±10.9	11.7

Animal commodities

BASF Method No. 397

Residue analytical method 397 was developed for the determination of the residues of chlormequat-chloride in/on animal matrices (Weidenauer 1998, 1998/11454).

Homogenised samples are extracted with a mixture of acetone/water (2:1, v/v). The extract is passed over a cation exchange column, the chlormequat-chloride is eluted with diluted HCl, and the eluate is evaporated to dryness. The dry residue is re-dissolved in water, and washed with dichloromethane. The aqueous phase is evaporated to dryness. The residue is then transfered onto an alumina column using an acetonitrile/methanol mixture and the eluate evaporated to dryness. The residue is taken up in methanol and then evaporated to dryness. The residue is re-dissolved in water and injected into an ion chromatography system.

The accuracy of the method was assessed on the basis of the determined recovery rates. Samples were fortified with chlormequat-chloride at concentrations of 0.05 and 0.5 mg/kg in all

matrices (cow meat, liver, kidney and fat, and hen meat, liver, fat and eggs) except milk (0.01 and 0.1 mg/kg). Mean recoveries were between 70 and 110% and RSD values were < 20% for each fortification level and each matrix.

The LOQ for chlormequat-chloride was 0.05 mg/kg in all matrices tested except milk (0.01 mg/kg).

Good linearity was observed over the range 0.25 to 5 µg/mL for chlormequat-chloride although a correlation coefficient was not reported.

Table 23 Method recoveries for method 397: Chlormequat-chloride in animal matrices

Matrix	Analyte	Fortification Level	No. of Tests	Recoveries		
				Range [%]	Mean ± SD [%]	RSD [%]
Meat (cow)	Chlormequat-chloride	0.05	6	70-105	89±13.0	14.6
		0.5	6	67-79	73±4.0	5.5
		Overall	12		81±12.7	15.7
Liver (cow)	Chlormequat-chloride	0.05	8	82-110	97±11.8	12.1
		0.5	6	65-92	76±9.7	12.7
		Overall	14		88±15.1	17.1
Kidney (cow)	Chlormequat-chloride	0.05	7	75-116	85±14.1	16.6
		0.5	7	65-92	75±10.5	14.0
		Overall	14		80±12.9	16.1
Fat (cow)	Chlormequat-chloride	0.05	5	79-103	92±11.8	12.8
		0.5	5	74-84	78±4.6	5.9
		Overall	10		85±11.3	13.2
Milk (cow)	Chlormequat-chloride	0.01	5	70-96	85±10.4	12.3
		0.1	7	71-108	89±16.5	18.7
		Overall	12		87±13.9	16.0
Meat (hen)	Chlormequat-chloride	0.05	5	65-78	71±5.1	7.2
		0.5	5	89-110	101±10.1	10.0
		Overall	10		86±17.7	20.5
Liver (hen)	Chlormequat-chloride	0.05	7	66-108	85±15.2	18.0
		0.5	6	70-114	95±17.8	18.8
		Overall	13		89±16.6	18.6
Fat (hen)	Chlormequat-chloride	0.05	5	65-93	84±11.2	13.3
		0.5	6	63-82	76±7.4	9.7
		Overall	11		80±9.6	12.1
Eggs (hen)	Chlormequat-chloride	0.05	6	70-111	91±13.3	14.7
		0.5	7	71-108	89±15.4	17.3
		Overall	13		90±13.9	15.5

BASF Method No. 397/0

A residue analytical method, 397/0, was developed for the determination of the residues of chlormequat-chloride in/on animal matrices (Tilting 1999, 1999/10026).

Residues of chlormequat-chloride are extracted from animal matrices using a mixture of acetone/ acidified water (1:2, v/v). The extract is absorbed onto an ion exchange column and the chlormequat-chloride is eluted with 2M HCl. An ion pair is formed from the analyte and tetraphenyl borate before extraction with dichlormethane. Cleavage of the complex with diluted hydrochloric acid and repartitioning into the aqueous phase is followed by alumina column clean-up. Quantitation is achieved after ion chromatography with conductivity detection.

The accuracy of the method was assessed on the basis of the determined recovery rates. Samples were fortified with chlormequat-chloride at concentrations of 0.01 and 0.1 mg/kg in milk and at 0.05 and 0.5 mg/kg in all matrices as well as 5.0 mg/kg in cow liver and kidney and 5.0 mg/kg in hen liver. Mean recoveries values per fortification level for chlormequat-chloride were between 70–110% while RSD values were < 20%.

The LOQ for chlormequat-chloride was 0.05 mg/kg in all matrices tested, except milk (0.01 mg/kg).

Good linearity was observed over the range 0.25 to 1.0 µg/mL for chlormequat-chloride (r^2 > 0.999).

Table 24 Method recoveries for method 397/0: Chlormequat-chloride in animal matrices

Matrix	Analyte	Fortification Level	No. of Tests	Recoveries		
				Range [%]	Mean ± SD [%]	RSD [%]
Muscle (cow)	Chlormequat-chloride	0.05	5	95-108	103±5.7	5.6
		0.5	5	88-99	95±5.0	5.3
		Overall	10		99±6.7	6.7
Liver (cow)	Chlormequat-chloride	0.05	5	90-109	101±7.8	7.7
		0.5	5	65-71	70±2.4	3.5
		5.0	5	67-99	86±13.6	15.8
		Overall	15		86±15.8	18.5
Kidney (cow)	Chlormequat-chloride	0.05	5	68-83	77±6.9	9.1
		0.5	5	77-91	84±6.1	7.3
		5.0	5	87-95	92±2.9	3.2
		Overall	15		84±8.3	9.8
Fat (cow)	Chlormequat-chloride	0.05	5	97-103	100±2.1	2.1
		0.5	5	98-101	100±1.2	1.2
		Overall	10		100±1.6	1.6
Milk (cow)	Chlormequat-chloride	0.01	5	67-87	75±8.5	11.3
		0.1	5	70-79	75±3.4	4.6
		Overall	10		75±6.1	8.2
Eggs (hen)	Chlormequat-chloride	0.05	5	68-91	79±8.3	10.6
		0.5	5	77-92	83±5.9	7.1
		Overall	10		81±7.2	8.9
Muscle (hen)	Chlormequat-chloride	0.05	5	82-100	89±8.3	9.2
		0.5	5	81-94	87±5.9	6.8
		Overall	10		88±6.9	7.8
Liver (hen)	Chlormequat-chloride	0.05	5	95-124	108±14.8	13.7
		0.5	5	105-111	108±2.4	2.2
		5.0	5	88-98	93±3.8	4.1
		Overall	15		103±11.1	10.8

A confirmatory method for residue analytical method 397/0 was developed, in which quantitation is achieved using LC-MS/MS (Tilting 2004, 2004/1006522).

The accuracy of the method was assessed on the basis of the determined recovery rates. Samples were fortified with chlormequat-chloride at concentrations of 0.01 and 0.1 mg/kg in cow muscle, milk and eggs and at 0.05 and 0.5 mg/kg in liver. Mean recoveries per fortification level for chlormequat-chloride were in the range 70–110% while RSD values were ≤ 11% except for cow liver fortifid at 0.05 mg/kg (24%). However if the cow liver recovery at 50% is considered to be an outlier (other values range from 77–101% after fortification at 0.05 mg/kg), then the RSD is 12%.

The LOQ for chlormequat-chloride, defined as the lowest validated fortification level, was 0.01 mg/kg in muscle, milk and eggs and 0.05 mg/kg in liver.

Good linearity was observed over the range 0.01 to 0.05 µg/mL for chlormequat-chloride (r > 0.986).

Table 25 Method recoveries for method 397/0: Chlormequat-chloride in animal matrices

Matrix	Analyte	Fortification Level	No. of Tests	Recoveries		
				Range [%]	Mean ± SD [%]	RSD [%]
Muscle (cow)	Chlormequat-chloride	0.01	5	78-88	84±4.9	5.9
		0.1	5	98-108	105±3.9	3.7
		Overall	10		94±11.8	12.5

Matrix	Analyte	Fortification Level	No. of Tests	Recoveries		
				Range [%]	Mean ± SD [%]	RSD [%]
Liver (cow)	Chlormequat-chloride	0.05	5	50-101	80±19.0	23.9
		0.5	5	83-107	99±11.0	11.1
		Overall	10		89±17.6	19.7
Milk (cow)	Chlormequat-chloride	0.01	5	64-76	72±4.8	6.7
		0.1	5	84-97	92±5.1	5.6
		Overall	10		82±11.7	14.3
Eggs (hen)	Chlormequat-chloride	0.01	5	81-90	85±3.3	3.9
		0.1	5	87-101	93±5.4	5.8
		Overall	10		89±5.9	6.6

An independent laboratory validation was conducted for method 397/0 (Schulz and Meyer 2007, 2007/1043394). Samples were fortified with chlormequat-chloride at concentrations of 0.01 and 0.1 mg/kg in all matrices (bovine meat, bovine kidney, milk, egg and fat) except pig liver (0.05 and 0.5 mg/kg). Mean recoveries (between 70 and 110%) and RSD values (<20%) for each fortification level and each matrix were acceptable (Table 26).

Two transitions were monitored for chlormequat-chloride in each matrix tested; 122/58 (quantification) and 122/63 (confirmation). The LOQ for chlormequat-chloride was 0.01 mg/kg in all matrices tested except liver (0.05 mg/kg). Good linearity was observed over the range 0.005 to 0.02 ng/mL for chlormequat-chloride ($r^2 \geq 0.999$).

Table 26 Method recoveries for method 397/0: Chlormequat-chloride in animal matrices

Matrix	Analyte	Transition [m/z]	Fortification Level	No. of Tests	Recoveries		
					Range [%]	Mean ± SD [%]	RSD [%]
Bovine meat	Chlormequat-chloride	122→58	0.01	5	84-101	93±7.8	8.4
			0.1	5	94-111	107±7.4	6.9
		Overall		10		100±10.2	10.2
		122→63	0.01	5	80-110	94±11.5	12.3
			0.1	5	96-111	107±6.1	5.7
		Overall		10		100±11.1	11.1
Bovine liver	Chlormequat-chloride	122→58	0.05	5	80-102	92±8.0	8.6
			0.5	5	93-104	101±4.4	4.4
		Overall		10		97±7.6	7.9
		122→63	0.05	5	87-103	96±6.0	6.3
			0.5	5	93-99	96±2.6	2.7
		Overall		10		96±4.3	4.5
Pig kidney	Chlormequat-chloride	122→58	0.01	5	74-103	91±10.6	11.6
			0.1	5	82-96	91±5.4	5.9
		Overall		10		91±7.9	8.7
		122→63	0.01	5	77-116	100±15.5	15.5
			0.1	5	83-93	88±3.7	4.2
		Overall		10		94±12.2	13.0
Milk	Chlormequat-chloride	122→58	0.01	5	97-102	100±2.6	2.6
			0.1	5	105-110	108±1.8	1.7
		Overall		10		104±4.6	4.5
		122→63	0.01	5	94-97	95±1.3	1.4
			0.1	5	104-111	108±2.9	2.7
		Overall		10		102±7.3	7.1
Egg	Chlormequat-chloride	122→58	0.01	5	103-110	106±3.3	3.1
			0.1	5	99-115	105±6.3	6.0
		Overall		10		106±4.8	4.5
		122→63	0.01	5	107-109	108±1.0	0.9
			0.1	5	96-115	105±7.0	6.6
		Overall		10		107±4.9	4.6

Matrix	Analyte	Transition [m/z]	Fortification Level	No. of Tests	Recoveries		
					Range [%]	Mean ± SD [%]	RSD [%]
Fat	Chlormequat-chloride	122→58	0.01 0.1	5 5	90-100 87-97	95±3.9 93±3.9	4.1 4.2
		Overall		10		94±3.9	4.1
		122→63	0.01 0.1	5 5	91-97 87-96	94±3.1 92±3.4	3.3 3.6
		Overall		10		93±3.3	3.5

Another independent laboratory validation was conducted for method 397/0 (Weber 2010, 2011/1036855) due to modifications during Project No. IF-07/00891214 compared with the original method (Tilting 2004, 2004/1006522). Samples of meat, kidney, milk, egg and fat were fortified with chlormequat-chloride at the nominal fortification levels of 0.01 and 0.10 mg/kg and liver was fortified at 0.05 and 0.50 mg/kg.

Analysis of samples was performed according to method 397/0. Residues were extracted from animal matrices with acidified water and acetone. After filtration, the residue was adsorbed on an ion exchange resin and eluted with 2 M hydrochloric acid. An ion pair was formed from the analyte and tetraphenyl borate and extracted with dichloromethane. After cleavage of the complex with hydrochloric acid and repartitioning into the aqueous phase, the final extracts were analysed for residues of chlormequat-chloride using with high performance liquid chromatography with mass selective detection (LC-MS/MS). Two MRM reactions were measured for chlormequat-chloride, one for quantification (*m/z* 122/58) and the second for confirmation (*m/z* 124/58). For all matrices, for both fortification levels, and for both MRM transitions monitored, the mean recoveries were between 75% and 110%, with RSD values of <20%. A summary of the independent laboratory validation results is given in Table 27.

The LOQ for chlormequat-chloride was 0.01 mg/kg in all matrices tested except liver (0.05 mg/kg).

Good linearity was observed over the range 0.1 to 15.0 ng/mL for chlormequat-chloride (r^2 > 0.999).

Table 27 Method recoveries for method 397/0: Chlormequat-chloride in animal matrices

Matrix	Analyte	Transition [m/z]	Fortification Level	No. of Tests	Recoveries		
					Range [%]	Mean ± SD [%]	RSD [%]
Meat	Chlormequat-chloride	122→58	0.01 0.1	5 5	73-77 69-71	75±1.7 70±1.1	2.2 1.6
		Overall		10		72±2.7	3.7
		124→58	0.01 0.1	5 5	70-83 70-74	76±4.9 72±1.6	6.5 2.2
		Overall		10		74±4.1	5.5
Liver	Chlormequat-chloride	122→58	0.05 0.5	5 5	59-84 62-88	73±9.1 78±10.6	12.5 13.7
		Overall		10		75±9.7	12.9
		124→58	0.05 0.5	5 5	58-85 61-88	73±9.8 77±11	13.4 14.4
		Overall		10		75±10.1	13.4
Kidney	Chlormequat-chloride	122→58	0.01 0.1	5 5	76-99 68-82	82±9.5 75±5.1	11.5 6.7
		Overall		10		79±8.1	10.2
		124→58	0.01 0.1	5 5	81-102 69-82	90±8.5 76±5.1	9.5 6.7
		Overall		10		83±9.8	11.8
Milk	Chlormequat-chloride	122→58	0.01 0.1	5 5	67-90 75-96	80±9.5 84±8.2	11.9 9.7
		Overall		10		82±8.7	10.7
		124→58	0.01 0.1	5 5	72-96 75-93	85±10.5 84±8.4	12.4 10.0

Matrix	Analyte	Transition [m/z]	Fortification Level	No. of Tests	Recoveries		
					Range [%]	Mean ± SD [%]	RSD [%]
		Overall		10		84±9.0	10.7
Egg	Chlormequat-chloride	122→58	0.01 0.1	5 5	71-86 69-82	75±6.2 74±5.0	8.3 6.8
		Overall		10		75±5.4	7.2
		124→58	0.01 0.1	5 5	73-92 70-80	81±8.4 74±3.9	10.4 5.3
		Overall		10		77±7.1	9.2
Fat	Chlormequat-chloride	122→58	0.01 0.1	5 5	82-95 91-98	89±4.7 95±3.3	5.3 3.5
		Overall		10		92±4.7	5.1
		124→58	0.01 0.1	5 5	90-100 84-91	95±5.0 88±2.8	5.2 3.2
		Overall		10		92±5.5	6.0

Stability of residues in stored analytical samples

Plant matrices

A freezer storage stability study was conducted on grapes (Richter 2012, 2012/1187637). Homogenised grape samples were fortified with a reference standard of chlormequat chloride at 0.50 mg/kg and stored frozen (-20 °C) for up to 24 months, alongside untreated samples for use for determination of concurrent recoveries and as analytical controls. Samples were withdrawn at intervals and analysed for chlormequat chloride using an LC-MS/MS method (BASF method 530/0).

Table 28 Stability of chlormequat chloride in grapes

Storage period (months)	Residues in stored samples (mg/kg)	Stored recovery (% of nominal)	Concurrent recovery (mean)
0	0.48, 0.48 (mean=0.48)	97, 97 (mean=97)	98
1	0.50, 0.49 (mean=0.50)	100, 98 (mean=99)	99
3	0.46, 0.45 (mean=0.46)	92, 90 (mean=91)	95
6	0.52, 0.51 (mean=0.51)	104, 102 (mean=103)	105
12	0.54, 0.52 (mean=0.53)	109, 104 (mean=107)	109
18	0.46, 0.48 (mean=0.47)	93, 96 (mean=95)	102
24	0.47, 0.47 (mean=0.47)	94, 95 (mean=94)	107

No significant degradation of chlormequat chloride residues occurred in grapes over 2 years of frozen storage.

A study was conducted to investigate the stability under frozen storage of residues of chlormequat chloride in wheat grain and straw and various processed fractions of wheat and barley (Zietz 2004a, 2004/1016556). Samples of wheat grain and straw were fortified with chlormequat chloride at 0.10 and 0.50 mg/kg, respectively. The fortified and control samples were then stored frozen (-18 °C). For processed fractions, samples of wheat bran and wholegrain bread, and barley malt and beer from other residue studies were re-analysed after 13 months of further frozen storage. Samples were withdrawn from storage at intervals and analysed using an LC-MS/MS method (method number CEN/TC 275/WG 4N).

Table 29 Stability of chlormequat chloride in wheat grain and straw

Storage period (months)	Wheat grain		Wheat straw	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
0	85, 88, 90, 91, 93, 93, 97 (mean=91)	-	88, 89, 92, 92, 93, 93, 96 (mean=92)	-
1	89, 91 (mean=90)	92, 92	96, 98 (mean=97)	90, 94
3	95, 96 (mean=96)	97, 102	87, 94 (mean=91)	87, 92
6	98, 99 (mean=99)	95, 96	94, 94 (mean=94)	88, 92

Storage period (months)	Wheat grain		Wheat straw	
	Stored recovery (%)	Concurrent recovery (%)	Stored recovery (%)	Concurrent recovery (%)
12	99, 103 (mean=101)	92, 97	101, 102 (mean=101)	97, 99
18	103, 104 (mean=103)	97, 97	107, 108 (mean=108)	105, 110
24	105, 116 (mean=110)	101, 105	112, 114 (mean=113)	108, 116

Residues of chlormequat chloride are stable in wheat grain and straw for at least 24 months on storage at -18 °C.

Table 30 Recovery of chlormequat chloride residues from processed fractions of wheat and barley at re-analysis after further storage

Matrix	Storage intervals (months)	First analysis (prior to storage, mg/kg)	Concurrent recovery (%)	Re-analysis (after storage, mg/kg)	Stored recovery (%)	Concurrent recovery (%)
Wheat bran	13	3.58, 3.27	81, 90 (mean=85)	3.44, 3.22	96, 99 (mean=97)	93
Wholegrain bread (wheat)	13	0.56, 0.53	74, 83 (mean=79)	0.82, 0.79	146, 149 (mean=148)	130
Barley malt	12	1.29, 1.20	77, 82 (mean=79)	1.34, 1.34	104, 112 (mean=108)	92
Beer	11	0.19, 0.29	80, 93 (mean=87)	0.22, 0.26	115, 89 (mean=102)	83

After taking account of the concurrent recoveries, residues of chlormequat chloride are stable in wheat bran and wholegrain bread for up to 13 months storage, barley malt for up to 12 months, and beer for up to 11 months, at -18 °C.

Animal matrices

A study on the stability of residues of chlormequat chloride in cattle meat, milk and hens' eggs on frozen storage (-18 °C) was conducted (Zenide 2002, 2002/1011999). Homogenised samples of cattle meat and eggs were fortified with chlormequat chloride at 0.50 mg/kg, and milk at 0.10 mg/kg, then frozen alongside untreated control samples. Control and treated samples (two of each) were withdrawn from frozen storage at intervals up to 12 months, one of the control samples was fortified, and all samples analysed using an HPLC method based on method 397/0.

Table 31 Stability of residues of chlormequat chloride in cattle meat, milk, and eggs

Matrix	Storage period (months)	Stored recovery (%)	Concurrent recovery (%)
Cattle meat	0	72, 72	93
	3	69, 70	70
	6	79, 83	76
	9	71, 71	69
	12	81, 86	84
Milk	0	81, 85	88
	3	87, 94	82
	6	84, 89	82
	9	82, 93	93
	12	69, 98	84
Eggs	0	88, 100	92
	3	82, 86	102
	6	70, 72	80
	9	91, 99	91
	12	74, 90	92

Noting the concurrent recoveries, which were largely consistent with the stored recoveries, residues of chlormequat chloride were stable in cattle meat, milk and hen eggs for up to 12 months of frozen storage at -18 °C.

USE PATTERN

Information on registered uses made available to this Meeting is shown in Table 32 and Table 33.

Table 32 Registered uses of chlormequat-chloride on grapes

Crop	Country	Formulation		Application						PHI [days]
		g ai/L	Type	Method	Growth stage/ timing	[g ai/100L] max	Water L/ha per appl. min. max.	Rate [g ai/ha] min. max.	Season Max. [g ai/100L/year] or (no. per crop)	
	Berries and other small fruits									
Grapes	Argentina	750	SL	Foliar	2 weeks before flowering	50	NA	50	1	NA
Wine-grapes	Australia	100	SL	Foliar	Zante currant (Apply 70-100% cap fall) Other varieties (Apply 1-2 weeks before flowering)	3-40	1100-1700	33-680	1	NA
Grapes	India	500	SL	Foliar	1: 3-5 leaf stage after April pruning 2: 5-7 leaf stage after April pruning 3: 3-5 leaf stage after October pruning	50 100 25	1000	500 1000 250	3	91

Table 33 Registered uses of chlormequat chloride on cereals

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Cereal Grains									
Wheat	Argentina	750	SL	Foliar	From tillering until first node (BBCH 21-31)	NA	2025	1	NA
Wheat	Australia	750	SL	Foliar	Apply at Zadoks stage Z25 to Z31	min. 30 (aerial) 100 (ground)	375-975	NA	H: NA G: 21
Rye, winter; Triticale, winter; Wheat, winter	Belarus	750	SL	Foliar	Spray at the beginning of stem elongation (BBCH 30-31)	200	750-938	1	NA
Triticale, spring	Belarus	750	SL	Foliar	Spray at flag leaf stage (BBCH 37)	200-300	750	1	NA
Wheat, spring	Belarus	750	SL	Foliar	Spray at BBCH 30-31	200-300	750-938	1	NA
Barley, spring	Belarus	750	SL	Foliar		200	675	1	NA
Winter wheat and triticale	Belarus	750	SL	Foliar	Spray at the start of stem elongation (BBCH 31-32)	200-300	1125	1	NA

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Winter wheat and triticale	Belarus	750	SL	Foliar	Spray at mid-tillering (BBCH 25)	200-300	487.5	1	NA
Oats	Belgium	750	SL	Foliar	Apply at 40cm crop height	200-600	1425	1	NA
Triticale	Belgium	750	SL	Foliar	Apply between BBCH 30 and BBCH32	200-600	750	1-2	NA
Wheat, spring	Belgium	750	SL	Foliar	Apply between BBCH 21 and BBCH 30	200-600	450-750	1	NA
Wheat	Belgium	368	SL	Foliar	Apply between BBCH 30 and BBCH 31-32	NA	736	1	NA
Wheat	Bulgaria	750	SL	Foliar		100-400	675-900	1-2	60
Wheat	Canada	460	SL	Foliar (single)	DO NOT use later than Feekes GS 7	200-400	1150-1380	1	NA
Wheat (Lennox)	Canada	460	SL	Foliar (split)	DO NOT use later than Feekes GS 7	200-400	1150 345	2	NA
Wheat (Lennox, Norstar)	Canada	460	SL	Foliar (single)	DO NOT use later than Feekes GS 7	200-400	920-1150	1	NA
Wheat (Monopol)	Canada	460	SL	Foliar (split)	DO NOT use later than Feekes GS 7	200-400	920-1150 230	2	NA
Wheat (Monopol)	Canada	460	SL	Foliar (late)	DO NOT use later than Feekes GS 7	200-400	1150-1380	1	NA
Wheat (Monopol)	Canada	460	SL	Foliar (single)	DO NOT use later than Feekes GS 7	200-400	1150	1	NA
Wheat (Absolvent, Vuka, Norstar)	Canada	460	SL	Foliar (split)	DO NOT use later than Feekes GS 7	200-400	230	2	NA
Wheat (Absolvent, Vuka)	Canada	460	SL	Foliar (single)	DO NOT use later than Feekes GS 7	200-400	1150-1380	1	NA
Wheat, spring	Chile	460[a]	SL	Foliar	Apply up to the first node stage (Feekes 4 to 6 or Zadok's 25-31. Not recommended for use in crops for grazing.	150-300	920-1150	1	NA
Wheat, intermediate	Chile	460[a]	SL	Foliar		150-300	1150	1	NA
Wheat, winter	Chile	460[a]	SL	Foliar		150-300	1150-1380	1	NA
Barley, winter	Croatia	750	SL	Foliar	Apply before appearance of first node (BBCH 21-29)	NA	750-1500	1-2 (can split between an autumn and a spring application)	63
Oats, spring; Oats, winter	Croatia	750	SL	Foliar	Apply from beginning of node formation to the appearance of the third node (BBCH 30-32)	NA	750-1500	NA	42

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Rye, winter	Croatia	750	SL	Foliar	Apply between beginning of node formation to flag leaf formation (BBCH 30-37)	NA	1125-1500	NA	63
Triticale	Croatia	750	SL	Foliar	Apply from BBCH 26-30	NA	750-1500	NA	63
Wheat, spring	Croatia	750	SL	Foliar	Apply from BBCH 21-29	NA	375-825	NA	63
Wheat, winter	Croatia	750	SL	Foliar	Apply between BBCH 21-31	200-600	750-1500	1-2 (can split application into 1125 + 375 at BBCH 21-29 and 30-31)	63
Oats	Czech Republic	750	SL	Foliar	Apply from BBCH 31-32	100-400	1500	NA	*
Rye, winter	Czech Republic	750	SL	Foliar	BBCH 30-31	100-400	1500	NA	*
Wheat, spring	Czech Republic	750	SL	Foliar	BBCH 23-29	100-400	600	NA	*
Wheat, winter	Czech Republic	750	SL	Foliar	BBCH 30-31 (600 g ai/ha can be used at BBCH 25-31)	200-600	600-1500	NA	*
Rye	Denmark	460	SL	Foliar	Make 1 application at BBCH 30-32 or 2 applications at BBCH 30-31 and 32-37	100-200	1150 or 690 + 460	1-2	NA
Oats	Denmark	460	SL	Foliar	Apply at BBCH 30-31	100-200	1150	1	NA
Wheat, spring	Denmark	460	SL	Foliar	Apply at BBCH 25-30	100-200	460-690	1	NA
Wheat, winter	Denmark	460	SL	Foliar	Make 1 application at BBCH 25-30 or 2 at BBCH 25-30 and 30-32	100-200	460-920 or 460-920 + 230-345	1-2	NA
Oats	Denmark	750	SL	Foliar	Apply at BBCH 30-31	100-400	1125	1	NA
Rye	Denmark	750	SL	Foliar	Make 1 application at BBCH 30-32 or 2 applications at BBCH 30-31 and 32-37	100-400	1125 Or 675 + 750	1-2	NA
Triticale	Denmark	750	SL	Foliar	Apply at BBCH 30-31	100-400	750	1	NA
Wheat, spring	Denmark	750	SL	Foliar	Apply at BBCH 25-30	100-400	450-675	1	NA
Wheat, winter	Denmark	750	SL	Foliar	Make 1 application at BBCH 25-30 or 2 applications at BBCH 25-30 and 30-32	100-400	One application at 450-900 Or two at 450-900 + 225-375	1-2	NA

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Barley	Estonia	750	SL	Foliar	Apply at BBCH 25-32	200 - 400	375-750	1	NA
Oats	Estonia	750	SL	Foliar	Apply at BBCH 30-31	200 - 400	750-1175	1	NA
Rye	Estonia	750	SL	Foliar	Apply at BBCH 25-32	200 - 400	750-1500	1	NA
Triticale	Estonia	750	SL	Foliar	Apply at BBCH 25-32	200-400	750-1125	1	NA
Wheat, spring	Estonia	750	SL	Foliar	Apply at BBCH 25-32	200 - 400	600-938	1	NA
Wheat, winter	Estonia	750	SL	Foliar	Apply at BBCH 25-32	200 - 400	750-1125	1	NA
Oats	Finland	750	SL	Foliar	Apply up to first node stage (BBCH 31)	200-400	750-1125	1	NA
Wheat, winter	Finland	750	SL	Foliar	Apply up to first node stage (BBCH 31)	200-400	750-1500	1	NA
Rye	Finland	750	SL	Foliar	Apply up to first node stage (BBCH 31)	200-400	1125-1500	1	NA
Wheat, spring	Finland	750	SL	Foliar	Apply up to first node stage (BBCH 31)	200-400	225-750	1	NA
Oats, winter	France	460	SL	Foliar	Treat when oats are 35-40 cm tall	100-150	1380	1	NA
Rye, winter	France	460	SL	Foliar	Treat when rye is 20-30 cm tall	100-150	1150	1	NA
Wheat, hard winter	France	460	SL	Foliar	Mid-tillering to 1 cm spike (Stage 25-30)	100-150	1610	1	NA
Wheat soft spring; Wheat soft, winter	France	460	SL	Foliar	Must be applied at the end of tillering or beginning of winter recovery. Optimum treatment period is Stage 29-30.	100-150	920	1	NA
Wheat, winter (TRZAW); Wheat, spring (TRZAS)	France	750	SL	Foliar	Optimum treatment period is Stage 29-30.	100-400	900	1	NA
Wheat, Durum (TRZDU)	France	750	SL	Foliar	Treat between Stage 25 and Stage 30	100-400	1500	1	NA
Barley, spring	France	230	SL	Foliar	Treat between Stage 31 and Stage 32	110	345	1	NA
Barley, winter; Rye, winter; Triticale; Wheat, hard, winter	France	230	SL	Foliar	Treat between Stage 31 and Stage 39	110	575	1	NA
Wheat, soft, winter	France	230	SL	Foliar	Treat between Stage 31/32 and Stage 37	110	460	1	NA

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Wheat, winter	France	345	SL	Foliar	Treat between mid-tillering and first node	100-400	690	1	NA
Wheat, winter	Hungary	460	SL	Foliar	a)BBCH 21-31 b)BBCH 21-32	200-300	a)322 – 920 b) 690 + 230	a) 1 b) 2	60
Wheat, winter	Ireland	750	SL	Foliar	Apply up to BBCH 31 (First node detectable)	200-450	a) 1500 b) 1125 + 563	a) 1 b) 2	NA
Wheat, spring	Ireland	750	SL	Foliar	Apply up to BBCH 31 (First node detectable)	200-450	a)750 b) 750 + 563	a) 1 b) 2	NA
Barley, spring	Ireland	750	SL	Foliar	Apply up to BBCH 30 (Leaf sheath erect)	220-450	1500	1	NA
Barley, winter	Ireland	750	SL	Foliar	Apply up to BBCH 30 (Leaf sheath erect)	220-450	562.5 (applied in autumn) + 1500 (applied in spring)	1	NA
Oats, winter and spring	Ireland	750	SL	Foliar	Apply up to BBCH 32 (Second node detectable)	200-450	1500	1	NA
Triticale	Ireland	750	SL	Foliar	Apply up to BBCH 31 (First node detectable)	220-450	1875	1	NA
Wheat, winter and spring (all except durum)	Ireland	368	SL	Foliar	Apply before second node is detectable (BBCH 32)	100-400	a)920 b) 644+276	a) 1 b) 2	NA
Wheat, spring	Japan	460	SL	Foliar	Apply before or after the 6th leaf stage (30-40 cm plant height)	1000-1200	920	1	NA
Wheat, winter	Japan	460	SL	Foliar	Apply at early stem elongation up to the second node (BBCH 30-32)	1000-1200	1380-2300	1	NA
Wheat, winter	Japan	460	SL	Foliar	Apply 10-20 days before heading (BBCH 51), at 40-60 cm plant height	1000-1200	2300	1	NA
Wheat, spring; Wheat, winter	Kazakhstan	750	SL	Foliar	Spray during tillering phase, i.e. BBCH 21-30	NA	750	1	NA
Oats	Latvia	750	SL	Foliar	Apply between BBCH 32-47	200-400	1500	1	NA
Rye	Latvia	750	SL	Foliar	Apply between BBCH 21-32	200-400	1500-2250	1	NA

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Triticale	Latvia	750	SL	Foliar	Apply between BBCH 25-30	200-400	750-1500	1	NA
Wheat, spring	Latvia	750	SL	Foliar	Apply between BBCH 21-30	200-400	375-1125	1	NA
Wheat, winter	Latvia	750	SL	Foliar	Apply between BBCH 21-30	200-400	375-1125	1	NA
Rye	Lithuania	750	SL	Foliar	BBCH 31-32	200-400	1500	1	NA
Triticale	Lithuania	750	SL	Foliar	BBCH 31-32	200-40	1500	1	NA
Barley	Lithuania	750	SL	Foliar	BBCH 25-29	200-400	750	1	NA
Wheat, spring	Lithuania	750	SL	Foliar	BBCH 25-29	200-400	750	1	NA
Wheat, winter	Lithuania	750	SL	Foliar	Can be applied as one application at BBCH 25-29, or split into two applications of 600-900 g ai/ha at BBCH 25-29 and 150-225 g ai/ha at BBCH 30-31	200-400	750-1125	1-2	NA
Oats	Luxembourg	750	SL	Foliar	At 40cm crop height	NA	1425	1	NA
Rye, winter	Luxembourg	750	SL	Foliar	Between BBCH 30 and BBCH 37	NA	1500	1	NA
Triticale, winter	Luxembourg	750	SL	Foliar	Between BBCH 30 and BBCH 32	NA	750	1-2	NA
Wheat, spring	Luxembourg	750	SL	Foliar	Between BBCH 21 and BBCH 30	NA	450-750	1	NA
Wheat, winter	Luxembourg	368	SL	Foliar	Between BBCH 30 and BBCH 31-32	NA	736	1	NA
Barley; Triticale, Wheat, winter	Macedonia	750	SL	Foliar	BBCH 30-32	200-600	750	2	63
Oats	Macedonia	750	SL	Foliar	Treat when plant is 40 cm high	200-600	1425	1	42
Rye, winter	Macedonia	750	SL	Foliar	BBCH 30-32	200-600	750	NA	63
Wheat, spring	Macedonia	750	SL	Foliar	BBCH 21-30	200-600	450-750	1	63
Cereal grains	Moldova	750	SL	Foliar	Apply from the start of tillering until the first node (BBCH 21-31)	200-300	1175	1	NA
Wheat	Morocco	460	SL	Foliar	Apply from the end of tillering until the beginning of stem elongation, and do not apply after the first node (BBCH 29-31)	NA	920	1	90

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Wheat, spring	Netherlands	750	SL	Foliar	Application preferably before the start of stem elongation, although after stem elongation commences is acceptable, Do not apply after the first awns are visible (BBCH 21-49)	200-400	413	1-2	NA
Wheat, winter	Netherlands	750	SL	Foliar	Application preferably before the start of stem elongation, although after stem elongation commences is acceptable, Do not apply after the first awns are visible (BBCH 21-49)	200-400	413-1000	1-2	NA
Oats; Wheat	New Zealand	750	SL	Foliar	Oats – Zadok's GS 32/ Feekes GS 7; Wheat Zadok's GS 30-32, Feekes GS 5-7;	200-350	750-1500	1	G: 42
Oats	Norway	460	SL	Foliar	Plants 20-25 cm high, 4-5 leaves (BBCH 14-15)	100-400	460-1380	1	NA
Rye	Norway	460	SL	Foliar	Plants 20-25 cm high, 4-5 leaves (BBCH 14-15)	100-400	460-1380	1	NA
Wheat	Norway	460	SL	Foliar	Plants 15-25 cm high, 3-5 leaves (BBCH 13-15)	100-400	460-1380	1	NA
Oats	Norway	750	SL	Foliar	Plants 20-25 cm high, 4-5 leaves (BBCH 14-15)	100-400	750-1200	1	NA
Rye	Norway	750	SL	Foliar	Plants 20-25 cm high, 4-5 leaves (BBCH 14-15)	100-400	750-1200	1	NA
Wheat	Norway	750	SL	Foliar	Plants 15-25 cm high, 3-5 leaves (BBCH 13-15)	100-400	750-1200	1	NA
Barley, winter; Wheat, winter	Romania	750	SL	Foliar	BBCH 26-32	100-400	900	1	NA
Wheat, winter	Russian Federation	750	SL	Foliar	Apply at early tillering up to the start of stem elongation (BBCH 21-31)	ground 300; aerial 50	750-1125	1	60
Wheat, spring	Russian Federation	750	SL	Foliar	Spray during stem elongation (BBCH 30-39)	ground 300; aerial 50	750-1125	1	60

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Barley, spring	Russian Federation	750	SL	Foliar	Spray during early stem elongation (BBCH 30-33)	ground 300; aerial 50	750-1125	1	60
Rye, winter	Russian Federation	750	SL	Foliar	Spray during stem elongation (BBCH 30-39)	ground 300; aerial 50	750-1125	1	60
Wheat	Republic of Serbia	750	SL	Foliar	Apply between BBCH 21-32	200-400	750-1500	1	63
Wheat	South Africa	750	SL	Foliar	Apply at first stem elongation (5-7 leaf stage, BBCH 35-57)	ground 300-400; aerial 30	1575	1	H/G: 49
Rye	Sweden	460	SL	Foliar	Apply at BBCH 25-31	200-400	920-1380	1	NA
Oats	Switzerland	460	SL	Foliar	Apply during stem elongation up to the third node (BBCH 30-33)	300-600	1150-1840	1	NA
Triticale	Switzerland	460	SL	Foliar	Apply from the end of tillering until the beginning of stem elongation (BBCH 29-30)	300-600	230-1150	1	NA
Wheat	Switzerland	460	SL	Foliar	Apply from the end of tillering until the beginning of stem elongation (BBCH 29-30)	300-600	230-1150	1	NA
Wheat	Turkey	750	SL	Foliar	Apply at BBCH 30-31	200-400	1875	1	NA
Cereals	Ukraine	750	SL	Foliar	Apply from the start of tillering until the first node (BBCH 21-31)	200-300	1175	1	NA

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Wheat, winter and Autumn drilled Wheat, spring	United Kingdom	750	SL	Foliar	a) Ideally apply just prior to the first node detectable stage and not later than the second node detectable stage (BBCH 30-32) b) Apply first dose at the tillers formed to leaf sheath lengthening stage followed by second dose at the leaf sheaf erect up to and including the first node detectable stage (BBCH 21-30 and BBCH 30-32)	220-450	a) 1650 b) 1200+450	a) 1 b) 2	NA
Wheat, spring (Spring drilled)	United Kingdom	750	SL	Foliar	Do not apply later than the first node detectable stage on the majority of tillers (maximum BBCH 31)	220-450	825	1	NA
Barley, winter	United Kingdom	750	SL	Foliar	a) Apply from mid-tillering to just prior to the first node detectable stage b) Apply first dose in the autumn and second dose from mid-tillering to just prior to the first node detectable stage (BBCH 31)	220-450	a) 1650 b) 450+1200	a) 1 b) 2	NA
Oats, winter and spring	United Kingdom	750	SL	Foliar	Apply before third node is detectable (BBCH 33)	220-450	1650	1	NA
Rye	United Kingdom	750	SL	Foliar	Apply before second node is detectable (BBCH 32)	220-450	1650	1	NA
Triticale	United Kingdom	750	SL	Foliar	Apply from mid-tillering until just prior to first node detectable (BBCH 31)	220-450	1650	1	NA

Crop	Country	Formulation		Application					PHI [days]
		g ai/L	Type	Method	Growth stage/timing	Spray volume (L/ha)	Rate (g ai/ha)	Maximum number of application, seasonal rate (g ai/ha)	
Wheat, winter	United Kingdom	230	SL	Foliar	a) Up to and including flag leaf ligule just visible stage (BBCH 39) b) Up to and including flag leaf sheath opening stage (BBCH 47)	220	a)460 or b)345	1	NA
Barley, winter	United Kingdom	230	SL	Foliar	a) Up to and including flag leaf ligule just visible stage (BBCH 39) b) Up to and including first awns visible stage (BBCH 49)	220	a)460 or b)345	1	NA
Barley, spring	United Kingdom	230	SL	Foliar	Up to and including flag leaf ligule just visible stage (BBCH 39)	220	345	1	NA
Wheat, winter	United Kingdom	368	SL	Foliar	Not later than second node detectable stage (BBCH 32)	200	a)920 b)644+276	a)1 b)2	NA
Wheat, winter	Uzbekistan	750	SL	Foliar		200-300	750-1125	1	NA

[a] Formulation also contains 320 g/L choline chloride

H: harvest; G: grazing

* Label instruction is not to graze green matter.

RESIDUES RESULTING FROM SUPERVISED TRIALS ON CROPS

The Meeting received information on supervised trials for the uses of chlormequat-chloride on berries and other small fruits (table grapes) and cereals (barley, oats, rye and wheat), and animal feeds (barley, oat, rye and wheat forage and straw).

Trials were well documented with laboratory and field reports. The former included method validation including recoveries with spiking at residue levels similar to those occurring in samples from the supervised trials. Dates of analyses or duration of sample storage were also provided. Samples were collected and stored frozen immediately or soon after sampling. Trials included control plots, although results for control samples are only noted in the Tables when residues above the LOQ were noted. Residues are have not been adjusted for recovery.

Residues from the trials conducted according to maximum GAP have been used for the estimation of maximum residue levels and dietary risk assessment and are underlined. If a higher residue level was observed at a longer PHI than the GAP, the higher value has been used in MRL setting and dietary risk assessment.

Table 34 Supervised residue trial data provided

Group	Commodity	Countries/regions	Table No.
FB Berries and other small fruits	Grapes (table variety)	India	35
GC Cereal grains	Barley	N. and S. Europe	36
	Oats	N. and S. Europe	37
	Rye	N. and S. Europe	38
	Wheat	N. and S. Europe	39
AF Cereal forages	Barley forage	N. and S. Europe	40
	Oat forage	N. and S. Europe	41
	Rye forage	N. and S. Europe	42
	Wheat forage	N. and S. Europe	43
AS Cereal straws and fodders	Barley straw	N. and S. Europe	44
	Oat straw	N. and S. Europe	45
	Rye straw	N. and S. Europe	46
	Wheat straw	N. and S. Europe	47

Grapes

A series of eight residue trials was conducted in India in grapes (table variety) during 2011/12 to determine the residues of chlormequat chloride after treatment with a 500 g/L SL formulation (Sathiyanarayanan, 2013). An untreated control plot and a treated plot were established at each site, and to each treated plot, three foliar spray applications were made during 2011 using a knapsack sprayer. The first and second applications were made at a target rate of 500 g ai/ha and 1000 g ai/ha, a few days apart and timed for after the April pruning, and the third at a target rate of 250 g ai/ha, around 6 months later, after the October pruning.

Grapes were sampled at two intervals during early 2012, immature fruit in January/February, at 3–4 months after the last application, and mature fruit at harvest in February-April, at 4-5 months after the last application. Plot and sample sizes were adequate. Samples were frozen (-20 °C) and kept frozen during transport and while awaiting analysis.

The use pattern in the trials is consistent with common viticultural practices used for approximately 70% of the grape crop in India, in vineyards in hot tropical areas (Shikhamany, 2001). The practice in these areas is for one crop per year to be harvested, in March-April, with two prunings per year. The first pruning takes place in March-May after harvest, when all canes are pruned back to single node spurs, while the second pruning takes place in October-November (the window for this pruning is fixed due to adverse weather conditions before October or later than November) in preparation for fruiting for the next year's harvest in March-April.

Samples were analysed by LC-MS/MS after extraction with water/methanol/2 N HCl (65:30:5 v/v/v, and cleanup of extracts using alumina columns. The method LOQ was 0.05 mg/kg, and recoveries ranged from 87–101% (at LOQ, n=5), and 94-100% (at 10 × LOQ, n=5). Analyses were conducted a maximum of 12 days after harvest. A concurrent storage stability study was conducted over 50 days using untreated grapes fortified at 0.50 mg/kg; recoveries of 97.0% and 91.2% were observed before and after storage respectively.

There are potential concerns with respect to all trials being conducted using the same grape variety (Thompson seedless). In trial 6, another application of Lihosin (chlormequat chloride) was made at 0.5 mL/L on 29/10/11 (i.e. 4 days before trial application number 3). At trial 8, another

chlormequat application was made on 18/11/11 at 0.5 mL/L (5 days before the trial application number 3).

Table 35 Residues of chlormequat chloride in grapes

Location (variety)	Application				Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	No. (RTI, days)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, DALA			
Kasabe Senu, Dist. Nashik, Maharashtra, India (Thompson seedless)	3 (4, 183)	0.5, 1.0, 0.25	500	96 150	< 0.05 (< 0.05, < 0.05) < 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04) < 0.04 (< 0.04, < 0.04)	2014/1043282,Location 1
Palkhed Bandhar, Dist. Nashik, Maharashtra, India (Thompson seedless)	3 (4, 153)	0.5, 1.0, 0.25	500	113 127	< 0.05 (< 0.05, < 0.05) < 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04) < 0.04 (< 0.04, < 0.04)	2014/1043282,Location 2
National Research Centre for Grapes, Pune, Maharashtra, India (Thompson seedless)	3 (5, 179)	0.5, 1.0, 0.25	500	107 128	< 0.05 (< 0.05, < 0.05) < 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04) < 0.04 (< 0.04, < 0.04)	2014/1043282,Location 3
Rajuri, Tq. Junnar, Dist. Pune, Maharashtra, India (Thompson seedless)	3 (5, 192)	0.5, 1.0, 0.25	500	112 134	< 0.05 (< 0.05, < 0.05) < 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04) < 0.04 (< 0.04, < 0.04)	2014/1043282,Location 4
Palashi, Tq. Khanapur, Dist. Sangli, Maharashtra, India (Thompson seedless)	3 (5, 176)	0.5, 1.0, 0.25	500	79 128	< 0.05 (< 0.05, < 0.05) < 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04) < 0.04 (< 0.04, < 0.04)	2014/1043282,Location 5
Takali, Tq. Miraj, Dist. Sangli, Maharashtra, India (Thompson seedless)*	4 (5, 165, 4)	0.5, 1.0, 25 g ai/100 L*, 0.25	500	112 134	< 0.05 (< 0.05, < 0.05) < 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04) < 0.04 (< 0.04, < 0.04)	2014/1043282,Location 6
Kasegaon, Tq. Pandharpur, Dist. Solapur, Maharashtra, India (Thompson seedless)	3 (4, 172)	0.5, 1.0, 0.25	500	117 139	< 0.05 (< 0.05, < 0.05) < 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04) < 0.04 (< 0.04, < 0.04)	2014/1043282,Location 7
Ugar Khurd, Ta Athani, Dist. Belgaon, Karnataka, India (Thompson seedless)*	4 (5, 157, 5)	0.5, 1.0, 25 g ai/100 L*, 0.25	500	91	< 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04)	2014/1043282,Location 8

Location (variety)	Application				Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	No. (RTI, days)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, DALA			
				120	< 0.05 (< 0.05, < 0.05)	< 0.04 (< 0.04, < 0.04)	

No residues above the LOQ were found in any of the untreated control samples.

*A fourth application of chlormequat chloride was inadvertently made at Locations 6 and 8. Spray volume not stated.

Cereal grains

Residue trials were conducted in wheat, barley, rye, and oats.

A series of trials was conducted in several growing seasons between 2003 and 2011 in northern and southern Europe in barley, oats, rye and wheat (Schulz 2005, 2004/1015956), (Klimmek and Zell 2010, 1014090), (Zell and Amann 2011, 2011/1071895), (Zell and Breyer 2011, 2011/1071894), (Klimmek and Breyer 2012a, 2012/1016109), (Klimmek and Breyer 2012b, 2012/1016107), (Klimmek and Breyer 2012c, 2012/1016108), Klimmek, Zell and Amann 2011, 2011/1070055), (Klimmek and Marzouki 2008, 2008/1014941), (Klimmek 2008, 2008/1016108), (Klimmek and Gizler 2009, 2009/1021674), (Raunft and Mackenroth 2005, 2005/1014176). A single foliar application of an SL formulation (usually 750 g/L) was made at a target rate of 1500 g ai/ha and generally at a target growth stage of BBCH 32 or 37 for southern and northern Europe respectively, using a boomsprayer. Some sites included additional plots treated at additional rates and/or using other SL formulations. At all sites, treated whole plant (forage) samples were collected on the day of application, while treated grain and straw samples were collected at commercial harvest. At some sites run as decline trials, additional whole plant samples were collected at target intervals of 14, 28 and 42 days after application, with the 42-day samples being separated into ear/panicle and remaining plant fractions. Untreated control whole plant samples were collected at the 0- and 42-day intervals, with control grains and straw being collected at harvest. Duplicate samples were generally collected, with one sample being analysed and the other kept as a retention sample. Plot and sample sizes were adequate. Except where noted, no other pesticides that would be expected to interfere with the trial were applied.

Samples were stored frozen until analysis. Chlormequat chloride residues were determined using an LC-MS/MS method involving extraction with methanol/water/HCl, followed by solid phase extraction cleanup (alumina cartridges) using method number BASF 530/0. Concurrent recoveries were acceptable. Sample analyses were completed within 9 months of collection.

Where residues results were adjusted for proportionality for estimation of maximum residue levels, both the raw numbers, and the proportionally adjusted values (italicised and underlined) are tabulated.

Table 36 Residues of chlormequat chloride in barley after a single application

Location, Year (variety)	Application					Residues, mg/kg as chlormequat chloride	Residues, mg/kg as chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
F-91150 Erzeville, Roinvillers, France, 2009 (spring barley, Sebastian)	750 SL	37	1.7	219	76	0.84	0.65	2010/1014090, 06

Location, Year (variety)	Application					Residues, mg/kg as chlormequat chloride	Residues, mg/kg as chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
50180 Utebo, Zaragoza, Spain, 2010 (barley, Graphic)	750 SL	32	1.4	182	69	0.40	0.31	2011/1071895, 01
66750 Saint-Cyprien, Pyrénées-Orientales, France, 2010 (barley, Prestige)	750 SL	32	1.6	207	59	0.40	0.31	2011/1071895, 02
50490 Villareal de Huerva, Spain, 2010 (barley, Montage)	750 SL	32	1.5	200	70	0.76	0.59	2011/1071895, 03
01560 St-Jean-sur-Reyssouze, Ain, France, 2010 (barley, Vanessa)	750 SL	32	1.4	187	84	0.08	0.062	2011/1071895, 04
21737 Wischhafen, Niedersachsen, Germany, 2011 (winter barley, Pelikan)	750 SL	37	1.5	202	76	0.16	0.12	2012/1016109, 01
21726 Oldendorf, Niedersachsen, Germany, 2011 (winter barley, Naomie)	750 SL	37	1.6	211	76	0.22	0.17	2012/1016109, 02
45300 Thignonville, Loiret, France, 2011 (spring barley, Sebastian)	750 SL	37	1.5	202	67	0.47	0.36	2012/1016109, 03
91150 Mespuits, Essonne, France, 2011 (spring barley, Sebastian)	750 SL	37	1.4	190	68	0.41	0.32	2012/1016109, 04
82130 Lafrancaise, Midi P., France, 2011	750 SL	32	1.6	220	73	< 0.05	< 0.04	2012/1016109, 05

Location, Year (variety)	Application					Residues, mg/kg as chlormequat chloride	Residues, mg/kg as chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
(winter barley, Azurel)								
82700 Bourret, Tarn et Garonne, France, 2011 (winter barley, Azurel)	750 SL	32	1.4	181	70	0.78	0.60	2012/1016109, 06
44492 Fonfria, Teruel, Spain, 2011 (barley, Estrelia)	750 SL	32	1.5	200	75	1.4	1.1	2012/1016109, 07
22809 Loarre, Aragon, Spain, 2011 (barley, Meseta)	750 SL	32	1.5	200	72	1.2	0.93	2012/1016109, 08
67229 Gerolsheim Römerstrasse 8, Rheinland-Pfalz, Germany, 2003 (spring barley, Scarlett)	350 SL	37	0.70	100	55	1.0	0.78	2004/1015956, 02
	750 SL	37	1.5	100	55	0.99	0.77	
Homelands Farm, Bucknell, Bicester, OX6 9NB, UK, 2003 (winter barley, Leonie)	350 SL	37	0.70	100	75	0.92	0.71	2004/1015956, 03
	750 SL	37	1.5	100	75	0.64	0.50	
67160 Seeback route de Hunspach, Alsace, France, 2003 (winter barley, Majestic)	350 SL	37	0.70	100	58	0.46	0.36	2004/1015956, 04
	750 SL	37	1.5	100	58	0.49	0.38	

Except where noted, no residues above the LOQ were found in any of the untreated control samples.

Table 37 Residues of chlormequat chloride in oats after a single application

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
F-45300 Yévre-la-Ville, France (winter oats, Expression)	750 SL	37	1.6	208	79	2.8	2.2	2010/1014090, 09
D-21709, Burweg, Germany (spring oats, Freddy)	750 SL	37	1.7	219	65	3.4	2.6	2010/1014090, 10
02690 Alpera, Albecete, Spain, 2010 (oats, Norlys)	750 SL	32	1.66	220	69	1.1	0.90 *1.0*	2011/1070055, 01
40018 Maccaretolo, Italy, 2010 (oats, Argentina)	750 SL	32	1.37	182	76	0.87	0.67 *0.90*	2011/1070055, 02
27109 Düdenbüttel, Niedersachsen, Germany, 2010 (oats, Dominik)	750 SL	37	1.51	200	46	4.1	3.2	2011/1070055, 03
16321 Bernau, Brandenburg, Germany, 2010 (oats, Flämingsford)	750 SL	39	1.67	221	52	4.3	3.3	2011/1070055, 04
45300 Boynes, Loiret, France, 2010 (oats, Grafton Redigo)	750 SL	37	1.56	207	67	2.6	2.0	2011/1070055, 05
68320 Muntzenheim, Alsace, France, 2010 (oats, Cornell)	750 SL	37	1.59	210	60	2.3	1.8	2011/1070055, 06
82290 Meauzac, Tarn et Garonne, France, 2010 (oats, Charmoise)	750 SL	32	1.5	196	-	Trial accidentally harvested prior to sampling		2011/1070055, 07
66750 Saint-Cyprien, Pyrénées-Orientales, France, 2010 (oats, Charmoise)	750 SL	32	1.50	198	72	2.0	1.6 *2.0*	2011/1070055, 08
15370 Vogelsdorf, Brandenburg,	750 SL	39	1.65	218	49	7.4	5.7	2011/1070055, 09

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Germany, 2010 (oats, Flämingsford)								
21769 Lamstedt, Niedersachsen, Germany, 2010 (oats, Atego)	750 SL	37	1.38	183	57	2.5	1.9	2011/1070055, 10
50491 Badules, Aragon, Spain, 2010 (oats, Blancanieves)	750 SL	32	1.55	207	58	1.7	1.3 *1.5*	2011/1070055, 11
32380 Bives, Gers, France, 2010 (oats, Charmoise)	750 SL	32	1.46	193	95	0.70	0.54 *0.68*	2011/1070055, 12
02640 Almansa, Albacete, Spain, 2011 (oats, Avena Roja)	750 SL	32	1.52	201	85	2.8	2.2 *2.7*	2012/1016107, 01
50491 Badules, Aragon, Spain, 2011 (oats, Prevision)	750 SL	32	1.34	203	69	2.7	2.1 *2.9*	2012/1016107, 02

No residues above the LOQ were found in any of the untreated control samples. Values in italics have been proportionally adjusted for application rate to match the Swiss GAP for oats.

Table 38 Residues of chlormequat chloride in rye after a single application

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
D-27449, Mulsum, Germany (winter rye, Askari)	750 SL	37	1.65	214	94	0.20	0.16 *0.22*	2010/1014090, 07
F-45300, Saint-Pryvé, Saint-Memin, France (winter rye, Conduct)	750 SL	37	1.52	197	90	2.6	2.0 *3.0*	2010/1014090, 08
50491 Badules, Aragon, Spain, 2010 (winter rye, Petkus)	750 SL	32	1.54	207	92	1.4	1.1 *1.6*	2011/1071894, 01

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
40016 Funo a Aruzato, Bologna, Italy, 2010 (rye, Fasto)	750 SL	32	1.50	198	75	1.1	0.85 *1.3*	2011/1071894, 02
27449 Mulsum, Niedersachsen, Germany, 2010 (winter rye, Guttino)*	750 SL	37	1.37	182	86	0.59 c0.24	0.46 c0.19	2011/1071894, 03
16321 Bernau, Brandeburg, Germany, 2010 (rye, Conduct)	750 SL	37	1.60	212	85	0.34	0.26 *0.37*	2011/1071894, 04
15370 Fredersdorf, Brandenburg, Germany, 2010 (rye, Recrut)	750 SL	37	1.64	217	77	0.67	0.52 *0.71*	2011/1071894, 05
21769 Lamstedt, Niedersachsen, Germany, 2010 (winter rye, Recrut)	750 SL	37	1.51	200	84	0.38	0.29 *0.43*	2011/1071894, 06
21210 Montlay en Auxois, Cote d'Or, France, 2010 (winter rye, Triskel)	750 SL	37	1.33	202	86	0.32	0.25 *0.42*	2011/1071894, 07
68320 Muntzenheim, Alsace, France, 2010 (rye, Nikita)	750 SL	37	1.51	200	82	0.94	0.73 *1.1*	2011/1071894, 08
56250 Elven, Bretagne, France, 2010 (rye, Askani)	750 SL	37	1.66	220	83	1.0	0.78 *1.1*	2011/1071894, 09
38510 Vézeronce-Curtin, France, 2010 (rye, Dukato)	750 SL	32	1.39	210	85	0.51	0.40 *0.65*	2011/1071894, 10
01190 Ressouze, Ain, France, 2010 (rye, Triskol)	750 SL	32	1.28	195	94	0.84	0.65 *1.1*	2011/1071894, 11
50491 Badules, Aragon, Spain, 2011 (rye, Petkus)	750 SL	32	1.22	185	84	1.9	1.5 *2.8*	2012/1016108, 01

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
50367 Retascon, Aragon, Spain, 2011 (rye, Ascary)	750 SL	32	1.39	210	76	0.89	0.69 *1.1*	2012/1016108, 02
01190 Ressouze, Ain, France, 2011 (rye, Fugato)	750 SL	32	1.47	195	87	2.8 c0.17	2.2 c0.13 *3.4*	2012/1016108, 03
38510 Sermerieu, Iscre, France, 2011 (rye, Rotego)	750 SL	32	1.53	203	92	1.2	0.93 *1.4*	2012/1016108, 04

Except where noted, no residues above the LOQ were found in any of the untreated control samples. Values in italics have been proportionally adjusted for application rate to match the Latvian GAP for rye.

*Trial site accidentally oversprayed with an additional application of chlormequat chloride.

Table 39 Residues of chlormequat chloride in wheat

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Brunne, Germany (winter wheat, Thasos)	460 SL	37	1.52	150	94	0.33	0.26 0.35	2005/1014176, ACK/03/04
	750 SL	37	1.50	150	94	0.45	0.35 0.47	
Seebach, northern France (winter wheat, Cap Horn)	460 SL	34	1.52	150	68	0.74	0.57 0.76	2005/1014176, FAN/03/04
	750 SL	34	1.50	150	68	0.73	0.57 0.77	
Aussonne, southern France (winter wheat, Autan)	460 SL	35	1.52	150	80	0.44	0.34 0.45	2005/1014176, FTL/03/04
	750 SL	34	1.50	150	80	0.62	0.48 0.65	
Withington, UK (spring wheat, Paragon)	460 SL	37	1.52	150	78	0.80	0.62 0.83	2005/1014176, OAT/01/04
	750 SL		1.50	150	78	0.76	0.59 0.80	
D-75233, Niefern-Öschelbronn, Germany (winter wheat, Tores)	750 SL	37	1.67	195	84	0.62	0.48 0.58	2010/1014090, 01
D-71277, Perouse-Rutesheim,	750 SL	37	1.40	163	98	0.30	0.23 0.33	2010/1014090, 02

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Germany (winter wheat, Tommi)								
F-45300, Rouvres-Saint-Jean, France (winter wheat, Campero)	750 SL	37	1.57	204	84	0.96	0.74 0.95	2010/1014090, 03
F-45300, Bouilly-en-Gâtinais, France (winter wheat, Apache)	750 SL	37	1.58	206	71	0.47	0.36 0.46	2010/1014090, 04
North Cave, East Yorkshire, UK (winter wheat, Oakley)[a]	750 SL	37	1.56	203	75	1.3 c0.94	1.0 c0.73	2010/1041090, 05
74193 Stetten a. H. Rieslingstrasse 18, Baden-Württemburg, Germany, 2003 (winter wheat, Transit)	350 SL	37	0.70	100	57	0.26	0.20 0.58	2004/1015956, 01
	750 SL	37	1.50	100	57	0.20	0.16 0.22	
82170 Pompignan 30 route de Toulouse, Midi-Pyrenées, France, 2003 (winter wheat, Sagem)[b]	350 SL	39	0.70	100	50	4.6	3.6 10.4	2004/1015956, 05
	750 SL		1.50	100	51	7.9	6.1 8.2	
D-47652 Weeze, Nordrhein-Westfalen, Germany, 2007 (spring wheat, Taifun)	750 SL	32	1.54	200	79	1.3	1.0 1.3	2008/1014941, 01
NL-6595, MS Ottersum, Limburg, The Netherlands, 2007 (winter wheat, Limos)	750 SL	32	1.62	210	75	0.88	0.68 0.85	2008/1014941, 02
F-12290, Aveyron, France, 2007 (spring wheat,	750 SL	37	1.00	195	98	0.21	0.16 0.32	2008/1014941, 03

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Florence Aurore)								
F-82100 Tarn et Garonne, France, 2007 (winter wheat, Apache)	750 SL	33	1.04	202	85	0.39	0.30 0.58	2008/1014941, 04
I-40068 Emilia Romagna, Italy, 2007 (spring wheat, Lippo)	750 SL	32	1.05	204	98	0.44	0.34 0.66	2008/1014941, 05
I-40054 Emilia Romagna, Italy, 2007 (winter wheat, Duilio)	750 SL	32	1.07	208	96	0.06	0.05 0.09	2008/1014941, 06
Via Calabria Nuovo No. 3, Quarto Inferiore, 40057 Bologna, Italy, 2007 (spring wheat, Croine)	750 SL	32	1.55	201	87	0.10	0.078 0.10	2008/1014940, 01
Castel S. Pietro, 40024 Bologna, Italy, 2007 (durum wheat, San Carlo)	750 SL	32	1.56	202	99	0.07	0.05 0.06	2008/1014940, 02
82000 Montauban, France, 2007 (winter wheat, Quality)	750 SL	32	1.48	192	65	0.07	0.05 0.07	2008/1014940, 03
82700 Finhan, France, 2007 (durum wheat, Joyaux)	750 SL	37	1.57	204	72	0.61	0.47 0.61	2008/1014940, 04
Granarolo dell'Emilia, 40057, Emilia Romagna, Italy, 2008 (spring wheat, Blasco)	750 SL	33	1.56	202	62	< 0.05	< 0.04	2009/1021674, 01
V. Matteotti 13, Molinella, Bologna 40062, Italy, 2008 (durum wheat, Duilio)	750 SL	32	1.52	198	96	< 0.05	< 0.04	2009/1021674, 02
Barry d'Islemade, 82000 Tarn et Garonne, France, 2008	750 SL	32	1.57	204	95	0.14	0.11 0.14	2009/1021674, 03

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
(winter wheat, Quality)								
Finhan, 82700 Tarn et Garonne, France, 2008 (durum wheat, Dakter)	750 SL	32	1.55	201	106	0.73	0.57 0.74	2009/1021674, 04
Herbert Neumann Dorfstr. 2, 16833 Brunne, Germany, 2004 (winter wheat, Thasos)	460 SL	37	1.52	150	94	0.33	0.26 0.35	2005/1014176, 01
	750 SL	37	1.50	150	94	0.45	0.35 0.47	
30 route de Hunspach, 67160 Seebach, France, 2004 (winter wheat, Cap Horn)	460 SL	34	1.52	150	68	0.74 c0.15	0.57 c0.12 0.76	2005/1014176, 02
	750 SL	34	1.50	150	68	0.73 c0.15	0.57 c0.12 0.77	
Ourmieres 3529, route de Merville 31840 Aussonne, France, 2004 (winter wheat Autan)	460 SL	35	1.52	150	80	0.44	0.34 0.45	2005/1014176, 03
	750 SL	35	1.50	150	80	0.62	0.48 0.65	
Upcote Farm, Withington, GL54 4BL, UK, 2004 (spring wheat, Paragon)	460 SL	37	1.52	150	78	0.80	0.62 0.83	2005/1014176, 04
	750 SL	37	1.50	150	78	0.76	0.59 0.80	

Except where indicated, no residues above the LOQ were found in any of the untreated control samples. Values in italics have been proportionally adjusted for application rate to match the Argentine GAP for wheat.

[a] Trial accidentally oversprayed with an additional application of 1.6 kg ai/ha chlormequat chloride 18 days prior to the trial application.

[b] Trial flagged by applicant as having abnormally high residues due to extremely low rainfall during the trial, contributing to lowered yields, and use of a durum wheat variety. Noting that this result differs significantly from the rest of the data, it is considered not representative of the residues expected after treatment in accordance with GAP, and has not been included the consideration for MRL estimation.

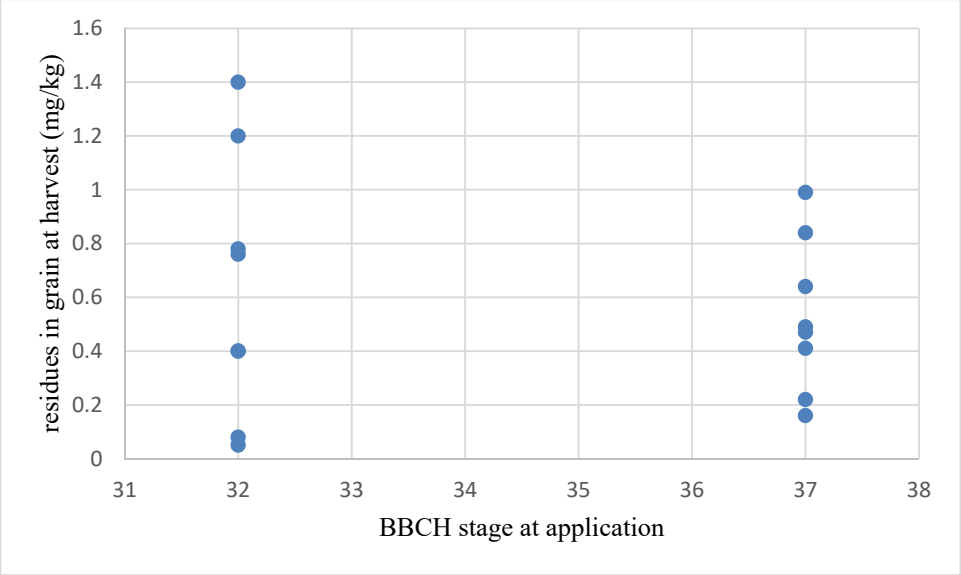


Figure 1 Dependence of residues in barley grain at harvest on crop growth stage at application

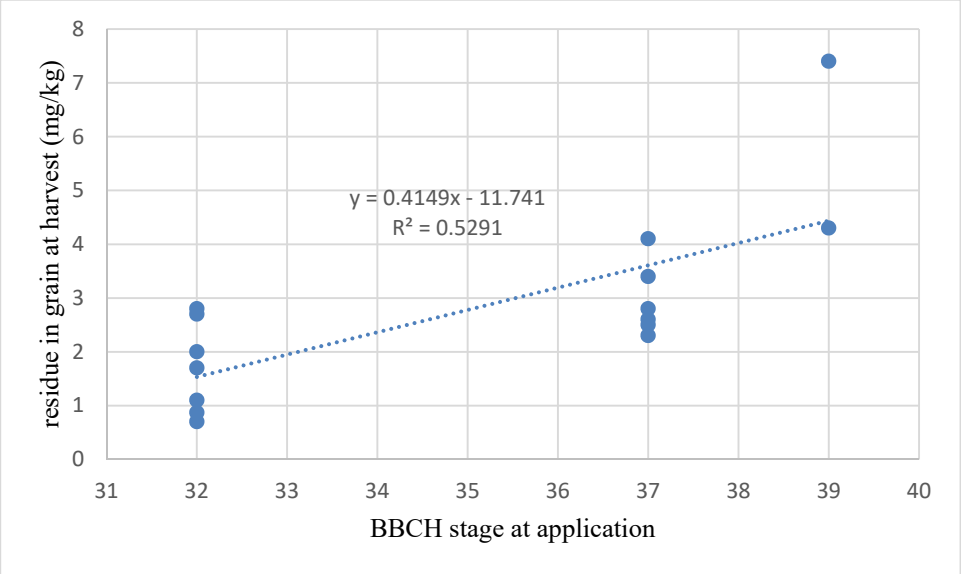


Figure 2 Dependence of residues in oat grain at harvest on crop growth stage at application

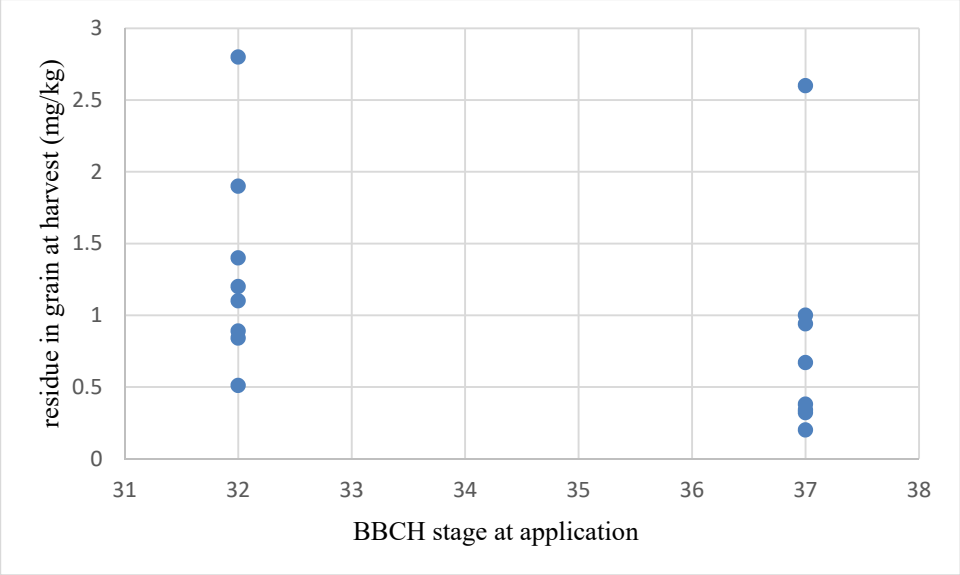


Figure 3 Dependence of residues in rye grain at harvest on crop growth stage at application

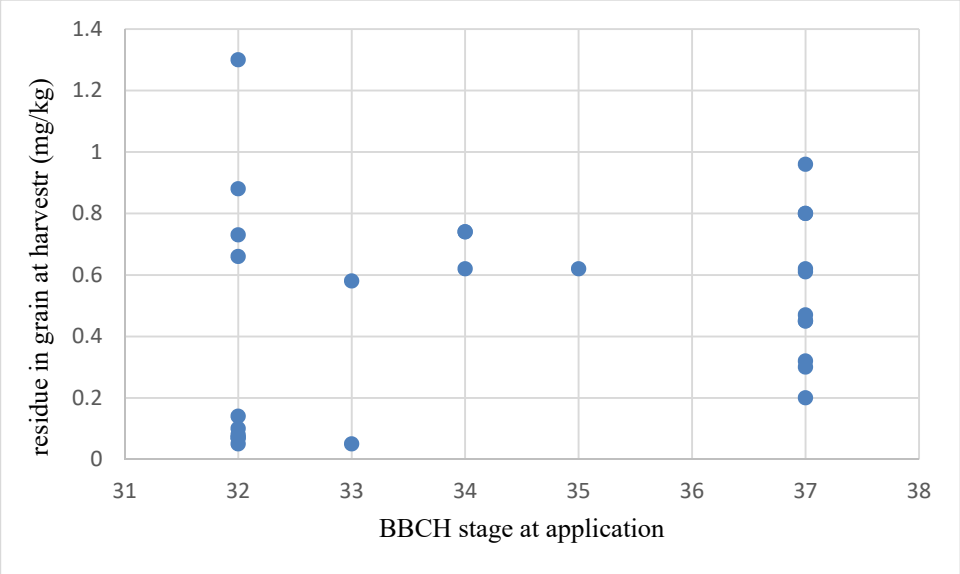


Figure 4 Dependence of residues in wheat grain at harvest on crop growth stage at application

Forage of cereal grains

Table 40 Residues of chlormequat chloride in barley forage after a single application (results reported on a fresh weight basis)

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
F-91150 Erzeville, Roinvillers, France, 2009 (spring barley,	750 SL	37	1.7	219	0	Whole plant w/o roots	27	21	2010/1014090, 06

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
Sebastian)									
					14	Whole plant w/o roots	16	12	
					28	Whole plant w/o roots	12	9.3	
					42	Ear/ panicle	0.37	0.29	
					42	Rest of plant w/o roots	13	10	
50180 Utebo, Zaragoza, Spain, 2010 (barley, Graphic)	750 SL	32	1.4	182	0	Whole plant w/o roots	28	22	2011/1071895, 01
					14	Whole plant w/o roots	2.5	1.9	
					28	Whole plant w/o roots	0.70	0.54	
					42	Ear/panicle	0.24	0.19	
					42	Rest of plant w/o roots	1.3	1.0	
66750 Saint-Cyprien, Pyrénées-Orientales, France, 2010 (barley, Prestige)	750 SL	32	1.6	207	0	Whole plant w/o roots	33	26	2011/1071895, 02
					14	Whole plant w/o roots	8.6	6.7	
					28	Whole plant w/o roots	7.3	5.7	
					41	Ear/ panicle	0.93	0.72	
					41	Rest of plant	14	11	
21737 Wischhafen, Niedersachsen, Germany, 2011 (winter barley, Pelikan)	750 SL	37	1.5	202	0	Whole plant w/o roots	19	15	2012/1016109, 01
					14	Whole plant w/o roots	3.8	2.9	
					28	Whole plant w/o roots	2.8	2.2	
					42	Ear/ panicle	0.41	0.32	
					42	Rest of plant w/o roots	3.3	2.6	
21726 Oldendorf, Niedersachsen, Germany, 2011 (winter barley, Naomie)	750 SL	37	1.6	211	0	Whole plant w/o roots	24	19	2012/1016109, 02
45300 Thignonville, Loiret, France, 2011 (spring	750 SL	37	1.5	202	0	Whole plant w/o roots	38	29	2012/1016109, 03

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
barley, Sebastian)									
					14	Whole plant w/o roots	4.6	3.6	
					27	Whole plant w/o roots	2.5	1.9	
					42	Ear/ panicle	0.20	0.16	
					42	Rest of plant w/o roots	1.8	1.4	
91150 Mespuits, Essonne, France, 2011 (spring barley, Sebastian)	750 SL	37	1.4	190	0	Whole plant w/o roots	24	19	2012/1016109, 04
82130 Lafrancaise, Midi P., France, 2011 (winter barley, Azurel)	750 SL	32	1.6	220	0	Whole plant w/o roots	41	32	2012/1016109, 05
					15	Whole plant w/o roots	4.3	3.3	
					29	Whole plant w/o roots	2.1	1.6	
					42	Ear/ panicle	0.35	0.27	
					42	Rest of plant	2.6	2.0	
82700 Bourret, Tarn et Garonne, France, 2011 (winter barley, Azurel)	750 SL	32	1.4	181	0	Whole plant w/o roots	30	23	2012/1016109, 06
44492 Fonfria, Teruel, Spain, 2011 (barley, Estrelia)	750 SL	32	1.5	200	0	Whole plant w/o roots	96	74	2012/1016109, 07
					13	Whole plant w/o roots	4.8	3.7	
					28	Whole plant w/o roots	2.0	1.6	
					42	Ear/panicle	0.69	0.54	
					42	Rest of plant	0.92	0.71	
22809 Loarre, Aragon, Spain, 2011 (barley, Meseta)	750 SL	32	1.5	200	0	Whole plant w/o roots	30	23	2012/1016109, 08
67229 Gerolsheim Römerstrasse 8, Rheinland-Pfalz, Germany, 2003 (spring	350 SL	37	0.70	100	0	Whole plant w/o roots	46.4	36	2004/1015956, 02

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
barley, Scarlett)									
					29	Ears	0.94	0.73	
					29	Rest of plant	4.7	3.6	
	750 SL	37	1.5	100	0	Whole plant w/o roots	32.4	25	
					29	Ears	0.62	0.48	
					29	Rest of plant	4.3	3.3	
Homelands Farm, Bucknell, Bicester, OX6 9NB, UK, 2003 (winter barley, Leonie)	350 SL	37	0.70	100	0	Whole plant w/o roots	35.1	27	2004/1015956, 03
					30	Ears	0.44	0.34	
					30	Rest of plant	4.1	3.2	
	750 SL	37	1.5	100	0	Whole plant w/o roots	41.8	32	
					30	Ears	0.42	0.33	
					30	Rest of plant	4.3	3.3	
67160 Seebach route de Hunspach, Alsace, France, 2003 (winter barley, Majestic)	350 SL	37	0.70	100	0	Whole plant w/o roots	18.1	14	2004/1015956, 04
					9	Ears	0.90	0.70	
					9	Rest of plant	5.1	4.0	
	750 SL	37	1.5	100	0	Whole plant w/o roots	27.5	21	
					9	Ears	0.44	0.34	
					9	Rest of plant	1.7	1.3	

Except where noted, no residues above the LOQ were found in any of the untreated control samples.

Table 41 Residues of chlormequat chloride in oat forage after a single application (results reported on a fresh weight basis)

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
F-45300 Yévre-la-Ville, France (winter oats, Expression)	750 SL	37	1.6	208	0	Whole plant w/o roots	22	17	2010/1014090, 09
D-21709, Burweg, Germany (spring oats,	750 SL	37	1.7	219	0	Whole plant w/o roots	63	49	2010/1014090, 10
					13	Whole plant w/o roots	13	10	

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
Freddy)					27	Whole plant w/o roots	5.6	4.3	
					41	Ear/panicle	4.0	3.1	
					41	Rest of plant w/o roots	6.0	4.7	
02690 Alpera, Albecete, Spain, 2010 (oats, Norlys)	750 SL	32	1.66	220	0	Whole plant w/o roots	40	31	2011/1070055, 01
					14	Whole plant w/o roots	5.4	4.2	
					28	Whole plant w/o roots	1.7	1.3	
					43	Ear/panicle	1.7	1.3	
					43	Rest of plant w/o roots	0.42	0.33	
40018 Maccaretolo, Italy, 2010 (oats, Argentina)	750 SL	32	1.4	182	0	Whole plant w/o roots	29	22	2011/1070055, 02
27109 Düdenbüttel, Niedersachsen, Germany, 2010 (oats, Dominik)	750 SL	37	1.5	200	0	Whole plant w/o roots	12	9.3	2011/1070055, 03
					13	Whole plant w/o roots	4.9	3.8	
					27	Whole plant w/o roots	2.6	2.0	
					41	Ear/panicle	2.7	2.1	
					41	Rest of plant	3.4	2.6	
16321 Bernau, Brandenburg, Germany, 2010 (oats, Flämingsford)	750 SL	39	1.7	221	0	Whole plant w/o roots	23	18	2011/1070055, 04
					14	Whole plant w/o roots	7.3	5.7	
					27	Whole plant w/o roots	3.0	2.3	
					42	Ear/panicle	4.1	3.2	
					42	Rest of plant w/o roots	4.7	3.6	
45300 Boynes, Loiret, France, 2010 (oats, Grafton Redigo)	750 SL	37	1.6	207	0	Whole plant w/o roots	29	22	2011/1070055, 05
					14	Whole plant w/o roots	7.7	6.0	
					28	Whole plant w/o roots	1.9	1.5	
					42	Ear/panicle	3.0	2.3	
					42	Rest of plant w/o roots	0.47	0.36	
68320 Muntzenheim, Alsace, France, 2010 (oats, Cornell)	750 SL	37	1.6	210	0	Whole plant w/o roots	21	16	2011/1070055, 06
82290 Meauzac, Tarn et Garonne, France, 2010 (oats, Charmoise)	750 SL	32	1.48	196	0	Whole plant w/o roots	25	19	2011/1070055, 07
					15	Whole plant w/o roots	2.9	2.2	
					28	Whole plant w/o roots	0.95	0.74	
					43	Ear/panicle	0.58	0.45	
					43	Rest of plant	0.34	0.26	

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
66750 Saint-Cyprien, Pyrénées-Orientales, France, 2010 (oats, Charmoise)	750 SL	32	1.5	198	0	Whole plant w/o roots	43	33	2011/1070055, 08
15370 Vogelsdorf, Brandenburg, Germany, 2010 (oats, Flämingsford)	750 SL	39	1.6	218	0	Whole plant w/o roots	28	22	2011/1070055, 09
21769 Lamstedt, Niedersachsen, Germany, 2010 (oats, Atego)	750 SL	37	1.4	183	0	Whole plant w/o roots	12	9.3	2011/1070055, 10
50491 Badules, Aragon, Spain, 2010 (oats, Blancanieves)	750 SL	32	1.5	207	0	Whole plant w/o roots	40	31	2011/1070055, 11
32380 Bives, Gers, France, 2010 (oats, Charmoise)	750 SL	32	1.46	193	0	Whole plant w/o roots	30	23	2011/1070055, 12
					14	Whole plant w/o roots	6.7	5.2	
					27	Whole plant w/o roots	1.0	0.78	
					42	Ear/panicle	0.47	0.36	
					42	Rest of plant	0.26	0.20	
02640 Almansa, Albacete, Spain, 2011 (oats, Avena Roja)	750 SL	32	1.52	201	0	Whole plant w/o roots	55	43	2012/1016107, 01
					14	Whole plant w/o roots	8.2	6.4	
					27	Whole plant w/o roots	5.2	4.0	
					42	Ear/panicle	3.3	2.6	
					42	Rest of plant	1.0	0.78	
50491 Badules, Aragon, Spain, 2011 (oats, Prevision)	750 SL	32	1.3	203	0	Whole plant w/o roots	65	50	2012/1016107, 02

Except where noted, no residues above the LOQ were found in any of the untreated control samples.

Table 42 Residues of chlormequat chloride in rye forage after a single application (results reported on a fresh weight basis)

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
D-27449, Mulsum, Germany (winter rye, Askari)	750 SL	37	1.65	214	0	Whole plant w/o roots	40	31	2010/10140 90, 07
					13	Whole plant w/o roots	7.4	5.7	
					27	Whole plant w/o roots	4.8	3.7	
					41	Ear/panicle	0.15	0.12	
					41	Rest of plant w/o roots	2.8	2.2	
F-45300, Saint-Pryvé, Saint-Memin, France (winter rye, Conduct)	750 SL	37	1.52	197	0	Whole plant w/o roots	13	10	2010/10140 90, 08
50491 Badules, Aragon, Spain, 2010 (winter rye, Petkus)	750 SL	32	1.54	207	0	Whole plant w/o roots	16	12	2011/10718 94, 01
					14	Whole plant w/o roots	3.0	2.3	
					28	Whole plant w/o roots	1.3	1.0	
					42	Ear/panicle	0.39	0.30	
					42	Rest of plant	2.0	1.6	
40016 Funo a Aruzato, Bologna, Italy, 2010 (rye, Fasto)	750 SL	32	1.50	198	0	Whole plant w/o roots	17	13	2011/10718 94, 02
27449 Mulsum, Niedersachsen, Germany, 2010 (winter rye, Guttino)[a]	750 SL	37	1.37	182	0	Whole plant w/o roots	40 c6.4	31 c5.0	2011/10718 94, 03
					15	Whole plant w/o roots	8.6	6.7	
					29	Whole plant w/o roots	3.5	2.7	
					42	Ear/panicle	0.05	0.04	
					42	Rest of plant w/o roots	8.3	6.4	
16321 Bernau, Brandeburg, Germany, 2010 (rye, Conduct)	750 SL	37	1.60	212	0	Whole plant w/o roots	8.6	6.7	2011/10718 94, 04
					15	Whole plant w/o roots	3.1	2.4	
					29	Whole plant w/o roots	2.8	2.2	
					42	Ear/panicle	< 0.05	< 0.04	
					42	Rest of plant w/o roots	3.3	2.6	
15370 Fredersdorf, Brandenburg, Germany, 2010 (rye, Recrut)	750 SL	37	1.64	217	0	Whole plant w/o roots	19	15	2011/10718 94, 05
21769 Lamstedt,	750 SL	37	1.51	200	0	Whole plant w/o roots	31	24	2011/10718 94, 06

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
Niedersachsen, Germany, 2010 (winter rye, Recrut)									
21210 Montlay en Auxois, Cote d'Or, France, 2010 (winter rye, Triskel)	750 SL	37	1.33	202	0	Whole plant w/o roots	9.9	7.7	2011/1071894, 07
					14	Whole plant w/o roots	2.2	1.7	
					29	Whole plant w/o roots	2.4	1.9	
					42	Ear/panicle	< 0.05	< 0.04	
					42	Rest of plant	2.7	2.1	
68320 Muntzenheim, Alsace, France, 2010 (rye, Nikita)	750 SL	37	1.51	200	0	Whole plant w/o roots	15	12	2011/1071894, 08
56250 Elven, Bretagne, France, 2010 (rye, Askani)	750 SL	37	1.66	220	0	Whole plant w/o roots	9.5	7.4	2011/1071894, 09
38510 Vézeronce-Curtin, France, 2010 (rye, Dukato)	750 SL	32	1.39	210	0	Whole plant w/o roots	23	18	2011/1071894, 10
01190 Ressouze, Ain, France, 2010 (rye, Triskol)	750 SL	32	1.28	195	0	Whole plant w/o roots	29	22	2011/1071894, 11
					14	Whole plant w/o roots	3.6	2.8	
					28	Whole plant w/o roots	1.4	1.1	
					42	Ear/panicle	0.15	0.1	
					42	Rest of plant w/o roots	1.2	0.9	
50491 Badules, Aragon, Spain, 2011 (rye, Petkus)	750 SL	32	1.22	185	0	Whole plant w/o roots	70	54	2012/1016108, 01
					15	Whole plant w/o roots	8.9	6.9	
					29	Whole plant w/o roots	9.2	7.1	
					41	Ear/panicle	2.2	1.7	
					41	Rest of plant w/o roots	3.5	2.7	
50367 Retascon, Aragon, Spain, 2011 (rye, Ascary)	750 SL	32	1.39	210	0	Whole plant w/o roots	52	40	2012/1016108, 02
01190 Ressouze,	750 SL	32	1.47	195	0	Whole plant w/o roots	53	41	2012/1016108, 03

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
Ain, France, 2011 (rye, Fugato)					14	Whole plant w/o roots	14	11	
					28	Whole plant w/o roots	3.9	3.0	
					42	Ear/panicle	3.9	3.0	
					42	Rest of plant	3.7	2.9	
38510 Sermerieu, Iscre, France, 2011 (rye, Rotego)	750 SL	32	1.53	203	0	Whole plant w/o roots	46	36	2012/1016108, 04

Except where noted, no residues above the LOQ were found in any of the untreated control samples.

[a] Trial site accidentally oversprayed with an additional application of chlormequat chloride.

Table 43 Residues of chlormequat chloride in wheat forage after a single application (results reported on a fresh weight basis)

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
D-75233, Niefern-Öschelbronn, Germany (winter wheat, Tores)	750 SL	37	1.67	195	0	Whole plant w/o roots	26	20	2010/1014090, 01
					14	Whole plant w/o roots	5.5	4.3 *5.2*	
					28	Whole plant w/o roots	4.6	3.6	
					42	Ear/panicle	0.28	0.22	
					42	Rest of plant w/o roots	6.4	5.0	
D-71277, Perouse-Rutesheim, Germany (winter wheat, Tommi)	750 SL	37	1.40	163	0	Whole plant w/o roots	30	23	2010/1014090, 02
F-45300, Rouvres-Saint-Jean, France (winter wheat, Campero)	750 SL	37	1.57	204	0	Whole plant w/o roots	44	34	2010/1014090, 03
F-45300, Bouilly-en-Gâtinais, France (winter wheat, Apache)	750 SL	37	1.58	206	0	Whole plant w/o roots	59	46	2010/1014090, 04
North Cave, East Yorkshire, UK (winter wheat, Oakley)[a]	750 SL	37	1.56	203	0	Whole plant w/o roots	78 c46	60 c36	2010/1041090, 05
					15	Whole plant w/o roots	42	33	
					27	Whole plant w/o roots	24	19	
					41	Ear/panicle	0.57 c0.43	0.44 c0.33	

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
					41	Rest of plant w/o roots	23 c21	18 c16	
74193 Stetten a. H. Rieslingstrasse 18, Baden-Württemburg, Germany, 2003 (winter wheat, Transit)	350 SL	37	0.70	100	0	Whole plant w/o roots	15.2	12	2004/1015956, 01
					18	Ear	0.20	0.16	
					18	Rest of plant w/o roots	7.2	5.6	
	750 SL	37	1.50	100	0	Whole plant w/o roots	20.9	16	
					18	Ear	0.73	0.57	
					18	Rest of plant w/o roots	8.5	6.6	
82170 Pompignan 30 route de Toulouse, Midi-Pyrenées, France, 2003 (winter wheat, Sagem)[b]	350 SL	39	0.70	100	0	Whole plant w/o roots	10.5	8.1	2004/1015956, 05
					8	Ear	26.9	21	
					8	Rest of plant w/o roots	13.6	11	
	750 SL		1.50	100	0	Whole plant w/o roots	27.2	21	
					9	Ear	49.4	38	
					9	Rest of plant w/o roots	28.7	22	
D-47652 Weeze, Nordrhein-Westfalen, Germany, 2007 (spring wheat, Taifun)	750 SL	32	1.54	200	0	Whole plant w/o roots	80	62	2008/1014941, 01
					15	Whole plant w/o roots	10	7.8 *10*	
					28	Whole plant w/o roots	4.2	3.3	
					42	Ear	1.3	1.0	
					42	Rest of plant w/o roots	6.0	4.7	
I-40068 Emilia Romagna, Italy, 2007 (spring wheat, Lippo)	750 SL	32	1.05	204	0	Whole plant w/o roots	52	40	2008/1014941, 05
					14	Whole plant w/o roots	17	13 *25*	
					28	Whole plant w/o roots	5.4	4.2	
					42	Ear	1.1	0.85	
					42	Rest of plant w/o roots	0.47	0.36	
Via Calabria Nuovo No. 3, Quarto Inferiore, 40057 Bologna, Italy, 2007 (spring wheat, Croine)	750 SL	32	1.55	201	0	Whole plant w/o roots	126	98	2008/1014940, 01
					14	Whole plant w/o roots	5.7	4.4 *5.7*	
					29	Whole plant w/o roots	0.36	0.28	
					42	Ear	0.08	0.06	
					42	Rest of plant w/o roots	0.12	0.09	
Granarolo	750 SL	33	1.56	202	0	Whole plant	60	47	2009/10216

Location (variety)	Application					Sample	Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days				
dell'Emilia, 40057, Emilia Romagna, Italy, 2008 (spring wheat, Blasco)						w/o roots			74, 01
					14	Whole plant w/o roots	8.6	6.7 *8.7*	
					29	Whole plant w/o roots	0.27	0.21	
					42	Ear/panicle	0.37	0.29	
					42	Rest of plant	1.5	1.2	
V. Matteotti 13, Molinella, Bologna 40062, Italy, 2008 (durum wheat, Duilio)	750 SL	32	1.52	198	0	Whole plant w/o roots	68	53	2009/10216 74, 02
Barry d'Islemade, 82000 Tarn et Garonne, France, 2008 (winter wheat, Quality)	750 SL	32	1.57	204	0	Whole plant w/o roots	30	23	2009/10216 74, 03
Finhan, 82700 Tarn et Garonne, France, 2008 (durum wheat, Dakter)	750 SL	32	1.55	201	0	Whole plant w/o roots	27	21	2009/10216 74, 04

Except where indicated, no residues above the LOQ were found in any of the untreated control samples. Values in italics have been proportionally adjusted by application rate to match the Argentine GAP.

[a] Trial accidentally oversprayed with an additional application of 1.6 kg ai/ha chlormequat chloride 18 days prior to the trial application.

[b] Trial flagged by applicant as having abnormally high residues due to extremely low rainfall during the trial, contributing to lowered yields, and use of a durum wheat variety.

Straws and fodders of cereal grains

Table 44 Residues of chlormequat chloride in barley straw after a single application (results reported on an as-is basis)

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
F-91150 Erzeville, Roinvillers, France, 2009 (spring barley, Sebastian)	750 SL	37	1.7	219	76	34	26	2010/1014090, 06
50180 Utebo, Zaragoza, Spain, 2010 (barley, Graphic)	750 SL	32	1.4	182	69	0.80	0.62	2011/1071895, 01
66750 Saint-Cyprien, Pyrénées-Orientales,	750 SL	32	1.6	207	59	39	30	2011/1071895, 02

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
France, 2010 (barley, Prestige)								
50490 Villareal de Huerva, Spain, 2010 (barley, Montage)	750 SL	32	1.5	200	70	1.6	1.2	2011/1071895, 03
01560 St-Jean-sur-Reyssouze, Ain, France, 2010 (barley, Vanessa)	750 SL	32	1.4	187	84	1.6	1.2	2011/1071895, 04
21737 Wischhafen, Niedersachsen, Germany, 2011 (winter barley, Pelikan)	750 SL	37	1.5	202	76	6.7	5.2	2012/1016109, 01
21726 Oldendorf, Niedersachsen, Germany, 2011 (winter barley, Naomie)	750 SL	37	1.6	211	76	7.1	5.5	2012/1016109, 02
45300 Thignonville, Loiret, France, 2011 (spring barley, Sebastian)	750 SL	37	1.5	202	67	3.5	2.7	2012/1016109, 03
91150 Mespuits, Essonne, France, 2011 (spring barley, Sebastian)	750 SL	37	1.4	190	68	4.1	3.2	2012/1016109, 04
82130 Lafrancaise, Midi P., France, 2011 (winter barley, Azurel)	750 SL	32	1.6	220	73	< 0.5	< 0.39	2012/1016109, 05
82700 Bourret, Tarn et Garonne, France, 2011 (winter barley, Azurel)	750 SL	32	1.4	181	70	3.3	2.6	2012/1016109, 06
44492 Fonfria, Teruel, Spain, 2011 (barley, Estrelia)	750 SL	32	1.5	200	75	2.4	1.9	2012/1016109, 07
22809 Loarre, Aragon, Spain, 2011	750 SL	32	1.5	200	72	7.6	5.9	2012/1016109, 08

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
(barley, Meseta)								
67229 Gerolsheim Römerstrasse 8, Rheinland-Pfalz, Germany, 2003 (spring barley, Scarlett)	350 SL	37	0.70	100	55	8.7	6.7	2004/1015956, 02
	750 SL	37	1.5	100	55	7.3	5.7	
Homelands Farm, Bucknell, Bicester, OX6 9NB, UK, 2003 (winter barley, Leonie)	350 SL	37	0.70	100	75	5.8	4.5	2004/1015956, 03
	750 SL	37	1.5	100	75	9.1	7.1	
67160 Seeback route de Hunspach, Alsace, France, 2003 (winter barley, Majestic)	350 SL	37	0.70	100	58	6.6	5.1	2004/1015956, 04
	750 SL	37	1.5	100	58	5.2	4.0	

Except where noted, no residues above the LOQ were found in any of the untreated control samples.

Table 45 Residues of chlormequat chloride in oat straw after a single application (results reported on an as-is basis)

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
F-45300 Yévre-la-Ville, France (winter oats, Expression)	750 SL	37	1.6	208	79	4.1	3.2	2010/1014090, 09
D-21709, Burweg, Germany (spring oats, Freddy)	750 SL	37	1.7	219	65	6.0	4.7	2010/1014090, 10
02690 Alpera, Albecete, Spain, 2010 (oats, Norlys)	750 SL	32	1.66	220	69	1.2	0.93 *1.0*	2011/1070055, 01
40018 Maccaretolo, Italy, 2010 (oats, Argentina)	750 SL	32	1.37	182	76	< 0.20	< 0.16	2011/1070055, 02

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
27109 Düdenbüttel, Niedersachsen, Germany, 2010 (oats, Dominik)	750 SL	37	1.51	200	46	2.9	2.2	2011/1070055, 03
16321 Bernau, Brandenburg, Germany, 2010 (oats, Flämingsford)	750 SL	39	1.67	221	52	6.5	5.0	2011/1070055, 04
45300 Boynes, Loiret, France, 2010 (oats, Grafton Redigo)	750 SL	37	1.56	207	67	4.7	3.6	2011/1070055, 05
68320 Muntzenheim, Alsace, France, 2010 (oats, Cornell)	750 SL	37	1.59	210	60	1.1	0.85	2011/1070055, 06
82290 Meauzac, Tarn et Garonne, France, 2010 (oats, Charmoise)	750 SL	32	1.5	196	N/A	Trial accidentally harvested prior to sampling	NA	2011/1070055, 07
66750 Saint-Cyprien, Pyrénées-Orientales, France, 2010 (oats, Charmoise)	750 SL	32	1.50	198	72	4.5	3.5 *4.3*	2011/1070055, 08
15370 Vogelsdorf, Brandenburg, Germany, 2010 (oats, Flämingsford)	750 SL	39	1.65	218	49	11	8.5	2011/1070055, 09
21769 Lamstedt, Niedersachsen, Germany, 2010 (oats, Atego)	750 SL	37	1.38	183	57	2.5	1.9	2011/1070055, 10
50491 Badules, Aragon, Spain, 2010 (oats, Blancanieves)	750 SL	32	1.55	207	56	3.1	2.4 *2.8*	2011/1070055, 11
32380 Bives, Gers, France, 2010 (oats, Charmoise)	750 SL	32	1.46	193	95	0.56	0.43 *0.54*	2011/1070055, 12

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
02640 Almansa, Albacete, Spain, 2011 (oats, Avena Roja)	750 SL	32	1.52	201	85	2.3	1.8 *2.2*	2012/1016107, 01
50491 Badules, Aragon, Spain, 2011 (oats, Prevision)	750 SL	32	1.34	203	69	0.50	0.39 *0.54*	2012/1016107, 02

Except where noted, no residues above the LOQ were found in any of the untreated control samples. Values in italics have been adjusted to match the Swiss GAP for oats.

Table 46 Residues of chlormequat chloride in rye straw after a single application (results reported on an as-is basis)

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
D-27449, Mulsum, Germany (winter rye, Askari)	750 SL	37	1.65	214	94	3.5	2.7 *3.7*	2010/1014090, 07
F-45300, Saint-Pryvé, Saint-Memin, France (winter rye, Conduct)	750 SL	37	1.52	197	90	7.8	6.0 *8.9*	2010/1014090, 08
50491 Badules, Aragon, Spain, 2010 (winter rye, Petkus)	750 SL	32	1.54	207	92	1.7	1.3 *1.9*	2011/1071894, 01
40016 Funo a Aruzato, Bologna, Italy, 2010 (rye, Fasto)	750 SL	32	1.50	198	75	0.72	0.56 *0.84*	2011/1071894, 02
27449 Mulsum, Niedersachsen, Germany, 2010 (winter rye, Guttino)[a]	750 SL	37	1.37	182	86	5.2 c1.2	4.0 c0.93	2011/1071894, 03
16321 Bernau, Brandeburg, Germany, 2010 (rye, Conduct)	750 SL	37	1.60	212	85	6.1	4.7 *6.6*	2011/1071894, 04
15370 Fredersdorf, Brandenburg,	750 SL	37	1.64	217	77	7.1	5.5 *7.5*	2011/1071894, 05

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Germany, 2010 (rye, Recrut)								
21769 Lamstedt, Niedersachsen, Germany, 2010 (winter rye, Recrut)	750 SL	37	1.51	200	84	1.4	1.1 *1.6*	2011/1071894, 06
21210 Montlay en Auxois, Cote d'Or, France, 2010 (winter rye, Triskel)	750 SL	37	1.33	202	86	4.3	3.3 *5.6*	2011/1071894, 07
68320 Muntzenheim, Alsace, France, 2010 (rye, Nikita)	750 SL	37	1.51	200	82	1.4	1.1 *1.6*	2011/1071894, 08
56250 Elven, Bretagne, France, 2010 (rye, Askani)	750 SL	37	1.66	220	83	4.8	3.7 *5.0*	2011/1071894, 09
38510 Vézeronce-Curtin, France, 2010 (rye, Dukato)	750 SL	32	1.39	210	85	3.4	2.6 *4.2*	2011/1071894, 10
01190 Ressouze, Ain, France, 2010 (rye, Triskol)	750 SL	32	1.28	195	94	1.6	1.2 *2.1*	2011/1071894, 11
50491 Badules, Aragon, Spain, 2011 (rye, Petkus)	750 SL	32	1.22	185	84	3.1	2.4 *4.2*	2012/1016108, 01
50367 Retascon, Aragon, Spain, 2011 (rye, Ascary)	750 SL	32	1.39	210	76	6.3	4.9 *7.9*	2012/1016108, 02
01190 Ressouze, Ain, France, 2011 (rye, Fugato)	750 SL	32	1.47	195	87	3.3	2.6 *4.0*	2012/1016108, 03
38510 Sermerieu, Iscre, France, 2011 (rye, Rotego)	750 SL	32	1.53	203	92	4.7	3.6 *5.3*	2012/1016108, 04

Except where noted, no residues above the LOQ were found in any of the untreated control samples. Values in italics have been proportionally adjusted for application rate in order to match the Latvian GAP for rye.

[a] Trial site accidentally oversprayed with an additional application of chlormequat chloride.

Table 47 Residues of chlormequat chloride in wheat straw (results reported on an as-is basis)

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Brunne, Germany (winter wheat, Thasos)	460 SL	37	1.52	150	94	26	20	2005/1014176, ACK/03/04
	750 SL	37	1.50	150	94	31	24 *32*	
Seebach, northern France (winter wheat, Cap Horn)	460 SL	34	1.52	150	68	4.1	3.2 *4.3*	2005/1014176, FAN/03/04
	750 SL	34	1.50	150	68	3.1	2.4	
Aussonne, southern France (winter wheat, Autan)	460 SL	35	1.52	150	80	27	21 *28*	2005/1014176, FTL/03/04
	750 SL	35	1.50	150	80	14	11	
Withington, UK (spring wheat, Paragon)	460 SL	37	1.52	150	78	14	11	2005/1014176, OAT/01/04
	750 SL	37	1.50	150	78	19	15 *20*	
D-75233, Niefern-Öschelbronn, Germany (winter wheat, Tores)	750 SL	37	1.67	195	84	8.1	6.3 *7.6*	2010/1014090, 01
D-71277, Perouse-Rutesheim, Germany (winter wheat, Tommi)	750 SL	37	1.40	163	98	9.4	7.3 *11*	2010/1014090, 02
F-45300, Rouvres-Saint-Jean, France (winter wheat, Campero)	750 SL	37	1.57	204	84	6.2	4.8 *6.2*	2010/1014090, 03
F-45300, Bouilly-en-Gâtinais, France (winter wheat, Apache)	750 SL	37	1.58	206	71	24	19 *24*	2010/1014090, 04
North Cave, East Yorkshire, UK (winter wheat, Oakley)[a]	750 SL	37	1.56	203	75	38 c28	29 c22	2010/1041090, 05

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
74193 Stetten a. H. Rieslingstrasse 18, Baden-Württemburg, Germany, 2003 (winter wheat, Transit)	350 SL	37	0.70	100	57	16.7	13 *37*	2004/1015956, 01
	750 SL	37	1.50	100	57	13.4	10 *14*	
82170 Pompignan 30 route de Toulouse, Midi-Pyrenées, France, 2003 (winter wheat, Sagem)[b]	350 SL	39	0.70	100	50	23.7	18 *53*	2004/1015956, 05
	750 SL	39	1.50	100	51	52.9	41 *55*	
D-47652 Weeze, Nordrhein-Westfalen, Germany, 2007 (spring wheat, Taifun)	750 SL	32	1.54	200	79	13	10 *13*	2008/1014941, 01
NL-6595, MS Ottersum, Limburg, The Netherlands, 2007 (winter wheat, Limos)	750 SL	32	1.62	210	75	9.5	7.4 *9.2*	2008/1014941, 02
F-12290, Aveyron, France, 2007 (spring wheat, Florence Aurore)	750 SL	37	1.00	195	98	10	7.8 *16*	2008/1014941, 03
F-82100 Tarn et Garonne, France, 2007 (winter wheat, Apache)	750 SL	33	1.04	202	85	4.2	3.3 *6.4*	2008/1014941, 04
I-40068 Emilia Romagna, Italy, 2007 (spring wheat, Lippo)	750 SL	32	1.05	204	98	1.9	1.5 *2.9*	2008/1014941, 05
I-40054 Emilia Romagna, Italy, 2007 (winter wheat, Duilio)	750 SL	32	1.07	208	96	< 0.50	< 0.39	2008/1014941, 06

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
Via Calabria Nuovo No. 3, Quarto Inferiore, 40057 Bologna, Italy, 2007 (spring wheat, Croine)	750 SL	32	1.55	201	87	< 0.50 (0.15)	< 0.39 (0.12)	2008/1014940, 01
Castel S. Pietro, 40024 Bologna, Italy, 2007 (durum wheat, San Carlo)	750 SL	32	1.56	202	99	< 0.50 (0.36)	< 0.39 (0.28)	2008/1014940, 02
82000 Montauban, France, 2007 (winter wheat, Quality)	750 SL	32	1.48	192	65	9.0	7.0 *9.6*	2008/1014940, 03
82700 Finhan, France, 2007 (durum wheat, Joyaux)	750 SL	37	1.57	204	72	16	12 *15*	2008/1014940, 04
Granarolo dell'Emilia, 40057, Emilia Romagna, Italy, 2008 (spring wheat, Blasco)	750 SL	33	1.56	202	62	< 0.50	< 0.39	2009/1021674, 01
V. Matteotti 13, Molinella, Bologna 40062, Italy, 2008 (durum wheat, Duilio)	750 SL	32	1.52	198	96	0.61	0.47 *0.63*	2009/1021674, 02
Barry d'Islemade, 82000 Tarn et Garonne, France, 2008 (winter wheat, Quality)	750 SL	32	1.57	204	95	4.1	3.2 *4.1*	2009/1021674, 03
Finhan, 82700 Tarn et Garonne, France, 2008 (durum wheat, Dakter)	750 SL	32	1.55	201	106	< 0.50 (0.32)	< 0.39 (0.25)	2009/1021674, 04
Herbert Neumann Dorfstr. 2, 16833 Brunne, Germany, 2004 (winter wheat, Thasos)	460 SL	37	1.52	150	94	26	20 *27*	2005/1014176, 01
	750 SL	37	1.50	150	94	31	24 *32*	

Location (variety)	Application					Residues, mg/kg chlormequat chloride	Residues, mg/kg chlormequat cation	Reference
	Form	Growth stage (BBCH)	Rate, kg ai/ha	Spray volume (L/ha)	PHI, days			
30 route de Hunspach, 67160 Seebach, France, 2004 (winter wheat, Cap Horn)	460 SL	34	1.52	150	68	4.1	3.2 *4.3*	2005/1014176, 02
	750 SL	34	1.50	150	68	3.1	2.4 *3.2*	
Ourmieres 3529, route de Merville 31840 Aussonne, France, 2004 (winter wheat Autan)	460 SL	35	1.52	150	80	27	21 *28*	2005/1014176, 03
	750 SL	35	1.50	150	80	15	12 *16*	
Upcote Farm, Withington, GL54 4BL, UK, 2004 (spring wheat, Paragon)	460 SL	37	1.52	150	78	14	11 *15*	2005/1014176, 04
	750 SL	37	1.50	150	78	19	15 *20*	

Except where indicated, no residues above the LOQ were found in any of the untreated control samples. Values in italics have been proportionally adjusted for application rate in order to match the Argentine GAP for wheat.

[a] Trial accidentally oversprayed with an additional application of 1.6 kg ai/ha chlormequat chloride 18 days prior to the trial application.

[b] Trial flagged by applicant as having abnormally high residues due to extremely low rainfall during the trial, contributing to lowered yields, and use of a durum wheat variety.

FATE OF RESIDUES IN PROCESSING

Effect of processing on the nature of residue

A study designed to simulate the effects of common food processing techniques relevant to cereal crops (beer brewing and baking) on the chemical nature of chlormequat chloride residues was carried out (Adam 2004, 2004/1027148). Two hydrolytic conditions were studied in order to simulate commercial processes (citrate buffer at pH 4, at 100 °C, for 120 minutes, simulating brewing, and citrate buffer at pH 5, at 100 °C, for 40 minutes, simulating baking). Test buffer solutions were fortified with ^{14}C-chlormequat chloride (labelled at both carbon atoms in the ethyl group), at 0.22 mg/L, then incubated at 100 °C using an oil bath. Total radioactivity was determined by liquid scintillation counting. The content of chlormequat chloride and any hydrolysis products was determined using TLC and HPLC.

Recoveries of radioactivity after incubation were essentially quantitative. TLC and HPLC analysis showed that slight increases of some unidentified components increased on incubation.

Table 48 Percentage composition of residues of ^{14}C-chlormequat chloride before and after hydrolysis under conditions simulating common food processing techniques

Component	Brewing (pH 4, 100 °C)		Baking (pH 5, 100 °C)	
	0 minutes	120 minutes	0 minutes	40 minutes
Chlormequat chloride	91.2	86.1	89.6	85.8
M1 (unknown)	3.5	3.0	2.9	2.6
M2 (unknown)	2.8	4.8	2.5	2.8
M3 (unknown)	1.3	3.3	ND	4.0
M4 (origin)	1.2	3.1	5.0	6.8

Residues of chlormequat chloride are largely unchanged after undergoing hydrolytic processes simulating brewing and baking.

Barley

A processing study in barley was conducted using samples treated, harvested and processed during the 2001 growing season in Germany (Zietz and Klimmek 2004a, 2004/1013831). At a single trial site, barley was treated with a single foliar broadcast application of a 750 g/L SL formulation of chlormequat chloride at BBCH stage 31, and with an application rate of 3.6 kg ai/ha. No other pesticides expected to interfere with the experiment were applied during the growing season.

Treated and control barley grain and straw were collected at normal harvest maturity, 71 days after application. Straw samples and grain samples intended for analysis without processing were frozen shortly after collection, while bulk grain samples for processing were kept at ambient temperatures for transport to the processing facility. After malting of the barley, the malt was kept at ambient temperatures until brewing.

Untreated samples of grain were processed before the treated samples. Barley was processed using simulated industrial processes.

To generate pot barley, firstly samples of unprocessed grain were collected and frozen, then raw grain was passed through a grain cleaner (sifter), then the cleaned grain and offal samples were collected. The moisture content of the cleaned grain was tested, and as it was between 10–15%, no moisture conditioning was required. Grain was then hulled using an abrasion mill, and the abraded pearl barley was passed through an elevator aspirator/sifter to remove adhering pearling dust. Samples of pearling dust and pot barley were then collected and frozen.

Malting was carried out in a pilot scale plant. The grain was first sorted using a grader consisting of two rotating cylinders to remove offal and grains smaller than 2.5 mm. Sorted grain was filled into 5 stainless steel cylinders each containing approximately 1.2 kg of grain. The steeping tank was then filled with fresh water at 18–22 °C and the barley was steeped inside the stainless steel cylinders using a program involving 2 hours soaking, then 15 hours aeration, 2 hours soaking, 23 hours aeration, and finally 2 hours soaking. The steeped grain was then placed, still in the cylinders, inside the germination box, where it was maintained at 14–16 °C for 97 hours, with agitation for 5 minutes every 3 hours. The germinated grain was then kilned, with a dwell time in the kiln of not more than 15 hours, and a temperature program involving steps at 55, 60, 70, and 80 °C, with 3-4 hours at each temperature step. Malt was then separated from the sprouts by rubbing over a sieve.

Beer was then brewed in a small scale plant simulating industrial processes as far as possible. Malt was first ground using a two-roller mill with adjustable spacing between the rollers. Grist (ground malt, approximately 3.5 kg) was mashed with stirring into 14 L of water at approximately 52 °C in a mashing tub. The mashing process was carried out over approximately 2.5 hours while the temperature was raised in steps from 52 to 76 °C. The mash was then drained off into a heated lautering tub. The wort and spent grain were separated by filtration with the spent grain being washed with 2 × 7.5 L aliquots of water. The wort and leachates were combined and transferred into a kettle heated by steam via a heat exchanger. The wort was boiled in the open kettle with collection of the condensate. A weighed portion of hop extract was suspended in a small portion of hot wort and added to the boiling kettle (7.5 g of hop extract by α-acids content to each 100 L wort). After 75–80 minutes

of boiling, the hot wort was drained into a whirlpool separator to separate the trub from the wort. Clear wort was pumped through a plate heat exchanger cooled using the brewery reticulated cooling water supply into a cooled fermentation vessel. The cooled clear wort (now at <20 °C) was seeded with 200 g of yeast slurry. The wort was pressure fermented (closed vessel) in the dark at approximately 10–13 °C for 6–8 days by which time the yeast had settled to the bottom and the fermentation was complete. The green beer was then drained off through the bottom valve into a 20 L stainless steel storage container, blanketed with carbon dioxide. The stainless steel container was then placed in cold storage (0 °C) for 6–11 days. The beer was then clarified by plate filtration, with the aid of diatomaceous earth. Finally, the beer was bottled with the aid of carbon dioxide to force the beer out of the stainless steel container.

Samples of processed barley commodities were frozen shortly after collection, with the exception of the malt retained for further processing into beer, and kept frozen until analysis. Samples were analysed with an LC-MS/MS method (CEN/TC 275/WG 4N) involving extraction with methanol/water and quantification using the internal standard method with a deuterated chlormequat internal standard (LOQ=0.05 mg/kg for straw, and 0.01 mg/kg for all other matrices). Acceptable concurrent recoveries were determined.

Raw grain and straw samples taken directly from the field site to the laboratory were analysed within 15 months of collection, while processed fractions were analysed within approximately 6, 9 or 12 months of collection (brewing, malting and pot barley processes respectively). Barley for processing was stored for up to 8 months at ambient temperatures, while malt for processing was stored at ambient temperatures for 2 months. Stability of the residues in the barley and malt stored at ambient temperatures before processing was verified by the comparable results between the raw barley frozen at the field site and the barley sampled just before processing after the ambient storage. Similarly, comparable results were obtained for the malt stored at ambient temperatures and sample just before commencement of brewing, and the malt sample frozen straight after completion of malting.

Low levels of residues of chlormequat chloride were observed in untreated control offal fractions and pearling dust, while no other control samples contained residues above the LOQ, although a number did contain residues above 30% of the LOQ.

Table 49 Residues of chlormequat chloride in processed fractions of barley from a site treated with a single application at BBCH 31

Location, Year (variety)	Application		Results	
	Rate, kg ai/ha	Spray volume (L/ha)	Sample	Residue (mg/kg chlormequat chloride)
D-23845 Grabau, Schleswig-Holstein, Germany, 2001 (barley, Barke)	3.6	305	Straw	17
			Grain	1.3
Balance study – Pot barley				
			Grain prior to processing	1.3
			Cleaned grain	1.3
			Offal	2.8 c0.04
			Pearling dust	3.5 c0.06
			Pot barley	1.2
Balance study – malting				
			Grain prior to processing	1.5
			Cleaned grain	1.6
			Offal	2.3 c0.04

Location, Year (variety)	Application		Results	
	Rate, kg ai/ha	Spray volume (L/ha)	Sample	Residue (mg/kg chlormequat chloride)
			Steeping water	< 0.01
			Malt spouts	3.2
			Malt	1.3
Balance study – brewing				
			Malt prior to brewing	1.3
			Spent grain	0.03
			Condensate	< 0.01
			Flocs	0.70
			Yeast	0.29
			Beer	0.19
Follow-up study 1 – pot barley				
			Grain prior to processing	1.3
			Pearling dust	4.1
			Pot barley	1.2
Follow-up study 1 – malting and brewing				
			Grain prior to processing	1.4
			Malt	1.3
			Malt prior to brewing	1.2
			Spent grain	0.02
			Flocs	0.84
			Beer	0.29
Follow-up study 2 – pot barley				
			Pearling dust	3.8
			Pot barley	1.2
Follow-up study 2 – malting and brewing				
			Grain prior to processing	1.0
			Malt	0.89
			Malt prior to brewing	0.92
			Spent grain	0.3
			Flocs	0.55
			Beer	0.16
Follow-up study 3 – pot barley				
			Pearling dust	4.1
			Pot barley	1.1
Follow-up study 3 – malting and brewing				
			Grain prior to processing	1.1
			Malt	1.0
			Malt prior to brewing	0.88
			Spent grain	0.02
			Flocs	0.71
			Beer	0.22

Except where otherwise noted, no untreated control samples contained residues above the LOQ.

Table 50 Processing factors for barley commodities

Processed fraction	Processing factor
Grain prior to processing	-
Cleaned grain (pot barley)	1.0
Offal (pot barley)	2.1
Pearling dust	2.7, 3.0, 3.3, 3.3
Pot barley	0.8, 0.9, 1.0, 1.0
Cleaned grain (malting)	1.1
Offal (malting)	1.6
Steeping water	0.01
Malt sprouts	2.2

Processed fraction	Processing factor
Malt	0.9, 0.9, 0.9, 1.0
Spent grain	0.01, 0.02, 0.02, 0.03
Condensate	0.01
Flocs	0.5, 0.5, 0.6, 0.6
Wort	0.1, 0.2, 0.2, 0.2
Yeast	0.2
Green beer	0.1, 0.2, 0.2, 0.2
Beer	0.1, 0.2, 0.2, 0.2

Residues of chlormequat chloride concentrate in offal, pearling dust, and malt sprouts, while they do not concentrate in cleaned barley grain, pot barley, beer, or any of the byproducts of malting and brewing other than the malt sprouts.

Oats

A processing study in oats was conducted using samples treated, harvested and processed during the 2003 growing season in Germany (Zietz 2004b, 2004/1013834). At a single trial site, oats were treated with a single foliar broadcast application of a 750 g/L SL formulation of chlormequat chloride at BBCH stage 49, and with an application rate of 3.6 kg ai/ha. No other pesticides expected to interfere with the experiment were applied during the growing season.

Treated and control oat forage was collected on the day of application, and grain and straw were collected at harvest maturity 67 days after application. Samples intended to be analysed without processing were frozen within an hour of collection, while bulk grain samples were collected for processing and kept at ambient temperatures for transport to the processing facility, where they were kept refrigerated.

Untreated samples were processed before the treated samples. Oats were processed using simulated industrial processes. At the beginning of the balance study, a sample of raw grain was collected for analysis. The moisture content of the grain was measured, and as it was < 15%, no adjustment of moisture content was necessary. The grain was cleaned using a grain cleaner, and samples of cleaned grain and offal collected. Grain fractions >2.5 mm and 2.2–2.5 mm were separately weighed and passed through the impact huller three times to remove the husks. The hull, kernel and grain fractions were separated after each passage and the hulls cleaned from the kernels using an elevator sifter. Oat dust was separated from the husks. Kernels, oat dust and husks were sampled. Hulled kernels were transferred to a conditioner for kilning and heated up to 110 °C and kept at this temperature for 60 minutes. The yield was determined, along with the moisture content. Kilned kernels were allowed to cool to 89–105 °C then steamed for 20 minutes, cooled to 80 °C and weighed. The moisture content was again determined. Steamed kernels were then rolled into flakes using a roller mill (gap between the rollers was set at 0.5 mm). Yield and moisture content were determined and the flakes were then transferred onto trays and dried in a controlled climate cabinet (45 °C, 40% RH, for 30 minutes). The flakes were then cooled, weighed, moisture content measured, and a sample of oat flakes collected.

In the follow-up studies, the same processing procedures were followed, with only raw oats and oat flakes being collected, as the most commercially important fractions, in order to gain more data on the partitioning of residues into these fractions.

Samples of processed oat commodities were frozen shortly after collection and kept frozen until analysis. Samples were analysed with an LC-MS/MS method (CEN/TC 275/WG 4N) involving extraction with methanol/water and quantification using the internal standard method with a deuterated internal standard (LOQ=0.05 mg/kg for straw and offal, and 0.01 mg/kg for all other matrices). Acceptable concurrent recoveries were determined.

Raw grain, forage and straw samples taken directly from the field site to the laboratory were analysed within 4 months of collection, while processed fractions were analysed within 2 months of collection. Oats for processing were stored ambient then refrigerated temperatures before processing.

Stability of the residues in the oats stored at ambient temperatures before processing was verified by the comparable results between the raw oats frozen at the field site and those sampled just before processing after the ambient storage.

Table 51 Residues of chlormequat chloride in processed fractions of oats from a site treated with a single application at BBCH 49

Location, Year (variety)	Application		Results	
	Rate, kg ai/ha	Spray volume (L/ha)	Sample	Residue (mg/kg chlormequat chloride)
D-65597 Aarbergen-Panrod, Hesse, Germany, 2003 (oats, Matilda)	3.6	304	Forage (shoots)	30 c0.01
			Straw	9.9
			Grain	2.3 c0.02
Balance study				
			Grain prior to processing	2.2 c0.02
			Cleaned grain	2.2 c0.02
			Offal	4.8 c0.10
			Oat kernels	2.2 c0.02
			Husks	1.6
			Oat dust	3.9 c0.32
			Oat flakes	2.6 c0.02
Follow-up study 1				
			Grain prior to processing	2.2
			Oat flakes	2.0
Follow-up study 2				
			Grain prior to processing	2.0
			Oat flakes	1.6
Follow-up study 3				
			Grain prior to processing	2.2
			Oat flakes	2.2

Except where otherwise noted, no untreated control samples contained residues above the LOQ.

Table 52 Processing factors for oat commodities

Processed fraction	Processing factor
Grain prior to processing	-
Cleaned grain	1.0
Offal	2.1
Oat kernels	1.0
Husks	0.7
Oat dust	1.8
Oat flakes	0.8, 0.9, 1.0, 1.2

Residues of chlormequat chloride do not concentrate in cleaned grain, oat kernels, husks or oat flakes on processing of raw oats, while residues do concentrate in offal and oat dust.

Wheat

A processing study in wheat was conducted using samples treated, harvested and processed during the 2001 growing season in Germany (Zietz and Klimmek 2004b, 2004/1013832). At single trial site,

wheat was treated with a single foliar broadcast application of a 720 g/L SL formulation of chlormequat chloride at BBCH stage 31, and an application rate of 3.5 kg ai/ha. No other pesticides expected to interfere with the experiment were applied during the growing season.

Wheat grain and straw (treated and control) were collected at normal commercial harvest, 82 days after application (1 kg each of grain and straw for analysis of the raw agricultural commodity (RAC) and 19 kg of grain for each balance study and 13 kg for each processing study).

RAC samples were frozen shortly after collection and kept frozen until analysis, while samples of grain for processing were shipped under ambient conditions to the processing laboratory. Grain for processing was stored under ambient conditions until processing was commenced approximately 3.5 months after harvest. Grain was processed into type 550 flour, wholemeal flour, bran and wholegrain bread using standard methods simulating industrial processes.

Untreated samples were processed before the treated samples. At the beginning of the balance study, a raw grain sample was collected for analysis. Raw grain samples were first cleaned using a grain cleaner, and samples of cleaned grain and offal (aspirated grain fractions) were collected. The cleaned grain was subdivided into batches for processing into type 550 flour and into wholemeal flour.

For processing into type 550 flour in the balance study, cleaned grain was tested for moisture content and water added to bring the water content to 15–16%. Moisture-adjusted grain was passed through a countercurrent mixer to blend the grain and remove the epidermis; the epidermis and grain were separated using an elevator sifter, and a sample of epidermis collected. The grain was then passed through the automated laboratory mill, where it passed over three breaking rolls (B1–B3) and three resolution rolls (C1–C3). Six fractions of straight flour and three each of coarse bran (B1–B3) and fine bran (middlings, or C1–C3) were collected and weighed. All six fractions of straight flour were then combined as were the three coarse bran and three fine bran fractions, with portions of each of straight flour, and coarse and fine bran collected for analysis. Half of each of the total fine and coarse brans were combined in a bran duster and separated (total bran and low grade meal). After weighing, samples of total bran and low grade meal were collected for analysis. The remaining low grade meal was used to adjust an aliquot of the straight flour fraction to give type 550 flour, which was sampled for analysis.

For wholemeal flour, cleaned moisture-adjusted grain was milled in the same automated laboratory mill used to produce the type 550 flour. The fractions of composite straight flour and total bran were collected and weighed and the bran ground with an impact grinding mill. Using a blender, the straight flour and ground bran were then blended to yield wholemeal flour, which was sampled for analysis. The majority was retained and stored frozen (-18 °C) for the next process, baking into bread.

Baking commenced within 10 days of production of the wholemeal flour. Sourdough wholegrain bread was prepared using a standard recipe. As the first step, 200 g flour, 400 mL water and 1 g sourdough starter were combined and fermented at 24–25 °C for 17–18 hours. Next, 540 g of the sourdough mixture, 1800 g wholemeal flour (assuming a moisture content of 14%, with the amount being adjusted for the actual moisture content), 27 g bakers yeast, 27 g salt, 18 g sugar, 18 g peanut fat, 180 mL 0.1% ascorbic acid, and 700-1350 mL water depending on the adsorption capacity of the flour were combined in a kneader. After the first kneading the dough was placed in a chamber for fermentation (31–32 °C and 82–85% RH, 30 minutes). After checking for quality by touch, dough was sampled for analysis, and the remainder placed in baking pans and proved (60 minutes in the fermentation chamber at the same temperature and humidity as for fermentation), then baked at 210 °C for 60 minutes (control bread was baked separately from treated bread). After cooling to room temperature, the bread was checked for quality (appearance, browning, volume, elasticity of the surface and crumb, curvature, and uniformity of vacuoles), then cut into coarse slices and frozen in plastic bags.

In the follow-up studies, the same processing procedures were followed, with only total bran, type 550 flour, wholemeal flour and wholegrain bread samples being collected, as the most

commercially important fractions, in order to gain more data on the partitioning of residues into these fractions.

Processed fraction samples were frozen shortly after collection and kept frozen until analysis. Samples were analysed with an LC-MS/MS method (CEN/TC 275/WG 4N) involving extraction with methanol/water and quantification using the internal standard method with a deuterated internal standard (LOQ=0.05 mg/kg for straw and bran, and 0.01 mg/kg for all other matrices). Acceptable concurrent recoveries were determined.

Apart from low grade meal, in which a residue of 0.02 mg/kg was observed, no residues above the LOQ were determined in any of the untreated control samples, although a number of other samples did contain residues at levels above 30% of the LOQ. Given the levels of chlormequat chloride residues observed in the treated samples, the levels observed in the control samples are not expected to interfere with the results of the processing study.

The raw grain and straw samples taken directly from the field site to the laboratory were analysed within 13 months of collection, while processed fractions were analysed within 9-10 months of collection.

Table 53 Residues of chlormequat chloride in processed fractions of wheat from a site treated with a single application at BBCH 31

Location, Year (variety)	Application		Results	
	Rate, kg ai/ha	Spray volume (L/ha)	Sample	Residue (mg/kg chlormequat chloride)
D-65597 Hünfelden-Nauheim, Hesse, Germany, 2001 (wheat, Thasos)	3.5	300	Straw	10
			Grain	1.0
Balance study				
			Grain prior to processing	1.1
			Cleaned grain	0.92
			Offal	1.1
			Epidermis	1.5
			Coarse bran	3.4
			Fine bran	2.4
			Straight flour	0.20
			Low grade meal	1.4
			Flour (type 550)	0.21
			Total bran (whole meal flour)	3.1
			Straight flour (wholemeal flour)	0.28
			Wholemeal flour	0.95
			Dough	0.56
			Wholegrain bread	0.56
Follow-up study 1				
			Grain prior to processing	0.96
			Total bran	2.8
			Flour (type 550)	0.28
			Wholemeal flour	1.1
			Wholegrain bread	0.53
Follow-up study 2				
			Grain prior to processing	0.99
			Total bran	3.1
			Flour (type 550)	0.30
			Wholemeal flour	0.90
			Wholegrain bread	0.49

Location, Year (variety)	Application		Results	
	Rate, kg ai/ha	Spray volume (L/ha)	Sample	Residue (mg/kg chlormequat chloride)
Follow-up study 3				
			Grain prior to processing	0.98
			Total bran	3.3
			Flour (type 550)	0.27
			Wholemeal flour	0.98
			Wholegrain bread	0.52

Table 54 Processing factors for wheat commodities

Processed fraction	Processing factor
Grain prior to processing	-
Cleaned grain	0.84
Offal	1.0
Epidermis	1.4
Coarse bran	3.1
Fine bran	2.2
Straight flour	0.18
Low grade meal	1.2
Flour (type 550)	0.19, 0.28, 0.29, 0.30
Total bran (wholemeal flour)	2.8, 2.9, 3.1, 3.4
Straight flour (wholemeal flour)	0.25
Wholemeal flour	0.86, 0.91, 1.0, 1.1
Dough	0.51
Wholegrain bread	0.49, 0.51, 0.53, 0.55

Residues of chlormequat chloride were observed to concentrate slightly in epidermis and low grade meal, and more significantly in the bran fractions. Residues of chlormequat chloride did not concentrate in cleaned grain, offal (aspirated grain fractions), type 550 flour, wholemeal flour, dough or bread.

Table 55 Summary of processing factors for chlormequat-chloride residues

Raw Agricultural Commodity (RAC)	Processed Commodity	Calculated Processing factors	Best Estimate Processing Factor
Barley	Cleaned grain (pot barley)	1.0	1.0
	Offal (pot barley)	2.1	2.1
	Pearling dust	2.7, 3.0, 3.3, 3.3	3.15
	Pot barley	0.8, 0.9, 1.0, 1.0	0.95
	Cleaned grain (malting)	1.1	1.1
	Offal (malting)	1.6	1.6
	Steeping water	0.01	0.01
	Malt sprouts	2.2	2.2
	Malt	0.9, 0.9, 0.9	0.9
	Spent grain	0.01, 0.02, 0.02, 0.03	0.02
	Condensate	0.01	0.01
	Flocs	0.5, 0.5, 0.6, 0.6	0.55
	Wort	0.1, 0.2, 0.2, 0.2	0.2
	Yeast	0.2	0.2
	Green beer	0.1, 0.2, 0.2, 0.2	0.2
	Beer	0.1, 0.2, 0.2, 0.2	0.2
	Cleaned grain (pot barley)	1.0	1.0
Oat	Cleaned grain	1.0	1.0
	Offal	2.1	2.1

Raw Agricultural Commodity (RAC)	Processed Commodity	Calculated Processing factors	Best Estimate Processing Factor
	Oat kernels	1.0	1.0
	Husks	0.7	0.7
	Oat dust	1.8	1.8
Wheat	Cleaned grain	0.84	0.84
	Offal	1.0	1.0
	Epidermis	1.4	1.4
	Coarse bran	3.1	3.1
	Fine bran	2.2	2.2
	Straight flour	0.18	0.18
	Low grade meal	1.3	1.3
	Flour (type 550)	0.19, 0.28, 0.29, 0.30	0.285
	Total bran (whole meal flour)	2.8, 2.9, 3.1, 3.4	3.0
	Straight flour (wholemeal flour)	0.25	0.25
	Wholemeal flour	0.86, 0.91, 1.0, 1.1	0.955
	Dough	0.51	0.51
	Wholegrain bread	0.49, 0.51, 0.53, 0.55	0.52

RESIDUES IN ANIMAL COMMODITIES

Cattle feeding study

The Meeting received a feeding study in lactating cattle (Weidenauer, 1999a), which was previously considered by the 2000 JMPR. 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 throughout the study. After the final dose, the cattle were slaughtered (with the exception of the depuration study animals, see previous paragraph). Samples were analysed for chlormequat chloride using an ion chromatographic method (method number 397, see above), with an LOQ of 0.01 mg/kg for milk and 0.05 mg/kg for tissues. Samples were frozen after collection and analysed up to 12 months after collection in the case of tissues and up to 13 months after collection in the case of milk. A storage stability study for animal matrices (see above) was provided to the Meeting and demonstrated stability of chlormequat chloride residues in cattle meat, milk and eggs over 12 months. The samples from the feeding study are therefore not likely to have been adversely affected by storage.

Table 56 Residues of chlormequat chloride in cattle tissues (individual results are for individual animals)

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

Cow no.	Dose	Residue (mg/kg)			
		Meat	Liver	Kidney	Fat
Mean		< 0.05	0.38	0.76	0.08
13 (2 days depuration)	120 ppm (depuration)	< 0.05	< 0.05	0.16	< 0.05
14 (7 days depuration)		< 0.05	< 0.05	0.09	< 0.05

Table 57 Residues of chlormequat chloride in milk (individual results are for individual animals)

Study day	Dose group		
	12 ppm	36 ppm	120 ppm
-1/0	0.02, < 0.01, < 0.01 (mean=< 0.01)	< 0.01, < 0.01[a], < 0.01	< 0.01 (5)
1/2	0.02, < 0.01[a], 0.01 (mean=0.01)	0.04, 0.06, 0.01 (mean=0.04)	0.07, 0.07, 0.20, 0.14, 0.07 (mean=0.11)
3/4	0.02, 0.05, 0.01 (mean=0.03)	0.14, 0.03, 0.05 (mean=0.07)	0.47, 0.21, 0.16, 0.32, 0.56 (mean=0.34)
5/6	0.01, 0.05, 0.05 (mean=0.04)	0.17, 0.10, 0.07 (mean=0.11)	0.06, 0.40, 0.10, 0.35, 0.33 (mean=0.25)
7/8	0.01, 0.02, < 0.01 (mean=0.01)	0.11, 0.09, 0.08 (mean=0.09)	0.28, 0.23, 0.25, 0.18, 0.20 (mean=0.23)
10/11	0.01, 0.05, 0.05 (mean=0.04)	0.17, 0.19, 0.13 (mean=0.16)	0.23, 0.14, 0.21, 0.11, 0.29 (mean=0.20)
12/13	0.05, 0.02, < 0.01[a] (mean=0.02)	0.10, 0.07, 0.06 (mean=0.08)	0.29, 0.31, 0.11, 0.19, 0.35 (mean=0.25)
14/15	0.04, 0.08, 0.04 (mean=0.05)	0.26, 0.21, 0.09 (mean=0.19)	0.13, 0.65, 0.07, 0.20, 0.07 (mean=0.22)
17/18	0.02, < 0.01[a], < 0.01[a] (mean=0.01)	0.07, 0.07, 0.02 (mean=0.05)	0.09, 0.13, 0.32, 0.20, 0.21 (mean=0.19)
20/21	0.03, 0.03, 0.03 (mean=0.03)	0.09, 0.08, 0.06 (mean=0.08)	0.26, 0.23, 0.35, 0.33, 0.05 (mean=0.24)
23/24	0.05, < 0.01, < 0.01 (mean=0.02)	< 0.01, 0.24, 0.13 (mean=0.12)	0.16, 0.29, 0.33, 0.16, 0.19 (mean=0.23)
25/26	0.02, 0.04, 0.05 (mean=0.04)	0.09, 0.12, 0.12 (mean=0.11)	0.30, 0.21, 0.13, 0.16, 0.21 (mean=0.20)
28/29	< 0.01, 0.01, 0.02 (mean=0.01)	0.06, 0.14, 0.05 (mean=0.08	0.15, 0.10, 0.23, 0.15, 0.15 (mean=0.16)
30 (+2)	NA	NA	0.04
35 (+7)	NA	NA	< 0.01

No residues above the LOQ were found in any of the untreated control milk samples.

[a] Detectable residues below the LOQ were found.

Table 58 Partitioning of chlormequat chloride residues between skim milk and cream

Study day - sample	Dose group		
	12 ppm	36 ppm	120 ppm
1 – skim milk	0.04, 0.02, 0.02 (mean=0.03)	0.04, 0.03, 0.02 (mean=0.03)	0.09, 0.09, 0.05, 0.03, 0.06 (mean=0.06)
1 – cream	< 0.01 (3)	0.02, < 0.01, 0.01 (mean=0.01)	0.07, 0.07, 0.09, 0.09, 0.11 (mean=0.09)
14 – skim milk	0.10, 0.04, 0.01 (mean=0.05)	0.14, 0.02, 0.10 (mean=0.09)	0.06, 0.38, 0.31, 0.02, 0.36 (mean=0.23)
14 – cream	0.02, 0.03, 0.03 (mean=0.03)	0.04, 0.04, 0.05 (mean=0.04)	0.07, 0.11, 0.05, 0.10, 0.09 (mean=0.08)
28 – skim milk	0.02, 0.03, 0.02 (mean=0.02)	0.22, 0.15, 0.04 (mean=0.14)	0.11, 0.16, 0.07, 0.13, 0.11 (mean=0.12)
28 – cream	0.02, 0.02, 0.03 (mean=0.02)	0.04, 0.07, 0.04 (mean=0.05)	0.02, 0.09, 0.06, 0.04, 0.10 (mean=0.06)

Mean residues in skim milk and cream from the untreated control group were below the LOQ.

Residues of chlormequat chloride in muscle and fat were mostly <LOQ, with the exception of fat for the highest dose group, were levels of ≤ 0.10 mg/kg were observed. Residue levels were higher in liver, and especially kidney. Finite residues were found in liver and kidney at all dose levels, with residues increasing with increased dose.

Milk residues increased with increasing dose. Residues in milk reached a plateau by 12–15 days of dosing. Residues in skim milk were generally higher in skim milk than in cream, consistent with the high water solubility and expected low fat solublity of chlormequat chloride.

After cessation of dosing, clearance of chlormequat chloride from milk and tissues was rapid, with no residues above the LOQ in muscle, fat or liver after 2 days depuration or in milk after 7 days depuration. Quantifiable residues in kidney were still present, although the residues had decreased from a mean value of 0.76 mg/kg on the last day of dosing, to 0.16 and 0.09 mg/kg after 2 and 7 days depuration respectively.

Poultry feeding study

The Meeting received a feeding study in laying hens (Weidenauer, 1999b), which was previously considered by the 2000 JMPR. Four groups of hens (one group per dose level), each group consisting of three subgroups 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. Two additional groups of 12 hens (one group per depuration interval) were dosed at the highest level for 28 days for generation of depuration data. The birds were slaughtered after the final dose, with the exception of the two groups of depuration phase birds, one group each of which was slaughtered 2 and 7 days after the final dose. Tissue samples (breast and leg muscle, liver and abdominal fat) were then collected. Eggs were collected daily during the dosing and depuration phases. Samples were analysed for chlormequat chloride using an ion pair chromatographic method (method number 397, see above). This method has an LOQ of 0.05 mg/kg in eggs and tissues. Tissues were analysed within 3 months of collection, and eggs within 9 months of collection, a period covered by the verified 12-month period of stable storage (see above).

Table 59 Residues of chlormequat chloride in laying hen tissues (individual results are for a subgroup of four hens)

Subgroup number	Dose group	Residue (mg/kg)		
		Muscle	Liver	Fat
4	6 ppm	< 0.05	0.09	< 0.05
5		< 0.05	< 0.05[a]	< 0.05
6		< 0.05	< 0.05[a]	< 0.05[a]
Mean		< 0.05	0.05	< 0.05
7	18 ppm	< 0.05	< 0.05[a]	< 0.05
8		< 0.05	0.10	< 0.05[a]
9		< 0.05	0.09	< 0.05
Mean		< 0.05	0.07	< 0.05
10	60 ppm	< 0.05[a]	0.12	< 0.05[a]
11		< 0.05	0.10	< 0.05[a]
12		< 0.05[a]	0.33	< 0.05
Mean		< 0.05	0.18	< 0.05
13-1	60 ppm (2 days depuration)	< 0.05	0.12	< 0.05[a]
13-2		< 0.05[a]	< 0.05	< 0.05
13-3		< 0.05	< 0.05[a]	< 0.05
Mean		< 0.05	0.05	< 0.05
14-1	60 ppm (7 days depuration)	< 0.05[a]	< 0.05[a]	< 0.05[a]
14-2		< 0.05[a]	0.08	< 0.05[a]
14-3		< 0.05	< 0.05[a]	< 0.05[a]
Mean		< 0.05	< 0.05	< 0.05

No residues above the LOQ were found in any of the untreated control tissue samples.

[a] Detectable residues below the LOQ were found.

Table 60 Residues of chlormequat chloride in eggs (individual results are for a subgroup of four hens)

Study day	Dose group		
	6 ppm	18 ppm	60 ppm
-1/0	< 0.05 (3)	< 0.05 (3)	< 0.05 (4), < 0.05[a]
1/2	< 0.05 (3)	< 0.05 (3)	< 0.05 (5)
3/4	< 0.05[a], < 0.05[a], < 0.05	0.06, < 0.05, < 0.05 (mean=< 0.05)	0.10, < 0.05[a], 0.07, 0.06, < 0.05[a] (mean=0.06)
5/6	< 0.05[a], < 0.05[a], < 0.05[a]	0.05, < 0.05, < 0.05 (mean=< 0.05)	< 0.05[a], 0.08, 0.16, 0.18, 0.11 (mean=0.11)
7/8	0.05, < 0.05[a], < 0.05[a] (mean=< 0.05)	0.09, 0.12, 0.10 (mean=0.10)	0.13, 0.08, 0.08, 0.17, 0.08 (mean=0.11)
10/11	< 0.05 (3)	0.06, 0.10, 0.07 (mean=0.08)	0.08, 0.07, 0.11, 0.09, 0.13 (mean=0.10)
12/13	< 0.05[a], < 0.05, < 0.05	< 0.05, 0.07, < 0.05 (mean=< 0.05)	0.08, 0.08, 0.10, 0.08, < 0.05 (mean 0.07)
14/15	< 0.05[a], < 0.05, < 0.05	< 0.05, 0.09, 0.06 (mean=0.05)	0.12, 0.19, 0.16, < 0.05, 0.07 (mean=0.11)
17/18	< 0.05[a], < 0.05, < 0.05	< 0.05, < 0.05[a], < 0.05	0.07, 0.14, < 0.05[a], 0.08, 0.06 (mean=0.08)
20/21	< 0.05 (3)	< 0.05 (3)	0.07, < 0.05, < 0.05, 0.05, 0.06 (mean=0.04)
23/24	< 0.05 (3)	< 0.05 (3)	0.09, 0.08, 0.05, 0.06, 0.06 (mean=0.07)
25/26	< 0.05 (3)	< 0.05, < 0.05, 0.06 (mean=< 0.05)	0.06, 0.15, 0.07, 0.08, 0.07 (mean=0.09)
28/29	< 0.05 (3)	< 0.05[a], < 0.05, < 0.05	0.13, 0.06, 0.05, 0.07, 0.06 (mean=0.07)
30 (+2)	NA	NA	< 0.05
35 (+7)	NA	NA	< 0.05[a]

No residues above the LOQ were found in any of the untreated control egg samples.

[a] Detectable residues below the LOQ were found.

No residues above the LOQ were found in any of the muscle or fat samples at any treatment level. Finite residues were found in liver and showed a trend of increase with increased dose. Residues in eggs were also increased with dose, with only one detection above the LOQ for the low dose group, and higher residues in the mid- and high-dose groups. Residues in eggs for the mid- and high-dose groups reached a plateau on day 7–8. Clearance of residues from the hens after cessation of dosing was rapid, with no residues of chlormequat chloride above the LOQ in eggs after 2 or 7 days on clean feed, and residues in liver decreasing from a mean value of 0.18 mg/kg on the last dosing day to 0.05 and < 0.05 mg/kg after 2 and 7 days of depuration respectively.

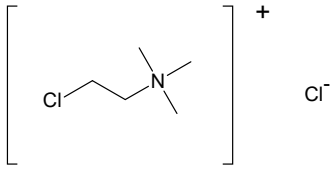
APPRAISAL

Chlormequat chloride is a plant growth regulator which acts primarily by reducing cell elongation, but also by lowering the rate of cell division. It inhibits the synthesis of gibberellins. It was scheduled for periodic review evaluation by the 2017 JMPR at the 48th Session of the CCPR (2016). Chlormequat was previously evaluated by the JMPR in 1970, 1972, 1994 (periodic review), 1997, 1999 and 2000. It was evaluated for toxicology in 1997 and 1999 at which time an acute reference dose was established.

The manufacturer supplied information on identity, physicochemical properties, plant, animal and confined crop metabolism, environmental fate, methods of residue analysis, freezer storage

stability, registered use patterns, supervised residue trials, fate of residues in processing, and animal transfer studies.

The IUPAC name is 2-chloroethyl-trimethylammonium chloride.



Chlormequat-chloride

Plant Metabolism

The Meeting received plant metabolism studies conducted on wheat and grapes, together with a considerable amount of supporting metabolism information previously provided to the 1994 JMPR.

Chlormequat chloride, radiolabelled in both carbons of the chloroethyl group, was applied to grapevines as three consecutive foliar applications at growth stages BBCH 13–15, 15–17 and 57. The application rates were 180, 360 and 90 g ai/ha (total 630 g ai/ha).

Leaves were sampled from the immature plants immediately before and 22 days after the last application. The mature grapes were harvested at BBCH 89 (90 DALA) and the remaining plant material was separated into leaves, branches and stalks. With methanol (×3) and water (×2) extractions, 99% of the radioactive residues in grapes and 95% of the radioactive residues of leaves were extracted.

In total, 0.18 mg eq/kg (98% TRR) in grapes and 1.65 mg eq/kg (84% TRR) in leaves was identified as the active substance chlormequat chloride. Minor unidentified components totalled 0.004 mg eq/kg in grapes and 0.10 mg eq/kg in leaves. In total 100% and 89% TRR was identified or characterised in grapes and leaves respectively. Unextracted residues in grapes after solvent extraction were < 1% TRR and in leaves 5% TRR.

In a study in wheat, [1,2-14C-ethyl]-chlormequat chloride was applied once at 1380 g ai/ha by foliar application to wheat plants grown in a phytotron. Forage was collected at 0, 28 and 84 days after application, while grain and straw were collected at harvest maturity 118 days after application.

Residues were readily extractable from forage and straw using methanol (79–90% TRR extracted from forage and 81% TRR from straw). Extractability from grain was lower, with 37% TRR extracted using methanol, together with a further 17% released using a methanol/water reflux. A significant proportion of the radioactivity in grain had been incorporated into biomolecules, with 16% TRR present as starch, and 36% TRR present as lignin. A smaller proportion of the residue in straw (5.1% TRR) had been incorporated into lignin. Incorporation into protein or cellulose was not significant in either straw or grain.

Parent was the largest individual identified component in wheat matrices, at 9.7–42 mg eq/kg (67–86% TRR) in forage, 36–37 mg eq/kg (78–81% TRR) in straw, and 0.37–0.41 mg eq/kg (28–30% TRR) in grain. Small amounts of betaine were identified in grain (up to 0.054 mg eq/kg, 4.7% TRR), and straw (0.06 mg eq/kg, 0.1% TRR), with unidentified components at up to 2.4 mg eq/kg (6.2% TRR) in forage, up to 1.8 mg eq/kg (3.8% TRR) in straw, and up to 0.026 mg eq/kg (1.5% TRR) in grain.

Summary of plant metabolism

Metabolism data in grapes and wheat were provided, together with a considerable amount of supporting literature. Parent was observed to be the major component of the radioactive residues in grape berries and leaves, and in wheat grain, straw and forage. Betaine was observed as a very minor component (<5% TRR) in grain and straw. A number of older metabolism studies (in pot grown wheat and barley, brassicas, and tomatoes) first considered by the 1994 JMPR showed similar behaviour,

with metabolism of chlormequat chloride only occurring to a limited extent, with minor amounts of choline also being observed. Greater degrees of metabolism were noted in other non-contemporary studies, including in wheat treated via a root application, in which significant metabolism to choline, then betaine, glycine and serine, with ultimate incorporation into biomolecules and evolution of radiolabelled CO_2 being observed.

Confined Rotational Crops

A study was undertaken to investigate the metabolism of chlormequat chloride in the representative crops spring wheat, lettuce and white radish after three plant back intervals using ^{14}C-chlormequat chloride (radiolabelled in both carbons of the chloroethyl group) sprayed onto bare soil in plastic containers at 2 kg ai/ha. The crops were each sown at 30, 120 and 365 days after the soil application, representing the first, second and third rotation.

In lettuce leaf parent was not observed at any of the three plant back intervals. In radish root and leaf, parent was observed at 0.008–0.009 mg eq/kg (19–20% TRR) at the 30 day plant back interval (PBI) while at the 120 day PBI it was no longer present. Parent was the major component in wheat straw at the 30 day PBI (0.072 mg eq/kg, 22% TRR) and at the 120 day PBI parent was no longer detected. Parent was observed in wheat grain at the 30 and 120 day PBIs only (0.015 and 0.009 mg eq/kg, 9 and 4% TRR respectively). Polar degradation products (not identified) were found in most samples at low levels (except for 120 day PBI wheat straw and chaff, containing ≤ 0.011 mg eq/kg (≤ 8.3% TRR) and 0.022 mg eq/kg (13% TRR) respectively, the totals in each matrix were ≤ 0.01 mg eq /kg (≤ 47% TRR)).

At the 365 day PBI, only lettuce and wheat grain residues were characterised, and no parent was detected, with only minor polar degradates found at ≤ 0.003 mg eq/kg (≤ 31% TRR).

In general, chlormequat chloride was converted to mainly polar degradation products and at longer plant back intervals parent was no longer detected or only found at low levels.

In another study, the metabolism of chlormequat chloride was investigated in the representative crops spring wheat, green beans, carrots and head lettuce from three consecutive rotations using 14C-chlormequat chloride added to loamy sand soil giving an application rate of 1.5 kg ai/ha, then stored in a drum for 30 days. After 30 days the soil was diluted using untreated soil to simulate ploughing. The crops were planted/sown at 30 days after the soil application. Beans, carrots and lettuce were cultivated in a greenhouse while spring wheat was grown in a phytotron with fluorescent lamps. Only total residues were reported: concentrations of total residue in the edible parts of the four crops ranged from 0.003 mg eq/kg in beans at harvest to 0.052 mg eq/kg in wheat grain at harvest, but were < 0.01 mg eq/kg for lettuce heads and carrot roots. In wheat forage and straw, bean forage, and carrot leaves, the total residues ranged from 0.016–0.066 mg eq/kg.

In summary, chlormequat chloride is metabolised in rotational crops to unidentified polar components, with only relatively low levels of parent found (≤ 0.072 mg eq/kg, ≤ 32% TRR at the 30-day PBI; ≤ 0.009 mg eq/kg, ≤ 9.2% TRR at the 120-day PBI; not detected at the 365-day PBI).

Environmental fate in soil

The Meeting received information on aerobic soil metabolism, hydrolysis, aqueous photolysis, and a field dissipation study. Only the aerobic soil metabolism study and the field dissipation study, which are relevant to the current evaluation were considered.

The route and rate of degradation of ^{14}C-chlormequat chloride was studied in an aerobic laboratory study in three European soils, at 20 ± 2 °C and a period of 120 days. Parent compound ^{14}C-chlormequat chloride was the only major radioactive fraction detected in the soil extracts. Mineralisation to CO_2 was the major route of degradation besides formation of bound residues. Four minor metabolites (< 3% AR) were detected in the soil extracts. DT_{50} values for chlormequat chloride ranged from 10.2–36.5 days, while DT_{90} values ranged from 33.8–121 days.

Field dissipation data conducted with a sandy loam soil, together with data for a clay soil in a greenhouse, showed rapid microbiological degradation in both cases. The observed field behaviour was consistent with the results of the laboratory study. Chlormequat was extensively mineralised and CO_2 was the ultimate product of degradation. Other degradation products could not be identified. DT_{50} values ranged from < 1–28 days, while DT_{90} values were less than 100 days.

Chlormequat is not considered to be persistent in soil.

Animal metabolism

The Meeting received animal metabolism studies with chlormequat in rats, hens and goats.

Rats

Evaluation of the metabolism studies in rats was carried out by the WHO Core Assessment Group.

Goats

A study on the metabolism of chlormequat chloride was conducted with the test compound labelled in both positions of the chloroethyl group. Two lactating goats were dosed orally twice daily for seven consecutive days, at 25 ppm in the diet. Milk was sampled twice daily, prior to dosing in the morning and afternoon. Animals were sacrificed approximately 23 hours after the last dose.

A total of 49 and 30% of the total administered dose was eliminated in the urine and faeces respectively (cumulative over 7 days). TRRs were 1.5 mg eq/kg in kidney, 0.36 mg eq/kg in liver, 0.23 mg eq/kg in muscle, 0.022 and 0.008 mg eq/kg in renal and omental fat respectively, and 0.24 and 0.20 mg eq/kg in 56 hour and 144 hour milk respectively.

Initial methanol extraction of kidney, liver, muscle and renal fat resulted in extraction efficiencies of 92, 77, 90 and 67% respectively. Pepsin hydrolysis of the post-extracted solid (PES) released 7.3, 15 and 7.7% TRR for kidney, liver, and muscle respectively. Protease released a further 1.5% for liver.

Milk was initially extracted with acetonitrile recovering 17 and 20% from the 56 hour and 144 hour samples respectively. Pepsin hydrolysis of the solvent-precipitated solids released 63 and 80% TRR from the 56 hour and 144 hour samples, respectively, while protease released a further 16% TRR from the 56 hour sample.

Metabolism in ruminants only occurred to a very limited extent. Parent was the only compound identified in kidney, liver and muscle (83, 42 and 76% TRR respectively). It was also the only identified compound in milk accounting for <5% TRR in the 56 hour and 144 hour milk samples (0.011 and 0.002 mg eq/kg respectively). Release of significant proportions of the radioactivity by pepsin and protease hydrolysis indicated that a substantial part of the radioactivity was present as macromolecules, formed by incorporation of chlormequat chloride by biosynthetic pathways.

Hens

A study on the metabolism of chlormequat chloride was conducted with the test compound labelled in both positions of the chloroethyl group. Ten laying hens were dosed orally once daily for 14 consecutive days, at 12 ppm. The hens were sacrificed approximately 23 hours after the last dose.

A total of 93% of the total administered dose was eliminated in the excreta (cumulative after 14 days). Egg yolk and egg white accounted for 0.34 and 0.05% of the administered dose respectively (cumulative after 14 days). A plateau level of approximately 0.97 mg eq/kg in composite egg yolk samples was reached at 264 hours (11 days) after the first administration, while concentrations in egg white were much lower and did not reach a plateau level. TRRs in tissues were 0.36 mg eq/kg in liver, 0.35 mg eq/kg in kidney, 0.12 mg eq/kg in muscle and 0.062 mg eq/kg in abdominal fat.

Methanol extraction of liver, kidney, muscle and egg yolk extracted 66, 65, 75 and 62% TRR respectively, while proteolytic enzyme hydrolysis after solvent extraction released further substantial amounts of the radioactivity (26%, 28%, 15%, and up to 13% respectively of the TRR).

In egg white, the solvent extraction only extracted ~5% TRR, however pepsin enzymatic hydrolysis released a significant proportion of the unextracted fraction (85 and 87% TRR for the 96 hour and 264 hour fractions respectively). In fat most of the radioactive residues remained unextracted after solvent extraction, enzyme hydrolysis and acid reflux (65% of TRR still unextracted, however absolute levels were low, with fat only containing a total of 0.062 mg eq/kg).

Parent chlormequat chloride was the only identified compound. It was found in kidney and liver (0.023 and 0.007 mg eq/kg, or 7 and 2% TRR respectively) and as a major fraction in egg yolk (0.47 mg eq/kg, or 48% TRR in the 264 hour sample, but was not found in the 96 hour sample). In the extracts of liver, kidney, muscle and egg yolk (96 hour sample), regions of radioactive residue, accounting for >0.05 mg eq/kg could not be identified. As with goats, a significant portion of the radioactive residues were released by protease and pepsin hydrolysis, indicating incorporation into macromolecules, *via* biosynthetic pathways.

Summary of animal metabolism

Metabolism in ruminants was very limited. Parent chlormequat chloride was the only compound identified in kidney, liver and muscle (83, 42 and 76% TRR respectively). It was also the only identified compound in milk, although only accounting for < 5% TRR in the 56 hour and 144 hour milk samples.

Parent chlormequat chloride was the only identified compound in the poultry metabolism study. It was identified in kidney, liver, and as a major fraction in the 264h sample of egg yolk, but not in the 96h yolk sample.

A similar pattern was observed in rats, with only parent chlormequat chloride, and two other components tentatively identified as other salts of chlormequat being found after oral administration.

Methods of analysis

The Meeting received information on analytical methods suitable for the determination of residues of chlormequat chloride in plant and animal matrices.

Plant matrices

A method (method 146) developed for the determination of chlormequat chloride in cereal matrices requires extraction with methanol and quantification using gas chromatography. Limits of quantification using this method were generally 0.05 mg/kg in cereal grains and 0.5 mg/kg for cereal straw.

Method 530/0 for the determination of chlormequat chloride in plant commodities, is based on extraction using water/ methanol/hydrochloric acid, and quantification using LC-MS/MS. Limits of quantification (LOQ) using method 530/0 were generally 0.05 mg/kg in all plant matrices except for cereal straw (LOQ = 0.1 or 0.5 mg/kg). Method 530/0 was used for determination of residues in the freezer storage stability study conducted on grapes, as well in the grape, wheat, barley, rye and oats residues trials.

Another LC-MS/MS method (CEN/TC 275/WG 4N), was used for determination of chlormequat chloride. This method involved extraction with methanol/water and quantification using a deuterated chlormequat internal standard (LOQ = 0.05 mg/kg for straw and 0.01 mg/kg for all other cereal matrices).

Animal matrices

Method 397 was developed for the determination of residues of chlormequat chloride in animal matrices. Samples are extracted using acetone/water, with determination using ion chromatography. Limits of quantification using method 397 were generally 0.05 mg/kg in all animal matrices except for milk (LOQ = 0.01 mg/kg). A modification of this method (397/0) employs LC-MS/MS. Limits of quantification using method 397/0 were 0.01 mg/kg in all animal matrices except for liver (LOQ = 0.05 mg/kg).

Stability of pesticide residues in stored analytical samples

The Meeting received information on the freezer storage stability of chlormequat chloride in plant and animal matrices.

A storage stability study showed that chlormequat chloride residues are stable for at least 24 months in grapes. A study for cereal matrices involved fortification of wheat grain and straw samples, and this demonstrated stability of chlormequat chloride residues in grain and straw samples for at least 24 months. The cereal study additionally re-analysed stored samples from processing studies, and demonstrated no significant changes in the residue levels over a further storage period of 13 months in wheat bran and wholegrain bread, 12 months in barley malt and 11 months in beer, when stored frozen at approximately -18 or -20 °C. The storage periods in the storage stability studies covers the sample storage intervals in the residue trials.

A study in animal matrices showed that residues of chlormequat chloride are stable in cattle meat, milk and hen eggs for at least 12 months of frozen storage at -18 °C, covering the storage intervals in the animal feeding studies.

Definition of the residue

Plant commodities

In the metabolism study conducted on grapes using ^{14}C-chlormequat chloride, the parent compound was observed to be the major component of the radioactive residues, accounting for approximately 100 and 88% of the TRR in grapes and grape leaves respectively. In a wheat metabolism study, parent compound accounted for 67–86% TRR in forage, 78–81% TRR in straw and 28–30% TRR in grain. Parent compound was also the only component identified in the confined rotational crop study.

Validated analytical methods for parent compound in plant matrices are available.

The Meeting therefore considered that a residue definition of the chlormequat cation is appropriate for plant commodities for compliance with MRLs (enforcement). It is proposed to maintain the residue definition as applying to the cation, which is the current residue definition.

It is noted that parent chlormequat chloride was the predominant residue in plants in the metabolism studies and was the only measured component in the supervised field trials. Minor metabolites that were observed (choline, betaine, serine, and glycine) are not of toxicological concern, with most of these being biochemicals. A residues definition of parent only is therefore supported for dietary risk assessment in plant commodities.

A residue definition for plant commodities for both enforcement and dietary risk assessment of chlormequat cation is proposed.

Animal commodities

Parent was the only compound identified in goat kidney, liver and muscle (83, 42 and 76% TRR respectively). It was also the only identified compound in milk accounting for < 5% TRR (0.002–0.011 mg eq/kg).

Parent chlormequat chloride was the only identified compound in the poultry metabolism study. It was found in kidney and liver and as a major component in egg yolk.

A residue definition of chlormequat cation is proposed for animal commodities for compliance with MRLs (enforcement) and for dietary risk assessment.

The octanol-water partition coefficient (log P_{ow}) at pH 7 (25 °C) is -3.47. There is no evidence from the feeding studies to suggest that there is significant potential for bioaccumulation in fat tissues.

Definition of the residue (for compliance with the MRL for plant and animal commodities and for estimation of dietary intake for plant and animal commodities): *Chlormequat cation*.

The residue is not fat soluble.

Results of supervised residue trials on crops

The Meeting received supervised trial data for foliar application of chlormequat chloride to grapes, barley, oats, rye and wheat. European, Australian, South African, New Zealand, and South American GAP information for cereal crops, and Indian GAP information for grapes were provided.

All results listed below are for residues reported as chlormequat cation.

Grapes

The GAP in India for grapes is for 3 foliar applications per season, the first and second at 500 and 1000 g ai/ha after the 'April pruning' (which is conducted shortly after harvest of the crop), and the third at 250 g ai/ha, and made after the 'October pruning' (before flowering), with a PHI of 91 days. The 'October pruning' takes place in October and November and is restricted to that window by weather conditions. Harvest generally takes place in March-April.

A series of trials was conducted in accordance with the Indian GAP (two applications 4–5 days apart at 500 and 1000 g ai/ha in April, and a third application at 250 g ai/ha in October). Grapes were sampled at two intervals at each site, immature grapes at 79–117 days after the last application, and mature grapes at 120–150 days after the last application. The application and sampling timings are considered representative of viticultural practice in the hot tropical region of India, in which approximately 70% of the Indian grape crop is grown.

Residue of chlormequat cation in mature grapes at harvest after treatment in accordance with GAP were < 0.04 (6) mg/kg.

It is noted that at two additional trial sites, a fourth application of chlormequat chloride was made. However, as no residues were found above the LOQ (0.04 mg/kg expressed as chlormequat cation) in these trials, the results are still considered to be representative of the residues expected after treatment in accordance with GAP.

The Meeting estimated a maximum residue level of 0.04* mg/kg for chlormequat cation in grapes, together with an STMR and an HR of 0.04 mg/kg.

Oilseeds

Cottonseed

The Meeting decided to withdraw its previous recommendation of 0.5 mg/kg for cotton seed (SO 0691) as no GAP information or supporting residue data for cotton was provided.

Rape seed

The Meeting decided to withdraw its previous recommendation 5 mg/kg for rape seed (SO 0495) and the associated processed commodity rape seed oil, crude (OC 0495) as no GAP information or supporting residue data for rape seed was provided.

Cereals

A large residue data set for trials conducted in various countries in Europe across several growing seasons was available for barley, oats, rye and wheat.

Barley

No trials are available matching the GAP for barley in Ireland (2 × applications to winter barley, one in autumn at 562.5 g ai/ha and the second the following spring at 1500 g ai/ha, with application up to first node, BBCH 31).

The GAP in the UK for barley is for a single application at 1650 g ai/ha, with application recommended for mid tillering to just prior to first node detectable (BBCH 25–30). A harvest withholding period is not stated.

Trials in barley were conducted in Europe, with application at a target growth stage of BBCH 32 (second node visible, northern Europe trials) and 37 (flag leaf emergence, southern Europe trials). It is noted that these trials are conducted with slightly later applications than recommended on the label, however for barley, growth stage at application around the tillering and stem elongation stage does not appear to have a critical effect on residues in harvested grain, with residues after application at BBCH 37 not differing significantly from the residues after application at BBCH 32. Trials for both application timings are therefore considered representative of the residues expected in barley after treatment in accordance with UK GAP.

Residues of chlormequat (as the cation) in barley in trials conducted in accordance with GAP were < 0.04, 0.062, 0.12, 0.17, 0.31 (2), 0.32, 0.36, 0.38, 0.59, 0.60, 0.65, 0.71, 0.78, 0.93, and 1.1 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg for barley, confirming the previous recommendation, with an STMR of 0.37 mg/kg.

Oats

The critical GAP for chlormequat in oats is in Switzerland, with a single application at 1840 g ai/ha made at BBCH 30–33 (beginning of stem elongation to the third node). No harvest withholding period is stated.

Trials in oats were conducted in Europe, with application at a target growth stage of BBCH 32 (second node visible, northern Europe trials) and 37 (flag leaf emergence, southern Europe trials). For oats, application timing does have an effect on residues at harvest, so only the trials within the application window stated on the label are considered to be in accordance with the GAP.

Residues of chlormequat (as the cation) in oats in trials with the application timing in accordance with the Swiss GAP were 0.54, 0.67, 0.90, 1.3, 1.6, 2.1, and 2.2 mg/kg.

As some of the trials were conducted with application rates outside ±25% of the GAP, the residues were adjusted proportionally for MRL estimation (adjustment factors ranged from 1.10–1.37×). After adjustment, residues of chlormequat cation were 0.68, 0.90, 1.0, 1.5, 2.0, 2.7, and 2.9 mg/kg.

The Meeting estimated a maximum residue level of 6 mg/kg for oats, together with an STMR of 1.5 mg/kg.

However, this GAP results in an exceedance of the ARfD, at 110% of the ARfD, for children in Canada consuming oat flakes.

The next most critical GAP is the UK GAP, with a single application at 1650 g ai/ha made at before the third node is detectable (BBCH 33). No harvest withholding period is stated.

Residues in trials matching the UK GAP are: 0.54, 0.67, 0.90, 1.3, 1.6, 2.1, and 2.2 mg/kg.

Therefore, the Meeting estimated a maximum residue level of 4 mg/kg, replacing the previous recommendation of 10 mg/kg, together with an STMR of 1.3 mg/kg.

Rye

The critical GAP for chlormequat chloride in rye is in Latvia, with a single application at 2250 g ai/ha made up to the second node stage (BBCH 21–32). A withholding period is not stated. No trials were conducted in accordance with this GAP, however trials with a lower application rate but the correct application timing are available and residues can be adjusted using the proportionality principle.

Trials in rye were conducted in Europe, with application at a target growth stage of BBCH 32 (second node visible, northern Europe trials) and 37 (flag leaf emergence, southern Europe trials). It is noted that the southern European trials are conducted with applications outside the growth stage window on the label, however for rye, growth stage at application around the tillering and stem elongation stage does not appear to have a critical effect on residues in harvested grain, with residues in mature grain after application at BBCH 37 not differing significantly from the residues after

application at BBCH 32. Trials for both application timings in rye are therefore considered representative of the residues expected in rye grain after treatment in accordance with the Latvian GAP (after appropriate adjustment for proportionality).

Residues of chlormequat (as the cation) in rye at harvest maturity in trials conducted in Europe were 0.16, 0.25, 0.26, 0.29, 0.40, 0.52, 0.65, 0.69, 0.73, 0.78, 0.85, 0.93, 1.1, 1.5, 2.0, and 2.2 mg/kg.

After adjustment to the application rate specified in the Latvian GAP (proportionality factors of 1.36–1.84×) residues of chlormequat cation were 0.22, 0.37, 0.42, 0.43, 0.65, 0.71, 1.1 (4), 1.3, 1.4, 1.6, 2.8, 3.0, and 3.4 mg/kg.

The Meeting estimated a maximum residue level of 6 mg/kg for rye, replacing the previous recommendation of 3 mg/kg, together with an STMR of 1.1 mg/kg.

Wheat

No trials are available matching the GAP for Japan (a single application of 2300 g ai/ha made 10–20 days before heading [which corresponds to BBCH 51], at 40–60 cm plant height) as it is not clear the application timings in the trials corresponds to the GAP.

The GAP for Argentina is a single application of 2025 g ai/ha, made between tillering until the first node (BBCH 21–31), with no withholding period specified. Trials in accordance with this GAP are not available to the Meeting, however European trials with different application rates that can be considered using the proportionality principle are available.

Trials in wheat were conducted in Europe, with application at a target growth stage of BBCH 32 (second node visible, northern Europe trials) and 37 (flag leaf emergence, southern Europe trials). It is noted therefore that some trials are conducted with applications outside the growth stage window on the label, however for wheat, growth stage at application around the tillering and stem elongation stage does not appear to have a critical effect on residues in harvested grain, with residues in mature grain after application at BBCH 37 not differing significantly from the residues after application at BBCH 32. Trials for both application timings in wheat are therefore considered representative of the residues expected in wheat grain after treatment in accordance with the Argentine GAP (after appropriate adjustment for proportionality).

Residues of chlormequat (as the cation) as measured were < 0.05 (2), 0.05 (3), 0.078, 0.11, 0.16, 0.20, 0.23, 0.30, 0.34, 0.35 (2), 0.36, 0.47, 0.48 (3), 0.57 (3), 0.62 (2), 0.68, 0.74, and 1.0 mg/kg.

After adjustment to the application rate specified in the Argentine GAP (proportionality factors of 1.21–2.89×, and excluding the two trials with <LOQ residues) residues of chlormequat cation were 0.06, 0.07, 0.09, 0.10, 0.14, 0.32, 0.33, 0.46, 0.47 (2), 0.58 (3), 0.61, 0.65 (2), 0.66, 0.74, 0.77 (2), 0.83 (2), 0.85, 0.95, and 1.3 mg/kg.

The Meeting estimated a maximum residue level of 2 mg/kg for wheat, replacing the previous recommendation of 3 mg/kg, together with an STMR of 0.58 mg/kg.

Triticale

The critical GAP for triticale is in Ireland, with a single application of 1875 g ai/ha, with a recommended latest application timing of BBCH 31.

No data for triticale was available to the Meeting. However, the Meeting noted that rye, triticale and wheat are all in the Codex subgroup of wheat and similar grains. Residue data is available for rye and wheat, and this data has been proportionally adjusted for GAPs that involve higher application rates than the Irish GAP for triticale.

It was noted that, after proportional adjustment to the Irish GAP for triticale, residues in rye were higher than those in wheat.

After adjustment to the Irish GAP for triticale, the rye residue data set is: 0.18, 0.31, 0.35, 0.36, 0.54, 0.59, 0.92 (4), 1.1, 1.2, 1.3, 2.3, 2.5, and 2.8 mg/kg.

The Meeting estimated a maximum residue level of 5 mg/kg for triticale, replacing the previous recommendation of 3 mg/kg, together with an STMR of 0.92 mg/kg, based on the proportionally adjusted rye data.

Animal feeds – forages

Grazing of forage from cereal grain crops is not common practice in Europe and is precluded in conjunction with agricultural chemical use unless specifically allowed by label instructions. Noting the critical GAPs considered for barley (UK), oats (UK), rye (Latvia) and triticale (Ireland), median and highest residues for barley, oat, rye and triticale forage have therefore not been estimated.

Wheat forage

The GAP considered for wheat is in Argentina (1 × 2025 g ai/ha application at BBCH 21–31) and the label does not restrict grazing.

Residue data is available from trials conducted in Europe for wheat forage sampled at intervals of 0, 14, 28, and 42 days after application.

The Meeting considered that residues in forage sampled at 14 ± 2 days after application, the shortest interval for which data is available and at which grazing would be likely to occur in common agricultural practice, would give the most robust and realistic estimate of median and highest residues in forages.

Residues of chlormequat cation in wheat forage from trials conducted in Europe 14 ± 1 days after an application at 1000 or 1500 g ai/ha were 4.3, 4.4, 6.7, 7.8, and 13 mg/kg.

After proportionality adjustment to the Argentine GAP residues of chlormequat cation in wheat forage (fresh weight) were 5.2, 5.7, 8.7, 10, and 25 mg/kg.

The Meeting estimated a median and a highest residue of 8.7 and 25 mg/kg respectively for wheat forage (fresh weight basis).

Animal feeds – straws and fodders

Residue data is available from trials conducted across several seasons in Europe for barley, oat, rye and wheat straw collected at harvest after application at BBCH 32–39 at target rates of 700, 1000, or 1500 g ai/ha (the majority of trials were conducted at a target application rate of 1500 g ai/ha).

Barley straw

The critical GAP for barley is in the UK (1 × 1650 g ai/ha application at BBCH 25–30).

Residues of chlormequat (as the cation) in barley straw at harvest, from trials conducted in Europe, after an application in accordance with the UK GAP for barley were < 0.39, 0.62, 1.2 (2), 1.9, 2.6, 2.7, 3.2, 5.1, 5.2, 5.5, 5.9, 6.7, 7.1, 26, and 30 mg/kg (as received), or < 0.44, 0.70, 1.3 (2), 2.1, 2.9, 3.0, 3.6, 5.7, 5.8, 6.2, 6.6, 7.5, 8.0, 29, and 34 mg/kg (dry weight basis).

The Meeting estimated a maximum residue level of 50 mg/kg for barley straw and fodder, dry, together with median and highest residues of 4.15 and 30 mg/kg respectively.

Oat straw

The critical GAP for oats (Switzerland, 1 × 1840 g ai/ha application, BBCH 30–33 could not be used for estimation of maximum residue levels due to acute dietary intake exceedance) and the next highest GAP (UK, 1 × 1650 g ai/ha application at <BBCH 33) was used instead.

Residues of chlormequat cation in oat straw at harvest from trials conducted in Europe, and matching the timing and application rate for the UK GAP () were < 0.16, 0.39, 0.43, 0.93, 1.8, 2.4, and 3.5 mg/kg (as received), or < 0.18, 0.43, 0.48, 1.0, 2.0, 2.7, and 3.9 mg/kg (dry weight basis).

The Meeting estimated a maximum residue level of 7 mg/kg for oat straw and fodder, dry, together with median and highest residues of 0.93 and 3.5 mg/kg respectively.

Rye straw

The critical GAP for rye is in Latvia (1 × 2250 g ai/ha application, at BBCH 21–32).

Residues of chlormequat cation in rye straw at harvest from trials conducted in Europe 14 ± 1 days after an application at a target rate of 1500 g ai/ha, residues were 0.56, 1.1 (2), 1.2, 1.3, 2.4, 2.6 (2), 2.7, 3.3, 3.6, 3.7, 4.7, 4.9, 5.5, and 6.0 mg/kg (as received).

After proportional adjustment of the residues to the Latvian GAP for rye, residues of chlormequat cation in rye straw were 0.84, 1.6 (2), 1.9, 2.1, 3.7, 4.0, 4.2 (2), 5.0, 5.3, 5.6, 6.6, 7.5, 7.9, and 8.9 mg/kg (as received), or 0.95, 1.8 (2), 2.2, 2.4, 4.2, 4.5, 4.8 (2), 5.7, 6.0, 6.4, 7.5, 8.5, 9.0, and 10 mg/kg (dry weight basis).

The Meeting estimated a maximum residue level of 20 mg/kg for rye straw and fodder, dry, together with median and highest residues of 4.2 and 8.9 mg/kg respectively.

Triticale straw

The critical GAP for triticale is in Ireland (1 × 1875 g ai/ha application, up to BBCH 31).

Residue data for triticale straw is not available. However, data is available for rye and wheat straw.

Residues of chlormequat cation at harvest in wheat straw (adjusted to the Irish GAP for triticale and eliminating the < LOQ residues) are 0.58, 2.7, 3.8, 4.0 (2), 5.7, 5.9, 7.0, 8.5, 8.9, 10, 12, 14, 15, 19 (2), 22, 26 (2), 30 (2), 34, and 51 mg/kg (as received), or 0.65, 3.0, 4.2, 4.4 (2), 6.4, 6.6, 7.8, 9.5, 9.9, 11, 13, 15, 16, 21 (2), 25, 29 (2), 33 (2), 38, and 57 mg/kg (dry weight).

The Meeting estimated a maximum residue level of 80 mg/kg for triticale straw and fodder, dry, together with median and highest residues of 12 and 51 mg/kg respectively.

Wheat straw

The critical GAP for wheat is in Argentina (1 × 2025 g ai/ha application, BBCH 21–31).

Residues of chlormequat cation in wheat straw at harvest from trials conducted in Europe after an application at 700, 1000, 1500 g ai/ha were < 0.39 (5), 0.47, 1.5, 3.2 (3), 3.3, 4.8, 6.3, 7.0, 7.3, 7.4, 7.8, 10, 12, 13, 15 (2), 19, 21 (2), 24 (2), and 41 mg/kg (as received).

After proportionality adjustment to the Argentine GAP (eliminating the <LOQ residues) residues of chlormequat cation in wheat straw were 0.63, 2.9, 4.1, 4.3 (2), 6.2, 6.4, 7.6, 9.2, 9.6, 11, 13, 15, 16, 20 (2), 24, 28 (2), 32 (2), 37, and 55 mg/kg (as received), or 0.72, 3.3, 4.7, 4.9 (2), 7.0, 7.3, 8.6, 10, 11, 13, 15, 17, 18, 23 (2), 27, 32 (2), 36 (2), 42, and 63 mg/kg (dry weight basis).

The Meeting estimated a maximum residue level of 80 mg/kg for wheat straw and fodder, dry, together with median and highest residues of 13 and 55 mg/kg respectively.

The Meeting withdrew the previous recommendation of 30 mg/kg for cereal straw and fodder, dry.

Maize fodder

The current MRL of 5 mg/kg for maize fodder (dry) (AS 0645) should be withdrawn as no GAP information for maize or supporting residue data was provided.

Fate of residues during processing

Barley

A processing study for chlormequat chloride in barley was provided to the Meeting. The processing factors determined from that study are tabulated below.

Processing factors for chlormequat chloride in barley

Processed fraction	Processing factor (parent)	Best estimate PF	RAC STMR	STMR-P
Pearl (pot) barley	*0.06,* 0.8, 0.9, 1.0, 1.0	0.9	0.37	0.33
Malt	*0.69,* 0.9, 0.9, 0.9, 1.0	0.9		0.33
Spent grain	0.01, 0.02, 0.02, 0.03	0.02		0.007
Beer	*0.015,* 0.1, 0.2, 0.2, 0.2	0.2		0.074

Processing factors in italics were obtained from studies considered by the 1994 and/or 2000 JMPRs.

The Meeting estimated STMR-P values of 0.33, 0.33, 0.007, and 0.074 mg/kg for pearl (pot) barley, malt, spent grain, and beer respectively.

Oats

A processing study for chlormequat chloride in oats was provided to the Meeting. The processing factors determined from that study are tabulated below.

Processing factors for chlormequat chloride in oats

Processed fraction	Processing factor (parent)	Best estimate PF	RAC STMR	STMR-P
Oat kernels	1.0	1.0	1.3	1.3
Oat flakes	*0.1, 0.25, 0.27,* 0.8, 0.9, 1.0, 1.2	0.80		1.04

Processing factors in italics were obtained from studies considered by the 1994 and/or 2000 JMPRs.

The Meeting estimated an STMR-P of 1.04 mg/kg for oat flakes, based on the UK GAP.

Rye

A processing study for rye was not provided to the Meeting. Key processing factors for rye from studies supplied to the 1994 JMPR are tabulated below.

Processing factors for chlormequat chloride in rye

Processed fraction	Processing factor (parent)	RAC MRL	Processed commodity MRL	RAC STMR	STMR-P
Rye bran	3.2	6	20	1.1	6.6
Rye flour	0.99		-		1.1
Rye wholemeal	1.3		8		1.4
Rye wholemeal bread	0.95		-		1.0

The Meeting estimated a maximum residue level of 20 mg/kg for rye bran, unprocessed, replacing the previous MRL of 10 mg/kg, together with an STMR-P of 6.6 mg/kg.

The Meeting estimated a maximum residue level of 8 mg/kg for rye wholemeal, replacing the previous recommendation of 4 mg/kg, together with an STMR of 1.4 mg/kg.

The Meeting withdrew the previous recommendation of 3 mg/kg for rye flour, as residues do not concentrate in rye flour and will be covered by the MRL for the raw commodity.

The Meeting estimated new STMR-P values of 1.1 and 1.0 mg/kg for rye flour and rye wholemeal bread respectively.

Wheat

A processing study for chlormequat chloride in wheat was provided to the Meeting. The processing factors determined from that study are tabulated below.

Processing factors for chlormequat chloride in wheat

Processed fraction	Processing factor (parent)	Best estimate PF	RAC MRL	Processed commodity MRL	RAC STMR	STMR-P
Flour (type 550)	0.19, 0.28, 0.29, 0.30, *0.41*	0.29	2	-	0.58	0.17
Bran	*2.5,* 2.8, 2.9, 3.1, 3.4, *4.6*	3.0		7		1.7
Wholemeal flour	0.86, 0.91, 1.0, 1.1	0.955		-		0.55
Wholemeal	*1.0, 1.4*	1.2		-		0.70
Wholemeal bread	0.49, 0.51, 0.53, 0.55, *0.63, 0.79*	0.54		-		0.31

Processing factors in italics were obtained from studies considered by the 1994 and/or 2000 JMPRs.

The Meeting estimated a maximum residue level of 7 mg/kg for wheat bran, unprocessed, replacing the previous recommendation of 10 mg/kg, together with an STMR-P of 1.7 mg/kg.

The Meeting withdrew the previous recommendations of 2 and 5 mg/kg for wheat flour and wheat wholemeal respectively, as residues do not concentrate in these commodities.

The Meeting estimated STMR-P values of 0.17, 0.55, 0.70, and 0.31 mg/kg for type 550 (white) flour, wholemeal flour, wheat wholemeal, and wholemeal bread respectively.

Farm animal dietary burden

Farm animal feeding studies in lactating cattle and laying hens were provided to the Meeting.

Lactating cattle

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 and 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 in liver.

The concentrations of chlormequat chloride in skim milk were similar to those in whole milk, but they were two times lower than those in cream because of the solubility of the compound in water.

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. No adequate information on stability was provided to the 2000 JMPR.

However, a storage stability study in animal commodities was provided to the current Meeting, and this confirmed that residues of chlormequat chloride were stable in cattle meat, milk and eggs for up to 12 months of frozen storage. Therefore, the results of the cattle feeding study are unlikely to have been adversely affected by sample degradation during storage.

Laying hens

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 provided to the 2000 JMPR.

However, a storage stability study in animal commodities was provided to the current Meeting, and this confirmed that residues of chlormequat chloride were stable in cattle meat, milk and eggs for up to 12 months of frozen storage. Therefore, the results of the laying hen feeding study are unlikely to have been adversely affected by sample degradation during storage.

Livestock dietary burden

Dietary burden calculations for cattle and poultry are provided below. The dietary burdens were estimated using the OECD diets listed in Appendix IX of the 2016 edition of the FAO Manual.

Summary of livestock dietary burden (ppm chlormequat cation)

	USA-Canada		EU		Australia		Japan	
	Max	Mean	Max	Mean	Max	Mean	Max	Mean
Beef cattle	10.5	3.34	24.5	8.59	100[a]	34.8[b]	1.72	1.72
Dairy cattle	21.3	8.02	24.5	8.59	66.8[c]	22.8[d]	1.09	1.09
Broiler hens	1.70	1.70	1.41	1.41	0.60	0.60	0.097	0.097
Laying hens	1.70	1.70	11.4[e]	4.89[f]	0.60	0.60	0.58	0.58

[a] Highest maximum dietary burden for beef cattle suitable for estimation of MRLs for mammalian meat and offal.

[b] Highest mean dietary burden for beef cattle suitable for estimation of STMRs for mammalian meat and offal.

[c] Highest maximum dietary burden for dairy cattle suitable for estimation of MRLs for milk.

[d] Highest mean dietary burden for dairy cattle suitable for estimation of STMRs for milk.

[e] Highest maximum dietary burden for broiler and layer poultry suitable for estimation of MRLs for poultry meat, offal and eggs.

[f] Highest mean dietary burden for broiler and layer poultry suitable for estimation of STMRs for poultry meat, offal and eggs.

Animal commodity maximum residue levels

Mammals

The highest maximum dietary burden for dairy cattle was 66.8 ppm while the highest mean dietary burden was 22.8 ppm (both numbers as chlormequat cation).

	Feed level (ppm, as the cation)	Residues in milk (mg/kg, as the cation)
MRL dairy cattle		
Feeding study	28	0.15
	93	0.26
Dietary burden and highest residue	66.8	0.22
STMR dairy cattle		
Feeding study	9.3	0.039
	28	0.15
Dietary burden and mean residue	22.8	0.12

The Meeting estimated a maximum residue level of 0.3 mg/kg for milk, together with an STMR of 0.12 mg/kg. The Meeting withdrew the previous recommendation of 0.5 mg/kg for milk of cattle, goats and sheep.

The highest maximum dietary burden for beef cattle was 100 ppm while the highest mean dietary burden was 34.8 ppm.

	Feed level (ppm, as the cation)	Residues (mg/kg as chlormequat cation)			
		Meat	Fat	Liver	Kidney
MRL beef cattle					
Feeding study	93	0.085[a]	0.078	0.39	0.82
Dietary burden and highest residue	100	0.091	0.083	0.42	0.88
STMR beef cattle					
Feeding study	28	< 0.04	< 0.04	0.062	0.31
	93	< 0.04	0.08	0.29	0.59
Dietary burden and mean residue	34.8	< 0.04	0.04	0.086	0.34

[a] This value is from the mid dose (28 ppm as the cation) feeding level, as a higher highest residue was observed for the mid dose feeding level than for the high dose feeding level.

The Meeting estimated a maximum residue level of 0.2 mg/kg for meat from mammals other than marine mammals, together with an STMR and an HR of 0.04 and 0.091 mg/kg respectively.

The Meeting withdrew the previous recommendations of 0.2 mg/kg for meat of cattle, pigs and sheep, and for goat meat.

The Meeting estimated a maximum residue level of 0.1 mg/kg, for mammalian fat together with an STMR and an HR of 0.04 and 0.083 mg/kg respectively.

Based on the data for kidney, the Meeting estimated a maximum residue level of 1 mg/kg for edible offal, mammalian, together with an STMR and an HR of 0.086 and 0.42 mg/kg for liver and an STMR and an HR of 0.34 and 0.88 mg/kg for kidney.

The Meeting withdrew the previous recommendations of 0.1 and 0.5 mg/kg for liver and kidney.

Poultry

The highest maximum dietary burden for poultry was 11.4 ppm, while the highest mean dietary burden was 4.89 ppm, for estimation of MRLs and dietary parameters for both meat and eggs (both values expressed as the cation).

No residues of chlormequat chloride above the LOQ (0.05 mg/kg in the study) were found at feeding levels of 6, 18 or 60 ppm (as the chloride, or 4.65, 14, or 46.5 ppm as the cation) in the meat or fat of laying hens.

The Meeting therefore estimated maximum residue levels of 0.04* mg/kg for poultry meat (confirming the previous recommendation), and 0.04* mg/kg for poultry fats, together with STMR and HR values of 0.04 mg/kg for both meat and fat (these values are for the cation).

	Feed level (ppm, as the cation)	Residues in liver (mg/kg, as the cation)	Residues in eggs (mg/kg, as the cation)
MRL poultry			
Feeding study	4.65	0.07	0.046
	14	0.078	0.093
Dietary burden and highest residue	11.4	0.072	0.079
STMR poultry			
Feeding study	4.65	0.039	< 0.039
	14	0.054	0.078
Dietary burden and mean residue	4.89	0.04	0.04

The Meeting estimated a maximum residue level of 0.1 mg/kg, confirming the previous recommendation, together with an STMR and HR of 0.04 and 0.072 mg/kg respectively, for poultry edible offal.

The Meeting estimated a maximum residue level of 0.1 mg/kg, confirming the previous recommendation, together with an STMR and an HR of 0.04 and 0.079 mg/kg respectively, for eggs.

RECOMMENDATIONS

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

The residue definition (for compliance with the MRL and dietary risk assessment) in plant and animal commodities remains as previously recommended: *chlormequat cation*.

The residue is not fat soluble.

CCN	Commodity name	Recommended maximum residue level, mg/kg		STMR (P), mg/kg	HR (P), mg/kg
		New	Previous		
GC 0640	Barley	2	2	0.37	-
AS 0640	Barley straw and fodder, dry	50 (dw)		4.15 (as)	30 (as)
SO 0691	Cotton seed	W	0.5		
MO 0105	Edible offal (mammalian)	1		Liver: 0.086 Kidney: 0.34	Liver: 0.42 Kidney: 0.88
PE 0112	Eggs	0.1	0.1	0.04	0.079
MM 0184	Goat meat	W	0.2		
FB 0269	Grapes	0.04*		0.04	0.04
MO 0098	Kidney of cattle, goats, pigs and sheep	W	0.5		
MO 0099	Liver of cattle, goats, pigs and sheep	W	0.1		
AS 0645	Maize fodder (dry)	W	7		
MF 0100	Mammalian fats (except milk fats)	0.1		0.04	0.083
MM 0095	Meat (from mammals other than marine mammals)	0.2		0.04	0.091
MM 0097	Meat of cattle, pigs and sheep	W	0.2		
ML 0106	Milks	0.3		0.12	-
ML 0107	Milk of cattle, goats and sheep	W	0.5		
GC 0647	Oats	4	10	1.3	-
AS 0647	Oat straw and fodder, dry	7 (dw)		0.93 (as)	3.5 (as)

CCN	Commodity name	Recommended maximum residue level, mg/kg		STMR (P), mg/kg	HR (P), mg/kg
		New	Previous		
PO 0111	Poultry, edible offal of	0.1	0.1	0.04	0.072
PF 0111	Poultry fats	0.04*		0.04	0.04
PM 0110	Poultry meat	0.04*	0.04*	0.04	0.04
SO 0495	Rape seed	W	5		
OC 0495	Rape seed oil, Crude	W	0.1		
GC 0650	Rye	6	3	1.1	-
CM 0650	Rye bran, unprocessed	20	10	6.6	
CF 1250	Rye flour	W	3		
AS 0650	Rye straw and fodder, dry	20 (dw)		4.2 (as)	8.9 (as)
CF 1251	Rye wholemeal	8	4	1.4	
AS 0081	Straw and fodder (dry) of cereal grains	W	30		
GC 0653	Triticale	5	3	0.92	-
AS 0653	Triticale straw and fodder, dry	80 (dw)		12 (as)	51 (as)
GC 0654	Wheat	2	3	0.58	-
CM 0654	Wheat bran, unprocessed	7	10	1.7	-
CF 1211	Wheat flour	W	2		
AS 0654	Wheat straw and fodder, dry	80 (dw)		13 (as)	55 (as)
CF 1212	Wheat wholemeal	W	5		

dw = dry weight basis; as = as received

STMR-P/STMR and HR (where required) values for processed commodities and livestock feeds for which an MRL is not required (for livestock dietary burden or dietary intake calculation)

Commodity	STMR/STMR-P (mg/kg)	HR (mg/kg)
Pearl barley	0.33	-
Malt	0.33	-
Spent grain	0.007	-
Beer	0.074	-
Oat flakes	1.04	-
Rye flour	1.1	-
Rye wholemeal bread	1.0	-
White (type 550) wheat flour	0.17	-
Wholemeal flour	0.55	-
Wheat wholemeal	0.70	-
Wheat wholemeal bread	0.31	-
Wheat forage (as received basis)	8.7	25

DIETARY RISK ASSESSMENT

Long-term dietary exposure

The International Estimated Daily Intakes (IEDIs) of chlormequat chloride were calculated for the 17 GEMS/Food cluster diets using STMRs/STMR-Ps estimated by the current Meeting. The results are shown in Annex 3 of the 2017 Report.

The ADI for chlormequat chloride is 0–0.05 mg/kg bw/day (or 0–0.0388 mg/kg bw/day expressed as chlormequat cation). The calculated IEDIs for chlormequat chloride were 1–7% of the maximum ADI. The Meeting concluded that the long term intakes of residues of chlormequat chloride, when used in accordance with GAPs that have been considered by JMPR, are unlikely to pose a public health concern.

Short term dietary exposure

The International Estimated Short Term Intakes (IESTIs) of chlormequat chloride were calculated for food commodities using HRs/HR-Ps or STMRs/STMR-Ps estimated by the current Meeting. The results are shown in Annex 4 to the 2017 Report.

The ARfD for chlormequat chloride is 0.05 mg/kg bw (or 0.0388 mg/kg bw expressed as chlormequat cation).

The calculated IESTIs for chlormequat ranged from 0–100% of the ARfD for children, and 0–50% for the general population. The Meeting concluded that the short-term intake of residues of chlormequat chloride, when used in accordance with GAPs that have been considered by JMPR, are unlikely to pose a public health concern.

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