CHLORMEQUAT-CHLORIDE

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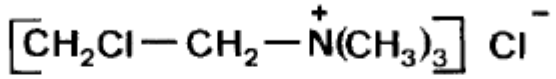

ISO common name	Chlormequat-chloride
Chemical name	(2-Chloroethyl)-trimethylammonium-chloride (IUPAC); ethanaminium, 2-chloro-*N,N,N*-trimethyl-chloride (CA; *999-81-5*)
Empirical formula	$C_5H_{13}Cl_2N$
RMM	158·1
m.p.	Begins to decompose at 245°C
v.p.	Less than 10^{-5} Pa at 20°C
Solubility	Water: more than 200 g/l at 20°C; ethanol: more than 200 g/l at 20°C; insoluble in ether and hydrocarbons
Description	The pure material is a hygroscopic white crystalline solid with a typical amine odour
Formulations	Aqueous solutions. Also formulated as aqueous solutions together with choline chloride

CHLORMEQUAT-CHLORIDE AQUEOUS SOLUTIONS

***143**/SL/(M)/–

1 **Sampling.** Take at least 500 ml.

2 **Identity test**

TLC

Stationary phase silica gel, 0·25 mm layer thickness, e.g. type G 1500 of Schleicher and Schüll or silica gel 60 of Merck

Mobile phase sodium iodide 0·1 mol/l in acetonitrile. Develop 2 times, each 15 cm high in ascending mode.

Visualisation reagent Dragendorff-reagent. Dissolve basic bismuth nitrate (1·7 g) in glacial acetic acid (20 ml) in a 1 l volumetric flask, successively add water (8 ml), a solution of 40 g potassium iodide in water, glacial acetic acid (200 ml), and make up to volume with water. Apply to the starting point about 100 µg of

* Provisional CIPAC method 1985. Prepared by the German Committee (DAPA), Chairman Dr W Dobrat. Based on a method supplied by BASF (FRG).

chlormequat-chloride in methanol, i.e. 5 µl of a 2·5% solution of chlormequat-chloride 750 g/l, or 5 µl of a 4% solution containing 460 g/l chlormequat-chloride and 320 g/l choline chloride. After developing spray with Dragendorff reagent. Under these conditions, the R_f value of chlormequat-chloride is about 0·6 and of choline chloride about 0·5.

3 Chlormequat-chloride

OUTLINE OF METHOD The active ingredient content is calculated using the content of organic chlorine, which is determined as the difference between directly titrated chloride and total chlorine by titration of chloride after hydrolysis. The titrations are carried out potentiometrically using a silver nitrate solution c ($AgNO_3$) = 0·1 mol/l.

REAGENTS

Sodium hydroxide c (NaOH) = 2·5 mol/l
Silver nitrate c ($AgNO_3$) = 0·1 mol/l (RE 24·1, CIPAC 1, *p. 762*)
Nitric acid conc., free of chloride
Methylred indicator solution (RE 18·1, CIPAC 1, *p. 756*)

APPARATUS

Potentiograph with 10 ml automatic burette and magnetic stirrer, e.g. type E 536, Metrohm
Combined solid silver electrode, e.g. type EA 246, Metrohm

PROCEDURE

(*a*) *Preparation of sample solution.* Weigh (to the nearest 0·1 mg) enough sample (w g) to contain about 140 mg of chloride (e.g. 950 mg chlormequat-chloride 750 g/l or 850 mg of a solution containing 460 g/l chlormequat-chloride and 320 g/l choline chloride) into a 100 ml volumetric flask and make up to volume with water.

(*b*) *Determination of chloride.* Pipette 20·0 ml of the sample solution into a 100 ml beaker, dilute with water (30 ml) and add indicator solution. Acidify with conc. nitric acid using methyl red as indicator and titrate potentiometrically with 0·1 mol/l silver nitrate (t_1). Carry out a blank experiment in the same manner without weighed-in sample (take 30 ml of water, add indicator etc.).

Potentiograph settings:

Range	1000 mV
Counter voltage	800 mV
Titration rate	1 ml/min

(*c*) *Determination of total chlorine.* Pipette 10 ml of the sample solution into a 250 ml conical flask with ground-glass stopper and reflux with 2·5 mol/l sodium hydroxide (50 ml) for 2 hours. Then rinse the reflux condenser with about 40 ml of water and combine this with the reaction solution. Acidify the combined solutions in the conical flask with conc. nitric acid using methyl red as indicator, and after cooling to ambient temperature titrate the solution potentiometrically

with silver nitrate 0·1 mol/l (t_2). Carry out an experiment in the same manner without weighed-in sample (take 50 ml of the NaOH solution and follow procedure (*c*)). Potentiograph settings see (*b*).

(*d*) *Calculation*

$$\text{Chloride content} = \frac{t_1 - b_1}{w} \times 177{\cdot}3 \times N \text{ g/kg (Procedure } (b))$$

$$\text{Total chlorine content} = \frac{t_2 - b_2}{w} \times 354{\cdot}5 \times N \text{ g/kg (Procedure } (c))$$

$$\text{Chlormequat-chloride content} = \frac{\text{total chlorine} - \text{chloride}}{35{\cdot}45} \times 158{\cdot}1$$

$$= (\text{total chlorine} - \text{chloride}) \times 4{\cdot}46 \text{ g/kg}$$

where:

t_1 = ml required for the chloride determination
b_1 = ml required for the blank of the chloride determination
t_2 = ml required for the total chlorine determination
b_2 = ml required for the blank of the total chlorine determination
w = mass of sample taken (g)
N = Normality of the silver nitrate solution

Repeatability r_{95} = 15·8 g/kg at 654 g/kg active ingredient content
Reproducibility R_{95} = 27·4 g/kg at 654 g/kg active ingredient content

Based on results of a study with 7 participants and 36 values.