CHLORMEQUAT-CHLORIDE
143

See chlormequat-chloride, CIPAC D, *p.* 39.

CHLORMEQUAT-CHLORIDE TECHNICAL
[*]**143**/TK/M2/-

ION CHROMATOGRAPHIC METHOD

1 Sampling. Take at least 100 g.

2 Identity tests
2.1 HPLC. Use the HPLC method below. The retention time of chlormequat-chloride for the sample solution should not deviate by more than 1 s from that for the calibration solution.
2.2 TLC. As for chlormequat-chloride aqueous solutions **143**/SL/(M)/2, CIPAC D, *p.* 39.
2.3 Infrared. Prepare potassium bromide discs from the sample and from pure chlormequat-chloride using 1.3 to 1.5 mg material and 300 mg potassium bromide. Scan the discs from 4000 to 400 cm^{-1}. The spectrum produced from the sample should not differ significantly from that of the standard.

3 Chlormequat-chloride

OUTLINE OF METHOD Chlormequat-chloride is dissolved in water and determined by ion chromatography on a silica based cation exchange column using an acetone-water-ethylenediamine mixture as eluent, conductivity detection and external standardisation.

REAGENTS
Chlormequat-chloride standard of known purity
Acetone HPLC grade
Water HPLC grade
1,2-Diaminoethane (ethylene diamine)
Oxalic acid dihydrate
Propan-2-ol

Nitric acid solution, $c(HNO_3) = 2.5$ mmol/l

Mobile phase. Dissolve oxalic acid dihydrate (440 mg) and ethylenediamine (150 mg) in water (900 ml) and add acetone (100 ml). Mix thoroughly and degas by vacuum filtration using, a 0.45 µm filter.

Calibration solution. Weigh (to the nearest 0.01 mg) in duplicate into two volumetric flasks (100 ml) about 150 mg (*s* mg) of chlormequat-chloride standard. Dissolve in water and fill to the mark with water. Mix well (solutions C_A and C_B).

APPARATUS

High performance liquid chromatograph equipped with an automated loop injector (20 µl) and a conductivity detector suitable for non-suppressed chromatography (Metrohm 690, IC or equivalent)

Column stainless steel, 250×4 or 4.6 (i. d.) mm, packed with Zorbax SCX 5 µm

Electronic integrator or *data system*

PROCEDURE

(a) Operating conditions (typical):

Eluent flow rate	0.8 ml/min
Column temperature	ambient
Injection volume	20 µl
Detector	conductivity detector, suitable for non-suppressed ion chromatography
Measuring range	20 mS
Background conductivity	400 mS
Retention time	chlormequat-chloride: about 10 min
Run time	20 min

(b) Column equilibration. Before carrying out the analyses, rinse the column with propan-2-ol (100 ml) and then with aqueous nitric acid (100 ml, $c = 2.5$ mmol/l). Equilibrate the column by pumping mobile phase through the system until a stable baseline is obtained.

(c) Linearity check. Check the linearity of the detector response by injecting 20 µl of solutions with chlormequat-chloride concentrations 0.5, 1 and 2 times that of the calibration solution. Ensure that the concentrations of the solutions are within the linear range of the detector, otherwise alter the weighings or the dilutions. Inject each calibration solution at least twice and determine the mean peak area to mass ratios. The single values should differ by less than 0.5 % from the mean value, otherwise

repeat the calibration. A plot of the mean peak area versus the mass should give a linear curve. The regression coefficient should not be less than 0.999.

(d) Sample preparation. Weigh (to the nearest 0.01 mg) into a volumetric flask (100 ml) sufficient sample to contain about 150 mg (*w* mg) of chlormequat-chloride. Dissolve in water and fill to the mark with water. Mix well (solution S).

(f) Determination. Inject in duplicate 20 µl portions of the calibration and sample solutions in the following sequence:

$$C_A, C_A, S_1, S_1, S_2, S_2, C_B, C_B, S_3 ......etc.$$

Record the areas of the peaks. Average the response factors of each pair of injections. Calculate the mean value of the pairs of response factors bracketing the injections of sample solution injections and use this value for calculating the chlormequat-chloride contents of the bracketed sample solution injections.

(g) Calculation

$$\text{Response factor } f = \frac{s \times P}{H_s}$$

$$\text{Chlormequat-chloride content} = \frac{H_w \times f}{w} \text{ g/kg}$$

where:
f = mean response factor
H_s = peak area of chlormequat-chloride in the calibration solution
H_w = peak area of chlormequat-chloride in the sample solution
s = mass of chlormequat-chloride in the calibration solution (mg)
w = mass of sample taken (mg)
P = purity of chlormequat-chloride standard (g/kg)

Repeatability r = 12 g/kg at 720 g/kg active ingredient content
Reproducibility R = 33 g/kg at 720 g/kg active ingredient content

Based on a study with 10 participants and 20 values.

CHLORMEQUAT-CHLORIDE AQUEOUS SOLUTIONS
[*]**143**/SL/M2/-

ION CHROMATOGRAPHIC METHOD

SCOPE The method can also be used for formulations containing choline chloride.

1 Sampling. Take at least 1 l.

2 Identity tests. As for **143**/TK/M2/2.

3 Chlormequat-chloride. As for **143**/TK/M2/3.

Repeatability r	= 5 g/kg at 405 g/kg active ingredient content
	= 10 g/kg at 560 g/kg active ingredient content
	= 15 g/kg at 630 g/kg active ingredient content
	= 13 g/kg at 672g/kg active ingredient content
Reproducibility R	= 18 g/kg at 405 g/kg active ingredient content
	= 29 g/kg at 560 g/kg active ingredient content
	= 26 g/kg at 630 g/kg active ingredient content
	= 18 g/kg at 672g/kg active ingredient content