

METHOD STATEMENT

Determinand:

Determination of of chlormequat and mepiquat.

Matrix:

Sample Type: Treated, Ground and Raw Waters.

Principle of Method:

Samples are adjusted to pH7 with a phosphate buffer prior to extraction by weak cation exchange solid phase extraction cartridges. The solid phase extraction cartridges are eluted with Acetonitrile : Formic Acid, 4 : 1. The sample extracts are analysed by hilic high performance liquid chromatography using a triple quadrupole mass spectrometer as a detector. The analytes are separated and then identified and quantified with mass spectrometric detection (MSD) in multiple reaction monitoring (MRM) mode. Quantitation is based on an internal standardisation procedure.

Interferences:

HPLC-MS/MS is an extremely selective technique and interferences should only be encountered very rarely. Any interfering compounds would have to display the identical MRM transition at the same retention time, this is extremely unlikely in potable water samples. However, any compound, which passes through the extraction procedure, and has a similar liquid chromatographic retention time and mass spectrometric properties to the compound of interest, will cause interference. Samples containing high humic or fulvic loading have been demonstrated to not cause significant ion suppression for the compounds.

Performance of Method:

Range of Application:

<u>Determinand</u>	Operational Calibration Range
CHLORMEQUAT	LOD - 0.125 µg/l
MEPIQUAT	LOD - 0.125 µg/l

Limit of Detection, Uncertainty of measurement and Recoveries of Compounds

Instrument 3 – Asset 1746-1747, 1749-1750, 1853 and 1888:

<u>Determinand</u>	LOD ng/litre	<u>Direct Standards</u>				<u>Elvington Treated Water (Hard Hardness)</u>		
		Low Standard		High Standard		PCV Spike		
		% Recovery	RSD	% Recovery	RSD	% Recovery	RSD	Uncert
CHLORMEQUAT	3	95.7%	4.5%	100.2%	3.7%	102.8%	5.6%	± 14.0%
MEPIQUAT	3	95.7%	3.5%	100.1%	3.1%	100.2%	5.1%	± 10.5%

References:

Agilent 1200 Series, Reference Manuals.

Agilent 6400 QQQ LC/MS Techniques and Operation, Agilent Technologies Course Number R1893A, Student Manuals Volumes 1 and 2.

Agilent 6460 Triple Quad LC/MS System, Quick Start Guide

Agilent 6400 Triple Quad LC/MS, Maintenance and Familiarization Guides.

Agilent 6400 Triple Quad LC/MS System, Concept Guide.

R.V. Cheeseman, A.L. Wilson, A Manual on Analytical Quality Control for the Water Industry, revised by M. J. Gardner, NS 30, Water Research Centre, 1989. ISBN 0-902156-85-3.

