

METHOD STATEMENT

Determinand:

Determination of Acrylamide.

Matrix:

Sample Type: Treated, Ground and Raw Waters.

Principle of Method:

The method is a direct aqueous injection procedure. Samples are analysed by high performance liquid chromatography using a triple quadrupole mass spectrometer as a detector. The aqueous sample is injected and the organic compounds 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:

Determinand	Instrument	Operational Calibration Range
ACRYLAMIDE	Q5 & Q6	LOD - 0.250 µg/l

Limit of Detection, Uncertainty of measurement and Recoveries of Compounds

Instrument WQQQ5, Agilent 6460 LC-QQQ

<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
ACRYLAMIDE	3	103.6%	5.7%	100.8%	1.6%	102.2%	2.2%	± 6.58%

Instrument WQQQ6, Agilent 6460 LC-QQQ

<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
ACRYLAMIDE	3	103.3%	5.4%	100.8%	2.1%	104.7%	3.3%	± 11.23%

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.

