

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.

Sampling and Sample Preparation:

Sampling, samples should be collected in 500 mL coloured glass bottles which have been proven to be suitable for this analysis, with PTFE lined screw caps and contain 0.9ml of 10mg/l Sodium Thiosulphate. Storage – samples should be analysed as soon as possible after collection. When this is not possible they should be stored under refrigeration at $3\pm 2^{\circ}\text{C}$, until analysis can begin. The maximum permissible storage time prior to analysis is given below, which is either derived from BS EN ISO 5667-3: 2003 "Water Quality – Sampling – Part 3: Guidance on the preservation and handling of water samples (BS 6068-6.3:2018) or from ALS in-house data ["ALS IHD"] which is held by the Quality section.

<u>Determinand</u>	<i>Maximum period of analyte stability prior to any extraction step (days)</i>	<i>Maximum period of analyte stability after to any extraction step (days)</i>	<i>Data is quoted from BS EN ISO 5667-3: 2018 ["ISO"] or ALS in-house data ["ALS IHD"]</i>
ACRYLAMIDE	31	N/A	ALS IHD

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>	Instruments	Operational Calibration Range
ACRYLAMIDE	QQQ10 & QQQ2	LOQ - 0.250 µg/l

METHOD STATEMENT



Limit of Quantification:, Recoveries of Compounds and Uncertainty of measurement: Instrument WQQQ10, Agilent 6475 LC-QQQ

<u>Determinand</u>	<u>Uncertainty</u>	<u>Direct Standards</u>				<u>Elvington Treated Water (Hard Hardness)</u>	
		Low Standard		High Standard		PCV Spike	
		Bias	RSD	Bias	RSD	Recovery	RSD
ACRYLAMIDE	12.155%	-0.56%	4.12%	0.87%	2.98%	102.06%	3.61%

Instrument WQQQ2, Agilent 6460 LC-QQQ

<u>Determinand</u>	<u>Uncertainty</u>	<u>Direct Standards</u>				<u>Elvington Treated Water (Hard Hardness)</u>	
		Low Standard		High Standard		PCV Spike	
		Bias	RSD	Bias	RSD	Recovery	RSD
ACRYLAMIDE	9.691%	2.62%	3.88%	0.75%	1.90%	102.58%	2.07%

The highest LOQ has been applied across all instruments.

<u>Determinand</u>	WQQQ10 Limit of Quantification ($\mu\text{g L}^{-1}$)	WQQQ2 Limit of Quantification ($\mu\text{g L}^{-1}$)	METHOD (Standardised) LOQ ($\mu\text{g L}^{-1}$)
ACRYLAMIDE	0.006	0.006	0.006

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.