

METHOD STATEMENT

**Determinand:**

Metaldehyde

Matrix:

Sample Types: Treated water, Raw Ground and Raw Surface water.

Principle of Method:

The method is a large volume injection on-line solid phase extraction procedure. Samples are analysed by high performance liquid chromatography using a triple quadrupole mass spectrometer as a detector. The aqueous sample is injected by large volume injection (LVI) onto an enrichment column. The analyte is trapped on the enrichment column and then "back flushed" on to the analytical column. The analyte is identified and quantified by selected reaction monitoring (SRM) mode mass spectrometric detection. Quantitation is based on an internal standardisation procedure.

Sampling and Sample Preparation:

Sampling, samples should be collected in 500 mL amber glass bottles which have been proven to be suitable for this analysis (ALS58), with PTFE lined screw caps and contain 90 mL of 10 g L⁻¹ sodium thiosulfate solution.

Storage – samples should be analysed as soon as possible after collection. When this is not possible, they should be stored under refrigeration at 3±2°C in the dark, until analysis can begin.

Please note that the below table references the maximum period of analyte stability prior to any extraction step (in days) for the quoted matrices.

<u>Determinand</u>	<u>Drinking water</u> <u>Quoted from</u> <u>ALS in house</u> <u>data [ALS-AS-</u> <u>IHD]</u>	<u>Surface water</u> <u>Quoted from</u> <u>ALS in house</u> <u>data [ALS-AS-</u> <u>IHD]</u>	<u>Ground water</u> <u>Quoted from BS</u> <u>EN ISO 5667-</u> <u>3:2003 ["ISO"]</u>	<u>Maximu period</u> <u>of analyte</u> <u>stability post any</u> <u>extraction step</u> <u>(days)</u>
METALDEHYDE	31	28	28	N/A

Interferences

HPLC-QQQ is an extremely selective technique and interferences should only be encountered very rarely. Any interfering compounds would have to display the identical SRM 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:**

LOQ to 0.500 µg L⁻¹

Samples producing results above this range should be diluted and re-extracted.

Limit of Quantification:

<u>Determinand</u>	<u>Limit of Quantification</u> <u>QQQ5 (µg L⁻¹)</u>	<u>Limit of Quantification</u> <u>QQQ4 (µg L⁻¹)</u>	<u>LOQ Method</u> <u>Standardised (µg L⁻¹)</u>
Metaldehyde	0.006	0.005	0.006

METHOD STATEMENT



Recoveries of Compounds, Bias and Uncertainty of measurement:

WQQQQ4

<u>Determinand</u>	Uncertainty of Measurement (UoM) (%)	<u>Direct Standards</u>				<u>Elvington Treated Water</u>	
		Low Standard		High Standard		PCV Spike	
		Recovery	Bias	Recovery	Bias	Recovery	Bias
Metaldehyde	8.488	99.48	3.06	100.15	5.82	100.19	3.18

WQQQQ5

<u>Determinand</u>	Uncertainty of Measurement (UoM) (%)	<u>Direct Standards</u>				<u>Elvington Treated Water</u>	
		Low Standard		High Standard		PCV Spike	
		Recovery	Bias	Recovery	Bias	Recovery	Bias
Metaldehyde	10.857	103.03	3.03	101.38	1.38	104.63	4.63

References:

Agilent 1200 Series, Reference Manuals.

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

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