



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 coloured glass bottles which have been proven to be suitable for this analysis, with PTFE lined screw caps and contain 0.500 mL of sample preservative, 30 g L⁻¹ w/v ascorbic acid. Samples are prepared by adding sample matrix modifier solution, 18 g L⁻¹ w/v sodium thiosulfate, internal standard spiking solution and formic acid.

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:

Determinand	Limit of Quantification QQQ5 (µg L ⁻¹)	Limit of Quantification QQQ4 (µg L ⁻¹)	LOQ Method Standardised (µg L ⁻¹)
Metaldehyde	0.007	0.005	0.007

Recoveries of Compounds, Bias and Uncertainty of measurement:

WQQQQ4

Determinand	Uncertainty of Measurement (UoM) (%)	Direct Standards				Elvington Treated Water	
		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

Determinand	Uncertainty of Measurement (UoM) (%)	Direct Standards				Elvington Treated Water	
		Low Standard		High Standard		PCV Spike	
		Recovery	Bias	Recovery	Bias	Recovery	Bias



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Determinand	Uncertainty of Measurement (UoM) (%)	Direct Standards				Elvington Treated Water	
		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



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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.