METHOD STATEMENT

Determinand:
NDMA (N-Nitrosodimethylamine)

Matrix:
Waters abstracted for potable supplies

Principle of Method:
The aqueous sample is spiked with the labelled internal standard N-nitrosodimethylamine-d6 (NDMA-d6) and extracted using solid phase extraction cartridges. NDMA is eluted from the solid phase with dichloromethane and the extract is dried and concentrated using a nitrogen blow-down apparatus prior to analysis.

The extracts are analysed by injecting an aliquot of the concentrated extract onto a fused silica capillary column of a GCMSMS system operating in positive ion electron ionisation (EI) mode with SRM detection.

Sampling and Sample Preparation:
Samples should be collected in 1000 ml PET plastic bottle containing ascorbic acid (1.33 ml 3% solution – 40mg equivalent).
Samples should be extracted as soon as possible after sampling. If storage is unavoidable, samples should be kept in a refrigerator at 3 ± 2°C for a maximum of 21 days (DEFRA Report).

Interferences:
Any compound which passes through the extraction and has similar gas chromatographic and mass spectrometric properties to the analyte. Monitoring of the ratio of qualifier ion SRM transition response identifies the presence of interferences that may require elevating the LOD to an equivalent level to the concentration of the identified peak.

Performance of Method:
Range of Method: 0.5ng/l – 20ng/l without dilution.

Determinand	LOD (ng/l)	Medium Tap Water				Standard			
		Low Spike		High Spike		Low Std		High Std	
		%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.	%RSD	%Rec.
NDMA	0.4266	5.16	101.25	7.86	110.01	11.78	98.42	4.65	101.77

Uncertainty of Measurement:

The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%.

Determinand	Uncertainty of Measurement (%)
NDMA	20.99

References:

Munch, J W and Bassett, M V (2004). Method 521: Determination of nitrosamines in drinking water by solid phase extraction and capillary column gas chromatography with large volume injection and chemical ionization tandem mass spectrometry (MS/MS). Version 1.0. EPA Document No. EPA/600/R-05/054. National Exposure Research Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268 (www.epa.gov/nerlcwww/m_521.pdf).

Cheng, R C, Andrews-Tate, C, Hwang, C J, Guo, Y, Grebel, J E and Suffet, I H (2005). Comparison of alternative nitrosamine analyses for water reuse samples. Water Reuse Association, California Section, 2005 Annual Conference, San Diego, CA.

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