METHOD STATEMENT

Determinand:
Mercury

Matrix:
Treated and untreated sewage, trade effluents, land and prepared Leachates, surface waters, ground waters and process waters

Untreated sewage is filtered prior to analysis to remove particulates.

Principle of Method:
A digested sample reacts with acidic tin (II) chloride to convert mercury (II) to mercury (0) vapour. The mercury vapour is removed from solution by a stream of argon and the mercury detected by atomic fluorescence.

Sampling and Sample Preparation:
All samples for mercury analysis are taken in dedicated 60ml glass mercury bottles containing 0.3ml of Potassium Bromate – Bromide solution and 0.3ml. of 36.5 - 38% Hydrochloric Acid solution. Samples are preserved on site at the point of sampling.

Samples that have been collected and preserved in mercury bottles are stable for up to 1 month from sampling (ISO 5667 – 3:2003).

Interferences:
Free bromine causes a negative interference by interfering with the transfer of mercury vapour. The effect is overcome by ensuring all free bromine vapour is reduced.

Performance of Method:
Range of Application: 0.010 – 0.500 µg/l Hg
Limit of Detection: 0.0071 µg/l Hg
Normal Reporting Level: 0.010 µg/l Hg

Determinand	Low Standard		High Standard	
	%RSD	%Bias	%RSD	%Bias
Mercury	6.51	1.41	4.87	2.27

Total Mercury

Determinand	%	Treated Sewage		Trade effluent (to sewer)		Untreated sewage		Trade effluent (to controlled waters)	
		20%	80%	20%	80%	20%	80%	20%	80%
Mercury	Rec.	98.72	98.25	97.93	97.56	90.46	95.17	92.58	92.1
	RSD	6.63	3.67	4.30	3.10	4.76	3.95	3.1	3.83

Determinand	%	Land Leachate	Prepared leachate	Ground water	Surface water	Process water
		80%	80%	80%	80%	80%
Mercury	Rec.	97.91	98.11	98.64	97.27	98.10
	RSD	3.15	1.84	2.49	3.71	2.55

Samples and spiked samples – Filtered Mercury

Determinand		Ground Water	Surface Water
		80%	80%
Mercury	%Rec	96.82	98.96
	%RSD	3.97	5.18

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 %
Mercury	14.82

References:

Thompson K C and Reynold R J. Atomic Absorption, Fluorescence and Flame Emission Spectroscopy – A Practical Approach. Charles Griffin 2nd Edition, London 1978. Pp 102-109

Thompson K C and Godden R G. Improvements in the AF Determination of Mercury by the Cold Vapour Technique. Analyst, 1975, 100, 544

Farey B J, Nelson L A and Rolfe M G. A Rapid Technique for the Breakdown of Organic Mercury Compounds in Natural Waters and Effluents. Analyst, 1978, 103, 656
Methods for the Examination of Waters and Associated Materials. Mercury in Waters, Effluents, Soils and Sediments, additional methods 1985, HMSO, ISBN 011-7519073

Nelson L A. Brominating Solution for the Preconcentration of Mercury from Natural Waters. Anal. Chem, 1975, 75, 592

Bothner M H and Robertson D E. Mercury Contamination of Seawater Samples Stored in Polyethylene Containers. Anal.Chem, 1975, 75, 592

P S Analytical Instrument Operating Manual: Merlin Plus System Manual. Part No M023M055. April 1992.