METHOD STATEMENT

Determinand:
Determination of Mercury

Matrix:
Sample Types: Raw, Potable and Surface waters.

Principle of Method:
This method uses the Perkin Elmer Elan DRC-e, Perkin Elmer Nexion 300 and ESI SC-0500-04 Autosampler.
The method describes a technique for the determination of mercury in solution. The basis of the method is the measurement of ions produced by an Inductively Coupled Plasma and detected using a mass spectrometer. Acidified samples preserved with gold solution are nebulised and the aerosol that is produced is transported to the plasma torch where excitation of the metal atoms occur. Excitation is due to the high temperatures (up to 6,000°C) produced by the radio frequency inductively coupled plasma. The metal ions thus produced pass through an interface region into the mass spectrometer. There the ions are separated by a quadropole and fall on to the mass detector. The intensities of the currents produced are processed and controlled by a computer system.
Internal standardisation is used to correct for transport and matrix effects.
A table of the isotopes measured and the internal standards used is given below.

Nexion ICP-MS				Elan ICP-MS			
Element	**Mass**	**Internal Standard used**	**Mass**	**Element**	**Mass**	**Internal Standard used**	**Mass**
Hg	200	Bi	209	Hg	200	Ir	193

Interferences:
Due to the large mass of the mercury isotopes, there are few interferences within potable water that could cause interferences. However, interference correction equations are available from the instrument's pre-loaded library.

Performance of the Method:
***Range of Application**:*

LOD – 1.25 µg/l Hg
The analytical range may be extended by sample dilution. The final concentration of acid / gold in the diluted solution should remain the same.
Perkin Elmer Elan
The reporting limit is < 0.02 µg/l Hg.
Perkin Elmer Nexion
The reporting limit is < 0.03 µg/l Hg.

Limit of Detection

Perkin Elmer Elan
Statistically obtained limit of detection of < 0.0176 µg/l Hg.
Perkin Elmer Nexion
Statistically obtained limit of detection < 0.0245 µg/l Hg.

Recoveries of Compounds and Uncertainty of measurement:

Perkin Elmer Elan

Sample type	Mean sample result (µg/l)	Mean sample spike result (µg/l)	Spike recovery (%)	Bias (%)	% uncertainty
Soft	0.008	0.970	98.12	-	± 8.89
Medium	0.008	0.933	94.32	-	± 11.13
Hard	0.012	0.938	94.45	-	± 12.95
Raw Surface	0.009	0.941	95.11	-	± 12.26
Borehole	0.006	0.956	96.86	-	± 9.48
Hard filtered	0.015	0.954	95.75	-	± 10.18
0.25 µg/l Std	0.259	-	-	3.55	± 10.74
1.00 µg/l Std	0.992	-	-	-0.80	± 5.56

Perkin Elmer Nexion

Sample type	Mean sample result (µg/l)	Mean sample spike result (µg/l)	Spike recovery (%)	Bias (%)	% uncertainty
Soft	0.005	1.008	100.35	-	± 4.89
Medium	0.006	1.014	100.91	-	± 5.19
Hard	0.008	1.034	102.69	-	± 5.27
Raw Surface	0.006	1.025	102.01	-	± 8.38
Borehole	0.006	1.034	102.86	-	60 ± 6.71
Hard filtered	0.011	1.020	101.03	-	± 7.66
0.25 µg/l Std	0.258	-	-	3.31	± 9.65
1.00 µg/l Std	1.023	-	-	2.34	± 7.09

References:

In house method based on SCA bluebook 163 Inductively Coupled Plasma Spectrometry 1996 and DWI Guidance note Sample Preservation and Preparation for Metals Analysis of Drinking Water.
Perkin Elmer Elan DRC-e series Hardware Guide manual.
Perkin Elmer Nexion 300 Series System Maintenenace Guide.
Perkin Elmer Elan DRC-e Series Software Manual.
Perkin elmer Nexion 300 Series Software Manual.