

METHOD STATEMENT

**Determinand:**

Determination of Mercury

Matrix:

Sample Types: Raw, Potable and Surface waters.

Principle of Method:

This method uses the Perkin Elmer Nexion 300 and ESI SC-0500-04 Autosampler and Agilent 7800 ICPMS and Agilent Technologies SPS4 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 quadrupole 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				Agilent ICP-MS			
Element	Mass	Internal Standard used	Mass	Element	Mass	Internal Standard used	Mass
Hg	200	Bi	209	Hg	200	Bi	209

Sampling and Sample Preparation:

Samples are normally collected in 125 ml polyethylene bottles

The 125 ml HDPE sampling bottles contain 1.25 ml of concentrated nitric acid and 0.6 ml of 1000 mg/l Gold Standard. The gold is present as a preservative. It forms an amalgam with mercury and will readily stop the loss of mercury

If analysis cannot be immediately undertaken, samples can be stored at room temperature until the day of analysis. Samples should be analysed within 30 days of the sampling date.

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 Method:**Range of Application:**

LOQ - 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 Nexion

The reporting limit is < 0.06 µg/l Hg.

Agilent

The reporting limit is < 0.06 µg/l Hg.

METHOD STATEMENT



Limit of Quantification:

Perkin Elmer Nexion

Statistically obtained limit of quantification < 0.0509 µg/l Hg.

Agilent

Statistically obtained limit of quantification < 0.0346 µg/l Hg.

Recoveries of Compounds, Bias and Uncertainty of measurement:

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	-	± 5.19
Medium	0.006	1.014	100.91	-	± 5.27
Hard	0.008	1.034	102.69	-	± 8.38
Raw Surface	0.006	1.025	102.01	-	± 6.71
Borehole	0.006	1.034	102.86	-	± 7.66
Hard filtered	0.011	1.020	101.03	-	± 5.48
0.25 µg/l Std	0.258	-	-	3.31	± 9.65
1.00 µg/l Std	1.023	-	-	2.34	± 7.09

Agilent

Sample type	Mean sample result (µg/l)	Mean sample spike result (µg/l)	Spike recovery (%)	Bias (%)	% uncertainty
Soft	0.007	0.986	98.07	-	± 3.96
Medium	0.009	0.996	98.84	-	± 3.16
Hard	0.009	1.009	100.05	-	± 2.70
Raw Surface	0.010	1.008	99.92	-	± 2.71
Borehole	0.010	1.018	100.97	-	± 3.07
Hard filtered	0.011	1.005	99.57	-	± 3.15
0.25 µg/l Std	0.258	-	-	3.04	± 10.71
1.00 µg/l Std	1.010	-	-	1.00	± 4.55

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 Nexion 300 Series System Maintenance Guide.

Perkin Elmer Nexion 300 Series Software Manual.