

# METHOD STATEMENT



## Determinand:

Dissolved and Total Mercury

## Matrix:

Sample Type: Final effluent, Trade Discharge and Crude Sewages.

## Principle of Method:

The basis of the method is the measurement of ions produced by an inductively coupled plasma (ICP) and detected using a mass spectrometer.

The Agilent 7900 uses helium gas in collision mode to reduce any potential interferences.

Acidified samples 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 K) 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 dual mode detector then detects these ions and the resulting electrical signals. These are processed into digital information that is used to indicate ion intensity and subsequently elemental concentration.

Analysis is carried out using the Agilent 7900 ICP-MS.

## Sampling and Sample Preparation:

Samples should be taken in 125ml azlons containing a preservative made of HNO<sub>3</sub> and Au (STL 241). Stability period is 55 days from sampling (Wakefield in-house data 2025), which is inclusive of the digested portion. Anything outside of this timeframe will require stability comments. If samples are taken in 1 L PET bottles, the results are considered compromised, and a sample comment added accordingly.

Samples requiring filtered mercury analysis should be filtered prior to sampling into STL241's. Samples not filtered prior to preservative or not sampled into STL 241's have compromised stability; an appropriate sample comment should be added.

If sample preparation cannot be immediately undertaken, samples should be refrigerated at 3 ± 2°C. Once digested, samples are stored at room temperature.

## Interferences

Due to the large mass of the mercury isotopes, there are few interferences within wastewater that could cause interferences.

Interferences are removed through the use of helium gas using a collision cell and the use of mass 201 which is not prone to tungsten interference.

## Performance of Method:

### Range of Application:

LOD – 2.5 µg/l

Normal reporting limit                      0.05µg/l

### Limit of Detection:

Agilent 7900: 0.0240 µg/l

### Recoveries of Compounds:

	Standards		Final Effluent		Final Effluent Filtered		Trade Discharge		Crude Sewage	
	Low Std	High Std	Low Spike	High Spike	Low Spike	High Spike	Low Spike	High Spike	Low Spike	High Spike
Agilent 7900										
<b>%Recovery</b>	97.46	97.34	98.53	100.14	99.38	99.92	99.41	100.71	98.84	99.93
<b>%RSD</b>	1.36	1.05	1.90	2.10	2.13	1.83	2.25	1.52	5.66	4.62

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## References:

SCA bluebook 163 Inductively Coupled Plasma Spectrometry 1996  
DWI Guidance note Sample Preservation and Preparation for Metals Analysis of Drinking Water.  
Agilent ICP-MS: ISIS 3 (Integrated Sample Introduction System).  
Agilent ICP-MS: MassHunter Workstation User Guide.  
Agilent ICP-MS: Familiarization Guide  
Agilent 7800/7900 ICPMS: Hardware Maintenance Manual  
Agilent SPS 4 Autosampler: User's Guide  
ISO 5667 – 3:2024 – Water quality