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 ELAN uses a dynamic reaction cell where oxygen is used as a reaction gas to reduce interference by interrupting the sequence of reactions that would otherwise create interference. 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 Perkin Elmer ELAN DRC-e and Agilent 7900 ICP-MS.

Sampling and Sample Preparation:
Samples should be taken in 125.ml azlons containing HNO3 and Au (STL 241), these are stable for 6 months (ISO 5667-3:2018), if samples are taken in 1L 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.
Digested samples are stable for 6 months (ISO 5667 – 3:2018).

Interferences
Due to the large mass of the mercury isotopes, there are few interferences within wastewater that could cause interferences.
ELAN: The oxygen DRC gas is used to remove potential tungsten oxide interferences; however these are not likely to be present in many waste water samples.
Agilent: Interferences are removed through the use of helium gas using a collision cell.

Performance of Method:

   Range of Application:
      LOD – 2.5 µg/l
      Normal reporting limit   0.05µg/l

   Limit of Detection:
      ELAN DRC-e: 0.0384 µg/l
      Agilent 7900: 0.0240 µg/l

METHOD STATEMENT

Recoveries of Compounds:

	Standards		Final Effluent		Final Effluent Filtered		Trade Discharge		Crude Sewage	
ELAN DRC-e 0114	Low Std	High Std	Low Spike	High Spike	Low Spike	High Spike	Low Spike	High Spike	Low Spike	High Spike
%Recovery	102.07	100.52	100.11	99.08	99.51	99.70	99.16	97.30	99.06	98.45
%RSD	2.22	1.32	3.47	1.62	3.51	2.08	3.13	2.15	4.85	2.32

Original validation data used for Comparison Data with 0082

	Standards		Final Effluent		Final Effluent Filtered	
ELAN DRC-e 0082	Low Std	High Std	Low Spike	High Spike	Low Spike	High Spike
%Recovery	97.61	98.77	105.46	102.91	104.62	102.19
%RSD	2.22	2.11	2.82	2.08	3.53	2.84

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

References:
SCA bluebook 163 Inductively Coupled Plasma Spectrometry 1996
DWI Guidance note Sample Preservation and Preparation for Metals Analysis of Drinking Water.
Perkin Elmer Elan DRC-e series Hardware Guide manual.
Perkin Elmer "Preparing your laboratory for the ELAN DRC II ICP-Mass Spectrometer" Guide
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