METHOD STATEMENT

Determinand:
Determination of Dissolved and Total Metals: The elements covered are aluminium, boron, calcium, cadmium, cobalt chromium, copper, iron, potassium, magnesium, manganese, sodium, nickel, phosphorus, lead, thallium, vanadium, zinc and sulphate.

Matrix:
Sample Type: Final effluents, trade discharges, crude sewage and other similar waste water samples

Principle of Method:
The method describes a technique for the simultaneous multi – element determination of metals in solution. Samples are acidified and digested at elevated temperatures to ensure any particulate matter is brought into solution. The acidified samples are then analysed by the ICP-OES (inductively coupled plasma optical emission spectroscopy) instrument to determine the concentration of metals present. Excitation of the sample within the 6,000°C plasma causes ionisation of atoms, which in turn causes the emission of electromagnetic radiation at specific wavelengths for each element. The intensity of the emission is measured and quantified by comparison against standards with known concentrations of elements.

Sampling and Sample Preparation:
Samples are normally taken in 1L PET bottles. Samples are stored at 3 ± 2°C until ready for preparation.
Samples should be prepared within 7 days of sampling (Coventry in-house data).
Prepared samples are stable for one month. B, Cd, Cr, Cu, Pb, Ni & Zn to 6 months (ISO 5667 – 3:2018). Prepared samples are stored at room temperature.

Interferences
Careful choice of plasma conditions, analytical wavelengths and concentration ranges are all designed to minimise interference.

Performance of Method:

Range of Application:

Element	Units	Range
Al	mg/l	LOD – 50
Ca	mg/l	LOD – 250
Fe	mg/l	LOD – 50
K	mg/l	LOD – 100
Mg	mg/l	LOD – 150
Mn	mg/l	LOD – 50
Na	mg/l	LOD – 200
Cd	mg/l	LOD – 1
Co	mg/l	LOD – 1
Cr	mg/l	LOD – 10
Cu	mg/l	LOD – 10
Ni	mg/l	LOD – 10
Pb	mg/l	LOD – 10
V	mg/l	LOD – 1
Zn	mg/l	LOD – 10
P	mg/l	LOD – 10
B	mg/l	LOD – 10

Element	Units	Range
Tl	mg/l	LOD – 5
SO4	Mg/l	LOD - 1000

All analytical ranges may be extended by sample dilution.

Limit of Detection, Recoveries of Compounds and Uncertainty of measurement:

	Units	LOD	Reporting Limit	Max. Allowable Blank	Final Effluent Spike Recovery %	Spike RSD %	Uncertainty %
Aluminium	mg/l	0.0809	0.09	0.09	99.08	3.30	7.52
Boron	mg/l	0.0408	0.041	0.041	97.82	3.12	8.42
Calcium	mg/l	2.7213	2.8	2.8	95.61	3.69	11.78
Cadmium	mg/l	0.0048	0.005	0.005	99.33	2.94	6.56
Cobalt	mg/l	0.0046	0.005	0.005	101.08	3.19	7.45
Chromium	mg/l	0.0223	0.023	0.023	98.77	3.47	8.18
Copper	mg/l	0.0256	0.026	0.026	98.14	3.41	8.68
Iron	mg/l	0.5626	0.6	0.6	98.14	3.67	9.19
Potassium	ug/l	907.7	1000	1000	95.78	3.68	11.58
Magnesium	mg/l	0.5918	0.6	0.6	97.18	3.32	9.46
Manganese	mg/l	0.0225	0.091	0.091	97.28	3.44	9.60
Sodium	ug/l	1791.5	1800	1800	94.45	3.90	13.36
Nickel	mg/l	0.0086	0.061	0.061	97.84	3.86	9.89
Phosphorus	mg/l	0.4729	0.48	0.48	89.65	3.24	16.83
Lead	mg/l	0.2838	0.29	0.29	98.38	3.64	8.90
Thallium	µg/l	98.6	100	100	97.57	4.03	10.50
Vanadium	mg/l	0.0134	0.014	0.014	101.69	3.09	7.88
Zinc	mg/l	0.0698	0.07	0.07	97.19	3.31	9.43
Sulphate	mg/l	17.22	18	18	98.83	3.11	12.28

For Urban Waste Water Regulations, an assessment of the method performance with different forms of phosphorus needs to be made. This is summarised below.

Potassium dihydrogen orthophosphate: Recovery 97.56%
Sodium pyrophosphate: Recovery 97.38%
Sodium hexametaphosphate: Recovery 102.43%
B- Glycerophosphate: Recovery 99.27%

References:

In house method based on SCA bluebooks, and Perkin Elmer Optima 7100, 7200 and 7300 series Hardware Guide manual.