

METHOD STATEMENT

Determinand:

Determination of Aluminium, Arsenic, Boron, Cadmium, Chromium, Cobalt, Copper, Iron, Manganese, Molybdenum, Nickel, Phosphorous, Lead, Selenium, Thallium, Uranium, Vanadium and Zinc

Matrix:

Sample Type: final effluents, trade discharge, crude sewage and other similar waste water samples. Filtered samples may also be analysed using this method.

NOTE: This method is not suitable for the analysis of selenium or phosphorous in crude sewages.

Principle of Method:

The method describes a technique for multi – element determination of trace metals 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 are nebulised and the aerosol that is produced is transported to the plasma torch where excitation of the metal atoms occurs. Excitation is due to the high temperatures (up to 6,000K) 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 where only ions having a specific mass to charge ratio are passed through at any moment in time. The dual mode detector then detects these ions and the resulting electrical signals are processed into digital information that is used to indicate ion intensity and subsequently elemental concentration.

Interferences:

Careful choice of plasma conditions, interference equations and isotopes are all designed to minimise interference.

Performance of Method:

Range of Application:

Element	µg/L
B	LOD - 1000
Al	LOD - 1000
P	LOD - 1000
V	LOD - 100
Cr	LOD - 500
Fe	LOD - 1000
Mn	LOD - 1000
Co	LOD - 100
Ni	LOD - 500
Cu	LOD - 500
Zn	LOD - 500
As	LOD - 100
Se	LOD - 100
Mo	LOD - 100
Cd	LOD - 100
Tl	LOD - 100
Pb	LOD - 500
U	LOD - 50

This range may be extended by sample dilution.



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

Element	LOD µg/l	MRV µg/l		Final Effluent		Trade Discharge		Untreated Raw	
				Low Spike	High Spike	Low Spike	High Spike	Low Spike	High Spike
B	18.8093	19	%recovery	97.82	96.32	99.90	98.62	102.21	100.64
			%rsd	4.99	4.82	4.37	3.06	3.58	4.07
Al	24.8704	25	%recovery	95.89	97.95	97.91	99.03	99.34	102.53
			%rsd	4.83	4.90	4.51	3.20	3.81	3.02
P	72.0790	80	%recovery	101.64	104.43	99.43	103.48	-	-
			%rsd	2.75	3.60	2.15	3.53	-	-
V	1.1332	1.2	%recovery	101.49	102.29	102.44	103.29	102.26	101.36
			%rsd	2.07	2.10	1.67	1.90	2.46	2.34
Cr	0.8058	0.81	%recovery	95.93	95.27	96.52	95.41	97.52	96.17
			%rsd	2.89	3.47	3.60	3.09	2.99	3.25
Mn	0.3047	0.31	%recovery	100.02	98.09	98.35	98.52	102.55	97.91
			%rsd	3.44	3.65	2.76	5.77	4.33	2.06
Fe	27.3463	31	%recovery	100.71	95.99	95.21	93.39	92.05	94.83
			%rsd	3.18	3.69	4.88	2.57	4.86	3.75
Co	0.1752	0.18	%recovery	99.44	100.21	100.55	99.60	102.46	100.46
			%rsd	2.46	2.85	2.79	2.55	2.34	1.92
Ni	2.7421	2.8	%recovery	96.00	95.48	97.34	96.71	100.23	97.91
			%rsd	2.64	3.05	2.68	2.41	2.66	2.36
Cu	2.4795	2.5	%recovery	99.97	97.58	98.34	94.51	102.42	98.15
			%rsd	3.23	3.52	4.38	5.16	4.93	2.87
Zn	6.1950	7	%recovery	104.96	97.64	98.18	93.09	96.67	93.04
			%rsd	4.71	5.00	4.27	4.97	3.09	3.39
As	0.7137	0.8	%recovery	103.87	102.77	104.63	103.75	102.13	100.40
			%rsd	3.05	2.34	3.27	3.60	3.11	3.28
Se	0.7991	0.8	%recovery	96.05	95.62	93.77	95.98	-	-
			%rsd	2.58	3.50	3.27	4.75	-	-
Mo	1.8571	1.9	%recovery	107.45	104.93	109.08	107.15	101.15	103.88
			%rsd	2.83	4.58	2.83	4.58	5.69	3.07
Cd	0.1152	0.12	%recovery	96.65	97.77	100.44	99.90	102.61	101.06
			%rsd	1.90	2.25	2.32	2.33	1.88	1.97
Tl	0.0277	0.03	%recovery	100.92	102.45	102.70	100.81	101.86	100.63
			%rsd	2.30	1.82	2.32	2.46	2.08	2.31
Pb	0.8074	0.81	%recovery	103.68	103.55	104.22	101.43	103.94	101.40
			%rsd	3.30	1.88	3.42	2.27	3.21	2.08
U	0.2847	0.29	%recovery	105.04	106.68	109.56	111.04	108.61	111.56
			%rsd	4.17	4.65	4.39	4.67	4.16	4.37

Note: Additional testing of filtered matrices showed no statistically significant change in performance. 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	105.25%
Sodium pyrophosphate:	Recovery	102.46%
Sodium hexametaphosphate:	Recovery	106.48%
B- Glycerophosphate:	Recovery	102.04%

References:

In house method based on SCA bluebooks

Perkin Elmer ELAN DRC-e Hardware guide manual.

ISO 17294-2:2003 Water quality – Application of inductively coupled plasma mass spectrometry (ICP-MS) – Part 2: Determination of 62 elements.

