METHOD STATEMENT

Determinand:
Metals: -
(Aluminium, Cadmium, Chromium, Copper, Iron, Lead, Nickel and Zinc).

Matrix:
Effluents and Surface Waters

Principle of Method:
Metals are determined by ICP-MS after heated dissolution in the presence of nitric acid. The digestion pre-treatment ensures that any suspended or colloidal forms are converted to soluble forms.
For Filtered metals, the samples are filtered on site.

Sampling and Sample Preparation:
All samples are acidified upon receipt by the laboratory.
Samples undergo a heated dissolution in the presence of nitric acid. The prepared samples are stored until required for analysis.

Interferences:
The interferences for a number of elements are well documented and understood. Within the limitations of the method, these interferences are adequately compensated for by careful choice of elemental isotopes, interference equations and the use of reaction or collision gas technology.

Performance of Method:

Total Metals

Determinand	Range of Application (ug/l)	LOD (ug/l)	Routine Reporting Limit (ug/l)	Low Standard		High Standard	
				%RSD	%Bias	% RSD	% Bias
Aluminium	25 to 5000	2.7228	25	2.82	-7.13	1.45	-3.04
Cadmium	0.02 to 2	0.0038	0.02	4.35	-2.77	1.26	-2.04
Chromium	0.5 to 50	0.0974	0.5	4.03	-0.57	3.04	-2.32
Copper	0.3 to 30	0.2259	0.3	4.72	-2.60	2.03	-2.57
Iron	25 to 5000	2.3373	25	4.09	-0.63	1.21	-2.21
Lead	0.2 to 20	0.0412	0.1	4.14	0.17	1.49	2.20
Nickel	0.5 to 50	0.4576	0.5	3.73	-1.77	1.68	-3.17
Zinc	0.8 to 50	0.3571	0.5	5.07	-0.24	5.03	-2.87

Determinand	Final Effluent				Surface Water			
	20% & CLOI		80%		20% & CLOI		80%	
	% RSD	% Bias	% RSD	% Bias	% RSD	% Bias	% RSD	% Bias
Aluminium	2.77	-8.99	1.69	-4.46	N/A	N/A	1.65	-5.71
Cadmium	1.38	-5.45	1.13	-3.99	1.89	-3.30	1.07	-4.03
Chromium	2.33	-6.89	2.78	-6.09	N/A	N/A	2.93	-6.18
Copper	4.02	-6.85	3.59	-7.79	N/A	N/A	4.09	-5.30
Iron	0.95	-7.00	1.02	-6.38	1.44	-8.47	1.40	-7.38
Lead	1.47	-1.17	1.60	-0.16	1.26	-4.03	2.02	-1.45
Nickel	1.66	-9.37	1.26	-9.74	3.42	-6.81	2.06	-9.78
Zinc	3.99	-9.56	2.87	-8.45	N/A	N/A	3.70	-9.11

Filtered Metals

Determinand	Range of Application (ug/l)	LOD (ug/l)	Routine Reporting Limit (ug/l)	Low Standard		High Standard	
				%RSD	%Bias	% RSD	% Bias
Aluminium	25 to 5000	3.9031	25	2.03	-6.86	1.25	-2.40
Cadmium	0.02 to 2	0.0073	0.02	1.92	-0.77	1.34	-1.85
Chromium	0.5 to 50	0.2328	0.5	2.97	-3.62	3.25	-2.62
Copper	0.3 to 30	0.1533	0.3	4.25	-4.04	3.12	-2.62
Iron	25 to 5000	19.0760	25	1.41	-0.48	1.48	-1.86
Lead	0.2 to 20	0.0807	0.1	1.43	-0.56	1.83	2.23
Nickel	0.5 to 50	0.3029	0.5	1.63	-1.67	1.63	-2.60
Zinc	0.8 to 50	0.4001	0.5	3.42	5.73	2.97	-1.43

Determinand	Final Effluent				Surface Water			
	20% & CLOI		80%		20% & CLOI		80%	
	% RSD	% Bias	% RSD	% Bias	% RSD	% Bias	% RSD	% Bias
Aluminium	1.40	-8.76	1.45	-3.38	N/A	N/A	1.42	-4.58
Cadmium	1.51	-4.98	1.03	-3.94	1.41	-3.53	1.45	-4.37
Chromium	2.98	-6.60	3.94	-5.9	N/A	N/A	4.12	-6.26
Copper	3.64	-2.98	2.51	-7.51	N/A	N/A	2.56	-6.79
Iron	1.01	-5.72	1.17	-5.70	1.02	-6.77	1.19	-6.42
Lead	1.38	-0.79	1.75	-0.22	1.15	-4.31	1.48	-1.87
Nickel	1.88	-8.12	1.24	-8.91	3.59	-5.67	1.23	-9.57
Zinc	3.61	4.92	3.51	-0.31	N/A	N/A	3.0	-8.40

Uncertainty of Measurement:

The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2, which gives a level of confidence of approximately 95%.

Determinand	Uncertainty of Measurement %	
	Total Metals	Filtered Metals
Aluminium	14.39	12.62
Cadmium	9.37	9.25
Chromium	15.15	3.84
Copper	15.56	14.49
Iron	14.42	12.23
Lead	6.88	6.54
Nickel	18.33	16.74
Zinc	20.70	13.64

References:

Handbook of Inductively Coupled Plasma Mass Spectrometry. K.E Jarvis, A.L. Gray, R.S.Houk. ISBN 0-216-912-1

Principles of Instrumental Analysis 6th Edition. Holler, Sloog, Crouch. ISBN 0-495-12570-9