

METHOD STATEMENT



Determinand:

Metals: -

Aluminium, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Nickel, Phosphorus, Potassium, Silver, Sodium, Strontium, Thallium, Tin, Titanium, Total Sulphur, Vanadium, Zinc, Zirconium.

Matrix:

Waste waters, surface waters, groundwaters, untreated sewages, final effluents and leachates, recreational waters and process waters.

Principle of Method:

Metals are determined by ICP-AES after dissolution in the presence of nitric acid. The pre-treatment ensures that any metals in suspended or colloidal forms are converted to soluble forms. An internal standard is used to compensate for interferences, such as those from matrices containing high levels of dissolved solids.

Sampling and Sample Preparation:

Samples undergo a hot acid digest in nitric acid in accordance with Method WAS-011 - Sample Preparation for Metals Analysis.

The prepared samples are stored in 50ml preparation tubes prior to analysis.

Samples are stable for times stated below, (In-House Data) from sampling.

Al	14 Days (In-House Data)
Ag	7 Days (In-House Data)
B	7 Days (In-House Data)
Ba	9 Days (In-House Data)
Be	17 Days (In-House Data)
Ca	17 Days (In-House Data)
Cd	17 Days (In-House Data)
Co	17 Days (In-House Data)
Cr	17 Days (In-House Data)
Cu	14 Days (In-House Data)
Fe	17 Days (In-House Data)
K	17 Days (In-House Data)
Li	7 Days (In-House Data)
Mg	10 Days (In-House Data)
Mn	17 Days (In-House Data)
Mo	15 Days (In-House Data)
Na	7 Days (In-House Data)
Ni	17 Days (In-House Data)
P	17 Days (In-House Data)
Pb	16 Days (In-House Data)
S	28 Days (ISO 5667:3)
Sn	7 Days (In-House Data)
Sr	17 Days (In-House Data)
Ti	14 Days (In-House Data)
Tl	17 Days (In-House Data)
V	17 Days (In-House Data)
Zn	17 Days (In-House Data)

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Interferences:

Spectral Interference may occur from the presence of other elements. The spectral lines have been chosen so that overlap is minimal. Elements within standards have been chosen to minimise chemical interference. Internal standards are used to compensate for interference from plasma anomalies caused by high dissolved solids content.

Performance of Method:

Determinand		MCERTS Accreditation	Range of Application (mg/l)	LOD (mg/l)	Normal Reporting Level	
					(mg/l)	(µg/l)
Ag	Silver		0.0007 - 0.5	0.0007	0.0007	0.7
Al	Aluminium	✓	0.1 - 50	0.0317	0.1	100
B	Boron		0.23- 25.0	0.2266	0.23	230
Ba	Barium		0.007- 10.0	0.0061	0.007	7.0
Be	Beryllium		0.0021 - 1.0	0.0021	0.0021	2.1
Ca	Calcium		0.38 - 2000	0.3734	0.38	380
Cd	Cadmium	✓	0.0006 - 1.0	0.0006	0.0006	0.6
Co	Cobalt	✓	0.002 - 1.0	0.0013	0.002	2
Cr	Chromium	✓	0.002 - 10	0.0013	0.002	2
Cu	Copper	✓	0.009 - 10	0.0083	0.009	9
Fe	Iron	✓	0.23 - 2000	0.2263	0.23	230
K	Potassium		0.18 - 2000	0.179	0.18	180
Li	Lithium		0.002 - 5	0.0011	0.002	2
Mg	Magnesium		0.6 - 2000	0.5502	0.6	600
Mn	Manganese	✓	0.007 - 50	0.0061	0.007	7
Mo	Molybdenum		0.003 - 1.0	0.0029	0.003	3
Na	Sodium		0.30- 2000	0.3041	0.3	300
Ni	Nickel	✓	0.003 - 10	0.0029	0.003	3
P	Phosphorus	✓	0.12- 50	0.1135	0.12	120
Pb	Lead	✓	0.006 - 10	0.0052	0.006	6
S	Total Sulphur		0.2 - 20	0.1615	0.2	200
Sn	Tin		0.007 - 1.0	0.0062	0.007	7
Sr	Strontium		0.002 - 2.0	0.0018	0.002	2
Ti	Titanium		0.002 - 1.0	0.002	0.002	2
Tl	Thallium		0.012 - 1.0	0.0119	0.012	12
V	Vanadium		0.004 - 1.0	0.0034	0.004	4
Zn	Zinc	✓	0.018 - 10	0.0179	0.018	18

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Determinand	Low Standard		High Standard	
	% RSD	% Rec.	% RSD	% Rec.
Ag	8.98	97.87	5.98	99.12
Al	1.18	94.77	1.13	101.21
B	0.87	97.40	0.98	96.72
Ba	3.16	94.10	1.23	106.07
Be	2.13	108.67	1.32	100.16
Ca	1.26	101.25	1.05	98.20
Cd	0.57	108.92	1.00	101.09
Co	0.87	105.37	0.89	98.14
Cr	1.14	98.29	1.13	102.32
Cu	0.92	99.28	1.36	103.60
Fe	1.36	100.15	1.32	97.16
K	1.45	103.50	0.61	95.32
Li	1.02	93.12	0.96	99.44
Mg	1.26	101.50	1.75	102.44
Mn	0.80	98.34	1.05	101.39
Mo	0.75	104.30	0.71	98.34
Na	1.02	96.92	1.18	99.10
Ni	0.86	95.40	1.19	93.67
P	4.37	91.62	4.26	92.36
Pb	0.86	97.87	0.98	94.47
S	1.01	96.92	1.16	99.10
Sn	1.24	96.14	2.20	102.78
Sr	0.79	89.32	1.09	97.69
Ti	0.73	108.95	1.09	98.12
Tl	1.92	98.70	1.64	96.59
V	0.55	108.41	0.76	99.51
Zn	2.96	92.35	5.93	94.21

Determinand		Final Effluent		Trade Discharge		Landfill Leachate	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Ag	% Recovery	105.20	98.25	118.86	98.88	-	98.3
	% RSD	18.07	5.87	22.60	6.64	-	7.55
Al	% Recovery	92.41	96.64	93.31	95.93	-	98.5
	% RSD	1.41	1.22	1.51	1.63	-	0.91
B	% Recovery	97.34	101.53	99.56	97.21	101.0	103.3
	% RSD	2.21	1.23	1.11	3.21	1.17	3.46
Ba	% Recovery	94.83	97.43	94.65	99.07	99.2	95.7
	% RSD	4.08	2.80	3.39	3.17	3.59	2.26
Be	% Recovery	107.88	101.81	105.22	101.57	-	101.5
	% RSD	1.28	0.85	1.43	0.95	-	1.15
Ca	% Recovery	88.52	95.62	90.12	95.65	98.9	103.0
	% RSD	6.21	1.15	1.31	1.37	1.05	2.18
Cd	% Recovery	104.00	103.06	103.33	104.26	-	105.2
	% RSD	0.89	0.69	0.66	0.72	-	0.61

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Determinand		Final Effluent		Trade Discharge		Landfill Leachate	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Co	% Recovery	100.64	98.15	102.36	100.44	-	98.2
	% RSD	1.28	0.92	1.48	0.96	-	1.32
Cr	% Recovery	98.29	100.23	97.86	100.25	-	100.5
	% RSD	2.02	0.94	2.10	1.09	-	1.50
Cu	% Recovery	98.82	102.13	98.55	101.37	-	103.6
	% RSD	0.73	1.01	1.00	1.18	-	0.89
Fe	% Recovery	100.22	98.97	101.04	98.83	98.7	97.6
	% RSD	1.77	1.37	1.54	1.55	1.47	1.57
K	% Recovery	109.40	100.47	102.09	105.00	96.0	98.3
	% RSD	1.90	0.95	1.54	1.09	3.64	1.00
Li	% Recovery	-	100.5	-	98.9	-	96.7
	% RSD	-	1.61	-	1.84	-	2.35
Mg	% Recovery	97.92	103.59	98.48	101.02	100.8	99.4
	% RSD	1.39	1.40	1.41	1.35	1.32	2.43
Mn	% Recovery	100.20	98.81	100.08	98.94	-	98.1
	% RSD	1.07	0.74	1.52	0.73	-	0.92
Mo	% Recovery	100.31	99.95	101.50	98.82	-	97.5
	% RSD	1.68	0.88	1.15	0.65	-	0.51
Na	% Recovery	110.71	89.89	99.87	90.57	95.7	92.2
	% RSD	2.12	1.64	2.15	1.08	0.91	1.87
Ni	% Recovery	96.53	94.00	96.83	93.93	-	93.3
	% RSD	1.44	0.87	1.35	1.00	-	1.43
P	% Recovery	93.75	97.04	104.00	96.90	97.54	98.51
	% RSD	3.94	2.96	3.90	2.61	1.21	0.93
Pb	% Recovery	99.70	96.34	98.88	95.90	-	100.8
	% RSD	1.37	0.82	1.09	0.90	-	1.12
S	% Recovery	-	94.4	-	91.2	-	92.3
	% RSD	-	9.54	-	8.92	-	1.50
Sn	% Recovery	99.22	101.83	91.17	103.44	-	104.1
	% RSD	2.60	3.08	2.81	3.59	-	3.07
Sr	% Recovery	102.53	106.77	102.77	106.68	-	104.2
	% RSD	1.91	1.18	1.91	1.98	-	0.85
Ti	% Recovery	103.41	97.29	103.09	96.74	-	97.4
	% RSD	1.03	1.04	0.91	0.92	-	0.70
Tl	% Recovery	97.27	95.75	96.85	95.53	-	95.3
	% RSD	2.22	1.50	2.13	1.35	-	1.68
V	% Recovery	107.95	101.95	107.00	101.65	-	103.3
	% RSD	0.55	0.69	0.45	0.85	-	0.47
Zn	% Recovery	94.78	90.83	94.93	90.43	-	91.2
	% RSD	3.20	2.34	3.06	2.08	-	2.32

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Determinand		Untreated		Groundwater	
		Low Spk	High Spk	Low Spk	High Spk
Ag	% Recovery	89.67	90.25	-	101.5
	% RSD	6.26	2.36	-	6.82
Al	% Recovery	96.66	95.20	-	98.6
	% RSD	3.24	0.76	-	3.1
B	% Recovery	95.13	108.62	-	99.5
	% RSD	2.48	1.77	-	0.85
Ba	% Recovery	91.03	100.39	-	101.0
	% RSD	4.21	0.29	-	2.44
Be	% Recovery	102.37	96.88	-	104.3
	% RSD	0.47	0.38	-	1.02
Ca	% Recovery	103.14	98.37	100.3	105.7
	% RSD	0.71	0.46	0.81	1.98
Cd	% Recovery	98.63	100.64	-	104.1
	% RSD	0.61	0.37	-	1.06
Co	% Recovery	97.29	95.54	-	100.9
	% RSD	1.19	0.35	-	0.59
Cr	% Recovery	95.89	95.86	-	103.9
	% RSD	0.29	0.23	-	1.30
Cu	% Recovery	99.56	101.54	-	104.0
	% RSD	0.85	0.77	-	1.29
Fe	% Recovery	101.00	100.45	100.9	101.7
	% RSD	0.59	0.39	1.09	1.59
K	% Recovery	111.48	104.69	106.5	91.3-
	% RSD	2.79	1.11	1.17	2.32
Li	% Recovery	-	107.8	-	98.6
	% RSD	-	0.98	-	2.28
Mg	% Recovery	102.86	103.72	104.5	103.9
	% RSD	0.83	0.26	1.36	1.86
Mn	% Recovery	97.91	96.49	-	102.3
	% RSD	0.73	0.48	-	0.96
Mo	% Recovery	98.58	103.79	-	100.1
	% RSD	0.62	0.57	-	0.89
Na	% Recovery	107.51	106.17	97.4	97.4
	% RSD	0.23	0.17	0.80	1.88
Ni	% Recovery	88.96	97.75	-	97.0
	% RSD	3.09	1.70	-	0.89
P	% Recovery	90.61	103.33	93.3	98.4
	% RSD	1.98	1.91	4.39	2.16
Pb	% Recovery	95.52	91.11	-	98.3
	% RSD	1.23	0.35	-	0.72
S	% Recovery	-	100.3	-	84.9
	% RSD	-	1.76	-	10.27
Sn	% Recovery	109.73	104.06	-	104.5
	% RSD	1.75	0.77	-	3.86

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Determinand		Untreated		Groundwater	
		Low Spk	High Spk	Low Spk	High Spk
Sr	% Recovery	102.84	100.65	100.7	108.7
	% RSD	3.68	3.85	1.30	1.03
Ti	% Recovery	99.35	97.95	-	99.8
	% RSD	0.35	0.20	-	0.94
Tl	% Recovery	92.36	95.95	-	95.8
	% RSD	1.61	0.37	-	1.82
V	% Recovery	99.28	104.53	-	102.2
	% RSD	1.15	0.94	-	0.80
Zn	% Recovery	95.19	90.15	-	93.3
	% RSD	0.92	1.09	-	2.52

Determinand		Surface water	Recreational	Clean Process	Dirty Process
		High Spike	High Spike	High Spike	High Spike
Ag	%Recovery	96.16	101.01	99.91%	92.74%
	% RSD	4.66	3.77	4.24%	6.24%
Al	%Recovery	99.69	99.57	100.85%	-
	% RSD	4.88	4.93	5.21%	-
B	%Recovery	102.15	94.86	93.19%	92.96%
	% RSD	1.66	2.02	2.44%	1.93%
Ba	%Recovery	93.24	93.80	93.90%	92.78%
	% RSD	1.69	2.15	2.08%	1.80%
Be	%Recovery	96.60	96.23	95.53%	97.07%
	% RSD	1.65	2.05	1.84%	2.19%
Ca L/L	%Recovery	91.25	93.41	92.62%	92.71%
	% RSD	2.98	1.81	1.88%	1.57%
Ca H/L	%Recovery	100.48	99.95	100.11%	101.31%
	% RSD	3.16	2.56	3.04%	2.52%
Cd	%Recovery	95.98	96.17	95.99%	95.87%
	% RSD	1.75	2.34	2.00%	2.02%
Co	%Recovery	95.07	95.05	94.99%	96.49%
	% RSD	2.02	2.44	2.26%	2.18%
Cr	%Recovery	95.22	95.86	95.47%	97.02%
	% RSD	1.68	2.24	2.01%	2.09%
Cu	%Recovery	96.69	95.83	96.96%	97.53%
	% RSD	1.36	1.82	1.85%	2.35%
Fe L/L	%Recovery	94.78	95.50	94.59%	93.30%
	% RSD	1.93	1.87	1.90%	1.74%
Fe H/L	%Recovery	98.48	98.19	98.75%	99.55%
	% RSD	2.89	2.01	2.50%	1.85%
K L/L	%Recovery	93.64	93.89	93.51%	91.56%
	% RSD	1.76	1.84	2.17%	1.81%
K H/L	%Recovery	95.89	95.51	95.59%	96.81%
	% RSD	1.89	2.28	2.33%	2.04%
Li	%Recovery	100.46	99.34	99.08%	97.42%
	% RSD	1.79	2.18	2.03%	1.75%

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Determinand		Surface water	Recreational	Clean Process	Dirty Process
		High Spike	High Spike	High Spike	High Spike
Mg L/L	%Recovery	93.29	94.31	92.26%	92.18%
	% RSD	1.63	2.43	1.75%	1.52%
Mg H/L	%Recovery	94.77	94.30	94.42%	95.42%
	% RSD	1.78	1.40	1.82%	1.37%
Mn	%Recovery	99.50	97.03	99.34%	-
	% RSD	2.37	2.17	1.79%	-
Mo	%Recovery	96.73	99.24	97.06%	-
	% RSD	2.25	13.32	2.19%	-
Na L/L	%Recovery	99.28	99.28	99.82%	97.91%
	% RSD	3.09	3.09	1.82%	1.46%
Na H/L	%Recovery	99.33	98.86	99.05%	100.04%
	% RSD	1.69	1.03	1.41%	1.17%
Ni	%Recovery	93.98	93.56	93.97%	95.87%
	% RSD	1.96	2.35	2.31%	2.06%
P	%Recovery	108.5	109.87	109.74%	-
	% RSD	4.52	4.47	4.32%	-
Pb	%Recovery	91.01	92.33	91.84%	90.09%
	% RSD	1.73	1.8	1.71%	1.54%
Sn	%Recovery	98.66	100.7	100.20%	99.29%
	% RSD	2.69	3.29	3.29%	2.46%
Ti	%Recovery	95.47	96.33	93.88%	97.27%
	% RSD	1.72	2.71	2.19%	2.06%
Tl	%Recovery	95.44	95.6	95.57%	-
	% RSD	4.4	5.03	4.67%	-
V	%Recovery	95.75	97.61	97.47%	95.97%
	% RSD	1.74	2.2	1.78%	2.03%
Zn	%Recovery	96.27	92.87	96.20%	96.02%
	% RSD	2.48	2.57	2.32%	2.84%

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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 %
Ag	19.38
Al	12.91
B	16.59
Ba	17.65
Be	13.84
Ca	16.55
Cd	14.44
Co	15.41
Cr	14.46
Cu	13.97
Fe	11.40
K	16.27
Li	14.12
Mg	16.08
Mn	10.44
Mo	15.14
Na	20.13
Ni	16.15
P	20.23
Pb	22.26
S	16.80
Sn	15.56
Sr	15.82
Ti	14.82
Tl	15.51
V	13.84
Zn	18.15

References:

Inductively Coupled Plasma Spectrometry 1996. HMSO, Methods for the Examination of Waters and Associated Materials ISBN 0117532444

Vista (v3.0) - 'online' tutorial program.

ISO 5667-3 2018 - Water quality Sampling Part 3: Guidance on the preservation and handling of water samples