

# METHOD STATEMENT



## Determinand:

Metals: -

Aluminium, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Molybdenum, Nickel, Phosphorus, Potassium, Selenium, Sodium, Strontium, Tellurium, Thallium, Tin, Titanium, Uranium, Vanadium, Zinc,

## Matrix:

Treated sewage, untreated sewage, trade effluents, surface waters, ground waters, land leachates, prepared leachates recreational and processed 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. Filtered (otherwise known as dissolved or soluble) metals may also be determined, by filtration through a 0.45µm membrane filter prior to analysis.

## Sampling and Sample Preparation:

Samples requiring analysis are collected into the digestion tubes from 1 litre PET bottle supplied by the customer.

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

Al	14 Days (In-House Data)	Mg	10 Days (In-House Data)
As	29 Days (In-House Data)	Mn	17 Days (In-House Data)
Ba	9 Days (In-House Data)	Mo	15 Days (In-House Data)
Be	17 Days (In-House Data)	Na	7 Days (In-House Data)
B	7 Days (In-House Data)	Ni	17 Days (In-House Data)
Ca	17 Days (In-House Data)	Sr	17 Days (In-House Data)
Cd	17 Days (In-House Data)	Sb	29 Days (In-House Data)
Cr	17 Days (In-House Data)	Se	29 Days (In-House Data)
Co	17 Days (In-House Data)	Tl	17 Days (In-House Data)
Cu	14 Days (In-House Data)	Sn	7 Days (In-House Data)
Fe	17 Days (In-House Data)	Ti	14 Days (In-House Data)
K	17 Days (In-House Data)	Te	11 Days (In-House Data)
P	17 Days (In-House Data)	U	29 Days (In-House Data)
Pb	16 Days (In-House Data)	V	17 Days (In-House Data)
Li	7 Days (In-House Data)	Zn	17 Days (In-House Data)

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## 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 and by using interference equations.

Appendix IV has details of the interference equations used.

Additional isotopes for some elements have been included in the method in order to investigate suspected interference on individual

## Performance of Method:

### Total Metals

Determinand	MCERTS Accreditation	Range of Application (µg/l)	LOD (µg/l)	Routine Reporting Level (µg/l)
Lithium		2.0 - 500	1.6734	2.0
Beryllium		0.60 - 500	0.1688	0.60
Boron		60 - 5000	28.4359	60
Sodium		460 - 500000	213.8044	460
Magnesium		80 - 500000	53.0400	80
Aluminium	Y	7.5 - 5000	7.4033	7.5
Phosphorus	Y	13 - 50000	7.2469	13
Potassium		370 - 500000	279.7398	370
Calcium		325 - 500000	323.5422	325
Titanium		1.1 - 500	0.2736	1.1
Vanadium		0.08 - 500	0.0755	0.08
Chromium	Y	0.51 - 500	0.3695	0.51
Iron	Y	25 - 50000	23.3780	25
Manganese	Y	1.7 - 500	1.6704	1.7
Cobalt	Y	0.08 - 500	0.0798	0.08
Nickel	Y	1.0 - 500	0.8223	1.0
Copper	Y	1.8 - 500	1.7369	1.8
Zinc	Y	6.0 - 2500	5.2010	6.0
Arsenic	Y	0.24 - 500	0.1136	0.24
Selenium		0.60 - 500	0.2964	0.60
Strontium		12 - 5000	11.5660	12
Molybdenum		2.7 - 500	0.2961	2.7
Cadmium	Y	0.07 - 50	0.0123	0.07
Tin		1.5 - 500	1.4557	1.5
Antimony	Y	1.6 - 500	0.8758	1.6
Tellurium		0.35 - 500	0.1535	0.35
Barium		2.0 - 500	1.8337	2.0
Thallium		0.75 - 500	0.4114	0.75
Lead	Y	0.30 - 500	0.2643	0.30
Uranium		0.06 - 50	0.0569	0.06

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Determinand	Low standard			High standard		
	Conc. µg/ l	RSD %	Bias %	Conc. µg/ l	RSD %	Bias %
lithium	100	8.75%	-2.11%	400	5.79%	+3.86%
Beryllium	100	6.95%	-5.69%	400	6.29%	+0.73%
Boron	1000	6.19%	+0.92%	4000	5.90%	+0.98%
Sodium	200000	6.93%	+3.25%	400000	4.70%	+0.60%
Magnesium	200000	4.89%	+4.47%	400000	4.02%	+1.57%
Aluminium	1000	2.65%	+5.09%	4000	3.65%	+3.79%
Phosphorus	10000	4.59%	-0.61%	40000	5.08%	+1.62%
Potassium	200000	6.02%	-0.09%	400000	4.83%	+1.73%
Calcium	200000	3.15%	-0.42%	400000	4.04%	+1.44%
Titanium	100	2.67%	+2.62%	400	2.58%	+0.54%
Vanadium	100	5.69%	+3.53%	400	2.94%	+4.09%
Chromium	100	3.80%	+5.45%	400	2.85%	+4.14%
Iron	10000	3.03%	+3.56	40000	2.55%	+2.49%
Manganese	100	4.07%	+2.24%	400	2.15%	+7.09%
Cobalt	100	2.34%	+6.79%	400	2.33%	+4.99%
Nickel	100	2.26%	-1.39%	400	2.19%	+5.10%
Copper	100	1.78%	-1.08%	400	2.05%	4.14%
Zinc	500	3.46%	+1.69%	2000	2.01%	+4.17%
Arsenic	100	2.26%	+1.05%	400	3.03%	+0.80%
Selenium	100	3.73%	-3.80%	400	3.01%	-4.69%
Strontium	1000	4.15%	+0.63%	4000	3.33%	-0.11%
Molybdenum	100	3.68%	-3.12%	400	3.10%	-3.71%
Cadmium	10	2.28%	-0.52%	40	2.29%	-2.96%
Tin	100	2.66%	+3.08%	400	2.33%	-0.34%
Antimony	100	4.70%	-5.13%	400	2.84%	-1.89%
Tellurium	100	3.22%	-0.51%	400	2.81%	-3.75%
Barium	100	2.64%	-5.97%	400	2.98%	-0.54%
Thallium	100	3.55%	+6.67%	400	3.24%	+5.18%
Lead	100	2.95%	+6.21%	400	2.60%	+5.00%
Uranium	10	3.50%	-1.61%	40	3.19%	+4.17%

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Determinand	Spike Level	Treated sewage (Finham)		Trade effluent (to controlled waters) (Trimpley)		Trade effluent (to sewer) (Walkers)		Untreated sewage (Finham/Alfreton)	
		RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %	RSD %	Rec. %
Lithium	80%	6.74	99.49	6.91	103.92	7.82	97.24	5.90	101.26
Beryllium	80%	8.01	100.37	7.57	102.75	8.11	97.55	7.40	101.66
Boron	80%	5.83	102.17	9.55	105.28	7.86	96.90	6.18	102.50
Sodium	80%	4.59	101.50	4.40	102.62	5.22	100.79	5.31	100.72
Magnesium	80%	3.62	103.76	3.41	103.4	3.20	103.17	3.85	103.29
Aluminium	20%	3.44	107.20	3.64	107.64	-	-	4.62	107.41
	80%	3.24	105.31	4.04	106.60	3.56	92.39	3.59	105.58
Phosphorus	20%	4.76	101.84	4.70	102.04	-	-	5.19	97.67
	80%	4.36	102.43	4.98	103.54	4.71	107.26	4.67	100.10
Potassium	80%	4.70	103.64	5.10	104.00	3.30	102.14	5.68	102.97
Calcium	80%	3.21	104.47	3.52	104.74	3.64	105.07	4.37	103.82
Titanium	80%	2.04	104.83	2.18	103.84	2.92	93.81	2.08	102.77
Vanadium	80%	3.20	109.29	2.50	107.82	4.05	106.29	3.02	108.97
Chromium	20%	4.37	106.98	3.56	105.60	-	-	4.60	106.76
	80%	3.43	107.27	2.23	106.69	4.89	102.54	2.95	106.92
Iron	20%	2.73	105.62	3.15	106.31	-	-	2.42	105.35
	80%	2.33	104.75	2.14	105.37	3.33	102.53	3.68	104.89
Manganese	20%	5.55	104.07	5.08	88.76	-	-	5.46	105.56
	80%	1.87	109.05	1.47	101.98	3.34	94.05	2.24	109.87
Cobalt	20%	2.32	107.42	2.31	106.07	-	-	2.18	107.99
	80%	2.60	105.83	2.11	105.38	4.92	102.85	2.63	105.97
Nickel	20%	2.79	96.32	3.11	96.24	-	-	2.60	96.49
	80%	3.37	103.41	3.82	103.84	5.10	98.06	2.96	103.68
Copper	20%	2.48	96.19	3.29	94.86	-	-	4.40	99.31
	80%	2.88	101.90	2.95	102.39	3.02	96.02	2.87	104.51
Zinc	20%	4.60	105.37	4.16	103.81	-	-	4.09	104.46
	80%	2.00	104.85	1.88	105.03	3.32	93.65	2.25	105.03
Arsenic	20%	2.73	105.02	1.99	103.33	-	-	1.58	104.17
	80%	3.84	105.84	2.90	105.00	1.65	102.12	3.32	105.44
Selenium	80%	4.34	95.60	3.57	96.30	3.97	95.28	2.73	94.58
Strontium	80%	3.62	100.57	3.53	100.40	3.13	98.84	3.08	100.34
Molybdenum	80%	2.44	100.01	2.93	98.68	3.51	98.99	2.61	99.13
Cadmium	20%	2.29	98.90	2.07	99.98	-	-	4.16	99.67
	80%	2.52	97.62	2.63	98.69	2.20	94.54	2.09	96.67
Tin	20%	2.75	105.99	3.05	104.03	-	-	3.12	100.14
	80%	1.74	104.63	2.51	103.21	2.99	99.75	3.43	92.87
Antimony	20%	3.16	98.24	4.63	101.40	-	-	3.11	100.04
	80%	3.24	103.10	4.45	106.05	4.25	100.85	2.63	103.57
Tellurium	80%	3.28	98.14	3.04	98.95	2.65	94.44	2.95	97.15
Barium	80%	2.68	102.66	3.05	103.18	3.22	103.77	2.47	103.49
Thallium	80%	2.86	105.16	3.19	107.48	3.15	99.36	2.84	104.91
Uranium	80%	3.31	108.07	3.91	108.72	3.20	98.59	3.45	106.81
Lead	20%	3.14	106.28	2.98	107.63	-	-	4.34	105.46
	80%	2.56	104.57	3.32	106.02	4.44	102.92	2.66	103.51

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Determinand	Ground Water		Surface Water		Landfill Leachate		Soil Leachate	
	RSD %	Rec. %	RSD %	Rec. %	RSD %.	Rec. %	RSD %.	Rec. %
Lithium	7.53	103.35	6.98	103.06	6.97	99.72	6.58	105.08
Beryllium	6.99	102.38	7.03	101.75	6.29	97.48	5.75	103.83
Boron	6.72	100.61	6.35	99.95	8.16	92.45	5.71	100.49
Sodium	5.06	104.09	5.61	104.07	5.67	102.53	6.36	105.30
Magnesium	3.64	103.99	4.13	105.22	4.07	102.64	4.79	106.37
Aluminium	3.13	102.52	3.20	102.00	3.57	98.34	5.23	102.16
Phosphorus	2.98	103.34	4.76	105.44	5.09	102.02	5.80	107.12
Potassium	4.32	103.61	5.05	104.31	4.74	102.05	5.04	105.29
Calcium	4.23	103.74	4.52	103.71	3.60	103.13	4.61	105.67
Titanium	3.36	101.64	3.64	100.78	4.31	101.66	3.55	100.21
Vanadium	3.95	106.63	3.98	105.90	4.17	107.34	3.46	104.08
Chromium	4.31	104.68	4.57	103.42	4.97	104.14	4.11	103.87
Iron	3.80	103.89	3.70	103.76	3.96	102.81	4.04	104.67
Manganese	2.59	106.53	2.92	105.57	3.48	105.35	2.71	106.13
Cobalt	4.16	102.95	4.84	102.62	4.43	103.85	3.93	104.77
Nickel	3.98	101.59	3.98	100.98	4.42	101.86	4.22	103.58
Copper	3.44	103.04	3.08	100.34	3.87	101.99	3.24	104.63
Zinc	2.82	103.20	3.10	101.87	3.29	100.27	2.67	104.15
Arsenic	1.81	102.99	1.44	102.19	3.14	103.03	1.86	101.34
Selenium	4.13	96.18	3.39	96.80	3.82	98.48	4.60	98.21
Strontium	3.63	100.68	2.87	100.05	2.90	99.01	3.94	102.26
Molybdenum	3.46	100.36	2.84	101.18	2.73	100.94	4.24	100.22
Cadmium	2.45	99.53	1.63	97.66	2.08	95.43	3.29	100.55
Tin	2.81	102.78	1.76	102.01	3.27	101.27	1.98	102.68
Antimony	2.95	107.13	2.06	106.00	4.47	107.93	3.02	103.88
Tellurium	2.46	99.14	2.01	97.87	2.31	97.01	3.38	100.37
Barium	2.49	104.04	2.43	101.36	3.00	102.26	2.23	100.32
Thallium	3.43	106.96	2.68	105.01	2.95	104.57	2.77	106.38
Lead	3.87	106.16	3.80	103.69	2.98	104.11	3.42	106.40
Uranium	3.89	108.44	4.52	107.83	3.08	108.46	3.65	107.11

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Determinand	Recreational		Clean Process		Dirty Process	
	RSD %	Rec. %	RSD %	Rec. %	RSD %.	Rec. %
Lithium	7.04	101.71	5.88	101.05	7.32	102.11
Beryllium	6.58	100.61	5.30	100.92	7.41	101.04
Boron	6.52	98.94	5.49	98.38	4.32	103.82
Sodium	5.20	103.89	5.24	104.37	5.62	102.56
Magnesium	3.62	105.32	4.02	105.79	4.20	103.89
Aluminium	1.98	101.33	2.35	101.12	2.97	100.92
Phosphorus	4.69	105.65	5.67	105.65	4.66	105.66
Potassium	3.95	104.56	4.98	104.12	4.81	102.31
Calcium	3.48	104.22	4.40	103.78	4.10	103.16
Titanium	3.16	100.32	3.43	100.76	3.03	99.48
Vanadium	4.00	105.45	3.32	105.49	3.42	103.78
Chromium	4.80	103.44	4.63	103.72	4.42	104.57
Iron	3.68	104.45	3.75	103.84	3.96	103.99
Manganese	2.48	105.84	2.86	105.77	3.75	109.34
Cobalt	4.00	103.35	4.45	102.82	3.93	105.25
Nickel	4.31	101.91	4.73	101.32	3.10	105.07
Copper	2.90	101.91	3.63	100.71	----	----
Zinc	2.87	103.20	3.38	102.87	3.87	108.59
Arsenic	1.55	102.30	1.69	102.60	1.33	103.83
Selenium	3.77	96.55	3.58	96.66	4.16	98.75
Strontium	3.29	101.20	3.21	101.08	3.13	101.93
Molybdenum	2.66	100.88	3.06	100.21	----	----
Cadmium	1.79	98.57	2.87	98.40	2.34	98.21
Barium	2.91	101.78	1.76	100.44	2.51	100.87
Lead	3.44	104.75	3.36	104.88	2.04	102.65

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## Filtered Metals

Determinand	Range of Application (µg/ l)	LOD (µg/ l)	Routine Reporting Level (µg/ l)
Lithium	2.0- 500	1.1419	2.0
Beryllium	0.25- 500	0.2368	0.25
Boron	56- 5000	55.1595	56
Sodium	320- 500000	318.8736	320
Magnesium	200- 500000	198.5980	200
Aluminium	3.5- 5000	3.2155	3.5
Phosphorus	16- 50000	15.8538	16
Potassium	283- 500000	282.5054	283
Calcium	150- 500000	147.5733	150
Titanium	0.32- 500	0.3190	0.32
Vanadium	0.15- 500	0.1346	0.15
Chromium	0.20- 500	0.1848	0.20
Iron	20- 50000	15.6160	20
Manganese	0.20- 500	0.1695	0.20
Cobalt	0.12- 500	0.1184	0.12
Nickel	1.0- 500	0.8090	1.0
Copper	4.0- 500	3.6317	4.0
Zinc	5.0- 2500	4.7613	5.0
Arsenic	0.20- 500	0.1023	0.20
Selenium	1.2- 500	1.1294	1.2
Strontium	5.0- 5000	4.0532	5.0
Molybdenum	2.5-500	2.2559	2.5
Cadmium	0.02-500	0.0098	0.02
Tin	1.5-50	1.4225	1.5
Antimony	1.3-500	1.2404	1.3
Tellurium	1.0-500	0.2124	1.0
Barium	1.0-500	0.2197	1.0
Thallium	0.70-500	0.6959	0.70
Lead	0.30-500	0.2440	0.30
Uranium	0.06-50	0.0393	0.06

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Determinand	Ground water		Land Leachate	
	RSD %	Rec. %	RSD %	Rec. %
Lithium	3.87	98.88	4.37	94.74
Beryllium	3.12	98.07	5.28	92.95
Boron	3.24	100.15	5.71	92.18
Sodium	3.29	99.89	2.75	98.98
Magnesium	2.62	99.45	2.34	98.10
Aluminium	1.60	102.21	4.30	98.25
Phosphorus	3.68	100.37	2.95	97.41
Potassium	3.12	98.88	3.12	97.32
Calcium	2.74	99.53	2.36	99.00
Titanium	2.18	101.22	2.92	100.76
Vanadium	3.04	105.86	4.25	106.25
Chromium	3.09	104.33	4.36	103.90
Iron	2.56	100.69	2.16	99.81
Manganese	2.30	105.24	3.32	104.47
Cobalt	2.20	101.31	4.08	103.02
Nickel	2.64	100.81	3.72	101.69
Copper	2.81	101.97	2.94	101.95
Zinc	2.18	102.37	2.81	99.67
Arsenic	1.50	100.72	1.82	100.56
Selenium	2.71	93.42	4.11	96.42
Strontium	2.38	97.33	3.06	95.98
Molybdenum	1.97	98.70	3.34	99.47
Cadmium	1.68	95.51	1.79	91.76
Tin	1.88	101.77	2.14	101.23
Antimony	3.84	103.73	4.69	103.76
Tellurium	1.90	95.53	2.50	93.81
Barium	2.18	101.14	4.88	98.68
Thallium	5.69	93.60	2.55	99.04
Lead	2.05	99.36	2.02	98.36
Uranium	3.04	98.76	3.85	100.84



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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%.

Total Metals: -

Determinand (Total)	Uncertainty of Measurement (%)
Lithium	14.34
Beryllium	14.19
Boron	14.28
Sodium	13.98
Magnesium	12.77
Aluminium	18.87
Phosphorus	16.50
Potassium	12.60
Calcium	12.19
Titanium	9.41
Vanadium	18.73
Chromium	14.86
Iron	12.19
Manganese	16.99
Cobalt	14.30
Nickel	10.06
Copper	9.56
Zinc	11.17
Arsenic	11.78
Selenium	11.75
Strontium	7.59
Molybdenum	6.63
Cadmium	8.67
Tin	11.93
Antimony	12.93
Tellurium	9.02
Barium	8.34
Thallium	12.60
Lead	13.09
Uranium	17.49

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Filtered Metals: -

Determinand (Filtered)	Uncertainty of Measurement (%)
Lithium	11.24
Beryllium	14.00
Boron	14.23
Sodium	6.75
Magnesium	6.59
Aluminium	7.05
Phosphorus	10.87
Potassium	8.34
Calcium	7.05
Titanium	6.41
Vanadium	15.08
Chromium	11.89
Iron	6.55
Manganese	12.59
Cobalt	9.75
Nickel	9.33
Copper	8.65
Zinc	7.98
Arsenic	7.38
Selenium	12.63
Strontium	7.63
Molybdenum	6.37
Cadmium	14.31
Tin	5.44
Antimony	9.92
Tellurium	12.56
Barium	8.59
Thallium	11.63
Lead	5.50
Uranium	6.45

## References

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