

METHOD STATEMENT

Determinand:

This method is applicable for the determination of Cadmium, Chromium, Copper, Lead, Nickel, Zinc, Arsenic, Selenium, Antimony, Boron, Silver, Barium, Beryllium, Cobalt, Lithium, Molybdenum, Tin, Strontium, Titanium, Thallium, Uranium, and Vanadium

Matrix:

Sample Types: Raw, Potable, Surface and Ground waters.

Principle of Method:

This method uses the Agilent ICP MS and Nexion ICP-MS.

The method describes a technique for the simultaneous 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 occur. Excitation is due to the high temperatures (up to 6,000°C) 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 quadrupole and fall on to the mass detector. The intensities of the currents produced are processed and controlled by a computer system.

Interferences:

Careful choice of plasma conditions, internal standardisation, interference equations, collision or reaction gas settings and isotopes are all designed to minimise interference.

Potable waters do not tend to contain high levels of elements likely to cause adverse interferences.

Performance of the Method:

Range of Application:

Agilent & Nexion ICPMS

Element	Calibration Range (µg/l)	Element	Calibration Range (µg/l)
Cd	LOD - 6.25	Ba	LOD - 500
Cr	LOD - 62.5	Be	LOD - 100
Cu	LOD - 500	Co	LOD - 100
Pb	LOD - 31.25	Li	LOD - 500
Ni	LOD - 25	Mo	LOD - 100
Zn	LOD - 500	Sn	LOD - 100
As	LOD - 12.5	Sr	LOD - 500
Se	LOD - 12.5	Ti	LOD - 100
Sb	LOD - 6.25	Tl	LOD - 100
U	LOD - 10	Ag	LOD - 25
V	LOD - 100	B	LOD - 1250

All analytical ranges may be extended by sample dilution.

Samples with a concentration higher than that of the top standard should be diluted so that the final concentration of acid in the diluted solution should remain the same. This can be achieved by using the calibration blank solution or by using deionised (Milli-Q) water and concentrated nitric acid. The sample should then be reanalysed.



METHOD STATEMENT



Limit of Detection and Reporting limit:

Perkin Elmer Nexion

Determinand	Units	Method LOD	Normal Reporting Limit
Cd	µg/l	0.013	0.013
Cr	µg/l	0.089	0.09
Cu	µg/l	0.171	0.2
Pb	µg/l	0.017	0.02
Ni	µg/l	0.088	0.09
Zn	µg/l	0.603	0.7
As	µg/l	0.075	0.08
Se	µg/l	0.160	0.16
Sb	µg/l	0.032	0.03
Ag	µg/l	0.332	0.4
Ba	µg/l	0.183	0.2
Be	µg/l	0.094	0.10
Co	µg/l	0.037	0.04
Li	µg/l	0.392	0.4
Mo	µg/l	0.379	0.38
Sn	µg/l	0.653	0.66
Sr	µg/l	0.230	0.23
Ti	µg/l	0.428	0.5
Tl	µg/l	0.071	0.08
U	µg/l	0.011	0.011
V	µg/l	0.051	0.06

Agilent

Determinand	Units	Method LOD	Normal Reporting Limit
Cd	µg/l	0.0018	0.002
Cr	µg/l	0.0195	0.02
Cu	µg/l	0.1792	0.2
Pb	µg/l	0.0080	0.01
Ni	µg/l	0.0801	0.09
Zn	µg/l	0.6793	0.68
As	µg/l	0.0051	0.01
Se	µg/l	0.0391	0.04
Sb	µg/l	0.0037	0.01
Ag	µg/l	0.3592	0.4
Ba	µg/l	0.2080	0.3
Be	µg/l	0.0431	0.05
Co	µg/l	0.0268	0.03
Li	µg/l	1.1692	1.17
Mo	µg/l	0.0279	0.03
Sn	µg/l	0.0822	0.09
Sr	µg/l	0.2034	0.21
Ti	µg/l	0.0408	0.05
Tl	µg/l	0.0289	0.03
U	µg/l	0.0040	0.01
V	µg/l	0.0294	0.03



METHOD STATEMENT



Recoveries of Compounds: Perkin Elmer Nexion ICPMS

	Soft Water		Medium Water		Hard Water		Raw Surface		Borehole Water		Filtered Hard Water	
	%Rec	%RSD	%Rec	%RSD	%Rec	%RSD	%Rec	%Rec	%Rec	%RSD	%Rec	%RSD
Li	105.41	2.51	105.52	2.05	104.54	1.88	106.48	2.51	105.53	2.21	106.79	1.57
Be	104.07	4.07	107.43	2.69	107.56	2.60	106.06	3.00	106.87	3.14	108.20	2.40
V	105.22	1.64	105.54	1.16	105.61	1.17	104.54	2.02	105.35	1.44	105.37	1.33
Ti	103.64	2.23	104.16	1.42	104.15	2.27	103.65	1.97	103.79	1.69	104.03	1.78
Cr	101.14	1.56	101.02	1.04	100.28	1.34	100.02	1.93	100.30	1.87	100.35	1.16
Co	101.34	1.29	101.49	1.32	100.68	1.50	100.94	1.22	100.52	1.35	101.33	1.40
Ni	100.64	1.51	101.67	1.64	99.25	2.03	99.85	1.80	99.34	2.30	100.32	1.47
Cu	100.32	1.31	99.68	1.16	97.43	1.47	98.79	1.34	97.82	1.39	99.01	1.10
Zn	100.09	1.42	99.41	1.36	97.01	1.69	98.85	1.73	98.24	1.41	99.19	1.64
As	104.98	2.00	105.81	1.96	105.87	2.18	105.42	2.13	104.35	2.55	105.67	1.86
Se	103.96	2.88	104.46	3.12	105.25	3.59	104.57	3.94	103.97	3.73	103.64	2.72
Sr	104.01	1.93	104.25	1.72	101.24	3.36	101.06	1.71	101.84	1.74	101.44	2.91
Mo	102.73	1.03	103.11	1.21	103.08	1.28	102.55	1.31	103.18	1.37	103.05	0.97
Ag	100.75	1.69	99.52	2.06	98.87	2.37	97.88	2.76	98.19	3.18	100.60	1.46
Cd	102.33	2.54	102.26	2.85	101.41	2.61	102.08	1.61	102.08	1.57	103.23	2.01
Sb	103.65	1.92	103.72	1.68	104.03	1.85	103.46	1.71	103.41	1.53	103.00	2.02
Sn	103.39	1.62	103.70	1.34	103.77	1.43	103.50	1.24	103.61	1.56	103.15	1.07
Ba	102.59	1.48	102.70	1.77	102.66	1.84	102.53	1.81	98.61	1.91	97.84	2.83
Tl	103.42	1.03	103.87	1.16	104.30	1.18	103.79	1.18	104.23	1.29	103.71	1.01
Pb	107.02	1.06	107.18	1.06	107.12	1.08	106.90	0.99	107.22	1.52	107.16	1.06
U	105.59	1.16	106.40	1.37	107.62	1.11	105.95	1.09	106.62	1.37	105.78	0.95

Agilent ICPMS

	Soft Water		Medium Water		Hard Water		Raw Surface		Borehole Water		Filtered Hard Water	
	%Rec	%RSD	%Rec	%RSD	%Rec	%RSD	%Rec	%Rec	%Rec	%RSD	%Rec	%RSD
Li	101.74	2.87	101.53	2.98	98.06	2.02	98.39	2.62	100.73	2.73	101.31	3.13
Be	103.18	2.66	103.61	2.63	99.85	2.25	99.20	2.53	102.80	2.72	103.39	2.90
V	103.95	1.18	104.41	1.19	103.41	0.84	103.39	2.24	104.02	1.18	104.08	0.93
Ti	101.27	1.54	101.47	1.43	101.47	1.29	101.92	2.31	101.20	1.40	101.54	1.40
Cr	99.14	1.69	98.70	1.35	97.41	1.48	97.92	2.30	98.14	1.40	98.54	1.37
Co	99.71	0.98	99.18	1.02	98.18	0.95	99.25	2.08	97.32	0.98	97.95	1.04
Ni	98.13	2.36	98.16	1.17	94.85	1.69	96.86	2.38	94.72	1.36	95.82	1.30
Cu	97.65	1.27	96.16	1.00	93.26	0.97	96.03	2.09	93.34	0.91	94.45	1.11
Zn	98.66	1.68	97.98	1.25	95.72	1.28	97.19	2.63	96.24	1.38	97.86	1.40
As	101.66	1.37	102.15	1.41	103.16	1.07	102.58	2.42	101.36	1.21	102.00	1.13
Se	99.95	1.77	101.13	1.41	102.09	1.13	101.65	2.42	100.84	1.36	101.11	1.95
Sr	99.70	1.64	99.29	1.85	96.47	2.64	96.67	3.81	99.41	1.69	98.03	1.78
Mo	102.02	1.38	102.33	1.54	102.40	1.29	101.81	2.37	102.02	2.03	102.41	1.44
Ag	96.95	2.17	95.94	1.82	94.74	1.77	93.97	4.01	93.82	2.30	96.36	1.50
Cd	100.11	1.68	100.07	1.29	98.70	1.46	99.31	2.86	99.41	1.31	99.85	1.21
Sb	100.99	1.52	101.52	1.29	101.64	1.32	101.71	2.62	101.06	1.22	101.12	1.21
Sn	101.51	1.64	101.98	1.55	101.59	1.59	101.86	2.65	101.44	1.60	101.59	1.41
Ba	101.59	1.63	102.00	1.57	102.38	1.28	102.30	2.69	98.17	2.04	98.45	1.46
Tl	101.52	1.40	102.01	1.21	102.15	1.05	102.08	2.63	102.21	1.12	102.05	1.41
Pb	102.28	1.72	102.48	1.15	102.00	1.07	102.60	2.52	101.90	1.40	102.13	1.39
U	99.15	1.59	99.58	1.26	99.10	1.14	99.18	2.26	99.12	1.14	99.36	1.37



METHOD STATEMENT



References:

In house method based on SCA bluebooks.

Nexion 300 ICP-MS System Customer Hardware Documentation (Software CD) Part No. W1032732-C

DWI Guidance note Sample Preservation and Preparation for Metals Analysis of Drinking Water

In-house Method WPC44- Metals Digestion Procedure

