

METHOD STATEMENT



Determinand:

Determination of Total and Dissolved (filtered) Aluminium, Manganese, Iron, Phosphorus, Boron, Calcium, Magnesium, Sulphate and Silica + Total Sodium and Total Potassium.

Matrix:

Sample Types: Raw, Potable, Surface, high purity, DI waters, Bottled and Ground waters.

Principle of Method:

This method uses the Perkin Elmer Optima 7300DV ICP-OES.

Samples are acidified and digested at elevated temperatures to ensure any particulate matter is brought into solution. The acidified samples are analysed on an ICP-OES instrument, where excitation of the sample within the 6,000°C plasma causes ionisation of atoms, which in turn causes the emission of electromagnetic radiation at specific wavelengths for each element. The intensity of the emission is measured and quantified by comparison against standards with known concentrations of elements. An internal standard solution is used to compensate the measurement for variances within the plasma on a sample-by-sample basis.

Sampling and Sample Preparation:

Samples are normally collected in polyethylene (HDPE) bottles.

On receipt at the laboratory the samples are acidified with concentrated nitric acid such that the final concentration of acid is ~1% V/V (e.g. 1.00 ± 0.10 cm³ of acid to each 100 ± 10 cm³ of sample). If dissolved (filtered) metals analysis is required on a sample, the sample must be filtered through a 0.45 µm filter disc prior to acidification. Following acidification, the samples should be digested in the oven provided at 80°C ± 5 °C for a minimum of 6 hours and allowed to cool prior to analysis. See method No. WPC44 for the metals preparation and digestion procedure.

If samples require emergency analysis they may be acidified as above (see section 6.2), microwaved for 1 minute each then run on the appropriate instrument. This has been shown in testing to be at least as effective as the oven digest. However this should only be used for emergency samples and not routine analysis.

If analysis cannot be immediately undertaken, samples can be stored at room temperature until the day of analysis. Samples should be analysed within 30 days of the sampling date.

Interferences

Choice of analytical wavelength and placement of background correction points has been designed to eliminate possible interference from other elements within the sample.

This is further aided by the fact that the elements being measured are generally at significantly higher concentrations than any possible interfering species.

Performance of Method:

Range of Application:

Determinand	Calibration range
Fe	LOQ - 5000 µg/l
Mn	LOQ - 1000 µg/l
Al	LOQ - 5000 µg/l
P	LOQ - 5000 µg/l
B	LOQ - 1000 µg/l
Ca	LOQ - 250 mg/l
Mg	LOQ - 50mg/l
Na	LOQ - 200mg/l

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K	LOQ - 20 mg/l
SO ₄	LOQ - 250 mg/l
Si	LOQ - 20 mg/l

The analytical range 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.

Limit of Quantification and Reporting limit:

Determinand	Units	Method LOQ	Normal Reporting Limit
Al	µg/l	14.4320	14.4
Fe	µg/l	5.2248	5.23
Mn	µg/l	0.8421	0.85
P	µg/l	20.1125	20.1
Ca	mg/l	0.3038	0.31
Mg	mg/l	0.0739	0.08
Na	mg/l	1.5872	1.59
K	mg/l	0.3637	0.37
SO ₄	mg/l	0.6766	0.68
B	µg/l	5.2762	5.28
Si	mg/l	0.0307	0.04

Recoveries of Compounds:

	Soft Water		Medium Water		Hard Water		Raw Surface	
	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
Al	101.2	3.17	102.49	3.03	102.59	3.50	103.60	3.33
Fe	105.85	2.91	106.25	3.03	105.60	3.38	101.43	2.24
Mn	104.09	3.24	105.02	2.89	104.94	3.29	101.11	2.26
P	100.06	3.21	103.78	3.16	103.46	3.08	102.26	3.03
Ca	101.21	2.78	97.81	2.39	97.80	2.49	101.07	2.71
Mg	102.16	1.37	102.54	2.29	102.26	1.65	103.80	2.71
Na	101.62	2.26	100.71	1.94	101.27	2.16	101.74	2.19
K	105.05	0.79	105.68	2.60	103.53	1.29	104.69	1.63
SO ₄	101.60	1.47	99.36	1.78	98.82	1.62	101.34	1.60
B	101.46	1.77	103.65	2.47	103.69	2.24	102.89	2.21
Si	101.15	1.24	101.25	1.82	10.086	1.66	102.24	1.12

	Borehole Water		Filtered Hard Water	
	% Rec	% RSD	% Rec	% RSD
Al	102.71	3.07	102.33	2.87
Fe	105.90	3.36	105.56	2.89
Mn	103.60	2.40	104.71	3.48
P	102.89	3.39	104.16	2.98
Ca	100.40	2.28	100.91	2.33
Mg	97.35	1.96	102.39	1.92
Na	100.94	1.92	101.46	2.14
K	102.60	1.24	103.83	1.03
SO ₄	102.88	1.51	103.09	1.48
B	102.89	2.57	102.60	2.26

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	Borehole Water		Filtered Hard Water	
	% Rec	% RSD	% Rec	% RSD
Si	100.72	1.88	101.26	1.50

References:

In house method based on SCA bluebook 163 Inductively Coupled Plasma Spectrometry 1 996 and DWI Guidance note Sample Preservation and Preparation for Metals Analysis of Drinking Water.

Perkin Elmer Optima 7100, 7200 and 7300 series Hardware Guide manual.

Water Quality-Sampling-Part 3: Guidance on the Preservation and Handling of Water Samples. BS EN ISO 5667-3-2003.

In-house Method WPC44- Metals Digestion Procedure