

METHOD STATEMENT



Determinand:

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

Matrix:

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

Principle of Method:

Samples are acidified and digested at elevated temperatures to ensure any particulate matter is brought into solution. The acidified samples are analysed using an ICP-OES instrument, where excitation of the sample within the 6,000°C plasma causes ionisation of atoms. This 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.

Sampling and Sample Preparation:

Samples are normally collected in polyethylene (HDPE) bottles.

125ml azlons bottles are sent to customers for total metals analysis. The bottles are then acidified on site. 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; these samples are received at the laboratory in non-acidified bottles. Some filtered metals samples are received in 125ml azlons bottles which have already been acidified by the customer and marked up accordingly. Upon receipt and following filtration/acidification if required, 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 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
Al	LOQ - 5000 µg/l
Mn	LOQ - 1000 µg/l
Fe	LOQ - 5000 µg/l
P	LOQ - 5000 µg/l
Ca	LOQ - 250 mg/l
Mg	LOQ - 50 mg/l
Na	LOQ - 200 mg/l
K	LOQ - 20 mg/l
SO ₄	LOQ - 250 mg/l
B	LOQ - 1000 µg/l
Si	LOQ - 20 mg/l

All analytical ranges may be extended by sample dilution. The final concentration of acid in the diluted solution should remain the same.

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Limit of Quantification and Reporting limit:

Determinand	Units	Method LOQ	Normal Reporting Limit
Fe	µg/l	3.7186	3.72
Mn	µg/l	0.2916	0.30
Al	µg/l	6.8936	6.90
Na	mg/l	0.7646	0.77
K	mg/l	0.1679	0.17
Ca	mg/l	0.6548	0.66
Mg	mg/l	0.1297	0.13
SO ₄	mg/l	0.5356	0.54
Si	mg/l	0.0640	0.07
P	µg/l	22.8061	22.8
B	µg/l	31.8472	31.9

Recoveries of Compounds:

	Soft Water		Medium Water		Hard Water		Raw Surface	
	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD	% Rec	% RSD
Fe	107	1.15	107	3.13	105	1.96	105	5.58
Mn	109	1.34	108	2.63	104	1.88	107	4.25
Al	104	2.34	104	2.76	103	2.01	102	4.68
Na	101	5.63	104	2.46	104	1.40	104	1.56
K	101	2.70	101	1.78	98.8	1.28	99.8	1.36
Ca	103	2.47	99.9	1.77	95.9	2.11	97.6	2.89
Mg	104	1.48	103	1.81	102	1.36	103	1.62
SO ₄	101	1.97	102	1.91	104	1.64	103	1.75
Si	105	1.32	103	1.45	102	0.86	101	1.14
P	102	1.18	103	1.97	101	1.32	104	4.49
B	102	1.38	102	1.63	102	1.03	101	1.44

	Borehole Water		Filtered Hard Water	
	% Rec	% RSD	% Rec	% RSD
Fe	105	1.94	105	2.53
Mn	105	1.47	107	2.15
Al	102	2.31	104	2.92
Na	104	1.82	104	1.32
K	99.1	2.22	101	1.48
Ca	95.7	1.93	96.1	2.02
Mg	96.1	1.88	104	1.60
SO ₄	103	2.03	103	1.99
Si	102	1.58	101	0.99
P	101	1.22	91	4.41
Si	101	1.08	101	1.02

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.

Agilent ICP Expert II software help files.

Water Quality-Sampling-Part 3: Guidance on the Preservation and Handling of Water Samples. BS EN ISO 5667-3-2018

In-house Method WPC44- Metals Digestion Procedure