

# METHOD STATEMENT



## Determinand:

Bromide, Sulphate, Chloride, Nitrite and Nitrate.

## Matrix:

Surface waters, groundwaters, prepared soil leachates, trade effluents, treated sewage effluents, untreated sewage and landfill leachates for Sulphate, Chloride and Bromide. Then for Nitrite, Nitrate and Sulphate process waters and recreational waters.

## Principle of Method:

The samples are analysed using an ion chromatography system, fitted with a conductimetric detector. Automatic calculation is performed by the system's data analysis software.

## Sampling and Sample Preparation

Samples are received in 1 litre PET bottles for sulphate, chloride, nitrate, and nitrite analysis. Sub-samples are taken from the 1L PET bottle into a 28ml glass vial. 100ml amber bottles (STL 061) are received for bromide analysis.

Samples are stable for times stated below, from sampling: -

Bromide	28 Days (ISO 5667:3)
Sulphate	30 Days (In-House Data)
Chloride	28 Days (ISO 5667:3)
Nitrite/TON	18 Days (In House Data)
Nitrate	30 days (In House Data)

## Interferences:

Any substance with the same retention time as the analytes on the ion chromatograph may cause interference.

## Performance of Method:

Range of Application without dilution: -	Sulphate	0.20 - 400 mg/l
	Bromide	0.20 - 50 mg/l
	Chloride	0.20 - 400 mg/l
	Nitrite	1.00 - 200mg/l
	Nitrate	0.20 - 400 mg/l
	Nitrite as N	0.30 - 60.87 mg/l
	Nitrate as N	0.05 - 90.32 mg/l
	Nitrite as NaNO <sub>2</sub>	1.50- 300 mg/l
	TON as NO <sub>3</sub>	0.35 - 151.19 mg/l

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## ICS2000:

Determinand	LOD (mg/l)	MRL (mg/l)	Low Standard		High Standard	
			% RSD	% Bias	% RSD	% Bias
Bromide	0.0188	0.20	2.70	1.36	1.69	0.90
Sulphate	0.142	0.20	1.64	3.64	0.76	4.47
Chloride	0.059	0.20	1.04	1.49	0.91	2.84
Nitrite	0.0712	1.00	1.47	-0.31	1.72	-1.17
Nitrate	0.0298	0.20	2.17	0.23	1.25	2.48

Matrix	Sulphate		Bromide		Chloride	
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD
Groundwater	98.44	0.79	97.08	1.97	99.33	0.80
Surface Water	97.15	0.80	99.94	1.15	98.89	0.90
Final Effluent	98.03	0.71	99.29	1.63	98.78	0.99
Soil Leachate	98.87	0.79	92.70	2.11	99.30	0.79
Landfill Leachate	97.84	0.77	99.09	1.32	99.26	0.91
Untreated Sewage	96.63	0.69	99.56	1.63	97.21	0.82
Trade Effluent	100.14	0.94	99.44	1.30	98.78	20.99

Matrix	Nitrite		Nitrate		Sulphate	
	% Recovery	% RSD	% Recovery	% RSD	% Recovery	% RSD
Clean Process	101.60	2.22	103.29	1.12	102.53	1.26
Dirty Process	103.66	1.33	100.78	0.95	101.03	0.87
Recreational	99.69	1.16	101.43	0.86	100.84	0.71

## Integriion:

Determinand	LOD (mg/l)	MRL (mg/l)	Low Standard		High Standard	
			% RSD	% Bias	% RSD	% Bias
Bromide	0.0344	0.20	1.62	10.14	2.38	3.61
Sulphate	0.1421	0.20	1.48	8.60	2.37	0.67
Chloride	0.0706	0.20	1.50	3.27	2.40	0.13
Nitrite	0.0466	1.00	1.34	2.84	2.80	1.93
Nitrate	0.1236	0.20	1.55	2.18	2.37	1.50

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Matrix	Sulphate		Bromide		Chloride	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Surface Water	98.19	2.01	100.89	1.93	92.60	1.94
Trade Effluent	97.77	1.77	102.16	1.67	99.64	1.71
Final Effluent	98.19	1.97	101.42	1.88	90.87	1.94
Untreated Sewage	98.50	1.68	97.81	1.57	91.20	1.64
Ground water	99.58	1.88	100.60	2.06	94.83	1.97
Prepared Leachate	99.99	2.04	102.26	1.98	100.18	1.88
Landfill Leachate	98.01	1.93	96.82	2.21	95.28	2.05

Matrix	Nitrite		Nitrate		Sulphate	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Clean Process	100.57	2.57	100.26	2.25	100.98	2.36
Dirty Process	95.38	3.00	98.85	2.20	96.03	1.98
Recreational	100.11	2.70	100.26	2.67	102.91	2.37

## 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 (%)
Bromide	9.26
Sulphate	8.95
Chloride	13.68
Nitrite	9.08
Nitrate	6.54

## References:

The determination of Anions and Cations, Transition Metals, Other Complex Ions and Organic Acids and bases in Water by chromatography 1990 ISBN 0-11-752331-3.

ISO 5667-3:2018- Water quality Sampling Part 3: Preservation and handling of water samples.