

# METHOD STATEMENT



## Determinand:

Bromide, Sulphate, Chloride, Nitrite and Nitrate.

## Matrix:

Surface waters, groundwaters, 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 10ml plastic sample tube. 100ml amber bottles (STL 061) are received for bromide analysis, if not received PET bottle can also be used. All samples to be stored at  $3\pm 2^{\circ}\text{C}$

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	30 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 $\text{NaNO}_2$	1.50- 300 mg/l
	TON as $\text{NO}_3$	0.35 - 151.19 mg/l

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## Integriion IC\_D:

Determinand	LOD (mg/l)	MRL (mg/l)	Low Standard		High Standard	
			% RSD	% Bias	% RSD	% Bias
Bromide	0.0215	0.20	2.51	-3.65	2.53	4.73
Sulphate	0.0462	0.20	1.47	-4.61	1.27	-0.93
Chloride	0.0643	0.20	1.42	3.71	1.12	3.96
Nitrite	0.0144	1.00	1.12	5.78	0.85	2.62
Nitrate	0.0529	0.20	1.52	-3.57	1.43	2.34

Matrix	Sulphate		Bromide		Chloride	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Surface Water	97.67	1.15	96.49	1.68	98.97	1.02
Trade Effluent	95.77	1.21	97.19	1.49	100.83	1.12
Final Effluent	96.96	1.34	90.56	1.65	99.74	1.21
Untreated Sewage	96.08	1.07	95.29	2.09	99.34	0.89
Ground water	97.70	1.39	95.88	2.21	99.94	1.26
Landfill Leachate	98.44	1.45	92.41	2.08	98.65	1.27

Matrix	Nitrite		Nitrate		Sulphate	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Clean Process	101.68	0.82	100.48	1.36	101.84	1.36
Dirty Process	101.94	0.78	98.99	1.42	95.88	1.24
Recreational	100.00	1.00	98.48	1.69	99.47	1.58

## Integriion IC\_E:

Determinand	LOD (mg/l)	MRL (mg/l)	Low Standard		High Standard	
			% RSD	% Bias	% RSD	% Bias
Bromide	0.0295	0.20	2.25	0.25	1.89	-0.54
Sulphate	0.0841	0.20	0.43	-4.34	0.41	-1.81
Chloride	0.0925	0.20	0.76	0.97	1.04	2.26
Nitrite	0.1953	1.00	1.88	6.57	0.97	0.23
Nitrate	0.0675	0.20	0.57	-2.99	0.39	-0.65

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Matrix	Sulphate		Bromide		Chloride	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Surface Water	101.80	0.47	99.97	1.58	101.43	1.23
Trade Effluent	101.35	0.30	99.43	1.58	102.59	1.16
Final Effluent	103.51	0.33	92.66	1.46	103.35	1.23
Untreated Sewage	99.99	0.58	98.65	1.62	101.86	1.41
Ground water	100.43	0.40	99.91	1.57	101.56	1.21
Landfill Leachate	100.45	0.46	90.49	1.68	101.47	1.93

Matrix	Nitrite		Nitrate		Sulphate	
	%Recovery	%RSD	%Recovery	%RSD	%Recovery	%RSD
Clean Process	98.01	0.91	99.22	0.41	101.71	0.39
Dirty Process	97.71	1.29	98.34	0.60	96.37	0.30
Recreational	98.80	1.70	98.23	0.38	100.16	0.50

## 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	14.41
Sulphate	7.58
Chloride	6.17
Nitrite	5.64
Nitrate	4.64

## 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 5667-3:2024- Water quality Sampling Part 3: Preservation and handling of water samples.