

METHOD STATEMENT



Determinand:

Determination of Bromate

Matrix:

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

Principle of Method:

This method uses Thermo Scientific Integriion and/or ICS6000 and associated accessories. Detection of Bromate is obtained by applying an acidic solution of potassium iodide containing a catalytic amount of molybdenum (VI) where the bromate reacts with iodide to form tri-iodide ions in a post column reaction (PCR) step. The tri-iodide is then measured by UV detection at 352nm. The amount of tri-iodide is directly proportional to the quantity of bromate in the sample.

Sampling and Sample Preparation:

Samples are normally collected in 100 ml, 250 ml or 300 ml amber glass bottles. No special preservation is required

If analysis cannot be immediately undertaken, samples should be stored at a temperature of 1 - 5°C until the day of analysis. Samples should be warmed up to room temperature prior to analysis and analysed within 31 days of the sampling date.

Interferences

Any peak that co-elutes at the same time as Bromate.

Performance of Method:

Range of Application:

LOQ - 20 µg/l BrO₃

The analytical ranges may be extended by sample dilution with deionised (Milli-Q) water.

Reporting Limit is 0.14 µg/l BrO₃

Limit of Quantification:

DNX4: 0.0686 µg/l BrO₃

DNX6: 0.132 µg/l BrO₃

Recoveries of Compounds, Bias and Uncertainty of measurement:

DNX4

Sample type	Mean sample result (µg/l)	Mean sample spike result (µg/l)	Conc. of spike (µg/l)	Spike recovery (%)	Bias (%)	% uncertainty
Soft- Langsett	0.6237	10.3701	10.0	101.30		± 1.56
Medium- Tophill Low	0.6237	10.6465	10.0	100.23		± 0.78
Hard- Purton (Bristol)	1.5040	11.5053	10.0	100.01		± 0.98
Borehole - Goose House BH2	0.0049	10.0709	10.0	100.66		± 1.00
Raw-(Surface) Derwent at Elvington	0.8725	10.8926	10.0	100.20		± 1.00

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Sample type	Mean sample result (µg/l)	Mean sample spike result (µg/l)	Conc. of spike (µg/l)	Spike recovery (%)	Bias (%)	% uncertainty
Bottle water - Strathmore	0.0049	10.0371	10.0	100.32		± 0.80
Spiked LOD sample	-	0.5059	0.5		1.19	
4 µg/l Std	-	4.0249	4.00		0.62	
16 µg/l Std	-	16.3419	16		2.14	

DNX6

Sample type	Mean sample result (µg/l)	Mean sample spike result (µg/l)	Conc of spike (µg/l)	Spike recovery (%)	Bias (%)	% uncertainty
Spiked LOD sample	-	0.49	0.5	-	-2.61	-
5 µg/l Std	-	4.85	5	-	-2.94	-
15 µg/l Std	-	14.43	15	-	-3.79	-
Graincliffe Treated	0.25	10.03	-	97.80	-	± 2.61

References:

Thermo Scientific Integriion user's instruction guides

Thermo scientific (Dionex) Technical Note 116 - Determination of Bromate by ISO Method 11206