METHOD STATEMENT

Determinand:
Determination of Bromide, Chlorate and Chlorite.

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

Principle of Method:
This method uses Metrohm Compact IC Pro and associated accessories.
Detection of the anions Chlorite, Bromide and Chlorate using direct injection ion chromatography with a sodium carbonate eluent, sequential suppression, conductivity detection and inline ultra-filtration.

Sampling and Sample Preparation:
Samples are normally collected in 100ml 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 the following days of the sampling date.
- Bromide   28 days
- Chlorate   19 days
- Chlorite   19 days

Interferences
Any peak that co-elutes at the same time as Chlorate, Bromide or Chlorite.

Performance of Method:

Range of Application:
Chlorate   LOQ - 500 µg/l as ClO3
Chlorite   LOQ - 500 µg/l as ClO2
Bromide   LOQ - 500 µg/l as Br
Samples with a concentration of the required analyte which is higher than that of the top standard of 500 µg/l should be diluted with deionised (Milli-Q) water and reanalysed.

Limit of Quantification:

Determinand µg/l	Quantification Limit (µg/l)	Reporting limit (µg/l)
Chlorite	5.1471	7.3
Bromide	0.9833	6.6
Chlorate	2.2137	5.1

Reporting limits are the highest of 3 instruments currently in use, these parameters are covered by methods WPC61 and WPC18.

Recoveries of Compounds, Bias and Uncertainty of measurement:

Chlorite

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	403.8	396.8	101.8	-	± 6.22
Medium- Hoober	0	396.4	396.8	99.9	-	±6.78
Hard- Elvington	0	398.0	396.8	100.3	-	±5.24
Borehole - Nutwell	0	394.2	396.8	99.3	-	±6.51

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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
Raw-Derwent	0	398.6	396.8	100.5	-	±6.96
Bottle -Buxton	0	392.3	396.8	98.9	-	±6.45
Bristol - Littleton	0	393.1	396.8	99.1	-	± 3.75
100 µg/l Std	97.3	-	-	-	-2.72	±6.99
400 µg/l Std	410.3	-	-	-	+2.24	±6.42

Bromide

Sample type	Mean sample result (µg/l)	Mean sample spike result (µg/l)	Conc. of spike (µg/l)	Spike recovery (%)	Bias (%)	% uncertainty
Soft- Langsett	7.34	410.3	396.8	101.5	-	± 6.49
Medium- Hoober	28.1	431.6	396.8	101.7	-	± 8.10
Hard- Elvington	23.7	431.9	396.8	102.9	-	± 7.33
Borehole - Nutwell	138.6	268.1	129.0	100.4	-	± 6.67
Raw-Derwent	49.7	454.7	396.8	102.1	-	± 9.31
Bottle -Buxton	128.3	259.2	129.0	101.5	-	± 7.44
Bristol - Littleton	28.2	427.3	398.6	100.6	-	± 4.28
100 µg/l Std	100.1	-	-	-	+0.11	± 3.29
400 µg/l Std	416.6	-	-	-	+4.14	± 8.56

Chlorate

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	402.2	398.6	101.4	-	± 6.07
Medium- Hoober	48.9	454.1	398.6	102.1	-	± 8.16
Hard- Elvington	74.1	382.3	297.6	103.6	-	± 8.13
Borehole - Nutwell	4.56	411.4	398.6	102.5	-	± 9.07
Raw-Derwent	2.21	407.8	398.6	102.2	-	± 9.15
Bottle -Buxton	0	408.6	398.6	103.0	-	± 8.29
Bristol - Littleton	3.32	407.3	398.6	101.1	-	± 4.64
100 µg/l Std	100.1	-	-	-	+0.09	± 2.84
400 µg/l Std	410.4	-	-	-	+2.59	± 7.47

References:
Metrohm user's instruction guides.
Metrohm Application Setup - Analysis of Chlorite, Bromide, and Chlorate in water samples
Water Quality-Sampling-Part 3: Guidance on the Preservation and Handling of Water Samples. BS EN ISO 5667-3-2012.
The Stability and Preservation of Drinking, Ground and Surface Water Samples 2018. Standing Committee of Analysts.