METHOD STATEMENT



Determinand:

Determination of Bromide, Chlorate and Chlorite.

Matrix:

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

Principle of Method:

This method applies to the Thermo Scientific Integrion.

Direct Injection Ion Chromatography using an electrolytically generated potassium hydroxide eluent combined with a hydroxide selective column and using suppressed conductivity detection for the determination of chlorite, chlorate and bromide.

Sampling and Sample Preparation:

Samples are normally collected in 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 $3 \pm 2^{\circ}$ 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 31 daysChlorate 19 daysChlorite 19 days.

Interferences

High concentrations of chloride, sulphate and other anions can cause poor chromatography including poor peak resolution and column overload. Metals in high concentration can irreversibly damage the analytical column set.

Performance of Method:

Range of Application:

Chlorate LOQ - $500 \mu g/l$ as ClO3 Chlorite LOQ - $500 \mu g/l$ as ClO2 Bromide LOQ - $500 \mu g/l$ as Br

The analytical range may be extended by sample dilution. Samples with a concentration higher than that of the top standard of $500 \,\mu\text{g/l}$ should be diluted with deionised (Milli-Q) water.

Limit of Quantification:

| | The | Thermo Scientic Integrion | | |
|-------------|-----------------------|-----------------------------|---------------------------|--|
| Determinand | Quantification | Quantification Limit (µg/l) | | |
| µg/l | DNX_3 - asset 2734 | DNX_5 - asset 3214 | Reporting limit (µg/l) | |
| Chlorate | 5.05 | 2.96 | 5.1 | |
| Chlorite | 4.96 | 4.39 | 5.2 | |
| Bromide | 8.87 | 2.75 | 8.9 | |

Recoveries of Compounds:

Thermo Scientific Integrion - DNX3

| | Bromide | Chlorate | Chlorite |
|--------------|---------|----------|----------|
| Soft Water | 99.47 | 100.47 | 94.95 |
| Medium Water | 99.76 | 99.80 | 90.08 |
| Hard Water | 99.47 | 99.76 | 92.32 |
| Raw Surface | 98.87 | 95.92 | 94.16 |

METHOD STATEMENT

Hard Water



| Raw Borehole | 102.37 | 98.94 | 98.77 | |
|------------------------------------|---------|----------|----------|--|
| Bottled Water | 99.70 | 98.47 | 97.04 | |
| Thermo Scientific Integrion - DNX5 | | | | |
| | Bromide | Chlorate | Chlorite | |

100.65

Bias, RSD and Uncertainty of measurement:

Thermo Scientific Integrion-DNX3

| | Thomas defending integriori Britie | | | | |
|---|------------------------------------|--------------------|--------|-------|---------------|
| | | Concentration µg/l | % Bias | % RSD | % Uncertainty |
| | Bromide | 400 | 0.55 | 2.00 | ±10.526 |
| ĺ | Chlorate | 400 | 0.05 | 2.19 | ±11.583 |
| | Chlorite | 400 | 0.26 | 2.06 | ±20.009 |

105.69

103.36

Thermo Scientific Integrion-DNX5

| | Concentration µg/l | % Bias | % RSD | % Uncertainty |
|----------|--------------------|--------|-------|---------------|
| Bromide | 400 | 2.12 | 1.71 | ±5.595 |
| Chlorate | 400 | 2.30 | 1.84 | ±12.381 |
| Chlorite | 400 | 3.35 | 1.80 | ±7.835 |

References:

Determination of Disinfection By-product Anions and Bromide in Drinking Water Using A Reagent Free Ion Chromatography System Followed by Postcolumn Addition of an Acidified Online Generated Reagent for Trace Bromate Analysis. Application Note 171, Dionex Corporation.

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