

# 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 BrO<sub>3</sub>

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

Reporting Limit is 0.14 µg/l BrO<sub>3</sub>

### Limit of Quantification:

DNX4: 0.0686 µg/l BrO<sub>3</sub>

DNX6: 0.132 µg/l BrO<sub>3</sub>

## 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                     | 0.2398                    | 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 Integrion user's instruction guides

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