

# METHOD STATEMENT



## Determinand:

Bromide, Sulphate, Chloride, Nitrite and Nitrate.

## Matrix:

Surface waters, groundwaters, prepared soil leachates, 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. 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 | 18 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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## ICS2000:

| Determinand | LOD (mg/l) | MRL (mg/l) | Low Standard |        | High Standard |        |
|-------------|------------|------------|--------------|--------|---------------|--------|
|             |            |            | % RSD        | % Bias | % RSD         | % Bias |
| Bromide     | 0.0188     | 0.20       | 2.70         | 1.36   | 1.69          | 0.90   |
| Sulphate    | 0.142      | 0.20       | 1.64         | 3.64   | 0.76          | 4.47   |
| Chloride    | 0.059      | 0.20       | 1.04         | 1.49   | 0.91          | 2.84   |
| Nitrite     | 0.0712     | 1.00       | 1.47         | -0.31  | 1.72          | -1.17  |
| Nitrate     | 0.0298     | 0.20       | 2.17         | 0.23   | 1.25          | 2.48   |

| Matrix            | Sulphate   |       | Bromide    |       | Chloride   |       |
|-------------------|------------|-------|------------|-------|------------|-------|
|                   | % Recovery | % RSD | % Recovery | % RSD | % Recovery | % RSD |
| Groundwater       | 98.44      | 0.79  | 97.08      | 1.97  | 99.33      | 0.80  |
| Surface Water     | 97.15      | 0.80  | 99.94      | 1.15  | 98.89      | 0.90  |
| Final Effluent    | 98.03      | 0.71  | 99.29      | 1.63  | 98.78      | 0.99  |
| Soil Leachate     | 98.87      | 0.79  | 92.70      | 2.11  | 99.30      | 0.79  |
| Landfill Leachate | 97.84      | 0.77  | 99.09      | 1.32  | 99.26      | 0.91  |
| Untreated Sewage  | 96.63      | 0.69  | 99.56      | 1.63  | 97.21      | 0.82  |
| Trade Effluent    | 100.14     | 0.94  | 99.44      | 1.30  | 98.78      | 20.99 |

| Matrix        | Nitrite    |       | Nitrate    |       | Sulphate   |       |
|---------------|------------|-------|------------|-------|------------|-------|
|               | % Recovery | % RSD | % Recovery | % RSD | % Recovery | % RSD |
| Clean Process | 101.60     | 2.22  | 103.29     | 1.12  | 102.53     | 1.26  |
| Dirty Process | 103.66     | 1.33  | 100.78     | 0.95  | 101.03     | 0.87  |
| Recreational  | 99.69      | 1.16  | 101.43     | 0.86  | 100.84     | 0.71  |

## Integriion:

| Determinand | LOD (mg/l) | MRL (mg/l) | Low Standard |        | High Standard |        |
|-------------|------------|------------|--------------|--------|---------------|--------|
|             |            |            | % RSD        | % Bias | % RSD         | % Bias |
| Bromide     | 0.0344     | 0.20       | 1.62         | 10.14  | 2.38          | 3.61   |
| Sulphate    | 0.1421     | 0.20       | 1.48         | 8.60   | 2.37          | 0.67   |
| Chloride    | 0.0706     | 0.20       | 1.50         | 3.27   | 2.40          | 0.13   |
| Nitrite     | 0.0466     | 1.00       | 1.34         | 2.84   | 2.80          | 1.93   |
| Nitrate     | 0.1236     | 0.20       | 1.55         | 2.18   | 2.37          | 1.50   |

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| Matrix            | Sulphate  |      | Bromide   |      | Chloride  |      |
|-------------------|-----------|------|-----------|------|-----------|------|
|                   | %Recovery | %RSD | %Recovery | %RSD | %Recovery | %RSD |
| Surface Water     | 98.19     | 2.01 | 100.89    | 1.93 | 92.60     | 1.94 |
| Trade Effluent    | 97.77     | 1.77 | 102.16    | 1.67 | 99.64     | 1.71 |
| Final Effluent    | 98.19     | 1.97 | 101.42    | 1.88 | 90.87     | 1.94 |
| Untreated Sewage  | 98.50     | 1.68 | 97.81     | 1.57 | 91.20     | 1.64 |
| Ground water      | 99.58     | 1.88 | 100.60    | 2.06 | 94.83     | 1.97 |
| Prepared Leachate | 99.99     | 2.04 | 102.26    | 1.98 | 100.18    | 1.88 |
| Landfill Leachate | 98.01     | 1.93 | 96.82     | 2.21 | 95.28     | 2.05 |

| Matrix        | Nitrite   |      | Nitrate   |      | Sulphate  |      |
|---------------|-----------|------|-----------|------|-----------|------|
|               | %Recovery | %RSD | %Recovery | %RSD | %Recovery | %RSD |
| Clean Process | 100.57    | 2.57 | 100.26    | 2.25 | 100.98    | 2.36 |
| Dirty Process | 95.38     | 3.00 | 98.85     | 2.20 | 96.03     | 1.98 |
| Recreational  | 100.11    | 2.70 | 100.26    | 2.67 | 102.91    | 2.37 |

## 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     | 9.26                           |
| Sulphate    | 8.95                           |
| Chloride    | 13.68                          |
| Nitrite     | 9.08                           |
| Nitrate     | 6.54                           |

## 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.