

METHOD STATEMENT



Determinand:

Determination of Permanganate Index.

Matrix:

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

Principle of Method:

This method uses any suitable water bath and burette and Permanganate Index is defined as the amount of oxygen required for chemical oxidation of both inorganic and organic matter.

In hot acid solution chemical oxidation by potassium permanganate results in the reduction of the permanganate Mn (VII) ion to the manganous Mn (II) ion.

The oxidation reaction is as follows $MnO_4^- + 8H^+ + 5e \rightarrow Mn^{2+} + 4H_2O$.

The remaining, unreduced permanganate is determined by addition of excess sodium oxalate and back titration with potassium permanganate to a visual end point.

The permanganate / oxalate reaction is as follows $8MnO_4^- + 6H^+ + 5H_2C_2O_4 \rightarrow 2Mn^{2+} + 10CO_2 + 8H_2O$

Sampling and Sample Preparation:

Samples are normally collected in 500 ml PET bottles. Other size PET bottles are also suitable.

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 analysed within 21 days of sampling.

Interferences

Oxidising and reducing agents (nitrite, ferrous iron) may influence the permanganate index but are not classed as interferences.

High chloride concentrations may cause an enhanced result and are regarded as interferences. The levels found in potable or lightly polluted waters are unlikely to cause any significant interference.

Performance of Method:

Range of Application:

LOQ - 10 mg/l. for a 25 ml sample volume

Samples with PI concentration greater than 10 mg/l O₂ must be re-analysed following an appropriate dilution with deionised water.

Reporting Limit is 0.59 mg/l O₂

Limit of Quantification:

0.5802 mg/l O₂

Recoveries of Compounds, Bias and Uncertainty of measurement:

| Sample type | Mean sample result (µg/l) | Mean sample spike result (µg/l) | Spike recovery (%) | Bias (%) | % uncertainty |
|-------------|---------------------------|---------------------------------|--------------------|----------|---------------|
| Soft | 0.660 | 5.572 | 103.15 | - | - |
| Medium | 0.452 | 5.346 | 102.77 | - | - |
| Hard | 0.715 | 4.848 | 107.47 | - | - |
| Surface | 2.654 | 5.303 | 108.59 | - | - |
| Surface | 2.654 | 7.676 | 105.45 | - | - |
| Borehole | 0.347 | 5.205 | 102.01 | - | - |
| 2 mg/l Std | 1.967 | - | - | -1.65 | ± 13.86 |

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| Sample type | Mean sample result ($\mu\text{g/l}$) | Mean sample spike result ($\mu\text{g/l}$) | Spike recovery (%) | Bias (%) | % uncertainty |
|-------------|--|--|--------------------|----------|---------------|
| 8 mg/l Std | 7.685 | - | - | -3.94 | ± 6.96 |

References:

Methods for the Examination of Waters and Associated Materials, HMSO, 1983 ISBN 0 11 751960 X