



Method Summary

Determination of Anions in Aqueous Matrices using the Discrete Analysers

Scope and Range

This method is used to determine the concentration of the soluble anions sulphate, chloride, phosphate, nitrate, and nitrite. It is applicable to waste water, effluent, leachates, groundwater, and potable water. Nitrite in seawater is also validated. Because the method utilises spectrophotometry and all samples analysed must be clear and virtually colourless. Nitrate is calculated from total oxidised nitrogen on the analyser.

| Anion | Limit of Detection (mg/l) | Range (mg/l) |
|--------------|----------------------------------|---------------------|
| Chloride | 2 | 2 → 200 |
| Sulphate | 2 | 2 → 500 |
| Phosphate | 0.05 | 0.05 → 5.00 |
| Nitrate | 0.3 | 0.3 → 40.0 |
| Nitrite | 0.05 | 0.05 → 2.00 |

The upper limit of the range for each anion may be extended using auto-dilution on the analyser, up to a maximum of x120.

The calculated component, Total Inorganic Nitrogen, is also reported under this method code.

References

Chloride: EPA Methods 325.1 & 325.2

Sulphate: AWWA/APHA 20th Edition Method 4500E

Phosphate: AWWA/APHA 19th Edition Method 4500-P E

Total Oxidised Nitrogen: AWWA/APHA 19th Edition Method 4500-NO3-H

Nitrite: AWWA/APHA 19th Edition Method 4500-NO2-B

Principle

Preparation and Extraction:

4ml of sample is filtered through a 0.45µm inline filter into a Kone cup.

Analysis:

Chloride reacts with mercuric thiocyanate forming a mercuric chloride complex, displacing thiocyanate equivalent to the original chloride concentration. Released thiocyanate then reacts with iron (III) forming a red ferric thiocyanate complex, and measurement is made photometrically at 510 nm.

Sulphate ions react with barium chloride forming barium sulphate precipitate. Sodium chloride and gelatine are used to stabilise the precipitate, which causes turbidity of the sample, and is then measured on the Kone 30 analyser at 510nm or on the Aquakem 600/Prime at 520nm.



Method Summary

Determination of Anions in Aqueous Matrices using the Discrete Analysers

Orthophosphate ions react with a solution containing molybdic acid and ascorbic acid to form 12-molybdophosphoric acid, which is reduced to phosphomolybdenum blue incorporating antimony. The resulting colour is measured on the analyser at 660 nm.

Nitrate is reduced to nitrite by a hydrazine-copper reagent. The nitrite ion (including that already present) is converted into a red azo-dye by sulphanilamide and N-(1-naphthyl)-ethylenediamine dihydrochloride. When calibrated against a nitrate standard, the total oxides of nitrogen, TON, is expressed as NO_3^- . The colour is measured at 540nm. Nitrate is calculated from $[(\text{NO}_2^- + \text{NO}_3^-) \text{ expressed as } \text{NO}_3^-] - [\text{previously determined } \text{NO}_2^-] \times 62/46$.

Nitrite ions react with a reagent containing sulphanilamide and N-1(naphthyl)ethylenediamine in dilute phosphoric acid. An azo dye is formed and is measured photometrically at 510 nm.

Interferences

Turbid samples will cause errors in readings, and therefore samples must be filtered prior to analysis. Coloured samples may cause interferences, and it may be necessary to dilute samples to reduce the effect. Where this is not possible, samples should be analysed by ion chromatography as an alternative method.

Thiocyanate present in the sample will cause interference (false positive results) **when analysing** the chloride method, due to the use of thiocyanate in the colour reaction.

High levels of Nitrate may cause an interference to the sulphate analysis, and it may be necessary to dilute the samples to reduce the effect.