



Method Summary

Determination of Anions in soils using the Kone Spectrophotometric Analysers

Scope and Range

This method is used to determine the concentration of the soluble anions sulphate, chloride, phosphate, nitrate, and nitrite in soils and sludges. Because the method utilises spectrophotometry, all samples analysed must be clear and virtually colourless. Nitrate is calculated from total oxidised nitrogen on the analyser.

Anion	Limit of Detection (mg/kg unless specified)	Range (mg/kg unless specified)
Chloride as Cl	5	5 → 200
Sulphate as SO ₄	0.004 g/l	0.004 → 0.500 g/l
Phosphate as PO ₄	1.0	1.0 → 10.0
TON as NO ₃	1.0	1.0 → 80.0
Nitrate as NO ₃	1.0	1.0 → 80.0
Nitrite as NO ₂	0.05	0.05 → 4.00

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

Principle

Preparation and Extraction

20g of dried and crushed sample is weighed out, and 40ml of deionised water is added. The sample is then shaken before being filtered.

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.

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 NO₃⁻. The colour is measured at 540nm. Nitrate is calculated from [(NO₂⁻ + NO₃⁻) expressed as NO₃⁻] - [previously determined NO₂⁻] x 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.



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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.