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Method Summary

Determination of Anions in Solids using Ion Chromatography

Scope

This method is suitable for the determination of anions in solids (mg/kg).
The limits of detection are as set out in Table 1. These are based on 2:1 water: solids extraction.
The calibrated range for all the anions is between the LOD and 200mg/l. This equates to 400mg/kg in the sample.

Table 1 - Limits of Detection

Anion	Limit of Detection	Units
Fluoride	1.0	mg/kg
Chloride	2.0	mg/kg
Nitrite	0.2	mg/kg
Bromide	0.3	mg/kg
Nitrate	0.5	mg/kg
Phosphate	3.0	mg/kg
Soluble Sulphate	0.001 [2.0]	g/l [mg/kg]

Principle

Preparation and Extraction
Samples should be taken in a 1 litre plastic tub and kept between 1-8°C until ready for preparation.

19.5-20.5g of dried and crushed sample is shaken for 1hour at 200rpm with 40ml of deionised water. The samples are then filtered through a filter paper and passed to analysis prep.

1-1.5ml of each sample is then filtered through a 0.45μm filter into a labelled vial. Vials are then capped and racked up until they are transferred to the instrument autosampler.

Analysis
An aliquot of the sample is injected onto a liquid chromatography column, where the different anions are separated by ion chromatography and subsequently pass a conductivity detector. The conductivity reading is plotted against time to give a chromatogram for each sample. The peaks on the calibration standards chromatograms are assigned to each of the anions in the order in Table 1. The peaks on the samples are then automatically assigned to each of the anions that are present in the sample, based on the time previously recorded for the standards. All of the sample peaks are then checked for correct integration by the analyst. The integrated area under each sample peak is compared to values from the calibration standards to give a result for each peak identified.

Interferences

Very large concentrations of one peak near another peak may mask that peak in the chromatogram and prevent it from being detected. In this case, gradual dilutions are carried out to reduce the size of the interfering peak and still detect an analysable amount of the peak of interest. This does not occur at levels within the calibrated range.