SGS	LAKEFIELD – INDUSTRIES & ENVIRONMENT DETERMINATION OF ANIONS IN AQUEOUS, SOIL AND SLUDGE SAMPLES BY ION CHROMATOGRAPHY	Doc. No.	ME-CA-[ENV]IC-LAK-AN-001
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1. SCOPE

This method is used to determine anions (Chloride, Sulphate, Nitrite, Nitrate, Bromide) in aqueous, soil and sludge samples by using ion chromatography. It is appropriate for all aqueous sample types including drinking water, wastewater, ground water, surface water and leachates. Solid matrices include soil, sediment, biosolid, pulp sample types.

2. DERIVATION

This method is derived from EPA method 300.1 as well as the Quebec Ministry of Environment method MA300-Ions1.3 latest version.

3. PRINCIPAL OF THE METHOD

The analysis is performed on a Ion Chromatograph, using a 50*μ*L sample loop, an AG4A guard column, AS4A anion separator analytical column, a conductivity detector, and/or a variable wavelength photometer.

During ion chromatography (IC), a pump delivers a mobile-phase solvent at a uniform rate at pressures that typically range from 500 to 5000*psi*. IC involves the injection of a small amount of liquid sample into a moving stream of liquid termed the mobile phase (eluent). This passes through a column packed with a cross-linked polymer resin in a stationary phase. The separation of a mixture into its components depends on the different degrees of retention of each component in the column. The extent to which a component is retained in the column is determined by its partitioning coefficient between the liquid mobile phase and the stationary phases.

Ion-exchange chromatography is a type of HPLC based on the partitioning of ions between a polar liquid phase and a stationary phase with ion-exchange sites. Anions are separated on anion exchange resins, which contain positively charged functional groups such as $CH_2N^+(CH_3)_3$, a quaternary ammonium ion. Separation is based on the various degrees to which ions partition into the ion-exchange phase.

4. INTERFERENCES

- Samples that contain carbonate, hydroxide, acetate, or other weak organic acids may coelute with Cl, NO_2, and Br. When this occurs, dilutions may be required to try to separate the coeluting peaks. However, sample dilutions will alter the LOQ by the same factor as the dilution that has been made. If sample dilution does not separate the coeluting peaks, then the analyte in question should be analyzed by a different method.
- Samples that contain particulate matter larger than 0.45µm should be filtered through a 0.45µm syringe filter (note: do not filter regulated drinking water

samples). This will prevent blockages in the instrument and the column. Since the filters often contain chloride, the filters must be flushed with deionized water (DI) followed by sample (to displace the DI) if analyzing for Cl.

- The last peak on the AS4A column is typically SO_4. Depending on the condition of the column, this peak usually elutes between 5 and 8 min on the conductivity detector. Take care not to stop analysis prior to the last peak, as this can cause interference with the next analysis. Having said this, some samples may contain unknown analytes that can potentially have even later eluting peaks. If this occurs the analyst must reanalyze the sample which has the interference carryover peak.
- Dilute hydrochloric and nitric acids are used as a common acid wash throughout the laboratory for glassware. When preparing dilutions for Cl or NO_3(N) analyses, all glassware should be thoroughly rinsed with deionized water prior to use to ensure there is no residual Cl or NO_3(N) left after washing.
- Sulphate concentrations tend to have high unstable readings when the pH of the sample is < 3. Therefore, the sample should be neutralized prior to analyzing. This can be achieved by diluting the samples with eluent.

5. SAMPLE REQUIREMENTS

Sampling information and concerns:	Representative sample must be taken.
Containers to be used:	Plastic or glass
Sample size:	Optimum aqueous sample volume 60mL Minimum aqueous sample volume 5mL *except drinking water samples. Minimum sample volume for drinking water samples is 60mL.* Optimum solid or soil sample size 25g Minimum solid or soil sample size 10g
Preservation required:	None
Temperature requirements for storage:	Aqueous and Reg. 153 soils: refrigerate at 5°C +/-3°C All other soil or solid samples: ambient
pH requirements:	pH 4.0 – pH 10.0 (optimal 7.0)
Pre-treatment:	None
Holding time (from time of collection to start of analysis):	**Cl, Br, SO_4:** 28 days **NO_2, NO_3:** 7 days (48 hours for samples falling under Quebec protocols) Solids: Quebec – 6 months to extraction, analyze within 28 days except for NO2 and NO3 – analyze within 48 hrs after extraction

	O. Reg 153 samples for Cl: – indefinite if dried, 30 days as received
Disposal of samples:	Refer to WI-CA-[ENV]AD-LAK-AD-014

6. REFERENCES

U.S. Environmental Protection Agency 1997; Method 300.1 Determination of Inorganic Anions in Drinking Water by Ion Chromatography.

Dionex Corporation 1989. Determination of anions using the HPIC – AS4A Separator; Dionex application note *in* Theoretical aspects of Dionex chromatography, Dionex Corporation Oakville.

Protocol for Analytical Methods Used in the Assessment of Properties under Part XV.1 of the Environmental Protection Act and Excess Soil Quality. Laboratory Services Branch, Ministry of the Environment, Conservation and Parks as amended.

CENTRE D'EXPERTISE EN ANALYSE ENVIRONNEMENTALE DU QUÉBEC. Détermination des anions : méthode par chromatographie ionique MA. 300 – Ions 1.3, révision 6, ministère de l'Environnement et de la Lutte contre les changements climatiques, 2020, 14 p.

Practices for the Collection and Handling of Drinking Water Samples Version 2.0, April 1, 2009; Laboratory Services Branch Ministry of the Environment; PIBS 4464e01