DETERMINATION OF INORGANIC ANIONS IN WATER SAMPLES

INTRODUCTION

The method allows determination of inorganic anions: chloride, nitrite, sulfate, nitrate, fluoride, and phosphate ions in samples of natural, potable and waste water.

MEASURING METHOD

The capillary electrophoresis method for determination of inorganic anions' concentrations is based on differential migration and separation of anions in the electric field due to different electrophoretic mobility. Identification and quantitative determination of the analyzed anions is performed by indirect detection by measuring the UV absorption.

CONCENTRATION RANGES

Ranges of measurable concentrations for analyzed anions are presented in the table.

Anions	Samples	Measurement range, mg/l
Nitrite	Potable, natural and waste water	0.2–50
Nitrate		0.2–50
Hydrophosphate		0.2–50
Sulfate		0.5–200
Fluoride		0.1–25
Chloride		0.5–200

If the concentration of an anion in an analyzed sample exceeds the upper limit of the measurement range, it is allowed to dilute the sample so that the concentration would be in the range from 5 to 50 mg/l (or for fluoride from 2.5 to 25 mg/l)

Soluble carbonates with concentration ratio 100:1 do not influence determination of phosphates. When the ratio is 1000:1 they do not influence determination of other analyzed anions.

Monobasic organic acids and neutral organic compounds do not influence the determination of analyzed anions. Presence of dibasic organic acids (up to 10 mg/l) and perchlorate and formate anions (up to 3 mg/l) is acceptable.

EQUIPMENT AND REAGENTS

The following equipment and reagents are used in measurements:

- The CAPEL® Capillary Electrophoresis System with high-voltage negative polarity;
- Reference anion standard solutions: Cl^- (1 mg/ml), NO_2^- (1 mg/ml), SO_4^{2-} (1 mg/ml), NO_3^- (1 mg/ml), F^- (1 mg/ml), HPO_4^{2-} (0.5 mg/ml);
- Distilled water;
- Chromium (VI) oxide, Analytical Grade;
- Cetyl-trimethyl-ammonium bromide, Reagent Grade;
- Diethanolamine, Reagent Grade;
- Sodium hydroxide, Ultra Pure Grade;
- Hydrochloric acid, Ultra Pure Grade;
- Acetic acid, Ultra Pure Grade
- Ammonium aqueous, Ultra Pure Grade
- Sodium EDTA (Trilon® B), Analytical Grade.

Data acquisition, collection, processing, and output are performed using a personal computer running under WINDOWS® 98/ME/NT/2000/XP operating system with installed Chrom&Spec® software package for acquisition and processing of chromatography data.

PREOPERATIONAL PROCEDURES

Preoperational procedures include: sampling and sample preparation, capillary conditioning, preparation of auxiliary and calibration solutions, and calibration of the CAPEL® Capillary Electrophoresis System.

Samples of natural, potable or waste water should be collected in compliance with ISO 5667 Standard.

Volume of the sample should be at least 100 ml.

The sample should be filtered through a “blue ribbon” dry filter (Whatman No 44 or S&S No 589 Blue Ribbon); first portions of the filtrate must be discarded. The sample must be analyzed within 24 hours. The system is calibrated by measuring signals of calibration solutions. Stability of the calibration characteristics is checked directly before sample measurement by recording an electrophoregram of one of the calibration mixtures.

MEASUREMENT PROCEDURE

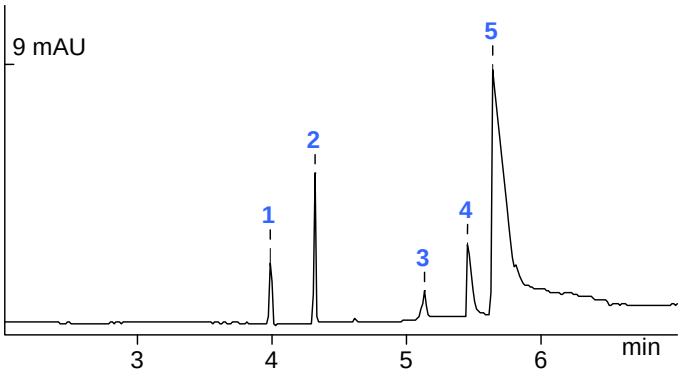
Pre-testing should be performed prior to main measurement: it may be necessary to adjust the sample pH and to eliminate interfering cations and anions.
No less than two specimens should be analyzed for each sample queued. If the measured chloride, nitrite, sulfate, nitrate and phosphate concentrations exceed 50 mg/l or concentration of fluoride exceeds 25 mg/l, it is necessary to pre-dilute the sample with distilled water.

DATA PROCESSING

Chrom&Spec® software outputs a report of concentrations (in mg/l) of analyzed anions in the solution prepared for analysis.

EXAMPLE OF REAL ANALYSIS

Sample: purified foul water (diluted 1:9)
Buffer: 5 mmol chromate,
20 mmol DEA,
1.65 mmol CTAB
Capillary: L_{EFF}/L_{TOTAL} 50/60 cm,
ID 75 µm
Injection: 300 mbar*s
Voltage: –17 kV
Detection: 254 nm, indirect



Measurement results:
1 – chloride (2.7 mg/l)
2 – sulfate (4.2 mg/l)
3 – fluoride (1.2 mg/l)
4 – phosphate (3.3 mg/l)
5 – hydrocarbonate