

DETERMINATION OF INORGANIC ANIONS IN WATER SAMPLES**INTRODUCTION**

The method allows determination of mass concentrations 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 evaluation of inorganic anion mass concentration is based on the migration and separation of anions in the electric field due to different electrophoretic mobility. Identification and quantitative evaluation of the analyzed anions is performed indirectly by detecting use UV absorption.

**CONCENTRATION RANGES**

Ranges of measurable concentrations for analyzed anions are given in Table 1.

Table 1. Measurement ranges for anions

Anions	Samples	Measurement range, mg/l
Nitrite	Potable, natural and waste water	0.5–50
Nitrate	Potable, natural and waste water	0.5–50
Hydrophosphate	Potable, natural and waste water	0.5–50
Sulfate	Potable, natural and waste water	0.5–50
Fluoride	Potable, natural and waste water	0.25–25
Chloride	Potable, natural and waste water	0.5–50

If the mass 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 match the range from 5 to 50 mg/l (or, for fluoride from 2.5 to 25 mg/l)

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

Monobasic organic acids and neutral organic compounds do not prevent from determination of analyzed anions. It is acceptable when up to 10 mg/l of dibasic organic acids and up to 3 mg/ml perchlorate and formate anions are present.

EQUIPMENT AND REAGENTS

The following equipment and reagents are used in measurements:

- The CAPEL Capillary Electrophoresis System with high-voltage negative polarity;
- Reference anion content 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
- Trilon B, Analytical Grade.

Data acquisition, collection, processing and output are performed using a personal computer running under WINDOWS® 95/98/ME/NT/2000 operating system with installed Chrom&Spec® for WINDOWS® software package for acquisition and processing of chromatography data.

PREOPERATIONAL PROCEDURES

Preoperational procedures include: selection and preparation of samples, preparation of the capillary to operation, 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 taken 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 electropherogram 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.

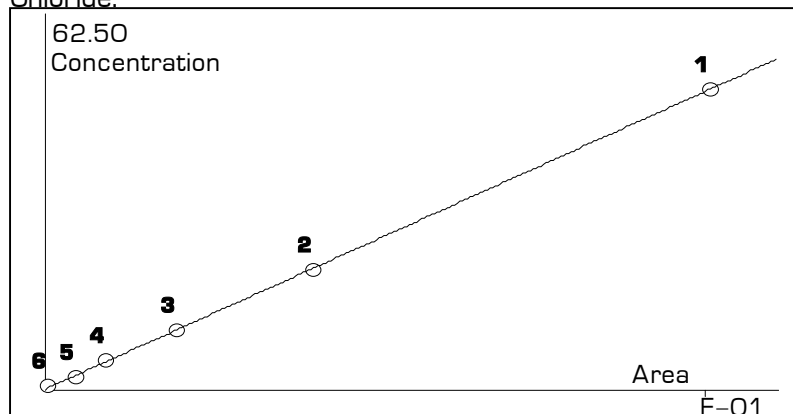
DATA PROCESSING

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

EXAMPLE OF REAL ANALYSIS

Calibration curve:

Chloride:



Component	Equation	RSD, %	Correlation, r
Nitrite	Q=355.5A	2.1	0.999
Nitrate	Q=619.9A	0.7	0.999
Hydrophosphate	Q=477.2A	2.3	0.999
Sulfate	Q=489.9A	1.2	0.999
Fluoride	Q=146.1A	1.2	0.999
Chloride	Q=494.9A	1.7	0.999

Sample: water sample from the Sunozhka River.
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 (6.8 mg/l)
- 2** – nitrite (1.6 mg/l)
- 3** – sulfate (8.3 mg/l)
- 4** – nitrate (6.9 mg/l)

