DETERMINATION OF INORGANIC ANIONS IN WATER SAMPLES ACCORDING TO EPA 6500 AND ASTM D 6508-00 TEST METHODS

EPA 6500
ASTM D 6508-00

INTRODUCTION

The method allows determination of inorganic anions: fluoride, bromide, chloride, nitrite, nitrate, sulfate, and *ortho*-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 using indirect detection by measuring the UV absorption.

CONCENTRATION RANGES

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

Anions	Linear range, mg/l	Measurement range, mg/l
Nitrite	5–50	0.1–50
Nitrate	5–50	0.1–50
Ortho-phosphate	5–50	0.1–200
Sulfate	5–50	0.1–1000
Fluoride	2.5–25	0.1–25
Bromide	5–50	0.1–50
Chloride	5–50	0.1–1000

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)

EQUIPMENT AND REAGENTS

The following equipment and reagents are used in measurements:

- The CAPEL® Capillary Electrophoresis System with high-voltage negative polarity
- Certified 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), PO_4^{3-} (0.5 mg/ml)
- Distilled water
- Sodium chromate tetrahydrate, Analytical Grade
- CHES (2-[*N*-cyclohexylamino]-ethane sulfonic acid), Analytical Grade
- Tetradecyltrimethyl ammonium bromide (TTABr), Analytical Grade, or 100 mM TTAOH solution
- Sodium hydroxide, Analytical Grade
- Calcium gluconate, Analytical Grade
- Anion exchange cartridge, hydroxyde form

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 EPA sampling guidelines (ASTM Practice D 3370).
The sample should be filtered through a prerinsed 0.2µm aqueous compatible membrane filter; 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.

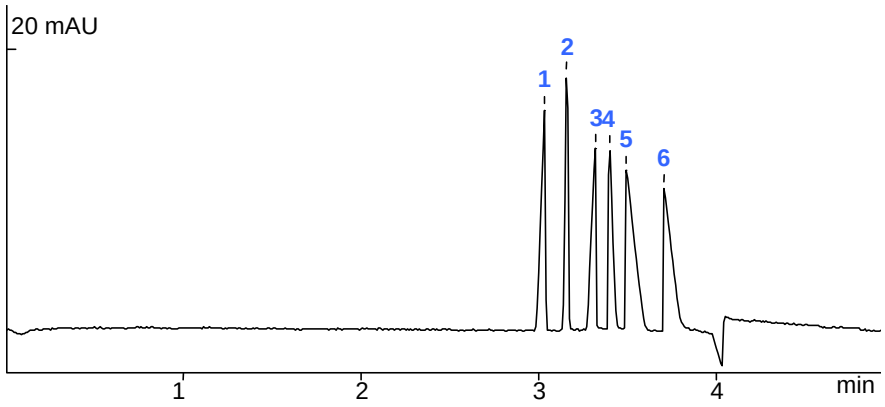
DATA PROCESSING

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

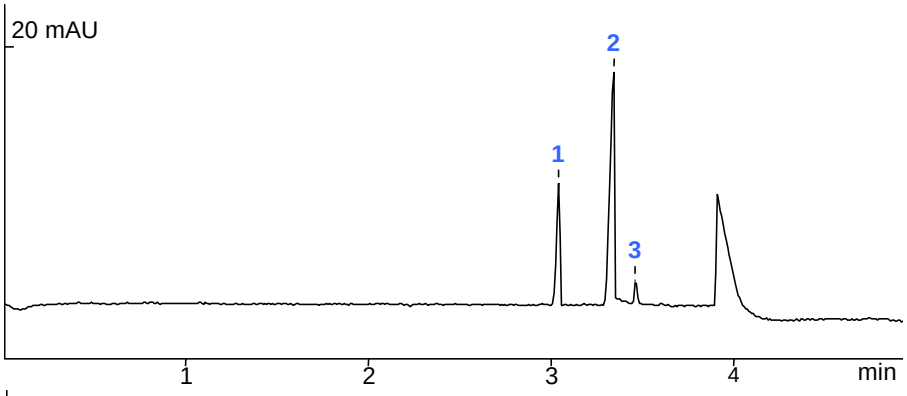
EXAMPLE OF REAL ANALYSIS

Buffer: 4.7 mmol chromate, 4 mmol TTAOH, 10 mmol CHES, 0.1 mmol Ca gluconate
Capillary: L_{EFF}/L_{TOTAL} 50/60 cm, ID 75 µm
Injection: 300 mbar*s
Voltage: –15 kV
Detection: 254 nm, indirect
Temperature: RT

Sample: test solution
1 – chloride (20 mg/l)
2 – nitrite (20 mg/l)
3 – sulfate (20 mg/l)
4 – nitrate (20 mg/l)
5 – fluoride (10 mg/l)
6 – phosphate (20 mg/l)



Sample: tap water
Measurement results:
1 – chloride (7.78 mg/l)
2 – sulfate (26.3 mg/l)
3 – nitrate (1.66 mg/l)



Sample: foul water (diluted 1:4)
Measurement results:
1 – chloride (532 mg/l)
2 – sulfate (23.1 mg/l)
3 – phosphate (6.8 mg/l)