
**Fertilizers and soil conditioners —
Determination of microamounts of
inorganic anions in fertilizers by ion
chromatography**

*Engrais et amendements — Détermination des microquantités
d'anions inorganiques dans les fertilisants par chromatographie
d'échange d'ions*

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ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Introduction

Microamounts of inorganic anions such as fluoride, chloride, bromide, iodide, nitrite and thiocyanate are usually contained in fertilizers. Excessive amounts of such inorganic anions can not only impede the growth of crops, but can also cause harmful effects on the arable land and the environment. Current standards around the world mostly focus on a few specific anions. This method, which uses ion chromatography, provides a high-throughput screening and rapid determination pathway targeting microamounts of six anions as an alternative to traditional chemical analysis.

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Fertilizers and soil conditioners — Determination of microamounts of inorganic anions in fertilizers by ion chromatography

1 Scope

This document specifies the ion chromatography method for the determination of microamounts of water soluble inorganic anions, such as fluoride (F⁻), chloride (Cl⁻), bromide (Br⁻), iodide (I⁻), nitrite (NO₂⁻) and thiocyanate (SCN⁻), in fertilizers.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 8157, *Fertilizers and soil conditioners — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 8157 apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

4 Principle

Water soluble inorganic anions are extracted by sonication, centrifugation and purification using a C18 column and a Ba-ion column (where applicable). During pre-treatment, the anions are filtered by a 0,22 µm aqueous filter. They are determined by ion chromatography. Anions are separated based on their affinity for the exchange sites on the analytical column. The suppressor device reduces the background conductivity of the eluent to a low level by replacing the cations with the hydrogen ions, thereby converting the anions in the fertilizer sample to their corresponding acids with higher conductivity. The separated anions (in their acid forms) are measured by an electrical-conductivity detector. Anions are identified based on their retention times compared to known standards. Quantitation is accomplished by measuring the peak height or area compared to a calibration curve generated from known standards.

5 Reagents

WARNING — Acetone and methanol are flammable and toxic. Refer to the applicable safety data sheet (SDS). The related operations shall be performed in the fume hood. This document does not point out all possible safety problems; therefore, the user shall bear the responsibility to take proper safety and health measures.

Analytical grade reagent (A.R.) chemicals shall be used in all tests, unless otherwise indicated. The purity of water used throughout shall be understood to mean reagent water with electrical resistivity ≥18,2 MΩ·cm. In the list below, G.R. refers to guarantee reagent.

5.1 Ultrapure water, HPLC grade water with electrical resistivity $\geq 18,2 \text{ M}\Omega\cdot\text{cm}$.

5.2 Potassium hydroxide (G.R.).

5.3 Sodium carbonate (G.R.).

5.4 Sodium bicarbonate (G.R.).

5.5 Acetone, HPLC grade.

5.6 Methanol, HPLC grade.

NOTE Methanol is be used for the activation of SPE-C18 column (6.8).

5.7 Potassium hydroxide eluent. Dissolve 5,611 g potassium hydroxide (0,1 mol, 5.2) in water (5.1). Dilute this solution to 1 l with water (5.1) and mix thoroughly. The potassium hydroxide solution can also be prepared by automated eluent generation system (OH⁻ type). The potassium hydroxide eluent is used for gradient chromatography conditions.

5.8 Carbonate eluent. Dissolve 0,530 g sodium carbonate (5,0 mmol) (5.3) and 0,168 g sodium bicarbonate (2,0 mmol) (5.4) in water (5.1), and add 40 ml of acetone (5.5) to the aqueous solution. Dilute to 1 l with water (5.1) and mix thoroughly by ultrasonic treatment for 10 min. The carbonate eluent is used for isocratic chromatography conditions.

5.9 Stock solutions

5.9.1 Fluoride stock solution (1 000 mg/l). [ISO 20702:2017](https://standards.iteh.ai/catalog/standards/sist/9a0bed21-9f63-4cb4-aa56-15599e4e4105/iso-20702-2017)

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Dissolve 2,210 0 g NaF (sodium fluoride, A.R., pre-treated at 105 °C for 2 h) into water (5.1). Transfer the aqueous solution into a 1 000 ml volumetric flask, dilute it to 1 l with water (5.1) and mix thoroughly. Keep the fluoride stock solution in a polyethylene bottle for storage.

5.9.2 Chloride stock solution (1 000 mg/l).

Dissolve 1,648 0 g NaCl (sodium chloride, A.R., pre-treated at 105 °C for 2 h) in water (5.1). Transfer the aqueous solution into a 1 000 ml volumetric flask, dilute it to 1 l with water (5.1) and mix thoroughly.

5.9.3 Bromide stock solution (1 000 mg/l).

Dissolve 1,490 0 g KBr (potassium bromide, A.R., pre-treated at 105 °C for 2 h) in water (5.1). Transfer the aqueous solution into a 1 000 ml volumetric flask, dilute it to 1 l with water (5.1) and mix thoroughly. Keep the bromide stock solution in an amber glass bottle for storage.

5.9.4 Iodide stock solution (1 000 mg/l).

Dissolve 1,308 0 g KI (potassium iodide, A.R., pre-treated at 105 °C for 2 h) in water (5.1). Transfer the aqueous solution into a 1 000 ml volumetric flask, dilute it to 1 l with water (5.1) and mix thoroughly. Keep the iodide stock solution in an amber glass bottle for storage.

5.9.5 Nitrite stock solution (1 000 mg/l).

Dissolve 1,489 0 g NaNO₂ (sodium nitrite, A.R., pre-dried in a desiccator for approximately 24 h) in water (5.1). Transfer the aqueous solution into a 1 000 ml volumetric flask, dilute it to 1 l with water (5.1) and mix thoroughly.

Nitrite is easily oxidized, especially in the presence of moisture, thus only fresh reagents are to be used. Place approximately 2 g of NaNO_2 in a 100 ml beaker and dry to constant weight (approximately 24 h) in a desiccator. Preservation by refrigeration at 4 °C is required for nitrite.

5.9.6 Thiocyanate stock solution (1 000 mg/l).

Dissolve 1,673 0 g KSCN (potassium thiocyanate, pre-treated at 105 °C for 2 h) into water (5.1). Transfer the aqueous solution into a 1 000 ml volumetric flask, dilute to 1 l with water (5.1) and mix thoroughly.

5.10 Anion standard working solutions.

Prepare anion standard working solutions (including F^- , Cl^- , Br^- , I^- , NO_2^- and SCN^-) in volumetric flasks according to the concentration ranges listed in Table 1. Alternatively, certificated standard solutions of single or mixed anions may also be used for the preparation of anion working solutions.

The concentration ranges for standard working solutions may be adjusted according to the levels expected in the samples.

Preservation by refrigeration at 4 °C is required for solutions containing nitrite.

Table 1 — Preparation of anion standard working solutions

Working solution	F^- mg/l	Cl^- mg/l	Br^- mg/l	I^- mg/l	NO_2^- mg/l	SCN^- mg/l
Blank	0	0	0	0	0	0
Standard 1	0,2	0,2	0,2	0,2	0,2	0,2
Standard 2	0,5	0,5	0,5	0,5	0,5	0,5
Standard 3	1,0	1,0	1,0	1,0	1,0	1,0
Standard 4	5,0	5,0	5,0	5,0	5,0	5,0
Standard 5	20,0	20,0	20,0	20,0	20,0	20,0

6 Apparatus and materials

6.1 Ion chromatograph, with electrical-conductivity detector.

6.2 Ultrasonic cleaner.

6.3 Centrifuge, with a rotational speed of 4 000 r/min.

NOTE Relative centrifugal force can be calculated as 0,000 001 18 times the rotational radius (in mm) times the square of the revolutions per minute (r/min).

6.4 High speed pulverizer.

6.5 Analytical balance, with a sensitivity of 0,1 mg.

6.6 Syringe-driven filter, with an aqueous filter membrane with 0,22 μm pores.

6.7 Ba-ion filter column, 1 ml.

NOTE The Ba-ion filter column (for example, a Ba-Polystyrene resin column) is used to eliminate excess amounts of sulfate and phosphate from the sample which can serve as potential interference.

6.8 SPE-C18 column, 1 ml.

NOTE The SPE-C18 column is used to eliminate interfering amounts of organic compounds from the sample which can serve as potential interference.

6.9 Ag/H filter column, 1 ml.

NOTE Since chloride and nitrite elute very closely together, they are potential interferences for each other. The Ag/H filter column is used to eliminate excess amounts of chloride to more accurately measure nitrite.

7 Test procedure

7.1 General

Two replicate experiments shall be done for the determination.

7.2 Preparation of test sample

Prepare a test portion by reducing the fertilizer sample to 100 g. Grind the sample until it passes through a sieve of aperture size 0,5 mm and mix until homogenous. Place in a clean and dry bottle with a lid.

7.3 Activation of the filter columns

7.3.1 Activation of the Ba-ion filter column

Flush the Ba-ion filter column with 10 ml water (5.1) before use, keeping the water outflow velocity less than 3 ml/min. The column shall be kept in stand-by mode after activation.

7.3.2 Activation of the SPE-C18 column

First, flush the SPE-C18 column with 5 ml methanol, keeping the methanol outflow velocity less than 3 ml/min. Then, flush the column with 15 ml water (5.1), keeping the water outflow velocity less than 3 ml/min. Keep the column flat for 20 min while it activates. The column shall be kept in stand-by mode after activation.

7.3.3 Activation of the Ag/H filter column

Flush the Ag/H filter column with 10 ml water (5.1) before use; keep the water outflow velocity less than 3 ml/min. The column shall be kept on stand-by after activation.

7.4 Preparation of the test solution

Accurately weigh to 0,000 2 g an appropriate amount (1 g to 2 g) of the ground test portion into a 100 ml volumetric flask and add water (5.1) to near 100 ml mark.

Choose a sample weight that ensures the concentrations of ions of interest lie within the following ranges: F⁻ (10 mg/kg to 2 000 mg/kg), Cl⁻ (10 mg/kg to 20 000 mg/kg), Br⁻ (20 mg/kg to 2 000 mg/kg), I⁻ (20 mg/kg to 2 000 mg/kg), NO₂⁻ (10 mg/kg to 2 000 mg/kg), SCN⁻ (40 mg/kg to 2 000 mg/kg).

Dissolve using an ultrasonic bath for 30 min, and then allow the contents of the volumetric flask to reach the room temperature.

Dilute to volume with water (5.1), and mix thoroughly by shaking.

Centrifuge a 10 ml portion of the solution at a rotational speed of 4 000 r/min for 5 min to 10 min.

Remove 5 ml of supernatant from the centrifuge tube and filter with syringe-driven filter (6.6).

Filter the solution with tandem filter columns of SPE-C18 column (activated), Ba-ion filter column (activated) and Ag/H filter column (activated) respectively, keeping the solution outflow velocity less than 3 ml/min.

Discard the first 3 ml of filtrate and keep the remaining 1 ml to 2 ml solution for testing.

NOTE The SPE-C18 column is used to eliminate organic compounds as interferences in the test solution. The Ba-ion filter column is used to eliminate excess amounts of sulfate and phosphate in the test solution. The Ag/H filter column is used to eliminate excess chloride to accurately measure nitrite. The use of these filter columns is dependent upon the sample being tested.

7.5 Ion chromatography condition A (Gradient chromatography condition)

7.5.1 Chromatographic column: hydroxide selective, gradient elution compatible, high capacity anion exchange column.

7.5.2 Column temperature: 30 °C.

7.5.3 Suppressor device: membrane-based self-regenerating anion suppressor device. An equivalent suppressor device may be used provided that comparable method detection limits can also be achieved and that adequate baseline stability is attained.

7.5.4 Detector: electrical-conductivity detector with cell temperature held at 35 °C.

7.5.5 Eluent: potassium hydroxide eluent (5.6); gradient elution schedule listed in Table 2.

Table 2 — Gradient elution schedule

Time min	OH ⁻ concentration (mmol·l ⁻¹)
0,00	12
13,00	12
14,00	80
46,00	80
47,00	12
54,00	12

7.5.6 Eluent flow rate: 1,0 ml/min.

7.5.7 Injection volume: 25 µl. The injection volume can be adjusted according to the concentrations of ions of interest in the test solution.

Use the same size of sample loop for all standards, samples, and fortified samples. If removal of the background matrix is not around 100 %, increasing the sample loop size will increase the overall mass loading to the guard and the analytical columns, potentially saturating a column's exchange capacity and shifting retention times for the unknowns.

7.6 Ion chromatography condition B (Isocratic chromatography condition)

7.6.1 Chromatographic column: carbonate selective, high capacity anion exchange column.

7.6.2 Column temperature: 40 °C.

7.6.3 Suppressor device: self-regenerating anion suppressor device. An equivalent suppressor device may be used provided that comparable method detection limits can also be achieved and that adequate baseline stability is attained.

7.6.4 Detector: electrical-conductivity detector with cell temperature held at 35 °C.

7.6.5 Eluent: carbonate eluent (5.7), isocratic elution.

7.6.6 Eluent flow rate: 0,7 ml/min.

7.6.7 Injection volume: 20 µl. The injection volume can be adjusted according to the concentrations of ions of interest in the test solution.

7.7 Determination of standard working solutions and sample test solutions

7.7.1 Determination of standard working solutions

Use F^- , Cl^- , Br^- , I^- , NO_2^- and SCN^- standard solutions (5.10) to prepare the mixed-anion standard working solutions given in Table 1. Plot the standard curves using the concentration of inorganic anions, corresponding to the peak heights/areas obtained in the test.

Filter the mixed-anion standard working solutions with syringe-driven filter and tandem filter columns of SPE-C18 column (activated) and Ba-ion filter column (activated), if necessary.

It is recommended to run a minimum of two standard curves for each set of unknowns, preferably at the beginning and end of each set of unknowns. Additional standard curves are recommended for large (>20) sets of unknowns.

7.7.2 Determination of sample test solutions

Test a blank solution and the sample test solutions under the same conditions as the standards. Use the retention times to identify the inorganic anions, and derive the concentrations of inorganic anions in the test solutions from the standard curves. Typical ion chromatograms are shown in Annex B (for gradient chromatography conditions) and Annex C (for isocratic chromatography conditions).

The blank solution is prepared in the same manner as the test solutions, except for adding any test samples.

If the response value (peak height/area) of any anion in a test solution exceeds the linear calibration range of a standard solution, appropriate dilutions should be prepared.

For the determination of anions in liquid fertilizer, the instrumental detection limit (IDL, D.3) should be verified before the analysis of the sample. For the determination of anions in liquid fertilizer containing NO_3^- , the proposed method (7.5 and 7.6) shall be carried on very carefully, and anions standard working solutions (5.10) shall be injected to identify the retention time of Br^- .

8 Calculation and expression of results

8.1 General

The mass fraction of fluoride (F⁻), chloride (Cl⁻), bromide (Br⁻), iodide (I⁻), nitrite (NO₂⁻) and thiocyanate (SCN⁻), w , in the unit of mg/kg, is calculated using [Formula \(1\)](#):

$$w = \frac{(\rho - \rho_0) \times V \times f}{m} \quad (1)$$

where

ρ is the mass concentration of fluoride (F⁻), chloride (Cl⁻), bromide (Br⁻), iodide (I⁻), nitrite (NO₂⁻) and thiocyanate (SCN⁻), expressed in milligrams per litre (mg/l) of the test solutions;

ρ_0 is the mass concentration of fluoride (F⁻), chloride (Cl⁻), bromide (Br⁻), iodide (I⁻), nitrite (NO₂⁻) and thiocyanate (SCN⁻), expressed in milligrams per litre (mg/l) of the blank solution;

V is the volume of the test/blank solutions, expressed in millilitres (ml);

f is the dilution factor of the test solutions;

m is the mass of the test portion, expressed in grams (g).

The determination result is the arithmetic average of two parallel determination results, and shall be rounded off to three significant figures.

8.2 Precision

8.2.1 Ring test <https://standards.iteh.ai/catalog/standards/sist/9a0bed21-9f63-4cb4-aa56-15f6e8983992/iso-20702-2017>

Details of ring test on the precision of the method are summarized in [Annex A](#).

8.2.2 Repeatability, r

Anions	F ⁻	Cl ⁻	Br ⁻	I ⁻	NO ₂ ⁻	SCN ⁻
Repeatability limit, r , mg/kg	10,424	$0,015 \bar{X} + 9,685$	$0,011 \bar{X} + 1,336$	$0,050 \bar{X} + 2,310$	$0,339 \bar{X} + 0,882$	$0,140 \bar{X} + 0,802$

8.2.3 Reproducibility, R

Anions	F ⁻	Cl ⁻	Br ⁻	I ⁻	NO ₂ ⁻	SCN ⁻
Reproducibility limit, R , mg/kg	$0,484 \bar{X} + 0,889$	$0,137 \bar{X} + 16,564$	$0,056 \bar{X} + 29,554$	$0,255 \bar{X} + 11,595$	20,020	$0,168 \bar{X} + 11,155$

9 Test report

The test report contains the following information, at a minimum:

- all information necessary for the complete identification of the sample;
- test method used with reference to this document, i.e. ISO 20702:2017;
- test results obtained (signal-to-noise ratio plots per unknown, instrument detection limits and percent recoveries are suggested to be presented, if necessary. An explanation of these statistical indices is provided in [Annex D](#));
- date of sampling and sampling procedure (if known);

- e) date of analysis completion;
- f) whether or not the requirement of the repeatability limit is fulfilled;
- g) all operating details not specified in this document, or regarded as optional, together with details of any incidents occurred when performing the method, which might have influenced the test results.

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Annex A (informative)

Ring test report — Report of international laboratories ring test

A.1 Overview

The international laboratories ring test of this document has been accomplished during March 2017 to May 2017. Fifteen laboratories participated in the two parallel tests on four test samples. This international ring test was conducted by Shanghai Research Institute of Chemical Industry, Co. Ltd. and Shanghai Entry-Exit Inspection and Quarantine Bureau, P. R. China. The statistician analysis and final report was prepared by Shanghai Research Institute of Chemical Industry, Co. Ltd.

The following are the 15 laboratories that participated in the two parallel tests on four samples:

- Central Institute for Supervising and Testing in Agriculture, National Reference Laboratory, Division of NRL Brno, Czech Republic;
- CF Industries Technical Service Laboratory, USA;
- China National Supervision and Testing Centre of Fine Chemicals;
- Hunan Testing Institute of Product and Commodity Supervision, P. R. China;
- Institution of Supervision and Inspection Product Quality of Guizhou Province, P. R. China;
- Metrohm China Limited;
- Ningbo Entry-Exit Inspection and Quarantine Bureau, P. R. China;
- Office of Indiana State Chemist, USA;
- Product Quality Supervising and Inspecting Centre of Taizhou city, P. R. China;
- Shaanxi Entry-Exit Inspection and Quarantine Bureau, P. R. China;
- Shandong Institute for Product Quality Inspection, P. R. China;
- Shanghai Research Institute of Chemical Industry, Co. Ltd., P. R. China;
- Shanghai Entry-Exit Inspection and Quarantine Bureau, P. R. China;
- Thermo Fisher Scientific Inc.;
- Yunnan Chemical Product Quality Supervision & Inspection Station, P. R. China.

NOTE Participating laboratories are listed in alphabetic order, which has no relation to the sequences listed in the tables below.

The test method described in this document was adopted for the determination of fluoride, chloride, bromide, iodide, nitrite and thiocyanate contents in the fertilizer samples.

Four different types of fertilizer samples were used during the ring test, each with several mean levels. The test samples were: sample A (ammonium phosphate), sample B (nitro-compound fertilizer), sample C (complex fertilizer) and sample D (potassium sulfate). The six anion contents to be determined and involved in the statistics in the four fertilizer samples generally lie in the range of 10 mg/kg to 1 700 mg/kg, and the standard working solutions in the standard were designed to cover the range of 0 mg/kg to 2 000 mg/kg.