Annex D.11

Inhibitors — Determination of the chloride content by ion chromatography and conductivity detection (IC-CD)

Draft method

1 Scope

This document specifies a method for the determination of the chloride content in inhibitors by ion chromatography.

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.

EN 1482-2, *Fertilizers and liming materials — Sampling and sample preparation — Part 2: Sample preparation*

EN 12944-1, *Fertilizers and liming materials— Vocabulary — Part 1: General terms*

EN 12944-2, *Fertilizers and liming materials— Vocabulary — Part 2: Terms relating to fertilizers*

EN 12944-3, *Fertilizers and liming materials — Vocabulary — Part 3: Terms relating to liming materials*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1, EN 12944-2 and EN 12944-3 apply.

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

— ISO Online browsing platform: available at https://www.iso.org/obp/

— IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The inhibitor sample is diluted in water. The content of chloride is determined in diluted aqueous solution by ion chromatography using a conductivity detector.

5 Reagents

All reagents shall be of recognized analytical grade and shall have negligible concentration of the element to be determined if compared to the lowest concentration of that element in the sample solution.

5.1 Water, distilled or demineralized.

The electrical conductivity shall at least be < 0,2 mS/m (at 25 °C).

5.2 Chloride standard solution, $\rho(Cl^-)$ = 1 000 mg/l.

5.3 Quality control sample Chloride, $\rho(Cl^-)$ = 1,0 mg/l prepared from a certified solution or a certified solid standard substance.

5.4 Eluent for IC-CD, as advised by the manufacturer to be used for determining chloride.

The eluent advised by the manufacturer of the instrument depends on the instrument used, hence different eluents may be used.

6 Apparatus and equipment

Usual laboratory glassware and equipment, in particular, the following.

6.1 Ion chromatography device with conductivity detector, and optional an auto sampler and column oven.

For lower noise and background level of detection, a suppressed conductivity detector may be used. For column selection, instrumental conditions and additional information on how to operate follow the instructions of the manufacturer.

6.2 Analytical balance, capable for weighing to an accuracy of accuracy 0,1 mg.

6.3 Syringe filter, for aqueous solutions; pore size 0,45 µm.

6.4 Graduated flask, capacity 100 ml.

6.5 Pipettes.

6.6 Usual laboratory glassware.

7 Sampling

Sampling is not part of the method specified in this document. A recommended sampling method for quality control purposes is given in EN 1482-1.

The sample preparation for quality control purposes shall be carried out according to EN 1482-2.

8 Procedure

8.1 Preparation of the calibration solutions calibration

To obtain the calibration curve, prepare the dilution series according Table 1 from the standard solution (5.2) (each in a 100 ml graduated flask).

Table 1 — Dilution steps for the calibration

Concentration of chloride	Volume of standard solution (5.2)
0,10 mg/l	10 µl
0,50 mg/l	50 µl
1,0 mg/l	100 µl
5,0 mg/l	500 µl
10,0 mg/l	1 000 µl

8.2 Preparation of the test solution and determination

Weigh approximately 1 000 mg of the sample into a 100 ml graduated flask (6.5) to the nearest 0,1 mg, add approximately 70 ml of water to dissolve or to suspense the sample. Shake the flask with the shaking equipment (6.xx) for a approximately 5 minutes. Fill up to the mark with water (5.1) and mix thoroughly.

Filtrate this solution straight through a syringe filter (6.4). Discard the first filtrate collected. Filter another volume and collect in a vial. This solution is used for the determination.

The determination should be performed twice.

The obtained results of these injections shall be within the calibration curve.

If the concentration of the sample injected falls outside the calibration range, an extra dilution step is advisable.

8.3 Quality control sample

The quality control sample should be performed in each sample series.

The determination should be performed twice.

9 Calculation and expression of the results

Carry out the evaluation on the basis of the calibration curve over the peak areas.

NOTE The calibration curve can be used to for the content determination if the correlation coefficient ≥ 0,999.

Calculate the mass fraction of chloride in the sample, in percent, according to the following Formula:

$$w_{\mathrm{Cl}} = \frac{\rho_{\mathrm{Cl}} \cdot V \cdot 100}{m}$$

where

w_{Cl}	is the mass fraction of chloride in the inhibitor in %;
ρ_{Cl}	is the mass concentration of chloride in the sample solution in mg/l;
m	is the mass of the sample in the sample solution, in mg;
V	is the volume of the sample solution, in l;
100	is the calculation factor in %.

10 Environmental aspects

The substances, solutions, eluents and packaging materials should be handled in accordance with the safety data sheets.

Residues of these materials should be disposed under consideration of safety data sheets and in accordance with local, state and federal regulations.

11 Precision (to be completed after ring tests)

11.1 Quality assurance

The recovery of the quality control sample shall be between 95 % and 105 %. If this requirement is invalid, recalibrate.

11.2 Inter-laboratory test

An inter-laboratory test has been carried out in 20xx with xx participating laboratories and x different samples of fertilizers. This test yielded the data given in Annex A. Repeatability and reproducibility were calculated according to ISO 5725-1.

The values derived from this inter-laboratory test may not be applicable to concentration ranges and matrices other than those given in Table 2.

11.3 Repeatability

The absolute difference between two independent single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will in no more than 5 % of the cases exceed the values of r given in Table 2.

11.4 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, will in no more than 5 % of the cases exceed the values of R given in Table 2.

Table 2 — Mean values, repeatability and reproducibility limits

Sample	$\bar{x}$	r	R

12 Test report

The test report shall contain at least the following information:

a) all information necessary for the complete identification of the sample;

b) the test method used with reference to this document including its year of publication;

c) the test results obtained;

d) date of sampling and sampling procedure (if known);

e) date when the analysis was finished;

f) whether the requirement of the repeatability limit has been 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 may have influenced the test result(s).

Annex A
(informative)

Statistical results of the inter-laboratory test

The precision of the method has been determined in the year 20xx in an inter-laboratory trial with x laboratories participating and carried out on x samples of inhibitors. The statistical results are given in Table A.1.

Table A.1 — Statistical results of the inter-laboratory trial

Parameter					
Year of the test					
Number of participating laboratories					
Number of laboratories after eliminating outliers					
Mean value, $\bar{x}$, (%)					
Repeatability standard deviation s_r, (%)					
RSD_r (%)					
Repeatability limit r (2,83 s_r) (%)					
Reproducibility standard deviation, s_R (%)					
RSD_R (%)					
Reproducibility limit R (2,83 s_R) (%)					

Bibliography

[1] EN 1482-1, Fertilizers and liming materials – Sampling and sample preparation – Part 1: Sampling

[2] ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method*