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Fertilizers — Determination of chlorides in the absence of organic material

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Engrais - Dosage des chlorures en l'absence de matières organiques

Düngemittel - Bestimmung von Chlorid bei Abwesenheit organischer Stoffe

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Foreword

This document (EN 16195:2012) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2013, and conflicting national standards shall be withdrawn at the latest by May 2013.

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This document supersedes CEN/TS 16195:2011.

The following changes have been made to the former edition:

a) the CEN Technical Specification has been adopted as a European Standard;

b) the document has been editorially revised.

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1 Scope

This European Standard specifies a method for the determination of chlorides in the absence of organic material. The method is applicable to all fertilizers which are free from organic material.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 12944-1:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials and soil improvers — Vocabulary — Part 2: Terms relating to fertilizers*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944 1:1999 and EN 12944-2:1999 apply.

4 Principle

The chlorides, dissolved in water, are precipitated in an acid medium by an excess of standard solution of silver nitrate. The excess is titrated with a solution of ammonium thiocyanate in the presence of ferric ammonium sulfate (Volhard's method).

5 Sampling

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

Sample preparation shall be carried out in accordance with EN 1482-2. Grinding of the laboratory sample is recommended for homogeneity reasons.

6 Reagents

Use only reagents of recognised analytical grade.

6.1 Water, distilled or demineralised and free from chlorides.

6.2 Nitrobenzene or diethyl ether.

6.3 Nitric acid, c = 10 mol/l.

6.4 Indicator solution.

Dissolve 40 g of ferric ammonium sulfate $Fe_2(SO_4)_3 . (NH_4)_2SO_4 . 24H_2O$, in water and make up to 1 l.

6.5 Silver nitrate standard solution, c = 0,1 mol/l.

Preparation: since this salt is hygroscopic and cannot be dried without risk of decomposition, it is advisable to weigh out an amount of approximately 18 g, dissolve in water and make up the volume to 1 l. Adjust to c = 0,1 mol/l substance concentration by titration of ammonium thiocyanate 0,1 mol/l.

Silver nitrate standard solution is also readily available commercially and may be used instead.

6.6 Ammonium thiocyanate, standard solution, c = 0,1 mol/l.

Preparation: since ammonium thiocyanate is hygroscopic and cannot be dried without risk of decomposition, it is advisable to weigh out an amount of approximately 9 g, dissolve in water and make up the volume to 1 l. Adjust to c = 0,1 mol/l substance concentration by titration of silver nitrate 0,1 mol/l.

Ammonium thiocyanate standard solution is also readily available commercially, and may be used instead.

7 Apparatus

7.1 Standard laboratory equipment.

7.2 Rotary shaker, 35 to 40 min^{-1}.

7.3 Burettes.

7.4 Graduated flask, capacity 500 ml and 1 000 ml.

7.5 Conical (Erlenmeyer) flask, capacity 250 ml.

8 Procedure

8.1 Preparation of the test portion and the solution

Place 5 g of the laboratory sample, weighed to the nearest 0,001 g, in a graduated flask (7.4) and add 450 ml of water (6.1). Mix for 0,5 h on the shaker (7.2), make up to 500 ml with water (6.1), mix and filter into a beaker.

8.2 Determination

Take an aliquot part of the test solution containing not more than 0,150 g of chloride: for example 25 ml (0,25 g), 50 ml (0,5 g) or 100 ml (1 g). If the amount of the test solution taken is smaller than 50 ml it is necessary to make up the volume to 50 ml with water (6.1).

Add 5 ml of nitric acid (6.3), 20 ml of the indicator solution (6.4), and two drops of ammonium thiocyanate standard solution (6.6) (this latter reagent is added with a burette adjusted to zero for this purpose).

With a burette (7.3) then add silver nitrate standard solution (6.5) until there is an excess of 2 ml to 5 ml. Add 5 ml of nitrobenzene or 5 ml of diethyl ether (6.2) and shake well to agglomerate the precipitate. Titrate the excess silver nitrate with ammonium thiocyanate (6.6) until a red-brown colour appears which remains after the flask has been shaken slightly.

NOTE Nitrobenzene or diethyl ether (but above all nitrobenzene) prevents the silver chloride from reacting with thiocyanate ions. Thus a clear colour change is obtained.

8.3 Blank test

Carry out a blank test (omitting the test portion) under the same conditions and allow for it when calculating the final result.

8.4 Control test

Before carrying out the estimations check the accuracy of the method by using an aliquot part of a freshly prepared solution of potassium chloride, such that this part contains a known quantity in the order of 100 mg of chloride.

9 Calculation and expression of the results

Express the result of the analysis as a percentage of chloride contained in the sample as it has been received for analysis.

Calculate the mass fraction of chlorides (Cl), w_{Cl}, in percent according to Formula (1).

$$w_{Cl} = 0{,}003\,546 \times \frac{(V_Z - V_{CZ}) - (V_a - V_{ca})}{M} \times 100 \quad (1)$$

where

V_z is the amount of silver nitrate 0,1 mol/l, in millilitres;

V_{cz} is the amount of silver nitrate 0,1 mol/l, used in the blank test, in millilitres;

V_a is the amount of ammonium thiocyanate 0,1 mol/l, in millilitres;

V_{ca} is the amount of ammonium thiocyanate 0,1 mol/l, used in the blank test, in millilitres;

M is the mass, in grams, of the test portion taken (8.1).

10 Precision

10.1 Inter-laboratory test

An inter-laboratory test has been carried out in 2009 with 14/13 participating laboratories and two different samples. The repeatability and reproducibility were calculated according to ISO 5725-2.

The values derived from these inter-laboratory tests may not be applicable to concentration ranges and matrices other than those given in Annex A.

10.2 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 not more than 5 % of the cases exceed the values of r given in Table 1.

10.3 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 not exceed values of R given in Table 1 in more than 5 % of the cases.

Table 1 — Mean values, repeatability and reproducibility limits

Sample	$\bar{x}$ %	r %	R %
Patent kali	2,781	0,066	0,284
NPK2 (12-11-18+4+8)	0,478	0,076	0,312

11 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;

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 might have influenced the test result(s).

Annex A
(informative)

Statistical results of the inter-laboratory test

The precision of the method was established in 2009 by Working Group 7 “Chemical analysis” of CEN/TC 260 “Fertilizers and liming materials” in an inter-laboratory test evaluated in accordance with ISO 5725-2. The statistical results are given in Table A.1.

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

Parameter	Sample	
	Patent kali	NPK2 (12-11-18+4+8)
Number of participating laboratories	14	13
Number of laboratories after elimination of outliers (accepted test results)	11	13
Mean value $\bar{x}$ (%)	2,781	0,478
Repeatability standard deviation s_r (%)	0,024	0,027
RSD_r (%)	0,9	6,0
Repeatability limit r (%)	0,066	0,076
Reproducibility standard deviation s_R (%)	0,101	0,111
RSD_R (%)	3,6	23,0
Reproducibility limit R (%)	0,284	0,312

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*

[3] *Regulation (EC) No 2003/2003 of the European Parliament and of the Council of 13 October 2003 relating to fertilisers*, Official Journal L 304, 21/11/2003, p. 0001-0194, Annex I and Annex IV, method 6.1

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