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ICS

English Version

Fertilizers - Determination of the sodium extracted by flame-emission spectrometry

Engrais - Détermination du sodium extrait par spectrométrie d'émission de flamme

Düngemittel - Bestimmung von extrahiertem Natrium mit Emissions-Flammenspektrometrie

This draft Technical Specification is submitted to CEN members for formal vote. It has been drawn up by the Technical Committee CEN/TC 260.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (FprCEN/TS 16199) has been prepared by Technical Committee CEN/TC 260 “Fertilizers and liming materials”, the secretariat of which is held by DIN.

This document is currently submitted to the Formal Vote.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association.

FprCEN/TS 16199:2010 (E)

1 Scope

This Technical Specification specifies a method for the determination of the sodium content in fertilizer extracts by flame-emission spectrometry.

The method is applicable to EC fertilizers for which a declaration of the sodium content is provided for in Regulation (EC) Nr 2003/2003, Annex I [1].

2 Normative references

The following referenced documents are indispensable for the application 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 12944-1:1999, *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*

CEN/TS 15960, *Fertilizers — Extraction of total calcium, total magnesium, total sodium and total sulfur in the forms of sulfates*

CEN/TS 15961, *Fertilizers — Extraction of water soluble calcium, magnesium, sodium and sulfur in the form of sulfates*

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

Following suitable dilution of the extract obtained according to CEN/TS 15960 or CEN/TS 15961, the sodium content of the solution is determined by flame-emission spectrometry.

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

6.1 Diluted hydrochloric acid.

Add one volume of hydrochloric acid for analysis ($\rho_{20} = 1,18$ g/ml) to one volume of water.

6.2 Aluminium nitrate, $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$.

6.3 Caesium chloride, CsCl .

6.4 Anhydrous sodium chloride, NaCl .

6.5 Caesium chloride and aluminium nitrate solution.

Dissolve in water 50 g of caesium chloride (6.3) and 250 g of aluminium nitrate (6.2) in a graduated flask (7.2). Make up to volume with water and mix.

6.6 Standard sodium solution, $\rho(\text{Na}) = 1$ mg/ml.

Dissolve in water 2,542 g of sodium chloride (6.4) in a graduated flask (7.2). Add 10 ml of hydrochloric acid (6.1). Make up to volume with water and mix.

6.7 Calibration solutions.

6.7.1 Place 10 ml of standard solution (6.6) in a graduated flask (7.3). Make up to volume and mix. Mass concentration of the solution, $\rho(\text{Na}) = 40$ $\mu\text{g}/\text{ml}$.

6.7.2 Place 0 ml, 5 ml, 10 ml, 15 ml, 20 ml, 25 ml of the intermediate solution (6.7.1) in graduated flasks (7.4). Add 10 ml of the solution (6.5). Make up to volume and mix. Mass concentration of the solutions, $\rho(\text{Na}) = 0$ $\mu\text{g}/\text{ml}$, 2 $\mu\text{g}/\text{ml}$, 4 $\mu\text{g}/\text{ml}$, 6 $\mu\text{g}/\text{ml}$, 8 $\mu\text{g}/\text{ml}$ and 10 $\mu\text{g}/\text{ml}$.

7 Apparatus

7.1 Spectrometer, equipped for flame emission, set at 589,3 nm.

7.2 1 000 ml graduated flask.

7.3 250 ml graduated flask.

7.4 100 ml graduated flasks.

8 Preparation of the test solution

Depending upon the expected sodium content of the extraction solution according to CEN/TS 15960 or CEN/TS 15961 (5 g of fertilizer in 500 ml), carry out the dilutions in accordance with Table 1.

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Table 1 — Preparation of the test solution

Na ₂ O %	Na %	Intermediate dilution		Final dilution		Degree of dilution
		Sample (ml) (v ₂)	Dilution to ml (v ₃)	Sample (ml) (v ₄)	Dilution to ml	
3 to 5	2,2 to 3,7	10	50	10	100	50
5 to 10	3,7 to 7,4	10	100	10	100	100
10 to 20	7,4 to 15	10	100	5	100	200
20 to 38	15 to 28	5	100	5	100	400

Make up the intermediate dilution with water. For the final dilution add 10 ml of the solution (6.5) to the graduated flask (7.4).

For a test portion of 1 g multiply the volume of the final dilution (v₄) by 5.

9 Procedure

Prepare the spectrometer (7.1) for the measurements at 589,3 nm. Calibrate the instrument by measuring the response of the calibration solutions (6.7.2). Then adjust the sensitivity of the instrument to use its entire scale when the most concentrated calibration solution is used. Then measure the response of the test solution to be analyzed (7.1). Repeat this operation three times.

10 Calculation and expression of the result

Draw a calibration curve by plotting the average response for each calibration solution along the ordinate and the corresponding concentrations, expressed in micrograms per millilitre on the abscissa. Determine from this the sodium concentration of the test solution. Calculate the quantity of sodium from the standard solutions taking account of the levels of dilution. Express the results as a mass fraction in percent of the sample.

Calculate the Na content as a mass fraction, w_{Na} , in percent of the fertilizer according to Equation (1).

$$w_{\text{Na}} = x \frac{v_3 \times v_1 \times 10^{-2}}{v_4 \times v_2 \times m} \quad (1)$$

Calculate the Na₂O content as a mass fraction, $w_{\text{Na}_2\text{O}}$, in percent of the fertilizer according to Equation (2).

$$w_{\text{Na}_2\text{O}} = w_{\text{Na}} \times 1,348 \quad (2)$$

where

- x is the mass concentration of the solution introduced into the spectrometer, in micrograms per millilitre;
- v_1 is the volume of the extraction solution in millilitres;
- v_2 is the aliquot volume in the intermediate dilution in millilitres;
- v_3 is the volume of intermediate dilution in millilitres;

v_4 is the aliquot volume, in millilitres, of the final dilution (to 100 ml);

m is the mass of the test sample in grams.

11 Precision

11.1 Inter-laboratory test

Repeated inter-laboratory tests have been carried out in 2007 and 2009 with different numbers of participating laboratories and several different samples (see Table A.1 to Table A.4). 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.

11.2 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 2 and Table 3.

11.3 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 in not more than 5 % of the cases exceed values of R given in Table 2 and Table 3.

Table 2 — Results ring test 2007

Sample	Extraction method	\bar{x} %	r %	R %
KALI ROH	CEN/TS 15960	30,2	0,9	4,4
	CEN/TS 15961	29,8	0,44	1,4
NPK1:20+0+8+Na	CEN/TS 15960	0,56	0,08	0,3
	CEN/TS 15961	0,52	0,04	0,12

Table 3 — Results ring test 2009

Sample	Extraction method	\bar{x} %	r %	R %
NPK:12-12-17S+2	CEN/TS 15960	5,46	0,22	0,22
	CEN/TS 15961	5,73	0,39	1,85

12 Test report

The test report shall contain at least the following information: