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

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilisers and related chemicals.

A list of organizations represented on this committee can be obtained on request to its secretary.

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English Version

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

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

Düngemittel - Bestimmung von extrahiertem Natrium mit Emissions-Flammenspektrometrie

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Contents

Page

Foreword

This document (EN 16199:2012) 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of an identical text or by endorsement, at the latest by May 2013, and conflicting national standards shall be withdrawn at the latest by May 2013.

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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 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 [3].

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*

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

EN 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 EN 15960 or EN 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 (ρ_{20} = 1,18 g/ml) to one volume of water.

6.2 Aluminium nitrate, $Al(NO_3)_3\ 9H_2O$.

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, ρ(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, ρ(Na) = 40 µg/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, ρ(Na) = 0 µg/ml, 2 µg/ml, 4 µg/ml, 6 µg/ml, 8 µg/ml and 10 µg/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 EN 15960 or EN 15961 (5 g of fertilizer in 500 ml), carry out the dilutions in accordance with Table 1.

Table 1 — Preparation of the test solution

Na_2O %	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 (v_4) 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 analysed (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 Formula (1):

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

Calculate the Na_2O content as a mass fraction, w_{Na2O}, in percent of the fertilizer according to Formula (2):

$$w_{Na_2O} = w_{Na} \times 1{,}348 \quad (2)$$

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 portion 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	EN 15960	30,2	0,9	4,4
	EN 15961	29,8	0,44	1,4
NPK1:20+0+8+Na	EN 15960	0,56	0,08	0,3
	EN 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	EN 15960	5,46	0,22	0,22
	EN 15961	5,73	0,39	1,85

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;

c) the method of preparation of the extraction solution (EN 15960 or EN 15961);

d) the test results obtained;

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

f) date when the analysis was finished;

g) whether the requirement of the repeatability limit has been fulfilled;

h) 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 2007 and in 2009 by Working Group 7 “Chemical analysis” of CEN/TC 260 “Fertilizers and liming materials” in several inter-laboratory tests evaluated in accordance with ISO 5725-2. The statistical results are given in Table A.1 and Table A.2.

Table A.1 — Statistical results of the inter-laboratory test in 2007

Parameter	Sample			
	KALI ROH		NPK1:20+0+8+Na	
Extraction method	**EN 15960**	**EN 15961**	**EN 15960**	**EN 15961**
Number of participating laboratories	11	10	9	9
Number of laboratories after elimination of outliers (accepted test results)	8	7	7	8
Mean value $\bar{x}$ (%)	30,2	29,8	0,56	0,52
Repeatability standard deviation s_r (%)	0,3	0,16	0,03	0,01
RSD_r (%)	1,0	0,5	5,0	3,0
Repeatability limit r (%)	0,9	0,44	0,08	0,04
Reproducibility standard deviation s_R (%)	1,6	0,5	0,1	0,04
RSD_R (%)	5,2	1,7	17,0	8,0
Reproducibility limit R (%)	4,4	1,4	0,3	0,12

Table A.2 — Statistical results of the inter-laboratory test in 2009

Parameter	Sample	
	NPK:12-12-17S+2	
Extraction method	**EN 15960**	**EN 15961**
Number of participating laboratories	5	5
Number of laboratories after elimination of outliers (accepted test results)	4	5
Mean value $\bar{x}$ (%)	5,46	5,73
Repeatability standard deviation s_r (%)	0,08	0,14
RSD_r (%)	1,0	2,0
Repeatability limit r (%)	0,22	0,39
Reproducibility standard deviation s_R (%)	0,08	0,66
RSD_R (%)	1,0	12,0
Reproducibility limit R (%)	0,22	1,85

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 8.10

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