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Fertilizers - Extraction and determination of elemental sulfur

Düngemittel - Extraktion und Bestimmung von elementarem Schwefel

Engrais - Extraction et dosage du soufre élémentaire

Ta slovenski standard je istoveten z: FprCEN/TS 16032

ICS:

65.080	Gnojila	Fertilizers

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TECHNICAL SPECIFICATION

SPÉCIFICATION TECHNIQUE

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**FINAL DRAFT
FprCEN/TS 16032**

December 2009

ICS

English Version

Fertilizers - Extraction and determination of elemental sulfur

Engrais - Extraction et dosage du soufre élémentaire

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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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COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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FprCEN/TS 16032:2009 (E)

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Foreword

This document (FprCEN/TS 16032:2009) 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 16032:2009 (E)

1 Scope

This document specifies a method for extraction and determination of the elemental sulfur contained in fertilizers.

The method is applicable to EC fertilizers for which a declaration of the total sulfur in elemental form 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*

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

After the removal of soluble compounds, elemental sulfur is extracted by using carbon disulfide, followed by gravimetric determination of the sulfur extracted.

WARNING — This method of analysis involves the use of carbon disulfide (CS_2). Special safety measures shall therefore be taken, in particular with regard to:

— the storage of CS_2,

— protective equipment for staff,

— occupational hygiene,

— prevention of fire and explosions,

— disposal of the reagent.

This method requires a highly skilled staff and a suitably equipped laboratory.

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5   Sampling and sample preparation

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   Carbon disulfide.

7   Apparatus

7.1   Extraction flask, capacity 100 ml, with ground glass stopper.

7.2   Soxhlet apparatus, with the appropriate filter elements.

7.3   Vacuum rotary evaporator

7.4   Electric oven, fan assisted, set at (90 ± 2) °C.

7.5   Porcelain Petri dishes, 50 mm to 70 mm in diameter, not exceeding 50 mm in height.

7.6   Electric hot plate, with adjustable temperature.

8   Procedure

8.1   Test portion

Weigh to an accuracy of 1 mg, 5 g to 10 g of the laboratory sample and place the test portion in the thimble of the Soxhlet apparatus (7.2).

8.2   Extraction of the sulfur

Wash thoroughly the contents with hot water to remove all soluble compounds. Dry in the oven (7.4) at 90 °C for at least 1 h. Place the filter in the Soxhlet apparatus (7.2).

Place a few glass beads in the flask of the apparatus (7.1) and weigh (m_0), then add 50 ml of carbon disulfide (6.1).

Connect the apparatus and leave the elemental sulfur to be extracted for 6 h. Switch off the heat and, after cooling, disconnect the flask. Connect the flask to the rotary evaporator (7.3) and evaporate until the contents of the flask have solidified in a spongy mass.

Dry the flask in the oven (7.4) at 90 °C (generally 1 h is necessary) until a constant mass is obtained (m_1).

8.3 Determination of the purity of the elemental sulfur

Certain substances may have been extracted by the carbon disulfide at the same time as the elemental sulfur. The purity of the elemental sulfur is determined as follows:

Homogenize the contents of the flask as thoroughly as possible, and remove 2 g or 3 g, weighed to an accuracy of 1 mg (n). Place in the Petri dish (7.5). Weigh the dish and contents together (m_2). Place on the hot plate (7.6) set at a temperature not exceeding 220 °C so as not to cause combustion of the sulfur. Continue sublimation for 3 h or 4 h until a constant mass is obtained (m_3).

NOTE   For some fertilizers, it may not be necessary to determine how pure the sulfur is. In this case, omit step 8.3.

9 Calculation and expression of the results

Calculate the mass fraction of impure elemental sulfur (S), w_{Si}, in percent of the fertilizer according to equation (1).

$$w_{Si} = \frac{m_1 - m_0}{m} \times 100 \tag{1}$$

Calculate the purity of sulfur extracted, w_p, in percent of the fertilizer according to equation (2).

$$w_p = \frac{m_2 - m_1}{n} \times 100 \tag{2}$$

Calculate the mass fraction of pure elemental sulfur (S), w_{Sp}, in percent of the fertilizer according to equation (3).

$$w_{Sp} = \frac{(m_1 - m_0)(m_2 - m_3)}{m \times n} \times 100 \tag{3}$$

where

   m is the mass of the test portion, in grams;

   m_0 is the mass of the Soxhlet flask, in grams;

   m_1 is the mass of the Soxhlet flask and the impure sulfur after drying, in grams;

   n is the mass of the impure sulfur to be purified, in grams;

   m_2 is the mass of the Petri dish, in grams;

   m_3 is the mass of the Petri dish after sublimation of the sulfur, in grams.

10 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).