

BS EN 16032:2011



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Fertilizers — Extraction and determination of elemental sulfur

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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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ISBN 978 0 580 74621 5

ICS 65.080

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 December 2011.

Amendments issued since publication

Date	Text affected
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EUROPEAN STANDARD

EN 16032

NORME EUROPÉENNE

EUROPÄISCHE NORM

November 2011

ICS 65.080

Supersedes CEN/TS 16032:2010

English Version

Fertilizers - Extraction and determination of elemental sulfur

Engrais - Extraction et dosage du soufre élémentaire

Düngemittel - Extraktion und Bestimmung von
elementarem Schwefel

This European Standard was approved by CEN on 29 October 2011.

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Contents

Page

Foreword.....	3
1 Scope	4
2 Normative references	4
3 Terms and definitions	4
4 Principle.....	4
5 Sampling and sample preparation	4
6 Reagents.....	5
7 Apparatus	5
8 Procedure	5
8.1 Test portion	5
8.2 Extraction of the sulfur	5
8.3 Determination of the purity of the elemental sulfur	5
9 Calculation and expression of the results	6
10 Test report	6
Bibliography	7

Foreword

This document (EN 16032:2011) 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of an identical text or by endorsement, at the latest by May 2012, and conflicting national standards shall be withdrawn at the latest by May 2012.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes CEN/TS 16032:2010.

The following has been added to the former edition of the European Standard:

- a) the CEN Technical Specification has been adopted as a European Standard;
- b) the document has been editorially revised.

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

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1 Scope

This European Standard 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 (CS₂). Special safety measures shall therefore be taken, in particular with regard to:

- the storage of CS₂;
- 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.

5 Sampling and sample preparation

Sampling is not part of the method specified in this European Standard. 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 (m) 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 electric 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 electric 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). Weigh the Petri dish (m_2) and place the aliquot in it. 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 Only compounds with boiling point < the temperature of the hotplate will be removed.

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).

$$w_{Si} = \frac{m_1 - m_0}{m} \times 100 \quad (1)$$

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

$$w_p = \frac{m_3 - m_2}{n} \times 100 \quad (2)$$

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

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

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 European Standard (i.e. EN 16032);
- 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).

Bibliography

- [1] *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 8.5
- [2] EN 1482-1, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*

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