

BS EN 15925:2011



BSI Standards Publication

# Fertilizers — Extraction of total sulfur present in various forms

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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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EUROPEAN STANDARD

**EN 15925**

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EUROPÄISCHE NORM

September 2011

ICS 65.080

Supersedes CEN/TS 15925:2009

English Version

**Fertilizers - Extraction of total sulfur present in various forms**

Engrais - Extraction du soufre total présent sous différentes formes

Düngemittel - Extraktion von Gesamtschwefel, der in verschiedener Form vorliegen kann

This European Standard was approved by CEN on 18 August 2011.

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## Foreword

This document (EN 15925:2011) 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of an identical text or by endorsement, at the latest by March 2012, and conflicting national standards shall be withdrawn at the latest by March 2012.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 15925:2009.

The following changes have been made to the former edition:

- a) the CEN Technical Specification has been adopted as a European Standard;
- b) Clause 9 "Test report" has been added;
- c) 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 document specifies a method for the extraction of the total sulfur contained in fertilizers in elemental form and/or in other chemical combinations.

The method is applicable to EC fertilizers for which a declaration of the total sulfur present in various forms (elemental, thiosulfate, sulfite, sulfate) is provided.

## 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*

EN 15749, *Fertilizers — Determination of sulfates content using three different methods*

## 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 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 is recommended for homogeneity reasons.

## 5 Principle

Elemental sulfur is converted in an alkaline medium into polysulfides and thiosulfate; these, together with any sulfites that can be present, are then oxidized with hydrogen peroxide. The various forms of sulfur are thus converted into sulfate that is determined by precipitation of barium sulfate.

## 6 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water.

### 6.1 Diluted hydrochloric acid.

Mix one volume of hydrochloric acid ( $d = 1,18$ ) with one volume of water.

### 6.2 Sodium hydroxide solution, NaOH 30 % minimum, $d = 1,33$ .

**6.3 Hydrogen peroxide solution**,  $w = 30 \%$ .

**6.4 Barium chloride**, aqueous solution of  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ ,  $\rho = 122 \text{ g/l}$ .

## 7 Apparatus

**7.1 Electric hot plate**, with adjustable temperature.

**7.2 400 ml beaker**.

**7.3 600 ml beaker**.

**7.4 250 ml volumetric flask**.

## 8 Procedure

### 8.1 Test portion

Weigh to an accuracy of 1 mg a quantity of the laboratory sample containing between 80 mg and 350 mg of sulfur (S) or 200 mg and 875 mg of  $\text{SO}_3$ .

As a rule (where  $\text{S} < 15 \%$ ), weigh 2,5 g. Place the test portion in a beaker (7.2).

### 8.2 Oxidation

Add 20 ml of sodium hydroxide solution (6.2) and 20 ml of water. Cover with a watch glass. Boil for 5 min on the hot plate (7.1). Remove from the hot plate. Using a jet of hot water, collect the sulfur sticking to the sides of the beaker and boil for 20 min. Leave to cool.

Add 2 ml increments of hydrogen peroxide (6.3) until no reaction is observed. 6 ml to 8 ml of hydrogen peroxide will be necessary. Allow oxidation to continue for 1 h, and then bring to the boil for 0,5 h. Leave to cool.

### 8.3 Preparation of the solution to be analyzed

Add approximately 50 ml of water and 50 ml of the hydrochloric acid solution (6.1).

#### a) If the level of sulfur (S) is less than 5 %:

Filter into a 600 ml beaker (7.3). Wash the residue on the filter several times with cold water. After washing, check for the absence of sulfate in the last drops of the filtrate using a barium chloride solution (6.4). The filtrate shall be perfectly clear. Sulfate is determined on the whole of the filtrate in accordance with EN 15749.

#### b) If the level of sulfur (S) is at above 5 %:

Transfer quantitatively into a 250 ml volumetric flask (7.4), make up to volume with water and mix. Filter through a dry filter into a dry container; the filtrate shall be completely clear. Stopper if the solution is not to be used immediately. Determine sulfates on an aliquot of this solution by precipitation in the form of barium sulfate in accordance with EN 15749.

## 9 Test report

The test report shall contain at least the following information:

a) the test method used with a reference to this document;

- b) all information necessary for the complete identification of the sample;
- c) date of sampling and sampling procedure (if known);
- d) date when the extraction was finished;
- e) all operating details not specified in this document, or regarded as optional, together with details of any incidents that occurred when performing the method which might have influenced the test result(s).



## Bibliography

- [1] EN 1482-1, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*
- [2] *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, Pp. 1-194, Annex IV, method 8.2





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