

Fertilizers — Determination of sulfates content using three different methods

ICS 65.080

National foreword

This British Standard is the UK implementation of EN 15749:2009. It supersedes DD CEN/TS 15749:2008 which is withdrawn.

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 sulfates content using three
different methods**

Engrais - Dosage des sulfates selon trois méthodes
différentes

Düngemittel - Bestimmung von Sulfat mit drei
verschiedenen Verfahren

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Management Centre: Avenue Marnix 17, B-1000 Brussels

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Foreword

This document (EN 15749:2009) 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of an identical text or by endorsement, at the latest by April 2010, and conflicting national standards shall be withdrawn at the latest by April 2010.

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Introduction

This document specifies three different methods for the determination of sulfur. Based on the statistical results of the inter-laboratory tests, obtained with the same samples, the three methods produce equivalent results, and hence can be used all three methods on decision of the user and availability of equipment.

1 Scope

This European Standard specifies three different methods (Methods A, B and C) for the determination of sulfur present in fertilizers extracts in the form of sulfates. Method A specifies the gravimetric method. Method B specifies the method using inductively coupled plasma optical spectrometry (ICP-OES). Method C specifies the method using ion chromatography (IC).

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 15925, *Fertilizers — Extraction of total sulfur present in various forms*

CEN/TS 15926, *Fertilizers — Extraction of water soluble sulfur where the sulfur is in various forms*

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 forms of sulfates)*

EN ISO 3696, *Water for analytical laboratory use - Specification and test methods (ISO 3696:1987)*

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

4.1 Method A: Gravimetric method

Sulfur is extracted from the sample according to the methods described in CEN/TS 15925, CEN/TS 15926, CEN/TS 15960 or CEN/TS 15961 and determined based on the gravimetric determination as barium sulfate.

4.2 Method B: ICP-OES

Sulfur is extracted from the sample according to the methods described in CEN/TS 15925, CEN/TS 15926, CEN/TS 15960 or CEN/TS 15961 and its concentration in the extract is measured by inductively coupled plasma-optical emission spectrometry (ICP-OES).

4.3 Method C: IC

Sulfur is extracted from the fertilizer according to the methods described in CEN/TS 15925, CEN/TS 15926, CEN/TS 15960 or CEN/TS 15961 in the form of sulfate. The sulfate concentration of the extract is measured by ion chromatography (IC) equipped with a suppressor device and a conductivity detector.

5 Sampling and sample preparation

Sampling is not part of the methods 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.

6 Method A – Gravimetric method

6.1 Reagents

Use only reagents of recognized analytical grade and distilled or demineralized water (grade 3 according to EN ISO 3696).

6.1.1 Diluted hydrochloric acid

Mix one volume of $\rho(\text{HCl}) = 1,18 \text{ g/ml}$ with one volume of water.

6.1.2 Barium chloride solution, $\rho(\text{BaCl}_2 \cdot 2 \text{ H}_2\text{O}) = 122 \text{ g/l}$.

6.1.3 Silver nitrate solution, $\rho = 5 \text{ g/l}$.

6.2 Apparatus

6.2.1 Porcelain crucibles

6.2.2 Hot water bath

6.2.3 Drying oven, set at $105 \text{ }^\circ\text{C} \pm 1 \text{ }^\circ\text{C}$.

6.2.4 Electric oven, set at $800 \text{ }^\circ\text{C} \pm 50 \text{ }^\circ\text{C}$.

6.3 Procedure

6.3.1 Sampling of the solution

Pipette an aliquot part of one of the extraction solutions containing between 20 mg and 100 mg of sulfur or 50 mg and 250 mg of SO_3 .

Place this aliquot in a beaker of suitable capacity. Add 20 ml of diluted hydrochloric acid (6.1.1). Make up to about 300 ml with water.

6.3.2 Preparation of the precipitate

Bring the solution to the boil. Add, drop by drop, about 20 ml of the barium chloride solution (6.1.2) while stirring the solution vigorously. Boil for a few minutes.