BRITISH STANDARD

BS EN
12947:2000

Liming materials — Determination of magnesium content — Atomic absorption spectrometric method

The European Standard EN 12947:2000 has the status of a British Standard

ICS 65.080

National foreword

This British Standard is the official English language version of EN 12947:2000.

The UK participation in its preparation was entrusted to Technical Committee CII/37, Fertilizers and related chemicals, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

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

The United Kingdom voted against the acceptance of this standard at the CEN Formal Vote stage because the precision data quoted for this method is incomplete and highly suspect.

Cross-references

The British Standards which implement international or European publications referred to in this document may be found in the BSI Standards Catalogue under the section entitled "International Standards Correspondence Index", or by using the "Find" facility of the BSI Standards Electronic Catalogue.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, the EN title page, pages 2 to 8, an inside back cover and a back cover.

The BSI copyright notice displayed in this document indicates when the document was last issued.

This British Standard, having been prepared under the direction of the Sector Committee for Materials and Chemicals, was published under the authority of the Standards Committee and comes into effect on 15 August 2000

ISBN 0 580 35921 2

Amendments issued since publication

Amd. No.	Date	Comments

EUROPEAN STANDARD NORME EUROPÉENNE EUROPÄISCHE NORM

EN 12947

June 2000

ICS 65.080

English version

Liming materials – Determination of magnesium content – Atomic absorption spectrometric method

Amendements calciques et/ou magnésiens – Détermination de la teneur en magnésium – Méthode par spectrométrie d'absorption atomique

Calcium-/Magnesium-Bodenverbesserungsmittel – Bestimmung des Magnesiumgehaltes – Atomabsorptionsspektrometrisches Verfahren

This European Standard was approved by CEN on 13 November 1999.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Ref. No. EN 12947:2000 E

Contents

Foreword

This European Standard 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 December 2000, and conflicting national standards shall be withdrawn at the latest by December 2000.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies a method for the determination of the magnesium content of all liming materials by atomic absorption spectrometry.

2 Normative references

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text, and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 1482, *Sampling of solid fertilizers and liming materials.*

ISO 3310-1, *Test sieves – Technical requirement and testing – Part 1: Test sieves of metal wire cloth.*

3 Principle

Dissolution of the test portion in diluted hydrochloric acid. Filtration and dilution. Determination of magnesium by flame atomic absorption spectrometry at a wavelength of 285,2 nm.

4 Reagents

4.1 General

Commercially available standard solutions may be used instead of standard solutions produced on-site in the laboratory.

4.2 Hydrochloric acid solution, diluted

Dilute 500 ml of concentrated hydrochloric acid (ρ_{20} = 1,18 g/ml) to 1 000 ml with water.

4.3 Hydrochloric acid solution

Approximately c(HCl) ≈ 1 mol/l

4.4 Hydrochloric acid solution

Approximately c(HCl) ≈ 0,5 mol/l

4.5 Standard magnesium solution

Containing 1,000 g of magnesium per litre.

4.5.1 Weigh 1,013 g of magnesium sulfate ($MgSO_4 \cdot 7H_2O$) into a 100 ml volumetric flask, dissolve in hydrochloric acid solution (4.4) and dilute to the mark with the same solution.

or

4.5.2 Heat magnesium oxide (MgO) at 600 °C for 2 h. Weigh 1,658 g of the freshly calcined MgO into a 500 ml beaker, dissolve in 100 ml of water and 120 ml of hydrochloric acid solution (4.3). Transfer quantitatively the solution to a 1 000 ml volumetric flask, dilute to the mark with water and mix thoroughly.

4.6 Lanthanum solution,

Containing 50 g lanthanum per litre.

4.6.1 Weigh 58,64 g lanthanum oxide (La_2O_3) into a 600 ml beaker. Add 50 ml of water and carefully 250 ml of concentrated hydrochloric acid (ρ_{20} = 1,18 g/ml). Stir, while cooling to room temperature. Transfer the solution quantitatively to a 1 000 ml volumetric flask, dilute to the mark with water and mix thoroughly.

or;

4.6.2 Weigh 133,69 g of lanthanum chloride ($LaCl_3 \cdot 7H_2O$) into a 600 ml beaker. Dissolve the salt in 500 ml of water under stirring. Transfer quantitatively to a 1 000 ml volumetric flask, dilute to the mark with water and mix thoroughly.

or

4.6.3 Weigh 155,85 g of lanthanum nitrate ($La(NO_3)_3 \cdot 6H_2O$) into a 600 ml beaker. Dissolve the salt in 500 ml of water under stirring.

Transfer quantitatively to a 1 000 ml volumetric flask, dilute to the mark with water and mix thoroughly.

NOTE A strontium solution may be used instead of lanthanum.

4.7 Strontium solution

Containing 50 g strontium per litre.

Weigh 75 g of strontium chloride ($SrCl_2 \cdot 6H_2O$) and dissolve in hydrochloric acid solution (4.4). Make up to 500 ml with the solution (4.4).

5 Apparatus

Usual laboratory apparatus and in particular the following:

5.1 Test sieve conforming to the requirement of ISO 3310-1, of nominal aperture size 250 µm.

5.2 Pestle and mortar, each of porcelain, or mechanical grinder.

5.3 Electric hot plate with adjustable temperature

5.4 Atomic absorption spectrometer with an air-acetylene burner, adjusted to a wavelength of 285,2 nm.

6 Sampling

Sample the liming materials in accordance with EN 1482.

7 Procedure

7.1 Preparation of test sample

Prepare the received laboratory sample by sieving it rapidly through the test sieve (5.1).

Crush the residue in a mortar or grind it with a mechanical grinder (5.2) and sieve again.

Repeat these operations until the sample passes the sieve completely.

Mix the test sample thoroughly.

7.2 Preparation of test solution

Weigh about 1 g to the nearest 0,001 g from the test sample (7.1) into a 600 ml beaker and add approximately 400 ml of water. Carefully add 50 ml of hydrochloric acid solution (4.2). Cover the beaker with a watch-glass to avoid possible contamination and loss of solution, and boil for 30 minutes. Continue stirring while allowing the solution to cool to ambient temperature.

Transfer the solution quantitatively to a 500 ml volumetric flask, dilute to the mark with water and mix thoroughly.

Filter through a dry filter, discarding the first 50 ml of the filtrate. The solution shall be clear without any turbidity.

Store this test solution in a stoppered flask.

7.3 Determination

7.3.1 Dilution

Pipette an aliquot of the test solution (7.2) into a 100 ml volumetric flask in order to obtain a dilution in the optimum measuring range of the atomic absorption spectrometer.

Add lanthanum solution (4.6) to the final dilution used for measurement in order to obtain a volume fraction of 10 % of lanthanum solution (4.6), dilute to the mark with water and mix.

7.3.2 Preparation of blank solution

Prepare a blank solution in accordance with the procedure described in 7.3.1 without any test portion.

7.3.3 Calibration

Prepare at least five calibration solutions within the optimum measuring range of the apparatus (5.4). Add lanthanum solution (4.6) in order to obtain a volume fraction of 10 % of lanthanum solution in the measuring solution.

7.4 Measurement

Prepare the atomic absorption spectrometer (5.4) in accordance with the instructions of the manufacturer. Allow about 10 min for the spectrometer to warm up until stable conditions without drift are reached.

Adjust the spectrometer to zero against the zero calibration solution prepared in accordance with 7.3.3 and then correct the result for the test solution for that of the blank solution.

Measure the standard solutions (7.3.3), the test solution (7.3.1) and the blank solution (7.3.2) alternately about three times each, without interruptions or changing instrument settings against the solution. During each measurement, wait till a stable signal is obtained.

Plot the calibration graph using the mean absorbance of each of the calibration solutions (7.3.3) as the ordinates and the corresponding concentration of magnesium in µg/ml as the abscissae.

8 Expression of results

The magnesium content w_{Mg}, expressed as a mass fraction of Mg, is given by the following equation:

$$w_{Mg} = \frac{\left(\rho_{Mg} - \rho_{bv}\right) \times D_1 \times D_2 \times \ldots D_n}{m \times 20} \qquad (1)$$

where

ρ_{Mg} is the concentration of magnesium, read off from the calibration graph for the test solution, in micrograms per millilitre;

ρ_{bv} is the blank value, read off from the calibration graph for the blank solution;

D_1, D_2, D_n are the dilution factors

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

The magnesium content expressed as a mass fraction of MgO is obtained by dividing the above result by 0,6.

9 Precision

9.1 General

The values derived from inter-laboratory tests.

9.2 Repeatability

The absolute difference between two single results found on identical test materials by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability limit r in not more than 5 % of cases.

The value is:

r = 0,21 %

9.3 Reproducibility

The absolute difference between two single test results on identical test material reported by two laboratories will exceed the reproducibility limit R in not more than 5 % of the cases.

The value is:

R = 0,25 %

10 Test report

The test report shall contain at least the following information:

— all data necessary for the identification of the sample;

— a reference to this European Standard;

— the results and the units in which the results have been expressed;

— any particular points observed in the course of the test;

— any operations not specified in the method or regarded as optional which might have affected the results.

Bibliography

VDLUFA, Manual II of analysing methods for fertilizers. (VDLUFA-Verlag, Bismarckstraße 41 A, D-64293 Darmstadt).

EC 77/535:1977, Council directive of 22.06.1977 on the approximation of laws in Member states regarding sampling and testing of fertilizers. OJEC n° L 213/1 of 22.08.1977.

blank

BSI — British Standards Institution

BSI is the independent national body responsible for preparing British Standards. It presents the UK view on standards in Europe and at the international level. It is incorporated by Royal Charter.

Revisions

British Standards are updated by amendment or revision. Users of British Standards should make sure that they possess the latest amendments or editions.

It is the constant aim of BSI to improve the quality of our products and services. We would be grateful if anyone finding an inaccuracy or ambiguity while using this British Standard would inform the Secretary of the technical committee responsible, the identity of which can be found on the inside front cover. Tel: 020 8996 9000. Fax: 020 8996 7400.

BSI offers members an individual updating service called PLUS which ensures that subscribers automatically receive the latest editions of standards.

Buying standards

Orders for all BSI, international and foreign standards publications should be addressed to Customer Services. Tel: 020 8996 9001. Fax: 020 8996 7001.

In response to orders for international standards, it is BSI policy to supply the BSI implementation of those that have been published as British Standards, unless otherwise requested.

Information on standards

BSI provides a wide range of information on national, European and international standards through its Library and its Technical Help to Exporters Service. Various BSI electronic information services are also available which give details on all its products and services. Contact the Information Centre. Tel: 020 8996 7111. Fax: 020 8996 7048.

Subscribing members of BSI are kept up to date with standards developments and receive substantial discounts on the purchase price of standards. For details of these and other benefits contact Membership Administration. Tel: 020 8996 7002. Fax: 020 8996 7001.

Copyright

BSI
389 Chiswick High Road
London
W4 4AL