BS EN 15704:2008

Liming materials — Determination of the breakdown of granulated calcium and calcium/magnesium carbonates under the influence of water

ICS 65.080

BSI British Standards

National foreword

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

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Calcium-/Magnesium-Bodenverbesserungsmittel - Bestimmung des Zerfalls von granulierten Calcium- und Calcium-/Magnesiumcarbonaten unter Wassereinwirkung

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Foreword

This document (EN 15704:2008) 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of an identical text or by endorsement, at the latest by June 2009, and conflicting national standards shall be withdrawn at the latest by June 2009.

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1 Scope

This European Standard specifies a method for the determination of the break down of granulated calcium and calcium /magnesium carbonates under the influence of water.

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 12048, *Solid fertilizers and liming materials — Determination of moisture content — Gravimetric method by drying at (105+/-2)° C (ISO 8190:1992 modified)*

EN 12944-1:1999, *Fertilizers and liming materials — Vocabulary — Part 1: General terms*

EN 12944-2:1999, *Fertilizers and liming materials — Vocabulary — Part 2: Terms relating to fertilizers*

EN 12944-3:2001, *Fertilizers and liming materials — Vocabulary — Part 3: Terms relating to liming materials*

EN 12948, *Liming materials — Determination of size distribution by dry and wet sieving*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 12944-1:1999, EN 12944-2:1999 and EN 12944-3:2001 apply.

4 Principle

The sample is added to water. The mixture is stirred and then screened through a series of test sieves.

NOTE The method is based upon a method described in the German Methods manual II (1985) [1].

5 Apparatus

5.1 Drying oven, capable of maintaining a temperature of 105 °C ± 5 °C;

5.2 Magnetic stirrer, with adjustable rotation, 600 rpm;

5.3 Magnetic stirring rod, 64 mm long (± 5 mm), with a central ring and a diameter of 8 mm (± 1 mm);

5.4 Test sieves, In accordance with ISO 3310-1 with apertures of 100 µm and 315 µm;

NOTE Additional sieve sizes can be added to meet national regulations or the requirements of the consumers.

5.5 Glass beaker, Capacity 2 000 ml and with a diameter of approximately 130 mm and a height of 200 mm.

6 Reagents

6.1 Water, conforming to EN ISO 3696:1995, grade 3.

7 Procedure

7.1 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 [2].

Sample preparation shall be carried out in accordance with EN 1482-2.

7.2 Determination of the moisture content

Determine the moisture content of the sample in accordance with EN 12048 and record the result as mass before drying (m_1) and mass after drying (m_2).

7.3 Breakdown of the granules in water

Weigh about 100 g of the as received test sample (m_3) to the nearest 0,1 g. Measure 1 000 ml of water (6.1) into the glass beaker (5.5). Add the magnetic stirrer rod (5.3) and start the magnetic stirrer (5.2). Adjust the magnetic stirrer to 600 rpm. Add the test portion into the outside area of the stirred water, and not into the middle vortex in the beaker. Stir for 10 min, maintaining the stirrer at 600 rpm.

It is important that the magnetic stirrer avoids any milling action on the test portion.

Assemble the sieves (5.4) with the largest aperture size on the top. Completely wash the contents of the beaker onto the top sieve. Wash the residues on each of the sieves in accordance with the wet sieving method described in EN 12948.

Dry (5.1) the residue on each sieve separately at 105 °C to a constant mass, and weigh to an accuracy of 0,1 g (m_4) after cooling down.

8 Calculation

The mass fraction passing through the respective sieve(s), w_S in percent, is calculated according to the following Equation:

$$w_S = 100 - \frac{m_1 \times m_4 \times 100}{m_2 \times m_3} \quad (1)$$

where

m_1 is the mass in grams of the sample before drying,

m_2 is the mass in grams of the dried sample,

m_3 is the mass in grams of the test portion,

m_4 is the mass in grams of the dried residue remaining on the sieve(s).

The mass of the dried residues (m_4) on the sieves shall be used to express the test results in accordance with the requirements of National Fertilizer Regulations for liming materials.

9 Precision

9.1 Inter-laboratory test

An inter-laboratory test has been carried out in 2006 with 12 participating laboratories. Repeatability and reproducibility were calculated according to ISO 5725-1:1994 [3].

The values derived from this inter-laboratory test may not be applicable to materials and sample matrices other than those given in Table 1.

9.2 Repeatability

The absolute difference between two independent single test results, obtained with the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, will not be more than 5 % of the cases exceed the values of r given in Table 1.

9.3 Reproducibility

The absolute difference between two single test results, obtained with the same method on identical test material in different laboratories by different operators using different equipment, will not be more than 5 % of the cases exceed the values of R given in Table 1.

Table 1 — Mean values, repeatability and reproducibility limits

Material and fineness before granulation	Particle size	$\bar{x}$ % passing	r	R	r % relative	R % relative
Dolomite	100 µm	98,118	1,1551	1,5313	1,1773	1,5607
	315 µm	99,550	0,3341	2,3743	0,3357	2,3850
Chalk fine	100 µm	86,943	1,4084	2,3110	1,6199	2,6581
	315 µm	90,454	7,2218	6,4734	7,9840	7,1566
Chalk coarse	100 µm	50,219	5,6911	20,7561	11,3325	41,3310
	315 µm	54,112	7,7781	24,6821	14,3742	45,6131

10 Test report

The test report shall contain at least the following information:

a) all data necessary for the complete identification of the sample;

b) date of the sampling and the sampling procedure (if known);

c) reference to this European Standard;

d) test results and the units in which the results have been expressed;

e) confirmation of the sieve sizes used in the test;

f) date of the completion of the test;

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] Methodenbuch Band II (1995): *Die Untersuchung von Düngemitteln*, 4. Auflage, Verband Deutscher Landwirtschaftlicher Untersuchungs- und Forschungsanstalten (VDLUFA), Kapitel 6.6, Seiten 1-2: *Bestimmung des Zerfalls von granulierten kohlensauren Kalken unter Feuchtigkeitseinfluss* (Methods Manual II (1995): *Analysis of Fertilizers*, Association of German Agricultural Experimental and Research Stations, Chapter 6.6, Pages 1-2: *Determination of the disaggregation of granulated calcium carbonates ($CaCO_3$, granulated) under the influence of moisture*)

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

[3] ISO 5725-1:1994, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

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