BS EN 12948:2010

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Liming materials — Determination of size distribution by dry and wet sieving

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National foreword

This British Standard is the UK implementation of EN 12948:2010. It supersedes BS EN 12948:2002 which is withdrawn.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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ISBN 978 0 580 63902 9

ICS 65.080

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2010.

Amendments issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 12948

November 2010

ICS 65.080

Supersedes EN 12948:2002

English Version

Liming materials - Determination of size distribution by dry and wet sieving

Amendements minéraux basiques - Détermination de la distribution granulométrique par tamisage par voie sèche ou par voie humide

Calcium-/Magnesium-Bodenverbesserungsmittel - Bestimmung der Korngrößenverteilung durch Trocken- und Nasssiebung

This European Standard was approved by CEN on 2 October 2010.

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Ref. No. EN 12948:2010: E

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Foreword

This document (EN 12948:2010) 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of an identical text or by endorsement, at the latest by May 2011, and conflicting national standards shall be withdrawn at the latest by May 2011.

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 EN 12948:2002.

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

a) the sampling method is not part of the standard. Informative reference to EN 1482-1 added;

b) normative reference to EN 1482-2 concerning sample preparation added;

c) clarification of the method of dispersion of agglomerates in method B added;

d) Bibliography revised.

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Introduction

The dry sieving of powdered material containing individual particles can be carried out quite easily. This method is simple, quick, cheap and enables the determination of the particle size of water-soluble materials. Therefore the dry sieving method should always be used first. However, the sieve apertures can become blocked by sample particles, a phenomenon known as blinding. Blinding is mainly caused by caking and the production of electrostatic charges, particularly on sieves with small apertures. Dry sieving of very wet material can also lead to blinding. These difficulties are not encountered with the wet sieving method, which is applicable to any kind of material such as powders (dry or wet), paste-like products or granules except those containing water-soluble constituents.

In order to ensure the comparability of results, all masses of size fractions are expressed as dry matter.

The approach to methods and principles of sieving refers to existing guidelines and European Regulations ([1] and [2]).

1 Scope

This European Standard specifies two methods for the determination of the particle size distribution of liming materials.

The dry sieving method (method A) is applicable to all liming materials except wet and paste-like products.

Method A is not applicable, if blinding, caking, electrostatic charges or agglomeration occur after pre drying.

The wet sieving method (method B) is applicable to products which are susceptible to blinding, caking, electrostatic charges or agglomeration after pre drying.

Method B can be used to determine the primary particle size distribution of granulated products.

Method B is not applicable to burnt lime and liming materials containing water-soluble constituents.

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 1235:1995, *Solid fertilizers — Test sieving (ISO 8397:1988 modified)*

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)*

ISO 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

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

3 Principle

3.1 Method A

Dry sieving of a liming material with one or more test sieves by hand or using a mechanical sieving machine.

3.2 Method B

Wet sieving of a liming material, if necessary after dispersion under continuous water spraying, by hand or using a mechanical sieving machine. Drying of the different fractions retained on the sieves.

4 Apparatus

Usual laboratory apparatus and in particular 4.1 to 4.6.

4.1 Balance, capable of weighing to the nearest 0,01 g.

4.2 Mechanical shaker (sieving machine), capable of imparting both horizontal and vertical motion to material inside a nest of sieves, fitted with a lid with a water intake and a receiver with a water outlet when used for method B.

NOTE Hand sieving can be carried out instead of mechanical sieving.

4.3 Stainless steel woven wire test sieves, conforming to ISO 3310-1 and of appropriate nominal aperture sizes.

4.4 Stopwatch.

4.5 Soft brush.

4.6 Oven, capable of being controlled at (105 ± 2) °C.

5 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.

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

6 Procedure

6.1 Test portion

The whole laboratory sample shall be divided in equal test portions of at least 100 g. The size of the test portions will vary according to the coarseness of the laboratory sample and shall be in accordance with EN 1235:1995, Table 1, subject to a minimum of 100 g.

6.2 Method A

6.2.1 Preparation of test portions

Dry wet samples if there is a possibility that blinding of the sieves could occur during the sieving.

Dry the wet sample in an oven (4.6) at (105 ± 2) °C for an appropriate period of time (see EN 12048).

Check whether agglomeration occurs after drying by carrying out a preliminary sieving test.

Use method B if agglomeration occurs.

6.2.2 Determination

WARNING — When sieving burnt or hydrated lime products, it is essential that precautions are taken to avoid inhalation and skin contact with the product. It is recommended that operations are carried out under a fume hood and appropriate gloves and eye protection are worn.

6.2.2.1 Carry out at least two single determinations on separate test portions prepared from the same laboratory sample.

6.2.2.2 Select a maximum of seven test sieves (4.3) from the range of principal sizes listed in ISO 565 to cover the range of particle size expected. For granular liming materials perforated plates according to ISO 3310-2 may be used.

Assemble the sieves in ascending order of aperture size on top of the receiver.

Weigh the test portion (6.1) to the nearest 0,01 g per 100 g of test portion, place it on the top sieve and fit the cover.

6.2.2.3 Place the sieve or the assembled nest of sieves on the mechanical shaker (4.2) and shake for exactly 10 min (use a stopwatch (4.4)). Set the mechanical shaker to a medium vibration frequency throughout the sieving.

6.2.2.4 If a nest of sieves is used, remove the sieves from the nest and weigh the quantity retained on each sieve and in the receiver to the nearest 0,01 g. Remove particles caught in the mesh of the sieve by brushing the reverse side of the sieve.

6.2.2.5 If only one sieve is used, discard the undersize fraction that has passed through the sieve. Repeat the sieving process for exactly 1 min. If more than 0,2 g passes through the sieve, repeat the procedure as many times as necessary. Remove particles caught in the mesh of the sieve by brushing the reverse side of the sieve. Weigh the oversize fraction.

6.3 Method B

6.3.1 General

Use an additional test portion to determine the moisture content in accordance with EN 12048.

Carry out at least two single determinations on separate test portions prepared from the same laboratory sample.

6.3.2 Determination

6.3.2.1 Non-granulated and non-agglomerated products

6.3.2.1.1 Select a maximum of seven test sieves (4.3) from the range of principal sizes listed in ISO 565 to cover the range of particle size expected.

Assemble the sieves in ascending order of aperture size on top of the receiver.

Weigh the test portion (6.1) to the nearest 0,01 g per 100 g of test portion, place it on the top sieve and fit the lid with the intake for water.

6.3.2.1.2 Place the assembled nest of sieves on the mechanical shaker (4.2) and shake under a continuous water flow of 2,0 l/min to 2,5 l/min for exactly 10 min (use a stopwatch (4.4)).

Set the mechanical shaker to a medium vibration frequency throughout the sieving.

6.3.2.1.3 Remove the sieves from the mechanical shaker and rinse the residues of each sieve quantitatively into separate 250 ml pre-weighed beakers.

Decant or pipette most of the water on the top of the material, ensuring that no material is spilled.

Dry each of the oversize fractions in an oven (4.6) at (105 ± 2) °C and then weigh each fraction separately (see EN 12048).

6.3.2.2 Granulated and agglomerated products

Weigh the test portion (6.1) to the nearest 0,01 g per 100 g of test portion and transfer it into a 800 ml beaker.

Add approximately 500 ml of water. Stir the granules for 10 min by means of a mechanical stirrer with a rotational speed not exceeding 800 min^{-1}. Avoid grinding.

Rinse out the sample completely on to the top sieve and fit the lid with the intake for water.

Continue according to 6.3.2.1.2 and 6.3.2.1.3.

If the sum of individual masses is less than 95 % of the original mass, it should be assumed that the sample contains water-soluble constituents.

7 Expression of results

7.1 Method A

7.1.1 Record the masses of the fractions retained on the sieves and the receiver (see 6.2.2.4 and 6.2.2.5).

7.1.2 Calculate each mass fraction as a percentage of the mass of the test portion (see EN 1235:1995, Annex ZA) according to Equation (1).

$$w_{n,1} = \frac{m_{n,1} \times 100}{m_{t,1}} \quad (1)$$

where

$w_{n,1}$ is the mass fraction retained on sieve n or in the receiver, in percent;

$m_{n,1}$ is the mass retained on sieve n or in the receiver, in grams;

$m_{t,1}$ is the mass of the test portion, in grams.

7.2 Method B

7.2.1 Record the masses of the fractions retained on the sieves (see 6.3.2.1.3).

7.2.2 Calculate each mass fraction as a percentage of the mass of the test portion (see EN 1235:1995, Annex ZA) according to Equation (2).

$$w_{n,2} = \frac{m_{n,2} \times 100}{m_{t,2}} \quad (2)$$

where

$w_{n,2}$ is the mass fraction retained on sieve n, in percent;

$m_{n,2}$ is the mass retained on sieve n after drying, in grams;

$m_{t,2}$ is the mass of the test portion expressed on a dry matter basis (see EN 12048), in grams.

8 Precision

8.1 General

The precision of the methods were established by an inter-laboratory trial carried out in 1999 in accordance with ISO 5725.

The values obtained for repeatability limit and reproducibility limit are expressed for the 95 % probability level and shall not be applicable to particle size ranges other than those given.

NOTE The repeatability limits and reproducibility limits obtained from the inter-laboratory trial for each product being tested are given in Annex A.

8.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within the shortest feasible time interval will exceed the repeatability limit (r) on average not more than once in 20 cases in the normal and correct operation of the method.

The values are:

— for method A: r = 6,50 % at a fineness of less than 1 mm;

— for method B: r = 7,64 % at a fineness of less than 1 mm.

8.3 Reproducibility

The absolute difference between two independent single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment will exceed the reproducibility limit (R) on average not more than once in 20 cases in the normal and correct operation of the method.

The values are:

— for method A: R = 13,90 % at a fineness of less than 1 mm;

— for method B: R = 13,10 % at a fineness of less than 1 mm.

9 Test report

The test report shall contain at least the following information:

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

b) a reference to this European Standard;

c) the method (method A or method B) and the type of sieve (wire cloth or perforated plate) used;

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

e) any particular points observed in the course of the test;

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

Annex A
(informative)

Results of an inter-laboratory trial to determine the size distribution by dry and wet sieving

An inter-laboratory trial was organized in 1999 under the auspices of the European Committee for Standardization to obtain precision data for the methods specified in this European Standard.

In this trial eight laboratories from five participating countries determined the size distribution by dry and wet sieving of five types of product. The values derived from this inter-laboratory trial for the repeatability limit and reproducibility limit of each product being tested are given in Tables A.1 and A.2.

Table A.1 — Repeatability limits and reproducibility limits derived from inter-laboratory trial for method A

Product type	Dry sieving method		
	Fineness	Repeatability limit r	Reproducibility limit R
	mm	%	%
Magnesium limestone, coarse	0,16 to 1,0	6,2	19,7
Dolomite, fine	less than 0,16	5,0	8,1
Dolomite, coarse	0,16 to 1,0	14,0	21,5
Blast furnace slag	less than 0,16	0,8	6,3
NOTE Results for chalk, coarse were not included in the calculation of r and R.			

Table A.2 — Repeatability limits and reproducibility limits derived from inter-laboratory trial for method B

Product type	Wet sieving method		
	Fineness	Repeatability limit r	Reproducibility limit R
	mm	%	%
Magnesium limestone, coarse	0,16 to 1,0	4,4	12,0
Dolomite, fine	less than 0,16	4,3	7,2
Dolomite, coarse	less than 0,16	9,0	15,9
Chalk, coarse	less than 0,16	20,0	28,2
Blast furnace slag	less than 0,16	0,5	2,2

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[2] VDLUFA, *Manual II of analyzing methods for fertilizers* (VDLUFA-Verlag, Bismarckstraße 41 A, D-64293 Darmstadt)

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

[4] ISO 5725 (all parts), *Accuracy (trueness and precision) of measurement methods and results*

[5] ISO 3310-2, *Test sieves — Technical requirements and testing — Part 2: Test sieves of perforated metal plate*

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