INTERNATIONAL
STANDARD

ISO
20977

First edition
2018-12

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

Amendements minéraux basiques — Détermination de la distribution granulométrique par tamisage à sec ou à l'état humide

Reference number
ISO 20977:2018(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 134, *Fertilizers, soil conditioners and beneficial substances*.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The dry sieving of powdered material containing individual particles can be carried out quite easily. This simple, quick and cheap method 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.

This document is based on EN 12948:2002.

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

1 Scope

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

Method A (the dry sieving method) 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 drying.

Method B (the wet sieving method) is applicable to products which are susceptible to blinding, caking, electrostatic charges or agglomeration after 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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.

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*

ISO 8397:1988, *Solid fertilizers and soil conditioners — Test sieving*

ISO 14820-1, *Fertilizers and liming materials — Sampling and sample preparation — Part 1: Sampling*

EN 12048, *Solid fertilisers and liming materials — Determination of moisture content — Gravimetric method by drying at (105 ± 2) °C*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

4.1 Suitable methods

Table 1 gives the recommended methods for different liming materials.

In case of different options, see remarks.

Table 1 — Suitable methods for size distribution determination

Material type	Presentation	Moisture	Water soluble	Size range on the market	Suitable method(s)			Remarks
	Powder/ Defined/ Granulated/ Suspension/ Screened	Dry/Wet	Yes/No/ Partially		Dry sieving (method A)	Wet sieving (method B)	Laser diffraction (not standardized)	
Limestone/ dolomite	Powder	Dry	No	0/10 µm to 0/63 µm	—	—	x	
	Powder	Dry	No	0/63 µm to 0/100 µm	x	—	x	
	Powder	Dry	No	0/100 µm to 0/5 mm	x	optional	—	
	Powder	Wet	No	0/100 µm to 0/5 mm	optional	x	—	after drying for dry sieving as received for wet sieving
	Defined	Dry	No	100 µm/x to y/5 mm	x	optional	—	
	Granulated	Dry	No	2/7 mm	x	—	—	before breakdown
	Granulated	Dry	No	0/63 µm to 0/1 mm (elementary particles)	x	optional 1	optional 2	1 and 2: after breakdown 2: up to 200 µm
	Suspension	Wet	No	0/10 µm to 0/250 µm	optional	—	x	fraction coarser than 100 µm
Chalk (soft material)	Powder	Wet	No	0/5 mm to 0/ 50 mm	—	x	—	as received
	Powder	Dry	No	0/63 µm to 0/1 mm	x	optional	—	
Burnt lime	Powder	Dry	Partially	0/1 mm to 0/3 mm	x	—	—	
	Screened	Dry	Partially	1/x mm to y/8 mm	x	—	—	
Hydrated lime	Powder	Dry	Partially	0/50 µm to 0/200 µm	x	—	optional	in alcohol
	Suspension	Wet	Partially	—	—	—	—	no need
BOF slags (blast oxygen furnace)	Powder	Dry	Partially	up to 1 mm	x	—	optional	in alcohol
	Powder	Wet	Partially	up to 4 mm	x	—	—	after drying

Key

x: preferred method

optional: may be used

—: not applicable

Table 1 *(continued)*

Material type	Presentation	Moisture	Water soluble	Size range on the market	Suitable method(s)			Remarks
	Powder/ Defined/ Granulated/ Suspension/ Screened	Dry/Wet	Yes/No/ Partially		Dry sieving (method A)	Wet sieving (method B)	Laser diffraction (not standardized)	
Sugar factory lime	Powder	Dry	No	very fine particles	x	—	x	
	Powder	Wet	No	very fine particles	x	—	x	after drying
	Suspension	Wet	No	very fine particles	—	—	x	
Ashes	Powder	Dry	Partially	up to 5 mm	x	—	—	
	Powder	Wet	Partially	up to 5 mm after preliminary sieving	x	—	—	after drying
	Granulated	Dry	Partially	2/7 mm	x	—	—	

Key

x: preferred method

optional: may be used

—: not applicable

In some countries, very fine materials (finer than 50 µm) can be found (such as calcium carbonate suspensions).

For such products, measurement by sieving is not suitable:

Fine particles block the sieve holes and the results cannot be considered as representative.

In this situation, only the laser granulometry measurement is suitable.

However, this measurement is not standardized because the results (xx percent passing yy mm) depend on a material matrix and on a device calculation algorithm which is specific to each device provider. Usually, for laser granulometry, particles are dispersed in water. When the product is totally or partially water soluble, alcohol should be used but in such a case (as burnt or hydrated lime), the agricultural product reactivity is much better than for insoluble products (carbonates) and fineness generally does not matter.

4.2 Method A (dry sieving)

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

4.3 Method B (wet sieving)

Dispersion of agglomerated or granulated liming materials with tap water.

Wet sieving of the dispersed liming materials under continuous water spraying by hand or using a mechanical sieving machine. Drying of the different fractions retained on the sieves.

5 Apparatus

Usual laboratory apparatus and, in particular, the following.

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

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

Hand sieving may be carried out instead of mechanical sieving.

5.3 Stainless steel woven wire test sieves, conforming to ISO 3310-1 and of nominal aperture sizes covering the granulometry range of the product.

5.4 Stopwatch.

5.5 Soft brush.

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

6 Sampling

Sample the liming material in accordance with ISO 14820-1.

7 Procedure

7.1 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 ISO 8397:1988, Table 1, subject to a minimum of 100 g.

7.2 Method A (dry sieving)

7.2.1 Preparation of test portions

Dry wet samples if blinding of the sieves is likely to occur during the sieving.

Dry the wet sample in an oven (5.6) at (105 ± 2) °C for a period of time specified in EN 12048.

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

Use method B if agglomeration occurs.

7.2.2 Determination

WARNING — When sieving burnt or hydrated lime products, precautions shall be taken to avoid inhalation and skin contact with the product. Operations should be carried out under a fume hood and appropriate gloves should be worn.

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

7.2.2.2 Select a maximum of seven test sieves (5.3) 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 (7.1) to the nearest 0,01 g per 100 g of test portion, place it on the top sieve and fit the cover.

7.2.2.3 Place the sieve or the assembled stack of sieves on the mechanical shaker (5.2) and shake for exactly 10 min [use a stopwatch (5.4)].

7.2.2.4 If a stack of sieves is used, remove the sieves from the stack 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.

7.2.2.5 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.

7.3 Method B (wet sieving)

7.3.1 General

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

Prior to measurement, check that the tap water pH is higher than 6,4 to avoid possible reaction of fine particles of carbonates.

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

7.3.2 Determination

7.3.2.1 Non-granulated and non-agglomerated products

7.3.2.1.1 Select a maximum of seven test sieves (5.3) 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 (7.1) 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.

7.3.2.1.2 Place the assembled stack of sieves on the mechanical shaker (5.2) and shake under a continuous water flow of 2,0 l/min to 2,5 l/min for exactly 10 min (use a stopwatch (5.4)).

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

7.3.2.1.3 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 (5.6) at (105 ± 2) °C and then weigh each fraction separately (see EN 12048).

7.3.2.2 Granulated and agglomerated products

7.3.2.2.1 Weigh the test portion (7.1) to the nearest 0,01 g per 100 g of test portion and transfer it into a 800 ml beaker.

7.3.2.2.2 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.

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

7.3.2.2.4 Continue according to 7.3.2.1.2 and 7.3.2.1.3.

8 Expression of results

8.1 Method A

8.1.1 Record the masses of the fractions retained on the sieves and the receiver (see 7.2.2.4 and 7.2.2.5).

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

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

where

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

$m_{n,1}$ 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.

8.2 Method B

8.2.1 Record the masses of the fractions retained on the sieves (see 7.3.2.1.3).

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

$$w_{n,2} = \frac{m_{n,2} \times 100}{m_{\mathrm{t},2}} \tag{2}$$

where

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

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

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

9 Precision

9.1 General

The precision of the methods was established by an interlaboratory trial carried out in 1999 in accordance with ISO 5725[1].

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

The repeatability limits and reproducibility limits obtained from the interlaboratory trial for each product and size fraction being tested are given in Annex A.

9.2 Repeatability

The absolute difference between two independent single test results (percentage passing a given size fraction), 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 average values are (15 levels retained):

- for method A (dry sieving):
  - r = 4,0 at a fineness of less than 3,15 mm and at least 5 % passing;

---

[1] Withdrawn.

  - r % = 20,5 % at a fineness of less than 3,15 mm and at least 5 % passing;
- for method B (wet sieving):
  - r = 3,6 at a fineness of less than 3,15 mm and at least 5 % passing;
  - r % = 25,2 % at a fineness of less than 3,15 mm and at least 5 % passing.

9.3 Reproducibility

The absolute difference between two independent single test results (percentage passing a given size fraction), 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 average values are (15 levels retained):

- for method A (dry sieving):
  - R = 7,8 at a fineness of less than 3,15 mm and at least 5 % passing;
  - R % = 37,2 % at a fineness of less than 3,15 mm and at least 5 % passing;
- for method B (wet sieving):
  - R = 5,7 at a fineness of less than 3,15 mm and at least 5 % passing;
  - R % = 39,7 % at a fineness of less than 3,15 mm and at least 5 % passing.

10 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 document, i.e. ISO 20977:2018;

c) the method used (method A or method B);

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 operation not specified in the method or regarded as optional which might have affected the results;

g) the date of the test.

Annex A
(informative)

Results of an interlaboratory trial to determine the size distribution by dry and wet sieving

An interlaboratory trial was organized in 1999 by CEN/TC 260/WG 3 to obtain precision data for the methods specified in this document.

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 interlaboratory trial for the repeatability limit and reproducibility limit of each product being tested are given in Tables A.1 and A.2.

Though all five materials were tested for six different granulometry ranges and reported below, the values of r and *R* obviously depend on the granulometry range.

— *r* and *R* values are much higher when the percentage passing a sieve is very low, i.e. less than 1 %. This method does not give accurate results and should not be used when a size fraction is lower than 5 %. These levels (less than 5 %) were removed for the general calculation of *r* and *R*.

— *r* and *R* values are much higher when the granulometry range is coarser than 3,15 mm because getting a representative sample of a coarse material is very difficult. These levels (coarser than 3,15 mm) were removed for the general calculation of *r* and *R*.

In the following tables, only the clear lines were taken into account for general *r* and *R* calculation. The grey lines were not taken into account.

Table A.1 — Repeatability limits and reproducibility limits derived from interlaboratory trial for method A (dry sieving)

Sieve material	Level	Mean % passing	Number of laboratories	*r*	*R*	*r* %	*R* %
Magnesian limestone	>6,3 mm	0,138	7	0,851	0,948	—	—
	3,15 mm 6,3 mm	1,046	6	1,182	1,383	—	—
	2 mm 3,15 mm	5,150	6	1,799	2,336	34,929	45,353
	1 mm 2 mm	22,205	6	2,967	3,347	13,362	15,074
	0,160 mm 1 mm	46,192	7	2,857	9,077	6,184	19,651
	<0,160 mm	25,648	7	4,327	5,236	16,871	20,417
Dolomite fine	>6,3 mm	0,000	7	0,000	0,000	—	—
	3,15 mm 6,3 mm	0,000	7	0,000	0,000	—	—
	2 mm 3,15 mm	0,001	7	0,006	0,008	—	—
	1 mm 2 mm	0,027	4	0,006	0,114	23,529	—
	0,160 mm 1 mm	9,515	7	4,505	8,323	47,341	87,466
	<0,160 mm	90,283	7	4,558	7,352	5,049	8,143
Dolomite coarse	>6,3 mm	0,054	7	0,646	0,760	—	—
	3,15 mm 6,3 mm	6,414	7	1,583	8,977	24,683	—
	2 mm 3,15 mm	16,429	7	6,240	5,601	37,981	34,091
	1 mm 2 mm	20,590	7	1,555	10,188	7,553	49,479
	0,160 mm 1 mm	22,064	7	3,086	4,749	13,988	21,524
	<0,160 mm	34,543	7	5,338	10,365	15,452	30,006
Chalk coarse	>6,3 mm	12,264	7	6,953	16,630	56,697	—
	3,15 mm 6,3 mm	15,165	6	4,064	6,472	26,796	42,677
	2 mm 3,15 mm	10,436	6	1,930	4,217	18,494	40,411
	1 mm 2 mm	13,062	7	2,041	8,368	15,622	64,064
	0,160 mm 1 mm	33,959	7	8,269	19,101	24,349	56,247
	<0,160 mm	21,082	6	10,443	12,601	49,536	59,770

Table A.1 *(continued)*

Sieve material	**Level**	**Mean** % passing	**Number of laboratories**	***r***	***R***	***r*** %	***R*** %
Blast furnace slag	>6,3 mm	0,000	3	0,000	0,000	—	—
	3,15 mm 6,3 mm	0,283	3	0,094	1,392	33,294	—
	2 mm 3,15 mm	0,350	3	0,094	1,718	26,952	—
	1 mm 2 mm	0,700	3	0,133	3,434	19,058	—
	0,160 mm 1 mm	0,258	3	0,156	1,276	60,555	—
	<0,160 mm	98,603	5	0,770	6,187	0,781	6,275