BRITISH STANDARD

BS EN
1367-2:2009

Tests for thermal and weathering properties of aggregates

Part 2: Magnesium sulfate test

ICS 91.100.15

BSI
British Standards

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National foreword

This British Standard is the UK implementation of EN 1367-2:2009. It supersedes BS EN 1367-2:1998 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/502/6, Test methods.

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

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Essais pour déterminer les propriétés thermiques et l'altérabilité des granulats - Partie 2 : Essai au sulfate de magnésium

Prüfverfahren für thermische Eigenschaften und Verwitterungsbeständigkeit von Gesteinskörnungen - Teil 2: Magnesiumsulfat-Verfahren

This European Standard was approved by CEN on 19 September 2009.

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Foreword

This document (EN 1367-2:2009) has been prepared by Technical Committee CEN/TC 154 “Aggregates”, the secretariat of which is held by BSI.

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of an identical text or by endorsement, at the latest by April 2010, and conflicting national standards shall be withdrawn at the latest by April 2010.

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 1367-2:1998.

This European Standard is part of the EN 1367 series of European Standards under the general title: "Tests for thermal and weathering properties of aggregates". The other parts are:

Part 1: Determination of resistance to freezing and thawing;

Part 2: Magnesium sulfate test;

Part 3: Boiling test for “Sonnenbrand basalt";

Part 4: Determination of drying shrinkage;

Part 5: Determination of resistance to thermal shock;

Part 6: Determination of resistance to freezing and thawing in the presence of salt (NaCl).

Test methods for other properties of aggregates will be covered by Parts of the following European Standards:

EN 932 Tests for general properties of aggregates;

EN 933 Tests for geometrical properties of aggregates;

EN 1097 Tests for mechanical and physical properties of aggregates;

EN 1744 Tests for chemical properties of aggregates;

EN 13179 Tests for filler aggregate used in bituminous mixtures.

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

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1 Scope

This European Standard describes the reference method used for type testing and in cases of dispute for assessing how an aggregate behaves when subjected to the cyclic action of immersion in magnesium sulfate, followed by oven drying. For other purposes, in particular factory production control, other methods may be used provided that an appropriate working relationship with the reference method has been established.

NOTE The majority of aggregates can be tested for performance using this method. Precision has been established for the rock types listed in Annex A. The test may not be suitable for all rock types and reservations have been expressed elsewhere in respect of some carbonate aggregates and some aggregates having a high proportion of magnesium bearing materials or of cryptocrystalline quartz.

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 932-1, *Tests for general properties of aggregates – Part 1: Methods for sampling*

EN 932-2, *Tests for general properties of aggregates – Part 2: Methods for reducing laboratory samples*

EN 932-3, *Tests for general properties of aggregates – Part 3: Procedure and terminology for simplified petrographic description*

EN 932-5, *Tests for general properties of aggregates – Part 5: Common equipment and calibration*

EN 933-2, *Tests for geometrical properties of aggregates – Part 2: Determination of particle size distribution – Test sieves, nominal size of apertures*

ISO 649-1, *Laboratory glassware – Density hydrometers for general purposes – Part 1: Specification*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
laboratory sample
sample intended for laboratory testing

3.2
test specimen
sample used in a single determination when a test method requires more than one determination of a property

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3.3
test portion
sample used as a whole in a single test

4 Principle

Two test specimens of aggregate in the size range 10 mm to 14 mm are subjected to five cycles of immersion in a saturated solution of magnesium sulfate, followed by oven drying at (110 ± 5) ºC. This subjects the test specimens of aggregate to the disruptive effects of the repeated crystallization and rehydration of magnesium sulfate within the pores of the aggregate. The degradation arising from the disruptive effects is measured by the extent to which material finer than 10 mm in particle size is produced.

NOTE The procedure can also be applied to other aggregate fractions (Annex B) or combinations of fractions (Annex C).

5 Sampling

The laboratory sample to be used for the test shall be taken in accordance with EN 932-1.

6 Apparatus

Unless otherwise stated, all apparatus shall conform to the general requirements of EN 932-5.

6.1 *Test sieves,* conforming to EN 933-2, of 10 mm and 14 mm size.

6.2 *Balance,* of 2 kg capacity, accurate to 0,1 g.

6.3 *Brass or stainless steel mesh baskets,* at least two, for immersing test specimens in the solution. A suitable design is shown in Figure 1.

6.4 *Containers,* such that the baskets listed in 6.3 can be readily placed in and out, complying with the minimum separation clearances specified in 9.1, and with a volume at least five times the volume of the immersed aggregate.

6.5 *Tank or tank rooms,* capable of maintaining the temperature of the solution inside the containers at (20 ± 2) ºC during the immersion stages.

6.6 *Ventilated drying oven with forced air circulation*, of adequate capacity. The oven shall be capable of being controlled at (110 ± 5) ºC.

6.7 *Density hydrometer,* complying with ISO 649-1 and graduated at 20 ºC for medium surface tension 55 mN/m to measure densities in the range of 1,284 g/ml to 1,300 g/ml to an accuracy of 0,001 g/ml.

6.8 *Desiccator,* large enough to contain at least two of the baskets listed in 6.3.

6.9 *Thermometer,* of range 0 ºC to 120 ºC and accurate to 1 ºC.

6.10 *Timing device,* such that the full range of timed periods can be measured to an accuracy of ±1 min.

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7 Reagents

7.1 *Distilled, or deionized water.*

7.2 *Barium chloride 5 % solution,* made by dissolving 5 g of barium chloride in 100 ml of distilled water.

7.3 *Saturated solution of magnesium sulfate,* which may be made by dissolving magnesium sulfate heptahydrate of reagent grade in distilled or deionized water.

7.3.1 Prepare the solution by slowly adding 1 500 g of the crystalline salt to each litre of water. A minimum of 3 l is required for each test.

NOTE It is advisable to prepare a second batch of solution using the above procedure as a reserve, in case of solution failure during the test procedure, see 9.3.

During preparation, maintain the temperature of the solution between 25 ºC and 30 ºC and stir thoroughly during the addition of the crystals. After preparation, lower the temperature to (20 ± 2) ºC, and maintain at this temperature for (48 ± 1) h.

7.3.2 Prior to use, check that the solution has achieved a density of (1,292 ± 0,008) g/ml by decanting a portion of the solution into a glass jar, measuring the density with the hydrometer, and returning the solution to the container.

8 Preparation of test specimens

8.1 Reduce the laboratory sample in accordance with EN 932-2 to produce two test specimens of sufficient mass such that each will produce a minimum of 500 g of the 10 mm to 14 mm size when processed as specified in 8.3.

NOTE Guidance on testing other fractions is given in Annex B and on testing all fractions in Annex C.

8.2 Dry each test specimen in the oven at (110 ± 5) ºC for (24 ± 1) h, and allow to cool in the desiccator to laboratory temperature.

8.3 Sieve each test specimen using the 10 mm and 14 mm sieves to reject oversize and undersize to give a mass of approximately 500 g each.

8.4 Wash each test specimen with distilled water until free from dust, allow to drain and dry in the oven as specified in 8.2.

8.5 Repeat the sieving as specified in 8.3, to ensure that only material in the 10 mm to 14 mm range is used.

8.6 Weigh out between (420 ± 0,1) g and (430 ± 0,1) g from each test specimen and record the masses (M_1). Transfer the test specimens to two labelled mesh baskets. Avoid shaking the baskets at all subsequent stages to minimize any loss by abrasion.

9 Procedure

9.1 Suspend each basket in a container holding the saturated magnesium sulfate solution at (20 ± 2) °C so that the top of the aggregate is completely immersed to a depth of 20 mm for a period of (17 ± 0,5) h. A minimum of 20 mm clearance shall be maintained between each basket, container sides and accumulated salt cakes.

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Take particular care to ensure that no whole piece of aggregate is lost from any basket at any stage. Cover the container to avoid evaporation and contamination.

9.2 After immersion, remove each basket from the solution and drain for (2 ± 0,25) h then cover the container immediately. Dry each basket as in 8.2 and cool to laboratory temperature for (5 ± 0,25) h.

9.3 Prior to the next immersion, break up any salt cake which may have accumulated at the bottom of the container, stir the solution thoroughly and allow to settle for 30 min. Check the density of the solution in the container as specified in 7.3.2. If the density is outside the specified range, replace the solution with unused saturated solution as prepared in accordance with 7.3.1.

Where severe disintegration of aggregate occurs during immersion, the measured densities of the solution may be inaccurate due to suspended fines or ion-exchange effects. Under these circumstances, replace with unused solution.

9.4 Repeat the process specified in 9.1 to 9.3 for five cycles, each cycle taking (48 ± 2) h.

NOTE If it is necessary to interrupt the test, for example at weekends, it can be done at the end of the drying stage. The containers should be kept at laboratory temperature. A total interruption of up to 72 h is possible.

9.5 After cooling at the completion of the five cycles as specified in 9.2, wash the aggregate in each basket with tap water until the washings are free from magnesium sulfate.

NOTE This can be verified by testing a 10 ml aliquot of the washings with a few drops of barium chloride solution for turbidity, and comparing this with the turbidity of an equal volume of fresh tap water similarly treated.

9.6 Dry each test specimen as specified in 8.2. Hand sieve on the 10 mm sieve and record the mass (M_2) of the aggregate retained on the sieve to the nearest 0,1 g.

10 Calculation and expression of results

10.1 Calculate the magnesium sulfate value (*MS*) in percentage by mass for each test specimen in accordance with the following equation, recording each value to the first decimal place:

$$MS = 100\,\frac{(M_1 - M_2)}{M_1}$$

where:

M_1 is the initial mass of the test specimen, to the nearest ± 0,1 g;

M_2 is the final mass of aggregate retained on the 10 mm sieve, to the nearest ± 0,1 g.

10.2 Calculate and record the mean of the two results obtained to the nearest whole number.

11 Test report

The test report shall include the following information:

a) reference to this European Standard;

b) the magnesium sulfate value (*MS*) in accordance with 10.2;

c) the individual magnesium sulphate values of the two specimens (for each aggregate fraction tested);

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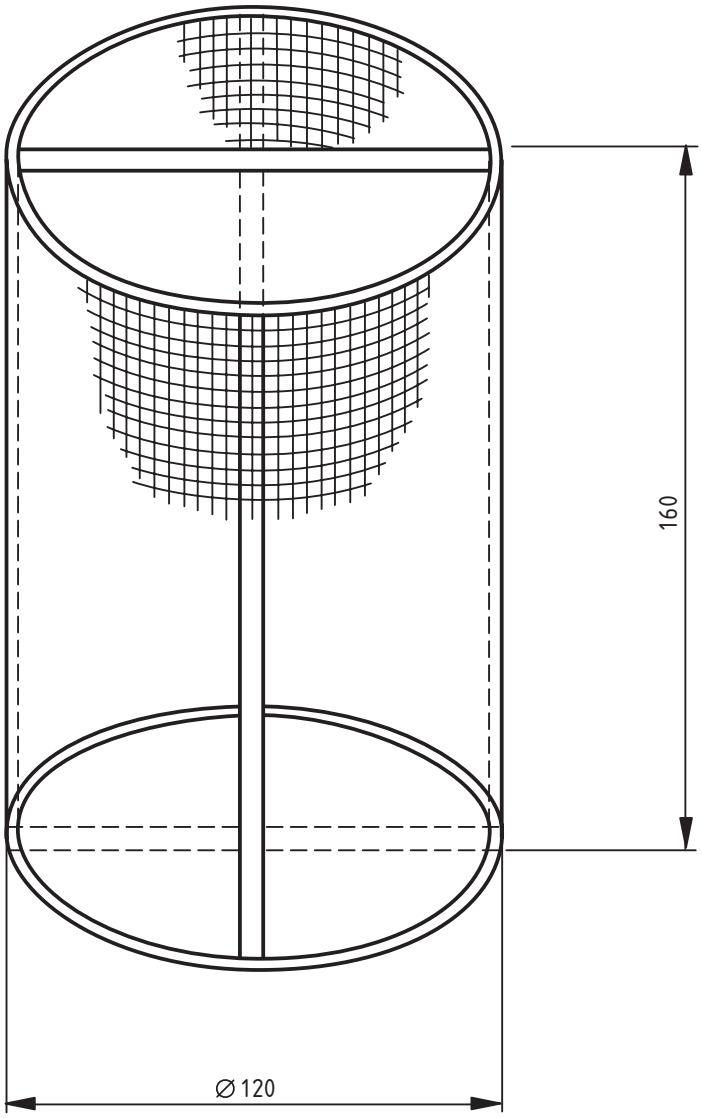
d) sample identification, including source, type and size of the submitted aggregate;

e) size fraction (or fractions) tested;

f) a copy of the certificate of sampling, if available.

Dimensions in millimetres



Mesh size 4

NOTE The dimensions are not critical and intended only as a guide. The main requirements are that the baskets should be large enough to allow the specimens to be totally immersed and to permit free circulation of the magnesium sulfate solution. The mesh should be strong enough to hold the aggregate but not so coarse that particles can pass through at the start of the test.

Figure 1 — Example of typical basket for magnesium sulfate testing

Annex A
(informative)

Precision

An experiment involving 11 laboratories was carried out in 1985/86.

Materials consisting of 10 t lots provided 100 kg laboratory samples. Two test portions were then produced from each laboratory sample.

Two laboratory averages were deleted as outliers. The sample variabilities which result from the sampling and sample reduction operations are proportional to *V*r and *V*s (as defined in ISO 5725-2) according to sampling error and sample reduction error as specified in EN 932-2.

The precision data from Table A.1 are approximately represented by the following simplified equations where x = *MS*.

$$r_1 = \sqrt{\{0{,}18\, x\, (100 - x)\}}$$

$$R_1 = \sqrt{\{0{,}31\, x\, (100 - x)\}}$$

$$R_2 = \sqrt{\{0{,}34\, x\, (100 - x)\}}$$

These may be used to interpolate values of r_1, R_1 and R_2 for levels of percentage retained between those which appear in Table A.1.

Table A.1 — Precision data for the magnesium sulfate soundness value
x = average *MS* at statistical levels

x %	r_1 %	R_1 %	R_2 %	vV_r %	vV_L %	vV_s %	**Rock type used in precision exercise**
70,9	7,3	19,6	19,8	2,61	6,47	1,06	Oolitic limestone
38,2	10,7	17,8	19,4	3,80	5,07	2,75	Lithic sandstone
19,1	9,1	16,4	19,5	3,25	4,86	0,59	Quartz dolerite
9,0	5,2	8,2	8,2	1,66	2,27	0,00	Olivine basalt
5,5	3,7	5,7	5,9	1,33	1,55	0,53	Shelly limestone
4,4	4,7	6,8	9,7	1,69	1,75	2,48	Olivine basalt
3,6	3,0	4,1	4,8	1,01	0,99	0,92	Lithic sandstone
3,2	1,5	2,6	2,8	0,53	0,75	0,36	Quartz dolerite

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Annex B
(informative)

Testing aggregates outside the size and range 10 mm to 14 mm

Table B.1 — Recommended test sieves, mesh baskets and mass of test specimens

Size fraction	Mass of test specimen	Test sieve		Mesh baskets		
		Passing	Retained	Mesh size	Height	Diameter
mm	g	mm	mm	mm	mm	mm
Larger than 14,00	800 to 830	28,00	20,00	3,35	160	120
	600 to 630	20,00	14,00	3,35	160	120
Smaller than 10,00	300 to 310	10,0	6,30	1,18	120	95
	200 to 210	6,30	5,00	1,18	120	95
	200 to 210	5,00	3,35	0,60	120	95
	200 to 210	3,35	2,36	0,60	120	95
	100 to 110	2,36	1,18	0,15	80	65
	100 to 110	1,18	0,60	0,15	80	65
	100 to 110	0,60	0,30	0,15	80	65
Smaller than 10,00 for use in assessing complete grading	300 to 310	10,0	6,30	1,18	120	95
	200 to 210	6,30	4,00	0,60	120	95
	200 to 210	4,00	2,00	0,60	120	95
	100 to 110	2,00	1,00	0,15	80	65
	100 to 110	1,00	0,50	0,15	80	65
	100 to 110	0,50	0,25	0,15	80	65

Annex C
(informative)

Assessment of complete grading

C.1 Apparatus

C.1.1 General

Apparatus specified in 6.1 to 6.10 together with the following:

C.1.2 *Sieves,* conforming to EN 933-2, of 20 mm, 6.3 mm, 4 mm, 2 mm, 1 mm, 0.5 mm and 0.25 mm sizes.

C.1.3 *Brass or stainless steel mesh baskets,* at least two, for immersing test specimens of fractions other than 10 mm to 14 mm in the solution. A suitable design is shown in Figure 1 but with the relevant dimensions given in Annex A.

C.2 Reagents

Reagents specified in 7.1 to 7.3.

C.3 Preparation of test specimens

C.3.1 Reduce the laboratory sample in accordance with 8.1 except that the two test specimens shall be of sufficient mass such that each will produce a minimum of 500 g of the 10 mm to 14 mm size together with the masses specified in Table B.1 of all relevant fractions when processed as specified in accordance with C.3.3.

C.3.2 Dry each test specimen in accordance with 8.2.

C.3.3 The particle size distribution of the test portion shall be determined in accordance with 8.3 using the 20 mm, 10 mm, 6.3 mm, 4 mm, 2 mm, 1 mm, 0,5 mm and 0,25 mm sieves. For coarse aggregates, the particles retained on the 20 mm sieve and passing the 1 mm sieve shall be discarded and not taken into account in the calculation of the test result. The remainder of the reduced sample shall be considered as the test portion. The particle size distribution shall be recorded giving the percentage of the mass of the test portion retained between each pair of sieves, together with that passing the 0,25 mm sieve for fine aggregates, to the nearest whole number.

C.3.4 Those fractions retained whose proportions are less than 5 % by mass of the test portion shall be discarded. Nevertheless, the proportions that the discarded fractions represent shall be taken into account in the calculation of the test result.

C.3.5 One test specimen, of mass in accordance with Table B.1, shall be taken out of each fraction retained after completion of C.3.4. If there is insufficient material in any of these fractions to provide a test specimen of the required size, the procedure shall be repeated starting from C.3.1. The particle size distribution recorded shall be that obtained from all the material sieved out.

C.3.6 Where one or more of the test fractions exceeds the required mass for that fraction in Annex B, the sample shall be reduced to produce the correct mass.

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C.3.7 Wash each test specimen in accordance with 8.4.

C.3.8 Repeat the sieving specified in C.3.3, to ensure that only material in the relevant fraction is used.

C.3.9 Weigh out the relevant mass of test specimens from Table B.1 to an accuracy of ± 0,1 g and record the masses (M_1). Transfer the test specimens to two labelled mesh baskets. Avoid shaking the baskets at all subsequent stages to minimize any loss by abrasion.

C.4 Procedure

Undertake 9.1 to 9.6 for each test specimen, replacing "*10 mm sieve*" in 9.6 by the sieve relevant to the lower size of the aggregate fraction.

C.5 Calculation and Expression of Test Results

C.5.1 Calculate the magnesium sulfate value of each test specimen in accordance with 10.1, replacing "*10 mm sieve*" by the sieve relevant to the lower size of the aggregate fraction.

C.5.2 Fractions not tested because they represent less than 5 % by mass of the test portion shall be assumed to have a magnesium sulfate value equivalent to:

a) the mean of the magnesium sulfate value found by the tests on specimens of the two fractions immediately adjacent to it in size; or

b) the magnesium sulfate value found by the test on a specimen of the fraction, either larger or smaller, immediately adjacent to it if only one of these fractions were tested; or

c) the mean magnesium sulfate value found by the tests on specimens of the two fractions next but one adjacent to it if both these fractions were tested and the adjacent fractions were not; or

d) the magnesium sulfate value found by the test on a specimen of the fraction, either larger or smaller, in this order of priority, most nearly adjacent to it.

C.5.3 For samples of fine aggregate, the material passing the 0,25 mm sieve shall not be tested but shall be taken as having a magnesium sulfate value equivalent to that of the specimen passing the 0,5 mm sieve but retained on the 0,25 mm sieve.

C.5.4 The magnesium sulfate value of each test portion of aggregate shall be the sum of the magnesium sulfate values found for each aggregate fraction times the proportion by mass of that fraction in the test portion.

C.5.5 The magnesium sulfate value for the aggregate shall be the mean of the two results for the test portions to the nearest whole number. The magnesium sulfate value for each fraction of the aggregate shall be the mean of the magnesium sulfate values for the two results for the test specimens to one decimal place.

NOTE A suitable worksheet (with two examples, one coarse aggregate and one fine aggregate) is shown in Tables C.1 and C.2.

C.5.6 The magnesium sulfate value for the maximum fraction shall be maximum magnesium sulfate value of any fraction tested.

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Table C.1 — Example with coarse aggregate (6 mm nominal single size)

Sieve Size		Grading of	Mass of Test Specimen			Magnesium	Weighted Mag.
Passing	**Retained**	**Test Portion**	**Initially**	**Before Test**	**After Test**	**Sulfate Value**	**Sulfate value**
mm	mm	% of total mass	g	g	g	% of original mass	%
37,5	20	*0*	–	–	–	–	*0*
20	10	*0*	–	–	–	–	*0*
10	6,3	*26,4*	*606,4*	*303,2*	*278,2*	*8,2*	*2,18*
6,3	4	*34,7*	*104,4*	*104,4*	*98,6*	*6,0*	*2,09*
4	2	*34.7*	*104,4*	*104,4*	*98,6*	*6,0*	*2,08*
2	1	*4.2* †	–	–	–	*6,0* ‡	*0,25*
Total		*100*				Total	*6,60*
37,5	20	*0*	–	–	–	–	*0*
20	10	*0*	–	–	–	–	*0*
10	6,3	*28,7*	*621.8*	304.7	280.2	*8.0*	*2.31*
6,3	4	*33,1*	*103,3*	103,3	97,2	*6.0*	*1.99*
4	2	*33,1*	*103,3*	103,3	97,1	*6,0*	*1,98*
2	1	*5,1*	*218.6*	104.1	98.2	*5.7*	*0.29*
Total		*100*				Total	*6.57*
						Mean	7

† Less than 5 % by mass of total sample, no test specimen.
‡ Taken as equivalent to that for 6,3 mm to 2 mm size under C.5.2, indent (b).

Table C.2 — Example with fine aggregate (Coarse sand)

Sieve Size		Grading of	Mass of Test Specimen			Magnesium	Weighted Mag.
Passing	**Retained**	**Test Portion**	**Initially**	**Before Test**	**After Test**	**Sulfate Value**	**Sulfate value**
mm	mm	% of total mass	g	g	g	% of original mass	%
10	6,3	*4,6* †	–	–	–	*11.9* ‡	*0.55*
6,3	4	*5,4*	*102,1*	*102,1*	*89,9*	*11.9*	*0,65*
4	2	*5,4*	*102,0*	*102,0*	*89,9*	*11,9*	*0.64*
2	1	*17,0*	*213.8*	*101.8*	*94.2*	*7.5*	*1.27*
1	0,5	*25,2*	*195.1*	*107.3*	*102.8*	*4.2*	*1.06*
0,5	0,25	*26,2*	*218.6*	*104.1*	*99.3*	*4.6*	*1.21*
0,25	–	*16,2*	–	–	–	*4.6* *	*0.75*
Total		*100*				Total	*6,12*
10	6,3	*4,4* †	–	–	–	*11.2* ‡	*0.49*
6,3	4	*5,5*	*104,1*	*104,1*	*92,4*	*11.2*	*0,62*
4	2	*5,4*	*104,1*	*104,1*	*92,4*	*11,2*	*0,61*
2	1	*17,3*	*213.6*	*106.8*	*98.3*	*8.0*	*1.38*
1	0,5	*25,1*	*203.4*	*101.7*	*96.8*	*4.8*	*1.21*
0,5	0,25	*26,1*	*200.6*	*100.3*	*96.1*	*4.2*	*1.09*
0,25	–	*16,2*	–	–	–	*4.2* *	*0.68*
Total		*100*				Total	*6.08*
						Mean	6

† Less than 5 % by mass of total sample, no test specimen.
‡ Taken as equivalent to that for 6,3 mm to 4 mm size under C.5.2, indent (b).
* No test but mass loss taken as equivalent to that for 0,5 mm to 0,25 mm size under C.5.3.

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[1] ISO 5725-2:1994. *Accuracy (trueness and precision) of measurement methods and results – Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

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