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EUROPEAN STANDARD
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EN 1367-2

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ICS

Descriptors: aggregates, tests, cyclic tests, determination, thermal properties, liability to deterioration, crystallization, immersion tests, magnesium sulphate

English version

**Tests for thermal and weathering properties of aggregates - Part
2: Magnesium sulfate test**

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 February 1998.

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Foreword

This European Standard has been prepared by Technical Committee CEN/TC 154 "Aggregates", the secretariat of which is held by BSI.

Testing in accordance with this standard is intended to provide information to assist in judging the performance of aggregates subject to weathering action. It is not intended that testing to this standard should be necessary where there is adequate information available from service records of the materials in use.

This European Standard is one of a series of tests for thermal and weathering properties of aggregates as listed below.

- prEN 1367-1 Tests for thermal and weathering properties of aggregates
Part 1: Determination of resistance to freezing and thawing
- prEN 1367-3 Tests for thermal and weathering properties of aggregates
Part 3: Boiling test for "Sonnenbrenner basalt" and disintegration of steel slag
- EN 1367-4 Tests for thermal and weathering properties of aggregates
Part 4: Determination of drying shrinkage
- prEN 1367-5 Tests for thermal and weathering properties of aggregates
Part 5: Determination of resistance to thermal shock

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

A European Standard "Tests filler aggregate used in bituminous bound fillers" is in course of preparation.

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 September 1998, and conflicting national standards shall be withdrawn at the latest by December 1999.

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 assessing how an aggregate behaves when subjected to the cyclic action of immersion in magnesium sulfate, followed by oven drying.

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 minerals or of cryptocrystalline quartz.

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.

- | | |
|------------|---|
| EN 932-1 | Tests for general properties of aggregates
Part 1: Methods for sampling |
| prEN 932-2 | Tests for general properties of aggregates
Part 2: Methods for reducing laboratory samples |
| EN 932-3 | Test for general properties of aggregates
Part 3: Procedure and terminology for simplified petrographic description |
| prEN 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 |

3 Definitions

For the purposes of this standard, the following definitions apply:

- 3.1 laboratory sample:** A sample derived from a bulk sample for laboratory testing.
- 3.2 test specimen:** The sample used in a single determination when a test method requires more than one determination of a property.
- 3.3 test portion:** The sample used as a whole in a single test.

4 Principle

A laboratory sample of aggregate in the size range 10 mm to 14 mm is subjected to five cycles of immersion in a saturated solution of magnesium sulfate, followed by oven drying at $(110 \pm 5) ^\circ\text{C}$. This subjects the laboratory sample of aggregate to the disruptive effects of the repeated crystallisation 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.

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 prEN 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 room, capable of maintaining the temperature of the solution inside the containers at $(20 \pm 2) ^\circ\text{C}$.

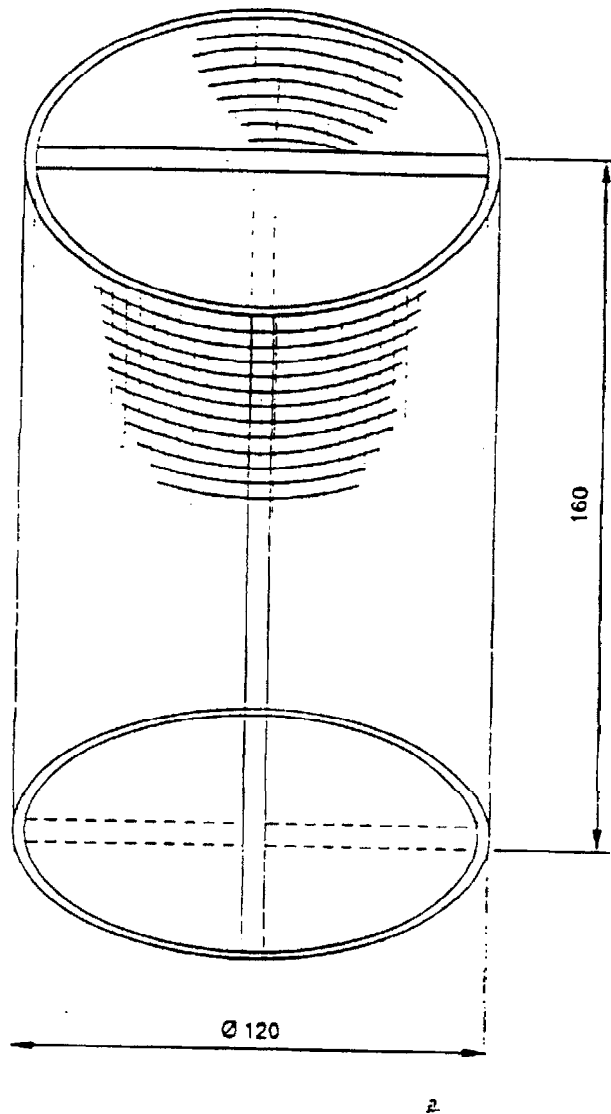
6.6 Ventilated drying oven with forced air circulation, of adequate capacity. The oven shall be capable of being controlled at $(110 \pm 5) ^\circ\text{C}$.

6.7 Density hydrometer, graduated at $20 ^\circ\text{C}$ 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 $0 ^\circ\text{C}$ to $120 ^\circ\text{C}$ and accurate to $1 ^\circ\text{C}$.

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



Mesh size 4

All dimensions in millimetres

NOTE: The dimensions are not critical and are 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

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 deionised water.

7.3.1 Prepare the solution by slowly adding 1500 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 °C to 30 °C and stir thoroughly during the addition of the crystals. After preparation lower the temperature to (20 ± 2) °C, and maintain at this temperature for (48 ± 1) h.

7.3.2 Prior to use check that the solution has achieved a density of $(1,292 \pm 0,008)$ g/ml by decanting a portion of the solution into a gas 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 prEN 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 size fractions is given in annex B.

8.2 Dry each test specimen in the oven at (110 ± 5) °C 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 $(420 \pm 0,1)$ g and $(430 \pm 0,1)$ 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 minimise any loss by abrasion.

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9 Procedure

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

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 $(2 \pm 0,25)$ h then cover the container immediately. Dry each basket as in 8.2 and cool to laboratory temperature for $(5 \pm 0,25)$ 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.

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:

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

where:

M_1 is the initial mass of the test specimen, to the nearest $\pm 0,1$ g;

M_2 is the final mass of aggregate retained on the 10 mm sieve,
to the nearest $\pm 0,1$ 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, including range obtained;
- c) sample identification, including source, description, simple petrographical description as in EN 932-3 and submitted aggregate size;
- d) size fraction tested;
- e) proportion by mass of laboratory sample used for the test portion, recorded to the nearest 5 %.
- f) a copy of the certificate of sampling, if available.

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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 prEN 932-2.

The precision data from table A.1 are approximately represented by the following simplified equations, where $x = MS$.

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

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

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

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 %	$\sqrt{V_r}$ %	$\sqrt{V_L}$ %	$\sqrt{V_s}$ %	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

Annex B (informative)

Recommended test sieves, mesh baskets and mass of test portions for testing aggregates outside the size and range 10,00 mm to 14,00 mm

Size fraction mm	Mass of test portion g	Test sieve		Mesh baskets		
		Passing mm	Retained mm	Mesh size mm	Height mm	Diameter 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
	100 to 110	0,60	0,30	0,15	80	65

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Annex C (informative)

Bibliography

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