



Standard Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This test method covers the testing of aggregates to estimate their soundness when subjected to weathering action in concrete or other applications. This is accomplished by repeated immersion in saturated solutions of sodium or magnesium sulfate followed by oven drying to partially or completely dehydrate the salt precipitated in permeable pore spaces. The internal expansive force, derived from the rehydration of the salt upon re-immersion, simulates the expansion of water on freezing. This test method furnishes information helpful in judging the soundness of aggregates when adequate information is not available from service records of the material exposed to actual weathering conditions.

1.2 The values given in parentheses are provided for information purposes only.

1.3 *This standard does not purport to address the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- C 33 Specification for Concrete Aggregates²
- C 136 Test Method for Sieve Analysis of Fine and Coarse Aggregates²
- C 670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials²
- C 702 Practice for Reducing Samples of Aggregate to Testing Size²

¹ This test method is under the jurisdiction of ASTM Committee C-9 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.20 on Normal Weight Aggregates.

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² *Annual Book of ASTM Standards*, Vol 04.02.

D 75 Practice for Sampling Aggregates³

D 3665 Practice for Random Sampling of Construction Materials³

E 11 Specification for Wire Cloth Sieves for Testing Purposes⁴

E 100 Specification for ASTM Hydrometers⁵

E 323 Specification for Perforated-Plate Sieves for Testing Purposes⁴

3. Significance and Use

3.1 This test method provides a procedure for making a preliminary estimate of the soundness of aggregates for use in concrete and other purposes. The values obtained may be compared with specifications, for example Specification C 33, that are designed to indicate the suitability of aggregate proposed for use. Since the precision of this test method is poor (Section 12), it may not be suitable for outright rejection of aggregates without confirmation from other tests more closely related to the specific service intended.

3.2 Values for the permitted-loss percentage by this test method are usually different for fine and coarse aggregates, and attention is called to the fact that test results by use of the two salts differ considerably and care must be exercised in fixing proper limits in any specifications that include requirements for these tests. The test is usually more severe when magnesium sulfate is used; accordingly, limits for percent loss allowed when magnesium sulfate is used are normally higher than limits when sodium sulfate is used.

NOTE 1—Refer to the appropriate sections in Specification C 33 establishing conditions for acceptance of coarse and fine aggregates which fail to meet requirements based on this test.

4. Apparatus

4.1 *Sieves*—With square openings of the following sizes

³ *Annual Book of ASTM Standards*, Vol 04.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ *Annual Book of ASTM Standards*, Vol 14.03.

conforming to Specifications E 11 or E 323, for sieving the samples in accordance with Sections 6, 7, and 9:

150 μm (No. 100)	8.0 mm ($\frac{5}{16}$ in.) 9.5 mm ($\frac{3}{8}$ in.)
300 μm (No. 50)	12.5 mm ($\frac{1}{2}$ in.) 16.0 mm ($\frac{5}{8}$ in.)
600 μm (No. 30)	19.0 mm ($\frac{3}{4}$ in.) 25.0 mm (1 in.)
1.18 mm (No. 16)	31.5 mm ($1\frac{1}{4}$ in.)
2.36 mm (No. 8)	37.5 mm ($1\frac{1}{2}$ in.) 50 mm (2 in.)
4.00 mm (No. 5)	63 mm ($2\frac{1}{2}$ in.)
4.75 mm (No. 4)	larger sizes by 12.5-mm ($\frac{1}{2}$ -in.) spread

4.2 *Containers*—Containers for immersing the samples of aggregate in the solution, in accordance with the procedure described in this test method, shall be perforated in such a manner as to permit free access of the solution to the sample and drainage of the solution from the sample without loss of aggregate.

NOTE 2—Baskets made of suitable wire mesh or sieves with suitable openings are satisfactory containers for the samples.

4.3 *Temperature Regulation*—Suitable means for regulating the temperature of the samples during immersion in the sodium sulfate or magnesium sulfate solution shall be provided.

4.4 *Balances*—For fine aggregate, a balance or scale accurate within 0.1 g over the range required for this test; for coarse aggregate, a balance or scale accurate within 0.1 % or 1 g, whichever is greater, over the range required for this test.

4.5 *Drying Oven*—The oven shall be capable of being heated continuously at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) and the rate of evaporation, at this range of temperature, shall be at least 25 g/h for 4 h, during which period the doors of the oven shall be kept closed. This rate shall be determined by the loss of water from 1-L Griffin low-form beakers, each initially containing 500 g of water at a temperature of $70 \pm 3^\circ\text{F}$ ($21 \pm 2^\circ\text{C}$), placed at each corner and the center of each shelf of the oven. The evaporation requirement is to apply to all test locations when the oven is empty except for the beakers of water.

4.6 *Specific Gravity Measurement*—Hydrometers conforming to the requirements of Specification E 100, or a suitable combination of graduated glassware and balance, capable of measuring the solution specific gravity within ± 0.001 .

5. Special Solutions Required

5.1 Prepare the solution for immersion of test samples from either sodium or magnesium sulfate in accordance with 5.1.1 or 5.1.2 (Note 3). The volume of the solution shall be at least five times the solid volume of all samples immersed at any one time.

NOTE 3—Some aggregates containing carbonates of calcium or magnesium are attacked chemically by fresh sulfate solution, resulting in erroneously high measured losses. If this condition is encountered or is suspected, repeat the test using a filtered solution that has been used previously to test the same type of carbonate rock, provided that the solution meets the requirements of 5.1.1 and 5.1.2 for specific gravity.

5.1.1 *Sodium Sulfate Solution*—Prepare a saturated solution of sodium sulfate by dissolving a USP or equal grade of the salt in water at a temperature of 77 to 86°F (25 to 30°C). Add sufficient salt (Note 4), of either the anhydrous (Na_2SO_4) or the

crystalline ($\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$) form,⁶ to ensure not only saturation but also the presence of excess crystals when the solution is ready for use in the tests. Thoroughly stir the mixture during the addition of the salt and stir the solution at frequent intervals until used. To reduce evaporation and prevent contamination, keep the solution covered at all times when access is not needed. Allow the solution to cool to $70 \pm 2^\circ\text{F}$ ($21 \pm 1^\circ\text{C}$). Again stir, and allow the solution to remain at the designated temperature for at least 48 h before use. Prior to each use, break up the salt cake, if any, in the container, stir the solution thoroughly, and determine the specific gravity of the solution. When used, the solution shall have a specific gravity not less than 1.151 nor more than 1.174. Discard a discolored solution, or filter it and check for specific gravity.

NOTE 4—For the solution, 215 g of anhydrous salt or 700 g of the decahydrate per litre of water are sufficient for saturation at 71.6°F (22°C). However, since these salts are not completely stable and since it is desirable that an excess of crystals be present, the use of not less than 350 g of the anhydrous salt or 750 g of the decahydrate salt per litre of water is recommended.

5.1.2 *Magnesium Sulfate Solution*—Prepare a saturated solution of magnesium sulfate by dissolving a USP or equal grade of the salt in water at a temperature of 77 to 86°F (25 to 30°C). Add sufficient salt (Note 5), of either the anhydrous (MgSO_4) or the crystalline ($\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$) (Epsom salt) form, to ensure saturation and the presence of excess crystals when the solution is ready for use in the tests. Thoroughly stir the mixture during the addition of the salt and stir the solution at frequent intervals until used. To reduce evaporation and prevent contamination, keep the solution covered at all times when access is not needed. Allow the solution to cool to $70 \pm 2^\circ\text{F}$ ($21 \pm 1^\circ\text{C}$). Again stir, and allow the solution to remain at the designated temperature for at least 48 h before use. Prior to each use, break up the salt cake, if any, in the container, stir the solution thoroughly, and determine the specific gravity of the solution. When used, the solution shall have a specific gravity not less than 1.295 nor more than 1.308. Discard a discolored solution, or filter it and check for specific gravity.

NOTE 5—For the solution, 350 g of anhydrous salt or 1230 g of the heptahydrate per litre of water are sufficient for saturation at 73.4°F (23°C). However, since these salts are not completely stable, with the hydrous salt being the more stable of the two, and since it is desirable that an excess of crystals be present, it is recommended that the heptahydrate salt be used and in an amount of not less than 1400 g/litre of water.

5.1.3 *Barium Chloride Solution*—Prepare 100 mL of 5 % barium chloride solution by dissolving 5 g of BaCl_2 in 100 mL of distilled water.

6. Samples

6.1 The sample shall be obtained in general accordance with Practice D 75 and reduced to test portion size in accordance with Practice C 702.

6.2 *Fine Aggregate*—Fine aggregate for the test shall be

⁶ Experience with the test method indicates that a grade of sodium sulfate designated by the trade as dried powder, which may be considered as approximately anhydrous, is the most practical for use. That grade is more economically available than the anhydrous form. The decahydrate sodium sulfate presents difficulties in compounding the required solution on account of its cooling effect on the solution.

passed through a 9.5-mm ($\frac{3}{8}$ -in.) sieve. The sample shall be of such size that it will yield not less than 100 g of each of the following sizes, which shall be available in amounts of 5 % or more, expressed in terms of the following sieves:

Passing Sieve	Retained on Sieve
600 μm (No. 30)	300 μm (No. 50)
1.18 mm (No. 16)	600 μm (No. 30)
2.36 mm (No. 8)	1.18 mm (No. 16)
4.75 mm (No. 4)	2.36 mm (No. 8)
9.5 mm ($\frac{3}{8}$ in.)	4.75 mm (No. 4)

6.3 Coarse Aggregate—Coarse aggregate for the test shall consist of material from which the sizes finer than the No. 4 sieve have been removed. The sample shall be of such a size that it will yield the following amounts of the indicated sizes that are available in amounts of 5 % or more:

Size (Square-Opening Sieves)	Mass, g
9.5 mm ($\frac{3}{8}$ in.) to 4.75 mm (No. 4)	300 \pm 5
19.0 mm ($\frac{3}{4}$ in.) to 9.5 mm ($\frac{3}{8}$ in.)	1000 \pm 10
Consisting of:	
12.5-mm ($\frac{1}{2}$ -in.) to 9.5-mm ($\frac{3}{8}$ -in.) material	330 \pm 5
19.0-mm ($\frac{3}{4}$ -in.) to 12.5-mm ($\frac{1}{2}$ -in.) material	670 \pm 10
37.5-mm ($1\frac{1}{2}$ -in.) to 19.0-mm ($\frac{3}{4}$ in.)	1500 \pm 50
Consisting of:	
25.0-mm (1-in.) to 19.0-mm ($\frac{3}{4}$ -in.) material	500 \pm 30
37.5-mm ($1\frac{1}{2}$ -in.) to 25.0-mm (1-in.) material	1000 \pm 50
63-mm ($2\frac{1}{2}$ in.) to 37.5-mm ($1\frac{1}{2}$ in.)	5000 \pm 300
Consisting of:	
50-mm (2 in.) to 37.5-mm ($1\frac{1}{2}$ -in.) material	2000 \pm 200
63-mm ($2\frac{1}{2}$ -in.) to 50-mm (2-in.) material	3000 \pm 300
Larger sizes by 25-mm (1-in.) spread in sieve size, each fraction	7000 \pm 1000

6.4 When an aggregate to be tested contains appreciable amounts of both fine and coarse material, having a grading with more than 10 weight % coarser than the 9.5-mm ($\frac{3}{8}$ -in.) sieve and, also, more than 10 weight % finer than the 4.75-mm (No. 4) sieve, test separate samples of the minus No. 4 fraction and the plus No. 4 fraction in accordance with the procedures for fine aggregate and coarse aggregate, respectively. Report the results separately for the fine-aggregate fraction and the coarse-aggregate fraction, giving the percentages of the coarse- and fine-size fractions in the initial grading.

7. Preparation of Test Sample

7.1 Fine Aggregate—Thoroughly wash the sample of fine aggregate on a 300- μm (No. 50) sieve, dry to constant weight at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$), and separate into the different sizes by sieving, as follows: Make a rough separation of the graded sample by means of a nest of the standard sieves specified in 6.2. From the fractions obtained in this manner, select samples of sufficient size to yield 100 g after sieving to refusal. (In general, a 110-g sample will be sufficient.) Do not use fine aggregate sticking in the meshes of the sieves in preparing the samples. Weigh samples consisting of 100 ± 0.1 g out of each of the separated fractions after final sieving and place in separate containers for the test.

7.2 Coarse Aggregate—Thoroughly wash and dry the sample of coarse aggregate to constant weight at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$) and separate it into the different sizes shown in 6.3 by sieving to refusal. Weigh out quantities of the different sizes within the tolerances of 6.3 and, where the test portion consists of two sizes, combine them to the designated total weight.

Record the weights of the test samples and their fractional components. In the case of sizes larger than 19.0 mm ($\frac{3}{4}$ in.), record the number of particles in the test samples.

8. Procedure

8.1 Storage of Samples in Solution—Immerse the samples in the prepared solution of sodium sulfate or magnesium sulfate for not less than 16 h nor more than 18 h in such a manner that the solution covers them to a depth of at least $\frac{1}{2}$ in. (Note 6). Cover the containers to reduce evaporation and prevent the accidental addition of extraneous substances. Maintain the samples immersed in the solution at a temperature of $70 \pm 2^\circ\text{F}$ ($21 \pm 1^\circ\text{C}$) for the immersion period.

NOTE 6—Suitably weighted wire grids placed over the sample in the containers will permit this coverage to be achieved with very lightweight aggregates.

8.2 Drying Samples After Immersion—After the immersion period, remove the aggregate sample from the solution, permit it to drain for 15 ± 5 min, and place in the drying oven. The temperature of the oven shall have been brought previously to $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$). Dry the samples at the specified temperature until constant weight has been achieved. Establish the time required to attain constant weight as follows: with the oven containing the maximum sample load expected, check the weight losses of test samples by removing and weighing them, without cooling, at intervals of 2 to 4 h; make enough checks to establish required drying time for the least favorable oven location (see 4.5) and sample condition (Note 7). Constant weight will be considered to have been achieved when weight loss is less than 0.1 % of sample weight in 4 h of drying. After constant weight has been achieved, allow the samples to cool to room temperature, when they shall again be immersed in the prepared solution as described in 8.1.

NOTE 7—Drying time required to reach constant weight may vary considerably for several reasons. Efficiency of drying will be reduced as cycles accumulate because of salt adhering to particles and, in some cases, because of increase in surface area due to breakdown. The different size fractions of aggregate will have differing drying rates. The smaller sizes will tend to dry more slowly because of their larger surface area and restricted interparticle voids, but this tendency may be altered by the effects of container size and shape.

8.3 Number of Cycles—Repeat the process of alternate immersion and drying until the required number of cycles is obtained.

8.4 After the completion of the final cycle and after the sample has cooled, wash the sample free from the sodium sulfate or magnesium sulfate as determined by the reaction of the wash water with barium chloride (BaCl_2). Wash by circulating water at $110 \pm 10^\circ\text{F}$ ($43 \pm 6^\circ\text{C}$) through the samples in their containers. This may be done by placing them in a tank into which the hot water can be introduced near the bottom and allowed to overflow. In the washing operation, the samples shall not be subjected to impact or abrasion that may tend to break up particles.

NOTE 8—Tap water containing sulfates when used for the wash water will cloud when tested with the barium chloride solution. The cloudiness of a solution of tap water and the barium chloride solution should be judged so that tested wash water with the same degree of cloudiness can be assumed to be free of sulfates from the test.

9. Quantitative Examination

9.1 Make the quantitative examination as follows:

9.1.1 After the sodium sulfate or magnesium sulfate has been removed, dry each fraction of the sample to constant weight at $230 \pm 9^\circ\text{F}$ ($110 \pm 5^\circ\text{C}$). Sieve the fine aggregate over the same sieve on which it was retained before the test, and sieve the coarse aggregate over the sieve shown below for the appropriate size of particle. For fine aggregate, the method and duration of sieving shall be the same as were used in preparing the test samples. For coarse aggregate, sieving shall be by hand, with agitation sufficient only to assure that all undersize material passes the designated sieve. No extra manipulation shall be employed to break up particles or cause them to pass the sieves. Weigh the material retained on each sieve and record each amount. The difference between each of these amounts and the initial weight of the fraction of the sample tested is the loss in the test and is to be expressed as a percentage of the initial weight for use in Table 1.

Size of Aggregate	Sieve Used to Determine Loss
63 mm (2½ in.) to 37.5 mm (1½ in.)	31.5 mm (1¼ in.)
37.5 mm (1½ in.) to 19.0 mm (¾ in.)	16.0 mm (⅝ in.)
19 mm (¾ in.) to 9.5 mm (⅜ in.)	8.0 mm (⅝ in.)
9.5 mm (⅜ in.) to 4.75 mm (No. 4)	4.0 mm (No. 5)

10. Qualitative Examination

10.1 Make a qualitative examination of test samples coarser than 19.0 mm (¾ in.) as follows (Note 9):

10.1.1 Separate the particles of each test sample into groups according to the action produced by the test (Note 9).

10.1.2 Record the number of particles showing each type of distress.

NOTE 9—Many types of action may be expected. In general, they may be classified as disintegration, splitting, crumbling, cracking, flaking, etc. While only particles larger than ¾ in. in size are required to be examined qualitatively, it is recommended that examination of the smaller sizes be

made in order to determine whether there is any evidence of excessive splitting.

11. Report

11.1 Report the following data (Note 10):

11.1.1 Weight of each fraction of each sample before test,

11.1.2 Material from each fraction of the sample finer than the sieve designated in 9.1.1 for sieving after test, expressed as a percentage of the original weight of the fraction,

11.1.3 Weighted average calculated in accordance with Test Method C 136 from the percentage of loss for each fraction, based on the grading of the sample as received for examination or, preferably, on the average grading of the material from that portion of the supply of which the sample is representative except that:

11.1.3.1 For fine aggregates (with less than 10 % coarser than the 9.5-mm (⅜-in.) sieve), assume sizes finer than the 300-µm (No. 50) sieve to have 0 % loss and sizes coarser than the 9.5-mm (⅜-in.) sieve to have the same loss as the next smaller size for which test data are available.

11.1.3.2 For coarse aggregate (with less than 10 % finer than the 4.75-mm (No. 4) sieve), assume sizes finer than the 4.75-mm (No. 4) sieve to have the same loss as the next larger size for which test data are available.

11.1.3.3 For an aggregate containing appreciable amounts of both fine and coarse material tested as two separate samples as required in 6.4, compute the weighted average losses separately for the minus No. 4 and plus No. 4 fractions based on recomputed gradings considering the fine fraction as 100 % and the coarse fraction as 100 %. Report the results separately giving the percentage of the minus No. 4 and plus No. 4 material in the initial grading.

11.1.3.4 For the purpose of calculating the weighted average, consider any sizes in 6.2 or 6.3 that contain less than 5 % of the sample to have the same loss as the average of the next smaller and the next larger size, or if one of these sizes is

TABLE 1 Suggested Form for Recording Test Data (with Illustrative Test Values)

Sieve Size	Grading of Original Sample, %	Weight of Test Fractions Before Test, g	Percentage Passing Designated Sieve After Test	Weighted Percentage Loss
Soundness Test of Fine Aggregate				
Minus 150 µm (No. 100)	6
300 µm (No. 50) to No. 100	11
600 µm (No. 30) to No. 50	26	100	4.2	1.1
1.18 mm (No. 16) to No. 30	25	100	4.8	1.2
2.36 mm (No. 8) to No. 16	17	100	8.0	1.4
4.75 mm (No. 4) to No. 8	11	100	11.2	1.2
9.5 mm (⅜ in.) to No. 4	4	...	11.2 ^A	0.4
Totals	100.0	5
Soundness Test of Coarse Aggregate				
63 mm (2½ in.) to 50 mm (2 in.)	2½ to 1½ in.	2825 g	4.8	1.0
50 mm (2 in.) to 37.5 mm (1½ in.)		1958 g		
37.5 mm (1½ in.) to 25.0 mm (1 in.)	1½ to ¾ in.	1012 g	8.0	3.6
25 mm (1 in.) to 19.0 mm (¾ in.)		513 g		
19.0 mm (¾ in.) to 12.5 mm (½ in.)	¾ to ⅜ in.	675 g	9.6	2.2
12.5 mm (in.) to 9.5 mm (in.)		333 g		
9.5 mm (⅜ in.) to 4.75 mm (No. 4)		298 g	11.2	1.3
Totals	100	8

^A The percentage loss (11.2 %) of the next smaller size is used as the percentage loss for this size, since this size contains less than 5 % of the original sample as received. See 11.1.3.4.

TABLE 2 Suggested Form for Qualitative Examination (with Illustrative Test Values)

Sieve Size	Qualitative Examination of Coarse Sizes								Total No. of Particles Before Test
	Particles Exhibiting Distress								
	Splitting		Crumbling		Cracking		Flaking		
	No.	%	No.	%	No.	%	No.	%	
63 mm (2½ in.) to 37.5 mm (1½ in.)	2	7	2	7	29
37.5 mm (1½ in.) to 19.0 mm (¾ in.)	5	10	1	2	4	8	50

absent, to have the same loss as the next larger or next smaller size, whichever is present.

11.1.4 Report the weighted percentage loss to the nearest whole number,

11.1.5 In the case of particles coarser than 19.0 mm (¾ in.) before test: (1) The number of particles in each fraction before test, and (2) the number of particles affected, classified as to number disintegrating, splitting, crumbling, cracking, flaking, etc., as shown in Table 2, and

11.1.6 Kind of solution (sodium or magnesium sulfate) and whether the solution was freshly prepared or previously used.

NOTE 10—Table 1, shown with test values inserted for purpose of illustration, is a suggested form for recording test data. The test values shown might be appropriate for either salt, depending on the quality of the aggregate.

12. Precision

12.1 *Precision*—For coarse aggregate with weighted average sulfate soundness losses in the ranges of 6 to 16 % for sodium and 9 to 20 % for magnesium, the precision indexes are as follows:

Coefficient of Variation (1S %), %^A

Difference Between Two Tests (D2S %), % of Average^A

<i>Multilaboratory:</i>		
Sodium sulfate	41	116
Magnesium sulfate	25	71
<i>Single-Operator:</i>		
Sodium sulfate	24	68
Magnesium sulfate	11	31

^A These numbers represent, respectively, the (1S %) and (D2S %) limits as described in Practice C 670.

12.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for this procedure, no statement on bias is being made.

13. Keywords

13.1 aggregates; magnesium sulfate; sodium sulfate; soundness; weathering

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