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MATERIALS BUREAU  
ALBANY, NY 12232

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## TEST METHOD

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SUBJECT: STANDARD TEST METHOD FOR SOUNDESS OF FINE AGGREGATES BY USE  
OF MAGNESIUM SULFATE

APPROVED:

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Dated:

### 1. SCOPE

1.1 This method covers the procedure to be followed in testing fine aggregates to determine their resistance to disintegration by a saturated solution of magnesium sulfate. It furnishes information helpful in judging the soundness of aggregates subject to weathering action, particularly when adequate information is not available from service records of the material exposed to weathering conditions. The format and wording of this method is similar to AASHTO T 104.

### REFERENCE DOCUMENTS

#### 2. AASHTO Standard:

M 92 Wire cloth sieves for testing purposes

#### 2.2 ASTM Standards

E 11 Wire cloth sieves for testing purposes

E 100 Specification for ASTM Hydrometers

C295 Standard Recommended Practice for PETROGRAPHIC EXAMINATION OF  
AGGREGATES FOR CONCRETE

### 3. APPARATUS

3.1 Sieves - With square openings of the following sizes conforming to AASHTO M 92 or ASTM E 11, for sieving the samples in accordance with selections 5, 6, and 8:

0.3 mm	(No. 50)	2.36 mm	(No. 8)
0.6 mm	(No. 30)	4.75 mm	(No. 4)
1.18 mm	(No. 16)	9.5mm	(3/8 in.)

3.2 Containers - Baskets for immersing the samples of aggregate in the solution, in accordance with the procedure described in this method, shall be constructed 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. The baskets shall have a stainless steel frame with stainless steel square wire mesh 0.25mm (No. 60). They shall be approximately 1.7cm (5 in.) in diameter, 5.08cm (2 in.) inside

and 6.35cm (2 1/2 in.) outside height, with at least six 3.81cm (1 1/2 in.) square windows of mesh on the sides and the recessed bottom entirely of mesh.

**3.3 Temperature Regulation** - Suitable means for regulating the temperature of the samples during immersion in the magnesium sulfate solution shall be provided. A circulation tank with a filter and a cover to reduce evaporation and prevent the accidental addition of extraneous substances is preferred.

**3.4 Balance** - The balance shall conform to AASHTO M231, Class G2.

**3.5 Drying Oven** - The oven shall have circulating fans and shall be capable of being heated continuously at  $110 \pm 5^{\circ}\text{C}$  ( $230 \pm 9^{\circ}\text{F}$ ). The rate of evaporation, at this range of temperature shall average 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-liter Griffin low-form beakers, each initially containing 500 g of water at a temperature of  $21 \pm 2^{\circ}\text{C}$  ( $70 \pm 3^{\circ}\text{F}$ ), 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.

**3.6 Specific Gravity Measurement** - A hydrometer conforming to the requirements ASTM E 100, or a suitable combination of graduated glassware and balance, capable of measuring the solution specific gravity within  $\pm 0.001$  shall be used.

#### **4. MAGNESIUM SULFATE SOLUTION**

**4.1** Prepare the solution of magnesium sulphate for immersion of test samples in accordance with Section 4.1.1. The volume of the solution shall be at least five times the solid volume of all samples immersed at any one time.

**4.1.1 MAGNESIUM SULPHATE SOLUTION** - Prepare a saturated solution of magnesium sulfate by dissolving a USP or equal grade of the salt in water at a temperature of 25 to  $30^{\circ}\text{C}$  ( $77$  to  $86^{\circ}\text{F}$ ). Add sufficient salt (Note 1), of either the anhydrous ( $\text{MgSO}_4$ ) 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  $23.3 \pm 0.6^{\circ}\text{C}$  ( $74.0 \pm 1.0^{\circ}\text{F}$ ). Again, stir and allow the solution to remain at the designated temperature for at least 48 hours before use. Prior to each use, break up the salt cake in the container, if any, stir the solution thoroughly, and determine the specific gravity of the solution. When used, the solution shall have a specific gravity of  $1.300 \pm 0.002$  and a temperature of  $23.3 \pm 0.6^{\circ}\text{C}$  ( $74.0 \pm 1.0^{\circ}\text{F}$ ). Discolored solution shall be discarded or filtered for use if, when checked, the chemical purity and specific gravity meet the requirements.

**Note 1** - For the solution, 350 g of anhydrous salt or 1230 g of the heptahdrate per liter of water are sufficient for saturation at  $23.3^{\circ}\text{C}$  ( $74.0^{\circ}\text{F}$ ). 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 per liter of water.

## 5. SAMPLES

5.1 **FINE AGGREGATES** - Fine aggregate for the test shall be passed through a 9.5mm (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 percent or more for each of the retained sieve fractions. Should the sample contain less than 5 percent of any of the sizes specified, that size shall not be tested.

<u>Passing Sieve</u>	<u>Retained on Sieve</u>
0.6 mm (No. 30)	0.3 mm (No. 50)
1.18 mm (No. 16)	0.6 mm (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 (3/8 in.)	4.75 mm (No. 4)

## 6. PREPARATION OF TEST SAMPLE

6.1 Dry the 23kg (50 lb.) field sample to remove enough retained water so that a representative sample of the fine aggregate, approximately 1400 grams, may be obtained by either the splitting or quartering method.

6.2 Separate the 1400 gram sample into the different sizes by sieving as follows:

6.2.1 Make a rough separation of the sizes by means of a nest of the standard sieves specified in section 5.1. Shake for ten minutes and weigh out approximately 170 grams of each sieve size.

6.2.2 Combine the 170 gram sieve fractions, place them into a nest of standard sieves and shake for an additional ten minutes. Weigh out approximately 125 grams of each size.

6.2.3 Combine the 125 gram sieve fractions; thoroughly wash the combined sample and dry to a constant weight at  $110 \pm 5^{\circ}\text{C}$  ( $230 \pm 9^{\circ}\text{F}$ ). Place the sample into the nest of standard sieves and shake for fifteen minutes. Hand tap each size until refusal. Weigh out 100 grams and place into separate baskets for testing. In preparing the sample, do not use any of the fine aggregate sticking in the meshes of the sieves.

**Note 2** - The designated steps (6.1 - 6.2.3) have been developed through research by NYSDOT to avoid overloading any sieve fraction, assure adequate material for testing all sieve fractions, and assure proper separation of all sizes during preparation of the test sample.

## 7. PROCEDURE

7.1 **Storage of Samples** - Immerse the samples in the prepared solution of magnesium sulfate for not less than 16h nor more than 18h in such a manner that the solution covers them to a depth of least 12.5mm (1/2 in.), (Note 3). Maintain the samples immersed in the solution at a temperature of  $23.3 \pm 0.6^{\circ}\text{C}$  ( $74 \pm 1^{\circ}\text{F}$ ).

**Note 3** - Cover the tank to reduce evaporation and prevent the accidental addition of extraneous substances. For lightweight aggregates it is also necessary to cover each basket with a plastic cover.

**7.2 Drying Samples After immersion** - After the immersion period, remove the aggregate sample from solution, permit it to drain for  $15 \pm 5$  minutes and place in the drying oven. The temperature of the oven shall have been brought previously to  $110 \pm 5^\circ\text{C}$  ( $230 \pm 9^\circ\text{F}$ ). 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 hours. Make enough checks to establish required drying time for the least favorable oven location and sample condition (Note 4). Constant weight will be considered to have been achieved when weight loss is less than 0.1 percent in 4 hours of drying. After constant weight has been achieved, allow the samples to cool to  $20\text{-}25^\circ\text{C}$  ( $68\text{-}77^\circ\text{F}$ ) when they shall again be immersed in the prepared solution as described in Section 7.1. Experience has shown that sample temperatures significantly different than  $23.3^\circ\text{C}$  ( $74^\circ\text{F}$ ) may change the temperature of the solution temporarily, thereby causing a change in salt saturation, even though the solution returns to  $23.3^\circ\text{C}$  ( $74^\circ\text{F}$ ) for most of the soaking period. Cooling of the sample may be aided by use of an air conditioner or fan.

**Note 4** - Drying time required to reach constant weight may vary considerably for several reasons. Efficiency of drying will be reduced as cycles accumulate because of the salt adhering to the particles and, in some cases, of increased 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 the container size and shape.

**7.3 Number of Cycles** - Repeat the process of alternate immersion and drying until 5 cycles have been completed. If the test must be interrupted, leave the samples in an oven dried condition (constant weight) at room temperature until testing can be resumed.

## **8. QUANTITATIVE EXAMINATION**

**8.1** Make the quantitative examination as follows:

**8.1.1** After the completion of the final cycle and after the sample has cooled, wash the sample free from the sodium sulfate or magnesium sulfate. Wash by circulating water at  $43 \pm 6^\circ\text{C}$  ( $110 \pm 10^\circ\text{F}$ ) through the samples in their containers by introducing hot water near the bottom and allowing the water to pass through the samples and overflow. The thoroughness of washing shall be checked by obtaining a sample of rinse water after it has passed through the samples and checked with a 10% solution of barium chloride. Further washing is required if sample becomes cloudy upon addition of the barium chloride solution. In areas where the water gives a reaction with barium chloride other analytical means shall be used to assure thoroughness of washing. In the washing operation, the samples shall not be subjected to impact or abrasion that may tend to break up particles.

**8.1.2** After the magnesium sulfate has been removed, dry each fraction of the sample to constant weight at  $110 \pm 5^\circ\text{C}$  ( $230 \pm 9^\circ\text{F}$ ). Sieve the fine aggregate for 15 minutes over the sieve on which it was retained before the test. Do not hand tap sieves. Weigh the material retained on each sieve and record each amount. The difference between the weights is the loss to be expressed

as a percentage of the initial weight for use in Table 1.

## 9. REPORT

9.1 The report shall include the following data (Note 5):

Weight of each fraction before test.

Weight of each fraction after test.

9.1.3 Weight lost from each fraction determined by subtracting the weight in 9.1.2 from the weight in 9.1.1. The loss shall be expressed as a percentage of the original weight of each fraction.

9.1.4 Weighted average calculated from the percentage of loss for each fraction, based on the NYSDOT "Standard Gradation (Note 5) except that:

9.1.4.1 For purposes of calculating the weighed average, assume that sizes finer than the 0.3 mm (No. 50) to have 0% loss.

9.1.4.2 For the purpose of calculating the weighted average, consider any sizes in Section 5.1 that contain less than 5 percent 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 absent, to have the same loss as the next larger or smaller size, whichever is present.

TABLE 1

SUGGESTED FORM FOR RECORDING TEST DATA  
(With illustrative Test Values)

Retained on Sieve (See Section 5.1)	4.75mm(No.4)	2.36mm(No.8)	1.18mm(No. 16)	0.6mm(No. 30)	0.3mm(No. 50)
Weight of Test Fraction (Before Test, grams)	0.0	100.0	100.0	100.0	100.0
Weight of Test Fraction (After Test, grams)		60.4	68.5	74.5	84.7
Loss in grams		39.6	31.5	25.5	15.3
Loss in Percent	39.6	39.6	31.5	25.5	15.3
Standard Gradation (Percent Retained, Note 5)	5%	15%	15%	22%	23%
Weighted Percent Loss	1.98	5.94	4.73	5.61	3.51
Total Weighted Loss (Percent)					

Note 5 - The use of sample gradation to calculate a "Weighted Percent Loss" was discontinued in 1965. At that time, it was determined that total weighted loss for a sample could vary by as much as 6% when the sample gradation was used to weight losses. Weighted Percent Loss is now calculated using a "Standard Gradation" which is the mean of the NYSDOT Specification limits

expressed as "Percent Retained" for Portland Cement Concrete fine aggregates. All fine aggregates, both portland cement concrete and bituminous sands, are evaluated in this manner to allow comparison of quality between deposits and monitor uniformity of the deposit and its operation. With geologic control and the use of the "Standard Gradation", the magnesium sulfate soundness losses for the deposit in Table 1 have ranged between 18.1% to 21.8% for the years 1965-1992. The magnesium sulfate soundness loss for the same deposit in 1964 was 14.7%.

#### STANDARD GRADATION

<u>Sieve</u>	<u>Percent Retained</u>
4.75mm (No. 4)	5
2.36mm (No. 8)	15
1.18mm (No. 16)	15
0.6mm (No. 30)	22
0.3mm (No. 50)	23