METHOD OF TEST FOR SOUNDNESS OF AGGREGATES BY USE OF MAGNESIUM SULPHATE

1. SCOPE

1.1 This method covers the testing of aggregates to determine their resistance to disintegration in saturated solutions of magnesium sulphate. It furnishes information helpful in judging the soundness of aggregates subject to weathering action, particularly when adequate information is not available from service records.

2. RELEVANT DOCUMENTS

ASTM C88	Standard Test Method for Soundness of Aggregates by Use of Sodium Sulfate or Magnesium Sulfate
ASTM E100	Standard Specification for ASTM Hydrometers
CSA A23.2-9A	Soundness of Aggregate by Use of Magnesium Sulphate

3. APPARATUS

3.1 SULPHATE TANK: A suitably constructed three-compartment tank, one compartment for solution make-up, one for the test solution, and the third for washing the completed test samples. The test solution compartment shall contain suitable refrigeration and heating units capable of controlling the temperature of the magnesium sulphate solution within ±1.0°C of the required temperature.

Note 1: Immersion type mercury contact thermo-regulators reading to 0.05°C controlling Jumo electronic relays are suitable for this purpose.

3.2 SIEVES: With square openings of the following sizes conforming to OPSS specifications, Table 1.

Table 1

Coarse Series	Fine Series
4.75 mm	300 µm
9.50 mm	600 µm
13.2 mm	1.18 mm
16.0 mm	2.36 mm
19.0 mm	4.75 mm

3.3 WIRE BASKETS: For immersing the samples of aggregates in the solution. The baskets shall bear a number or other means of identification. The baskets shall be made of copper wire or stainless steel and of appropriate mesh size for the aggregate under test (19 - 9.5 mm aggregate use sieve mesh 6.7 mm, 9.5 - 4.75 mm aggregate use sieve mesh 2.36 mm).

3.4 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.

3.5 MECHANICAL CONVECTION OVEN: The oven shall be capable of being continuously heated at 110 ± 5.0°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-litre Griffin low-form beakers, each initially containing 500 g of water at a temperature of 21 ± 2.0°C, placed at each corner and the centre 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 HYDROMETER: Capable of determining the relative density of the test solution, conforming to the requirements of ASTM E100.

3.7 LABORATORY CONTROL AGGREGATE: A supply of standard Drain Brothers Stoney Lake Quarry coarse aggregate is available from the Soils and Aggregates Section, Ministry of Transportation, 1201 Wilson Avenue, Ontario M3M 1J8, Fax (416) 235-4101.

4. PREPARATION OF SOLUTION

4.1 Prepare a saturated solution of magnesium sulphate by dissolving a U.S.P. or equal grade of the salt in water at a temperature of 40 ± 3.0°C. Add sufficient salt of either the anhydrous ($MgSO_4$) or the crystalline ($MgSO_4 \cdot 7H_2O$, 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 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 21 ± 1.0°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 relative density of the solution. When used, the solution shall have a relative density not less than 1.295 nor more than 1.308. Discard a discoloured solution or filter it and check for relative density.

Note 2: For the solution, 350 g of anhydrous salt or 1230 g of heptahydrate per litre of water are sufficient for saturation at 23°C. However, since these salts are not completely stable, with the heptahydrate 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 be used and in an amount of not less than 1400 g/L of water.

Note 3: Freshly mixed sulphate solutions have low pH values, which may result in a higher loss of material for aggregates containing carbonate minerals. When testing these types of materials, the pH value of freshly mixed solutions should be checked for pH (with either a pH meter or pH paper, range 5 - 7) and neutralized by the addition of a suitable additive.

5. PREPARATION OF SAMPLE

5.1 FINE AGGREGATE: Fine aggregate for the test shall be passed through a 4.75 mm sieve. The test sample shall be obtained from the materials to be tested by use of a sample splitter or the method of quartering and shall weigh approximately 2500 g. The sample is then washed on a 300 µm sieve and dried to a constant mass. The sample is separated into the sizes shown in Table 2 by sieving in a mechanical sieve shaker for a period of 8 to 12 min. From the fractions obtained in this manner, select samples of sufficient size to yield 100 g after sieving to refusal (in general, a 110 to 120 g sample will be sufficient). The samples are then re-sieved to refusal on the same sieves using a mechanical sieve shaker for a period of 12 min. Weigh samples consisting of 100 ± 0.1 g from each of the separated fractions after final sieving and place in separate containers for the test.

Note 4: Sieving to 'refusal' means that no particles pass the sieve during 1 min. of continuous sieving. No hand manipulation of particles is allowed.

Table 2

Passing Sieve	Retained on Sieve
600 µm	300 µm
1.18 mm	600 µm
2.36 mm	1.18 mm
4.75 mm	2.36 mm

Should the sample have less than 30% retained on the 300 µm sieve, it is deemed to be too fine and no test is done on any fraction. If any fraction constitutes less than 5% of the sample, it shall not be tested.

5.2 COARSE AGGREGATE: Coarse aggregate for test shall consist of material from which sizes finer than the 4.75 mm sieve have been removed. Separate the sample into the different sizes shown in Table 3 by sieving to refusal. Weigh out quantities of the different sizes shown in Table 3. If any fraction constitutes less than 5% of the sample, it shall not be tested.

Table 3

Pass	Retained	Mass, g
9.5 mm	**4.75 mm**	**300**
19.0 mm	9.5 mm	1000
Consisting of:		
13.2 mm	9.5 mm	330
19.0 mm	13.2 mm	670
37.5 mm	19 mm	1500
Consisting of:		
26.5 mm	19 mm	500
37.5 mm	26.5 mm	1000
63 mm	37.5 mm	5000
Consisting of:		
53 mm	37.5 mm	2000
63 mm	53 mm	3000

6. PROCEDURE

6.1 FINE AGGREGATE: Place each fraction in a separate suitable wire basket.

6.2 COARSE AGGREGATE: Place the 9.5 mm to 4.75 mm fraction in a suitable wire basket. Place the combined 19.0 mm to 9.5 mm fraction in another wire basket. Place combined fractions larger than 19 mm in one or more baskets as required.

6.3 STORAGE OF SAMPLES IN SOLUTION: Immerse the samples in the prepared solution of magnesium sulphate for not less than 16 h or more than 18 h in such a manner that the solution covers them to a depth of at least 15 mm. Maintain the samples immersed in the solution at a temperature of 21 ± 1.0°C for the immersion period. The volume of solution shall be at least 20 times greater than the total sample volume.

6.4 DRYING SAMPLES AFTER IMMERSION: After the immersion period, remove the samples from the solution, drain for 30 ± 5 min., and place in drying oven. Dry at 110 ± 5.0°C until constant mass has been achieved, usually 6 to 8 h. Drying time may be established as follows: with oven containing the maximum sample load expected, check the loss in mass of samples by removing and weighing them in the baskets, without cooling, at intervals of 2 to 4 h. Make enough checks to establish required drying time for the least favourable oven location and sample condition. Constant mass will be considered to be achieved when the loss is less than 0.1% of sample mass in 4 h of drying. When constant mass is achieved, allow samples to cool to room temperature and immerse in solution.

Note 5: As the number of cycles progresses, the drying time required increases due to loss of drying efficiency because of the accumulation of salt adhering to particles, increase of surface area due to breakdown, and differences in surface area due to particle sizes.

6.5 NUMBER OF CYCLES: Repeat the process of alternate immersion and drying for 5 cycles.

7. QUANTITATIVE EXAMINATION

7.1 After completion of the final cycle, and after the sample has cooled, wash the sample free from the magnesium sulphate as determined by the reaction of the wash water with a 3% (by mass) barium chloride ($BaCl_2$). Wash by circulating hot tap water (40 to 60°C) in their containers. A continuous flow of fresh hot water shall be maintained throughout the washing period. In the washing operation, the sample shall not be subjected to impact or abrasion that may tend to break up particles. After the magnesium sulphate has been removed, dry the samples to a constant weight at 110 ± 5.0°C.

7.2 FINE AGGREGATE: Sieve the fine aggregate over the same sieve on which it was retained before the test, nesting the sieves so that the finest is on the top and the coarsest is on the bottom. Sieve in a mechanical sieve shaker for a period of 12 min. Weigh the material retained on each sieve and record each amount on the Fine Aggregate Report Card (Figure 1).

7.3 COARSE AGGREGATE: Sieve material larger than 63 mm over the same sieve on which it was retained before the test. Sieve the sample of pass 63 mm retained 37.5 mm material over the 37.5 mm sieve. Sieve the sample of pass 37.5 mm retained 19 mm material over the 19 mm sieve. Sieve the sample of pass 19.0 mm retained 9.5 mm material over the 9.5 mm sieve. Sieve the sample of pass 9.5 mm retained 4.75 mm over the 4.75 mm sieve. Sieve only sufficiently to assure that all undersize material passes the sieves. Weigh the material retained on each sieve and record each amount on the Coarse Aggregate Report Card (Figure 2).

8. QUALITATIVE EXAMINATION

8.1 A qualitative examination may be done on coarse aggregate samples to determine the mode of breakdown, i.e. splitting, disintegration, crumbling, cracking, flaking, etc. This examination is not done routinely by MTO. Samples are held in storage for some time after the test. If there are conflicting or "surprising" results in this test, the samples may be recalled and examined in an effort to resolve problems of this nature.

9. CALCULATION

9.1 Calculate the percent loss for each fraction in the magnesium sulphate soundness test as follows:

$$\text{percent loss} = \frac{\text{original mass - mass retained after test}}{\text{original mass}} \times 100$$

9.1.1 Calculate the percent loss to one decimal place.

9.2 Calculate the percent loss for each fraction as the product of the percentage (based on the "as-received" coarse aggregate sample mass or "as-received" fine aggregate sample mass) of each fraction and the percent loss for that fraction.

9.2.1 Calculate the weighted average value of the as-received sample as the sum of the weighted average value for each fraction divided by 100.

9.2.2 For the purpose of calculating the weighted average, consider any sizes (not tested) that contain less than 5% of the as-received sample to have the same values as the average of the next smaller and the next larger size or if one of these sizes is missing, to have the same value as the next larger or smaller size, whichever is present. For fine aggregates, sizes smaller than the 300 µm sieve shall be assumed to have zero percent loss.

10. USE OF LABORATORY CONTROL AGGREGATE

10.1 Every 10 samples, but at least every week in which a sample is tested, a sample of the standard reference aggregate shall also be tested. The material shall be tested on the 19.0 mm to the 9.5 mm and 9.5 mm to 4.75 mm gradings. For the purposes of calculating a weighted average loss, assume the 19.0 mm fraction constitutes 75% of the sample and the 9.5 mm to 4.75 mm fraction constitutes 25% of the sample.

10.2 Control Chart Use: The weighted average loss of the last 20 samples of reference material shall be plotted on a control chart in order to monitor the performance of the laboratory.

10.3 The mean loss of the Drain Brothers Stoney Lake Quarry standard reference aggregate is 8.9% (MERO-036, 2010). Individual test data should not normally be greater than 12.9% or less than 4.9%.

11. REPORT

The report shall include the following:

11.1 The weighted average loss of the reference sample, tested closest to the time at which the aggregate sample was tested, to 1 decimal place.

11.2 The weighted average loss of the last 20 samples of reference material on a control chart.

11.3 Report the loss in percent of each fraction of the sample tested to 0.1%.

11.4 Report the weighted loss in percent of the sample tested to the nearest 0.1%.

12. PRECAUTIONS

12.1 Wire baskets shall be examined after each test for defects in the mesh.

12.2 Extreme care must be taken when immersing the fine aggregate samples as often some of the particles float on the solution due to surface tension. Carefully, with the fingers, sink these particles into their baskets when this happens.

Ministry of Transportation Ontario

$MgSO_4$ SOUNDNESS
FINE AGGREGATE

RUN NO. _____

LAB. NO.	DATE REQ'D.	BASKET NO.	2.36 mm	1.18 mm	600 µm	300 µm

REMARKS:

PH-CC-336 78-06

Figure 1 - Fine Aggregate Data Card

Ministry of Transportation
Ontario

$MgSO_4$ SOUNDNESS
COARSE AGGREGATE

RUN NO._____

LAB. NO.	BASKET	DUE DATE	GRADING	MASS RETAINED ON			
				37.5 mm	19.0 mm	9.5 mm	4.75 mm

PH-CC-335 88-06

Figure 2 - Coarse Aggregate Data Card