METHOD OF TEST FOR FREEZING AND THAWING OF COARSE AGGREGATE

1. SCOPE

1.1 This method covers the testing of coarse aggregates to determine their resistance to disintegration by repeated freezing and thawing in a sodium chloride solution. It provides information helpful in judging the soundness of aggregates subject to freezing and thawing action, particularly when adequate information is not available from service records of the material exposed to actual weathering conditions.

2. REFERENCES

MTO Report EM-87	Development of an Unconfined Freeze-Thaw Test for Coarse Aggregate
MTO Report MERO-002	Aggregate and Soil Proficiency Sample Testing Program for 2002, February 2003
ASTM E11	Standard Specification for Wire Cloth and Sieves for Testing Purposes
CSA A23.2-24A	Test for Resistance of Unconfined Coarse Aggregate to Freezing and Thawing

3. APPARATUS

3.1 FREEZER: A freezer (chest, stand-up, or walk-in type) capable of maintaining a temperature of -18.0 ± 2.0°C. There shall be a fan that provides adequate air circulation so that the maximum variation within 25 cm of the top and the bottom of the space does not exceed 2.0°C. The tempera-ture of the freezer must be continually monitored at different points within the chamber, either by thermometer or thermocouple. If thermometers are used, the bulb should be in a metal sleeve to avoid sudden temperature changes when the door or lid is opened.

3.2 SIEVES: With square openings and of suitable sizes to furnish the information required by the specification covering the material to be tested. The sieves shall conform to ASTM E11. Half-height sieves shall not be used for sieving material coarser than 9.5 mm.

Sieve Sizes
37.5 mm
26.5 mm
19.0 mm
13.2 mm
9.5 mm
4.75 mm

3.3 THERMOMETERS: Mercury or alcohol type with a range of -35°C to +50°C marked in 1° divisions readable to 0.5°C. All thermometers must be calibrated with an ASTM precision reference thermometer accurate to 0.1°C, at 0°C.

Note 1: Thermometers are available from CANLAB.

Type	Model	Temperature Range
Precision	T2352-62	-38°C to +2°C
Lab Grade	T2025-1C	-35°C to +50°C

3.4 CONTAINERS: Autoclavable plastic mason jars, with airtight screw-on caps able to with-stand a continuous temperature of 110°C. One L jars are used for fractions retained on the 13.2 mm and 9.5 mm sieves and 500 ml jars for the fraction passing 9.5 mm retained 4.75.

Note 2: Autoclavable plastic mason jars are available from CANLAB.

To order, use: J-3030-1 500 ml
J-3030-2 1 L

Note 3: Containers must not come into contact with metal shelves or freezer walls because more rapid cooling will occur.

3.5 PLASTIC MESH BASKETS: Capable of holding four 500 ml jars or two 1 L and one 500 ml jars, and suitable wooden or plastic spacers placed between them to keep the jars from coming in contact with each other. The baskets should be stackable with sufficient clearance for the larger jars.

3.6 BALANCE: A balance or scale accurate to within 0.1% of the sample mass, or 1 g, whichever is greater, over the range required for the test.

3.7 MECHANICAL CONVECTION OVEN: Capable of maintaining a temperature of 110 ± 5°C.

3.8 CONTROL AGGREGATE: A supply of standard Drain Brothers Stoney Lake Quarry coarse aggregate, 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 sufficient 3% sodium chloride solution for the aggregate to be tested. For example, dissolve 30 g of sodium chloride in 970 g of water. Domestic table salt is acceptable. (Studies have shown that a concentration of 3% caused the most severe freeze-thaw damage.) A graduated beaker or cylinder may be used for the water using 1 g = 1 ml, but check accuracy of volume markings. Large quantities of sodium chloride solution should be prepared to minimize any error in measure-ment. Prior to using the solution, the container should be agitated thoroughly.

5. PREPARATION OF SAMPLE

5.1 Aggregate for the test shall consist of material retained on the 4.75 mm sieve (coarse aggregate). Separate the sample into fractions, shown in Table 1, by sieving (see Note 4). Weigh out quantities, as indicated in Table 1, of the different sizes present in the sample. If any fraction constitutes less than 5% of the original sample, it shall not be tested.

Note 4: Continuous sieving of aggregates in mechanical sieve shakers will cause continual break-down. It is good practice to limit mechanical sieving.

Table 1 - Gradings and Masses of Test Samples

Pass	Retained	Mass (g)
37.5 mm	26.5 mm	5000
26.5 mm	19.0 mm	2500
19.0 mm	13.2 mm	1250
13.2 mm	9.5 mm	1000
9.5 mm	4.75 mm	500

The retained material on each sieve is then weighed and the sample placed in the appropriate size jar. Mark a number on the jar and lid, and refer this number to the laboratory number and sieve size by recording on the laboratory test data cards. Lids of jars should be marked with an arrow to indicate the direction of rotation between cycles.

5.2 Every time a freeze-thaw test is conducted, a sample of the standard control aggregate shall also be tested. The material shall be taken from a stock supply and prepared as described in 5.1.

6. PROCEDURE

6.1 Place aggregate passing 19.0 mm retained 13.2 mm in a 1 L jar. Aggregate passing 13.2 mm and retained 9.5 mm is also placed in a 1 L jar. Aggregate passing 9.5 mm retained 4.75 mm is placed in a 500 ml jar. Aggregate larger than 19 mm shall be placed in 2 or more containers so that all of the required sample is tested.

6.2 Saturation of samples in solution: Jars containing samples should be filled with the prepared 3% sodium chloride solution so as to completely immerse all aggregate. Seal the jars with lids to prevent evaporation and keep at room temperature for 24 ± 2 h.

6.3 Draining samples after immersion: Rapidly drain off solution by inverting each jar over a screen smaller than 4.75 mm for approximately 5 sec (do not remove aggregate from jars). For convenience, a screen of 1.18 mm mesh can be cut to fit inside a modified Mason jar lid to facilitate draining and washing. Two or three ml of solution should remain in the jar. Seal the jars to maintain a 100% relative humidity condition.

Note 5: Make sure the lids are screwed on properly to form an airtight seal. When using a lid with a screen to drain off solution or when washing, ensure that the lid is on tight and/or hold a finger against the side of the lid to prevent loss of sample as lids may not fit all jars satisfactorily.

6.4 Freeze thaw cycles: Subject the samples to 5 cycles of freezing and thawing. Place the jars on their sides in baskets with spacers between to separate them. Place the baskets in the freezer at -18.0 ± 2.0°C for 16 ± 2 h (usually overnight), and remove them in the morning to allow approximately 8 h to thaw out at room temperature. Rotate the jars one quarter turn each cycle to ensure that all aggregate particles receive adequate exposure to the solution.

Note 6: Damage due to freezing and thawing is more severe if the rate of cooling in the temperature range -2°C to -12°C is relatively slow. It has been found to be good practice to turn the freezer off, with the door closed, at the end of freezing cycle and then turn the freezer on when the thawed samples are put back in the freezer.

Note 7: If for any reason the sequence of freezing and thawing must be interrupted, the aggregates should be kept frozen inside the container until the cycle can be resumed.

6.5 Wash aggregate after thawing at the end of the fifth cycle. Fill the jar with water and invert over the sink using a lid fitted with a screen as in 6.3 above. Repeat this washing 5 times, without removing the aggregate from the jars.

6.6 Drying samples: Remove the lids from the jars and oven-dry the samples to constant mass at 110°C ± 5°C, usually overnight.

7. QUANTITATIVE EXAMINATION

7.1 After drying the samples, place the aggregate on the same sieve used in the preparation of the sample and shake in the same sieve shaker used for sample preparation for the amount of time established by the procedure given in Appendix I (± 10 sec). Following removal from the sieve shaker, shake gently all of the aggregates on the sieve for a maximum of 5 sec to ensure that thorough sieving has taken place.

Note 8: Sieves may be inverted before the samples are sieved (i.e. 4.75 mm sieve at the top and 13.2 mm sieve at the bottom of the nest). Any material that passes through the 4.75 mm sieve will naturally fall through the larger sieves below into the pan.

Note 9: Further sieving beyond the established sieving time may lead to additional aggregate loss due to mechanical breakdown.

7.2 Weigh the individual sieve fractions and record the mass to nearest 1 g.

8. CALCULATION

8.1 Calculate the per cent loss and weighted average for each fraction as follows:

$$\text{Per cent Loss} = \frac{\text{Original Mass - Mass Retained After Test}}{\text{Original Mass}} \times 100$$

Calculate the per cent loss to the nearest 0.1%.

8.2 Calculate the weighted average loss as follows: Compute the percent of each fraction specified in Table 1 from the coarse aggregate portion of the material, i.e., based on the total mass of material retained on 4.75 mm sieve. Multiply the percentage of each fraction computed by the per cent loss for that fraction. The sum of these products divided by 100 is the weighted average per cent freeze-thaw loss for the sample.

Table 2 - Example

Fraction		Per Cent Loss	Per Cent Retained	Product
Pass	**Retained**			
19.0 mm	13.2 mm	15.0	20.0	300
13.2 mm	9.5 mm	18.0	30.0	540
9.5 mm	4.75 mm	16.0	50.0	800
				Total = 1640

$$\text{Weighted Average} = \frac{1640}{100} = 16.4\%$$

8.3 For the purpose of calculating the weighted average, consider any sizes (not tested) that contain less than 5% of the coarse aggregate portion of the sample to have the same value as the next larger or smaller size, whichever is present.

9. USE OF CONTROL AGGREGATE

9.1 Every time a freeze-thaw test is conducted, a sample of the control aggregate shall also be tested. The material shall be taken from a stock supply and prepared as described in 5.1.

9.2 For the purpose of calculating the weighted average loss of the control aggregate, use the grading shown in Table 3.

Table 3 - Grading of Control Aggregate

Sieve Fraction	Per Cent Retained
19 - 13.2 mm	45
13.2 - 9.5 mm	35
9.5 - 4.75 mm	20

9.3 Control Chart Use: The per cent loss of the last twenty samples of control material shall be plotted on a control chart in order to monitor the variation in results.

9.4 The mean loss of the Drain Brothers Stoney Lake Quarry control aggregate is 11.9% (MTO Report MERO-036, 2010). Individual test data should not normally be greater than 15.3% or less than 8.5%.

9.5 The control aggregate is used to adjust the sieving time required for the quantitative analysis of samples for a given set of equipment (see Appendix I).

10. REPORTING OF RESULTS

10.1 Report the loss on each sieve to the nearest decimal place. Report the weighted loss to the nearest decimal place.

10.2 Report the weighted loss of the Drain Brothers Stoney Lake Quarry control aggregate to the nearest decimal place.

11. PRECISION

For coarse aggregate passing 19.0 mm and retained on 4.75 mm, with freeze-thaw losses in the range of 4% to 18%, the single-operator coefficient of variation has been found to be 7.5%[A]. Therefore, results of two properly conducted tests on samples of the same aggregate by the same operator using the same equipment are not expected to differ by more than 21.2%[A] of their average. The multi-laboratory coefficient of variation has been found to be 20.2%[A]. Therefore, the results of two properly conducted tests by different laboratories on samples of the same aggregate are not expected to differ by more than 57.1%[A] of their average.

[A] These numbers represent, respectively, the (1s%) and (d2s%) limits as described in ASTM C670. The precision estimates provided are based on the results of the proficiency sample testing program conducted by MTO since 2000.

Appendix I: Determination of Sieving Time for Quantitative Analysis

A1. The following procedure has been established to reduce interlaboratory variation of the weighted per cent loss of an individual sample due to differences that may arise from the use of different types of laboratory equipment, e.g. mechanical shaker, diameter of sieves. The amount of time required for quantitative sieving is established through incremental timed-testing of the control aggregate. Sieving time is interpolated from the mean weighted freeze-thaw loss of the control aggregate as given in Section 9. This time is then used for testing all other samples.

A2. Prepare and test a minimum of 3 control aggregate samples.

A3. For each sample, after drying, place the aggregate on the same sieves and shake in the same sieve shaker used for the sample preparation for 1 min. Following removal from the sieve shaker, shake gently all the aggregates on each sieve for a maximum of 5 sec to ensure thorough sieving has taken place (see Note 8).

A4. Weigh each individual sieve fraction separately and record the mass to the nearest 1 g. Return each fraction to the corresponding sieve and continue shaking in the sieve shaker for 1 additional min.

A5. Repeat step A4 for a cumulative total of 5 min.

A6. Repeat steps A3 through A5 for the remaining control aggregate samples (see example, Table A1).

A7. Calculate the cumulative per cent loss for each individual sieve for all the control aggregate samples (see example, Table A2).

A8. Calculate the cumulative weighted freeze-thaw loss for each control aggregate sample at each incremental test time (see example, Table A3).

A9. Calculate the average weighted freeze-thaw loss of each sample at each incremental test time (see example, Table A3).

A10. Plot the average weighted freeze-thaw loss vs. sieving time (see example, Figure A1).

A11. For the freeze-thaw loss of the control aggregate given in Section 9, interpolate the required sieving time from the plot (see example, Figure A1).

A12. Use the sieving time determined in these steps in the quantitative examination of samples as given in Section 7.

A13. Repeat this procedure to establish a quantitative sieving time for each individual sieve set, shaker, or combination thereof every 12 months or whenever changes in equipment or control aggregate occur.

Example. Weighted Per Cent Freeze-Thaw Loss: LS-614 Control Aggregate

Sieve Set: Set A, 300-mm diam., 13.2 mm, 9.5 mm, 4.75 mm, pan
Shaker: Shaker X, Gilson/ROTAP RX-30-2-CAN, 300 mm

Table A1 - Mass of Individual Sieves as a Function of Time

Sample	Sieve	Initial	1 Minute	2 Minutes	3 Minutes	4 Minutes	5 Minutes
1	13.2	1250.6	1190.8	1178.3	1157.1	1145.2	1135.8
	9.5	1000.8	903.1	890.7	884.1	876.1	870.0
	4.75	500.5	415.1	409.8	406.8	404.5	402.6
2	13.2	1250.5	1173.0	1157.5	1141.6	1136.8	1121.9
	9.5	1000.8	889.3	882.3	878.2	872.9	865.1
	4.75	500.7	421.6	412.5	406.8	403.9	401.2
3	13.2	1251.0	1182.5	1173.7	1144.4	1133.4	1132.5
	9.5	1000.1	888.6	877.1	870.8	860.9	853.6
	4.75	500.9	418.7	413.6	407.5	403.4	401.5

Table A2 - Cumulative Per Cent Loss for Individual Sieves

Sample	Sieve	1 Minute	2 Minutes	3 Minutes	4 Minutes	5 Minutes
1	13.2	4.78	5.78	7.48	8.43	9.18
	9.5	9.76	11.00	11.66	12.46	13.07
	4.75	17.06	18.12	18.72	19.18	19.56
2	13.2	6.20	7.44	8.71	9.09	10.28
	9.5	11.14	11.84	12.25	12.78	13.56
	4.75	15.80	17.62	18.75	19.33	19.87
3	13.2	5.48	6.18	8.52	9.40	9.47
	9.5	11.15	12.30	12.93	13.92	14.65
	4.75	16.41	17.43	18.65	19.46	19.84

Table A3 - Average Weighted Freeze-Thaw Loss

Time (Min.)	Sample			Average(%)
	1	2	3	
1	8.81	9.73	9.51	9.35
2	9.91	10.88	10.44	10.41
3	11.03	11.81	11.95	11.60
4	11.84	12.28	12.86	12.33
5	12.47	13.21	13.23	12.97

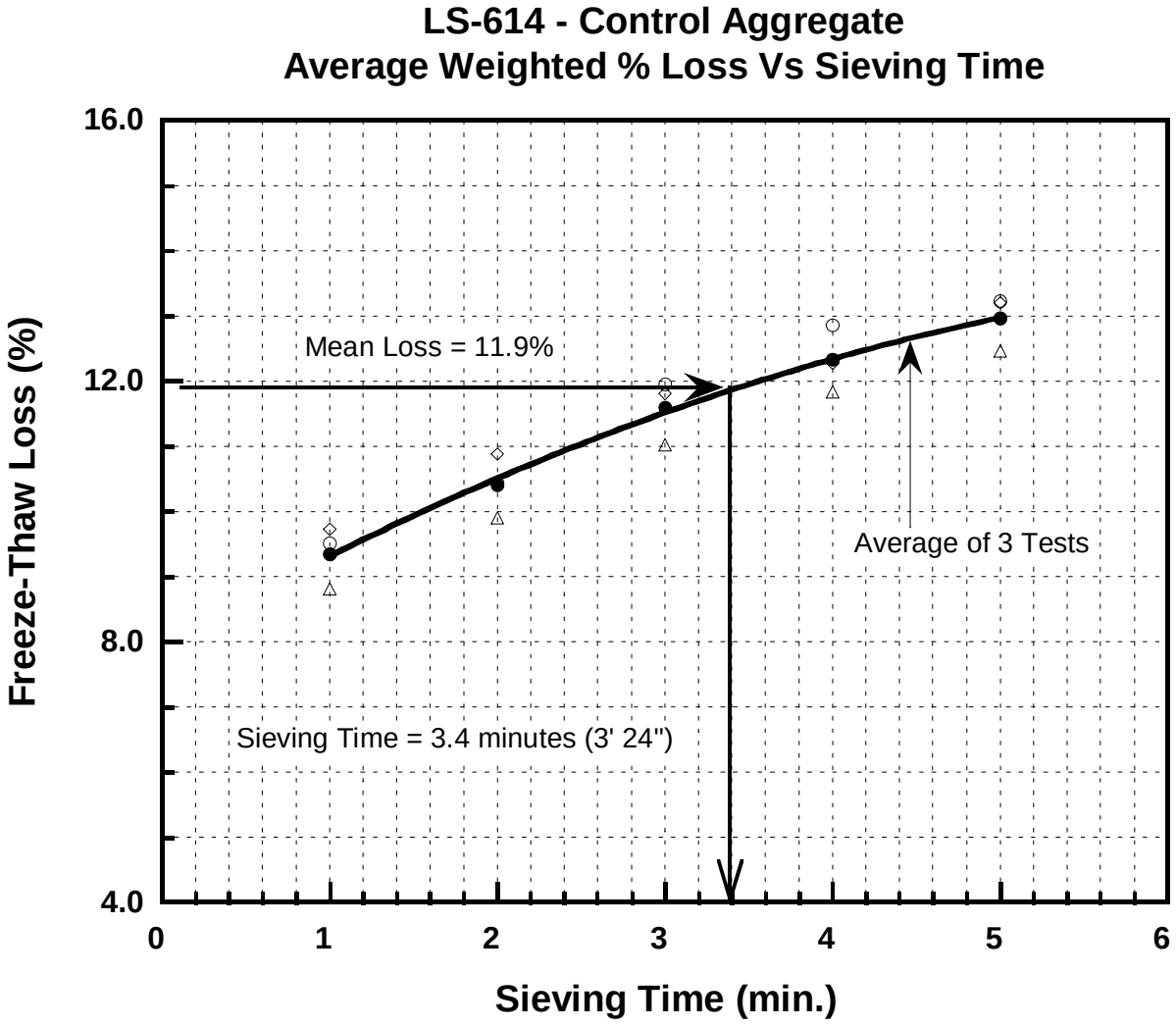

Figure A1 - Plot of Average Weighted Freeze-Thaw Loss of Control Aggregate Vs Time (The time of 3 min 23 sec is established for quantitative sieving of samples when prepared, tested, and evaluated using sieve set A and shaker X).

Note: The plot shown above is that of the Control Aggregate from Drain Brothers Stoney Lake Quarry.

Ministry of Transportation Ontario

FREEZE THAW TEST
COARSE AGGREGATE

DATE TEST STARTED ____________

LAB. NO.	JAR NO.	MASS RETAINED						PERCENT LOSS
		13.2 mm		9.5 mm		4.75 mm		
		INITIAL	FINAL	INITIAL	FINAL	INITIAL	FINAL	

REMARKS :

PH-CC-434 85-12

Figure 1 - Test Data Card