

NOVA SCOTIA DEPARTMENT

OF

TRANSPORTATION AND INFRASTRUCTURE RENEWAL

TEST METHODS MANUAL March 2018

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The Test Methods Manual is a compilation of non-standardized test methods intended to provide the user with procedures and test methods applicable to work undertaken on Nova Scotia Department of Transportation and Infrastructure Renewal (the Department) projects. In the event Test Method clarification or correction is required, the user is advised to contact the Department's Highway Construction Services division at (902) 860-2999.

March 2018

TEST METHOD (TM-1) - THE RESISTANCE OF COARSE AGGREGATE TO DEGRADATION BY ABRASION IN THE MICRO-DEVAL APPARATUS

1.0 SCOPE

1.1 This method covers the testing of coarse aggregates to determine their abrasion loss in the presence of water and an abrasive charge. It provides information helpful in judging the suitability of coarse aggregate subjected to weathering action.

2.0 RELEVANT DOCUMENTS

- 2.1 Ontario Ministry of Transportation, Test Method LS-618, Method of Test for the Resistance of Coarse Aggregate to Degradation by Abrasion in the Micro-Deval Apparatus
- 2.2 ASTM C136, Standard Method for Sieve Analysis of Fine and Coarse Aggregate

3.0 APPARATUS

- 3.1 MICRO-DEVAL ABRASION MACHINE: A jar rolling mill capable of running at 100 ± 5 rpm (Figure 1).
- 3.2 CONTAINERS: Stainless steel, Micro-Deval abrasion jars having a 5 Litre capacity with a rubber ring in the rotary locking cover. Internal diameter = 194 mm, internal height = 170 mm. The inside and outside surfaces of the jars shall be smooth and have no observable ridges or indentations.
- 3.3 ABRASION CHARGE: Stainless steel balls are required. These shall have a diameter of 9.5 ± 0.5 mm. Each jar requires a charge of $5000 \pm 5g$ of balls.
- 3.4 SIEVES: The sieves shall conform to CAN/CGSB-8.2-M88

20 000 µm	16 000 μm
14 000 µm	10 000 μm
6 300 μm	5 000 μm
1 250 μm	

- 3.5 OVEN: An oven capable of maintaining a temperature of $110 \pm 5^{\circ}$ C.
- 3.6 BALANCE: A balance or scale accurate to 0.1 g

3.7 LABORATORY CONTROL AGGREGATE: A supply of Brechin standard reference aggregate available from the Soils and Aggregates Section, Ministry of Transportation, 1201 Wilson Avenue, Downsview, Ontario, M3M 1J8.

4.0 **PREPARATION OF TEST SAMPLE**

4.1 Aggregate for the test shall normally consist of material passing the 20 000 μ m sieve, retained on the 10 000 μ m sieve. An oven dry sample of $1500 \pm 5g$ shall be prepared as follows:

Passing	Retained	Mass
20 000 µm	16 000 μm	375 g
16 000 μm	14 000 μm	375 g
14 000 μm	10 000 μm	750 g
	TOTAL	1500 g

4.2 In cases where the maximum nominal size of the coarse aggregate is less than 16 000 μ m, a sample of 1500 \pm 5g shall be prepared as follows:

Passing	Retained	Mass
16 000 μm	10 000 μm	375 g
10 000 μm	6 300 μm	375 g
6 300 μm	5 000 μm	750 g
	TOTAL	1500 g

5.0 TEST PROCEDURE

- 5.1 Wash the sample.
- 5.2 Oven dry the sample to constant mass.
- 5.3 Prepare a representative 1500 ± 5 g sample. Record the Mass "A" to the nearest 1.0 g.
- 5.4 Saturate the sample in 2.0 ± 0.05 L tap water (temperature 20 ± 5^0 C) for a minimum of 1 hour.
- 5.5 Place the sample in the Micro-Deval abrasion container with $5000 \pm 5g$ of steel balls and the water. Place the Micro-Deval container on the machine.

Note: It is permissible to saturate the sample in the water within the Micro-Deval abrasion container rather than a separate container.

- 5.6 Run the machine at 100 ± 5 rpm for 2 hours ± 1 minute.
- 5.7 Carefully pour the sample over two superimposed sieves:

The 5 000 μ m and 1 250 μ m sieves are to be used. Take care to remove the entire sample from the stainless steel jar. Wash the retained material (a hand-held sprayer might be useful) until the washings are clear. Remove the stainless steel balls using a magnet or other suitable means.

- 5.8 Combine the material retained on the 5 000 μ m and 1 250 μ m sieves, being careful not to lose any material.
- 5.9 Oven dry the sample to a constant mass at $110 \pm 5^{\circ}$ C.
- 5.10 Weigh the sample to the nearest 0.1 g. Record the mass as "B".

6.0 CALCULATIONS

6.1 Calculate the micro-Deval abrasion loss, as follows, to the nearest 0.1%.

Percent Loss =
$$\underline{\mathbf{A} \cdot \mathbf{B}}$$
 X 100 A

7.0 USE OF LABORATORY CONTROL AGGREGATE

- 7.1 A sample of the standard reference aggregate shall also be tested every 10 samples. The material shall be taken from a stock supply and prepared according to 4.1.
- 7.2 Control Chart Use: The percent loss of the last twenty samples of reference material shall be plotted on a control chart in order to monitor the variation in results (Figures 2 and 3).

8.0 REPORT

The report shall include the following:

- 8.1 The percent loss of the test sample to one decimal place
- 8.2 The maximum size of the aggregate tested.
- 8.3 The percent loss of the reference sample, tested closest to the time at which the aggregate sample was tested, to one decimal place.
- 8.4 The percent loss of the last twenty samples of reference material on a control chart.

9.0 **PRECISION**

In a limited (Ontario Ministry of Transportation) study, it was found that the multi-laboratory coefficient of variation varies depending on the level as shown in Table 1. The difference between two samples of the same material tested in two experienced laboratories should only exceed the D2S value shown in column 3 one time in twenty.

Material Property (% Mean Loss)	Coefficient of Variation (%)	D2S (% of Value)
5	10	28
12	6.4	18
17	5.6	16

TEST METHOD (TM-1B) - THE RESISTANCE OF FINE AGGREGATE TO DEGRADATION BY ABRASION IN THE MICRO-DEVAL APPARATUS

1.0 SCOPE

1.1 This method covers the testing of fine aggregates to determine their abrasion loss in the presence of water and an abrasive charge. It provides information helpful in judging the suitability of fine aggregate subjected to weathering action.

2.0 RELEVANT DOCUMENTS

- 2.1 Ontario Ministry of Transportation, Test Method LS-619, Method of Test for the Resistance of Fine Aggregate to Degradation by Abrasion in the Micro-Deval Apparatus
- 2.2 ASTM C136, Standard Method for Sieve Analysis of Fine and Coarse Aggregate

3.0 APPARATUS

- 3.1 MICRO-DEVAL ABRASION MACHINE: A jar rolling mill capable of running at 100 ± 5 rpm.
- 3.2 CONTAINERS: Stainless steel, Micro-Deval abrasion jars having a 5 Litre capacity with a rubber ring in the rotary locking cover. Internal diameter = 194 mm, internal height = 170 mm. The inside and outside surfaces of the jars shall be smooth and have no observable ridges or indentations.
- 3.3 ABRASION CHARGE: Stainless steel balls are required. These shall have a diameter of 9.5 ± 0.5 mm. Each jar requires a charge of $1250 \pm 5g$ of balls.
- **NOTE:** Prior to use, new containers and new steel balls should be conditioned. Conditioning is accomplished by running the equipment with a charge of 500 g of silica sand with 750 ml of water for a period of 4 hours. At the end of 4 h, this procedure must be repeated with a new sand sample. From time to time, it may be necessary to recondition the containers and steel balls. The need for this will be indicated by significant change in loss with the control material. It has been found that reconditioning is usually needed when the equipment has been used for testing carbonate coarse aggregate in LS-618, which leads to polishing of the container and ball surface.

3.4 SIEVES: The sieves shall conform to ASTM E11 specifications:

2.36 mm 150 μm

1.18 mm 75 μm

600 µm

300 µm

A minimum 200 diameter 75 μ m sieve is to be used for washing the aggregate. A 6.7 mm sieve will be found useful for separating the steel balls from the aggregate.

- 3.5 OVEN: An oven capable of maintaining a temperature of $110 \pm 5^{\circ}$ C.
- 3.6 BALANCE: A balance or scale accurate to 0.1 g.
- 3.7 LABORATORY CONTROL AGGREGATE: A supply of Brechin standard reference aggregate available from the Soils and Aggregates Section, Ministry of Transportation, 1201 Wilson Avenue, Downsview, Ontario, M3M 1J8.

4.0 **PREPARATION OF TEST SAMPLE**

4.1 Aggregate for the test shall normally consist of material passing the 4.75 mm sieve, Split a representative 725 ± 25 g sample for the Micro-Deval test and place in a sealed container.

5.0 TEST PROCEDURE

- 5.1 Wash the sample over 75 μ m sieve until the wash water is clear, as described in LS-601, Method of Test for Materials Finer than 75 μ m Sieve in Mineral Aggregates by Washing.
- 5.2 Oven dry the sample to constant mass at a temperature of $110 \pm 5^{\circ}$ C.
- 5.3 The sample shall be sieved into separate sizes. The sample shall be made up to a fineness modulus (FM) of 2.8 using the gradation given below and then tested. Record the Mass 'A' to the nearest 0.1 g.

Passing	Retained	Mass	
4.75 mm	2.36 mm	50 g	
2.36 mm	1.18 mm	125 g	
1.18 mm	600 μm	125 g	
600 µm	300 µm	100 g	
300 µm	150 μm	75 g	
150 µm	75 μm	25 g	
	TOTAL	500 g	

- Note: Where prior testing has shown the loss of material from a source to be less than 17%, a representative 500 ± 5 g sample from the original 700 g sample may be tested without sieving into individual fractions. Testing of low Micro-Deval loss material without sieving into separate fractions has a small effect on the measured loss and may considerably reduce the complexity of the testing. If testing shows this loss to be greater than 17%, then a further sample shall be tested, which has been prepared to an FM of 2.8 using the gradation specified in 5.3. This loss shall be reported as the final test result.
- 5.4 Saturate the sample in tap water for 24 ± 4 hours.
- 5.5 Pour off excess water and place the sample in the Micro-Deval abrasion container with $1250 \pm 5g$ of steel balls and the water. Place the Micro-Deval container on the machine.
- 5.6 Run the machine at 100 ± 5 rpm for 15 minutes ± 10 sec.
- 5.7 Carefully pour the sample through a 6.7 mm sieve over a pan. Wash the aggregate over a 75 μ m sieve, according to the procedure described in LS-601, being careful not to lose any retained 75 μ m material.
- 5.8 Oven dry the sample to a constant mass at 110 ± 5^0 C.
- 5.9 Weigh the sample to the nearest 1.0 g. Record the mass as "B".

6.0 CALCULATIONS

6.1 Calculate the Micro-Deval abrasion loss, as follows, to the nearest 0.1%.

Percent Loss = $\underline{\mathbf{A} \cdot \mathbf{B}}$ X 100 A

7.0 USE OF LABORATORY CONTROL AGGREGATE

7.1 Every 10 samples, but at least every week in which the sample was tested, a sample of the standard reference aggregate shall be tested. The material shall be taken from a stock supply and prepared according to the following procedure. The material shall be sieved into separate sizes, and each size fraction thoroughly washed and dried to a constant mass. The reference sample shall be made up using the following gradation:

Passing	Retained	Mass	
4.75 mm	2.36 mm	40 g	
2.36 mm	1.18 mm	115 g	
1.18 mm	600 μm	180 g	
600 µm	300 µm	120 g	
300 µm	150 μm	38 g	
150 µm	75 µm	7 g	
	TOTAL	500 g	

7.2 Control Chart Use: The percent loss of the last twenty samples of reference material shall be plotted on a control chart in order to monitor the variation in results. The average loss of the control material should be 16.8%. Individual test data should not normally be greater than 18.4% or less that 15.2%.

8.0 REPORT

The report shall include the following:

- 8.1 The percent loss of the test sample to one decimal place.
- 8.2 The percent loss of the reference sample tested closest to the time at which the aggregate sample was tested, to 1 decimal place.
- 8.3 The percent loss of the last twenty samples of reference material on a control chart.

9.0 **PRECISION**

9.1 For the fine aggregate with abrasion losses in the range from 7% to 18%, the single-operator coefficient of variation has been found to be 3.0%^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 8.5%^A of their average, 95% of the time. The multi-laboratory coefficient of variation has been found to be 7.7%^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 21.8%^A of their average, 95% of the time.

^A These numbers represent, respectively, the (1s%) and (d2s%) limits as described in ASTM C670. The data are based on the analyses of the test results from 60 to 77 laboratories that tested twelve pairs of fine aggregate proficiency test samples covering a twelve year period from 2000 to 2011.

TEST METHOD (TM-2) - THE PETROGRAPHIC ANALYSIS OF COARSE AGGREGATE

1.0 SCOPE

- 1.1 This test method outlines the procedures to be employed in the petrographic analysis of coarse aggregate. It is a method of appraising the quality of coarse aggregate, and provides a numerical means of expressing the quality of a coarse aggregate sample.
- 1.2 This method does not attempt to fully undertake a geological classification of the aggregate particles. It is a simplified version of the Ontario Ministry of Transport Test Method LS-609. The test invokes a scratch hardness evaluation of the aggregate, and is designed to gauge the (apparent) durability of the aggregate.

2.0 RELEVANT DOCUMENTS

2.1 ASTM C136, Sieve Analysis of Fine and Coarse Aggregates

3.0 APPARATUS

- 3.1 Hand Lens: 10 X magnification
- 3.2 Pocket Knife: Good quality with a blade hardness of between 5.5 and 6 on the Moh's Scale
- 3.3 Hydrochloric Acid: Technical Grade, 5% by volume
- 3.4 Balance: Of sufficient capacity and sensitive to 0.1g

4.0 **PREPARATION OF SAMPLE**

4.1 A representative sample of oven dried aggregate shall be prepared to the following approximate masses:

Passing	Retained	Approx. Mass (g)
20 000 μm	14 000 μm	800
14 000 μm	10 000 μm	500
10 000 μm	5 000 μm	200

5.0 TEST PROCEDURE

5.1 Conduct the scratch hardness evaluation using the following procedure and criteria:

The scratch hardness evaluation should be conducted by firmly holding the test knife and applying moderate pressure while using a sawing motion along the aggregate particle.

Good: Aggregate particle cannot be scratched with the knife blade, dislodged Surficial grains of the particle do not constitute a scratch

Fair: Aggregate particle can be scratched

Poor: Particle can be easily grooved

Deleterious: Particle will crumble with finger pressure

- 5.2 Each examined particle shall be placed in their respective qualitative categories, i.e. good, fair, poor, or deleterious.*
- 5.3 The dilute hydrochloric acid may be applied by an eye-dropper to particles suspected of being acid reactive, i.e.: carbonates. This is undertaken for the purposes of general classification.
- 5.4 The examined particles from the various qualitative factor categories can be further subdivided into rock type.
- 5.5 On completion of the examination, each group of rock type shall be weighed to the nearest 0.1 g.

*Basalt or any similar trap rock may be upgraded from "fair" to "good" should tested components produce a scratch. Heavily weathered particles shall not be considered "good"

6.0 CALCULATION

- 6.1 The percentage of each rock type shall be calculated to the nearest 0.1 percent.
- 6.2 The percentage of each rock type within their respective qualitative groups will be added together to produce a total percentage for that group. All groups added together should equal 100 percent of the aggregate sample.
- 6.3 The percentage of each of the qualitative groups shall be factored in the following manner:

Good Group	Percentage of Group X 1
Fair Group	Percentage of Group X 3
Poor Group	Percentage of Group X 6
Deleterious Group	Percentage of Group X 10

6.4 The summation of the factored groups is the Petrographic Number or PN. The Petrographic Number shall be reported to the nearest whole number.

7.0 **REPORT FORM**

7.1 A report form for the calculation of the Petrographic Number is appended to this test method.

DEPARTMENT OF TRANSPORTATION AND PUBLIC WORKS PETROGRAPHIC ANALYSIS REPORT

Client	Cont. No	Map Sheet		
Pit Name	County	Map Reference		
FACTOR 1		MASS, g	PERCENTAGE	
Quartzite				
Volcanic, felsic				
Volcanic, mafic (Trap Rock)				
Sandstone				
Metagreywacke				
Granite				
Diorite, Gabbro				
	Total		P ₁	
FACTOR 3				
Sandstone				
Volcanic, mafic (Trap Rock)				
Granite				
Gneiss				
Carbonates				
	Total		P ₃	
FACTOR 6				
Sandstone, Siltstone, friable				
Volcanic, decomposed				
Granite, decomposed				
Slate				
Shale				
Carbonates				
	Total		P ₆	
FACTOR 10				
Slate				
Shale				
Conglomerate				
	Total		\mathbf{P}_{10}	

Factor 1	1	Х	$P_1 =$	
Factor 3	3	Х	$P_3 =$	
Factor 6	6	Х	$P_6 =$	
Factor 10	10	Х	$P_{10} =$	

TOTAL

Performed By

Agency _____

TEST METHOD (TM-3) - DETERMINATION OF PERCENT FRACTURED PARTICLES IN PROCESSED COARSE AGGREGATES

1.0 SCOPE

1.1 This method covers the visual determination of the percent, by mass, of fractured particles in a processed coarse aggregate.

2.0 RELEVANT DOCUMENTS

2.1 ASTM C136, Sieve Analysis of Fine and Coarse Aggregates

3.0 **DEFINITIONS**

- 3.1 **Asphalt concrete aggregate:** A Fractured Particle is defined as a piece of coarse aggregate which has at least **two well defined fresh faces** resulting from fracture, with each of the fractured faces comprising no less than 20 percent of the particle surface area. Particles with smooth faces and rounded edges, or with only small chips removed, are not considered fractured.
- 3.2 **Base granular aggregate:** A Fractured Particle is defined as a piece of coarse aggregate which has at least **one well defined fresh face** resulting from fracture, with the face comprising no less than 20 percent of the particle surface area. Particles with smooth faces and rounded edges, or with only small chips removed, are not considered fractured.

4.0 APPARATUS

4.1 Balance: Of sufficient capacity and sensitive to 1 g or less.

5.0 PREPARATION OF TEST SAMPLE

- 5.1 Prepare the coarse aggregate sample by conducting a sieve analysis in accordance with ASTM C136. Material passing the 5000 µm sieve can be discarded. Washing the aggregate particles is permissible when dust or coatings make it difficult to determine well defined fractured faces. However, the sample must be dried before weighing.
- 5.2 The test sample shall be representative of the original sample gradation and the sample mass shall consist of one of the size ranges as outlined in Table 1.

TABLE	1:
	.

COARSE AGGREGATE FRACTION		APPROX. MASS, g
PASSING	RETAINED	
56 000 μm	5 000 μm	2000
28 000 μm	5 000 μm	1500
20 000 µm	5 000 μm	1000
14 000 μm	5 000 μm	750

6.0 TEST PROCEDURE

- 6.1 Spread the test sample on a clean, flat surface large enough to permit the particles to be visually examined.
- 6.2 Separate the particles into fractured and unfractured portions.
- 6.3 Weigh each fractured and unfractured portion of the sample to the nearest 1 g.

7.0 CALCULATION

7.1 The mass of fractured particles divided by the total sample mass multiplied by 100 shall be reported as percent fractured particles.

REFEREE PROCEDURE - PETROGRAPHIC ANALYSIS OF COARSE AGGREGATE (TM-2) AND PERCENT FRACTURED PARTICLES IN PROCESSED COARSE AGGREGATE (TM-3)

1.0 SCOPE

This recommended practice describes the protocol for initiating a referee process for both the petrographic analysis of coarse aggregate and the percent fractured particles, proper sampling techniques, sample preparation and distribution, as well as appropriate test procedures. The purpose is to secure, through acceptable practices, a mechanism for dispute resolution for either test.

2.0 RELEVANT DOCUMENTS

2.1 ASTM Standards:

D75-87	Standard Practice for Sampling Aggregates	
C702-87	Standard Practice for Reducing Field Samples of Aggregate to Testing Size	
C136-87	Sieve Analysis of Fine and Coarse Aggregates Standard Specification, Division 4, Section 4	
TIR	General Test Methods, TM-2 & TM-3	

3.0 DISPUTE MECHANISM

2.2

- 3.1 In the event of a disagreement regarding the Petrographic Number or Fractured Particles percentage, the Contractor shall note their objections in writing to the Project Engineer such that the Engineer will notify the Manager of Highway Construction Services of impending referee testing. An analysis will then be done as follows:
- 3.2 The Manager will arrange to have the stockpile sampled. If an Inspector Specialist is not available to sample, the Project Engineer or a representative will sample the stockpile. The Contractor will observe the sampling procedure.
- 3.3 The sample will be taken to Highway Construction Services. All consultants that are retained by the Department for asphalt concrete quality control, or a minimum of two consultants, will be asked to test a representative portion of the sample.
- 3.4 The sample will be split and each Technician will perform the Petrographic Test or the Fractured Particles Test. The results will be given to the Manager, who will make the final decision with respect to the use of the aggregate, using the test results as a guide.

4.0 SAMPLING PROCEDURE

- 4.1 The sampling of the aggregate shall be undertaken by an Inspector Specialist, Project Engineer, or representative. A minimum 500 tonnes of stockpiled aggregate is required before sampling can be undertaken. Sampling shall be conducted in accordance with ASTM D75-87. Modifications to the ASTM procedure are as follows:
- 4.2 Representative vertical slices of the stockpile shall be taken by means of heavy equipment supplied by the proponent. The use of heavy equipment is deemed necessary in order to gain access to all areas of the stockpile. A minimum of four vertical slices shall be taken, with each slice "backdragged" in order to expose the vertical profile. A representative sample of each slice, having a minimum mass of 30 kg, shall be collected and prepared for reduction to test size.
- 4.3 The reduction of the field sample shall be undertaken in accordance with ASTM C702-87. This method ensures further representative sampling as well as sample reduction by use of either a sample splitter or sample quartering on a canvas tarp.

5.0 CONTRACTOR NOTIFICATION:

The Contractor shall be notified when the sampling and the testing is to be undertaken.

TEST METHOD (TM-4) - MOISTURE IN ASPHALT MIXES BY OVEN OR HOT PLATE DRYING

1.0 SCOPE

1.1 This method describes the procedure for determining the moisture content of plant produced asphalt concrete mixes using a field laboratory oven or hot plate.

2.0 APPARATUS

- 2.1 Scale capable of measuring to 0.1 g accuracy.
- 2.2 An oven or hot plate with a temperature range of 50° C to 150° C.
- 2.3 Pans, large mixing pan for bulk sampling and one drying pan for the moisture test.
- 2.4 Thermometer with a minimum range of 50° C to 150° C.
- 2.5 Hand tools, scoop, spatula, gloves.

3.0 PREPARATION OF TEST SAMPLE

3.1 Obtain a representative test specimen of the asphalt mix from a portion of the extraction test sample.

4.0 TEST PROCEDURE

- 4.1 Weigh a dying pan and record on Line "D".
- 4.2 Place the specimen into a mixing pan and use a heated scoop to thoroughly blend the mix.
- 4.3 Place a minimum of 500 g of mix in the drying pan. Weigh the pan and mix and record as Wt. of Moist Sample + Plan (line "A") to the nearest 0.1 g.
- 4.4 Place the drying pan and mix in the oven or on the hot plate set at $130^{0}C \pm 5^{0}C$ for a minimum of 1 hour. Replace the sample in the oven or on the hot plate until two consecutive weights are the same. Check the temperature with a thermometer.
- 4.5 Weigh the hot sample and record as Wt. of Dry Sample + Pan (line "B") to the nearest 0.1 g.

5.0 CALCULATIONS

- 5.1 Calculate the weight of water removed to the nearest 0.1% (line "C") as follows: Wt. of Water (g) = (Wt. of Moist Sample + Pan) - (Wt. of Dry Sample + Pan)
- 5.2 Determine the oven dry weight of the mix sample (line "E") as follows:

Wt. of Dry Sample (g) = (Wt. of Dry Sample + pan) - (Wt. of Pan)

5.3 Calculate the Moisture Content (line "F") of the plant mix using the formula:

 $Moisture \ Content \ (\%) = \frac{Wt. \ of \ Water}{Wt. \ of \ Dry \ Sample} x \ 100$

6.0 **REPORT**

Report the percent moisture to the nearest 0.1%.

DRYING IN TIME OUT А Wt. of Moist Sample + Pan g В Wt. of Dry Sample + Pan g С Wt. of Water A - B g D Wt. of Pan g Е Wt. of Dry Sample B - D g F Moisture Content C / E %

Moisture Content - Asphalt Mix

TEST METHOD (TM-5) - OBTAINING LOOSE ASPHALT CONCRETE MIX SAMPLES & OBTAINING CORES FROM COMPACTED ASPHALT CONCRETE SURFACES

1.0 SCOPE

This procedure covers sampling of loose asphalt concrete mix and the sampling of compacted asphalt concrete samples by coring.

2.0 RELEVANT DOCUMENTS

- 2.1 ASTM D979, Standard Practice for Sampling Bituminous Paving Mixtures
- 2.2 ASTM D3665, Practice for Random Sampling of Construction Materials
- 2.3 ASTM D5361, Practice for Sampling Compacted Bituminous Mixtures for Testing
- 2.4 NSTIR Standard Specifications for Highway Construction & Maintenance

3.0 APPARATUS

- 3.1 Square mouth shovel or scoop (supplied by the Contractor)
- 3.2 Square metal plate and collar assembly (supplied by the Contractor)
- 3.3 Sample boxes, bags, labels (supplied by the Contractor)
- 3.4 Core drill, minimum nominal diameter of 100 mm (4 in) (supplied by Contractor / Consultant)
- 3.5 Calculator, computer, tables to generate random numbers
- 3.6 Measuring tape
- 3.7 Spray paint or lumber crayon and template.

4.0 **PROCEDURE**

- 4.1 Contractors or Consultants shall use the methods noted below for sampling asphalt concrete material:
 - Method "A" Loose Sampling From the Road Mat
 - Method "B" Loose Sampling From Truck Boxes
 - Method "C" Core Sampling From the Mat
 - Method "D" Core Sampling From the Longitudinal Joint
 - Method "E" Loose Samples from the Material Transfer Vehicle (MTV)

METHOD "A" Loose Samples from the Mat

- **1.0** A production Lot will be divided into segments of approximate equal quantity. The number of segments is defined by the project. In each segment, a random tonnage sample will be determined by using random numbers.
- **2.0** The Engineer shall determine the number of lots and random tonnage samples for the project. See NSTIR TM-5 Method "B" or Method "E" for other examples of this calculation. See NSTIR Standard Specifications for Highway Construction and Maintenance Division 4 Section 19 for further clarification on work categories and lot sizes.

Example 1: Random Tonnage Sample: Loose Samples Method "A", "B" or "E"

A 2400 tonne Lot divided into 4 equal segments = 600 t Sub-Lots. One loose sample will be required from each 600 t Sub-Lot.

Generate four random numbers: 0.285, 0.610, 0.316 and 0.854

Sample #1:	0 to 600 t: 600 t segment x 0.285 = 171 t. Sample #1 at 171 tonnes.
Sample #2:	600 to 1200 t: 600 t + (600 t segment x 0.610) = 966 t. Sample #2 at 966 tonnes.
Sample #3:	1200 to 1800 t: 1200 t + (600 t segment x 0.316) = 1390 t. Sample #3 at 1390 tonnes.
Sample #4:	1800 to 2400 t: 1800 t + (600 t segment x 0.854) = 2312 t. Sample #4 at 2312 tonnes.

- **Note:** If it is not feasible to obtain the sample at the designated tonnage because the paver is being moved, plant break down, etc., then once production resumes the Engineer shall obtain a sample from the 5th load.
- **3.0** The Contractor shall obtain the minimum number of quantity-based loose asphalt concrete samples from each Lot as required by the project and in the presence of the Engineer.
 - 3.1 The loose samples shall be obtained from the full depth of the mat avoiding contamination from underlying materials. A square metal plate and/or collar assembly is recommended for obtaining the sample. The metal plate shall be wide enough to obtain the required sample size and to prevent degradation or contamination. A collar, if used, shall have perpendicular sides and proper height so that mix does not spill over the top. The collar shall be sufficiently smaller than the plate to facilitate positioning after the mat has been placed.

- 3.2 The plate shall be positioned two to three minutes prior to sampling in front of the paving train and anchored so that truck tires or paver tracks shall not cause any disturbance as they pass over. The location of the plate shall be identified on the shoulder of the road with spray paint or lumber crayon or wire.
- 3.3 Once the paver has passed over the plate, the location of the plate shall be determined. If a steel collar is used, it shall be centered and pressed down through the mix until it reaches the plate. Care shall be taken to ensure the entire area of the collar falls inside the boundary of the plate. All loose mix on the plate or within the collar shall be sampled. The plate, collar and all other tools shall be scraped as clean as possible and the fines included in the sample. The total sample mass shall be 20 to 25 kg for all mixes.
- **Note:** If the mix to be sampled contains an obvious defect, such as, but not limited to, segregation, excessive or insufficient liquid asphalt, contamination, etc., the Contractor may exclude it from sampling and shall repair the defective area. Another sample shall be taken immediately in front of the paving train.
- 3.4 The total sample shall then be split down into two equal portions by hand quartering or using a riffle sample splitter. The asphalt concrete mix shall be placed into sample boxes or bags. One portion shall be labeled "Quality Assurance Testing" and the other portion "Appeal Testing".
- 3.5 A label shall be attached to the sample containing the following information:
 - Contract number, Lot number, Sample number, Date, Time, Tonnage, Mix type and Sample location (station, offset, and lane)
- 3.6 The sample area must be reinstated such that the resulting mix is uniform, non-segregated, and well compacted.

METHOD "B" Sampling From Truck Boxes

- **1.0** A production Lot will be divided into segments of approximate equal quantity. The number of segments is defined by the project. In each segment, a random tonnage sample will be determined by using random numbers.
- **2.0** The Engineer shall determine the number of lots and random tonnage samples for the project. See NSTIR TM 5 Method "A" or Method "E" for other examples of this calculation. See NSTIR Standard Specifications for Highway Construction and Maintenance Division 4 Section 19 for further information on work categories and lot sizes.

Example 1: Random Tonnage Sample: Loose Samples Method "A", "B" or "E"

The remaining asphalt plant production balance to be placed on a work category 3 project is 900 tonne. The Lot is divided into three (3) equal segments = 300 t Sub-Lots. One loose sample will be required from each 300 t Sub-Lot.

Generate three random numbers: 0.199, 0.610 and 0.418.

Sample #1:	0 to 300 t: 300 t segment x 0.199 = 60 t. Sample #1 at 60 tonnes.
Sample #2:	300 to 600 t: 300 t + (300 t segment x 0.610) = 483 t. Sample #2 at 483 tonnes.
Sample #3:	600 to 900 t: 600 t + (300 t segment x 0.418) = 725 t. Sample #3 at 725 tonnes.

- **Note:** If it is not feasible to obtain the sample at the designated tonnage because the paver is being moved, plant break down, etc., then once production resumes the Engineer shall obtain a sample from the 5th load.
- **3.0** The Contractor shall obtain the minimum number of quantity-based loose asphalt concrete samples from each Lot as required by the project and in the presence of the Engineer.
 - 3.1 The loose sample shall be obtained from a truck box at the plant site, after the truck is fully loaded. Each truck box to be sampled shall be divided into equal quadrants numbered one to four as in Figure 1. The Engineer shall generate a random number for each loose sample, then multiplied by four (rounded to the nearest whole number) to determine which quadrant shall be sampled. Each sample shall be drawn entirely from one quadrant.

1	2
4	3

Figure 1 - Quadrants for Random sampling from a Loaded Truck

Sample #1 at 171 tonnes: $4 \ge 0.422 = 2$ Sample #2 at 966 tonnes: $4 \ge 0.527 = 2$ Sample #3 at 1390 tonnes: $4 \ge 0.661 = 3$ Sample #4 at 2312 tonnes: $4 \ge 0.335 = 1$

- 3.2 The Contractor shall use a square mouth shovel or scoop, driven into the mix at an angle perpendicular to the undisturbed loose mix surface in the truck box. The total sample mass shall be 20 to 25 kg drawn throughout and entirely from the designated quadrant. The shovel or scoop shall then be scraped as clean as possible and the fines are to be included in the sample.
- 3.3 The total sample shall then be split down into two equal portions by hand quartering or using a riffle sample splitter. The asphalt concrete mix shall be placed into sample boxes or bags. One portion shall be labelled "Quality Assurance Testing" and the other portion "Appeal Testing".
- 3.4 A label shall be attached to the sample containing the following information:
 - Contract number, Lot number, Sample number, Date, Time, Tonnage, Mix type and Sample location (truck number, ticket number and sampling quadrant)

METHOD "C" Core Sampling of the Mat

1.0 For mat density core samples, the Engineer shall determine the random tonnage sample locations. A Lot will be divided into segments of approximate equal quantity. In each segment, a random tonnage sample will be determined by using random numbers.

Example 1: Random Tonnages Sample: Core Samples for Mat Density

Lot divided into 5 equal segments = 480 t Sub-Lots. One core sample will be required from each 480 t Sub-Lot.

Generate five random numbers for core tonnage: 0.088, 0.584, 0.737, 0.557 and 0.370 Generate five random numbers for core offset: 0.245, 0.288, 0.019, 0.492 and 0.095

Segme	nt #1,	0 to 480 tonnes: 480 t x $0.088 = 42$ tonnes Offset for 3.65m width: 0.245 x (3.65w - 0.3m) + 0.15m = 0.97 metres
Segme	nt #2,	480 to 960 tonnes: 480 t + (480 t x 0.584) = 761 tonnes Offset for 3.65m width: 0.288 x (3.65w - 0.3m) + 0.15m = 1.11 metres
Segme	nt #3,	960 to 1440 tonnes, 960 t + (480 x 0.737) = 1314 tonnes Offset for 3.70m width: 0.019 x (3.70w - 0.3m) + 0.15m = 0.21 metres
Segme	nt #4,	1440 to 1920 tonnes: 1440 t + (480 t x 0.557) = 1707 tonnes Offset for 3.70m width: 0.492 x (3.70w - 0.3m) + 0.15m = 1.82 metres
Segme		1920 to 2400 tonnes: 1920 t + (480 t x 0.370) = 2098 tonnes Offset for 3.70m width: 0.095 x (3.70w - 0.3m) + 0.15m = 0.47 metres
<u>Notes:</u>		Cores not taken within 10 metres of a loose sample location. Cores not taken within 6 metres of a transverse joint. Cores not taken within 0.15 metres of longitudinal joint. Cores not taken within 0.15 metres of outside edge of mat. Offset distance measured from left edge of mat in the direction of stationing.
2.0		ontractor shall retrieve a minimum of 5 (five) core samples from each Lot, in the presence of the Engineer.
	2.1.	The Engineer will spray paint a 50 mm diameter circle (using a template) to identify the core sample location.
	2.2.	The core samples shall include the entire 50 mm painted circle, and be free of distortion and damage and have a minimum diameter of 100 millimetres.

2.3. Cores that are damaged during sampling or handling shall be discarded and new samples shall be taken adjacent (within 0.3 metres) to the original

sample location.

- 2.4. The Contractor shall reinstate the pavement at each core sample location in conjunction with removal of the core, by de-watering the core hole and filling it with hot mix asphalt concrete. The hot mix asphalt concrete shall be compacted by applying 25 blows with Marshall Hammer to achieve a dense, surface smooth plug.
- 2.5. Each core sample is to be identified with Lot and sample number using a lumber crayon. The sampling form shall be completed noting the coring date and station.

METHOD "D" Core Sampling of the Longitudinal Joint

1.0 For joint density core samples, the Engineer shall determine the random chainage sample locations associated with each Lot for the project. A Lot (total entire length of joint per lift) will be divided into 5 or more segments of approximately equal length. The maximum length of a segment will be 1000 metres.

Example 1: Random Chainage Sample: Core Samples for Joint Density

Lot divided into a minimum of 5 equal segments (maximum length 1000 metres). One core sample will be required from each segment.

Generate five random numbers for core segments: 0.038, 0.061, 0.488, 0.220 and 0.690

	Length of Contract 4.8 km 4.8 km $/5 = 0.96$ km segment
Segment #1,	0 to 0.96: 0.96 x 0.038 = 0.036 or Station 0+036
Segment #2,	0.96 to 1.92: 0.96 + (0.96 x 0.061) = 1.019 or Station 1+019
Segment #3,	1.92 to 2.88: 1.92 + (0.96 x 0.488) = 2.389 or Station 2+389
Segment #4,	2.88 to 3.84: 2.88 + (0.96 x 0.220) = 3.091 or Station 3+091
Segment #5,	3.84 to 4.80: 3.84 + (0.96 x 0.690) = 4.503 or Station 4+503

- **Notes:** Cores not taken within 6 metres of a transverse joint. The distance will be measured in the direction of chainage.
 - 1.1 After final rolling, the Engineer will identify the longitudinal joint by measuring and recording the width of the 1st mat at the designated station. Upon completion of the adjacent mat (including final rolling) the Engineer will visually identify the longitudinal joint and confirm by re-measuring the 1st mat. The core location shall be marked by spray painting a 50 mm diameter circle (using a template) over the joint to identify the core sample location.
- **2.0** The Contractor shall retrieve a minimum of 5 (five) core samples from each Lot, in the presence of the Engineer.
 - 2.1. The cores shall be centred over the longitudinal joint and samples shall include the entire 50 mm painted circle, and be free of distortion and damage and have a minimum diameter of 100 millimetres.
 - 2.2. Cores that are damaged during sampling or handling shall be discarded and new samples shall be taken adjacent (within 0.3 metres) to the original sample location.

- 2.3. The Contractor shall reinstate the pavement at each core sample location in conjunction with removal of the core, by de-watering the core hole and filling it with hot mix asphalt concrete. The hot mix asphalt concrete shall be compacted by applying 25 blows with Marshall Hammer to achieve a dense, surface smooth plug.
- 2.4. Each core sample is to be identified with Lot and sample number using a lumber crayon. The sampling form shall be completed noting the coring date and station.

METHOD "E" Loose Samples from the Material Transfer Vehicle (MTV)

- **1.0** A production Lot will be divided into segments of approximate equal quantity. The number of segments is defined by the project. In each segment, a random tonnage sample will be determined by using random numbers.
- **2.0** The Engineer shall determine the number of lots and random tonnage samples for the project. See NSTIR TM 5 Method "A" or Method "B" for other examples of this calculation. See NSTIR Standard Specifications for Highway Construction and Maintenance Division 4 Section 19 for further information on work categories and lot sizes.

Example: Random Tonnage Sample: Loose Samples Method "A", "B" or "E".

Lot of 1500 tonnes divided into three (3) equal segments = 500 t Sub-Lots. One loose sample will be required from each 500 t Sub-Lot.

Generate three random numbers: 0.376, 0.710, and 0.890

- Sample #1: 0 to 500 t: 500 t segment x 0.376 = 188 t. Sample #1 at 188 tonnes.
- Sample #2: 500 to 1000 t: 500 t + (500 t segment x 0.710) = 855 t. Sample #2 at 855 tonnes.
- Sample #3: 1000 to 1500 t:1000 t + (500 t segment x 0.890) = 1445 t. Sample #3 at 1445 tonnes.
- **Note:** If it is not feasible to obtain the sample at the designated tonnage because the paver is being moved, plant break down, etc., then once production resumes the Engineer shall obtain a sample from the 5th load.
- **3.0** The Contractor shall obtain the minimum number of quantity-based loose asphalt concrete samples from each Lot as required by the project and in the presence of the Engineer.
 - 3.1 For each random tonnage sample, the sample shall consist of one sample collected with an approved mechanical splitter (i.e. Quartermaster etc.), which will then split the sample into four (4) equal portions.
 - 3.2 Clean the splitter and, if necessary, apply a light coating of approved release agent to the surfaces that will come into contact with asphalt concrete.
 - 3.3 Close and secure hopper gates on the mechanical splitter.

- 3.4 Obtain asphalt concrete sample from the material transfer vehicle by directing the conveyor into the mechanical splitter hopper. Avoid segregation, loss of mix or the accidental addition of foreign material. The total sample mass shall be 20 to 25 kg per sample for all mixes.
- 3.5 Place the four sample containers in the splitter so that there is no loss of material. The asphalt concrete mix shall be placed into sample boxes or bags.
- 3.6 Release the handle(s), allowing the asphalt concrete to drop through the divider chutes and discharge into the four receptacles.
- 3.7 Any asphalt concrete that is retained on the surface of the splitter shall be removed and placed into the appropriate receptacle.
- 3.8 Close and secure the hopper gates on the mechanical splitter.
- 3.9 Remove two portions of the sample from diagonally opposite corners. Label one portion "Quality Assurance Testing" and the other portion "Appeal Testing".

The label shall be attached to the sample and must contain the contract number, lot number sample number, date, time, tonnage, mix type, sample method and location.

3.10 The remaining two portions of the sample are to be used for the contractor's purposes (i.e. quality control testing etc.).

TEST METHOD (TM-6) - PROCEDURES FOR GRAVEL SAMPLING FOR TPW INSPECTORS

1.0 SCOPE

This procedure covers the sampling of gravels during production, as well as post production sampling from stockpiles. The importance of correct and consistent sampling cannot be stressed enough. Test results, particularly for gradation, may have little practical value if the material tested is not representative of the material produced.

2.0 **REFERENCE DOCUMENTS**

- 2.1 ASTM C702 Standard Practice for Reducing Field Samples of Aggregate to Testing Size
- 2.2 ASTM D75 Standard Practice for Sampling Aggregates
- 2.3 NSTIR Standard Specification, Highway Construction and Maintenance, Division 3 Section 2 and Division 4 Section 4

3.0 EQUIPMENT

- 3.1 Heavy equipment (front-end-loader)
- 3.1 Square blade shovel
- 3.1 Brush or broom
- 3.1 Mechanical Splitter (optional)
- 3.1 Tarpaulin or a hard flat surface (sheet plywood etc.)
- 3.1 Sample bags

4.0 SAMPLE SIZE

Maximum Nominal Size of Aggregates*	Approximate Minimum Mass of Field Sample (kg)	
Sand		
Winter Sand (5 mm)	10	
Coarse Aggregate		
Type 1 (14 mm)	25	
Type 2 (56 mm)	100	
Type M (20 mm)	50	

5.0 SAMPLING

5.1 Sampling during Production

Do not sample from the very start or near the end of production as there is a much greater risk of obtaining segregated material. The procedure detailed below is modified from a procedure outlined in ASTM D75.

- 5.1.1 Utilize a front-end loader or other suitable heavy equipment to obtain the field sample. This will ensure that the minimum size field sample is obtained as detailed in the table in Section 3. Sampling utilizing heavy equipment is also potentially safer and requires less effort than trying to retrieve a sample manually.
- 5.1.2 Once consistent production from the crusher is achieved, position the loader bucket beneath the falling stream of gravel.
- 5.1.3 Obtain about ¹/₂ a loader bucket of gravel.
- 5.1.4 Dump the gravel on a level area at a safe location away from vehicular traffic.
- 5.1.5 Back-drag the pile with the loader bucket to create a layer of gravel approximately 200 to 300 mm thick.
- 5.1.6 With a square blade shovel, remove several shovels worth of material (or more depending on the volume) from the flattened pile in a random pattern. It may be helpful to establish a rough grid on the flattened pile and take a shovelful from some of the squares. Avoid the very edges of the pile where material may segregate.
- 5.1.7 Reduce this sample by splitting or quartering to a size suitable for testing.
- 5.2 Sampling from Stockpile

Due to the large volumes of material typically contained in stockpiles it is by nature difficult to obtain representative samples. Stockpiling, even when properly done, may induce segregation. Generally speaking, the larger the nominal size of the aggregate the greater the potential for segregation and the greater the effort required to obtain a representative sample.

- 5.2.1 Utilize a front-end loader or other suitable heavy equipment to obtain the field sample.
- 5.2.2 Take a vertical slice of the stockpile, from the bottom to the top, at a minimum of four locations around the pile (more if the pile is very large).
- 5.2.3 Dump and back-drag each bucket of gravel as described in subsection 5.1.
- 5.2.4 Sample each slice pile as described in subsection 5.1.
- 5.2.5 Reduce each sample by splitting or quartering to a size suitable for testing.

5.3 Other Sampling

ASTM D75 details techniques for sampling from conveyor belts, trucks, rail cars and barges. If you are presented with circumstances beyond your experience please contact Highway Construction Services, (902) 860-2999, for assistance.

6.0 SPLITTING OR QUARTERING

Field samples must be reduced to a size appropriate for testing. This section will describe ways to accomplish this and will help ensure that the test sample is representative of the field sample. Methods described below are based on those outlined in ASTM C702.

6.1 Mechanical Splitters

Sample splitters have an even number of equal width chutes which discharge on each side of the splitter. The width of the chutes determines the maximum nominal size gravel that can be processed. The splitter is equipped with at least two pans that fit under the chutes and collect the sample. Fine aggregates (sand) that are moister than saturated surface dry (SSD) should not be reduced by mechanical splitter (SSD sands would flow easily without clumping). Quartering is the preferred method for sand that is wetter than SSD.

- 6.1.1 Use a 64 mm (2.5 in) splitter for Gravel Type 2 or Type M. Use a 19 mm (3/4 in) or 25 mm (1 in) splitter for sands or Gravel Type 1/1S.
- 6.1.2 Place the field sample in the hopper chute and uniformly distribute it from edge to edge.
- 6.1.3 Allow the material to flow through the chute into the catch pans.
- 6.1.4 Discard the material in one of the pans.
- 6.1.5 Place the material from the remaining pan into the hopper chute and repeat this procedure until a test sample of the desired size is obtained.
- 6.2 Quartering

In the absence of a mechanical splitter the quartering method may be used. A square blade shovel, a broom or brush and a hard flat surface are required. The hard surface, a sheet of plywood, sheet metal or whatever is available, must be large enough to contain the sample when it is spread out. A canvas tarpaulin may be used where another suitable work surface is unavailable.

- 6.2.1 Place the field sample on the hard, clean, level surface.
- 6.2.2 Mix the material by turning the entire sample over at least three times.
- 6.2.3 With the last turning, shovel the entire sample into a conical pile.

- 6.2.4 Carefully flatten the conical pile to a uniform thickness. The diameter of the flattened pile should be approximately four to eight times the thickness.
- 6.2.5 Divide the flattened pile into four equal quarters with a shovel.
- 6.2.6 Remove two opposite quarters and sweep away any fines left behind by the shovel.
- 6.2.7 Combine the two remaining quarters, mix well and repeat the process until a sample size suitable for testing is achieved.

7.0 TESTING

After completing the procedures outlined above, a sample of gravel of an appropriate size for testing will be produced. As a final check before testing compare the test sample to the material being produced or already in stockpile. Verify that the test sample is a fair representation of what is seen in the pit or quarry.

Once you are satisfied that you have a proper sample proceed with testing. Compare the gradation test results with the requirements outlined in Division 3, Section 2 of the Standard Specification.

TEST METHOD (TM-7) - WORKABILITY OF COLD BITUMINOUS PATCHING MATERIAL BY BLADE RESISTANCE METHOD

1.0 SCOPE

1.1 This test method covers the determination of the workability property of cold patching material (CPM) in cold temperatures (e.g. at -100C). The handling property of CPM is duplicated in this method by the action of a blade penetrating a mixture.

2.0 SUMMARY OF METHOD

- 2.1 This test measures the workability of CPM in cold temperatures by using a modified Marshall tester holding a metal blade onto a proving ring. The blade is used to penetrate a compacted sample. The resistance to the blade penetration in Newton (N) after 30 seconds of blading is a measure of the workability of a CPM, and is called the workability index (W.I.); the higher W.I. is, the poorer the workability.
- 2.2 The consolidation of CPM in the stockpile is simulated by compacting a CPM sample, at room temperature in a wooden box, with a modified flat foot square hand held Marshall hammer. The sample is then conditioned in a cold room or a freezer before testing. Duplicate samples are prepared for each mix.

3.0 SIGNIFICANCE AND USE

- 3.1 CPM is commonly used for patching minor surface defects in routine asphalt pavement maintenance in the winter. The ease of handling and application, compaction and performance of CPM are all affected by workability. It is, therefore, important to have some means of measuring this property.
- 3.2 The method enables the mix workability, as indicated by the ease of shoveling a mix in a stockpile, to be quantitatively measured rather than by the subjective judgment of an operator. The measurement will allow the operator to assess the effort required for removing a mix from a stockpile or truck box by shoveling or scooping.

4.0 APPARATUS

- 4.1 WOODEN BOXES: Two boxes measuring 265 mm x 165 mm x 50 mm deep inside dimensions, and sufficiently rigid for forming and testing of cold mix specimen.
- 4.2 COMPACTION HAMMER: A hand held hammer with a modified flat tamping foot of 150 mm x 50 mm x 6 mm thick attached to the bottom of the hammer.
- 4.3 FREEZER OR COLD ROOM: Capable of maintaining a temperature at $-10^0 \pm 2^0$ C.

- 4.4 MARSHALL TESTING MACHINE: A breaking head modified to hold a steel blade measuring 130 mm wide, 50 mm height and 3 mm thick to the adapter at the bottom of the proving ring.
- 4.5 STAND: Any stand of suitable height and configuration for supporting the wooden box.
- 4.6 METAL SCOOP: Flat bottom type for transferring mixes.
- 4.7 THERMOMETER: A type suitable for measuring a range of mix temperature from $+25^{\circ}$ to -25° C with sensitivity of $\pm 2^{\circ}$ C.
- 4.8 BALANCE: 5 kg capacity, with sensitivity of 1 g.
- 4.9 METAL PANS OR BOWLS.

5.0 **PREPARATION OF TEST SPECIMENS**

5.1 Prepare two specimens using the material to be tested.

5.2 MIX PREPARATION

- 5.2.1 Bring samples of cold patching material (about 5 kg) to room temperature $(21^0 \pm 3^0 \text{C})$.
- 5.2.2 Weigh into two separate wooden boxes the amount (about 2 kg) that will result in a compacted specimen height of 48 or 50 mm.
- 5.2.3 Spread the mix uniformly over the box and level the mix, avoiding any segregation. Discard any excess material.

5.3 COMPACTION OF SPECIMENS

- 5.3.1 Place the box with the material on any hard and level surface, and place the compaction hammer with the modified foot close to one side of the box and apply two blows with the hammer. Apply another two blows to the material on the other side of the box.
- 5.4 Mark three locations along the length of the box, approximately at 1/4 points, to indicate the position for the blade penetration.
- 5.5 Place the compacted material and the box in a freezer or cold room for a minimum of 12 hours (i.e. overnight) at -10° C.

6.0 **PROCEDURE**

6.1 Attach the penetration blade to the adapter at the bottom of proving ring of the Marshall apparatus with the blade parallel to the front of the machine.

- 6.2 Adjust the dial indicator to zero.
- 6.3 Transfer the box with the compacted material from the freezer or cold room to the Marshal apparatus. Place it under the blade and on the support stand, ensuring that it is level and firmly seated on the support stand.
- 6.4 Switch on the motor and bring the specimen close to the blade. Position the specimen at the marked location.
- 6.5 Continue the upward movement of the jack head and start the stop watch when the dial indicator begins to move. Watch the dial carefully and record the reading after 30 seconds of penetration.
- 6.6 Stop the motor, lower the jack head and move the specimen to the next testing position. Clean the blade, if necessary. Repeat the blade penetration procedure for the remaining two locations. Record the readings on the dial indicator.
- 6.7 Repeat the above procedure on the second specimen.

7.0 CALCULATION

- 7.1 Multiply each of the three dial readings by the proving ring calibration factor to calculate the blade resistance values.
- 7.2 Calculate the average of the three values.
- 7.3 Repeat the same for the second specimen.

8.0 **REPORT**

The report shall include the following information.

- 8.1 Identification and description of the mix or sample.
- 8.2 The W.I. by averaging of the two calculated values from the two specimens.

TEST METHOD (TM-8) - CHEMICAL ANALYSIS OF MINERALIZED ROCK MEASUREMENT OF ACID-PRODUCING POTENTIAL

1.0 OBJECT

This test method provides a standard test for the prediction of the acid producing potential of sulphide minerals and their waste waters.

2.0 BACKGROUND

The production of acid mine waters arises from the oxidation of metallic sulfide minerals, particularly those containing iron. In theory, this oxidation can occur either chemically or biologically, but in practice the bacterium *Thiobacillus ferrooxidans* is always present in acid mine waters, suggesting that the biological mechanism is predominant.

Thiobacillus ferrooxidans is a unique bacterium; its energy for growth is obtained from the oxidation of reduced sulfur compounds (i.e. sulfides) and ferrous iron. It is the only organism known that has the capability of oxidizing the sulfide portion of insoluble metallic sulfides. It does so by the direct attachment to the solid surface. The bacterium requires an aquatic environment; air is the source of the oxygen and carbon dioxide required. It requires a source of ammonia nitrogen as well as small amounts of phosphate, calcium, magnesium, etc. which are usually present in natural waters.

Thiobacillus ferrooxidans attacks the sulfide minerals by attachment and direct oxidation of the insoluble sulfides. If iron is present, it is oxidized simultaneously. The sulfide ion is probably attached by an enzyme containing a sulfhydryl group and a polysulfide chain is built up. The sulfur atoms on this chain are ultimately oxidized through to the sulfate ion which is released into solution. Ferrous iron, if present, is oxidized by a different enzyme system and is released as ferric iron.

Microbiological acid production from sulfide minerals can be illustrated as follows, using pyrite as an example.

 $4FeS_2 + 15O_2 + 14H_2O \rightarrow 4Fe(OH)_3 + 8H_2SO_4 \qquad (1)$

This equation represents the complete hydrolysis of all the ferric iron and the production of two moles of sulfuric acid per mole of pyrite. In practice, we find that the iron does not all precipitate as ferric hydroxide, but rather as a basic ferric sulfate or jarosite-type mineral, represented by the formula $AFe_3(SO_4)_2(OH)_6$ where A can be H⁺, NH_4^+ , K⁺, Na^+ , etc. Assuming all the iron precipitates as hydronium-jarosite, the following equation would explain the oxidation of pyrite.

 $6FeS_2 + 22\frac{1}{2}O2 + 15H_2O \rightarrow 2HFe_3(SO_4)_2(OH)_6 + 8H_2SO_4$ (2)

On this basis, 1.33 moles of sulfuric acid are produced per mole of pyrite. In practice, neither equation 1, nor equation 2, applies completely so the actual amount of sulfuric acid produced in a natural leaching situation will be between 1.33 and 2 moles per mole of

pyrite or between 0.67 and 1 mole per mole of sulfide present.

With chalcopyrite (CuFeS₂), maximum acid production is predicted by equation 3, which has all the iron precipitating as ferric hydroxide.

$$2CuFeS_2 + 8\frac{1}{2}O_2 + 5H_2O \rightarrow 2CuSO_4 + 2Fe(OH)_3 + 2H_2SO_4$$
(3)

This is the equivalent of one mole of sulfuric acid per mole of chalcopyrite or 0.5 moles per mole of sulfide. Again, in practice most of the iron tends to precipitate as a jarosite according to equation 4.

 $6CuFeS_2 + 25 \frac{1}{2}O_2 + 9H_2O \rightarrow 6CuSO_4 + 2HFe_3(SO4)_2(OH)_6 + 2H_2SO_4$ (4)

This results in one mole of acid per 3 moles of pyrite or 0.17 mole of acid per mole of sulfide.

Sulfides such as millerite (NiS) and sphalerite (ZnS) can also be oxidized to their corresponding sulfates. However, free acid is not formed and thus the pH will not drop as low as when the iron salts hydrolyse. However, these sulfate salts do have an acid pH and must be neutralized in order to reduce the mobility of the heavy metals.

It is evident from the foregoing discussion that the maximum amount of acid that can be produced is 1 mole per mole of sulfur present. However, in practice the amount of free acid is usually considerably less, but if one assumes a 1:1 ratio a safety factor will ensue. The initial portion of the following test procedure is based on the assumption that 1 mole of acid will be produced per mole of total sulfur present, which is the maximum figure theoretically possible. If a potential danger is indicated by this relatively simple test, then the confirmation portion of the test should be initiated to evaluate the real danger of microbiological acid production.

Bacteria are also capable of producing acid by the oxidation of dissolved components present in waters emanating from mining and milling operations. In this presentation we are only concerned with the formation of strong acids, i.e. sulfuric, and not with the formation of weak organic acids resulting from heterotrophic growth on available organic matter.

There are two possible sources of strong acid arising from soluble components: the oxidation of ferrous iron and the oxidation of reduced sulfur compounds such as thiosulphate or polythionates.

If ferrous iron is present it can be oxidized either chemically or biologically as follows:

 $2Fe^{+2} + \frac{1}{2}O_2 + 2H^+ - 2Fe^{+3} + H_2O$ (5)

This reaction consumes acid but the ferric iron then hydrolyzes

 $2Fe^{+3} + 6H_2O - 2Fe(OH)_3 + 6H^+$ (6)

for a net gain of two moles of hydrogen ions per mole of ferrous iron.

With reduced sulfur compounds either chemical or biological oxidation can take place depending on conditions. The amount of acid released would depend on the ionic species present, the nature of the associated cations and the mode of oxidation. Three possible situations are given by equations (7), (8) & (9).

$S_2O_3^{=} + 2O_2 + H_2O - 2SO_4^{=} + 2H^+$	(7)
$S_{3}O_{6}^{=}+20_{2}+2H_{2}O-3SO_{4}+4H^{+}$	(8)

 $S_4O_6^{=} + 3 \frac{1}{2}O_2 + 3H_2O - 4SO_4 = + 6H^+$

These equations assume complete oxidation of all the reduced sulfur compounds, a result normally occurring only in the presence of sulfur-oxidizing bacteria. Chemical oxidation in the acidic environment is normally incomplete.

(9)

3.0 TEST PROCEDURES FOR EVALUATING ACID-PRODUCING POTENTIAL OF ORE AND WASTE ROCK

- 3.1 Initial Test
 - 3.11 Sample The sample selected must be taken in such a manner that it is truly representative of the type of mineralization being examined. A composite made up of split drill core or randomly selected grab samples should be satisfactory. The number of samples to be examined will depend on the variability of the mineralization and must be left to the discretion of the geologist taking the samples. The bulk sample is crushed to a size which can be conveniently handled, (i.e. -2 inch) and then thoroughly mixed and approximately a 2 lb portion split out, using the usual coning and quartering technique. This sample is then pulverized to pass a 100 mesh screen and used for assay, the titration test, and the confirmation test if necessary.
 - 3.1.2 Assay The pulverized sample is assayed in duplicate for total sulfur. The use of a Leco furnace is recommended, although a chemical oxidation technique followed by barium sulfate precipitation is satisfactory. The total sulfur assay value is expressed as pounds of sulfuric acid per ton of sample, assuming a 1:1 conversion factor, which is the acid-producing potential of the sample.
 - 3.1.3 Titration test Duplicate 10 gram portions of the pulverized sample are suspended in 100 ml of distilled or de-ionized water and stirred for approximately fifteen minutes. The natural pH of the sample is then recorded and the sample titrated to pH 3.5 with 1.0 Normal sulfuric acid and left stirring. If an automatic titrator is used the test is continued unless than 0.1 ml of acid is added over a 4 hour period. If manual titration is used, the addition of acid is repeated every half-hour (approximately) until the pH

change over a 4 hour period is 0.1 pH unit or less. The total volume of acid added is recorded and converted to lbs per ton of sample. This is the acid-consuming ability of the sample, i.e. $\underline{ml of 1.0 N H_2SO_4 x 0.049 x 2000}$ Acid-Consuming ability (lb/ton) = wt if sample in g or for a 10g sample = ml of 1.0 N H_2SO_4 x 9.8

3.1.4 Interpretation - If the acid consumption value (in lbs of acid per ton of sample) exceeds the acid-producing potential (lbs per ton) then the sample will not be a source of acid mine drainage and no additional work is necessary. If the acid consumption is less than the acid-producing potential, the possibility of acid mine water production exists and the confirmation test should be conducted.

3.2 Confirmation Test

This test examines the amenability of the sulfides in the test sample to oxidation through the action of **T. ferrooxidans**, which is the only organism presently known to be capable or oxidizing insoluble metallic sulfides. The organism also has the ability to oxidize ferrous iron. Experience has shown that not all sulfide minerals are amenable to microbiological attack, nor do they all oxidize completely and so the acid-producing potential indicated by the assay may overestimate the danger.

- 3.2.1 Sample The remaining portion of the pulverized sample is ball milled (wet) for 2 3 hours to produce a -400 mesh sample which is dried overnight at 105^oC.
- 3.2.2 Shake-flask leaching test Duplicate 30 gram portions (or smaller amount if the sulfide exceeds 2%) are placed in 250 ml Erlenmeyer flasks with 70 ml of a nutrient medium containing 3 g/l (NH₄)₂SO₄ ; 0.10g/l KCl; 0.50 g/l K₂HPO₄; 0.50 g/l MgSO₄.7H₂O; 0.1 g/l Ca (NO₃)₂. Add sufficient sulfuric acid (use either 12 or 36 Normal) to bring the pH to 2.5. Shake the flask for approximately 4 hr and the pH should be between 2.5 and 2.8. If necessary add additional acid until the pH remains in that range, and then inoculate the flask with 5 ml of an active culture of *Thiobacillus ferrooxidans*. Record the weight of the flask and contents. Plug the flask with a loose cotton plug and incubate at 35^oC on a gyratory shaker.

The experimental leaching flask is returned to its original weight before sampling by adding distilled or de-ionized water. Monitor the pH and concentration of a dissolved metal, eg. iron or copper, for the first three days to ensure that the pH remains below 2.8. Thereafter, monitor every second day until microbiological activity has ceased, ie. the pH no longer drops or the dissolved metal concentration remains constant.

When microbiological activity has ceased, add half the weight of feed used originally (ie. 15 grams), shake 24 hr and record the pH. If it is greater than pH 3.5, terminate the test. If it is pH 3.5 or less, again add half the weight of feed (ie. 15 grams) and shake for 24 hr. If the pH is less than 3.5 or greater than pH 4, the

experiment is terminated. If the pH is between 3.5 and 4.0, the sample is shaken an additional 48 hr and the final pH value recorded.

3.2.3 Interpretation - The object of this test is to determine if the sulfide oxidizing bacteria can generate enough sulfuric acid from the sulfides present in the sample to satisfy its acid demand. If they can, microbiological action will continue on a self-sustaining basis and acid mine water will result. In this test, the acid demand is satisfied initially by adding sulfuric acid. This permits the bacteria to generate the maximum amount of sulfuric acid from the sample concerned. Once microbiological action has ceased, half the original feed weight is added. If there has not been sufficient acid production, the pH approaches the natural pH of the sample (i.e. be above pH 3.5) and the sample is reported as not being a potential source of acid mine water. If the pH remains at 3.5 or below, the remainder of the sample is added and the sample is shaken for up to 72 hr before measuring the final pH. If the pH is still in the leaching range, i.e. pH 3.5 or below, there is a strong possibility that natural leaching will occur and acid mine drainage will be produced. If the pH is above 3.5, there is no possibility of acid mine drainage occurring.

If the sample produces excess acidity, there is the possibility of metal recovery by microbiological leaching. A measure of this potential can be obtained be estimating the percentage of the contained metal which has been solubilized during the leaching test. Under such circumstances, it may be desirable to promote microbiological action as means of recovering valuable metals from a waste material. In such a system, suitable precautions must be taken to prevent the metal and acid rich leach waters from entering the natural drainage system of the surroundings.

4.0 TEST PROCEDURE FOR EVALUATING ACID-PRODUCING POTENTIAL OF MINE WATER

4.1 Sample

The water sample must be taken in a clean bottle with a tight cap. The sample must fill the bottle completely so that all air is excluded and the sample must be returned to the laboratory for testing within 3 hr. If any iron originally in solution precipitates before the test is initiated the sample must be discarded.

- 4.2 Test Procedure
 - 4.2.1 Place a 500 ml portion of the water sample in a 1 litre Erlenmeyer flask, measure the pH, and add 5 ml of each of the following solutions (w/v): 30% (NH₄)₂SO₄; 5% KH₂PO₄; 5%MgSO₄ 7H₂O; 1% KCl; 0.1% Ca(NO₃)₂. Adjust the pH to 4.0 with sulfuric acid and record the amount added. Inoculate with 50 ml of an active culture of *Thiobacillus ferrooxidans* which has been grown on ferrous sulfate. Stopper with a cotton plug and incubate at 35^oC on a gyratory shaker until the pH stabilizes (approximately 10 days).

- 4.2.2 Set up a control flask containing 500 ml of distilled or de-ionized water in the same manner and incubate at 35^oC. Add the same amount of acid regardless of the final pH.
- 4.2.3 Titrate the test and control flasks back to the original pH of the test sample. Convert the difference in titer into mg/l of sulfuric acid which represents the acid produced by the sample.
- 4.3 Interpretation

The acid-producing potential of the sample in mg/l sulfuric acid must be compared with the alkalinity of the sample as determined by "Standard Methods" in order to assess the potential effect on the test water. An alternative method would be to add the equivalent amount of sulfuric acid to a portion of the test sample and measure the final pH.

Summary

A test for estimating the potential for acid mine drainage production has been presented. The first part of the initial test involves obtaining knowledge of the total sulfur content of the sample and thus estimating the theoretical amount of sulfuric acid that can be produced from that sample. The second part of the initial test involves determining by titration the acid-consuming ability of the gangue mineralization which would be required to neutralize the acid if it should be produced. By comparing the acid-producing capability and the acid consumption an estimate of the potential for excess acid production is obtained.

If the initial tests indicate that excess acid production is theoretically possible, then the confirmation test becomes necessary to determine if the bacteria are actually converting enough of the sulfur into sulfuric acid that the sample is a net acid producer. This is done by inoculating a portion of the test sample with *Thiobacillus ferrooxidans*, under conditions which will promote maximum acid production. An equivalent amount of the original sample is then added and its ability to consume the acid produced is monitored. If the fresh portion of sample is able to consume all the acid that the bacteria have produced, then there is no danger of acid mine drainage occurring.

A test to determine the acid-producing potential of mine waters, due to the presence of dissolved iron and reduced sulfur compounds, involves the microbiological oxidation of these compounds by *Thiobacillus ferrooxidans* under standard conditions and the measurement of the resulting change in titrable acidity. This value must be assessed in terms of the alkalinity of the sample. A chemical oxidation technique does not appear satisfactory because of the incomplete oxidation of the reduced sulfur compounds.

This article was written by D W Duncan, Group Leader, Mineral Microbiology, Division of Applied Biology for the Department of Environment, Environmental Protection Service, Ottawa, Ontario K1A 0H3 July 24, 1972.

TEST METHOD (TM-9) - PROCEDURES FOR SUBSURFACE INVESTIGATIONS

1.0 SCOPE

1.1 This procedure details the requirements for conducting a variety of subsurface investigations for Transportation and Infrastructure Renewal (TIR).

2.0 RELEVANT DOCUMENTS

- 2.1 ASTM D1452 Practice for Soil Investigation and Sampling by Auger Borings
- 2.2 ASTM D1586 Test Method for Penetration Test and Split-Barrel Sampling of Soils
- 2.3 ASTM D1587 Practice for Thin-Walled Tube Sampling of Fine-Grained Soils for Geotechnical Purposes
- 2.4 ASTM D2113 Practice for Rock Core Drilling and Sampling of Rock for Site Exploration
- 2.5 ASTM D2573 Test Method for Field Vane Shear Test in Saturated Fine-Grained Soils
- 2.6 ASTM D4220 Practice for Preserving and Transporting Soil Samples
- 2.7 ASTM D5434 Standard Guide for Field Logging of Subsurface Explorations of Soil and Rock
- 2.8 ASTM D3441 Standard Test Method for Mechanical Cone Penetration Tests of Soil
- 2.9 GEOSPEC Newsletter CGS Volume 16, Number 4, Canadian Foundation Engineering Manual
- 2.10 TIR Traffic Control Manual

3.0 SERVICES AND EQUIPMENT

At the request of TIR the drilling company shall supply equipment and trained personnel to conduct subsurface investigations. At the request of TIR the drilling company shall provide any special services required for the boring operation including the construction of drilling platforms, arranging cranes or backhoes and constructing sedimentation control.

Adequate casing and drill rods for a drilling depth of 40 m shall be available on site. An estimate of the number of Shelby tubes to be made available on site may be obtained from TIR's Construction or Engineering sections.

Drilling equipment shall be capable of performing the required work in accordance with the latest edition of ASTM D1452 and shall be in good working condition. TIR reserves the right to demand the removal of unacceptable equipment from the work site and/or to demand that repairs be made to broken or damaged equipment when the rate of production is adversely affected or when the equipment presents, in the opinion of TIR's representative, a safety hazard.

The drilling equipment shall be capable of performing the required soil sampling by

following the latest edition of ASTM and AASHTO test methods.

3.1 Survey

TIR will furnish all necessary information regarding locations and elevations of drill holes and elevations of benchmarks in the vicinity of the work. TIR shall identify the location of the proposed drill holes.

Unless otherwise authorized by TIR, boreholes shall not be drilled in a location different from the designated location. All elevations shall be based on the given benchmark(s). When drilling over water which is subject to tidal variations, the drilling company will be required to install a "Tidal Board", which will show the water surface elevation at all times. Prior to each sampling and rock coring operation, a "Tidal Board" reading shall be taken to determine sample or rock core elevation.

4.0 SUBSURFACE & GEOTECHNICAL INVESTIGATIONS FOR HIGHWAYS AND STRUCTURES

4.1 Sampling

When drilling in granular material such as sands, gravels, tills or non-plastic silts, 50.8 mm outside diameter split barrel samples shall be taken at the surface, at 1.5 m intervals and at suspected changes in strata, with the Standard Penetration Test (SPT) "N" values recorded on the boring logs - as per the latest edition of ASTM Standard Method D1586. For Subsurface Investigations for Structures TIR may request continuous sampling on one or more holes.

When drilling in cohesive materials such as clay, plastic silt or organic-silt (if material contains a high clay content) samples shall be taken with 50.8 mm inside diameter or 76.2 mm outside diameter new, high quality, thin walled (Shelby) tubes according to the latest edition of ASTM Standard Method D1587. Tubes are to be taken at 1.5 m intervals or more frequently if requested by the Department or its representative. Sampling methods different than the ASTM Standard Methods shall be approved by TIR.

Insitu shear vane tests may be required between the tube locations - as per the latest edition of ASTM Standard Method D2573. The position of the casing and other modifications to the sampling regime may be necessary if this testing is to be accomplished.

When conditions in granular soils are such that SPT "N" values and samples cannot be obtained (e.g. - sand up casing), Dynamic Cone Penetration Tests shall be performed as a supplement to the SPT. The penetrometer shall consist of a 57.2 mm diameter recoverable drive point with a cone angle of 60 degrees. The cone penetrometer shall be advanced, following standard procedures, 3.0 m beyond the bottom of the casing or to a change in strata, and resistance readings recorded. The cone penetrometer shall then be withdrawn, the casing advanced 3.0 m or to a change in strata, and a SPT attempted. This procedure shall be repeated until split-barrel samples and "N" values can be obtained at 1.5 m intervals.

Sampling as described above shall be carried out over the full depth of soil strata in all boreholes unless otherwise directed. Reporting of sampling shall include all information required by each pertinent standard method.

The drilling company shall notify TIR as soon as possible of any unusual site features or subsurface conditions while the company is still on site.

Boreholes shall not be advanced by jetting.

Drilling fluid level within the boring or casing shall be maintained at or above the insitu ground water level at all times during drilling, removal of drill rods and sampling.

4.2 Groundwater Measurement

The ground water level shall be determined at each borehole. Artesian pressure information shall be recorded whenever encountered, and shall include the depths at which artesian pressures were encountered, the measured heads, and the time at which each measurement was made.

Requirements for observation wells or open standpipe piezometers shall be determined by TIR, in consultation with the drilling company. Piezometers, standpipes, sand, bentonite and associated supplies shall be provided by TIR or purchased at cost from the drilling company.

The upper end of the pipe shall, where practical, extend above the expected maximum piezometric elevation of the zone under study.

Observation wells shall be backfilled with permeable material and sealed at the surface to prevent entry of surface water.

Open standpipe piezometers shall be backfilled with permeable material over the perforated tip length, isolated with a 1 m bentonite seal and backfilled to the surface with a sealing grout.

The drilling company, unless otherwise instructed, shall be responsible to read and report on the water levels in the various instruments up to the time of demobilization. Subsequent monitoring shall be performed by TIR. The results of the ground water monitoring shall be incorporated into the drilling company's final logs.

4.3 Bedrock Coring

In borings for structures, bedrock is to be proven by coring for one and one-half $(1 \frac{1}{2})$ continuous metres of bedrock with percent recovery, and rock description shown on the logs unless otherwise instructed. Core size shall be between 35 mm and 50 mm in diameter.

In some circumstances, depending on the quality of the bedrock, the Department may order the coring of boreholes to depths greater than 1.5 m into bedrock. In cases such as this, no addition to the quoted rate will be paid by TIR. When drilling for foundations other than bridges, TIR may request rock coring to depths less than 1.5 m. No reimbursement will be considered for these changes.

The drilling company shall notify TIR, as soon as possible, of any unusual, soft or poor quality bedrock conditions encountered. The presence of mudstone, siltstone or low percentage of core recovery will also be reported immediately while the company is still on the site.

It is the responsibility of the drilling company to ensure that the drilling equipment on site is adequate to core the bedrock encountered and recover the core for TIR's inspection. Bedrock cores shall be delivered to TIR's Consultant or designated representative, unless otherwise instructed. All core runs shall be clearly indicated in the core box, and depths or elevations marked. Also, core boxes and their covers shall each be clearly identified with the project name, borehole identification, and core depth data, as per the latest edition of ASTM Standard Practice D2113.

Where used core trays are supplied, old identification, markings, etc., shall be completely eradicated prior to reuse.

5.0 AUGER DRILLING FOR OFF-HIGHWAY AND/OR ON-HIGHWAY PROJECTS

This work involves drilling along highway survey lines, usually located in wooded areas or along existing highway alignments. The work usually consists of auger drilling many shallow holes over a long corridor. The auger drill shall be mounted on a rubber tired or tracked vehicle. The drill shall be capable of auguring a hole of at least 130mm in diameter to a depth of 30m. The presence of bedrock shall be confirmed by coring for one and one-half (1 ¹/₂) continuous metres of bedrock with percent recovery shown on logs. In most instances soil sampling will not be required; the elevation of bedrock is the primary concern. On occasion, shear vane testing, split barrel and Shelby tube sampling may be requested by TPW.

6.0 SAMPLE HANDLING

All samples shall be properly labelled and identified with borehole and sample number, depth (or elevation) in metres and recovery indicated in millimetres.

Unless otherwise instructed, the Shelby tube samples are to be delivered to a place designated by TIR. The transportation and handling of tubes shall be such that sample disturbance will be minimized and in accordance with the latest edition of ASTM Standard Practice D4220 (Group D).

The drilling company shall be responsible for all transportation of samples from the borehole to TIR's Consultant or designated representative, unless otherwise instructed. The samples shall, at all times, be protected against freezing, mechanical damage and changes in moisture content.

After a visual classification of split-barrel and other disturbed samples, the drilling company shall deliver these to TIR's Consultant or designated representative, unless

otherwise instructed. Storage and handling shall be in accordance with ASTM Standard Practice D4220 (Group B).

TEST METHOD (TM-10) - THE ROUNDNESS, MOISTURE RESISTANCE AND CHEMICAL STABILITY OF GLASS OVERLAY-TYPE BEADS

1.0 SCOPE

1.1 This specification outlines methods for the roundness, moisture resistance and chemical stability of reflectorized overlay-type beads for use with traffic

2.0 RELEVANT DOCUMENTS

- 2.1 CGSB 1-GP-71 49.1 Methods of Testing Paints and Pigments Refractive Index
- 2.2 CGSB 1-GP-71 149.1 Imperfections
- 2.3 ASTM D1155 Test Method for Roundness of Glass Spheres
- 2.4 ASTM D1214 Test Method for Sieve Analysis of Glass Spheres

3.0 GENERAL REQUIREMENTS

The beads shall be true spheres and their surface shall be smooth, lustrous and free from cavities and scratches. The beads shall be manufactured from glass of a composition designed to be resistant to the effects of traffic wear and weathering. The beads shall be moisture proofed with a suitable moisture-proofing material so as to overcome the effects of water as a vapour or liquid on the beads before the beads are added to the paint stripe. No foreign material shall be contained in or among the beads.

4.0 TEST PROCEDURES

4.1 Roundness

A minimum of 75 % by mass of the glass beads shall be true spheres. The percentage true spheres shall be determined by one of the following method:

By counting the beads under 50 x and 100 x magnification as follows:

Glass beads larger than 300 µm sieve size shall be counted under 50x magnification (see gradation requirements)

Glass beads smaller than 300 μm sieve size shall be counted under 100 x magnification.

Approximately 1000 beads contained loosely in a culture dish shall be counted under reflected light for each sieve specified to determine the percentage by mass of perfectly round spheres.

4.2 Moisture Resistance

The beads shall not agglomerate during storage and application. They shall be treated in such a manner as to overcome the effect of water, both as a vapor and a liquid, on the beads before the beads are added to the paint stripe. They shall flow freely from the dispensing equipment at any time when surface and atmosphere conditions are satisfactory for painting.

Moisture resistance will be tested by the method described as follows:

A 100 g sample of glass beads will be placed in a 500 ml beaker and an equivalent volume of distilled water shall be added to the beaker. The beaker will then stand for five (5) minutes at the end of which time the water will be carefully poured off and the glass beads transferred to a clean, dry beaker and allowed to stand for five minutes. The beads will then be poured slowly into a standard 125 mm glass funnel having a stem of 125 mm length and 10 mm inside diameter.

The beads shall flow through the stem without stoppage. Slight initial agitation to start the flow through the funnel at the beginning of the test is permissible.

4.3 Chemical Stability

When the glass beads are exposed to atmospheric conditions, humidity, diluted acid or alkali solutions, or paint film constituents, there shall be no dulling of the surface which would adversely affect reflective properties of the beads.

Calcium chloride resistance shall be determined in the following manner:

Place 10 g of beads in a 100 ml glass beaker; Cover the sample with 500 ml of calcium chloride (1.0 Normal Solution); Soak the beads for three hours; Rinse the beads three times with 100 ml of distilled water and dry; Examine the beads under a microscope and compare them with an untreated sample.

Dulling of the surface or other detrimental effects shall constitute failure of this test.

TEST METHOD (TM-11) - CALCULATION OF FILM THICKNESS

1.0 SCOPE

1.1 This method outlines the procedure for calculating the asphalt film thickness around the aggregate particles in Hot Mix Asphalt Concrete.

2.0 SIGNIFICANCE AND USE

2.1 It is essential that an asphalt paving mixture have an adequate asphalt film thickness around the aggregate particles to reduce oxidation, fatigue cracking and raveling.

3.0 CALCULATION

3.1 Determine the **% Effective Asphalt** for the hot mix design. Effective asphalt content is equal to the Optimum Asphalt Content minus the asphalt absorbed by the aggregate.

Example: Optimum asphalt content of mix:	5.6%
Aggregate absorption*:	0.65%
Effective asphalt content:	4.95%

* Aggregate absorption requires the Effective Specific Gravity of Aggregate & Percent Asphalt absorption as calculated in MS-2, Asphalt Institute - Mix Design Methods.

3.2 Calculate the **Surface Area** (SA) of the aggregate gradation as follows:

SA = 2 + 0.02a + 0.04b + 0.08c + 0.14d + 0.30e + 0.60f + 1.60g

Where: a, b, c, d, e, f, and g are % of total aggregate passing sieve sizes of 5.0 mm, 2.5 mm, 1.25 mm, 630 μ m, 315 μ m, 160 μ m, 80 μ m.

Numbers: 2, 0.02, 0.04, etc. are constants. Example: Sieve Size % Passing 20 mm 100 14 mm 92 10 mm 80 5.0 mm 58 2.5 mm 43 1.25 mm 33 630 µm 24 315 µm 15 160 µm 10 80 µm

3.3 Calculate the **Bitumen Index (BI)** as follows:

 $BI = \frac{\% \text{ Effective Asphalt}}{100} \div \text{ Surface area}$

Example: BI = (4.95/100) / 32.58

BI = 0.0015

3.4 The **Asphalt Film Thickness (AFT)** is calculated by multiplying the Bitumen Index by 4870 (constant) and the result is shown in microns.

 $AFT = BI \times 4870$

Example: $AFT = 0.0015 \times 4870$

AFT = 7.3 microns

4.0 REPORT

The AFT result shall be reported on the Asphalt Concrete Mix Design Report and on the Quality Assurance Test Result form.

TEST METHOD (TM-12) - THE DETERMINATION OF INDIRECT TENSILE STRENGTH OF RECYCLED ASPHALT MIXES

1. SCOPE

1.1 This method covers procedures for indirect tensile strength testing of field samples of expanded and emulsified asphalt mixes.

2. **RELEVANT DOCUMENTS**

- 2.1 Ontario Ministry of Transportation, Test Method LS-297, Method of Test for the Determination of Indirect Tensile Strength of Expanded Asphalt Mixes
- 2.2 ASTM D2041 Standard Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixtures
- 2.3 ASTM D2216 Standard Test Methods for Laboratory Determination of Water (Moisture) Content of Soil and Rock by Mass (Method B)
- 2.4 ASTM D6926 Standard Test Method for Preparation of Bituminous Specimens using Marshall Apparatus
- 2.5 ASTM D6931 Standard Test Method for Indirect Tensile (IDT) Strength of Bituminous Mixtures
- 2.6 ASTM D1557 Standard Test Methods for Laboratory Compaction Characteristics of Soil Using Modified Effort
- 2.7 Wirtgen Cold Recycling Manual (Latest Edition)

3. DEFINITIONS AND ACRONYMS

- 3.1 OMC: Optimum Moisture Content
- 3.2 Constant Mass: The mass of the sample when there is no change in mass in excess of 0.1% for successive 30 minute drying periods.
- 3.3 Prepared Mix: Material that passes the 20 mm sieve that has a moisture content equal to 80% of W_{omc} .
- 3.4 W_{labsample}: The percentage of moisture in the sample in the lab as determine prior to moisture conditioning, by mass.
- 3.5 W_{omc}: Optimum moisture content determined during the mix design process in accordance with the Modified Proctor Test (ASTM D1557).

4. APPARATUS

- 4.1 Oven: of suitable capacity and capable of maintaining $110^{\circ}C \pm 5^{\circ}C$ for drying samples.
- 4.2 Marshall molds, compaction hammer, compaction pedestal, circular mold discs,

weigh scale, spatula, metal trough, extrusion jack.

- 4.3 Circular mold discs should be nonabsorbent, plastic discs 4 mil in thickness cut to mold diameter size.
- 4.4 Sieve: 20 mm opening sieve, meeting ASTM E11 specifications with a minimum surface area equivalent to a sieve with a 305 mm diameter frame.
- 4.5 Containers: as required for mixing and splitting samples.
- 4.6 Thermometer, or temperature probe: capable of measuring specified temperatures with a precision of 0.2° C.
- 4.7 Forced draft oven: capable of maintaining $40^{\circ}C \pm 1^{\circ}C$ for curing the specimens.
- 4.8 Temperature control system: an air cabinet, air bath or water bath capable of maintaining a temperature of $25^{\circ}C \pm 1^{\circ}C$.
- 4.9 Compression loading apparatus and breaking head: able to uniformly load the specimen with a constant deformation rate of 50.8 mm/minute.
- 4.10 Loading strips: as per ASTM D6931

5. PROCEDURE FOR PREPARING LAB SAMPLE

- 5.1 Manually screen the entire sample. Particles retained on the 20 mm sieve that are stuck together may be separated by hand. Oversize material shall not be crushed. Discard material retained on the 20 mm sieve.
- 5.2 Determine W_{labsample} by following the procedure outlined in ASTM D2216, with the following exceptions: use a 1000 g representative portion, and record the mass, m to the nearest 0.1g.
- 5.3 When W_{labsample} is greater than or equal to 80% of W_{omc}, proceed to step 6 and immediately prepare the briquettes.
- 5.4 When W_{labsample} is less than 80% of W_{omc}, determine the mass of water, M_{water} that needs to be added to the remaining screened material (for manufacturing briquettes as in Step 6) as follows:
- 5.4.1 Weigh the screened material that passed the 20 mm sieve to determine its mass, M_{labsample} in g, in a tared flat pan.
- 5.4.2 Moisture condition the screened material, from W_{labsample} to 0.8W_{omc} by adding M_{water} of water as calculated by the following equation:

$$M_{water} = (0.8W_{omc} - W_{labsample}) \times M_{labsample}$$

The mass of water (M_{water}) can be added by spraying the sample with a spray bottle filled with distilled water and mixing the moisture into the screened material. The prepared mix shall be compacted into briquettes as soon as 80% of the optimum moisture content, 0.8W_{omc} is achieved. Proceed to Step 6.

6. **PROCEDURE FOR THE COMPACTION OF SPECIMENS**

- 6.1 Prepare the Marshall mold and hammer by cleaning the mold, collar, base-plate, and face of the compaction hammer. Use 100 mm (4") Marshall molds for the briquettes.
- 6.2 Reduce the prepared mix using the splitting/quartering method to get sufficient material (approximately 6,600 g) for preparing 6 Marshall briquettes. Note that, to achieve a compacted height of 63.5 ± 2.5 mm, usually 1,100 g is sufficient for each briquette. The 6 briquettes will be divided into 2 sets of 3 briquettes each. One set of 3 briquettes will be tested as dry specimens and the other set of 3 briquettes will be tested as wet specimens.
- 6.3 Place a circular mold disc at the bottom of the Marshall mold.
- 6.4 Using the metal trowel for removing the prepared mix from the trough, place approximately 1,100 g into each of the tared molds. Make certain that the bottom of the trowel is in contact with the bottom of the trough so that the prepared mix is not removed in layers. (Try 1 briquette at 1,100g and go from there to determine approximate mass required).
- 6.5 Using the spatula, rod the prepared mix in the mold 25 times 15 around the outside, 10 around the centre. When rodding around the outside, keep the spatula blade flat against the inside the mold. When rodding around the inside, do not change direction of the blade, and finish by leaving a small cone of prepared mix in the centre of the mold.
- 6.6 Place another circular mold disc at the top of the Marshall mold.
- 6.7 The briquettes shall be compacted at room temperature. Each briquette shall receive 75 blows per side. Make sure the hammer is kept as vertical as possible and ensure free fall of the hammer during compaction.

7. PROCEDURE FOR CURING THE SPECIMENS

- 7.1 After compaction, remove the top and bottom circular mold discs and collar of the mold, place the mold with the sample face down on a smooth flat tray and allow the specimen to cure for 24 hours in the mold in a force draft oven at 40°C. Briquettes may be removed from molds if compacted material has sufficient cohesion to remain intact. Do not stack the briquettes.
- 7.2 Allow the molds to cool until warm to the touch, and carefully extrude the briquettes by means of an extrusion jack avoiding distortion of the compacted

specimens.

- 7.3 Place the samples on their faces on a smooth flat tray and cure in a forced draft oven for a further 48 ± 4 hours at 40°C. Do not stack the briquettes.
- 7.4 Remove the briquettes from the oven, and immediately determine each briquette mass (g) and record the mass in air.
- 7.5 Allow the briquettes to cool in air at room temperature for a minimum of 4 hours and a maximum of 20 hours. The use of fans or other methods of accelerating cooling of specimens **is not permitted**. When cooled, the diameter, d (mm) and the height, h (mm) around the circumference at 4 evenly spaced places shall be measured for each briquette to zero decimal places and the average height (h_{ave}) calculated. Use these measurements to calculate the volume of each briquette with the following formula:

 $V = \pi r^2 h_{ave} / 1000$

Where V = volume in cm^3 r = radius of specimen in mm to one decimal place (r = d/2) h_{ave} = average height of specimen in mm to one decimal place

8. DETERMINATION OF SPECIMEN DENSITY

8.1 Calculate the density of the compacted specimens using the following formula:

D=M/V

Where:	$D = density in g/cm^3$
	M = Mass of briquette in g
	$V = Volume of briquette in cm^3$

- 8.2 If the density of any one briquette is more than ± 0.025 g/cm³ from the average density of all the specimens, recheck the calculations and, if necessary, re-weigh. Exclude specimens from further testing which significantly differ.
- 8.3 Group the 6 briquettes into two (2) sets of three (3) so that the average density of each set is approximately equal.

9. DETERMINATION OF INDIRECT TENSILE STRENGTH (ITS)

- 9.1 The standard ITS test is used to test the briquettes under both dry and wet conditions. The ITS is determined by measuring the ultimate load to failure of a specimen which is subjected to a constant deformation rate of 50.8 mm/minute on its diametrical axis according to ASTM D6931.
- 9.2 The dry specimen procedure is as follows:

- 9.2.1 Bring the specimens to the test temperature of 25° C by placing the briquettes in a dry temperature control system at 25° C $\pm 1^{\circ}$ C for at least 1 hour, but not for longer than 2 hours before testing.
- 9.2.2 Remove a specimen from the air cabinet and place it into the loading apparatus.
- 9.2.3 Centre the briquette on the edge of the lower loading strip. Position the upper loading strip. Take note of the alignment marks on the loading apparatus and position accordingly.
- 9.2.4 Position the assembly centrally under the loading ram of the compression testing device.
- 9.2.5 Apply the load to the specimen, without shock, at a rate of advance of 50.8 mm / minute until the maximum load is reached.
- 9.2.6 Record this maximum load, P (in N), accurate to zero decimal place.
- 9.3 The wet specimen procedure is as follows:
- 9.3.1 Condition the 3 briquettes under a minimum of 25 mm of distilled water cover for 24 hours at $25^{\circ}C \pm 1^{\circ}C$ for 24 hours. Alternatively, the 3 briquettes can be covered with water at $25^{\circ}C \pm 1^{\circ}C$ in a vacuum according to ASTM D2041, with the exception that the vacuum pressure is only increased until the residual pressure manometer reads 50 mm mercury and then once the vacuum is achieved, continue the vacuum and agitation for 60 ± 1 minutes.
- 9.3.2 Remove the specimen and surface dry.
- 9.3.3 Test for the ultimate tensile load, as described above in steps 9.2.3 through 9.2.6.
- 9.4 Calculate the ITS for each specimen to the nearest 1 kPa using the following formula:

	$ITS = 2000 \text{ x P} / \pi \text{ x } h_{\text{ave}} \text{ x } d$
Where:	ITS = Indirect Tensile Strength in kPa
	P = maximum applied load in N
	h_{ave} = average height of the specimen in mm to one decimal place
	d = diameter of the specimen in mm to one decimal place

9.4.1 Calculate the tensile strength ratio (TSR) for each set of specimens using the following formula:

$$TSR = ITS_{wet} / ITS_{dry} \times 100\%$$

Where: ITS_{wet} = average ITS of all wet specimens in the set ITS_{dry} = average ITS of all dry specimens in the set

10. **REPORTING**

Report the following measurements in the report, as well as any observations:

- 10.1 W_{labsample} and W_{omc} of each specimen to the nearest 0.1%.
- 10.2 Density of each specimen.
- 10.3 ITS of each specimen.
- 10.4 TSR value.

TEST METHOD (TM-13) - PROCEDURE FOR THE DETERMINATION OF RECLAIMED ASPHALT PAVEMENT SPECIFIC GRAVITY

1.0 SCOPE

1.2 This method covers the procedure for determination of the bulk specific gravity of Reclaimed Asphalt Pavement (RAP).

2.0 RELEVANT DOCUMENTS

- 2.1 ASTM C127, Standard Test Method for Specific Gravity and Absorption of Coarse Aggregate
- 2.2 ASTM C128, Standard Test Method for Specific Gravity and Absorption of Fine Aggregate
- 2.3 ASTM D75, Standard Practices for Sampling Aggregates
- 2.4 ASTM D2041, Standard Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixes
- 2.5 ASTM D2172, Standard Test Method for Quantitative Extraction of Bitumen from Bituminous Paving Mixtures Test Method A
- 2.6 ASTM E11, Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves
- 2.7 NAPA Quality Improvement Series 124 (QIP-124), Designing HMA Mixtures with High RAP Content
- 2.8 Asphalt Institute Manual Series 2 (MS-2)

3.0 APPARATUS

- 3.1 SIEVES: All sieves shall conform to ASTM E11 specifications.
- 3.2 ASPHALT EXTRACTION APPARATUS: All apparatus used for quantitative determination of bitumen content as described in ASTM D2172 Test Method A.
- 3.3 OVENS: Mechanical ovens, convection ovens or forced draft ovens shall be required for drying aggregates and HMA mixtures, and for preheating HMA mixtures prior to ignition testing.
- 3.4 SPECIFIC GRAVITY APPARATUS: All apparatus used to determine the specific gravity of aggregate as described in ASTM D2041.
- 3.5 BALANCE: A balance or scale accurate to 0.1 g.

4.0 TEST PROCEDURE

- 4.1 The bulk specific gravity of RAP shall be determined following NAPA Publication QIP-124. This method requires determination of:
 - Theoretical Maximum Density (G_{mm}) as per ASTM D2041.
 - Asphalt Binder Content (P_b) as per ASTM D2172 Test Method A, including determination of mineral matter.
 - Asphalt Binder Specific Gravity (G_b) Assume 1.020.
 - Asphalt Absorption (P_{ba}) Assume 0.7% unless RAP source is known to have alternate properties.
- 4.2 The coarse and fine aggregate specific gravity shall be determined following ASTM C127 and ASTM C128 respectively.

5.0 CALCULATION

5.1 The bulk specific gravity of RAP shall be determined as follows:

$$G_{se} = \frac{(100 - P_b)}{\left[\left(\frac{100}{G_{mm}}\right) - \left(\frac{P_b}{G_b}\right)\right]}$$

$$G_{sb} = G_{se} / \left[(P_{ba} \ x \frac{G_{se}}{100xG_b}) + 1 \right]$$

Where:

 $G_{mm} = \text{Theoretical Maximum Specific Gravity of RAP}$ $G_b = \text{Specific Gravity of RAP Asphalt Binder}$ $P_b = \text{RAP Asphalt Binder Content}$ $G_{se} = \text{Effective Specific Gravity of Aggregate}$ $G_{sb} = \text{Bulk Specific Gravity of Aggregate}$ $P_{ba} = \text{Absorbed Asphalt Binder, \% weight of Aggregate}$

5.2 The combined aggregate specific gravity shall be determined following the Asphalt Institute Manual Series (MS-2):

$$G_{sb} = \frac{(P_1 + P_2 + \dots + P_n)}{(\frac{P_1}{G_1} + \frac{P_2}{G_2} + \dots + \frac{P_n}{G_n})}$$

Where:

 G_{sb} = Bulk Specific Gravity of the Aggregate $G_1, G_2, ..., G_n$ = Bulk Specific Gravity of Each Aggregate in Blend $P_1, P_2, ..., P_n$ = Percentage (by mass) of Each Aggregate in Blend

6.0 **REPORT**

The report shall include the following:

- 6.1 NSTIR Project Number.
- 6.2 Date of materials sampled and received.
- 6.3 Report date.
- 6.4 Identification and proportions (%) of RAP and virgin aggregate.
- 6.5 The bulk specific gravity of the RAP reported to three decimal places.
- 6.6 The combined virgin aggregate bulk specific gravity reported to three decimal places.

TEST METHOD (TM-14) – PROCEDURE FOR IGNITION OVEN CALIBRATION

1.0 SCOPE

1.1 This method covers the procedure for calibrating hot-mix asphalt ignition ovens.

2.0 RELEVANT DOCUMENTS

- 2.1 ASTM C136, Standard Method for Sieve Analysis of Fine and Coarse Aggregate
- 2.2 ASTM D75 Standard Practice for Sampling Aggregates
- 2.3 ASTM D140, Standard Practice for Sampling Bituminous Materials
- 2.4 ASTM D2172, Standard Test Method for Quantitative Extraction of Bitumen from Bituminous Paving Mixtures Test Method A
- 2.5 ASTM D5444, Standard Test Method for Mechanical Size Analysis of Extracted Aggregate
- 2.6 ASTM D6307, Standard Test Method for Asphalt Content of Hot-Mix Asphalt by Ignition Method Test Method A
- 2.7 ASTM E11, Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves

3.0 APPARATUS

- 3.1 SIEVES: All sieves shall conform to ASTM E11 specifications.
- 3.2 ASPHALT EXTRACTION APPARATUS: All apparatus used for quantitative determination of bitumen content as described in ASTM D2172 Test Method A.
- 3.3 OVENS: Mechanical ovens, convection ovens or forced draft ovens shall be required for drying aggregates and HMA mixtures, and for preheating HMA mixtures prior to ignition testing.
- 3.4 IGNITION FURNACE: A forced air ignition furnace that heats the HMA sample either by convection method or direct-irradiation method as described in ASTM D6307 Test Method A.
- 3.5 BALANCE: A balance or scale accurate to 0.1 g.

4.0 TEST PROCEDURE

- 4.1 All asphalt binder contents shall be determined and reported based a percentage of total mix weight.
- 4.2 RAP asphalt binder content shall be determined on a representative sample of the supplied calibration material following ASTM D2172 Test Method A, including determination of mineral matter.
- 4.3 RAP shall be air dried, and screened into appropriate size fractions.

- 4.4 Virgin aggregates shall be oven dried to a constant mass, cooled and screened into appropriate size fractions.
- 4.5 Virgin aggregates and RAP shall be combined as per mix design proportions accounting for any remaining moisture in the RAP for each test sample separately.
- 4.6 A target dry weight of aggregate and RAP of 2,000, 1,500 and 1,000 grams shall be used for BHF, C-HF and D-HF mixture designations respectively. This will ensure that mixtures reasonably exceed the minimum sample testing size requirements after the addition of asphalt binder.
- 4.7 Blended aggregate and RAP shall be heated to a temperature not to exceed 28°C greater than the mixing temperature required for the PGAB.
- 4.8 Virgin asphalt binder and any modifiers shall be added in the proportions stated in the mix design.
- 4.9 The mixture will be cured at the compaction temperature for two (2) hours.
- 4.10 A minimum of four (4) calibration samples shall be prepared. The first mixture shall be used as a "butter" mix to prepare bowls and utensils for the remaining three (3) calibration samples to be tested according to ASTM D6307 Test Method A. The actual total Asphalt Binder content shall be calculated as follows:

[Mass of RAP(g) x RAP Asphalt Content(%) + Mass of Virgin Asphalt] [Mass of Total Mixture(g)]

NOTE: Depending on the RAP asphalt binder content determined in the calibration sample, the total asphalt binder content may not equate to the mix design total asphalt binder content.

4.11 The correction factor shall be determined as follows:

[Average Oven Recorded Asphalt Content (%)] - [Actual Total Asphalt Content (%)]

- 4.12 If the correction factor exceeds 1.0%, then additional calibration samples shall be prepared, and the procedure repeated at a lower ignition oven temperature as per ASTM D6307, Section 9.15.
- 4.13 The correction factor shall be applied to subsequent oven-recorded asphalt contents for all production samples to determine a corrected asphalt binder content.
- 4.14 Only corrected asphalt binder contents results shall be reported for production samples.
- 4.15 A sieve analysis shall be performed on a minimum of three (3) calibration samples to assess the potential for aggregate breakdown. NSTIR Test Method TM-15 shall be followed for determining if aggregate gradation correction factors are required.

The quality control consultant shall identify sources suspected of aggregate breakdown prior to production.

4.16 Correction factors shall be determined each time an ignition oven is re-located, there is a change in the PGAB type, there is a change in the aggregate source, or there is a significant change in the blend proportions, in particular, the ratio of RAP to virgin aggregate contents.

5.0 REPORT

The report shall include the following:

- 5.1 NSTIR Project Number.
- 5.2 Date of materials sampled and received.
- 5.3 Report date.
- 5.4 Identification of aggregate, RAP materials, and mix type.
- 5.5 Test number.
- 5.6 Calibration data.
- 5.7 The mass of HMA sample before and after ignition reported to the nearest 0.1 gram.
- 5.8 The measured asphalt content reported to the nearest 0.01 percent.
- 5.9 The calculated gradation analysis on the residual aggregate reported to the nearest 0.1 percent.

TEST METHOD (TM-15) – PROCEDURE FOR IGNITION OVEN CORRECTION FACTOR FOR DETERMINATION OF AGGREGATE BREAKDOWN

1.0 SCOPE

- 1.1 This method covers the ignition oven correction factor procedure for determination of aggregate breakdown.
- 1.2 Asphalt binder content results may be affected by the type of aggregate in the mixture and the ignition furnace. Therefore, asphalt binder and aggregate correction factors must be established by testing a set of correction specimens for each job mix formula (JMF) mix design. Correction factor(s) must be determined before any acceptance testing is completed and repeated each time a change in the mix ingredients or design occurs. Any changes greater than 5 percent in stockpiled aggregate proportions should require a new correction factor.
- 1.3 Asphalt binder correction factor: Certain aggregate types may result in unusually high correction factors (greater than 1.0 percent). Such mixes should be corrected and tested at a lower temperature, as described below. Each ignition furnace will have its own unique asphalt binder correction factor determined in the location where testing will be performed.
- 1.4 Aggregate correction factor: Due to potential aggregate breakdown during the ignition process, an aggregate correction factor will be determined for each ignition furnace in the location where testing will be performed when the following conditions occur: a. Aggregates that have a proven history of excessive breakdown or b. Aggregates from an unknown source.

2.0 RELEVANT DOCUMENTS

- 2.1 ASTM C136, Standard Method for Sieve Analysis of Fine and Coarse Aggregate
- 2.2 ASTM D75 Standard Practice for Sampling Aggregates
- 2.3 ASTM D140, Standard Practice for Sampling Bituminous Materials
- 2.4 ASTM D6307, Standard Test Method for Asphalt Content of Hot-Mix Asphalt by Ignition Method Test Method A
- 2.5 ASTM D5444, Standard Test Method for Mechanical Size Analysis of Extracted Aggregate
- 2.6 ASTM E11, Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves

3.0 APPARATUS

- 3.1 SIEVES: All sieves shall conform to ASTM E11 specifications.
- 3.2 OVENS: Mechanical ovens, convection ovens or forced draft ovens shall be required for drying aggregates and HMA mixtures, and for preheating HMA mixtures prior to ignition testing.
- 3.3 IGNITION FURNACE: A forced air ignition furnace that heats the HMA sample either by convection method or direct-irradiation method as described in ASTM D6307.
- 3.4 BALANCE: A balance or scale accurate to 0.1 g.

4.0 TEST PROCEDURE

- 4.1 Obtain samples of aggregate in accordance with ATSM D75.
- 4.2 Obtain samples of asphalt binder in accordance with ASTM D140.
- 4.3 Prepare an initial, or "butter mix" at the design asphalt binder content. Mix and discard the butter mix prior to preparing any of the correction specimens to ensure an accurate asphalt binder content.
- 4.4 Prepare two correction specimens at the JMF design asphalt binder content and gradation. Aggregate used for correction specimens shall be sampled from the material designated for use in production. An additional "blank" (aggregate only) specimen shall be batched at the JMF gradation. Determine an aggregate gradation in accordance with ASTM D5444 on the "blank" specimen.
- 4.5 Place the freshly mixed specimens directly into the specimen basket assembly. If specimens are allowed to cool prior to placement in the specimen basket assembly, the specimens must be dried to constant mass at a temperature of 110±5°C. Do not preheat the specimen basket assembly.
- 4.6 Test the specimens in accordance with ASTM D6307 Method A.
- 4.7 Once both of the correction specimens have been burned, determine the asphalt binder content for each specimen by calculation or from the printed oven tickets, if available.
- 4.8 If the difference between the asphalt binder contents of the two specimens exceeds 0.15 percent, repeat Section 4.3 to 4.7 with two more specimens and, from the four results, discard the high and low result. Determine the correction factor from the two original or remaining results as appropriate. Calculate the difference between the actual and measured asphalt binder contents for each specimen. The asphalt binder correction factor, CF, is the average of the differences expressed as a percentage by mass of the HMA.

4.9 If the asphalt binder correction factor exceeds 1.0 percent, the test temperature should be lowered to 482±5°C for convection type furnace. If there is no improvement in the correction factor, it is permissible to use the higher temperature.

Note: The temperature for determining the asphalt binder content of HMA specimens by this procedure shall be the same temperature determined for the correction specimens.

- 4.10 For the direct IR irradiation-type furnaces, the DEFAULT burn profile should be used for most materials. The operator may select burn-profile OPTION 1 or OPTION 2 to optimize the burn cycle. Option 1 is designed for aggregate that require a large aggregate correction factor (greater than 1 percent) typically very soft aggregate (such as dolomite). Option 2 is designed for samples that may not burn completely using the DEFAULT burn profile. The burn profile for testing HMA samples shall be the same burn profile selected for correction samples.
- 4.11 Perform a gradation analysis on the residual aggregate in accordance with ASTM D5444. The results will be utilized in developing an aggregate correction factor and should be calculated and reported to the nearest 0.1 percent.
- 4.12 From the gradation results, subtract the percent passing for each sieve for each specimen from the percent passing each sieve of the "blank" specimen gradation results from Section 4.4.
- 4.13 Determine the average difference for the two values. If the difference for any single sieve exceeds the allowable difference for that sieve as listed in Table 1, then aggregate gradation correction factors (equal to the resultant average differences) for all sieves shall be applied to all acceptance gradation test results determined by ASTM D5444, prior to final rounding and reporting. If the 75 μ m sieve is the only sieve outside the limits in Table 1, apply the aggregate correction factor to only the 75 μ m sieve.

Sieve (mm)	Allowable Difference (%)
\geq 2.36 mm	± 5.0
$>75 \ \mu m$ and $< 2.36 \ mm$	± 3.0
\leq 75 μ m	± 5.0

Table 1: Permitted Sieving Difference

5.0 REPORT

The report shall include the following:

- 5.1 NSTIR Project Number.
- 5.2 Date of materials sampled and received.
- 5.3 Date of report.
- 5.4 Identification of aggregate and mix type.
- 5.5 Test number.
- 5.6 Calibration data.
- 5.7 The mass of HMA sample before and after ignition reported to the nearest 0.1 gram.
- 5.8 The asphalt binder correction factor, CF, expressed as a percentage by mass of the HMA.
- 5.9 The calculated gradation analysis on the "blank" aggregate reported to the nearest 0.1 percent.
- 5.10 The calculated gradation analysis on the residual aggregate reported to the nearest 0.1 percent.
- 5.11 The calculated average differences between the percent passing each sieve of the "blank" specimen gradation results from Section 4.4 and the percent passing of the residual aggregate reported to the nearest 0.1 percent.

TEST METHOD (TM-16) – PROCEDURE FOR GYRATORY COMPACTOR CALIBRATION

1.0 SCOPE

1.1 This method covers the procedure for performing Superpave calibration trials on prepared hot-mix asphalt (HMA) specimens using the gyratory compactor.

2.0 RELEVANT DOCUMENTS

- 2.1 ASTM C127, Standard Test Method for Specific Gravity and Absorption of Coarse Aggregate
- 2.2 ASTM C128, Standard Test Method for Specific Gravity and Absorption of Fine Aggregate
- 2.3 ASTM C136, Standard Method for Sieve Analysis of Fine and Coarse Aggregate
- 2.4 ASTM D75, Standard Practices for Sampling Aggregates
- 2.5 ASTM D140, Standard Practice for Sampling Bituminous Materials
- 2.6 ASTM D2041, Standard Test Method for Theoretical Maximum Specific Gravity and Density of Bituminous Paving Mixes
- 2.7 ASTM D2172, Standard Test Method for Quantitative Extraction of Bitumen from Bituminous Paving Mixtures - Test Method A
- 2.8 ASTM D2726, Standard Test Method for Bulk Specific Gravity of Compacted Bituminous Mixes Using Saturated Surface-Dry Specimens
- 2.9 ASTM D5444, Standard Test Method for Mechanical Size Analysis of Extracted Aggregate
- 2.10 ASTM E11, Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves
- 2.11 AASHTO T312, Standard Method of Test for Preparing and Determining the Density of Hot-Mix Asphalt (HMA) Specimens by Means of Superpave Gyratory Compactor
- 2.12 NSTIR TM-13, Procedure for Determination of RAP Specific Gravity
- 2.13 Asphalt Institute Manual Series 2 (MS-2)

3.0 APPARATUS

- 3.1 SIEVES: All sieves shall conform to ASTM E11 specifications.
- 3.2 ASPHALT EXTRACTION APPARATUS: All apparatus used for quantitative determination of bitumen content as described in ASTM D2172 Test Method A.
- 3.3 SPECIFIC GRAVITY APPARATUS: All apparatus used to determine the specific gravity of aggregates and bituminous paving mixes as described in ASTM C127, C128, D2041, and D2726.
- 3.4 OVENS: Mechanical ovens, convection ovens or forced draft ovens shall be required for drying virgin aggregates, RAP, and HMA mixtures.

- 3.5 SUPERPAVE GYRATORY COMPACTOR: An electrohydraulic or electromechanical compactor and all apparatus used for determining the density of hot mix asphalt specimens as described in AASHTO T312.
- 3.6 BALANCE: A balance or scale accurate to 0.1 g.

4.0 TEST PROCEDURE

Prepare and compact two HMA specimens utilizing calibration materials following the mix design proportions as follows:

- 4.1 RAP asphalt binder content shall be determined on a representative sample of the supplied calibration material following ASTM D2172 Test Method A, including determination of mineral matter.
- 4.2 RAP material shall be air dried, and screened into appropriate size fractions.
- 4.3 RAP effective specific gravity and bulk specific gravity shall be determined on a representative sample following NSTIR TM-13.
- 4.4 Virgin aggregates shall be oven dried to a constant mass, cooled and screened into appropriate size fractions.
- 4.5 Coarse and fine virgin aggregate specific gravity and absorption shall be determined on a representative sample following ASTM C127 and C128.
- 4.6 Virgin aggregates and RAP shall be combined as per mix design proportions accounting for any remaining moisture in the RAP for each test sample separately.
- 4.7 Blended virgin aggregate and RAP shall be heated to a temperature not to exceed 28°C greater than the mixing temperature required for the PGAB.
- 4.8 Virgin PGAB and any modifiers shall be added in the proportions stated in the mix design. The mixture shall be cured at the compaction temperature for two hours.
- 4.9 The HMA specimens shall be compacted following AASHTO T312.
- 4.10 Bulk specific gravity testing of the compacted specimens shall be conducted following ASTM D2726.

5.0 REPORT

A quality assurance calibration report will be provided to NSTIR staff (Project Engineer, Highway Construction Services) and will be forwarded to the contractor by the Project Engineer.

The calibration report shall include the following:

- 5.1 NSTIR Project Number.
- 5.2 Date of materials sampled and received.
- 5.3 Report date.
- 5.4 Identification and proportions (%) of RAP, virgin aggregate, asphalt mix type, PGAB type and source, asphalt modifiers (anti-strip, etc.).
- 5.5 The RAP asphalt content (%) reported to two decimal places.
- 5.6 The RAP maximum theoretical specific gravity (G_{mm}) reported to three decimal places.
- 5.7 The RAP effective specific gravity (G_{se}) reported to three decimal places.
- 5.8 The virgin aggregate and RAP bulk specific gravity(G_{sb}) reported to three decimal places.
- 5.9 The virgin aggregate and RAP absorption reported to two decimal places.
- 5.10 The number of gyrations (N_{des}) of the HMA specimens.
- 5.11 The bulk specific gravity at N_{des} of the HMA specimens reported to three decimal places.
- 5.12 The bulk density (kg/m^3) of the HMA specimens.
- 5.13 The HMA specimens average mass (grams) reported to one decimal place.
- 5.14 The compacted HMA specimen average height (mm) reported to one decimal place.