

PROCEDURE FOR THE PETROGRAPHIC ANALYSIS OF FINE AGGREGATE

1. SCOPE

This procedure outlines the method to be employed in the petrographic analysis of fine aggregate. Unlike the petrographic analysis of coarse aggregate, this method does not provide a petrographic number. The procedure appraises the quality of the fine aggregate. The test method is subdivided into Parts A and B. In Part A, the method firstly determines amounts of silicate and carbonate rock types. Secondly, the amount of deleterious material including, for example, shale, mica, and chert is recorded. The latter is required in order to assess the potential for problems such as lack of freeze-thaw durability and alkali-aggregate reaction. Part B outlines the procedure for petrographic identification of fine aggregate derived from reclaimed asphalt pavement (RAP) or reclaimed shingle tab (RST).

2. RELEVANT DOCUMENTS

- 2.1 MTO LS-282 Method of Test for Quantitative Extraction of Asphalt Cement and Analysis of Extracted Aggregate From Bituminous Paving Mixtures
- 2.2 MTO LS-291 Method of Test for Quantitative Extraction of Asphalt Cement and Mechanical Analysis of Extracted Aggregate From Bituminous Paving Mixtures - Ontario Procedure
- 2.3 MTO LS-602 Method of Test for Sieve Analysis of Aggregates
- 2.4 MTO LS-609 Procedure for the Petrographic Analysis of Coarse Aggregate
- 2.5 MTO Report EM-91 Petrographic Examination of Aggregate and Concrete in Ontario, Engineering Materials Office Report, Ministry of Transportation (1989)
- 2.6 ASTM C294 Standard Descriptive Nomenclature for Constituents of Concrete Aggregates
- 2.7 ASTM C295 Standard Guide for Petrographic Examination of Aggregates for Concrete
- 2.8 ASTM C670 Standard Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials for Preparing Precision Statements for Test Methods for Construction Materials

3. APPARATUS

- 3.1 Stereoscopic microscope, with crosshairs in at least one eyepiece, final magnification of not less than 50x.
- 3.2 A source of illumination.
- 3.3 Pre-cleaned glass microscope slides, 50 x 75 mm.
- 3.4 Epoxy, hardener, and resin (5-minute setting).
- 3.5 Mixing surface, disposable, e.g. cardboard.
- 3.6 Mixing stick, disposable, e.g. coffee stirrer.

- 3.7 Mechanical stage, i.e. electrically triggered specimen carriage (Notes 1 & 2).
- 3.8 Automatic point counter with at least 10 separate channels.
- 3.9 Pointer, metal, to scratch particles.
- 3.10 Hydrochloric acid (5 percent).

Note 1: In MTO laboratories, point counting is undertaken using a Swift automatic point counter. In the event that such equipment is not available, it is permissible to use non-automatic equipment such as a mechanical stage and a mechanical means of counting provided that particles selected for examination are done so on a random basis.

Note 2: The Swift mechanical stage, if used, should be equipped with a 1/3 mm stepping gear rather than the 1/6 mm gear usually supplied.

METHOD A

4. SAMPLE PREPARATION

- 4.1 Dry the sample to a constant mass at a temperature of $110 \pm 5^{\circ}\text{C}$, and then cool to room temperature.
- 4.2 The fine aggregate portion (pass 4.75 mm material) of the sample should be reduced in mass by splitting to approximately 250 to 300 g.
- 4.3 Place test sample in the uppermost of a progressive series of sieves including the 2.36 mm, 1.18 mm, 600 μm , 300 μm , 150 μm , and 75 μm sizes. Sieving should be undertaken according to LS-602. Place each size in a separate bag and label.
- 4.4 In order to accommodate a glass slide in the point counter, it is necessary to leave a 5 mm wide sand free border around the perimeter of the slide. This can be achieved by overlapping masking tape a width of about 5 mm around the edge of the slide prior to applying resin. In preparation for sticking sand grains to glass slides, mix about 1 ml of 5 minute epoxy resin with 1 ml of epoxy hardener on a disposable surface, not on the glass slide. This is normally sufficient for the preparation of 2 slides. Apply resin evenly over 2 glass slides (Note 3). Do not attempt to do more than this number at one time. Using a teaspoon, dig deeply into the sand sample and spread the sand grains evenly over the slide.

Note 3: A 27 x 45 mm glass microscope slide, on edge, can be used to spread an even film of epoxy resin on the 50 x 75 mm slide.

- 4.5 In the case of the large size fractions (i.e. retained 2.36 and 1.18 mm), grains should be pressed into the resin to obtain a better bond. This can be achieved by applying pressure to another glass slide placed on top of the grains.

After 3 or 4 minutes, turn slide upside down in order to remove loose grains. Remove the masking tape. Repeat the procedure to prepare the remainder of the slides.

In the event that there are less than 200 particles in an individual sieve fraction, a specimen should nevertheless be prepared on a glass slide. All particles should be observed and classified. A note should be made on the report form of the total number of particles examined.

Note 4: In order to examine at least 200 particles of the retained 2.36 mm fraction, it is necessary to prepare 2 slides.

Note 5: The thickness of the epoxy resin on slides should decrease with grain size, so that small grains are not totally submerged in epoxy, which would make identification more difficult.

5. SETTING UP APPARATUS

5.1 Attach the mechanical stage to the base of the microscope and plug the automatic point counter into the stage.

5.2 Zero the vernier gauge on the mechanical stage and insert the glass sample slide into the stage.

5.3 Enter a 'sampling target' of 200 particles in the point counter. Set the 'stage interval' at #6 (2 mm) for all size fractions. Where the majority of particles retained on the 2.36 mm sieve are on the large size, the 'stage interval' must be adjusted to accommodate the larger particle sizes (usually #9 or 3 mm intervals).

5.4 Allocate and label separate channels on the automatic point counter for each rock type category, e.g. silicate, carbonate, shale, etc. Refer to Form PH-CC-437 for recommended categories.

6. PROCEDURE

6.1 Switch on the automatic point counter.

6.2 Start the examination in the upper left hand corner of the glass slide. Identify the particle under the crosshairs and press the button for the appropriate rock type. This reading is recorded and the slide is automatically moved a regulated distance set by 'stage interval'. Identify the next particle under the crosshairs and repeat this procedure.

Note 6: In the identification and classification of each particle, the following features may be relevant: particle shape, surface texture, scratch hardness, colour, mineralogy, and porosity.

Note 7: In the identification and recognition of sand containing carbonate rock types and minerals, a light acid etch may be found useful. Place the prepared slide face down in a petri dish containing dilute (5 percent) hydrochloric acid and agitate rapidly for 5 to 10 seconds. Remove from the acid and wash gently in water to remove the acid. Allow the sample to air dry before examination. When using this technique with sizes smaller than 300 μm , there is a danger of dissolving too much carbonate.

Note 8: In the event that difficulty exists in the classification of a sand particle, the particle may be removed from the glass slide and referred to a petrographer for detailed study using such techniques as immersion mounts or x-ray diffraction.

6.3 If there is no particle under the crosshairs, press the 'stage only' button. The total reading will not change and the slide will be moved the regulated distance to another particle.

- 6.4 Continue the point count from left to right until the carriage stops at the right hand side. The range of movement of the stage is only about half of the length of the slide, i.e. about 15 readings.
- 6.5 Return the carriage to the left hand side. Use spacing lever to move slide upwards to the start of the next row; 4 mm movement for the retained 2.36 mm fraction, and 3 mm for all other fractions. East/West and North/South spacings must be judged so that 200 particles can be examined across the slide. Repeat this examination procedure on the second line of particles and on the rest of the first half of the slide until 100 particles are counted (or 50 particles in the case of the retained 2.36 mm fraction).
- 6.6 Remove the slide from the stage, rotate it through 180 degrees, and replace it in the stage. Undertake the examination of this second half as described in 6.2 to 6.5 above.
- 6.7 When the sampling target is reached, i.e. when 200 particles have been examined, a buzzer will sound. The petrographic examination is complete for that size fraction.
- 6.8 Press the button for the first 'rock type'. Record the number of particles for the channel. Press the percentage button to determine percentage for the channel. Repeat this procedure for each channel in turn. After all readings have been recorded, press the re-set button to clear memory. For the retained 2.36 mm fraction, do not clear memory until 2 slides have been examined.
- 6.9 Repeat the procedure outlined in 6.2 - 6.8 for each size fraction.

7. CALCULATION

- 7.1 Determine the number of particles of each material type and record each amount on the Report Form (PH-CC-437). Calculate the percentage of each material for each fraction.
- 7.2 Calculate the weighted percent of each rock, mineral or material type present in the sample as indicated below. Calculation of the weighted percentage of chert is used as an example:

$$\text{Weighted percent chert} = \sum_{i=1}^n \frac{A_i \times B_i}{100}$$

- where A_i = % of total sample of sand (minus 4.75 mm portion) retained on an individual sieve (non cumulative)
- B_i = % chert in that sieve fraction
- n = number of individual sieve fractions

- 7.3 For the purpose of calculating the weighted percent chert consider the minus 75 μm fraction to have the same composition as the retained 75 μm sieve fraction.

Note 9: The weighted percent of other materials present in the sample shall be calculated using the procedure above (7.2). Materials of interest might be shale, mica, or contaminating material such as glass or coal.

8. PRECISION

8.1 Single-Observer Precision

For sands with a chert content of between 2 and 5 percent, the single-observer standard deviation has been found to be 0.62%.* Therefore, results of two properly conducted examinations by the same observer on samples of the same sand should not differ by more than 1.8%.*

For sands with a shale content of between 9 and 14 percent, the single-observer standard deviation has been found to be 0.62%.* Therefore, results of two properly conducted examinations by the same observer on samples of the same sand should not differ by more than 1.8%.*

8.2 Multi-Observer Precision

For sands with a chert content of between 2 and 5 percent, the multi-observer standard deviation has been found to be 0.82%.* Therefore, results of two properly conducted examinations by two experienced observers on samples of the same sand should not differ by more than 2.3%.*

For sands with a shale content of between 9 and 14 percent, the multi-observer standard deviation has been found to be 1.68%.* Therefore, results of two properly conducted examinations by two experienced observers on samples of the same sand should not differ by more than 4.7%.*

* These numbers represent, respectively, the (1S) and (D2S) limits as described in ASTM C670, for Preparing Precision Statements for Test Methods for Construction Materials.

METHOD B

9. SAMPLE PREPARATION

9.1 Extract aggregate from RAP or RST as per LS-282, LS-291, or as specified in the contract documents and retain the fine aggregate portion (material passing 4.75 mm sieve) of the extracted aggregate.

9.2 Reduce the fine aggregate portion by splitting to 500 ± 50 g.

9.3 Wash the fine aggregate sample over the 75 μ m sieve as per LS-601.

9.4 Dry the sample to a constant mass at a temperature of $110 \pm 5^\circ\text{C}$, and allow the sample to cool to room temperature.

9.5 Place the test sample in the uppermost of a progressive series of sieves including the 2.36 mm, 1.18 mm, and 75 μ m sizes. Sieving should be undertaken according to LS-602. Place each fraction in a separate container and label.

9.6 Prepare grain mounts on glass slides for the 2.36 and 1.18 mm fractions as per Part A of this test method Sections 4.4 and 4.5.

10. SETTING UP APPARATUS

- 10.1 Set up the apparatus as per Part A of this test method Sections 5.1 through 5.4.
- 10.2 Allocate and label separate channels on the automatic point counter for each aggregate category as indicated in Table 1.

11. PROCEDURE

- 11.1 Complete the test procedure as per Part A, Sections 6.1 through 6.9 on the prepared 2.36 and 1.18 mm fractions. Sampling target is 200 particles for each fraction, consistent with Part A, Section 6 requirements.
- 11.2 Classify the particles aggregate categories as listed in Table 1.
- 11.3 Report the results in Table 1.

12. REPORTING

METHOD A The report of the examination should include the following:

- 11.1 The aggregate source name, location, and mineral aggregate inventory number.
- 11.2 The sample number and date of the test.
- 11.3 The name of the analyst.
- 11.4 The percentage of various rock types on each individual sieve size.
- 11.5 The weighted average percent chert and other materials present in the sample.
- 11.6 The percentage of any component to one decimal place.

METHOD B

- 11.7 The report of the examination should include the following:
- 11.8 The aggregate source name, location, and mineral aggregate inventory number.
- 11.9 The sample number and date of the test.
- 11.10 The name of the analyst.
- 11.11 The percentage of various aggregate categories on the 2.36 and 1.18 mm sieve size.

13. SAMPLE STORAGE

PARTS A AND B

- 13.1 Samples should be retained indefinitely on their glass slides. Wooden boxes for storage are available commercially.

Note 10: A wooden slide box suitable for storing 75 x 50 mm glass slides is available from Fisher Scientific (catalogue #12-550C).



MTO LS-616 FINE AGGREGATE PETROGRAPHIC ANALYSIS

SAMPLE No: _____ DATE: _____ MAIDB No. _____ SOURCE NAME: _____

ANALYST: _____

ROCK, MINERAL OR MATERIAL TYPE	SIEVE SIZE												WEIGHTED AVERAGE
	4.75 - 2.36 mm		2.36 - 1.18 mm		1.18 - 600 µm		600 - 300 µm		300 - 150 µm		150 - 75 µm		
	#	%	#	%	#	%	#	%	#	%	#	%	
Silicate rocks and minerals (e.g. granite, gabbro, gneiss, quartz, feldspar, amphibole)*													
Carbonate rocks and minerals (e.g. limestone, dolostone, calcite, dolomite)													
Conglomerate, sandstone, quartzite (e.g. greywacke, arkose)													
Shale, argillite, siltstone, clay, ochre													
Micaceous minerals (e.g. biotite, muscovite, chlorite)													
Chert (leached and unleached), flint, jasper													
Cemented Particles													
Sulphate rocks and minerals (e.g. gypsum, anhydrite)													
Sulphide rocks and minerals (e.g. pyrite, pyrrhotite, chalcopyrite)													
Oxide minerals (e.g. magnetite, ilmenite, hematite, chromite)													
Contamination (e.g. glass, slag, coal, reclaimed concrete)													
Other (please list, describe)													
TOTAL													
GRADATION: percent retained on individual sieve													Ret.
													Pass
Estimated percent totally crushed particles													

*Includes all silicate rock and mineral types except those listed as separate individual categories within the table (e.g. chert, micaceous minerals, conglomerate, sandstone, quartzite).

COMMENTS AND NOTES:

Table 1: Petrographic Classification of Fine Aggregate from RAP or RST

Sample Number:					
Analyst:					
Date:				Retained 2.36 mm portion	
				Retained 1.18 mm portion	
Aggregate Category	Description	#	%	#	%
Category 1	Lithologies included in DSM #3.05.25 and their major mineral components: Diabase, dolomitic sandstone, gabbro, gneiss, meta-arkose, meta-gabbro, traprock, granite; quartz, feldspar, amphibole, pyroxene, garnet, epidote, magnetite				
Category 2:	Carbonate Rocks and Minerals, includes calcite, dolomite				
Category 3:	Other, includes iron sulphides and other metallic sulphide minerals (pyrite, marcasite, chalcopyrite, etc.); chert (leached and unleached), flint, jasper; mica (biotite, muscovite, chlorite, etc.); siltstone, shale argillite, clay, ochre; sulphate rocks, and minerals (gypsum, barite, etc.); cemented particles; contamination (wood, glass, ceramic, concrete, etc.); other unidentifiable particles				
Total					