PROCEDURE FOR THE PETROGRAPHIC ANALYSIS OF FINE AGGREGATE

1. SCOPE

1.1 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. Firstly, the method 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.

2. RELEVANT DOCUMENTS

2.1 ASTM C 294, C 295

2.2 MTO LS-602, LS-609

2.3 EM - 91, Petrographic Examination of Aggregate and Concrete in Ontario, Engineering Materials Office, Ministry of Transportation.

3. APPARATUS

3.1 Epoxy, hardener and resin (5 minute setting).

3.2 Mixing surface, disposable e.g. cardboard.

3.3 Mixing stick, disposable e.g. coffee stirrer.

3.4 Precleaned glass microscope slides, 50 x 75 mm (e.g. Fisher Scientific, catalogue #12-550C).

3.5 Stereoscopic microscope, with cross hairs in at least one eyepiece, final magnification of not less than 50x. A source of illumination.

3.6 Mechanical stage i.e. electrically triggered specimen carriage (Notes 1 & 2).

3.7 Automatic point counter with at least 10 separate channels.

3.8 Pointer, metal, to scratch particles.

3.9 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.

4. SAMPLE PREPARATION

4.1 Dry the sample to a constant mass at a temperature of 110 ± 5°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 gm.

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 MTO Method 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.

4.6 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 automatic point counter.

6.2 Start examination in upper left hand corner of the glass slide. Identify the particle under the crosshairs and press 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 slide from 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 button for the first 'rock type'. Record number of particles for the channel. Press percentage button to determine percentage for the channel. Repeat this procedure for each channel in turn. After all readings have been recorded, press 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 chert of the sample as follows:

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

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.

7.2.1 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: When desired, or required, the weighted percent of other materials present in the sample may be calculated using the procedure above (7.2). Materials of interest might be shale, mica or contaminating material such as glass or coal.

8. REPORTING

8.1 The report of the examination should include the following:

8.1.1 The aggregate source name, location and mineral aggregate inventory number.

8.1.2 The sample number and date.

8.1.3 The name of the analyst.

8.1.4 The percentage of various rock types on each individual sieve size.

8.1.5 The weighted average percent chert and other materials present in the sample.

8.1.6 The percentage of any component to one decimal place.

9. SAMPLE STORAGE

9.1 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).

10. PRECISION

10.1 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 %.*

10.2 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 Practice C670, for Preparing Precision Statements for Test Methods for Construction Materials.

Ministry of Transportation
Ontario

MTO LS – 616 FINE AGGREGATE PETROGRAPHIC ANALYSIS

SAMPLE NO.: _____ DATE: _____

SOURCE NAME: _____

GRAN. I. NO.: _____

LOCATION: _____

ANALYST: _____

	SIEVE SIZE											
	4.75 – 2.36		2.36 – 1.18		1.18 – 600		600 – 300		300 – 150		150 – 075	
	#	%	#	%	#	%	#	%	#	%	#	%
SILICATE ROCKS (AND ASSOCIATED MINERALS, QUARTZ, FELDSPAR, AMPHIBOLE), SANDSTONE, QUARTZITE												
CARBONATE ROCKS AND ASSOCIATED MINERALS, CALCITE, DOLOMITE												
SHALE, ARGILLITE (soft), CLAY, OCHRE												
MICA (Biotite, Muscovite' Chlorite)												
CHERT (Leached and Unleached), FLINT, JASPER												
CONTAMINATION, ie. glass, slag, coal, etc.												
CEMENTED PARTICLES												
TOTAL												
GRADATION, PERCENT RETAINED ON INDIVIDUAL SIEVE												
ESTIMATED PERCENT TOTALLY CRUSHED PARTICLES												

COMMENTS AND NOTES:

WEIGHTED AVERAGE PERCENT CHERT _____

PH-CC-437 81-08

Made from recovered materials

Figure 1 Fine Aggregate Petrographic Analysis Data Card