Test Procedure for

MATERIAL FINER THAN 75 μM (NO. 200) SIEVE IN MINERAL AGGREGATES (DECANTATION TEST FOR CONCRETE AGGREGATES)



TxDOT Designation: Tex-406-A

Effective Date: August 1999

1. SCOPE

- 1.1 Use this method to perform the decantation test for concrete aggregates on material finer than the 75 μ m (No. 200) sieve.
- 1.2 The values given in parentheses (if provided) are not standard and may not be exact mathematical conversions. Use each system of units separately. Combining values from the two systems may result in nonconformance with the standard.

PART I—LABORATORY METHOD

2. SCOPE

2.1 Use this procedure to perform the laboratory method for the decantation test.

3. APPARATUS

- 3.1 *Balance*, Class G2 in accordance with Tex-901-K, minimum capacity of 6000 g.
- 3.2 Drying oven, maintained at $110 \pm 5^{\circ}$ C (230 $\pm 9^{\circ}$ F).
- **3.3** Drying oven, maintained at $60 \pm 5^{\circ}$ C (140 $\pm 9^{\circ}$ F).
- 3.4 *Pan*, approximately 305 mm (12 in.) in diameter and 127 mm (5 in.) deep.
- 3.5 *Set of standard U.S. sieves,* meeting requirements of Tex-907-K, in the following sizes:
 - 4.75 mm (No. 4)
 - 75 µm (No. 200).
- 3.6 *Sample splitter or quartering cloth.*
- 3.7 *Plaster of paris molds*, with filter paper (optional).

4. PROCEDURE

- 4.1 If sample contains fine and coarse aggregates, which have separate specification requirements, separate the materials into coarse and fine aggregate samples as defined by the specifications. Test these samples separately.
- 4.2 Mix sample thoroughly.
- 4.3 If testing sample in accordance with Tex-401-A, reduce the sample to the minimum test sample mass described in that method. If not testing in accordance with Tex-401-A, reduce the sample to one of the masses according to its nominal maximum size listed in Table 1.
- 4.4 Dry the aggregate to constant mass at 110° C (230°F). Weigh the oven-dry mass to the nearest 0.1 g and record as W_1 in Section 5.

Note 1—When drying bituminous (RAP) materials, dry at 60°C (140°F) to a constant mass.

- 4.5 Place the sample into a pan and fill with tap water until all of the material is submerged. Soak the material for a minimum of 12 hours.
- 4.6 After the aggregate is thoroughly saturated, vigorously agitate the material and then decant the wash water over the 75 μ m (No. 200) sieve.
- 4.7 Add water to the pan and repeat washing and decanting until wash water is clear.
- 4.8 Recover any aggregate retained on the 75 μ m (No. 200) sieve and return to the pan.
- 4.9 If the material finer than the 75 μ m (No. 200) sieve is to be tested for percent limestone in Part III, follow Sections 4.10–4.14; if not, go to Section 4.14.
- 4.10 Place the pan containing the minus (-) 75 μ m (No. 200) sieve material and wash water aside where it will not be disturbed. Allow all of the fine material to settle to the bottom of the pan (approximately 2–3 hours). If material fails to settle in this amount of time, proceed to Section 4.12.
- 4.11 Decant or siphon off the clear water.
- 4.12 In cases where the material fails to settle in a reasonable period, decant the water onto plaster of paris molds lined with filter paper. After the water has disappeared, dry the filter paper in the $60 \pm 5^{\circ}$ C ($140 \pm 9^{\circ}$ F) oven, then brush the fines from the filter paper with a stiff brush into the pan of fines.
- 4.13 Dry the remaining material to a constant weight, using a $60 \pm 5^{\circ}$ C (140 $\pm 9^{\circ}$ F) oven if there is difficulty in removing the moisture from the pan.
- 4.14 Dry the washed plus (+) 75 μ m (No. 200) material to a constant mass in a 110 ± 5°C (230 ± 9°F) oven; weigh the net mass of the washed aggregate to the nearest 0.1 g and record as W_2 in Section 5.

Nominal Max. Size	Minimum Mass, g
37.50 mm (1-1/2 in.) or larger	5000
25.00 mm (1 in.) or larger	3500
19.00 mm (3/4 in.)	2500
9.5 mm (3/8 in.)	1000
4.75 mm (No. 4)	500
less than 4.75 mm (No. 4)	200

5. CALCULATIONS

5.1 Calculate the Percent Loss of material finer than 75 μm (No. 200):

Percent Loss = $100 [(W_1 - W_2) / W_1]$

Where: W_1 = original dry mass of the sample, g W_2 = final dry weight, g.

6. REPORT

6.1 Report Percent Loss to the nearest 0.1%.

PART II—FIELD METHOD FOR CONCRETE AGGREGATES

7. SCOPE

7.1 Part II provides a rapid approximation intended only when correlated with Part I. When material is borderline, use Part I.

8. APPARATUS

- 8.1 *Balance*, same as used in Part I.
- 8.2 *Wide mouth funnel.*
- 8.3 *Pycnometer*, 2 L (1/2 gal.) glass Mason jar with a pycnometer cap, calibrated in accordance with Tex-403-A.

9. PROCEDURE

9.1 If sample contains fine and coarse aggregates, which have separate specification requirements, separate coarse and fine aggregates into different samples as defined by the specifications. Test the samples separately.

9.2	Mix sample thoroughly.
9.3	Secure a portion weighing approximately 1200 g. (The sample need not be weighed, and the moisture content of the material is not considered since these factors have no bearing upon the test values.)
9.4	Place the sample into the pycnometer jar and cover with water.
9.5	If the material is not drier than saturated surface-dry (SSD), proceed immediately to Section 9.6. If the moist condition of the material is in doubt, or if the material is in stockpile condition and drier than SSD, allow to stand undisturbed for at least 12 hours.
9.6	Fill the jar with water at approximately $23 \pm 2^{\circ}$ C ($73 \pm 3^{\circ}$ F), to within 12.5 mm (0.5 in.) of the rim and screw the lid on. Then finish filling the jar with water to the top.
9.7	Stop the hole in the cap with a finger and roll the jar to free the entrapped air. Raise and lower the jar in such a manner that the material will flow back and forth in the jar while being rolled.
9.8	Refill the cap to remove any air bubbles. Take precautions to prevent loss of fine material while removing the entrapped air.
9.9	Use a towel to dry the outside of the jar and add water until the jar is full and has a rounded bead of water at the top. Weigh and record the mass to the nearest 0.5 g as Z_1 under Section 10.
9.10	When testing sands, close the opening in the cap with a finger and agitate the contents of the jar by rolling the pycnometer with a swinging motion. When testing coarse aggregate, gently roll the pycnometer to avoid breaking the jar.
9.11	Place the jar upright and remove the cap.
9.12	Slowly pour the liquid over a 75 μ m (No. 200) sieve, taking care to lose none of the plus (+) 75 μ m (No. 200) material.
9.13	Replace the cap and fill to full again, leaving a rounded bead of water at the top of the cap. Repeat Sections 9.10–9.12 until the water above the aggregate is reasonably clear.
9.14	Recover any material that retained on the 75 μ m (No. 200) sieve and return it to the pycnometer.
9.15	Screw the lid onto the jar and fill again with water at $23 \pm 2^{\circ}$ C ($73 \pm 3^{\circ}$ F).
9.16	Dry the outside of the pycnometer and complete filling with water, leaving a rounded bead of water on top of the pycnometer cap.
9.17	Weigh and record the mass to the nearest 0.5 g as Z_2 under Section 10.

10. CALCULATIONS

10.1 Calculate the Percent Loss of material finer than the 75 μm (No. 200):

Percent Loss = $100[(Z_1 - Z_2)/(Z_1 - Y)]$

Where:

 Z_1 = mass of pycnometer containing sample and water to fill, before washing, kg (lb.)

 Z_2 = mass of pycnometer containing sample and water to fill, after washing, kg (lb.)

Y = calibrated mass of pycnometer filled with only water at approximately the same temperature at which Z_1 and Z_2 were determined (Tex-403-A, Section 6).

11. REPORT

11.1 Report percent loss to the nearest 0.1%.

Note 2—The percentage by mass of material lost by decantation is equal to the percentage by absolute volume, assuming that the specific gravity of the material is the same as that of the particles remaining.

Note 3—Any pan of suitable size and texture will be satisfactory. Avoid the use of metal pans, which react with aggregates.

Note 4—When correlating the field method (Part II) with the laboratory method (Part I), perform the tests according to both Part I and Part II and record the results. The difference between the results for Part I and Part II becomes a factor, which should be added to the results obtained when using the Part II method. If the results from using Part II plus the factor produce a failing value, rerun the test using Part I.

PART III—PERCENT OF LIMESTONE IN DECANTATION MATERIAL

12. SCOPE

- 12.1 This procedure is intended to distinguish between the limestone and non-limestone fractions of minus (-) 75 μ m (No. 200) material.
- 12.2 The calcium carbonate $(CaCO_3)$ limestone portion of the sample can be determined accurately by a dilute hydrochloric acid titration. The dilute acid will react with the CaCO₃ but will not affect the non-limestone particles.
- 12.3 The basic chemical equation is:

 $CaCO_3 + 2H^+ \rightarrow Ca^{+2} + CO_2(gas) + H_2O$

12.4 Using a known sample weight and known acid concentration, the CaCO₃ content of the sample can be calculated from the amount of acid used in titration.

13. APPARATUS

- 13.1 *Oven*, maintained at $110 \pm 5^{\circ}C (230 \pm 9^{\circ}F)$.
- 13.2 *Desiccator*, with indicating type silica-gel desiccant.
- **13.3** *Balance*, Class G1 in accordance with Tex-901-K, minimum capacity of 100 g, suitable for rapid weighing.
- **13.4** *Beaker*, 400 mL (12 oz.)
- 13.5 *Stirrer*, magnetic type, with a Teflon-covered bar.
- 13.6 Buret, Class A, 50 mL (1.7 fl. oz.) capacity graduated to 0.1 mL (0.003 fl. oz.)
- 13.7 pH meter, accuracy of ± 0.1 pH unit or better within a temperature range of 0–100°C (32–212°F). The meter should have either a manual or automatic temperature compensator.

14. MATERIALS

- 14.1 *Hydrochloric acid solution,* 1.00 ± 0.005 N, with the normality of the solution accurately determined by direct comparison with a 1.000 ± 0.005 N sodium hydroxide solution prepared and standardized in accordance with Tex-600-J, Part II.
- 14.2 *Distilled or deionized water.*

15. PREPARING SAMPLE

- 15.1 Perform Part I, Section 4, and save the material passing the 75 μm (No. 200) sieve from the concrete coarse aggregate, as stated in Section 4.9 of the procedure.
- 15.2 Dry the material to a constant mass in the oven and then cool in a desiccator.
- 15.3 Pulverize with a mortar and pestle.

16. PROCEDURE

- 16.1 Weigh out 2 ± 0.01 g of material into a 400 mL (12 fl. oz.) beaker of known mass.
- 16.2 Carefully add 125–150 mL (4–5 fl. oz.) of distilled or deionized water, add the stirring bar to the beaker, and place on magnetic stirrer.
- 16.3 Start the stirrer and insert the pH meter electrodes into the mixture.

16.4 Titrate the mixture with the acid solution until a stable pH of 2.7 is attained (approximately 3 minutes). As acid is added, the pH of the mixture will go down to about 5.5, at which time CO₂ will start to be generated.
16.4.1 Add acid more slowly at this point, so the effervescence will not splatter sample from the beaker.
16.4.2 If the pH meter has a manual temperature compensator, make the adjustment to correct for the temperature of the solution at the endpoint.

16.4.3 At a pH of 2.7, read the volume of acid used in mL (fl. oz.)

17. CALCULATIONS

17.1 Calculate the Percent Loss of minus (-) 75 μm (No. 200) material as percent limestone:

% Limestone = 5[N(V) / X]

Where:

N = Normality of HCl used

V = Volume of HCl used in titration, mL (fl. oz.)

X = Sample weight, g.

Note 5—Unless a value is specified elsewhere in the plans or specifications, the minus (-) 75 μ m (No. 200) material obtained from the limestone coarse aggregate can be considered as dust of fracture when the percent limestone is equal to or more than 75.