Test Procedure for

LASER DIFFRACTION PARTICLE SIZE DISTRIBUTION ANALYZER

TxDOT Designation: Tex-238-F

Effective Date: August 1999

1. SCOPE

1.1 This method determines the particle size distribution of material finer than the 200 µm (No. 75) 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.

2. APPARATUS

2.1 *Horiba Laser Diffraction Particle Size Distribution Analyzer,* or exact equal.

2.2 *Rotary micro riffler.*

2.3 *Small sample splitter.*

3. MATERIALS

3.1 *Mediums*—distilled water, glycerin, ethylene glycol, isopropyl alcohol, viscous oil.

3.2 *Dispersants*—sodium pyrophosphate, sodium hexametaphosphate, Darvan 821-A, aerosol OT-100% (sodium dioctyl-sulfo-succanate), menhaden oil, viscous oil.

4. PROCEDURES

4.1 *Representative Sampling:*

4.1.1 Select a representative sample from the lot. This becomes increasingly difficult as the sample size decreases. Samples must range from at least 0.02–1 g to analyze the material.

4.1.2 If the material passing the 150 µm (No. 100) sieve is separate from the rest of the aggregate, then obtain from the source (bin, drum, silo, etc.) a sample of approximately 1 kg (2.2 lb.)

4.1.3 If the desired fraction is mixed with the coarser fractions of the aggregate, then separate it from the blend. Select enough aggregate so that approximately 1 kg (2.2 lb.) of material will pass the 150 μm (No. 100) screen with dry sieving. Dry sieving must be done by hand.

4.1.4 Dry-sieve the full-graded aggregate sample to separate as much of the fraction passing the 150 μm (No. 100) screen as is practical through dry-sieving without causing any appreciable abrasion of the aggregate particles.

4.1.4.1 Several coarser screens must be included so that the amount retained on the 150 μm (No. 100) screen is divided into several fractions.

4.1.4.2 The initial steps facilitate the subsequent steps of washing the individual fractions of the full graded sample over the 150 μm (No. 100) screen.

4.1.5 Remove and store the fraction passing, by dry sieving, the 150 μm (No. 100) screen.

4.1.6 Wash the other aggregate fractions over a 150 μm (No. 100) screen to collect the remainder of the particles finer than 150 μm (No. 100) screen that adhere to the larger aggregates. Reduce the washing time by washing the larger fractions over the screen separately.

4.1.7 Wash until rinse water looks clean.

4.1.7.1 Include the washing procedure because the particles that are not removed by dry sieving alone tend to be much finer than those that are removed; therefore, testing the dry sieved material alone may not be representative of the true gradation.

4.1.7.2 The sample's particles will conglomerate and cake up during drying. This is expected and not detrimental to the remainder of the analysis.

4.1.8 After drying, carefully and thoroughly mix the sample to break up the conglomerates until the maximum size is approximately 1 mm (0.04 in.) or less.

4.1.9 Mix together the two minus 150 μm (No. 100) fractions, one from dry sieving, and the other from washing, to achieve the minus 150 μm (No. 100) aggregate sample.

4.2 *Quartering:*

4.2.1 Spread the entire 1 kg (2.2 lb.) sample out in a clean, dry tray and mix well.

4.2.2 Extract about 800 g of the minus 150 μm (No. 100) aggregate and place into a dish by either randomly scooping from the tray or by quartering. Store the remainder in a covered container or closeable bag.

4.2.3 Quarter the 800 g sample once (or split it twice) to achieve a 200 g sample.

4.2.4 Riffle the sample.

4.2.5 Return the remainder to the storage container.

4.3 *Riffling:*

4.3.1 Divide the samples further by riffling with a Rotary Micro Riffler.

4.3.2 Load sixteen clean containers into the collector and begin riffling the 200 g sample.

4.3.3 When the riffling is complete, and the sample is completely separate in the containers, remove two opposite samples from the collector and return their contents to the riffler chute. Store the contents of the other fourteen containers.

4.3.4 Following the steps outlined above, riffle the sample again into sixteen containers.

4.3.5 The contents of the sixteen containers must weigh approximately 1.5 g each. Cover the containers and label each with the material's designation. After selecting a proper medium and dispersant solution, there are sixteen representative containers of samples to use for analyses. Depending on the volume of the medium/dispersant solution used, each container may yield one to five samples.

4.3.6 Extract the samples from each container with the use of a small, slender spatula. Use the spatula to mix the contents of the container thoroughly prior to drawing out the sample. Do not acquire the sample from the container by sprinkling because this causes segregation.

5. SELECTING MEDIUMS AND DISPERSANTS

5.1 The mediums and dispersants are shown in Table 1. The table is a selection guide for identifying possible medium/dispersant solutions that could work well in analyzing the particle size distribution of a given type of minus 150 μm (No. 100) aggregate in Horiba's LA-500. In the table, each of the mediums is coupled with the recommended amount of an appropriate dispersant.

Table 1—Mediums and Dispersants Selection Guide

Water Insoluble Materials:	Mediums	Dispersants
FINES	1-> 100% Distilled Water	1-> none
- Clayey Materials	2-> 99.5% Distilled Water	2-> 0.5% Sodium Pyrophosphate
- Aglime	3-> 96% Distilled Water	3-> 4% Sodium Hexametaphosphate $(NaPO_3)_6$
- Baghouse Dusts	4-> 99% Distilled Water	4-> 1% Darvan 821-A
COARSE	5-> 49.5% Glycerin 49.5% Distilled Water	5-> 1% Darvan 821-A
- Sandy Materials	6-> 69.5% Glycerin 29.5% Distilled Water	6-> 1% Darvan 821-A
	7-> 49% Ethylene Glycol 49% Distilled Water	7-> 2% Sodium Hexametaphosphate

Water Insoluble Materials:	Mediums	Dispersants
FINES - Hydrated Lime	8-> 99.5% Isopropyl Alcohol	8-> 0.5% Aerosol OT-100% (Sodium dioctyl-sulfo-succanate)
	9-> 99% Isopropyl Alcohol	9-> 1% Menhaden Oil
	10-> 79.5% Isopropyl Alcohol 19.5% Glycerin	10-> 1% Menhaden Oil
COARSE	11-> Viscous Oil	11-> Viscous Oil

5.2 *Selection Procedure:*

5.2.1 Test water-insoluble fillers with medium/dispersant combinations 1 through 7, as shown in Table 1.

5.2.2 Test water-soluble fillers with solutions 8 through 11 as shown in Table 1.

5.2.3 After narrowing down an appropriate group of solutions for a filler, compare the solutions for their respective dispersing ability by the test container settlement test described in Section 4.2.2.

5.2.4 Use the 1.5 g samples attained by riffling to select an appropriate medium/dispersant combination for testing.

5.2.5 Select several dispersants from Table 1 based on initial observation of the minus 150 μm (No. 100) aggregates (water solubility and relative fineness).

5.2.6 Combine and agitate the minus 150 μm (No. 100) aggregate and trial medium/dispersant solutions in separate test containers.

5.2.7 Mark the containers and set in a stand, undisturbed.

5.2.8 After several minutes, observe the containers to determine the best medium/dispersant. The filler that has settled the least is the best dispersed.

6. PARTICLE SIZE ANALYSIS BY LASER DIFFUSION

6.1 Horiba's analyzer determines the particle size distribution of materials while held in a fluid suspension. The detection range of the analyzer is 0.1–200 μm (No. 75) particles. The fluid suspension or slurry is comprised of the following components:

- sample (powder)
- fluid medium
- dispersing agent.

6.2 The sample size varies according to the translucence of the fluid suspension. Depending on the sample's properties, anywhere from 0.02–1 g of powder can yield the proper

concentration required for a 200–250 mL (6.8–8.5 fl. oz.) suspension. Adequate sample sizes, mediums, and dispersants can only be found through experimentation.

6.3 Select an appropriate medium/dispersant combination, as described in the previous section, using the test tube settlement test. Determine the corresponding sample size by running through the testing procedure described in this section.

6.4 In order to assure consistent results of riffled powders, the operator must adhere to the following procedures for operation of the LA-500. The first procedure is the initial warm-up procedure, and the second is the sample testing procedure, assuming that a medium/dispersant solution has already been selected and that several samples have already been prepared in the size required.

6.5 *Procedures:*

6.5.1 *Initial Warm-Up:*

6.5.1.1 Turn the instrument on and allow it to warm up.

6.5.1.2 If testing water-insoluble samples, add 200–250 mL (6.8–8.5 fl. oz.) distilled water to the ultrasonic chamber; if testing water-soluble samples, add approximately 150 mL (5.1 fl. oz.) of isopropyl alcohol.

6.5.1.3 Set mechanical agitation at 7.

6.5.1.4 Set circulation speed at 7.

6.5.1.5 Continue circulation and agitation for 20 minutes.

6.5.1.6 While running, close the black cover to engage laser, remove the right panel, adjust beam angles by turning the vertical and horizontal displacement screws until all indicator arrows are lit, and return the cover.

6.5.1.7 If there are no leaks, drain the water or isopropyl alcohol.

6.5.2 *Running Samples for Particle Size Analysis:*

6.5.2.1 The LA-500 testing conditions are set or loaded in from memory.

- Distribution Type = 1
- Method = Volume
- Reading Time = 100 s.

6.5.2.2 If testing water-insoluble samples, add 200 to 250 mL (6.8 to 8.5 fl. oz.) distilled water to the ultrasonic chamber; if testing water-soluble samples, use approximately 150 mL (5.1 fl. oz.) of isopropyl alcohol.

6.5.2.3 Begin mechanical agitation at 7.

6.5.2.4 Start circulation through measuring cell at speed 7 for at least 4 minutes.

6.5.2.5 Allow flow through cell for a few minutes.

6.5.2.6 Observe diffraction pattern for stability. If this is the first test since power up, the concentration level of the medium/dispersant alone may appear significant, but the initial Blank Value will compensate.

6.5.2.7 Check system for leaks.

6.5.2.8 Record the Blank Value if the diffraction pattern seems stable, with no wild fluctuations in any of the channels.

6.5.2.9 Observe diffraction image for stability, especially the concentration level (the concentration level must be stable and at its thinnest point).

6.5.2.10 Begin ultrasonic agitation at a 2-minute interval.

6.5.2.11 Immediately begin to add the sample slowly into the ultrasonic chamber with a spatula until the concentration is in the “good” region. If the desired concentration level is not reached after the 2 minutes of ultrasonic agitation has expired, repeat the test, adding the sample at a faster rate.

6.5.2.12 If there is too much of the sample, add more medium/dispersant solution to 'thin' out the slurry. With increased ultrasonic agitation the concentration will tend towards the “thin” region; however, this is not suggested practice because increased ultrasonic agitation will lower the repeatability of the results.

6.5.2.13 Allow the diffraction pattern to stabilize (2 minutes minimum), then begin the analysis.

6.5.2.14 When all replicate tests on the current sample are completed, drain the system.

6.5.2.15 While running, remove the black cover to engage the laser, remove the right panel, adjust beam angles, and return the cover.

6.5.2.16 If there are no leaks, drain the water or isopropyl alcohol.

6.5.2.17 Repeat the testing procedures for the next sample, if the sample is of the same material and the same medium/dispersant solution is to be used.

6.5.2.18 Remove and clean the measuring cell if a new material is to be tested.

Note 1—Avoid testing in excessively dusty areas. Trapped lint in a sample can considerably influence the results of the analysis. In normal laboratories, especially where aggregate testing is conducted, air-borne particulates are excessive. For these reasons, take special care when preparing and storing the samples.

Note 2—Using cleaned and towel-dried equipment (beakers, pan, tubes, etc.), as well as rinsing the equipment with distilled water helps minimize the risk of unwanted foreign contaminants. When towel drying, use a lint-free towel because air-drying tends to attract more air-borne particles to the equipment.

Note 3—Using water in testing is imperative; however, using water in equipment and sample washing is also encouraged. If a sample is washed with regular water, minerals are deposited on the sample while drying. These deposits can affect the results of a

gradation analysis. This is evident when considering that the solubility of the minerals vary with each of the mediums used to conduct the test. Use of water avoids contamination of the sample by unwanted mineral precipitates or residues. If possible, use compressed air to disperse the water after washing.

Note 4—When drying samples in conventional or microwave ovens, avoid any excessive airflow over the sample. For conventional ovens, maintain a temperature of 110 ± 5 °C (230 ± 9 °F). Minimize any loss of the sample when drying by not positioning samples directly under exhausts or fans, by using tall beakers or containers, and/or keeping the airflow to a minimum. During mixing of large quantities of a sample, be careful not to lose significant amounts of the sample to the air by overly vigorous agitation. Covered storage for both cleaned equipment and prepared samples is imperative for proper sampling.