Test Procedure for

DETERMINING THE PRESENCE OF HARMFUL CLAYS USING METHYLENE BLUE

TxDOT Designation: Tex-252-F

Effective Date: July 2021

1. SCOPE

1.1 This test identifies the presence of harmful clays in aggregate and mineral fillers. Hot Mix Asphalt produced with fines of harmful clays may be susceptible to moisture damage and permanent deformation.

2. APPARATUS

2.1 *Balance,* complying with Tex-901-K, Class G1.

2.2 *Balance,* complying with Tex-901-K, Class B for preparing the methylene blue solution.

2.3 *Burette,* minimum capacity of 50 mL with 0.1 mL graduations.

2.4 *Distilled water.*

2.5 *Filter paper,* Whatman No. 2.

2.6 *Glass stirring rod.*

2.7 *Griffin beaker,* 500 mL.

2.8 *Magnetic mixer with stir bar.*

2.9 *Metal trowel.*

2.10 *Methylene blue,* reagent grade-dated stored in a dark cabinet at lab temperature with safety data sheet (SDS) available.

2.11 *Methylene blue solution,* stored in an amber bottle in a dark cabinet at lab temperature for a maximum of 4 mo. from the production date.

2.12 *Mortar and pestle.*

2.13 *Oven,* capable of maintaining a minimum temperature of 230 ± 9°F.

2.14 *Pans or containers*, rust and heat resistant and capable of holding water and fines.

2.15 *Standard U.S. sieves*, meeting the requirements of Tex-907-K.

2.16 *Timer* or *stopwatch*.

2.17 *Volumetric flask*, 200 mL for producing the methylene blue solution.

3. PROCEDURE

3.1 *Preparing Test Sample.*

3.1.1 Obtain a minimum 2,500 g representative sample in accordance with Tex-221-F.

3.1.2 Place the sample in a wash pan or container and completely cover with clean potable water.

3.1.3 Gently mix the sample with the hands to break up clay lumps and friable particles and loosen the coating on fines on the aggregate or mineral filler.

3.1.4 Rinse any sample particles clinging to the hands back into the wash pan or container.

3.1.5 Soak the sample a minimum of 24 hr.

3.1.6 After soaking, remix the sample with the hands as noted in Section 3.1.3 and repeat Section 3.1.4.

3.1.7 Stack a No. 8 (2.36 mm) sieve on a No. 200 (75 μm) sieve and place in a pan or container to collect the wash water and fines.

Note 1—Depending on the gradation of the sample, No. 30 (0.595 mm) sieve or a No. 50 (0.297 mm) sieve may be used to stack on the No. 200 (75 μm) instead of the No. 8 (2.36 mm) sieve.

3.1.8 Wash the wetted sample over the stacked sieves in small batches to prevent overloading and damage to the No. 200 (75 μm) sieve. Collect the washed material passing the No. 200 (75 μm) sieve and wash water.

3.1.9 Place the pan or container with the washed material passing the No. 200 (75 μm) sieve and wash water in the oven at 230 ± 9°F (110 ± 5°C) and dry to constant mass.

Note 2—Several days may be required to evaporate the water from the sample.

3.1.10 Discard the material retained on the sieves.

3.1.11 After the material has dried, allow the material to cool to room temperature.

3.1.12 Remove the material from the pan or container and place into the mortar.

Note 3—The sides of the pan or container may need to be scraped with a metal trowel to remove the fines adhering to the pan or container.

3.1.13 Using the pestle, grind the material until it passes a No. 50 (0.297 mm) sieve and place in a pan or container. After all the material passes the No. 50 (0.297 mm) sieve, mix the material thoroughly.

3.2 *Preparing Methylene Blue Solution.*

3.2.1 In a 200-mL volumetric flask, dissolve 1,000 mg (1.000 g) of methylene blue in distilled water to produce 200 mL of solution. Stir the solution with a mixer for at least 1 hr.

Note 4—Each 1 mL of methylene blue solution must contain 5 mg (0.005 g) of methylene blue.

Note 5—It is recommended small quantities (200 mL) of methylene blue solution are produced at a time to avoid having to dispose of excess solution.

3.2.2 Transfer the methylene blue solution into an amber bottle and store in a dark cabinet.

3.3 *Determining Presence of Harmful Clays Using Methylene Blue.*

3.3.1 Weigh 10.00 ± 0.01 g of the material collected from Section 3.1.13 and place into a 500-mL beaker. Record as W.

3.3.2 Add 30 mL of distilled water into the beaker and stir with the mixer to make a slurry.

3.3.3 While the slurry is mixing, fill the burette with the methylene blue solution. Record the initial volume.

3.3.4 Add 0.5 mL of the methylene blue solution to the slurry and stir for 1 min.

Note 6—To reduce the testing time and determine a starting volume of methylene blue solution, the increments of methylene blue solution may be increased to 3 mL. Continue to perform the titration as discussed in Section 3.3.4 to Section 3.3.9 with 3 mL increments until the light blue halo is observed as shown in Figure 1. Once the blue halo is observed, determine the starting volume of methylene blue solution by subtracting the initial volume from the final volume. Discard the sample and repeat the test beginning with Section 3.3.1 and the starting volume of methylene blue solution determined. Verify the blue halo is not observed after adding the methylene blue solution and mixing for 5 min. If the blue halo is observed, discard the sample and repeat the test using 3 mL less of methylene blue solution than the starting volume. Continue the procedure with 0.5 mL increments described in Section 3.3.4.

Note 7—If testing a sample for meeting a specification, the amount of added methylene blue could be adjusted to the specification limit by back-calculating the volume of methylene blue from the dry weight W and the specification MBV limit. Continue to perform Sections 3.3.5 and 3.3.6, if the formation of a light blue halo around the drop is not observed the sample meets the specification. If the formation of a light blue halo around the drop is observed as shown in Figure 1 and confirmed by performing Section 3.3.9, the sample fails the specification.

3.3.5 Remove a drop of the slurry using the glass stirring rod and place it on the filter paper.

3.3.6 Observe the appearance of the drop on the filter paper.

3.3.7 When the formation of a light blue halo around the drop is observed as shown in Figure 1, proceed to Section 3.3.9.

3.3.8 If the formation of a light blue halo around the drop is not observed, repeat Section 3.3.4 to Section 3.3.7.

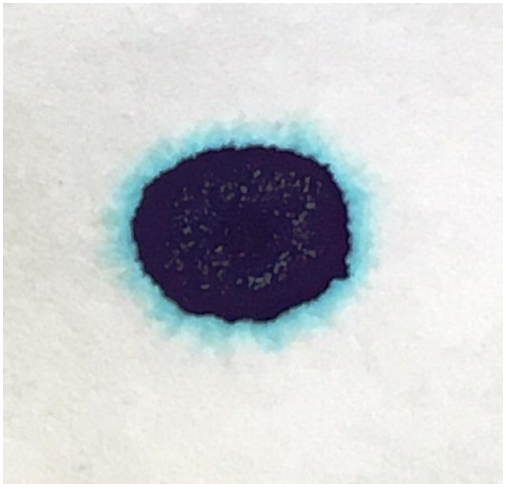

Figure 1. Photograph of Light Blue Halo

3.3.9 After observing the blue halo, continue stirring the slurry for 5 min. and verify the formation of the light blue halo by repeating Section 3.3.5 to Section 3.3.6.

3.3.9.1 If the light blue halo appears again, the test is complete. Record the final volume. Proceed to Section 3.3.10.

3.3.9.2 If the light blue halo does not appear, repeat Section 3.3.4 to Section 3.3.9 until the light blue halo is observed. Record the final volume. Proceed to Section 3.3.10.

3.3.10 Subtract the initial volume from the final volume to determine the volume of methylene blue solution. Record as V.

3.3.11 Repeat Section 3.3 for one additional sample.

4. CALCULATION

4.1 $MBV = (5 \times V)/W$

Where:

MBV = Methylene Blue Value (MBV) in mg of solution per g (mg/g) of the sample.

V = mL of Methylene Blue solution required for titration.

W = Weight of oven dried sample.

5. REPORT

5.1 Report the following:

5.1.1 Type and source of the material tested, and

5.1.2 Average Methylene Blue Value of two tests to the nearest 0.1 mg/g.