Test Procedure for

DETERMINING THE PRESENCE OF HARMFUL CLAYS USING METHYLENE BLUE

TxDOT Designation: Tex-252-F

Effective Date: November 2019 – June 2021

1. SCOPE

1.1 The Methylene Blue 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 M 231, Class G1.

2.2 *Buret,* minimum capacity of 50-mL with 0.1-mL graduations.

2.3 *Distilled water.*

2.4 *Filter paper,* Whatman No. 2

2.5 *Glass stirring rod.*

2.6 *Griffin Beaker,* 500 mL.

2.7 *Magnetic mixer with stir bar.*

2.8 *Methylene Blue,* reagent grade-dated stored in a dark cabinet at lab temperature.

2.9 *Methylene Blue Solution,* stored in an amber bottle in a dark cabinet at lab temperature for no more than four months.

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

2.11 *Pans or containers,* capable of holding water and fines.

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

2.13 *Timer* or *stopwatch.*

2.14 *Volumetric flask,* 1,000-mL.

3. PROCEDURE

3.1 *Preparing Test Sample*

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

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

Note 1—Avoid using metal pans that are prone to rust.

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.

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 to collect the wash water and fines.

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 and water passing the No. 200 (75 µm) sieve in a rust resistant container.

3.1.9 Discard the material on the No. 8 (2.36 mm) and No. 200 (75 µm) sieves.

3.1.10 Allow the material to settle and decant excess water.

3.1.11 Dry the minus No. 200 (75 µm) material to constant mass in the oven at 230 ± 9°F (110 ± 5°C).

Note 2—Section 3.3 requires an oven-dried sample of at least 20 g.

3.2 *Preparing Methylene Blue Reagent*

3.2.1 Dissolve 1,000 mg (1.000 g) of Methylene Blue in distilled water to produce 200 mL of solution, with each 1 mL of solution containing 5 mg of Methylene Blue. Stir the solution with a mixer for at least 1 hr.

Note 3—Solution must not be stored for more than four mo. Solution must be stored in an amber bottle in a dark cabinet at lab temperature.

Note 4—It is recommended small quantities (1L) of Methylene Blue solution are produced at a time to avoid having to dispose of excess solution.

3.3 *Determining Presence of Harmful Clays Using Methylene Blue*

3.3.1 Place 10.0 ± 0.05 g of the sample from Section 3.2.1 into a 500-mL beaker.

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

3.3.3 With the slurry is mixing, fill the buret with the Methylene Blue solution.

3.3.4 Add 0.5 mL of the Methylene Blue solution to the slurry and stir for one min.

Note 5—The initial increment of Methylene Blue solution may be increased to 12 mL to accelerate the test. If the light blue halo is observed as shown in Figure 1, discard the sample and repeat the test beginning with 0.5 mL increments as described in Section 3.3.4.

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.

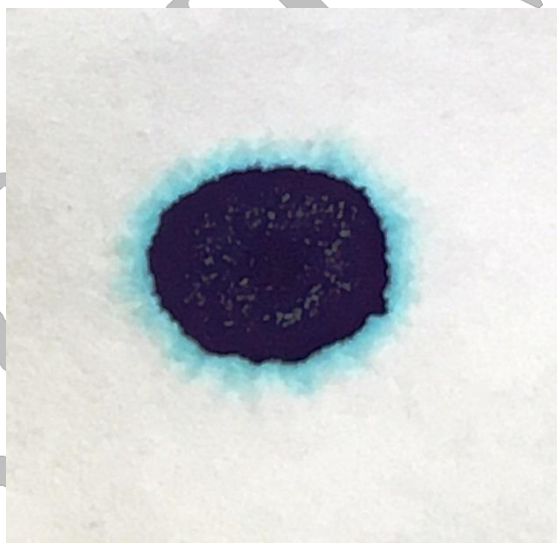

Figure 1. Photograph of Light Blue Halo

3.3.9 Continue stirring for five min. and verify the formation of the light blue halo by repeating Section 3.3.5 to Section 3.3.6.

3.3.10 If the light blue halo appears again, the test is complete. Record the volume (in mL) of Methylene Blue solution.

3.3.11 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 volume (in mL) of Methylene Blue solution.

3.3.12 Repeat Section 3.3 for one additional sample.

4. CALCULATION

4.1 $M = (5 \times V)/10$

$M = 0.5V$

Where:
M = Methylene Blue Value in mg of solution per g of the sample
V = mL of Methylene Blue solution required for titration

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 (M) of the two tests to the nearest 0.1 mg/g.