

The Qualitative Detection of Harmful Clays of the Smectite Group in Aggregates Using Methylene Blue



AASHTO Designation: T 330-07¹

1. SCOPE

- 1.1. The purpose of this standard is to identify the presence of harmful clays of the smectite group (poor minus 75- μ m material) and to provide an indication of the surface activity of the aggregate.
- 1.2. *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns associated with its use. It is the responsibility of the user of this procedure to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.*

2. REFERENCED DOCUMENTS

- 2.1. *AASHTO Standard:*
- M 231, Weighing Devices Used in the Testing of Materials

3. SUMMARY OF TEST METHOD

- 3.1. Methylene Blue solution is titrated in increments into distilled water containing sample material passing the 75- μ m (No. 200) sieve in increments. A small amount of water containing the sample material and titrated Methylene Blue is removed via a glass rod and dropped onto filter paper. When the clay fraction of the sample aggregate can no longer absorb more Methylene Blue, a blue ring is formed on the filter paper.

4. SIGNIFICANCE AND USE

- 4.1. The Methylene Blue Value determined by this standard can be used to estimate the amount of harmful clays and organic matter present in an aggregate. A high value for Methylene Blue Value indicates a large amount of clay or organic material present in the sample.

Note 1—Research (Aschenbrener, 1992) has developed the following relationship of methylene blue values and anticipated hot mix asphalt pavement performance as related to moisture susceptibility. (See Table 1.)

Table 1—Expected Performance of Methylene Blue

Methylene Blue (mg/g)	Expected Performance
≤6	Excellent
7–12	Marginally acceptable
13–19	Problems/possible failures
≥20	Failure

5. APPARATUS

- 5.1. Amber-colored burette of at least 50-mL capacity with 0.1-mL graduations.
- 5.2. Magnetic mixer with stir bar.
- 5.3. Balance complying with M 231, Class G1.
- 5.4. Glass rod of approximately 250-mm (10-in.) length and approximately 8-mm (0.3-in.) diameter.
- 5.5. Timer or stopwatch.
- 5.6. Pan and 75- μ m (No. 200) sieve.
- 5.7. Volumetric Flask of 1000-mL capacity.
- 5.8. Whatman No. 2 filter paper.
- 5.9. Three 500-mL griffin beakers.
- 5.10. Methylene Blue, reagent grade-dated and stored for no more than four months in a brown bottle wrapped with foil in a dark cabinet at lab temperature.

Note 2—One gram of Methylene Blue is dissolved in enough distilled water to produce 200 mL of solution, with each one mL of solution containing 5 mg of Methylene Blue.
- 5.11. Distilled water at lab temperature.
- 5.12. Oven capable of maintaining $110 \pm 5^\circ\text{C}$ ($230 \pm 9^\circ\text{F}$).

Note 3—For the purposes of this procedure, lab temperature is defined as $15\text{--}25^\circ\text{C}$ ($60\text{--}77^\circ\text{F}$).

6. SAMPLE PREPARATION

- 6.1. This test shall be performed on a sample of material passing the 75- μ m (No. 200) sieve, taken from the washed portion of a representative sample of individual or combined material (as required). The material shall be washed through the 75- μ m (No. 200) sieve and collected in a clean container. This container shall be large enough to hold several gallons of water and material passing the 75- μ m (No. 200) sieve. Allow the material to settle and remove the excess water. The portion passing the 75- μ m (No. 200) sieve is collected, transferred to a smaller container, and dried for testing. The collected material is mixed thoroughly prior to testing. The minimum oven-

dry mass of the collected material shall be at least 30 g to ensure enough material for a possible retest.

7. PROCEDURE

- 7.1. Place 10.0 g (± 0.05 g) of the minus 75- μ m (No. 200) material that has been dried to constant mass in a 500-mL griffin beaker.
- 7.2. Add 30 mL of distilled water and stir with the mixer to make a slurry.
- 7.3. With the slurry still mixing, fill the burette with the Methylene Blue solution, add 0.5 mL of the solution to the slurry, and stir for one minute.
- 7.4. Remove a drop of the slurry, using the glass stirring rod, and place on the filter paper.
- 7.5. Observe the appearance of the drop on the filter paper. The end point is indicated by the formation of a light blue halo around the drop. Continue adding the Methylene Blue solution to the slurry in 0.5-mL increments with one-minute stirring after each addition, then testing, until the end point is reached.
- 7.6. After the end point is reached, continue stirring for five minutes and retest.

Note 4—With experience, the person performing the test can reach the end point more quickly by skipping early increments.

8. CALCULATION

- 8.1. *Example:*

$$M = CV/W \quad (1)$$

where:

- M = Methylene Blue Value in mg of solution per g of the minus 75- μ m (No. 200) material;
 C = mg of Methylene Blue/mL of solution;
 V = mL of Methylene Blue solution required for titration; and
 W = grams of dry material.

- 8.2. The calculation may be simplified by inserting the mg of Methylene Blue per mL of solution and the grams of dry material.

$$M = (5 \times V)/10 \quad (2)$$

$$M = 0.5V \quad (3)$$

9. REPORT

- 9.1. *The report shall include the following:*
- 9.1.1. Type and source of the material tested; and
- 9.1.2. Methylene Blue value to the nearest 0.1 mg/g.

10. PRECISION AND BIAS

- 10.1. *Precision*—No precision has been established for this test.
- 10.2. *Bias*—No bias can be established because no reference material is available for this test.

11. KEYWORDS

- 11.1. Clay and organic material; fine aggregate; methylene blue; moisture susceptibility.

12. REFERENCES

- 12.1. Aschenbrener, T. Comparison of Colorado Component Hot Mix Asphalt Materials with some European Specifications. In *Research Report CDOT-DTD-R-92-14*. Colorado Department of Transportation, Denver, CO, December 1992, p. 65.
- 12.2. Aschenbrener, T. and R. Zamora. Evaluation of Specialized Tests for Aggregates Used in Hot Mix Asphalt Pavements in Colorado. In *Transportation Research Record 1486*. Transportation Research Board Business Office, Washington, DC, January 1995, pp. 130–136.
- 12.3. Aschenbrener, T., R. Terrel, and R. Zamora. Comparison of the Hamburg Wheel Tracking Device and the Environmental Conditioning System to Pavements of Known Stripping Performance. In *Research Report CDOT-DTD-R-94-1*. Colorado Department of Transportation, Denver, CO, January 1994, p. 110.

¹ Formerly TP 57.