Designation: C1777 – 15

Standard Test Method for Rapid Determination of the Methylene Blue Value for Fine Aggregate or Mineral Filler Using a Colorimeter[1]

This standard is issued under the fixed designation C1777; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides a rapid test to determine the amount of methylene blue adsorbed by a specimen of fine aggregate or mineral filler and can be used both in the laboratory and in the field.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

C125 Terminology Relating to Concrete and Concrete Aggregates

C702 Practice for Reducing Samples of Aggregate to Testing Size

D75 Practice for Sampling Aggregates

E11 Specification for Woven Wire Test Sieve Cloth and Test Sieves

2.2 *Other Standards:*

AASHTO T330 Standard Method of Test for the Qualitative Detection of Harmful Clays of the Smectite Group in Aggregates Using Methylene Blue[3]

EN 933-9 Tests for geometrical properties of aggregates. Part 9: Assessment of fines – Methylene blue test[4]

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in this standard, refer to Terminology C125.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *mineral filler, n*—a finely divided mineral product at least 65 % of which passes the 75-µm sieve.

4. Summary of Test Method

4.1 A specimen of fine aggregate or mineral filler is combined with a methylene blue solution of known concentration and mixed for a prescribed period of time. The specimen adsorbs some of the methylene blue from solution. The resulting mixture is filtered and a portion of the filtered solution is diluted by a fixed amount. A colorimeter is used to determine the absorbance of the diluted solution, from which the concentration of methylene blue prior to dilution is calculated. The change in concentration of methylene blue before and after mixing with fine aggregate or mineral filler is converted to a methylene blue value and reported in units of mg/g.

5. Significance and Use

5.1 This test method is used to determine rapidly the amount of methylene blue adsorbed by a specimen of fine aggregate or mineral filler. The result is reported as a methylene blue value in units of mg of methylene blue adsorbed per g of fine aggregate or mineral filler. The methylene blue value is a function of the amount and characteristics of clay minerals present in the test specimen. High methylene blue values indicate increased potential for diminished fine aggregate or mineral filler performance in a cementitious mixture due to the presence of clays.

NOTE 1—Results from this test method are not expected to be correlated with those obtained using AASHTO T 330 or EN 933-9. These three test methods are likely to give very different numerical values even though the units are the same. The AASHTO T 330 test is performed only on the fraction of an aggregate passing the 75 µm sieve, the EN 933-9 test is

[1] This test method is under the jurisdiction of ASTM Committee C09 on Concrete and Concrete Aggregates and is the direct responsibility of Subcommittee C09.20 on Normal Weight Aggregates.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American Association of State Highway and Transportation Officials (AASHTO), 444 N. Capitol St., NW, Suite 249, Washington, DC 20001, http://www.transportation.org.

[4] Available from European Committee for Standardization, Avenue Marnix 17, B-1000 Brussels, Belgium.

performed only on the fraction finer than 2 mm, and this test is performed on the fraction finer than the 4.75 mm sieve. Therefore, when testing the same fine aggregate source, the AASHTO test method would give the highest methylene blue value because any clay present in the specimen would be concentrated in the fraction finer than the 75 µm sieve. The AASHTO and EN test methods do not take into account the amount of material passing the 75 µm sieve or 2 mm sieve, respectively. For example, a fine aggregate with high methylene blue value measured by the AASHTO method but low percentage passing the 75 µm sieve may have less effect on concrete performance than a fine aggregate with low methylene blue value measured by the AASHTO method but a high percentage passing the 75 µm sieve. In contrast, this test method expresses methylene blue value based on the entire fine aggregate or mineral filler specimen. Additionally, the AASHTO and EN test methods use titration to determine the amount of methylene blue of known concentration that is adsorbed by a specimen and require the operator to visually determine the end point of the test. In contrast, this test method uses a colorimeter to detect the change in concentration of methylene blue solution before and after being mixed with the fine aggregate or mineral filler specimen.

NOTE 2—Recommendations for maximum methylene blue values for specific applications are not provided in this test method. Maximum methylene blue values should be established based on successful performance of fine aggregate or mineral filler in the applications under consideration.

6. Interferences

6.1 Methylene blue will degrade when exposed to light. Store in darkness. No appreciable degradation occurs during the time it takes to complete the test method.

6.2 Methylene blue will stain glassware and plastic ware. Therefore, do not reuse such apparatus.

7. Apparatus

7.1 *4.75-mm sieve* conforming to E11.

7.2 *Mass Balance* having a capacity of 50 g or more and capable of measuring to the nearest 0.1 g or less.

7.3 *Micropipette* capable of measuring to the nearest 1 µL.

7.4 *Colorimeter* capable of reading absorbance of a specimen at a wavelength of 610 ± 1 nm at operating temperatures of at least 0 to 50ºC. The colorimeter shall be able to read absorbance between zero and the absorbance associated with a 0.144 % mass concentration of methylene blue solution.

7.5 *Disposable items for each test*—two plastic 50-mL test tubes, one plastic 1-mL vial, one 3-mL syringe with Luer-Lok adapter, one 0.2-µm syringe filter, one colorimeter glass cuvette (or sample cell), one micropipette tip, and two transfer pipettes.

7.6 *Additional disposable items for confirming methylene blue starting concentration*—plastic 50-mL test tube, colorimeter glass cuvette, micropipette tip, and transfer pipette.

7.7 *Additional disposable items for standardizing the colorimeter*—plastic 50-mL test tube, colorimeter glass cuvette, micropipette tip, and transfer pipette.

7.8 *Drying Apparatus*—A ventilated oven capable of maintaining a uniform temperature of 110 ± 5°C. Other suitable drying apparatuses shall be permitted, such as an electric hot plate or heat lamp. The temperature of the specimen shall not exceed 150°C. In cases where the fine aggregate or mineral filler itself is altered by temperature greater than 115°C, use a ventilated, controlled-temperature oven at 110 ± 5°C.

NOTE 3—Drying by means other than a ventilated oven may be appropriate for field use.

8. Reagents and Materials

8.1 *Purity of Reagents*—reagent grade methylene blue shall be used in all tests.

8.2 *Purity of water*—references to water shall be understood to mean distilled or deionized water.

8.3 *Methylene blue test solution*—a 0.50 % mass concentration methylene blue solution based on mass of trihydrate methylene blue in water.

NOTE 4—Methylene blue is available in both anhydrous and trihydrate form and can also be obtained in solution form. This test is based on the mass of the trihydrate form.

9. Sampling, Test Specimens, and Test Units

9.1 Sample fine aggregate in accordance to Practice D75.

9.2 Thoroughly mix the sample and reduce it as necessary using the applicable procedures in Practice C702.

9.3 If it appears necessary, dampen the material to avoid segregation or loss of fines during specimen preparation.

9.4 Obtain at least 30 g of material passing the 4.75-mm sieve in the following manner:

9.4.1 Separate the sample on the 4.75-mm sieve by means of a lateral and vertical motion of the sieve, accompanied by a jarring action so as to keep the sample moving continuously over the surface of the sieve. Continue the sieving until not more than 1 mass % of the residue passes the sieve during the 1-min sieving operation. Perform the sieving operation either by hand or by a mechanical apparatus. When thoroughness of mechanical sieving is being determined, test by the hand method described above using a single layer of material on the sieve.

9.4.2 Break down any lumps of material in the coarse fraction to pass the 4.75-mm sieve. Use a mortar and rubber-covered pestle or any other means that will not fracture aggregate particles. Add this additional material passing the sieve to the separated fine portion of the sample and mix thoroughly.

9.5 Dry the test specimen to constant mass by means of the selected source of heat, and cool to room temperature before testing. The sample is thoroughly dry when further heating causes, or would cause, less than 0.1 g additional loss in mass.

9.6 Repeat the procedures in 9.4 and 9.5 to obtain three test specimens.

10. Standardization

10.1 *Standardization of the colorimeter for the relationship between absorbance and the methylene blue concentration*—Insert a glass cuvette approximately 2/3 full with water into the colorimeter and zero the instrument. Use the micropipette to transfer a 130 ± 1 µL aliquot of 0.50 % mass methylene blue solution to a 50-mL test tube. Dilute the aliquot with water so that the net mass of the diluted solution is 45.0 ± 0.1 g. Place a cap on the test tube and gently shake the diluted solution for 5 ± 1 s. Using a new transfer pipette, fill a glass colorimeter

cuvette approximately 2/3 full with the diluted solution. Wipe the cuvette with a clean towel if necessary to remove any marks. Insert the cuvette with the diluted methylene blue solution into the colorimeter and measure the absorbance. Rotate the cuvette within the meter a quarter revolution and take another measurement. Repeat until four measurements are made. Calculate the average of the four values, and record as A_{std} to the nearest 0.01 A. Perform this standardization for each colorimeter at least once every 6 months or whenever the light source or batteries are replaced (if applicable). Use a freshly-made 0.5 % mass methylene blue test solution for standardization of the colorimeter.

10.2 *Determination of actual initial methylene blue concentration*—Before testing the fine aggregate or mineral filler, determine the actual initial concentration of methylene blue test solution that will be used. Insert a cuvette approximately 2/3 filled with water into the colorimeter and zero the instrument. Use the micropipette to transfer a 130 ± 1 µL aliquot of the methylene blue solution to a 50-mL test tube. Dilute the aliquot with water so that the net mass of the diluted solution is 45.0 ± 0.1 g. Cap the test tube and gently shake the diluted solution for 5 ± 1 s. Follow the procedure in 10.1 to obtain four values of absorbance of the diluted test solution. Calculate the average of the four values, and record as A_i. Determine the actual initial concentration of the test solution, C_i, prior to dilution, using the following equation:

$$C_i = (0.50\ \%) \times \frac{A_i}{A_{std}} \quad (1)$$

Repeat the process with two more aliquots. Calculate the average of the three values and record this as the average initial concentration of methylene blue in the test solution to the nearest 0.01 %. Perform this determination of actual initial concentration each day or whenever a new source or batch of methylene blue test solution is used.

NOTE 5—10.2 is conducted to ensure the methylene blue solution to be used in the test is at the correct initial concentration.

10.3 *Adjustment of initial concentration of methylene blue solution*—If the actual initial concentration is below 0.48 %, discard and prepare a new test solution. If the actual initial concentration is greater than 0.50 %, add sufficient water to adjust to 0.50 %.

11. Procedure

11.1 *Test Specimens*—Weigh 20.0 ± 0.1 g of dry fine aggregate or mineral filler as obtained in Section 9 and record the actual mass of the specimen. Tare a 50-ml test tube, weigh 30.0 ± 0.1 g of methylene blue test solution directly into the test tube, and record the actual mass of solution. Add the weighed aggregate to the methylene blue solution, ensuring all fines are incorporated.

11.2 *Mixing*—Cap the test tube and shake the mixture by hand for 60 ± 1 s and allow to rest for 180 ± 5 s. Shake the mixture again for 60 ± 1 s to complete the mixing process.

11.3 *Filtration*—Remove the plunger from the 3-mL-syringe and attach the 0.2-µm syringe filter. Using a transfer pipette, add approximately 2 mL of the specimen mixture to the syringe and replace the plunger. Push the plunger slowly until 0.5 to 1 mL of the filtered solution is collected in a new 1-mL vial.

11.4 *Dilution*—Using the micropipette, transfer 130 ± 1 µL of the filtered solution into a new 50-mL test tube. Dilute the filtered solution with water until the net mass is 45.0 ± 0.1 g. Cap the 50-mL test tube and gently shake the diluted solution for 5 ± 1 s. Using a new transfer pipette, fill a glass colorimeter cuvette approximately 2/3 full with the diluted solution.

11.5 *Colorimeter zero adjustment*—Place a cuvette filled approximately 2/3 with water into the colorimeter and zero the instrument.

11.6 *Absorbance measurement*—Remove the cuvette filled with water and replace with the cuvette filled with the diluted solution. Measure and record the absorbance to the nearest 0.01 A. Rotate the cuvette within the colorimeter a quarter revolution and take another measurement. Repeat a total of four times and calculate the average of the four values; record as A_f.

11.7 *Calculation of undiluted final methylene blue concentration*—Using the average value of A_f from 11.6, calculate the final methylene blue concentration, before dilution, to the nearest 0.01 % using the following equation:

$$C_f = (0.50\ \%) \times \frac{A_f}{A_{std}} \quad (2)$$

11.8 *Replicate testing*—Repeat steps 11.1 through 11.7 on two additional specimens.

12. Calculation of Methylene Blue Value

12.1 For each determination of the final concentration of methylene blue solution, determine the methylene blue value (MBV) in units of mg/g by using the following equation:

$$MBV = \frac{(C_i - C_f)(M_{MB})}{M_{FM}} \times 10 \quad (3)$$

where:

C_i = average actual initial concentration of methylene blue test solution in percent as determined in 10.2.
C_f = final concentration of methylene blue solution in percent as determined in 11.7.
M_{MB} = mass of methylene blue test solution as measured in 11.1.
M_{FM} = mass of fine aggregate or mineral filler as measured in 11.1.

12.2 Calculate the methylene blue value as the average of three individual test determinations from 12.1.

12.3 If the methylene blue value is greater than or equal to 7.5 mg/g when using 20.0 ± 0.1 g of fine aggregate or mineral filler, record the methylene blue value as "greater than 7.5 mg/g."

12.4 It is permitted to repeat the procedure in Section 11 using only 10.0 ± 0.1 g of fine aggregate or mineral filler. If the methylene blue value is greater than or equal to 15 mg/g when using 10.0 ± 0.1 g of fine aggregate or mineral filler, record the methylene blue value as "greater than 15 mg/g."

NOTE 6—If the test is performed with 10.0 ± 0.1 g of fine aggregate or

mineral filler, the sensitivity of the measurement will be reduced.

13. Report

13.1 Report the following information:

13.1.1 Source of fine aggregate or mineral filler,

13.1.2 Actual initial concentration of methylene blue test solution to the nearest 0.01 %,

13.1.3 Date of test,

13.1.4 Date when test solution was prepared,

13.1.5 Mass of fine aggregate or mineral filler used to the nearest 0.1 g, and

13.1.6 The methylene blue value to the nearest 0.1 mg/g.

14. Precision and Bias

14.1 *Precision*—A complete interlaboratory study has not been completed for this test method. Therefore, the precision of the method has not been determined.

NOTE 7—A preliminary estimate of within-laboratory precision was determined from test results on a single material tested in a single laboratory. The material was a field fine aggregate. Eight test results were generated, each a mean of 3 determinations, as directed in the test method. The average test result was 2.04 mg/g. The standard deviation of a test result, defined as the average of three individual determinations, was 0.08 mg/g.

14.2 *Bias*—No statement on bias is presented because no reference material having an accepted methylene blue value is available.

15. Keywords

15.1 clay ; fine aggregate; limestone filler; methylene blue; mineral filler