Designation: C 837 – 99

Standard Test Method for Methylene Blue Index of Clay[1]

This standard is issued under the fixed designation C 837; 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 covers the measurement of the adsorption of methylene blue dye by a clay, which is calculated as a methylene blue index for a clay.

1.2 *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:*

C 324 Test Method for Free Moisture in Ceramic Whiteware Clays[2]

3. Significance and Use

3.1 Tests run on many clays generally indicate that a straight-line relationship exists between the methylene blue index (MBI) and such fundamental clay properties as cation exchange capacity, dry bond strength, and casting rate. Where the colloidal portion of the clay is kaolinite, there is also a direct correlation with specific surface (as determined by nitrogen adsorption). Where the colloidal portion contains significant amounts of illite or montmorillonite, the same close correlation does not exist. The MBI better correlates with the ceramic-forming properties than does the specific surface.

3.2 That portion of a clay lying within the colloidal range (generally defined as the 0.5- to 0.001-µm range), determines the strictly colloidal properties of the clay and, together with the amount and type of organic material associated with the clay and the 2- to 0.2-µm fraction, largely determines the properties of the clay when used in ceramic-forming processes. While the specific surface of a clay is a function of the particle size and morphology and a relationship exists between dye adsorption and specific surface, the MBI should not be considered to be a particle size analysis since the value obtained is dominated by the character of only the very fine end of the particle size distribution. This procedure describes the determination of the dye adsorption (in this case, methylene blue) of a clay.

4. Apparatus

4.1 *Balance*, accurate to 0.01 g.

4.2 *Mixer.*[3]

4.3 *pH Meter or pH Paper*.

4.4 *Beaker*, 600 mL.

4.5 *Buret*, 25 mL.

4.6 *Medicine Dropper or Glass Stirring Rod*.

4.7 *Filter Paper*, Baroid No. 987.[4]

5. Reagents

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

5.3 *Methylene Blue Solution (1 mL = 0.01 meq)*[4]—Store in darkness.

5.4 *Sulfuric Acid (0.1N)*.

6. Procedure

6.1 Weigh out 2.00 g of clay that has been dried in accordance with the procedure in Test Method C 324 and place in the 600-mL beaker. If the clay cannot be tested immediately after drying, it should be stored in a desiccator.

6.2 Add 300 mL of distilled water to the beaker and stir with

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[1] This test method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C 21.04on Clays.

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[2] *Annual Book of ASTM Standards*, Vol 15.02.

[3] This test method is based on the use of the Model F Lightnin mixer, which is available from Mixing Equipment Co., Inc., Rochester, NY. However, it has been found that some clays are not completely dispersed in this apparatus, and so results may be spurious. It has been found that the Waring Blender, available from Waring Products Division, Dynamics Corporation of America, New Hartford, CT 06057, is successful in dispersing all clays.

[4] Available from Baroid Div., National Lead Co., No. 425-15.

[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

the mixer until the clay is uniformly dispersed.

6.3 Determine the pH of the slurry and add sufficient sulfuric acid to bring the pH within the range from 2.5 to 3.8. Continue stirring while the pH is being adjusted and continue stirring for 10 to 15 min after the last addition of acid.

6.4 Again test the slurry for pH, adding additional acid if necessary to restore the pH to the 2.5 to 3.8 range.

6.5 With the slurry still under the mixer, fill the buret with the methylene blue solution, add 5 ml of the solution to the slurry, and stir for 1 to 2 min.

6.6 Remove a drop of the slurry, using the dropper or the glass stirring rod, and place on the edge of the filter paper.

6.7 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 1.0-mL increments with 1 to 2 min of stirring after each addition, then testing, until the end point is reached. For ball clays with relatively high methylene blue indexes, testing may start after two or even three 5-mL additions have been made to save time. Allow 1 to 2 min of stirring after each 5-mL increment.

6.8 After the end point is reached, continue stirring for 2 min and retest.

NOTE 1—Greater precision can be obtained by using larger samples with only minor influence on the value obtained.

7. Calculation

7.1 Calculate the methylene blue index as follows:

$$\text{MBI} = \frac{E \times V}{W} \times 100$$

where:

MBI = methylene blue index for the clay in meq/100 g clay,
E = milliequivalents of methylene blue per millilitre (see 5.3),
V = millilitres of methylene blue solution required for the titration, and
W = grams of dry material.

7.2 The calculations may be facilitated by using a multiplication factor where the specimen size is 2.00 g and the methylene blue titrating solution is 0.01*N*:

$$\text{MBI} = \frac{0.01 \times V}{2} \times 100 = 0.5\,V$$

7.3 Record the methylene blue index for the clay.

8. Precision and Bias

8.1 The precision of this test method is the calculated methylene blue index (MBI) ± 0.25 meq/100 g clay. The MBI value cannot be directly related to any single, simply measured, characteristic of the clay; thus the bias of this test method cannot be specified.