Designation: D 1512 – 95 (Reapproved 2000)

Standard Test Methods for Carbon Black—pH Value[1]

This standard is issued under the fixed designation D 1512; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods, Test Method A—Boiling Slurry and Test Method B—Sonic Slurry, are used to indicate the pH of the carbon black surface by measuring the pH of water in contact with the carbon black.

NOTE 1—The pH of the carbon black is often used in this industry to indicate the relative acidity or alkalinity of carbon black and will be used in the remainder of these test methods to describe this property.

NOTE 2—Test Method A and Test Method B do not always give the same results.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

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:*

D 1193 Specification for Reagent Water[2]

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[3]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[3]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]

E 70 Test Method for pH of Aqueous Solutions with the Glass Electrode[4]

3. Significance and Use

3.1 The pH level of a carbon black is known to affect the vulcanization of some rubber compounds.

TEST METHOD A—BOILING SLURRY

4. Apparatus

4.1 *pH Meter*, (digital is recommended) having an accuracy of ±0.05 pH and equipped with a combination electrode and RNC connector.

4.2 *Container*, stainless steel or copper, 125 cm^3 or larger.

4.3 *Hot Plate*.

4.4 *High Speed Mill, Mixer or Mortar and Pestle*.

4.5 *Beakers*, glass, 100 cm^3 graduated with watch glasses.

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 reagent water as defined by Type 1 in Specification D 1193.

5.3 *Distilled Water*, high purity.

5.4 *Buffer Solutions*, pH of 4.00, 7.00, and 10.00.

5.5 *Acetone*, reagent grade.

6. Sampling

6.1 Samples shall be taken in accordance with Practices D 1799 or D 1900.

7. Procedure

7.1 Pulverize pelleted or lumpy carbon black to a fine powder, using either the high speed mixer or mortar and pestle.

7.2 Weigh 5 g of carbon black into a 100 cm^3 glass beaker.

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[1] These test methods are under the jurisdiction of ASTM Committee D24 on Carbon Black and are the direct responsibility of Subcommittee D24.31 on Non-Carbon Black Components of Carbon Black.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 09.01.

[4] *Annual Book of ASTM Standards*, Vol 15.05.

[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.

7.3 Add 50 cm^3 of boiling, distilled water prepared in a stainless steel beaker and 2 to 3 drops of acetone to facilitate wetting of the sample.

NOTE 3—A stainless steel beaker is used to eliminate contamination during boiling.

7.4 Cover the glass beaker with a watch glass and boil the mixture for 15 min, but do not allow all the liquid to evaporate.

7.5 Let the mixture cool to room temperature in an atmosphere free from chemical fumes which might contaminate the sample.

7.6 Standardize the pH meter with the buffer solutions. Rinse the electrode with distilled water and wipe clean after each test.

7.7 Place the electrode in the sludge, rotate gently in alternate directions until a constant pH is obtained, and record the pH to the nearest 0.05 unit.

NOTE 4—Refer to Test Method E 70 for a definition of pH and a highly detailed procedure for making pH measurements.

7.8 Rinse the electrode with distilled water and wipe clean. Keep the electrode soaking in distilled water when not in use.

8. Report

8.1 Report the following information:

8.1.1 Proper identification of the sample,

8.1.2 Result obtained, reported to the nearest 0.05 unit, and

8.1.3 Test Method used, A or B.

9. Precision and Bias

9.1 *Test Method A*:

9.1.1 This precision and bias has been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical details.

9.1.2 The precision results in this precision and bias give an estimate of the precision as described in the following paragraphs. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

9.1.3 A Type 1 interlaboratory precision program was conducted in 1990. Both repeatability and reproducibility represent short term testing conditions. Eight laboratories tested four carbon blacks (Samples A through D), twice on two different days. A test result is the value obtained from a single determination. Acceptable difference values were not measured.

9.1.4 The results of the precision calculations are given in Table 1 with the materials arranged in ascending mean level order of the pH value.

9.1.5 The precision for the pooled values for pH value may be expressed as follows:

9.1.5.1 *Repeatability*— The repeatability, *r*, of the pH value has been established as 0.5 pH units. Two single test results (or determinations) that differ by more than 0.5 pH units must be considered suspect and dictates that some appropriate investigative action be taken.

9.1.5.2 *Reproducibility*— The reproducibility, *R*, of the pH value has been established as 1.53 pH units. Two single test results (or determinations) produced in separate laboratories that differ by more than 1.53 pH units must be considered as suspect and dictates that appropriate investigative or technical/commercial actions be taken.

9.2 *Bias*—In test method terminology, bias is the difference between average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

TEST METHOD B—SONIC SLURRY

10. Apparatus

10.1 *pH Meter*, (digital is recommended) having an accuracy of ± 0.05 pH and equipped with a combination electrode and RNC connector.

10.2 *Container*, stainless steel or copper, 125 cm^3 or larger.

10.3 *Ultrasonic Stirring Bath*[6] , two-position.

10.4 *Magnetic Spinbars*, 4.8 mm (3⁄16 in.) or 6.4 mm (¼ in.) by 22.4 mm (⅞ in.) long, coated with a fluorocarbon polymer, such as TFE-fluorocarbon.

10.5 *Beakers*, glass, 30 cm^3 graduated with watch glasses.

11. Reagents

11.1 *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.

11.2 *Purity of Water*— Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 in Specification D 1193.

11.3 *Distilled Water*, high-purity.

11.4 *Buffer Solutions*, pH of 4.00, 7.00, and 10.00.

11.5 *Acetone*, reagent grade.

12. Sampling

12.1 Samples shall be taken in accordance with Practices D 1799 or D 1900.

13. Procedure

13.1 Weigh 1.5 g of carbon black into a 30 cm^3 beaker.

13.2 Insert a magnetic spinbar into the beaker and add 20 cm^3 of distilled water and 2 to 3 drops of acetone to aid dispersion.

NOTE 5—The water should be boiled in a stainless steel beaker and cooled prior to use to remove dissolved carbon dioxide.

13.3 Cover the beaker with a watch glass and insert it into the ultrasonic bath which contains water to a depth of 40 mm that is 5 to 10°C below ambient temperature.

[6] A unit which has been found suitable for this test is available upon custom order from Micro-Star 2000, Inc., 255 Bradwick Dr., Unit 21, Concord, Ontario, Canada L4K 1K7.