



Designation: D1512 – 23

## Standard Test Methods for Carbon Black—pH Value<sup>1</sup>

This standard is issued under the fixed designation D1512; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

*This standard has been approved for use by agencies of the U.S. 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D1193 Specification for Reagent Water

D1799 Practice for Carbon Black—Sampling Packaged Shipments

D1900 Practice for Carbon Black—Sampling Bulk Shipments

D4483 Practice for Evaluating Precision for Test Method

<sup>1</sup> 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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<sup>2</sup> 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.

Standards in the Rubber and Carbon Black Manufacturing Industries

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

### 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  $\pm 0.05$  pH and equipped with a combination electrode and RNC connector.

4.2 *Container*, stainless steel or copper, 125 cm<sup>3</sup> or larger.

4.3 *Hot Plate*.

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

4.5 *Beakers*, glass, appropriate beaker size to keep the ratio of Carbon Black to water 1:10, graduated with watch glasses.

4.6 *Magnetic Stir Plate*.

4.7 *Magnetic Stir Bars*.

### 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.<sup>3</sup> 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 D1193.

<sup>3</sup> *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.

**TABLE 1 Carbon Black Weight and Water Volumes**

| Carbon black weight, g | Volume of water cm <sup>3</sup> | Beaker or flask volume, cm <sup>3</sup> |
|------------------------|---------------------------------|---|
| 10                     | 100                             | 200                                     |
| 20                     | 200                             | 250                                     |

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 **D1799** or **D1900**.

## 7. Calibration

7.1 Calibrate the pH meter using buffer solutions according to manufacturer's instructions. Rinse the electrode with distilled water and gently touch the tip of the electrode to remove excessive water with a lint-free paper towel after each test.

## 8. Procedure

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

8.2 Prepare boiling distilled water in a stainless steel beaker.

8.3 Weigh carbon black and boiling distilled water into a glass beaker or boiling flask and add 2 to 3 drops of acetone to facilitate wetting of the sample. The carbon black weight and volume of water is maintained at a constant ratio of 1:10. See **Table 1** for selection of carbon black weight, volume of water and beaker or boiling flask volume.

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

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

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

8.6 Place a magnetic stir bar into the glass beaker and place on magnetic stir plate (or similar mechanical stirring device) and adjust the stir speed to achieve a continuous uniform slurry. Carefully place the pH electrode into the slurry taking care not to allow the electrode to contact the stir bar. Record the pH to the nearest 0.05 units after an equilibration period of 2 min.

NOTE 4—Refer to Test Method **E70** for a definition of pH and a highly detailed procedure for making pH measurements.

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

## 9. Report

9.1 Report the following information:

9.1.1 Proper identification of the sample,

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

9.1.3 Test Method used, A or B.

## 10. Precision and Bias

### 10.1 Test Method A:

10.1.1 These precision statements have been prepared in accordance with Practice **D4483**. Refer to this practice for terminology and other statistical details.

10.1.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials used in the particular interlaboratory program described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method. Any appropriate value may be used from **Table 2**.

10.1.3 A type 1 inter-laboratory precision program was conducted as detailed in **Table 2**. Both repeatability and reproducibility represent short term (daily) testing conditions. The testing was performed using two operators in each laboratory performing the test once on each material on each of two days (total of four tests).

10.1.4 The results of the precision calculations for this test are given in **Table 2**. The materials are arranged in ascending "mean level" order.

10.1.4.1 *Repeatability*—The pooled absolute repeatability,  $r$ , of this test has been established as 0.40 pH units. Any other value in **Table 2** may be used as an estimate of repeatability, as appropriate. The difference between two single test results (or determinations) found on identical test material under the repeatability conditions prescribed for this test will exceed the repeatability on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results that differ by more than the appropriate value from **Table 2** must be suspected of being from different populations and some appropriate action taken.

NOTE 5—Appropriate action may be an investigation of the test method procedure or apparatus for faulty operation or the declaration of a significant difference in the two materials, samples, etc., which generated the two test results.

10.1.4.2 *Reproducibility*—The pooled absolute reproducibility,  $R$ , of this test has been established as 1.65 pH units. Any other value in **Table 2** may be used as an estimate of reproducibility, as appropriate. The difference between two single and independent test results found by two operators working under the prescribed reproducibility conditions in different laboratories on identical test material will exceed the reproducibility on an average of not more than once in 20 cases in the normal and correct operation of the method. Two single test results produced in different laboratories that differ by more than the appropriate value from **Table 2** must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

10.2 *Bias*—In test method terminology, bias is the difference between an 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.