Designation: D1512 – 15b

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

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 (ε) 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 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 ±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.

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.[3] 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.

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.

[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] 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] *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. Calibration

7.1 Calibrate the pH meter using buffer solutions according to manufacturer's instructions.

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

8.7 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. Once a constant pH is obtained record to the nearest 0.05 unit.

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

8.8 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:*

TABLE 1 Carbon Black Weight and Water Volumes

Carbon black weight, g	Volume of water cm^3	Beaker or flask volume, cm^3
10	100	200
20	200	250

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

TABLE 2 Precision Parameters for ASTM D1512, Carbon Black -- pH Value, Method A, (Type 1 Precision)

Units		pH Units					
Material	Period	Number of Laboratories	Mean Level	Sr	r	SR	R
HS Tread	Fall 2003	22	6.2	0.15	0.43	0.73	2.07
SRB A6 (N134)	Fall 2004	23	6.5	0.15	0.41	0.53	1.49
LS Carcass	Spring 2004	26	7.2	0.14	0.39	0.50	1.41
SRB C6 (N326)	Spring 2003	27	8.5	0.11	0.32	0.50	1.41
N774	Fall 2002	19	8.6	0.16	0.45	0.62	1.75
Average			7.4				
Pooled Values				0.14	0.40	0.58	1.65

TABLE 3 Precision Parameters for ASTM D1512, Carbon Black -- pH Value, Method B, (Type 1 Precision)

Units		pH Units					
Material	Period	Number of Laboratories	Mean Level	Sr	r	SR	R
HS Tread	Fall 2003	15	6.8	0.13	0.38	0.49	1.39
SRB A6 (N134)	Fall 2004	18	7.1	0.13	0.37	0.36	1.03
LS Carcass	Spring 2004	16	7.3	0.12	0.35	0.43	1.21
SRB C6 (N326)	Spring 2003	15	8.8	0.10	0.28	0.33	0.93
N774	Fall 2002	8	9.1	0.11	0.30	0.59	1.67
Average			7.8				
Pooled Values				0.12	0.34	0.45	1.27

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.

TEST METHOD B—SONIC SLURRY

11. Apparatus

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

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

11.3 *Ultrasonic Stirring Bath*[4], two-position.

11.4 *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.

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

12. Reagents

12.1 *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.[3] 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.

12.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type 1 in Specification D1193.

12.3 *Distilled Water,* high-purity.

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

12.5 *Acetone,* reagent grade.

13. Sampling

13.1 Samples shall be taken in accordance with Practices D1799 or D1900.

14. Procedure

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

14.2 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 6—The water should be boiled in a stainless steel beaker and cooled prior to use to remove dissolved carbon dioxide.

14.3 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.

NOTE 7—Use small pieces of ice to control bath temperature.

14.4 Adjust the magnetic stirrer to give vigorous stirring by the spinbar, and turn on the sonic power for 3 min to equilibrate the mixture.

NOTE 8—If a combination ultrasonic stirring bath is not available, use alternate periods of 1 min of ultrasonic agitation and 1 min of magnetic spin stirring for a total time of 6 min.

14.5 Remove the beaker from the ultrasonic bath and place it on a magnetic stirrer and adjust the stir speed to achieve a continuous uniform slurry. Carefully place the pH electrode in the slurry taking care not to allow the electrode to contact the stir bar. Measure the pH to the nearest 0.05 units after an equilibration period of 2 min.

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

15. Report

15.1 See Section 9.

16. Precision and Bias[5]

16.1 *Test Method B:*

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

[4] The sole source of supply of the apparatus known to the committee at this time is Micro-Star 2000, Inc., 255 Bradwick Dr., Unit 21, Concord, Ontario, Canada L4K 1K7. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D24-1023.

16.1.2 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 3.

16.1.3 A type 1 inter-laboratory precision program was conducted as detailed in Table 3. 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).

16.1.4 The results of the precision calculations for this test are given in Table 3. The materials are arranged in ascending "mean level" order.

16.1.4.1 *Repeatability*—The pooled absolute repeatability, r, of this test has been established as 0.34 pH units. Any other value in Table 3 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 3 must be suspected of being from different populations and some appropriate action taken.

NOTE 9—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.

16.1.4.2 *Reproducibility*—The pooled absolute reproducibility, R, of this test has been established as 1.27 pH units. Any other value in Table 3 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 3 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

16.2 *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.

17. Keywords

17.1 carbon black; pH