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Standard Test Method for Carbon Black—Tint Strength[1]

This standard is issued under the fixed designation D3265; 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 determination of the tint strength of carbon black relative to an industry tint reference black (ITRB).

1.2 The values stated in SI units are to be regarded as the standard. The values given 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:*[2]

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
D4821 Guide for Carbon Black—Validation of Test Method Precision and Bias

3. Summary of Test Method

3.1 A carbon black sample is mixed with a white powder (zinc oxide) and a liquid vehicle (epoxidized soybean oil, ESO) to produce a black or gray paste. This paste is then spread to produce a surface suitable for measuring the reflectance of the mixture by means of a photo-electric reflectance meter. The reflectance of the tested sample is then compared to the reflectance of the ITRB prepared in the same manner. The tint strength of the tested sample is expressed as units of the reflectance of the ITRB divided by the reflectance of the sample and multiplied by 100. The test value is therefore expressed in %.

3.2 *Correction Factor:*

3.2.1 A correction factor in Eq 3 is needed when raw materials Paraplex G-62 and ZnO Lot#8 or earlier produced before 2012, in combination with ITRB2, are used. For additional details refer to ASTM D3265-17a.

[1] This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.21 on Carbon Black Surface Area and Related Properties.

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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.

3.2.2 There should be no correction applied when ITRB is used with Paraplex G-62 and ZnO Lot#8 or earlier.

3.2.3 There should be no correction applied when using the currently available tint raw materials (see Section 6) for pastes prepared with either ITRB or ITRB2.

4. Significance and Use

4.1 For the broad range of commercial rubber grade carbon blacks, tint strength is highly dependent upon particle size. Tint strength can be used as an indication of particle size; however, tint strength is also dependent on structure and aggregate size distribution. Therefore, differences in tint strength within grades of carbon black may reflect differences other than particle size.

NOTE 1—This test method was developed primarily for the characterization of N100, N200, and N300 series carbon blacks.

4.2 Tint strength values within the carbon black industry have been developed using a Automatic Muller apparatus which is used to prepare carbon black-zinc oxide pastes. An alternative mixing apparatus, Hauschild SpeedMixer[3,4] (DAC 150 FVZ), and a corresponding procedure have been extensively studied within D24 and shown to provide equivalent tint strength for all carcass or soft blacks and most tread blacks with the exception of higher surface area N100 types and specialty blacks. Therefore, it is the responsibility of the user of this alternate apparatus to ensure their products will adequately disperse. Disputes arising between a user and producer should be resolved using the Automatic Muller apparatus until ASTM develops adequate precision statements.

4.3 The term ITRB is used in the entire text for both, the original ITRB, used as the first reference material for tint testing, but which is now used up, and the successor reference material, ITRB2.

5. Apparatus

5.1 *Analytical Balance,* with a sensitivity of ±0.1 mg.

5.2 *Automatic Muller.*[5,4]

5.3 *Photometric Instrument,* capable of detecting differences in the amount of light reflectance between shades of gray.[6,4] The instrument is to be operated following the manufacturer's instructions for optimum performance.

5.4 *Oven, Gravity-Convection Type,* capable of temperature regulation within ±1°C at 125°C (±2°F at 257°F) and temperature uniformity within ±5°C (±9°F).

5.5 *Reflectance Standards,* as required for each reflectance instrument for checking calibration.

5.6 *Spatulas,* 100 to 150 mm (4 to 6 in.), two, flexible, tapered.

5.7 *Syringe,* 5-cm^3, automatic refilling, reproducible to ±0.02 cm^3.

5.8 *Wiping Tissue,* absorbent and lint free.

5.9 *Paste Application Apparatus*—Any one of the following groups of equipment may be used:

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[3] The sole source of supply of the apparatus – Hauschild SpeedMixer®, which has been qualified for this method, including the DAC150 FVZ – known to the committee at this time is Hauschild, www.hauschild-speedmixer.com.

[4] 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] The following instruments have been found satisfactory for this test method: Hoover Automatic Muller, Model M5, Hoover Color Corp., P.O. Box 218, State Highway 693, Hiwassee, VA 24347. Automatic Pigment Muller JEL 25/53-II, J. Engelsmann AG, Frankenthaler Str. 137 – 141, 67059 Ludwigshafen, Germany, www.engelsmann.de.

[6] The following instruments have been found satisfactory for this test method: Erichsen Tint Tester 527, available from T. J. Bell, Inc., 1340 Home Ave., Akron, OH 44310, and Hunter MiniScan EZ, available from Hunter Associates Laboratory, Inc., 11491 Sunset Hills Road, Reston, Virginia 20190-5280.

The Hunter MiniScan XE or XE Plus may still be used for the test, but are no longer commercially available. Instructions are the same for all Hunter Miniscan types. The Densicron reflectometer, though no longer commercially available, may be used for the test. For instructions, see Test Method D3265 – 01.

5.9.1 *Apparatus for Film Drawdown Method:*

5.9.1.1 *Film Applicator,*[7,4] 0.076 mm (0.003 in.) in depth.

5.9.1.2 *Polished Glass Plate,* approximately 760 by 500 by 10 mm (30 by 20 by 0.375 in.).

5.9.2 *Apparatus for Roller Spreader Method:*

5.9.2.1 *Tint Roller Spreader.*[8,4]

5.10 *Desiccator.*

5.11 *SpeedMixer (DAC 150 FVZ),* PP15 cups and holder.[3,4]

5.12 *IKA A-10 Analytical Mill,* A-14 SS cutter, A-18 chamber reducer.[9,4]

5.13 *5 mm Glass Grinding Beads.*[10,4]

6. Reagents and Materials

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[11] 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.

6.2 *Industry Tint Reference Black.*[12]

6.3 *Plasticizer,* soybean oil epoxide.[13,14]

6.4 *Denatured Alcohol,* for cleaning purposes.

6.5 *Zinc Oxide,* Industry Tint Zinc Oxide.[15]

6.6 *ASTM D24 Standard Reference Blacks.*[16]

7. Sampling

7.1 Samples of shipments shall be taken in accordance with Practices D1799 and D1900.

[7] The sole source of supply of the Film Applicator, Catalog No. 5553, known to the committee at this time is Byk-Gardner, 9104 Guilford Rd., Columbia, MD 21046, http://www.byk.com.

[8] The sole source of supply of the Tint Roller Spreader, Model 1A, known to the committee at this time is Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 79066-2316.

[9] The sole source of supply of the apparatus (Part #EW-04301-00) known to the committee at this time is Cole-Parmer Instrument Company, 625 East Bunker Court, Vernon Hills, IL 60061. http://www.coleparmer.com

[10] The sole source of supply of glass grinding beads known to the committee at this time is Quackenbush Co., Inc., 6711 Sands Rd., Crystal Lake, IL 60014. http://www.quackco.com

[11] *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.

[12] The original ITRB is depleted and therefore no longer commercially available. However, any available stocks of original ITRB may still be used. The successor of the original ITRB is ITRB2. ITRB2 was commercialized in 2012.

[13] The sole source of supply of the epoxidized soybean oil (Greenflex 7170) known to the committee at this time is Balentine Enterprises, Inc., dba Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007, www.carbonstandard.com.

[14] Supporting data (on the shelf life of this material) have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D24-1004.

[15] The sole source of supply of Industry Tint Zinc Oxide known to the committee at this time is Balentine Enterprises, Inc., dba Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007, www.carbonstandard.com.

[16] The sole source of supply of ASTM D24 Standard Reference Blacks known to the committee at this time is Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007.

8. Calibration and Standardization

8.1 *Standard Pastes*—Prepare pastes of the ITRB, following 9.2 – 9.3.2 for the following masses, prepare the 0.1000-g Automatic Muller paste or the 0.1200-g SpeedMixer paste in duplicate:

Industry Tint Reference Black		Calibration Tint Units [%]
Automatic Muller Pastes	SpeedMixer Pastes	
0.0900 g	0.1080 g	90.0
0.1000 g	0.1200 g	100.0
0.1100 g	0.1320 g	110.0
0.1200 g	0.1440 g	120.0
0.1300 g	0.1560 g	130.0

NOTE 2—Optionally, 0.1400 and 0.1500 g of Automatic Muller paste or 0.1680 and 0.1800 g of SpeedMixer paste may be added in the standardization step when samples with tint results above 130 tint units are tested. The modified standardization should only be used for samples exceeding 130 tint units.

8.2 *Erichsen Tint Tester 527:*

8.2.1 Turn on the power switch and allow for 30 min warm-up.

8.2.2 Place the reflectance head on the black calibration panel and adjust the digital readout to 0.00 using the "zero" control potentiometer.

8.2.3 Following Section 9, prepare the paste drawdown for reflectance measurement.

8.2.4 Use one of the 0.1000-g Automatic Muller or 0.1200-g SpeedMixer ITRB pastes of 8.1 to set the instrument to read 3.00 using the calibration control potentiometer.

8.2.5 Determine the reflectivity on the remaining 0.1000-g Automatic Muller or 0.1200-g SpeedMixer ITRB paste. This reflectance reading must be from 2.99 through 3.01 for the duplicate pastes to be considered acceptable.

8.2.6 If the duplicate 0.1000-g Automatic Muller or 0.1200-g SpeedMixer pastes are acceptable, the two duplicate pastes are blended together using a spatula, and the instrument is set to read 3.00 using the calibration control potentiometer.

8.2.7 If the duplicate pastes are unacceptable, prepare another 0.1000-g Automatic Muller or 0.1200-g SpeedMixer paste following 9.2 – 9.3.2, and follow 8.2.5.

8.2.8 Determine the reflectance value for all of the remaining ITRB pastes of 8.1.

8.2.9 Calculate the tint strength of the standard pastes as follows:

$$\text{Tint}[\%] = 3.00/S \times 100 \tag{1}$$

where:

S = reflectance value of sample.

8.2.10 Calculate a regression of the standard values (y value) on the measured values (x value) by using the least squares method.

8.2.11 The measured tint strength of all subsequent samples is corrected by substituting each measured value into this linear equation and calculating the corrected value for the tint strength.

8.2.12 New regression coefficients are determined periodically, typically on a monthly basis.

NOTE 3—Proper paste preparation and regression may be validated by analyzing at least one SRB tread grade (SRB A-C). After applying the normalization to the tint pastes and calculation as described in Section 10 the tested SRB samples should be within the accuracy limits given in Guide D4821.

8.2.13 Proper calibration and standardization of the equipment, reagents, materials, and method shall be checked on a periodic