Designation: D3265 – 23

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.

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

[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] 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.

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:

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.

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:

[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.

[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.

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:

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

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 basis using ASTM D24 Standard Reference Blacks. The standard tint strength values and the acceptable test limit of the standard reference blacks may be found in Guide D4821.

8.3 *Hunter MiniScan:*

8.3.1 Calibrate the instrument using the black and white tiles.

8.3.2 Place the instrument in Setup Mode. Set up instrument with XYZ color scale, D65 illuminant, 10° observer, average a minimum of three readings. (Note that Y corresponds to the lightness/darkness function, and thus represents the information of interest).

8.3.3 Read each of the ITRB pastes at least three times and record only the average Y value of the three readings. The Y value for the 0.1000-g Automatic Muller or 0.1200-g SpeedMixer paste should read approximately 2.60. Calculate the tint strength of the ITRB pastes as follows:

$$\text{Tint}[\%] = I/Y \times 100 \quad (2)$$

where:

I = reflectance of 0.1000-g Automatic Muller or 0.1200-g SpeedMixer ITRB paste.

Y = reflectance value of sample.

8.3.4 Follow 8.2.10 – 8.2.13 to complete the calibration of the Hunter MiniScan.

9. Procedure

9.1 *Paste Preparation:*

9.1.1 *Automatic Muller Paste Preparation:*

9.1.1.1 Dry the zinc oxide, ITRB, and the carbon black sample(s) for 1 h in the specified oven set at 125 ± 1°C (257 ± 2°F). Remove to a desiccator and allow to cool to room temperature.

NOTE 4—The ITRB must be dried each day a test is performed, preferably at the same time as the tested sample. Dry only the required amount, as the ITRB should not be dried repeatedly.

NOTE 5—As an optional step in the procedure a carbon black sample may be homogenized prior to drying and weighing using either a mortar and pestle or by low-intensity grinding, for example, in a coffee mill.

9.1.1.2 Weigh a sample of exactly 0.1000 g of carbon black into a weighing dish and then weigh onto the carbon black 3.7500 g of zinc oxide. For N500, N600, N700 series carbon blacks, weigh exactly 0.2000 g of carbon black and 3.7500 g of zinc oxide.

9.1.1.3 Using the syringe, place 2.20 ± 0.02 cm^3 or, if preferred, 2.20 g of plasticizer in the center of the mulling plate.

NOTE 6—If the use of 2.20 cm^3 (2.20 g) of plasticizer produces an unmanageable fluid paste, then it is acceptable to use 2.00 cm^3 (2.00 g) of plasticizer for both the ITRB standard and the sample.

9.1.1.4 Place the zinc oxide and carbon black in the center of the pool of plasticizer.

9.1.1.5 Mix the three materials with a spatula, confining them as close to the center as possible; mix until they are well incorporated. Clean the material from the spatula onto the top mulling plate, maintaining minimum loss of material.

9.1.1.6 Set the muller for 25 revolutions with one extra mass on the arm equivalent to a force of 445 N (100 lbf) on the plate. Close the plates, raise the mass arm, and start the muller.

9.1.1.7 At the end of the 25-revolution cycle, lower the mass arm, and open the plates. Scrape the upper plate with a spatula to remove as much paste as possible, and transfer it to the center of the lower plate. Then, with the lower plate rotating, use the spatula to spread the paste to a flattened circle on the plate, then work all of the paste to the exact center. Repeat this step two additional times. Clean the material from the spatula onto the top mulling plate.

9.1.1.8 Repeat 9.1.1.6 and 9.1.1.7 three times, for a total of four cycles of 25 revolutions each.

9.1.1.9 Remove the paste to a smooth, clean surface, and clean the muller plates, using a solvent and wiping tissue. (**Warning—** Denatured alcohol is highly flammable. Use only small quantities for cleaning. Make sure that the working environment is sufficiently vented. Do not start the muller immediately after cleaning, since ethanol/air vapors might be ignited.)

9.1.1.10 Prepare the paste for reflectance measurement by one of the methods in 9.2.1 or 9.2.2 and measure the paste reflectance by one of the methods in 9.3.1 or 9.3.2.

9.1.2 *SpeedMixer (DAC 150 FVZ) Paste Preparation:*

9.1.2.1 Mill pelleted carbon black using IKA milling procedure in 9.1.2.2 – 9.1.2.10. If testing powder samples proceed to 9.1.2.11.

9.1.2.2 Clean Analytical Mill using tissue and vacuum until visually clean. Pieces of tissue may be added to mill and ground to aid cleaning the chamber and blades.

9.1.2.3 Add 4 to 5 g pelleted CB to mill.

9.1.2.4 Add chamber reduction insert and close lid.

9.1.2.5 Grind for 15 s and discard sample.

9.1.2.6 Repeat the steps in 9.1.2.2 and 9.1.2.4.

9.1.2.7 Grind using two (2) 15 s pulses.

9.1.2.8 Remove lid and insert. Use a spatula to loosen CB from mill wall and blades.

9.1.2.9 Repeat the steps in 9.1.2.4, 9.1.2.7, and 9.1.2.8.

9.1.2.10 Discharge sample from mill. (Sample should be dried in oven.)

9.1.2.11 Set SpeedMixer acceleration “ramp up” = 10 s.

9.1.2.12 Tare a 15 g cup on analytical balance.

9.1.2.13 Weigh 4.5000 g zinc oxide directly into the 15 g cup.

9.1.2.14 Weigh exactly 0.1200 g CB directly into the 15 g cup on top of zinc oxide (0.2400 g for N500, N600, N700 series). Minimize CB contact with the cup wall by making a depression in the zinc oxide for the CB to be placed.

9.1.2.15 Close cup lid and place into holder without disturbing the zinc oxide and CB.

9.1.2.16 Place holder correctly in machine basket and dry grind for 3 min at 2500 r/min—make sure the cup is kept upright to keep CB from getting on lid.

9.1.2.17 Remove holder from instrument, remove cup from holder, and carefully remove lid.

9.1.2.18 Add eight (8) 5 mm glass beads.

9.1.2.19 Add 2.64 g Paraplex.

9.1.2.20 Close cup lid and place into holder, then place the holder into the machine without disturbing the mixture.

9.1.2.21 Mix for 2.0 min at 3500 r/min (fixed speed 2 must be set to provide 3500 r/min).

9.1.2.22 Remove cup from SpeedMixer and prepare the paste for reflectance measurement by one of the methods in 9.2.1 or 9.2.2, and measure the paste reflectance by one of the methods in 9.3.1 or 9.3.2:

NOTE 7—The SpeedMixer paste will contain glass beads. When SpeedMixer pastes are used with the film drawdown method, the glass beads can be easily separated from the paste while preparing the drawdown with the film applicator. When SpeedMixer pastes are used with the roller spreader method, the glass beads may need to be separated from the paste prior to application to the roller.

9.2 *Film Application:*

9.2.1 *Film Drawdown Method:*

9.2.1.1 Clean the glass plate with a wiping tissue to remove any dust particles or film.

9.2.1.2 Use a spatula to place a portion of the paste near the top edge of the glass plate.

9.2.1.3 Using a film applicator and applying moderate, consistent pressure, draw down the paste to the bottom edge in 2 to 3 s.

9.2.1.4 Pick up the applicator without removing the excess paste adhered to it; return it to the beginning of the drawdown, and again draw down the paste to the bottom edge of the glass plate in 2 to 3 s. The drawdown shall have a uniform surface approximately 65 mm (2.5 in.) wide. If the surface is not uniform, prepare another drawdown.

NOTE 8—The wet film thickness of the drawdown has a nominal thickness of 0.04 mm (0.0015 in.), or approximately one half the actual gap clearance of the applicator.

9.2.2 *Roller Spreader Method*—No preparation of the paste is required, for it is placed directly on the turning roller of the tint roller spreader.

9.3 *Reflectance Measurement:*

9.3.1 *Erichsen Tint Tester 5270—Film Drawdown Method:*

9.3.1.1 Disregarding the first 75 mm (3 in.) at the top of the drawdown, set the reflectance head aperture over the drawdown of the ITRB (100 %) paste.

9.3.1.2 Adjust the Erichsen reflectometer to read an average of 3.00 for at least four readings taken at different positions.

9.3.1.3 The instrument is now correctly adjusted for reading other pastes. The value of 3.00 is used for ITRB for making calculations in 10.1.

9.3.1.4 Remove the paste and clean the drawdown area when all readings have been made.

9.3.2 *Hunter MiniScan—Film Drawdown Method:*

9.3.2.1 Disregarding the first 75 mm (3 in.) at the top of the drawdown, set the reflectance head aperture over the drawdown.

9.3.2.2 Take at least three readings at different positions. Record each reading and use the average as the reflectance value for the sample in 10.1.

9.3.2.3 Remove the paste and clean the drawdown area when all readings have been taken.