Designation: D 3265 – 03

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

This standard is issued under the fixed designation D 3265; 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 and health practices and determine the applicability of regulatory limitations prior to use.*

## 2. Referenced Documents

2.1 *ASTM Standards:*

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[2]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias[2]

## 3. Summary of Test Method

3.1 A carbon black sample is mixed with a white powder (zinc oxide) and a liquid vehicle (soybean oil epoxide) 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.

## 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.

## 5. Apparatus

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

5.2 *Automatic Muller.*[3]

5.3 *Photometric Instrument*, capable of detecting differences in the amount of light reflectance between shades of gray.[4,5] 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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[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] *Annual Book of ASTM Standards*, Vol 09.01.

[3] The sole source of supply of the Hoover Automatic Muller, Model M5 known to the committee at this time is Hoover Color Corp., P.O. Box 218, State Highway 693, Hiwassee, VA 24347. 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.

[4] The following instruments have been found satisfactory for this test method: Erichsen Tint Tester 527, available from T. G. Bell, Inc., 1340 Home Ave., Akron, OH 44310, and Hunter MiniScan XE or XE Plus, available from Hunter Associates Laboratory, Inc., 11491 Sunset Hills Road, Reston, Virginia 22090-5280. The Densicron reflectometer may be used per Test Method D 3265 – 00.

[5] The Densicron reflectometer, though no longer commercially available, may be used for the test. For instructions, see Test Method D 3265 – 01.

5.9.1 *Apparatus for Film Drawdown Method*:

5.9.1.1 *Film Applicator,*[3,6] 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.*[3,7]

5.10 *Desiccator*.

## 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.[8] 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.*[9]

6.3 *Plasticizer*, soybean oil epoxide.[10]

6.4 *Solvent*, cleaning, low toxicity, and nonflammable.[11]

6.5 *Zinc Oxide*, Industry Tint Zinc Oxide.[12]

6.6 *ASTM D24 Standard Reference Blacks.*[13]

## 7. Sampling

7.1 Samples of shipments shall be taken in accordance with Practices D 1799 and D 1900.

## 8. Calibration and Standardization

8.1 *Standard Pastes*—Prepare pastes of the ITRB, following 9.1-9.9 for the following masses, prepare the 0.1000-g paste in duplicate:

| Industry Tint Reference Black (g in 10.2 mixture) | Calibration Tint Units | Acceptable Range |
|---|---|---|
| 0.0900 | 90.0 | 89.3 – 90.7 |
| 0.1000 | 100.0 | 99.2 – 100.8 |
| 0.1100 | 110.0 | 109.1 – 110.9 |
| 0.1200 | 120.0 | 119.0 – 121.0 |
| 0.1300 | 130.0 | 129.0 – 131.0 |

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 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 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 pastes are acceptable, the two 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 paste following 9.1 through 9.9, 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:

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

where:

$S$ = reflectance value of sample.

8.2.10 If the measured tint values are not within the guidelines (±0.8 % relative of the target tint strength values) given in 8.1, then a regression of the standard values ($y$ value) on the measured values ($x$ value) must be calculated using the least squares method.

NOTE 2—It is almost always necessary to develop a statistical equation to measure tint strength values.

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

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

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 D 4821.

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 3 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 $Y$

[6] The sole source of supply of the Film Applicator, Catalog No. AR-5257 known to the committee at this time is Byk-Gardner, Ph. (800) 343-7721, http://byk-gardnerusa.com..

[7] 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.

[8] *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.

[9] Industry Tint Reference Black, an N-330 carbon black, is available from Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007.

[10] *Chemical Abstracts Registry Number* 12768-71-7, "Paraplex G-62," available from C. P. Hall Co., Technical Center, Customer Service Department, 5851 West 73rd St., Chicago, IL, 60638. Minimum order is 5 gal and order should state "Refer to ASTM D3265." A report on the shelf life of this material is filed at ASTM International Headquarters. Request RR: D24-1004.

[11] 1,1,1-trichloroethane has been found satisfactory for this test method.

[12] Available from Forcoven Products, Inc., P.O. Box 1556, Humble, TX 77347.

[13] Available from Laboratory Standards and Technologies, 227 Somerset St., Borger, TX 79007.

value for the 0.1000 g paste should read approximately 2.60. Calculate the tint strength of the ITRB pastes as follows:

$$\text{Tint} = 2.60/Y \text{ X100} \quad (2)$$

where:
$Y$ = 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 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 3—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.

9.2 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.

9.3 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 4—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.

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

9.5 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.

9.6 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.

9.7 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.

9.8 Repeat 9.6 and 9.7 three times, for a total of four cycles of 25 revolutions each.

9.9 Remove the paste to a smooth, clean surface, and clean the muller plates, using a solvent and wiping tissue.

9.10 Prepare the paste for reflectance measurement by one of the following methods:

9.10.1 *Film Drawdown Method*:

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

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

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

9.10.1.4 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 5—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.

9.10.2 *Roller Spreader Method*:

9.10.2.1 No preparation of the paste is required, for it is placed directly on the turning roller of the tint roller spreader.

9.11 Adjust the reflectance instrument as follows:

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

9.11.1.1 Follow 9.1-9.9.

9.11.1.2 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.

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

9.11.1.4 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.

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

9.12 Determine the reflectance value of the prepared paste of the test sample as follows:

9.12.1 *Erichsen—Film Drawdown Method*:

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

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

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

9.13 *Hunter MiniScan—Film Drawdown Method*:

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

9.13.2 Take at least 3 readings at different positions and record the average as the reflectance value for the sample in 10.1.

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

## 10. Calculation

10.1 Calculate the tint strength to the nearest 0.1 unit as follows:

$$T = [(I/S \times 100) \times M] + B \quad (3)$$

where:
$T$ = tint units,
$I$ = reflectance value for 0.1000-g ITRB,
$S$ = reflectance value for the sample,
$M$ = slope of regression equation from 8.2.11, and
$B$ = y-intercept from 8.2.10.

**TABLE 1 Precision Parameters[A] for ASTM 3265, Carbon Black—Tint Strength, (Type 1 Precision)**

NOTE 1—Symbols are defined as follows:
*Sr* = within laboratory standard deviation
*r* = repeatability, measurement units
*(r)* = repeatability, %
*SR* = between laboratory standard deviation
*R* = reproducibility, measurement units
*(R)* = reproducibility, %

| Material | Number of Laboratories | Tint Strength Units Mean Value | *Sr* | *(r)* | *SR* | *(R)* |
|---|---|---|---|---|---|---|
| SRB F6 (N683) | 16 | 51.7 | 0.49 | 2.69 | 1.55 | 8.49 |
| SRB D6 (N762) | 16 | 56.8 | 0.67 | 3.34 | 1.67 | 8.34 |
| SRB E6 (N660) | 16 | 60.0 | 0.64 | 3.03 | 1.71 | 8.07 |
| SRB C6 (N326) | 14 | 113.1 | 0.56 | 1.39 | 1.58 | 3.96 |
| SRB B6 (N220) | 16 | 117.8 | 1.12 | 2.68 | 1.35 | 3.25 |
| SRB A6 (N134) | 16 | 129.8 | 1.37 | 3.00 | 2.58 | 5.63 |
| Average | | 88.2 | | | | |
| Pooled Values | | | 0.87 | 2.79 | 1.79 | 5.73 |

[A] Precision data based on normalized tint strength.

For N500, N600, N700 series carbon blacks, divide the tint value by 2, after applying regression equation, in order to get the final value.

NOTE 6—The regression analysis may be omitted if the ITRB calibration curve meets the requirements of 8.1.

## 11. Report

11.1 Report the following information:

11.1.1 Proper identification of the sample,

11.1.2 Method used in reading the paste reflectance, and

11.1.3 The result obtained from the individual determination is reported to the nearest 0.1 unit.

## 12. Precision and Bias

12.1 These precision statements have been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical details.

12.2 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 1.

12.3 A type 1 interlaboratory precision program was conducted as shown in Table 1. 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).

**TABLE 2 Interlaboratory Precision Program**

| Material | Number of Laboratories |
|---|---|
| SRB A6 (N134) | 16 |
| SRB B6 (N220) | 16 |
| SRB C6 (N326) | 14 |
| SRB D6 (N762) | 16 |
| SRB E6 (N660) | 16 |
| SRB F6 (N683) | 16 |

12.4 The results of the precision calculations for this test are given in Table 1. The materials are arranged in ascending "mean level" order.

12.5 *Repeatability*—The pooled relative repeatability, *(r)*, of this test has been established as 2.79 %. Any other value in Table 1 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 1 must be suspected of being from different populations and some appropriate action taken.

NOTE 7—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, and so forth, which generated the two test results.

12.6 *Reproducibility*—The pooled relative reproducibility, *(R)*, of this test has been established as 5.73 %. Any other value in Table 1 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 1 must be suspected of being from different populations and some appropriate investigative or technical/commercial action taken.

12.7 *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.

## 13. Keywords

13.1 carbon black; photo–electric reflectance meter; tint strength