



Designation: D 3265 – 03

Standard Test Method for Carbon Black—Tint Strength¹

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 approval. 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 Shipments²
- D 1900 Practice for Carbon Black—Sampling Bulk Shipments²
- D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²
- D 4821 Guide for Carbon Black—Validation of Test Method Precision and Bias²

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.

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.

Current edition approved July 10, 2003. Published August 2003. Originally approved in 1973. Last previous edition approved in 2002 as D 3265 – 02.

² *Annual Book of ASTM Standards*, Vol 09.01.

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 $\pm 1^\circ\text{C}$ at 125°C ($\pm 2^\circ\text{F}$ at 257°F) and temperature uniformity within $\pm 5^\circ\text{C}$ ($\pm 9^\circ\text{F}$).

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-cm³, automatic refilling, reproducible to ± 0.02 cm³.

5.8 *Wiping Tissue*, absorbent and lint free.

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

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 committee¹, 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 Densicon reflectometer may be used per Test Method D 3265 – 00.

⁵ The Densicon 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:*

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.

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 ($\pm 0.8\%$ 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