



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 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 Shipments²

D 1900 Practice for Carbon Black—Sampling Bulk Shipments²

D 3324 Practice for Carbon Black—Improving Test Reproducibility Using ASTM Reference Blacks²

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

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 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:

5.9.1 *Apparatus for Film Drawdown Method*:

5.9.1.1 *Film Applicator*,⁵ 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*:

³ Hoover Automatic Muller, Model M5, available from Hoover Color Corp., P.O. Box 218, State Highway 693, Hiwassee, VA 24347, has been found satisfactory for this test method.

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; Densichron Reflectometer, Catalog No. 3837, composed of the following parts: Model 1 Photometer-Linear Scale, Catalog No. 3830D; Reflection Head, Catalog No. 3832A; Regulated 6-V Transformer, Catalog No. 3834M; Reflection Tile Set (Standard), Catalog No. 3827T, available from Sargent-Welch Scientific Co., Densichron Div., 7300 N. Lander Ave., Skokie, IL 60076. This model is no longer available and the suggested replacement model 3867 B has not been found to be satisfactory. Hunter MiniScan XE or XE Plus, available from Hunter Associates Laboratory, Inc., 11491 Sunset Hills Road, Reston, Virginia 22090-5280.

⁵ Film Applicator, Catalog No. AR-3800F, available from Pacific Scientific Co., 2431 Linden Ln., Silver Spring, MD 20910.

¹ This test method is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.11 on Absorptive Properties of Carbon Black.

Current edition approved May 10, 2000. Published June 2000. Originally published as D 3265 – 73. Last previous edition D 3265 – 99a.

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

5.9.2.1 Tint Roller Spreader.⁶

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.⁷ 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.*⁸

6.3 *Plasticizer, soybean oil epoxide.*⁹

6.4 *Solvent, cleaning, low toxicity, and nonflammable.*¹⁰

6.5 *Zinc Oxide, Industry Tint Zinc Oxide.*¹¹

6.6 *ASTM D-24 Standard Reference Blacks.*¹²

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 Densichron Reflectometer

8.2.1 Following 9.10 and 9.11, use one of the 0.1000-g (100 tint units) ITRB pastes to set the instrument to an 80 reflectance value.

8.2.2 Following 9.10 and 9.12, determine the reflectivity of the remaining 0.1000-g paste. The reflectance reading must be from 79.5 to 80.5 for the duplicate pastes to be considered acceptable.

8.2.3 If the duplicate 0.1000-g pastes are acceptable, the two are blended together using a spatula, and repeat 8.2.1 using this blend.

8.2.4 If the duplicate pastes are unacceptable, prepare another 0.1000-g paste following 9.1-9.9. Repeat 8.2.1 and 8.2.2 with the new paste, and blend it with whichever of the previous 0.1000-g pastes which will satisfy the limits of 8.2.2. Repeat 8.2.1 using this blend.

8.2.5 Following 9.10 and 9.12, determine the reflectance value for all of the remaining ITRB pastes of 8.1.

8.2.6 Calculate the tint strength for the standard pastes as follows:

$$\text{Tint Units} = 80/S \times 100 \quad (1)$$

where:

S = reflectance value of sample.

8.2.7 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.

8.2.8 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.

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

8.2.10 Proper calibration and standardization of the equipment, reagents, materials, and method shall be checked on a periodic basis using the ASTM D-24 Standard Reference Blacks. The standard tint strength values and the acceptable test limit of the standard reference blacks may be found in Practice D 3324.

8.3 Erichsen Tint Tester 527:

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

8.3.2 Place the reflectance head on the black calibration panel and adjust the digital readout to 0.00 using the “zero” control potentiometer.

8.3.3 Following 9.10, prepare the paste drawdown for reflectance measurement.

8.3.4 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.

8.3.5 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.

8.3.6 Follow 8.2.3 and 8.2.4.

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

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

$$\text{Tint Units} = 3.00/S \times 100 \quad (2)$$

where:

S = reflectance value of sample

8.3.9 Follow 8.2.7-8.2.10 to complete calibration of the Erichsen Tint Tester.

⁶ Tint Roller Spreader, Model 1A, available from Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 79066-2316 has been found satisfactory for this test method.

⁷ “Reagent Chemicals, American Chemical Society Specifications,” Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see “Analar Standards for Laboratory U.K. Chemicals,” BDH Ltd., Poole, Dorset, and the “United States Pharmacopeia.”

⁸ Industry Tint Reference Black, an N-330 carbon black, is available from Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 79066-2316.

⁹ *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 (312/767-4600). Minimum order is 5 gals and order should state “Refer to ASTM D3265.” A report on the shelf life of this material is filed at ASTM Headquarters. Request RR: D24-1004.

¹⁰ 1,1,1-trichloroethane has been found satisfactory for this test method.

¹¹ Available from Forcoven Products, Inc., P.O. Box 1556, Humble, TX 77347.

¹² F-4 will be used until depleted, at which time F-5 will be used. Available from Titan Specialties, Inc., P.O. Box 2316, Pampa, TX 79066-2316.