



Designation: D 4821 – 99

## Standard Guide for Carbon Black—Validation of Test Method Precision and Bias<sup>1</sup>

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### 1. Scope

1.1 This guide covers a procedure for using ASTM Standard Reference Blacks (SRBs) to continuously monitor the precision of those carbon black test methods for which standard values have been established. It also offers guidelines for trouble-shooting various test methods.

1.2 This guide establishes the limits within which the statistical calibration procedure of Practice D 3324 shall be applied and establishes the limits for an out-of-calibration condition beyond which the statistical calibration procedure of Practice D 3324 shall not be applied.

1.3 This guide uses statistical control chart methodology as discussed in STP-15-D<sup>2</sup> to determine if a laboratory's test results differ significantly from the accepted values of the SRBs and provides a format for reporting test data for specified tests on the SRB blacks.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 *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 1510 Test Method for Carbon Black—Iodine Adsorption Number<sup>3</sup>

D 2414 Test Method for Carbon Black—*n*-Dibutyl Phthalate Absorption Number<sup>3</sup>

D 3037 Test Methods for Carbon Black—Surface Area by Nitrogen Adsorption<sup>3</sup>

D 3265 Test Method for Carbon Black—Tint Strength<sup>3</sup>

D 3324 Practice for Carbon Black—Improving Test Reproducibility Using ASTM Reference Blacks<sup>3</sup>

<sup>1</sup> This guide is under the jurisdiction of ASTM Committee D-24 on Carbon Black and is the direct responsibility of Subcommittee D24.61 on Carbon Black Sampling and Statistical Analysis.

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<sup>2</sup> *Symposium on Manual on Presentation of Data and Control Chart Analysis, ASTM STP 15D*, ASTM, 1976.

<sup>3</sup> *Annual Book of ASTM Standards*, Vol 09.01.

D 3493 Test Method for Carbon Black—*n*-Dibutyl Phthalate Absorption Number of Compressed Sample<sup>3</sup>

D 3765 Test Method for Carbon Black—CTAB (Cetyltrimethylammonium Bromide) Surface Area<sup>3</sup>

D 4820 Test Methods for Carbon Black—Surface Area by Multipoint B. E. T. Nitrogen Adsorption<sup>3</sup>

### 3. Significance and Use

3.1 One of the major causes of poor test precision is the lack of calibration or standardization of instruments, apparatus, reagents, and technique among laboratories.

3.2 In addition to the calibration of a test method by physicochemical means, Practice D 3324 describes a statistical method for achieving calibration of a test method.

3.3 This guide outlines the use of control charts to graphically present calibration test data determined for the ASTM SRBs for those test methods given in Section 2. These control charts shall be used to validate the testing precision and bias of an individual laboratory.

### 4. Procedure

4.1 Select six SRBs from the SRB 4 and SRB 5 series to cover the range of interest, being sure to include one each of the A, B, C, D, E, and F (see Note 1) SRBs.

NOTE 1—F-5 will not be available for purchase until F-4 is depleted.

4.2 Prepare a control chart for each of the selected SRBs for each test method as presented by Part 3 of ASTM STP 15D.

4.3 The target values given in Table 1 and Table 2 are the nominal SRB values reported in Practice D 3324. Values are used as control chart limits plus or minus three single test repeatability standard deviations as given in the precision section of each applicable test method for the SRB 4s or as determined during the validation of the SRB 5s.

4.4 Plot the uncorrected values for the selected six SRBs until a repeatability control limit is exceeded (see Note 2). Perform a retest immediately. If the retest falls outside the repeatability control limits, then begin a search for an assignable cause. (See Section 5 for a list of assignable causes.) If the search for an assignable cause and the subsequent action taken still do not produce results within the control limits, then a statistical regression or correction equation shall be calculated as described in Practice D 3324.

NOTE 2—Selected SRBs from SRB 4 and SRB 5 may be plotted on