



Standard Guide for Carbon Black—Validation of Test Method Precision and Bias¹

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1. Scope

1.1 This guide covers a procedure for using ASTM Standard Reference Blacks² (SRBs) to continuously monitor the precision of those carbon black test methods for which standard values have been established. It also offers guidelines for troubleshooting various test methods.

1.2 This guide establishes the x-chart control limits to be used when continuously monitoring those tests listed in Section 2. Alternatively, these control limits may be used as a basis for comparison to testing precision computed within a laboratory.

1.3 This guide uses statistical control chart methodology as discussed in STP-15-D³ to determine if a laboratory's test results differ significantly from the accepted values of the SRBs.

1.4 This guide provides a statistical procedure for improving test reproducibility when a laboratory cannot physically calibrate its apparatus to obtain the standard values of the ASTM SRBs, within the ranges given in the precision statement of the test method.

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

1.6 *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:⁴

¹ This guide is under the jurisdiction of ASTM Committee D24 on Carbon Black and is the direct responsibility of Subcommittee D24.61 on Carbon Black Sampling and Statistical Analysis.

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² Standard Reference Blacks are available from Laboratory Standards & Technologies, Inc., 227 Somerset St., Borger, TX 79007. Phone/fax: (806) 273-3006. E-mail: jwbal@infinitytx.net.

³ *Symposium on Manual on Presentation of Data and Control Chart Analysis, ASTM STP 15D*, ASTM, 1976.

⁴ 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.

- D 1510 Test Method for Carbon Black—Iodine Adsorption Number
- D 1765 Classification System for Carbon Blacks Used in Rubber Products
- D 2414 Test Method for Carbon Black—Oil Absorption Number
- D 3265 Test Method for Carbon Black—Tint Strength
- D 3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample
- D 6556 Test Methods for Carbon Black—Total and External Surface Area by Nitrogen Adsorption

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. The sum of all sources of testing error is unique for an individual laboratory. A least-squares regression of a laboratory's actual test values for reference materials to the established mean values will result in a unique least-squares regression line (and equation) for that laboratory. Generally, there are two reasons for using the SRBs in testing: (1) to monitor testing performance (see Section 4) to ensure that no systematic error or bias is affecting the test results, or (2) to establish a statistical calibration (see Section 5) when the correction of assignable causes (see Section 6) does not yield in-control test results.

3.2 In addition to the calibration of a test method by physicochemical means, a statistical method for achieving calibration of a test method is presented.

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. All laboratories are encouraged to utilize statistical control charts and the SRBs because this allows a comparison of testing precision within a laboratory to the "industry average" values found in Table 1.

3.4 The techniques of this guide can be used to continuously monitor testing execution and precision for other tests that are not listed in Section 2 or for materials that fall outside the range of the SRBs for those tests listed in Section 2. In these cases, each laboratory will have to establish the applicable mean and control limit values for the "local reference." The monitoring will then consist of a comparison of present test results for the