



Standard Test Methods for Carbon Black—Heating Loss¹

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This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 These test methods cover the determination of the heating loss of carbon black at 125°C. This heating loss consists primarily of moisture, but other volatile materials may also be lost. These test methods are not applicable to treated carbon blacks that contain added volatile materials, if moisture loss is to be measured.

1.2 These test methods may also be used for the determination of the heating loss of recovered carbon fillers (rCF/rCB) at 125°C. However, these materials were not included in the precision studies and therefore, the precision statements contained in this standard may not be valid for these materials.

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

1.4 *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:²

[D1799 Practice for Carbon Black—Sampling Packaged Shipments](#)

[D1900 Practice for Carbon Black—Sampling Bulk Shipments](#)

[D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries](#)

¹ These test methods are under the jurisdiction of ASTM Committee D24 on Carbon Black and are the direct responsibility of Subcommittee D24.31 on Non-Carbon Black Components of Carbon Black.

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² 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.

3. Summary of Test Method

3.1 A carbon black sample is weighed before and after heating for 1 h at 125°C. The observed difference in mass is the heating loss.

4. Significance and Use

4.1 In addition to determining the heating loss (primarily moisture content) of carbon black, these drying conditions are used to prepare samples prior to performing other carbon black tests.

4.2 When larger samples are prepared for other tests, use an open vessel of suitable dimensions so that the depth of the black is no more than 10 mm during conditioning.

4.3 Carbon black is hygroscopic. The amount of moisture absorbed is related to the surface area of the black and to the relative humidity, ambient temperature, and time to which the material is exposed.

Method A—Convection–Gravity Oven Method

5. Apparatus

5.1 *Oven*, gravity-convection type, capable of temperature regulation of within $\pm 1^\circ\text{C}$ at 125°C and temperature uniformity within $\pm 5^\circ\text{C}$.

5.2 *Weighing Bottle*, low-form, 30 mm in height and 60 mm in diameter, equipped with a ground-glass stopper.

5.3 *Analytical Balance*, having a sensitivity of 0.1 mg.

5.4 *Desiccator*.

6. Sampling

6.1 Samples shall be taken in accordance with Practices [D1799](#) or [D1900](#).

6.1.1 Place the samples of carbon black in airtight sample containers. Allow the closed container to reach room temperature before starting the test.

7. Procedure

7.1 Dry the weighing bottle and the stopper, with the stopper removed, in the specified oven set at 125°C for 30 min.

Place the bottle and stopper in the desiccator and allow to cool to room temperature. Weigh the bottle with stopper to the nearest 0.1 mg.

7.2 Weigh 2 g of carbon black into the weighing bottle to the nearest 0.1 mg.

7.3 Place the weighing bottle, sample, and stopper in the specified oven set at 125°C for 1 h with the stopper removed.

7.4 Replace the stopper and transfer the bottle and contents to the desiccator. Remove the stopper and allow to cool to room temperature. Replace the stopper on the weighing bottle and reweigh to the nearest 0.1 mg.

NOTE 1—Keep the stopper on the weighing bottle when transferring to and from the desiccator to prevent loss of carbon black due to air currents.

7.5 Repeat the procedure on a second sample.

8. Calculation

8.1 Calculate the percent heating loss to the nearest 0.1 % as follows:

$$H = [(B - C)/(B - A)] \times 100 \quad (1)$$

where:

H = heating loss, %,

A = mass of weighing bottle and stopper, g,

B = mass of weighing bottle, stopper, and sample before heating, g, and

C = mass of weighing bottle, stopper, and sample after heating, g.

9. Report

9.1 Report the following information:

9.1.1 Proper identification of the sample, and

9.1.2 Result reported to the nearest 0.1 %.

10. Precision and Bias

10.1 This precision and bias statement has been prepared in accordance with Practice D4483. Refer to Practice D4483 for terminology and other statistical details.

10.2 *Precision*—The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers, carbon blacks, etc.) used in the particular interlaboratory program described in 10.3 –

10.3.2. 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.

10.3 *Convection-Gravity Oven—Test Method A*—A Type 1 interlaboratory precision program was conducted in 1994. Both repeatability and reproducibility represent short-term testing conditions. Eight laboratories tested three carbon blacks (Materials A, B, and C) twice on each of two different days. A test result is the value obtained from a single determination. Acceptable difference values were not measured. (See Table 1 for the individual precision results for these three carbon blacks, which span a broad range for heat loss.)

10.3.1 *Repeatability*—The pooled absolute repeatability, r , of Method A heat loss has been established as 0.070 %. Two single test results (or determinations) that differ by more than 0.070 % must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

10.3.2 *Reproducibility*—The pooled absolute reproducibility, R , of Method A heat loss has been established as 0.356 %. Two single test results (or determinations) produced in separate laboratories that differ by more than 0.356 % must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

10.4 *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.

Method B—Moisture Balance Method

11. Apparatus

11.1 *Moisture Balance*, having a sensitivity of 0.1 mg and preferably an indirect heating source.

NOTE 2—An indirect heating source is preferred due to the ability of a carbon black to absorb infrared radiation and elevate the temperature of

TABLE 1 Test Methods D1509 Test Method Precision—Type 1 (Convection-Gravity Oven—Method A)^A

| Material | Mean Level, % Mass Loss | Within Laboratories ^B | | | Between Laboratories ^B | | |
|---------------|----------------------------|----------------------------------|-------|---------|-----------------------------------|-------|---------|
| | | S_r | r | (r) | S_R | R | (R) |
| B | 0.03 | 0.012 | 0.033 | 95.4 | 0.033 | 0.093 | 271 |
| A | 0.24 | 0.011 | 0.032 | 12.9 | 0.051 | 0.145 | 59.0 |
| C | 0.40 | 0.039 | 0.112 | 28.2 | 0.210 | 0.593 | 149 |
| Average | 0.23 | | | | | | |
| Pooled values | | 0.025 | 0.070 | 30.9 | 0.126 | 0.356 | 158 |

^A This is short-term precision (days) with outliers removed from the data set.

^B Symbols are defined as follows:

S_r = within-laboratory standard deviation,

r = repeatability (in measurement units),

(r) = repeatability (in relative percent),

S_R = between-laboratory standard deviation,

R = reproducibility (in measurement units), and

(R) = reproducibility (in relative percent).

the carbon black higher than the surrounding atmosphere.

11.2 *Desiccator*.

12. Sampling

12.1 Samples shall be taken in accordance with Practices **D1799** or **D1900**.

12.1.1 Place the samples of carbon black in air-tight containers. Allow the closed container to reach room temperature before starting the test.

13. Procedure

13.1 Set up the moisture balance according to the manufacturer's instructions. The temperature should not exceed 125°C.

13.2 Place approximately 2 g of carbon black into the moisture balance and determine its mass to the nearest 0.1 mg.

13.3 Close the lid and start the machine.

13.4 Once the mass loss, under these drying conditions, is less than 1 mg over 30 s the test portion is considered dry and the percent mass loss should be recorded to the nearest 0.1 %.

14. Calculation

14.1 Calculate the percent heating loss to the nearest 0.1 % as follows:

$$H = [(A - B)/A] \times 100 \quad (2)$$

where:

H = heating loss, %,

A = mass of test portion before heating, g, and

B = mass of test portion after heating, g.

15. Report

15.1 Report the following information:

15.1.1 Proper identification of the sample, and

15.1.2 Result obtained from an individual determination, reported to the nearest 0.1 %.

16. Precision and Bias

16.1 No precision and bias statement has been developed on this procedure; however, it is anticipated that it will be developed (see Practice **D4483**).

17. Keywords

17.1 carbon black; heating loss; moisture; volatile materials

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