



Designation: D 6086 – 05

## Standard Test Method for Carbon Black—Void Volume (VV)<sup>1</sup>

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### 1. Scope

1.1 This test method covers a procedure to measure a carbon black property called Void Volume. The void volume is obtained by measuring the compressed volume of a weighed sample contained in a cylindrical chamber when a specified compression force is applied by a movable piston with a displacement transducer on the piston mechanism.

1.2 Void volume is an important carbon black property that relates to the compounded physical properties for carbon black-filled elastomers.

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:<sup>2</sup>

D 1799 Practice for Carbon Black—Sampling Packaged Shipments

D 1900 Practice for Carbon Black—Sampling Bulk Shipments

### 3. Terminology

3.1 *Definitions of Terms Specific to This Standard*—Refer to Sections 4 and 9 for a more complete understanding of the use of these terms in this test method.

3.1.1 *compressed volume (carbon black), n*—the volume that a mass of carbon black occupies when it is contained in a specified cylindrical chamber and subjected to a specified compression force by a movable piston.

<sup>1</sup> This test method is under the jurisdiction of Committee D24 on Carbon Black and are the direct responsibility of Subcommittee D24.11 on Carbon Black Structure.

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<sup>2</sup> 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.

3.1.2 *theoretical volume (carbon black), n*—the volume that a specific mass of carbon black particles with no voids, for practical purposes, is given by the ratio of the mass to the density, when the density is determined by an accepted test.

3.1.3 *void volume (carbon black), n*—a measure of the irregularity and non-sphericity of carbon black aggregate particles, it is expressed as the difference, (compressed volume-theoretical volume) for a mass of carbon black, the differential volume is normalized to a selected unit mass.

3.1.3.1 *Discussion*—The aggregate irregularity resists compression and thus the compressed volume is a function of the degree of this irregularity. The compressed void volumes are specific to the compressed volume measuring instrument and to the conditions of test.

### 4. Summary of Test Method

4.1 The compressed volume of a weighed dry test sample of a candidate carbon black is obtained in a void volume instrument under calibration test conditions. From the compressed volume, the void volume is obtained by subtracting the theoretical volume.

### 5. Significance and Use

5.1 The void volume of a carbon black expressed in absolute terms, VV, is a carbon black property that reflects differences in structure for candidate carbon blacks. Structure is a generic term that is a function of the shape irregularity and deviation from sphericity of carbon black aggregates. The more that a carbon black resists compression by having substantial aggregate irregularity and non-sphericity, the greater the compressed volume and void volume.

5.2 Structure is a property that influences the physical properties developed in carbon black compounds for use in tires, mechanical rubber goods, and other manufactured rubber products.

### 6. Apparatus

6.1 *Analytical Balance*, or equivalent, capable of a weighing sensitivity of 0.1 mg.

6.2 *Gravity Convection Drying Oven*, capable of maintaining  $125 \pm 5^\circ\text{C}$ .

6.3 *Weighing Dish, Camel Hair or Similar Brush*, to be used for weighing the samples.

6.4 *Void Volume Instrument*, to be used to measure the compressed volume of carbon blacks from which the void volume is calculated. The void volume instrument or device shall conform to the following generic specifications and be capable of operating as outlined in 6.4.1-6.4.3.

6.4.1 The instrument shall have a rigid framework that contains a metal block with a vertically oriented cylindrical sample chamber (see Fig. X1.1).

6.4.2 When opened, the sample port shall allow the loading of a sample of carbon black.

6.4.3 By means of a suitable mechanism with sufficient power for the compression forces as required for testing, the piston shall be capable of being moved to compress the sample. A device to record the movement of the piston and indicate displacement shall be provided. The compressed volume of any sample is determined at the end of a test, by the distance from the end of the piston to the end of the sample port; this is designated as a “height” in the procedure discussed in Section 9. A test is concluded and the height is measured when a predetermined pressure is attained. See Appendix X1 for a brief description of a commercial void volume instrument that meets these specifications.

## 7. Sampling

7.1 Samples of candidate carbon blacks shall be taken in accordance with Practice D 1799 or D 1900.

## 8. Calibration

8.1 Follow the manufacturer’s instructions to set up the instrument and to calibrate the zero value reading of the height.

8.2 Follow the manufacturer’s instructions to calibrate the height measurement using a physical standard (metal plug).

8.3 Follow the manufacturer’s instructions to calibrate the compression pressure using a test gage.

NOTE 1—Once precision and bias statements are available for standard reference blacks, alternative calibration techniques can be evaluated.

## 9. Procedure

### 9.1 Sample Preparation:

9.1.1 Dry an adequate sample of the carbon black at 125 ± 5°C for 1 h and cool in a desiccator.

9.1.2 Weigh 2.0000 g (to the nearest 0.0001 g) of the dried carbon black (or use a sample weight specified by the manufacturer).

### 9.2 Evaluating Candidate Carbon Blacks:

9.2.1 Place the dried and weighed carbon black in the calibrated instrument. Start the test sequence to apply the predetermined pressure. The height of the piston will be automatically recorded and used to calculate the sample’s compressed volume.

## 10. Void Volume Calculations

10.1 The void volume (VV) is calculated from the measured compressed volume as follows. The compressed volume of the weighed candidate sample is evaluated by Eq 1.

$$V_A = h \times 3.1416 D^2 / 4000 \quad (1)$$

where:

$V_A$  = the (actual) compressed volume of the weighed candidate sample,  $\text{cm}^3$ ,

$h$  = the “height” of the compressed carbon black in cylinder, mm, and

$D$  = the diameter of the cylinder, mm.

The theoretical volume of the carbon black is evaluated by Eq 2.

$$V_T = m / d_{CB} \quad (2)$$

where:

$V_T$  = the theoretical volume of the weighed sample,  $\text{cm}^3$ ,

$d_{CB}$  = accepted density of carbon black = 1.90  $\text{g}/\text{cm}^3$ , and

$m$  = mass of weighed candidate black sample, g.

The void volume of the candidate carbon black per unit mass (100 g) is given by Eq 3.

$$VV = V_A - V_T \quad (3)$$

where:

$VV$  = void volume of candidate carbon black,  $10^{-5} \text{m}^3/\text{kg}$  ( $\text{cm}^3/100 \text{ g}$ ),

$V_A$  = the (actual) compressed volume of the weighed candidate sample,  $\text{cm}^3$ , (Eq 1), and

$V_T$  = the theoretical volume of the weighed sample,  $\text{cm}^3$ , (Eq 2).

In addition to void volume, a compressed volume index (CVI) can be calculated, which should be expressed to the nearest 0.01 percent, is given by Eq 4.

$$CVI = [VV_{\text{Candidate}} / VV_{\text{Ref\_Standard}}] 100 \quad (4)$$

where:

$VV_{\text{Candidate}}$  = void volume of candidate carbon black,  $10^{-5} \text{m}^3/\text{kg}$  ( $\text{cm}^3/100 \text{ g}$ ), and

$VV_{\text{Ref\_Standard}}$  = void volume of reference carbon black,  $10^{-5} \text{m}^3/\text{kg}$  ( $\text{cm}^3/100 \text{ g}$ ).

NOTE 2—Void volume of SRB B-5 = 56.60  $10^{-5} \text{m}^3/\text{kg}$  ( $\text{cm}^3/100 \text{ g}$ ).

## 11. Report

11.1 Report the following information:

11.1.1 Sample identification,

11.1.2 Sample mass if different from 9.1.2, and

11.1.3 Test result for void volume obtained from a single test determination expressed to the nearest  $10^{-5} \text{m}^3/\text{kg}$  ( $\text{cm}^3/100 \text{ g}$ ).

## 12. Precision and Bias

12.1 A precision and bias statement has not been developed as of this time. This work will be undertaken in the near future.

## 13. Keywords

13.1 compressed volume; structure; void volume