



Standard Test Method for Carbon Black-Void Volume at Mean Pressure¹

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1. Scope

1.1 This test method covers a procedure to measure a carbon black structure property known as Void Volume at mean pressure. Compressed void volumes are obtained by measuring the compressed volume of a weighed sample in a cylindrical chamber as a function of pressure exerted by a movable piston. A profile of void volume as a function of pressure provides a means to assess carbon black structure at varying levels of density and aggregate reduction.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:*²

D1799 Practice for Carbon Black—Sampling Packaged Shipments

D1900 Practice for Carbon Black—Sampling Bulk Shipments

D2414 Test Method for Carbon Black—Oil Absorption Number (OAN)

D3493 Test Method for Carbon Black—Oil Absorption Number of Compressed Sample (COAN)

D4821 Guide for Carbon Black—Validation of Test Method Precision and Bias

D6086 Test Method for Carbon Black—Void Volume (VV)

3. Terminology

3.1 Refer to Sections 4 and 9 for a more complete understanding of the use of these terms in this test method.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *applied pressure, n*—the pressure exerted on a sample mass by a movable piston in a cylindrical chamber, where the load cell or force measuring system is in contact with the movable piston.

3.2.2 *compressed volume (carbon black), n*—the apparent volume that a specified mass of carbon black occupies when it is contained in a specified cylindrical chamber and subjected to a single uniaxial compression at a specified pressure by means of a movable piston.

3.2.3 *geometric mean pressure, n*—the geometric mean of the applied and transmitted pressures at a specific void volume; the geometric mean pressure is defined in Eq 1:

$$\text{Geometric Mean } P_{GM} = (P_a \times P_t)^{0.5} \quad (1)$$

3.2.4 *theoretical volume (carbon black), n*—the volume that a specific mass of carbon black would occupy if there were no void space within the carbon black, and is given by the ratio of mass to skeletal density, where the skeletal density is determined by an accepted test method.

3.2.5 *transmitted pressure, n*—the resulting pressure transmitted through a sample in a cylindrical chamber, where the load cell or force measuring system is in contact with the sample opposite the movable piston, typically via a stationary second piston.

3.2.6 *void volume (carbon black), n*—a measure of the intra-aggregate void space or occluded volume within the primary structure of carbon black, characterized by the irregularity and non-sphericity of carbon black aggregate particles, and expressed as the difference (compressed volume minus theoretical volume) as a function of specified uniaxial compression pressure, and normalized to 100 g mass.

3.2.6.1 *Discussion*—Carbon blacks resist packing, compression, and fracture due to aggregate irregularities and entanglements, size distribution, and aggregate strength resulting from particle-to-particle necks within aggregate branches. Compressed void volume is also affected by reacting forces to the cylinder wall and the piston tip, which in turn depend on factors including sample shape (that is, the ratio of sample height to cylinder diameter) or interfacial area, which can influence the uniformity of the compaction density. Since compressed void volumes as a function of applied pressure are known to be specific to sample mass and cylinder geometry,

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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.

such a compressed void volume is biased due to error in the applied pressure relationship. The applied pressure bias is a result of force losses due to friction between the sample and cylinder wall interface. There is presently no known technique to properly correct applied pressure measurements for an instrument design using a single load cell since friction coefficients (μ) are not constant for carbon black products or applied pressures. For this reason, the most useful technique for comparing compressed void volumes is based on a numerical technique known as mean compaction force or mean pressure. The mean pressure technique requires an instrument design consisting of two load cells to enable the measurement of compressed void volume as a function of applied and transmitted force or pressure. Such a design allows the computation of void volumes at mean pressures, a method which has been demonstrated to minimize the effects of carbon black sample mass and cylinder geometry.

4. Summary of Test Method

4.1 The measured compressed volume (apparent volume) of a weighed dry test sample is obtained in a void volume instrument as a function of specified pressure. The instrument consists of an apparatus which can apply uniaxial compression to a test sample in a cylindrical sample chamber where applied and transmitted forces (or pressures) are measured. The compressed void volume is obtained by subtracting the theoretical volume from the apparent volume, then expressing the result unitized to 100 g mass, as a function of specified mean pressure.

5. Significance and Use

5.1 The void volume of a carbon black expressed as a function of geometric mean pressure, VVGM, is a carbon black structure property. Structure is a generic term that is a function of the shape irregularity and deviation from sphericity of carbon black aggregates. The greater a carbon black resists compression by having substantial aggregate irregularity and non-sphericity, the greater the compressed volume and void volume. Also, the more that a carbon black resists compression, the greater the energy required to compress the sample per unit void volume.

5.2 Structure is a property that strongly influences the physical properties developed in carbon black-elastomer compounds for use in tires, mechanical rubber goods, and other manufactured rubber products. Structure measurements by OAN (Test Method [D2414](#)) and COAN (Test Method [D3493](#)), are based on oil absorption.

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 and transferring samples.

6.4 *Void Volume Instrument*, to be used to measure the compressed volume (apparent volume) of carbon blacks as a

function of applied pressure, from which the void volume is calculated at specified intervals of geometric mean pressure (that is, geometric mean of applied and transmitted pressures). The void volume instrument or device shall conform to the following generic specifications and be capable of operating as outlined in Section 9.

6.4.1 The instrument shall have a rigid framework that contains a cylindrical sample chamber. Hysteresis and elasticity in the framework under the range of applied forces should be accounted for in the displacement measurement.

6.4.2 The cylinder shall have a uniform diameter.

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 measure displacement shall be provided. The compressed volume of any sample is determined by the distance from the end of the piston to the end of the cylinder; this is designated as a “height” in the calculations discussed in Section 10. The sample height and cylinder diameter are used to calculate an apparent sample volume.

6.4.4 Load cells or other suitable force or pressure measurement devices are used to measure the applied and transmitted pressures.

6.4.5 The instrument design shall provide continuous compression at a controlled and constant rate thereby allowing continuous measurements of apparent volume and pressures at specified data intervals.

6.4.6 The instrument uses an electric motor or hydraulic fluid to operate a linear actuator attached to a piston.

6.4.7 The instrument incorporates two load cells to directly measure applied and transmitted forces or pressures.

6.4.8 The instrument design shall provide a means to save and store the compression data up to a minimum of 100 MPa geometric mean pressure for subsequent analysis.

7. Sampling

7.1 Samples of candidate carbon blacks shall be taken in accordance with Practice [D1799](#) or [D1900](#).

8. Calibration

8.1 *Calibration*—Follow the manufacturer’s instructions to set up the instrument and to calibrate the measurement systems. The use of a physical standard such as a calibrated steel plug with traceability is recommended to calibrate or verify the height displacement transducer. A reference load cell with traceability is recommended to calibrate or verify the force transducers.

9. Procedure

9.1 *Sample Preparation*—Dry an adequate sample of the carbon black for at least 1 h in a gravity-convection oven set at $125 \pm 5^\circ\text{C}$, in an open container of suitable dimensions, so that the depth of black is no more than 10 mm. Cool to room temperature in a desiccator before use.

9.2 Weigh a mass of sample specified by the instrument manufacturer, typically 1.000 or 2.000 g, to the nearest 0.1 mg.

9.3 Define the analysis conditions to include void volume measurements at 50.0, 75.0, and 100.0 MPa geometric mean pressure with a compression rate of 1 to 2 MPa/s. In practice this is accomplished by defining a scan from the lowest detection pressure to at least 100 MPa geometric mean pressure.

9.4 Transfer the weighed sample to the instrument. Brush the sample pan and funnel to ensure the entire sample is introduced into the cylinder. Proceed with the test.

9.5 At the end of the test insure residual carbon black has been removed from the cylinder and piston tips.

10. Void Volume Calculations

10.1 The void volume (VV) is calculated from the apparent compressed volume as follows. The apparent compressed volume of the sample is evaluated by Eq 2.

$$V_A = h \times 3.1416 D^2/4 \quad (2)$$

where:

V_A = the apparent compressed volume of the carbon black sample, cm^3 , from a single uniaxial compression.

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

D = the diameter of the cylinder, cm.

10.2 The theoretical volume of the carbon black is evaluated by Eq 3.

$$V_T = m/d_{CB} \quad (3)$$

where:

V_T = the theoretical volume of the carbon black sample, cm^3 ,

d_{CB} = accepted true (skeletal) density of the carbon black = 1.90 g/cm^3 , and

m = mass of the carbon black sample, g.

10.3 The void volume of a carbon black per unit mass (100 g) is given by Eq 4.

$$VV = (V_A - V_T)/m (100) \quad (4)$$

where:

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

V_A = the apparent compressed volume of the carbon black sample, cm^3 , from a single uniaxial compression (Eq 2), and

V_T = the theoretical volume of the carbon black sample, cm^3 , (Eq 3).

NOTE 1—Some carbon blacks have reported skeletal densities of approximately 1.8 to 2.0 g/cm^3 . The accepted skeletal density of rubber carbon black is 1.90 g/cm^3 .

11. Report

11.1 Report the following information:

11.1.1 Sample Identification.

11.1.2 Void volume expressed to the nearest $10^{-6} \text{ m}^3/\text{kg}$ ($0.1 \text{ cm}^3/100 \text{ g}$) measured at 50.0, 75.0 and 100.0 MPa Geometric mean pressures.

11.1.3 Sample mass in grams weighed to nearest 0.1 mg.

11.1.4 Cylinder diameter to the nearest 0.001 mm.

12. Precision and Bias

12.1 *Precision and Bias*—A precision and bias statement has not been developed as of this time.

13. Keywords

13.1 apparent volume; geometric mean pressure; structure; theoretical volume; void volume

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