Designation: D 6086 – 97

Standard Test Methods for Carbon Black—Compressed Volume Index[1]

This standard is issued under the fixed designation D 6086; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover a procedure to measure a carbon black property called the Compressed Volume Index. This index is the ratio (×100) of two void volumes, the void volume of a sample of a candidate carbon black divided by the void volume of a Standard Reference Black, SRB B5. 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 The compressed volume index 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:*

D 1509 Test Methods for Carbon Black—Heating Loss[2]

D 1799 Practice for Carbon Black—Sampling Packaged Shipments[2]

D 1900 Practice for Carbon Black—Sampling Bulk Shipments[2]

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 these test methods.

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.

3.1.2 *compressed volume index (CVI) (carbon black), n*—a normalized void volume defined as the ratio (×100) of the void volume measurements of a candidate carbon black to the void volume of a reference carbon black, SRB B5; both void volumes should be obtained in the same instrument under identical test conditions.

3.1.3 *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.

3.1.4 *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.

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

4. Summary of Test Methods

4.1 The compressed volume instrument used for any testing program is adjusted or calibrated by conducting a compression operation on a specified mass of the reference carbon black, SRB B5. This operation adjusts the compression force (pressure) to give the accepted void volume of this standard carbon black.

4.2 The compressed volume of a weighed dry test sample of a candidate carbon black is obtained in the same compressed volume instrument under calibration test conditions. From the compressed volume, the void volume is obtained by subtracting the theoretical volume. The compressed volume index is obtained for the compressed volumes of candidate and SRB B5 carbon blacks.

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[1] These test methods are 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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[2] *Annual Book of ASTM Standards*, Vol 09.01.

5. Significance and Use

5.1 The void volume of a carbon black expressed in terms of a normalized value, the compressed volume index (CVI), 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, void volume, and CVI.

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 ± 5°C.

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

6.4 *Fume Hood*, with adequate draft.

6.5 *Compressed Volume Instrument*, to be used to measure CVI for candidate and SRB B5 carbon blacks. The compressed volume instrument or device shall conform to the following generic specifications and be capable of operating as outlined in 6.5.1 through 6.5.3.

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

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

6.5.3 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 8. 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 compressed 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. Procedure

8.1 *Sample Preparation*:

8.1.1 Dry an adequate sample of the candidate or SRB B5 carbon black at 125 ± 5°C for 1 h and cool in a desiccator.

8.1.2 Weigh 2.500 g (to the nearest 0.001 g) of the dried carbon black (see also instrument instructions).

8.2 *Instrument Preparation*:

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

8.3 *Calibration Test Pressure (SRB B5)*:

8.3.1 The instrument is set up to conduct tests by adjusting the test end point or calibration pressure to obtain the accepted standard compressed volume for SRB B5.

8.3.2 Place 2.500 ± 0.001 g of SRB B5 into the sample chamber.

8.3.3 Select "Calibrate Pressure" on the instrument control device and start the test operation. The piston will move to the calculated height to attain a calibration compressed volume of 56.60×10^{-5} m^3/kg (10^{-5} $m^3/kg = cm^3/100$ g). The height is held for 20 s and the average pressure is measured. This is repeated two more times for the calibration value of SRB B5. The instrument should be calibrated at the beginning of each test group. After the calibration, a SRB B5 should be tested every 20 tests. A variance of 1.5 % from 100 will indicate recalibration is needed.

8.4 *Evaluating Candidate Carbon Blacks*:

8.4.1 Place the dried and weighed candidate black in the calibrated instrument and secure the sample by closing the gate. Identify the sample (keyboard input) and start the test sequence to apply the predetermined (calibration) pressure for a period of 20 s. The height of the piston will be automatically recorded and used to calculate the CVI for that candidate sample.

9. Compressed Volume Index Calculations

9.1 The CVI is calculated from the measured void volume value 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, 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, cm^3,
d_{CB} = accepted density of carbon black = 1.90 g/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.

$$V(v) = V_A - V_T \quad (3)$$

where:

$V(v)$ = void volume of candidate carbon black, $10^{-5} m^3/kg$ ($cm^3/100$ g), and
m = the mass of the candidate carbon black sample, g.

The CVI, which should be expressed to the nearest 0.01 percent, is given by Eq 4.

$$CVI = [V(v) \text{ candidate} / V(v) \text{ SRB } B5]\ 100 \quad (4)$$

where:

V(v) candidate = candidate carbon black void volume, $10^{-5}m^3/kg$ ($cm^3/100$ g), and

V(v) SRB B5 = SRB B5 void volume = 56.60 $10^{-5}m^3/kg$ ($cm^3/100$ g).

10. Report

10.1 Report the following information:

10.1.1 Sample identification, all samples,

10.1.2 Sample mass if different from 8.4.1, and

10.1.3 Test results for CVI obtained from a single test determination expressed to the nearest 0.01 % (of the SRB B5 value).

11. Precision and Bias

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

12. Keywords

12.1 compressed volume; compressed volume index; structure; void volume

APPENDIX

(Nonmandatory Information)

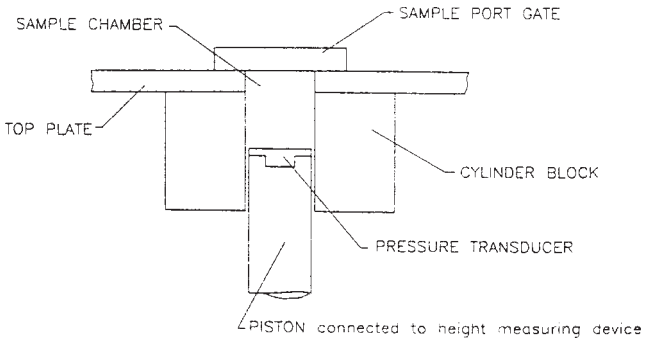
X1. BRIEF DESCRIPTION OF A COMPRESSED VOLUME DEVICE

X1.1 A typical compressed volume instrument or device is shown in Fig. X1.1.[3]

X1.1.1 The CVI instrument meets the specifications in accordance with 6.5. It consists of a rectangular rigid framework with a vertically oriented cylindrical sample chamber. The top plate contains a mechanism for opening and closing the top end or sample port of the chamber with a rigidly held restraining block or gate.

X1.1.2 When open, the sample port allows the loading of a sample of carbon black to be tested. When loaded, a sample rests on the top end of a piston rod that may be propelled upward along the cylindrical sample chamber. Prior to loading, the top of the piston rod is approximately 40 mm below the sample port opening. The piston contains a flat circular end plate that is connected to a pressure transducer and forms the lower end of the sample chamber. The mass of carbon black to be tested rests on top of this piston rod.

X1.1.3 By means of a servo drive motor controlled by a typical motor controller, a ball screw drive is activated to move the piston upward to compress the sample. An encoder determines the magnitude of movement of the piston rod. 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 upper surface or end of the sample port; this is designated as a "height" in the procedure discussed in the main body of these test methods. A test is concluded and the height is measured when a predetermined pressure is attained.



NOTE 1—This is not the only physical design that can be used to determine CV.

FIG. X1.1 Schematic Diagram of a Compressed Volume Instrument Showing Important Components

[3] A compressed volume instrument as indicated in Fig. X1.1 is available from Titan Specialties Inc., P.O. Box 2316, Pampa, TX 79065-2316.