

Standard Test Methods for Rubber Property—Compression Set¹

This standard is issued under the fixed designation D 395; 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.

Section

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

1. Scope

1.1 These test methods cover the testing of rubber intended for use in applications in which the rubber will be subjected to compressive stresses in air or liquid media. They are applicable particularly to the rubber used in machinery mountings, vibration dampers, and seals. Two test methods are covered as follows:

lest method	Occuon
A—Compression Set Under Constant Force in Air	7–10
B—Compression Set Under Constant Deflection in Air	11–14

Test Method

1.2 The choice of test method is optional, but consideration should be given to the nature of the service for which correlation of test results may be sought. Unless otherwise stated in a detailed specification, Test Method B shall be used.

1.3 Test Method B is not suitable for vulcanizates harder than 90 IRHD.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 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 1349 Practice for Rubber—Standard Temperatures for Testing²
- D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets²
- D 3183 Practice for Rubber—Preparation of Pieces for Test Purposes from Products²
- D 3767 Practice for Rubber-Measurement of Dimensions²
- D 4483 Practice for Determining Precision for Test Methods Standards in the Rubber and Carbon Black Industries²

² Annual Book of ASTM Standards, Vol 09.01.

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens³

3. Summary of Test Methods

3.1 A test specimen is compressed to either a deflection or by a specified force and maintained under this condition for a specified time and at a specified temperature.

3.2 The residual deformation of a test specimen is measured 30 min after removal from a suitable compression device in which the specimen had been subjected for a definite time to compressive deformation under specified conditions.

3.3 After the measurement of the residual deformation, the compression set, as specified in the appropriate test method, is calculated according to Eq 1 and Eq 2.

4. Significance and Use

4.1 Compression set tests are intended to measure the ability of rubber compounds to retain elastic properties after prolonged action of compressive stresses. The actual stressing service may involve the maintenance of a definite deflection, the constant application of a known force, or the rapidly repeated deformation and recovery resulting from intermittent compressive forces. Though the latter dynamic stressing, like the others, produces compression set, its effects as a whole are simulated more closely by compression flexing or hysteresis tests. Therefore, compression set tests are considered to be mainly applicable to service conditions involving static stresses. Tests are frequently conducted at elevated temperatures.

5. Test Specimens

5.1 Specimens from each sample may be tested in duplicate (Option 1) or triplicate (Option 2). The compression set of the sample in Option 1 shall be the average of the two specimens expressed as a percentage. The compression set of the sample in Option 2 shall be the median (middle most value) of the three specimens expressed as a percentage.

5.2 The standard test specimen shall be a cylindrical disk cut from a laboratory prepared slab.

5.2.1 The dimensions of the standard specimens shall be:

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¹ These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D11.10 on Physical Testing.

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³ Annual Book of ASTM Standards, Vol 14.04.

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Туре	1^	2 ^{<i>B</i>}
Thickness, mm (in.)	12.5 \pm 0.5 (0.49 \pm 0.02)	$6.0 \pm 0.2 \; (0.24 \pm 0.01)$
Diameter, mm (in.)	29.0 ± 0.5 (1.14 ± 0.02)	$\begin{array}{c} 13.0\ \pm\ 0.2\ (0.51\ \pm\ 0.01) \end{array}$

^AType 1 specimen is used in Test Methods A and B.

^BType 2 specimen is used in Test Method B.

5.2.2 When cutting the standard specimen, the circular die having the required inside dimensions specified in 5.2.1 shall be rotated in a drill press or similar device and lubricated by means of a soap solution. A minimum distance of 13 mm (0.51 in.) shall be maintained between the cutting edge of the die and the edge of the slab. The cutting pressure shall be as light as possible to minimize cupping of the cut edges. The dies shall be maintained carefully so that the cutting edges are sharp and free of nicks.

5.3 An optional method of preparing the standard specimen may be the direct molding of a circular disk having the dimensions required for the test method used and specified in 5.2.1.

NOTE 1—It should be recognized that an equal time and temperature, if used for both the slab and molded specimen, will not produce an equivalent state of cure in the two types of specimen. A higher degree of cure will be obtained in the molded specimen. Adjustments, preferably in the time of cure, must be taken into consideration if comparisons between the specimens prepared by different methods are to be considered valid.

Note 2—It is suggested, for the purpose of uniformity and closer tolerances in the molded specimen, that the dimensions of the mold be specified and shrinkage compensated for therein. A two-plate mold with a cavity 13.0 ± 0.1 mm (0.510 ± 0.004 in.) in thickness and 29.20 ± 0.05 mm (1.148 ± 0.002 in.) in diameter, with overflow grooves, will provide Type 1 specimens for Test Method A and Test Method B. A similar mold but having a cavity of 6.3 ± 0.3 mm (0.25 ± 0.012 in.) in thickness and 13.2 ± 0.1 mm (0.52 ± 0.004 in.) in diameter will provide Type 2 specimens for Test Method B.

5.4 When the standard test specimen is to be replaced by a specimen taken from a vulcanized rubber part of greater thickness than the one indicated in 5.2.1, the sample thickness shall be reduced first by cutting transversely with a sharp knife and then followed by buffing to the required thickness in accordance with Practice D 3183.

5.5 An alternative method of preparing specimens is by plying up cylindrical disks cut from a standard sheet prepared in accordance with Practice D 3182 using the specimen sizes specified in 5.2.1 and cutting as described in 5.2.2, or where a drill press is not available cutting the specimens with a single stroke from a cutting die..

5.5.1 The disks shall be plied, without cementing, to the thickness required. Such plies shall be smooth, flat, of uniform thickness, and shall not exceed seven in number for Type 1 specimens and four in number for Type 2 specimens.

5.5.2 Care shall be taken during handling and placing of the plied test specimen in the test fixture by keeping the circular faces parallel and at right angles to the axis of the cylinder.

5.5.3 The results obtained on plied specimens may be different from those obtained using solid specimens and the results may be variable, particularly if air is trapped between disks.

5.5.4 The results obtained on the specimens prepared by one

of the methods may be compared only to those prepared by the same method.

5.6 For routine or product specification testing, it is sometimes more convenient to prepare specimens of a different size or shape, or both. When such specimens are used, the results should be compared only with those obtained from specimens of similar size and shape and not with those obtained with standard specimen. For such cases, the product specification should define the specimen as to the size and shape. If suitable specimens cannot be prepared from the product, the test method and allowable limits must be agreed upon between the producer and the purchaser.

TABLE 1 Type 1 Precision Results, % Compression Set—Test Method A

Material Mean	Mean	Within Laboratory ^A			Between Laboratory ^A		
Material	Level	S _r	r	(<i>r</i>)	S_R	R	(<i>R</i>)
1	1.73 (%)	0.050	0.142	8.2	0.190	0.54	31.1
2	26.1	0.898	2.54	9.7	2.37	6.71	25.7

^AS_r= within laboratory standard deviation.

r = repeatability (in measurement units).

(r) = repeatability (in percent).

 S_R = between laboratory standard deviation.

R = reproducibility (in measurement units).

(R) = reproducibility (in percent).

6. Conditioning

6.1 Store all vulcanized test specimens or product samples to be tested at least 24 h but not more than 60 days. When the date of vulcanization is not known, make tests within 60 days after delivery by the producer of the article represented by the specimen.

6.2 Allow buffed specimens to rest at least 30 min before specimens are cut for testing.

6.3 Condition all specimens before testing for a minimum of 3 h at $23 \pm 2^{\circ}$ C (73.4 \pm 3.6°F). Specimens whose compression set properties are affected by atmospheric moisture shall be conditioned for a minimum of 24 h in an atmosphere controlled to 50 \pm 5 % relative humidity.

7. Precision and Bias⁴

7.1 These precision statements have been prepared in accordance with Practice D 4483. Please refer to Practice D 4483 for terminology and other testing and statistical concepts.

7.2 Prepared test specimens of two rubbers for Test Methods A and B were supplied to five laboratories. These were tested in duplicate each day on two separate testing days. A test result, therefore, is the average of two test specimens, for both Test Methods A and B.

7.3 One laboratory did not run the Test Method A testing; therefore, the precision for Test Method A is derived from four laboratories.

7.4 The Type 1 precision results are given in Table 1 and Table 2.

⁴ Supporting data are available from ASTM Headquarters. Request RR: D11–1138.

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TABLE 2	Type 1	Precision	Results,	% Comp	oression	Set—Test
		1	Method E	3		

Material Mean		Within Laboratory ^A			Between Laboratory ^A		
Material	Level	S _r	r	(<i>r</i>)	S_R	R	(<i>R</i>)
1	13.7 (%)	0.591	1.67	12.2	1.54	4.36	31.8
2	52.8	0.567	1.60	3.0	5.92	16.8	31.7

^AS_r= within laboratory standard deviation.

r = repeatability (in measurement units). (r) = repeatability (in percent).

S_R= between laboratory standard deviation. R = reproducibility (in measurement units).

(R) = reproducibility (in percent).

7.5 Bias—In test method statistical terminology, bias is the difference between an average test value and the reference or true test property value. Reference values do not exist for these test methods since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

TEST METHOD A—COMPRESSION SET UNDER CONSTANT FORCE IN AIR

8. Apparatus

8.1 Dial Micrometer-A dial micrometer, for measuring specimen thickness, in accordance with Practice 3767, Method A 1.

8.2 Compression Device, consisting of a force application spring and two parallel compression plates assembled by means of a frame or threaded bolt in such a manner that the device shall be portable and self-contained after the force has been applied and that the parallelism of the plates shall be maintained. The force may be applied in accordance with either 8.2.1 or 8.2.2.

8.2.1 Calibrated Spring Force Application—The required force shall be applied by a screw mechanism for compressing a calibrated spring the proper amount. The spring shall be of properly heat-treated spring steel with ends ground and perpendicular to the longitudinal axis of the spring. A suitable compression device is shown in Fig. 1. The spring shall conform to the following requirements:

8.2.1.1 The spring shall be calibrated at room temperature $23 \pm 5^{\circ}$ C (73.4 $\pm 9^{\circ}$ F) by applying successive increments of force not exceeding 250 N (50 lbf) and measuring the corresponding deflection to the nearest 0.2 mm (0.01 in.). The curve obtained by plotting the forces against the corresponding deflections shall have a slope of 70 \pm 3.5 kN/m (400 \pm 20 lbf/in.) at 1.8 kN (400 lbf). The slope is obtained by dividing the two forces above and below 1.8 kN by the difference between the corresponding deflections.

8.2.1.2 The original dimensions of the spring shall not change due to fatigue by more than 0.3 mm (0.01 in.) after it has been mounted in the compression device, compressed under a force of 1.8 kN (400 lbf), and heated in the oven for one week at 70°C \pm 2°C (158 \pm 3.6°F). In ordinary use, a weekly check of the dimensions shall show no greater change than this over a period of 1 year.

8.2.1.3 The minimum force required to close the spring (solid) shall be 2.4 kN (530 lbf).

8.2.2 External Force Application—The required force shall be applied to the compression plates and spring by external

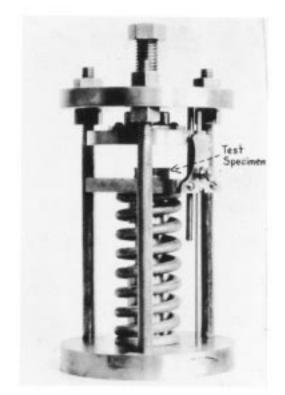


FIG. 1 Device for Compression Set Test, Using Calibrated Spring Loading, Test Method A

means after the test specimen is mounted in the apparatus. Either a calibrated compression machine or known masses may be used for force application. Provision shall be made by the use of bolts and nuts or other devices to prevent the specimen and spring from losing their initial deflections when the external force is removed. The spring shall have essentially the same characteristics as described in 8.2.1, but calibration is not required. A suitable compression device is shown in Fig. 2.

8.3 *Plates*—The plates between which the test specimen is compressed shall be made of steel of sufficient thickness to withstand the compressive stresses without bending. The surfaces against which the specimen is held shall have a chromium-plated finish and shall be cleaned thoroughly and wiped dry before each test.

8.4 Oven, conforming to the specification for a Type IIB laboratory oven given in Specification E 145.

8.4.1 Type IIB ovens specified in Test Method E 145 are satisfactory for use through 70° C. For higher Temperatures Type II A ovens are necessary.

8.4.2 The interior size shall be as follows or of an equivalent volume:

Interior size of air oven:	
min.	300 bt 300 mm by 300mm (12 by 12 by 12 in.)
max.	900 by 900 by 1200 mm (36 by 36 by 48 in.)

8.4.3 Provision shall be made for placing test specimens in the oven without touching each other or the sides of the aging chamber.

8.4.4 The heating medium for the aging chamber shall be air circulated within it at atmospheric pressure.

8.4.5 The source of heat is optional but shall be located in the air supply outside of the aging chamber.

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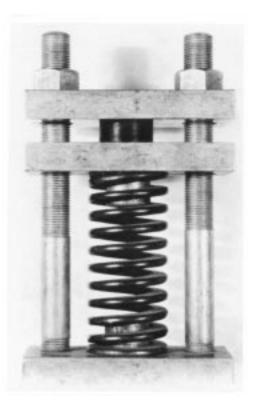


FIG. 2 Device for Compression Set Test, Using External Loading, Test Method A

8.4.6 A suitable temperature measurement device located in the upper central portion of the chamber near the test specimens shall be provided to record the actual aging temperature.

8.4.7 Automatic temperature control by means of thermostatic regulation shall be used.

8.4.8 The following special precautions shall be taken in order that accurate, uniform heating is obtained in all parts of the aging chamber.

8.4.8.1 The heated air shall be thoroughly circulated in the oven by means of mechanical agitation. When a motor driven fan is used, the air must not come in contact with the fan motor brush discharge because of danger of ozone formation.

8.4.8.2 Baffles shall be used as required to prevent local overheating and dead spots.

8.4.8.3 The thermostatic control device shall be so located as to give accurate temperature control of the heating medium. The preferred location is adjacent to the temperature measuring device listed in section 8.4.6.

8.4.8.4 An actual check shall be made by means of maximum reading thermometers placed in various parts of the oven to verify the uniformity of the heating.

9. Procedure

9.1 Original Thickness Measurement— Measure the original thickness of the specimen to the nearest 0.02 mm (0.001 in.). Place the specimen on the anvil of the dial micrometer so that the presser foot will indicate the thickness at the central portion of the top and bottom faces.

9.2 Application of Compressive Force— Assemble the specimens in the compression device, using extreme care to

place them exactly in the center between the plates to avoid tilting. If the calibrated spring device (Fig. 1) is used, apply the compressive force by tightening the screw until the deflection as read from the scale is equivalent to that shown on the calibration curve for the spring corresponding to a force of 1.8 kN (400 lbf). With the external loading device (Fig. 2), apply this force to the assembly in the compression machine or by adding required masses, but in the latter case, take care to add the mass gradually without shock. Tighten the nuts and bolts just sufficiently to hold the initial deflections of the specimen and spring. It is imperative that no additional force be applied in tightening the bolts.

9.3 Test Time and Test Temperature— Choose a suitable temperature and time for the compression set, depending upon the conditions of the expected service. In comparative tests, use identical temperature and heating periods. It is suggested that the test temperature be chosen from those listed in Practice D 1349. Suggested test periods are 22 h and 70 h. The specimen shall be at room temperature when inserted in the compression device. Place the assembled compression device in the oven within 2 h after completion of the assembly and allow it to remain there for the required test period, take the device from the oven and remove the specimens immediately and allow it to cool.

9.4 *Cooling Period*—While cooling, allow the specimens to rest on a poor thermally conducting surface, such as wood, for 30 min before making the measurement of the final thickness. Conduct the cooling period at a standard laboratory temperature of $23 \pm 2^{\circ}$ C (73.4 $\pm 3.6^{\circ}$ F). Specimens whose compression set property is affected by atmospheric moisture shall be cooled in an atmosphere controlled to $50\pm 5\%$ relative humidity.

9.5 *Final Thickness Measurement*—After the rest period, measure the final thickness at the center of the specimen in accordance with 9.1.

10. Calculation

10.1 Calculate the compression set as a percentage of the original thickness as follows:

$$C_A = [(t_o - t_i)/t_o] \times 100 \tag{1}$$

where:

 C_A = Compression set (Test Method A) as a percentage of the original thickness,

 t_o = original thickness (9.1), and

 t_i = final thickness (9.5).

11. Report

11.1 Report the following information:

11.1.1 Original dimensions of the test specimen, including the original thickness, t_{o} ,

11.1.2 Actual compressive force on the specimen as determined from the calibration curve of the spring and spring deflection reading (8.2.1) or as applied by an external force (8.2.2),

11.1.3 Thickness of the test specimen 30 min after removal from the clamp, t_{i} ,

11.1.4 Type of test specimen used, together with the time

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and temperature of test,

11.1.5 Compression set, expressed as a percentage of the original thickness,

11.1.6 Test method used (Test Method A), and

11.1.7 Number of specimens tested.

TEST METHOD B—COMPRESSION SET UNDER CONSTANT DEFLECTION IN AIR

12. Apparatus

12.1 *Dial Micrometer*—A dial micrometer, for measuring the specimen thickness, in accordance with Practice D 3767, Method A 1.

Note 3—For vulcanizates having a hardness below 35 IRHD, the force on the presser foot should be reduced to 0.2 \pm 0.05 N (0.04 \pm 0.01 lbf).

12.2 *Spacer Bars*, to maintain the constant deflection required under Test Method B.

12.2.1 Spacer bars for Type 1 samples shall have a thickness of 9.5 \pm 0.02 mm (0.375 \pm 0.001 in.).

12.2.2 Spacer bars for Type 2 samples shall have a thickness of 4.50 ± 0.01 mm (0.1770 ± 0.0005 in.).

12.3 Compression Device, consisting of two or more flat steel plates between the parallel faces of which the specimens may be compressed as shown in Fig. 3. Steel spacers for the required percentage of compression given in 13.2 shall be placed on each side of the rubber specimens to control their thickness while compressed. The steel surfaces contacting the rubber specimens shall be ground to a maximum roughness of 250 μ m (10 μ in.) and then chromium plated and polished.

12.4 Oven, See section 8.4.

12.5 *Plates*—The plates between which the test specimen is compressed shall be made of steel of sufficient thickness to withstand the compressive stresses without bending. The surfaces against which the specimen is held shall have a chromium-plated finish and shall be cleaned thoroughly and wiped dry before each test.

13. Procedure

13.1 Original Thickness Measurement— Measure the original thickness of the specimen to the nearest 0.02 mm (0.001 in.). Place the specimen on the anvil of the dial micrometer so that the presser foot will indicate the thickness at the central portion of the top and bottom faces.

13.2 Application of Compressive Force— Place the test specimen between the plates of the compression device with the spacers on each side, allowing sufficient clearance for the bulging of the rubber when compressed (Fig. 3). Where a lubricant is applied, it shall consist of a thin coating of a

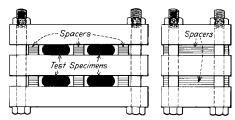


FIG. 3 Device for Compression Set Test Under Constant Deflection, Test Method B

lubricant having substantially no action on the rubber. For most purposes, a silicon or fluorosilicon fluid is suitable. Tighten the bolts so that the plates are drawn together uniformly until they are in contact with the spacers. The amount of compression employed shall be approximately 25 %. A suitable mechanical or hydraulic device may be used to facilitate assembling and disassembling the test fixture.

13.3 Test Time and Temperature—Choose a suitable temperature and time for the compression set, depending upon the conditions of the expected service. In comparative tests, use identical temperature and test periods. It is suggested that the test temperature be chosen from those listed in Practice D 1349. Suggested test periods are 22 h and 70 h. The test specimen shall be at room temperature when inserted in the compression device. Place the assembled compression device in the oven within 2 h after completion of the assembly and allow it to remain there for the required test period, take the device from the oven and remove the test specimen immediately and allow them to cool.

13.4 *Cooling Period*—While cooling, allow the test specimen to rest on a poor thermally conducting surface, such as wood, for 30 min before making the measurement of the final thickness. Maintain the conditions during the cooling period in accordance with 9.4.

13.5 *Final Thickness Measurement*—After the rest period, measure the final thickness at the center of the test specimen in accordance with 13.1.

14. Calculation

14.1 Calculate the compression set expressed as a percentage of the original deflection as follows:

$$C_B = \left[(t_o - t_i) / (t_o - t_n) \right] \times 100$$
(2)

where:

$$C_{\rm B}$$
 = compression set (Test Method B) expressed as percentage of the original deflection,

 t_0 = original thickness of specimen (13.1),

 t_i = final thickness of specimen (13.5), and

 $t_{\rm n}$ = thickness of the spacer bar used.

NOTE 4—Lubrication of the operating surfaces of the compression device is optional while giving more reproducible results; lubrication may somewhat alter the compression set values.

15. Report

15.1 Report the following information:

15.1.1 Original dimensions of the test specimen including the original thickness, t_{0} ,

15.1.2 Percentage compression of the specimen actually employed,

15.1.3 Thickness of the test specimen 30 min after removal from the clamp, t_{i} ,

15.1.4 Type of test specimen used, together with the time and temperature of test,

15.1.5 Whether or not the surfaces of the compression device are lubricated. If they are, what type lubrication was used,

15.1.6 Compression set, expressed as a percentage of the original deflection,

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15.1.7 Test method used (Test Method B), and

15.1.8 Number of specimens tested.

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