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Standard Test Method for
Rubber Property—Compression Set at Low Temperatures[1]

This standard is issued under the fixed designation D 1229; 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.

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

~~^{ϵ1} Note—Keywords were added and equations in 9.1 were corrected editorially in March 1998.~~

1. Scope

1.1 This test method covers evaluation of the ability of vulcanized rubbers that have been compressed at room temperature and then subjected to low temperature (air or carbon dioxide atmosphere), to recover from deformation when taken from the clamping device while still at the low temperature.

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

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:*

D 395 Test Methods for Rubber Property—Compression Set[2]

D 832 Practice for Rubber Conditioning for Low-Temperature Testing[2]

D 3767 Practice for Rubber—Measurement of Dimensions[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

3. Summary of Test Method

3.1 At room temperature, a test specimen is compressed to 25 % of its original thickness and exposed for a specified time at a specified low temperature.

3.2 Upon release from compression, the specimen is allowed to recover while remaining at the test temperature.

3.3 The residual deformation of the specimen is measured 10 s and 30 min after removal from the compressive device.

3.4 Compression set of the specimen is calculated according to the equations in 9.1.

4. Significance and Use

4.1 Rubber products, such as hydraulic seals on aircraft, submarine hatch gaskets, and hydraulic brake cups, may be exposed to varying temperature extremes. This test method provides a means of determining the extent to which recovery from compression at normal ambient temperatures is inhibited upon release of the compressive force during subsequent exposure at low temperatures.

5. Compression Set

5.1 For the purpose of this test method, compression set of vulcanizates shall be the loss in thickness of the specimen expressed as a percentage of the original deflection. This set, unlike conventional compression set at elevated temperatures, is temporary since the specimen will regain its original dimensions when brought back to room temperature or slightly above.

6. Apparatus

6.1 *Compression Set Jigs*, with suitable steel spacer bars in accordance with Method B of Test Methods D 395.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

TABLE 1 Type 1 Precision, Compression Set, %

NOTE 1—

S_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).

Materials	Measured At	Range of Values, %	Within Laboratories			Between Laboratories		
			S_r	r	(r)	S_R	R	(R)
Three unknown compounds	10 s	75 to 104	0.60	1.70	1.9[A]	2.60	7.4	8.2[A]
Three unknown compounds	30 min	42 to 93	1.60	4.5	6.6[A]	5.60	15.8	23.2[A]

[A] An estimated value of relative repeatability (r) and reproducibility (R) using the mid-point of the range.

6.2 *Dial Micrometer*, as described in Practice D 3767.

6.3 *Cold Box*, cooled by solid carbon dioxide (Dry Ice), liquid carbon dioxide, liquid nitrogen, or mechanically refrigerated, preferably by the top-opening type, and capable of temperature control within ±1°C (1.8°F) of the specified testing temperature as prescribed in Practice D 832. The test chamber shall be equipped with a vise, "C" clamp, or other suitable device for holding the compression set jig.

7. Test Specimen

7.1 The standard test specimen shall be a cylindrical disk 29.0 ± 0.5 mm (1.14 ± 0.02 in.) in diameter and 12.5 ± 0.5 mm (0.49 ± 0.02 in.) in thickness, as specified in Test Methods D 395.

8. Procedure

8.1 *Original Thickness Measurement* —Measure the original thickness, t_o, at the center of the specimen to the nearest 0.02 mm (0.001 in.).

8.2 *Application of Load*—Place a test specimen between clean, unlubricated plates of the compression device with the spacers on each side of it. Use only one specimen with each pair of steel plates, and place in the center between the plates. Tighten the bolts so as to draw the plates together uniformly until they are in contact with the spacers. The compression employed shall be 25 % for all hardnesses.

8.3 Within 30 min after the jigs are loaded, place them in the low-temperature cabinet, and maintain at the service temperature for which the vulcanizates are being evaluated. In the event the service temperature is unknown, the following temperatures are suggested: –40°C (–40°F) and –55°C (–67°F). The conditioning period shall be either 22 or 94 h. At least 1 h before the conditioning period is over, place the dial micrometer in the test chamber and clamp one of the set jigs in the vise or "C" clamp provided in the low-temperature chamber. Use suitable gloves for all operations in the test chamber. At the end of the conditioning period, remove the nuts from the jig, after which release the vise or "C" clamp, and start the stop watch simultaneously. Measure the thickness of the specimens 10 s and again 30 min after release from the vise or "C" clamp and record as t_{10} and t_{30}, respectively. Since the test is conducted at a specific temperature, within ±1°C (1.8°F), the schedule of opening the jigs shall be such that the test chamber will stay within the permissible variations in temperature.

NOTE 1—It is convenient when conducting many tests to have the dial micrometer outside the chamber and have an extended insulated stem pass through the cover or the top to measure the thickness of specimens after release from compression.

NOTE 2—Slowly crystallizing rubbers may need to be tested at –10°C (14°F) or –25°C (–13°F), with a conditioning period of 166 h.

NOTE 3—In the event that the dail micrometer gage being used is unacceptable for use at a low temperature, it may be placed outside of the test chamber. The test specimen is to remain in the cold box until it is measured. Immediately upon removal from the chamber the first measurement, t_{10}, is to be taken and the specimen immediately returned to the cold box. At the 30 min time interval the specimen is again removed from the chamber and immediately measured to obtain the value for t_{30}.

8.4 *Check Test*—Run tests in duplicate. The results should agree within 5.0 %.

9. Calculation

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

$$C = [(t_0 - t_{10}) / (t_0 - t_s) \times 100 \tag{1}$$

$$C = [(t_0 - t_{10}) / (t_0 - t_s) \times 100 \tag{1}$$

or

$$C = [(t_0 - t_{30}) / (t_0 - t_s) \times 100 \tag{2}$$

$$C = [(t_0 - t_{30}) / (t_0 - t_s) \times 100 \tag{2}$$

where:

C = compression set expressed as a percentage of the original deflection, and

t_s = thickness of the spacer bar used.

10. Report

10.1 The report shall include the following:

10.1.1 The original thickness of the test specimen,

10.1.2 The percentage compression of the specimen actually employed,

10.1.3 The thickness of the test specimen 10 s, t_{10}, and 30 min, t_{30}, after removal from the clamp, and

10.1.4 The compression set expressed as a percentage of the original deflection.

11. Precision and Bias [3]

11.1 This precision and bias section was prepared in accordance with Practice D 4483. Refer to Practice D 4483 for terminology and other statistical calculation details.

11.2 Although prepared in accordance with Practice D 4483, the data generated for this test method were obtained prior to the adoption of Practice D 4483 in 1985. No records exist for the original test program. The values of within-and-between laboratory standard deviation have been used to construct Table 1. A Type 1 precision was evaluated.

11.3 Compression set test specimens were prepared in one laboratory from three different rubber compounds. Ten laboratories tested two specimens from each of these compounds on 4 different days. The precision statements are based on the average of two measurements and are expressed in terms of the compression set, in percent.

11.4 The precision of this test method may be expressed in the format of the following statements which use what is called an "appropriate value" or *r, R, (rr),* or (*R*), that is, that value to be used in decisions about test results (obtained with the test method). The *appropriate value* is that value of *r* or *R* associated with a mean level in Table 1 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

11.5 *Repeatability*—The repeatability, *r*, of this test method has been established as the appropriate value tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

11.6 *Reproducibility*— The reproducibility, *R*, of this test method has been established as the appropriate value tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

11.7 Repeatability and reproducibility expressed as a percentage of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

11.8 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias, therefore, cannot be determined.

12. Keywords

12.1 compression set; low temperature

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[3] Supporting data are available from ASTM Headquarters. Request RR: D-11-1042.