



Test Method for Vulcanized Rubber and Thermoplastic Elastomer— Determination of Force Decay (Stress Relaxation) in Compression¹

This standard is issued under the fixed designation D 6147; 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 approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This standard specifies two methods for determining the decrease in counterforce exerted by a test specimen of vulcanized rubber or thermoplastic elastomer which has been compressed at a constant deformation under specified conditions of time and temperature.

1.2 This document was developed based on testing in air and liquids.

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 573 Standard Test Method for Rubber—Deterioration in an Air Oven²
- D 1349 Standard Practice for Rubber—Standard Temperatures for Testing²
- D 3182 Standard Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets²
- D 3767 Standard Practice for Rubber—Measurement of Dimensions²
- D 4483 Standard Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²
- E 145 Specification for Gravity—Convection and Forced-Ventilation Ovens³

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *Compressive Stress, n* —the time-dependent force necessary to maintain a constant compressive strain, divided by the original cross-sectional area over which the force is applied.

3.1.2 *Force Decay, n* —the decrease in stress which has occurred after a specified time-interval, during application of a constant deformation, expressed as a percentage of the stress at the commencement of that time-interval. (Stress relaxation is a synonym.)

4. Summary of Test Method

4.1 *Method A*—The test specimen is compressed at the test temperature, and it is maintained at this temperature throughout the test period, all force measurements being made at the test temperature.

4.2 *Method B*—The test specimen compression and the initial measurement of the counterforce take place at $23 \pm 2^\circ\text{C}$ ($73 \pm 4^\circ\text{F}$); the test specimen is then stored in a chamber controlled at the test temperature, but it is removed from the chamber for each of the subsequent force measurements, which are made at $23 \pm 2^\circ\text{C}$ ($73 \pm 4^\circ\text{F}$).

4.3 The two methods, A and B, of carrying out the measurement do not give the same values of force decay and comparison of values obtained from the two methods must be avoided. The method selected for use depends on the purpose of the test. Thus, for fundamental studies and in applications where sealing at elevated temperatures is a problem, method A may be preferred, and in applications where temperature cycling from normal to an elevated temperature is a problem, method B may be preferred.

5. Significance and Use

5.1 When a constant strain is imposed on rubber, the force necessary to maintain that strain is not constant but decreases with time; this phenomenon is called force decay (stress relaxation). Conversely, when rubber is subjected to a constant stress, an increase in the deformation takes place in time; this behavior is called creep. These phenomena are of practical significance in rubber articles, such as seals and gaskets.

5.2 The processes responsible for force decay may be either physical or chemical in nature, and under all normal conditions both processes will occur simultaneously. However, at ambient or low temperatures and/or short times, force decay is dominated by physical processes, while at elevated temperatures and/or long times, chemical processes are dominant. Hence, it is neither safe to extrapolate time/force decay curves in order to predict force decay after periods considerably longer than

¹ This practice is under the jurisdiction of ASTM Committee D-11 on Rubber and is the direct responsibility of Subcommittee D11.14 on Time and Temperature—Dependent Physical Properties.

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

³ *Annual Book of ASTM Standards*, Vol 14.02.

those covered by the test, nor to use tests at higher temperatures as accelerated tests to give information on force decay at lower temperatures.

5.3 In addition to the need to specify the temperature and time-intervals in a force decay test, it is also necessary to specify the initial stress and the previous mechanical history of the test specimen, since these may also influence the measured force decay, particularly in rubbers containing fillers.

6. Apparatus

6.1 *Compression Device*, consisting of two parallel, flat plates, between the faces of which the test specimens are compressed. The two devices shown schematically in Fig. 1 and Fig. 2 were found to be acceptable through interlaboratory testing. Material of construction of the jig shall not be subject to attack from the test environment (for example acidic oil).

6.2 The finish of the surface of the compression plates shall not exceed $0.6\ \mu\text{m}$ (24 micro inches) arithmetical mean deviation from the mean line of the profile. The plates shall be sufficiently rigid to withstand the stress without bending, and of sufficient size to ensure that the whole of the compressed test specimen is within the area of the plates.

6.3 The compression device shall be connected with suitable equipment for compressing the test specimen to the specified compression within 30 s. It shall be capable of setting and maintaining the compression during the whole duration of test and shall be such that it can be kept in an oven at the specified test temperature. Care shall be taken to ensure that there is no loss of heat from the test specimen, for example by conduction through metal parts which are connected with the outside of the oven.

6.4 *Counterforce measuring device*, capable of measuring compression forces in the desired range with an accuracy of $\pm 1\%$. The device may be such as to contain the test specimens during the whole duration of the test, in which case continuous measurements are possible. Alternatively, a testing machine may be used in which the counterforce is measured after prescribed time-intervals on test specimens, compressed in a suitable jig, by applying a slight increase in the compression of the test specimen. This additional compression shall be as small as possible and in no case more than a force of 1N or half percent ($\frac{1}{2}\%$) of original load for balance type machine and 0.1 mm (0.004 in) or 2% of original deflection for stress-strain type machines, in a time not greater than 30 s after commencing the additional compression.

6.5 *Oven*, with temperature control to maintain the specified temperature within the prescribed tolerances. Satisfactory circulation of the air shall be maintained by means of a fan (D 573 or E 145).

6.6 *Temperature reading device (optional)*, with for example a thermocouple as the sensing element, mounted in a central position in the oven.

7. Test Specimen

7.1 *Type of Test Specimen*—The test specimen shall be one of the following:

7.1.1 A cylindrical disc of diameter $13.0 \pm 0.5\ \text{mm}$ ($0.512 \pm 0.020\ \text{in}$) and thickness $6.3 \pm 0.3\ \text{mm}$ ($0.248 \pm 0.012\ \text{in}$).

7.1.2 Cylindrical disc of diameter $29.0 \pm 0.5\ \text{mm}$ ($1.142 \pm$

$0.020\ \text{in}$) and thickness $12.5 \pm 0.5\ \text{mm}$ ($0.492 \pm 0.020\ \text{in}$).

7.1.3 Washer of $12.5 \pm 0.5\ \text{mm}$ ($0.492 \pm 0.020\ \text{in}$) inside diameter and $19.0 \pm 0.5\ \text{mm}$ ($0.748 \pm 0.020\ \text{in}$) outside diameter with $2.0 \pm 0.2\ \text{mm}$ ($0.080 \pm 0.008\ \text{in}$) thickness. Within the sample test specimens, thickness variation shall be within $0.025\ \text{mm}$ ($0.001\ \text{in}$).

7.1.4 Other test specimen sizes may be used as dictated by end use.

7.2 The first three test specimens described above were evaluated in interlaboratory testing. The washer type test specimen (7.1.3) is preferred.

7.3 *Preparation of Test Specimens*—Test specimens may be prepared by taking a sample of the compound or material of interest. If this is a compound it shall be cured and molded in accordance with D 3182, or prepared by cutting from molded sheets. Cutting is carried out by means of a sharp, rotating, circular die or revolving knife, lubricated with soapy water, and brought carefully into contact with the rubber. Alternatively, the die or knife is kept stationary and the rubber rotated against it. The rubber is mounted on suitable backing material and the cutting pressure is kept small enough to avoid “cupping” of the cut surface.

7.4 Due to shape factor effect, equivalent results may not be obtained for the different test specimen shapes described above.

7.5 *Number of Test Specimens*—At least three test specimens shall be used for each test.

7.6 *Time-Interval Between Vulcanization and Testing*—The time-interval between vulcanization and testing shall be at least 24 h at ambient conditions.

8. Procedure

8.1 Carefully clean the operating surfaces of the compression device. The test specimen surface shall be free from mold release agent or dusting powder.

8.2 Measure the thickness of each test specimen at the central portion with an accuracy of $0.01\ \text{mm}$ ($0.0004\ \text{in}$). The thickness shall be determined according to D 3767.

8.3 Method A

8.3.1 Preheat the compression device to the test temperature.

8.3.2 Preheat the test specimen to the test temperature in accordance with D 1349. A preheating period of 30_{-0}^{+2} min. is recommended.

8.3.3 Compress the preheated test piece by $25 \pm 1\%$ in the compression device (6.1) at the test temperature; use a compression of $15 \pm 1\%$ if a compression of 25% cannot be obtained. Apply the compression within 30 s. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which may be used for measurement of counterforce, mentioned in 6.4).

8.3.4 After the compression has been applied for 30_{-0}^{+2} min., measure the counterforce with an accuracy of $\pm 1\%$, still at the test temperature.

8.3.5 Repeat the measurement of the counterforce after different times, according to 10.1. Take all measurements at the test temperature.

8.3.6 Due to operator variability, all counterforce

Item	Description
A	Sample
B	Spacer (Specified by Customer)
C	Nut
D	Flange
E	Pillar
F	Clamp
G	Outer Spring Spacer
H	Spacer
I	Adjusting Screw
J	Contact Housing Assy.
K	Inner Spring Spacer
L	Springs
M	Platen

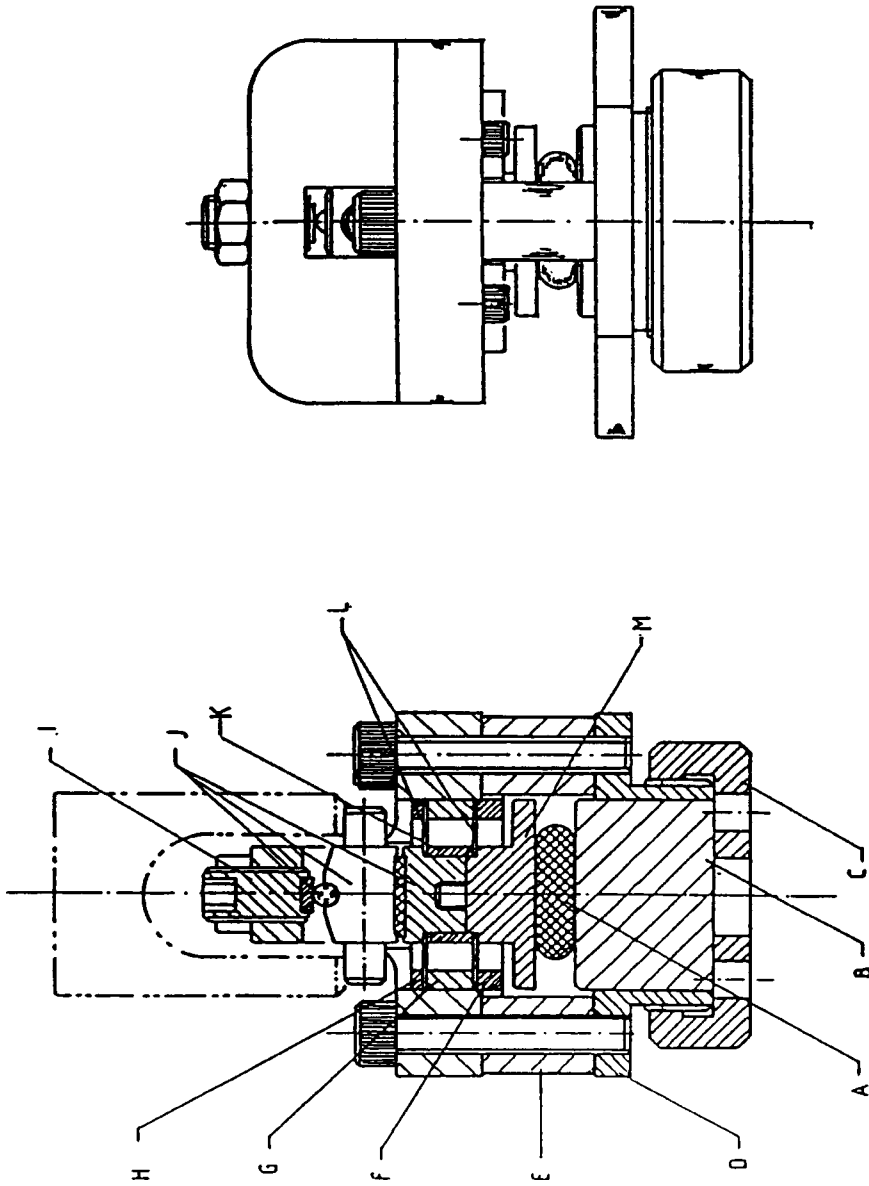


FIG. 1 Shawbury-Wallace Stress Relaxation Fixture

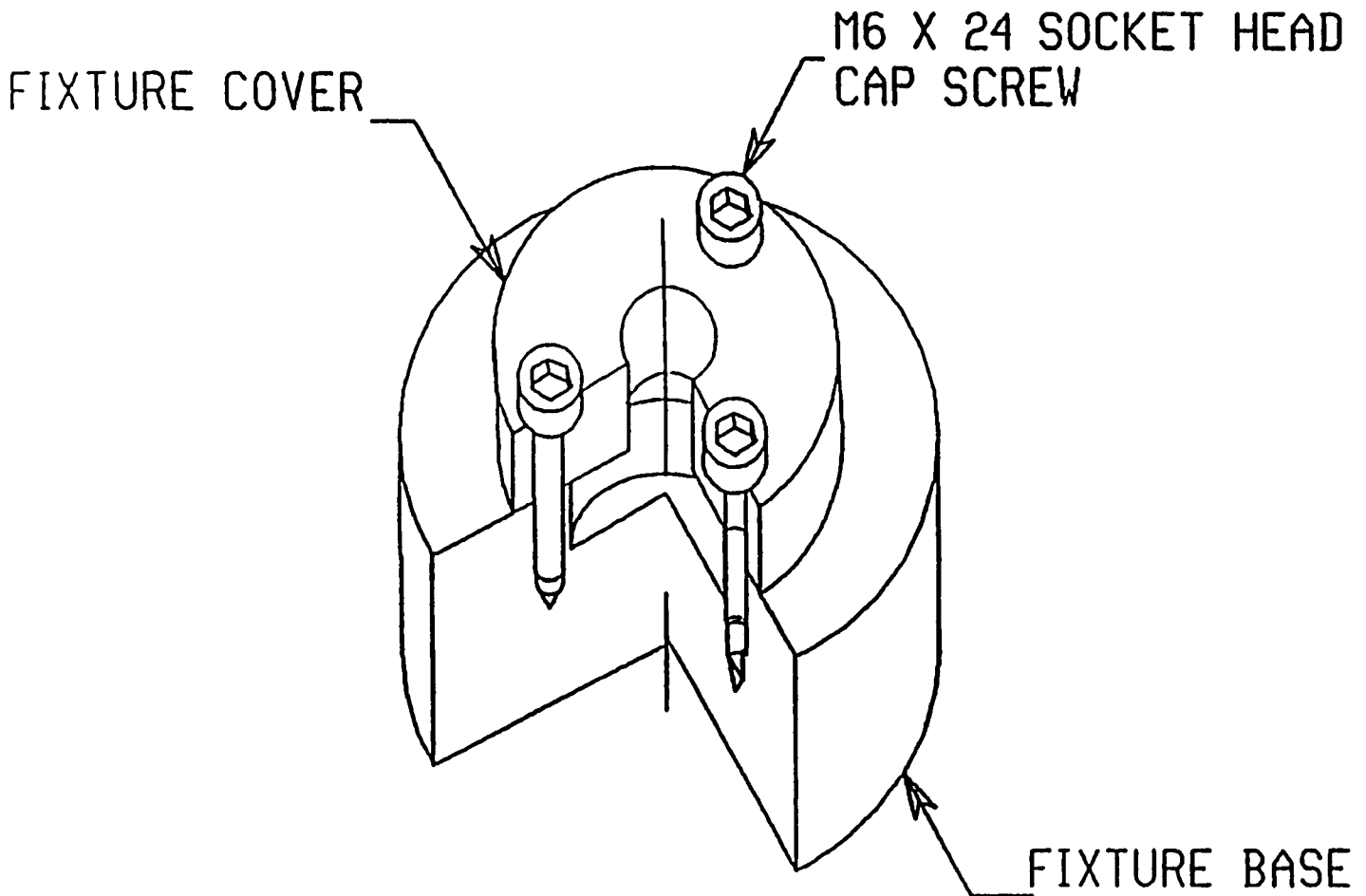


FIG. 2 Wykeham Farrance Stress Relaxation Fixture

measurements shall be performed three (3) times for each specimen and the median value used.

8.4 Method B

8.4.1 Compress the test piece by $25 \pm 1\%$ at $23 \pm 2^\circ\text{C}$ ($73 \pm 4^\circ\text{F}$); a compression of $15 \pm 1\%$ may be used when a compression of 25% cannot be obtained. Apply the compression within 30 s. When reached, the final compression shall be fixed and maintained during the entire test period (apart from the further small compression which may be used for measurement of counterforce, mentioned in 6.4).

8.4.2 After the compression has been applied for 30^{+2}_{-0} min. measure the counterforce with an accuracy of $\pm 1\%$, still at the standard laboratory temperature.

8.4.3 Store the compressed test specimen in the oven (6.5) at the specified elevated temperature.

8.4.4 When making measurements of counterforce after the times specified, remove the apparatus from the oven, maintain it at the standard laboratory temperature for $2^{+0}_{-0.2}$ h, determine the counterforce and then return to the test environment for a further time. It is important that the apparatus and test specimen reach thermal equilibrium within 2 h, and force cooling may be necessary. In method B, the 2 h required after each time-interval, to attain standard laboratory temperature, shall be included in the duration of test.

8.4.5 Repeat the measurement of the counterforce after

different times, according to 10.1. Take all measurements at ambient temperature.

8.4.6 Due to operator variability, all counterforce measurements shall be performed three (3) times for each test specimen and the median value used.

9. Liquid Immersions Testing Procedure

9.1 In some applications force decay testing under liquid immersion conditions is necessary. Under these conditions the following additional preparation of the jig is necessary.

9.2 Apply a lubricant to the top and bottom surface of the jig. It shall consist of a thin coating of the test fluid having substantially no action on the rubber.

9.3 The test specimen/test fixture shall be completely immersed in liquid under consideration. The immersed test specimen shall be in the compressed condition, similar to testing in air as discussed in Section 8. Care shall be taken while performing force decay under liquid immersions (especially at elevated temperatures) to avoid hazardous test conditions. Environmentally safe practices shall be followed while performing liquid immersion testing. If the effect of liquid on inside and outside surfaces of rubber is desired, the washer type test specimen (7.1.3), is recommended. In this case the fixture used shall have an inlet and vent hole of at least 3.2 ± 0.3 mm (0.125 ± 0.012 in) diameter to allow flow of liquid

towards inside diameter of the sample.

9.4 Follow test procedures in 8.3 for Method A or 8.4 for Method B.

10. Duration and Temperature of Test

10.1 *Duration of Test*—The duration of the test shall be mutually agreed upon by the customer and the supplier. A test period of $168 \frac{0}{-2}$ h has been found suitable for many elastomers. The test period begins after the initial compression. If longer testing times are needed, a logarithmic time-scale may be used. In method B, the 2 h required after each time-interval, to attain standard laboratory temperature, shall be included in the duration of test.

10.2 *Temperature of Test*—The temperature of test shall be chosen from D 1349. Unless for technical reasons another temperature is required, the ambient temperature shall be $23 \pm 2^{\circ}\text{C}$ ($73 \pm 4^{\circ}\text{F}$).

11. Calculation of Test Results

11.1 The compression force decay (stress relaxation), $R(t)$, after a specified duration of test, t , expressed as a percentage of the initial counterforce, is calculated by Eq. 1.

$$R(t) = \frac{F_0 - F_t}{F_0} \times 100 \quad (1)$$

where:

F_0 = initial counterforce measured 30 min. after compression of the test specimen, and

F_t = counterforce measured after the specified duration of test.

11.2 The median value of the calculated results for compression force decay shall be used.

11.3 For some applications, it may be more useful to calculate compressive force retention values, i.e. $FR(t)$ at various times after the compression is applied, rather than force decay values. This may be calculated by Eq. 2

$$FR(t) = 100 - R(t) \quad (2)$$

where:

$FR(t)$ = force retention expressed as a percent, after a specified duration of test.

11.4 The compressive force retention values may be presented graphically as a function of time. The use of a logarithmic scale for the time-axis may facilitate representation of the test data.

12. Test Report

12.1 The test report shall include the following information:

12.1.1 Sample details:

12.1.1.1 A full description of the sample and its origin.

12.1.1.2 Compound details, cure time and temperature and date of cure, where appropriate.

12.1.1.3 Method of preparation of test specimens from sample.

12.1.2 Test Method and Test Details:

12.1.2.1 The number of this standard

12.1.2.2 The method used, A or B

12.1.2.3 The standard laboratory temperature, if other than $23 \pm 2^{\circ}\text{C}$ ($73 \pm 4^{\circ}\text{F}$)

12.1.2.4 The duration and temperature of test

12.1.2.5 The type and dimensions of test specimens

12.1.2.6 Nature of lubricant, if used

12.1.2.7 A description of the principles of the testing device (oven included)

12.1.2.8 The method used for measuring the counterforce

12.1.2.9 Any non-standardized procedures, for example, mechanical condition.

12.1.3 Test Results:

12.1.3.1 The number of test specimens tested, if more than three

12.1.3.2 The median value of the test results, expressed in accordance with Section 11.

12.1.4 Date of test.

12.2 Refer to Table 1 for a recommended table for reporting test results.

13. Precision and Bias ⁴

13.1 The precision results for this standard have been prepared in accordance with Practice D 4483 with certain exceptions as noted below. Please refer to this practice for terminology and other testing and statistical concept explanations.

13.2 The precision results in this precision and bias section give an estimate of the precision for this test standard with the materials (rubbers) used in the particular program as described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to that group of materials and the specific protocols of the test method.

13.3 A Type 1 interlaboratory test program (ITP) was conducted in 1994. The general plan for the testing involved seven participating laboratories conducting parallel tests on two test specimens: a washer shaped specimen and a button specimen. The exceptions to the normal D 4483 protocol are: (1) only one material (rubber compound) was tested, rather than the recommended 3 or 4, and (2) the within-lab testing was conducted on a general plan with three test specimens on one day rather than the specified D 4483 repeat testing of n test specimens *each* on Day 1 and Day 2. Repeatability as given in the tables of precision results therefore is a within-day between replicate value and not the usual Day 1 - Day 2 repeatability. A test result is defined as one measurement value. The relaxation was carried out in air at 70°C .

13.4 An extensive analysis of the data for this ITP was conducted to detect any outlier laboratories that depart significantly from the remaining laboratories. This analysis identified a core group of four laboratories (among the seven) for the washer test specimen and a core group of five laboratories for the button test specimen. Both of these core groups have better between-lab agreement than the entire seven laboratories. The precision results as given in Table 2 and Table 3 are based on the data from these core group laboratories.

13.5 The precision results for Method B are given in Table 2 for the washer test specimen and are based on three replicates

⁴ Supporting data are available from ASTM Headquarters. Request RR: D11-1086

TABLE 2 PRECISION TABULATION - FOR R(t), %; WASHER TEST SPECIMEN^A METHOD B; IN AIR @ 70C

RELAX TIME	MEAN	WITHIN-LAB			BETWEEN-LAB		
		Sr	r	(r)	SR	R	(R)
24 HR	18.9	3.17	8.87	47.0	3.73	10.4	55
96 HR	26.0	2.69	7.53	29.0	3.96	11.1	43
168 HR	29.0	2.17	6.07	20.9	4.31	12.1	42

^A Precision given for three relaxation times for one rubber compound (material) 3 replicates per relaxation time, 5 laboratories participating

Within-lab precision is 'within-day between test specimen variation', not usual between day variation

Sr = within-lab (repeatability) standard deviation; r = repeatability, in test units, % for R(t); (r) = repeatability in relative units, percent of a percent; SR = between-lab (reproducibility) standard deviation; R = reproducibility in test units, % for R(t); (R) = reproducibility in relative units, percent of a percent

TABLE 3 PRECISION TABULATION FOR R(t), % FOR BUTTON TEST SPECIMEN^A METHOD B; IN AIR @ 70C

RELAX TIME	MEAN	WITHIN-LAB			BETWEEN-LAB		
		Sr	r	(r)	SR	R	(R)
24 HR	15.5	1.83	5.12	33.1	2.43	6.80	44.0
96 HR	21.1	2.10	5.89	27.9	2.40	6.73	31.9
168 HR	22.7	2.32	6.52	28.7	3.43	9.62	42.3

^A Precision given for three relaxation times for one rubber compound (material) 2 replicates per relaxation time, 5 laboratories participating

Within-lab precision is 'within-day between test specimen variation', not usual between day variation

Sr = within-lab (repeatability) standard deviation; r = repeatability, in test units, % for R(t); (r) = repeatability in relative units, percent of a percent; SR = between-lab (reproducibility) standard deviation; R = reproducibility in test units, % for R(t); (R) = reproducibility in relative units, percent of a percent

(on one day) per relaxation time. The precision results in Table 3 for the button test specimen are based on only two replicates per relaxation time.

13.6 *Repeatability*—The repeatability for this test has been

established as the values in Table 2 and Table 3 for the indicated relaxation times and test conditions. Two test results that differ by more than the repeatability values in Table 2 and Table 3 must be considered to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

13.7 *Reproducibility*—The reproducibility of this test has been established as the values in Table 2 and Table 3 for the indicated relaxation times and test conditions. Two test results from different laboratories that differ by more than the reproducibility values in Table 2 and Table 3 must be considered to represent different sample populations. Such a decision dictates that some appropriate action be taken.

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

14. Keywords

14.1 stress relaxation; force relaxation; load decay; stress decay; compressive stress relaxation; reaction force; force retention; force decay; rubber; thermoplastic elastomer

NOTICE: This standard has either been superseded and replaced by a new version or discontinued. Contact ASTM International (www.astm.org) for the latest information.



**TABLE 1 Recommended Table for Reporting Test Results
COMPRESSION FORCE DECAY (STRESS RELAXATION) RESULTS**

NAME OF TEST LAB: _____ ASTM D _____, METHOD A OR B (Circle One)
 OPERATOR: _____ SPECIMEN SIZE: _____
 MATERIAL: _____ FIXTURE TYPE: _____
 CURE TIME/TEMP: _____ % COMPRESSION: _____
 LIQUID MEDIA: _____ TEST TEMPERATURE: _____

ADDITIONAL COMMENTS: _____

DATE	DURATION OF HEAT EXPOSURE/ TEST TIME	REACTION FORCE (N or Lbf)			COMPRESSION FORCE DECAY (STRESS RELAXATION) $R(t) = \frac{F_0 - F_t}{F_0} \times 100$			R(t) MEDIAN	R(t) STANDARD DEVIATION	FORCE RETENTION $FR(t) = 100 - R(t)$
		Test Specimen 1	Test Specimen 2	Test Specimen 3	Test Specimen 1	Test Specimen 2	Test Specimen 3			
	0									
	24 Hrs.									
	48 Hrs.									
	72 Hrs.									
	96 Hrs.									
	120 Hrs.									
	144 Hrs.									
	168 Hrs.									

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