Designation: D1329 – 16

Standard Test Method for Evaluating Rubber Property—Retraction at Lower Temperatures (TR Test)[1]

This standard is issued under the fixed designation D1329; 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 This test method describes a temperature-retraction procedure for rapid evaluation of crystallization effects and for comparing viscoelastic properties of rubber and rubber-like materials at low temperatures. This test method is useful when employed in conjunction with other low-temperature tests for selection of materials suitable for low-temperature service.

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:*[2]

D832 Practice for Rubber Conditioning For Low Temperature Testing

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

3. Summary of Test Method

3.1 This test method is carried out by (*1*) elongating the specimen, (*2*) locking it in the elongated condition, (*3*) freezing it to a state of reduced elasticity, (*4*) releasing the frozen specimen and allowing it to retract freely while raising the temperature at a uniform rate, (*5*) measuring the length of the specimen at regular temperature intervals while it is retracting, and (*6*) computing the percentage retraction at these temperatures from the data obtained. In practice, the temperatures corresponding to 10 % and 70 % retraction are of particular importance, and are designated as TR10 and TR70, respectively.

4. Significance and Use

4.1 The difference between the temperature at which a vulcanizate retracts 10 % (TR10) and the temperature at which a vulcanizate retracts 70 % (TR70) increases as the tendency to crystallize increases.

4.2 TR70 correlates with low-temperature compression set.

4.3 TR10 has been found to correlate with brittle points in vulcanizates based on polymers of similar type.

4.4 In general, the retraction rate is believed to correlate with low-temperature flexibility of both crystallizable and noncrystallizable rubbers.

5. Apparatus

5.1 *Specimen Rack,* designed to maintain a slight tension on the specimen of 7 to 21 kPa (1 to 3 psi), and to permit it to be stretched and anchored at any elongation desired up to a maximum to 350 %. Means of measuring the length of the specimen at any time during the test within an accuracy of ±1 mm (±0.04 in.) shall be provided. The rack may be designed to hold a number of specimens at the same time.

5.2 *Insulated Cooling Bath,* equipped with stirrer, thermometer, and an immersion heater. A rheostat shall be included in the heater circuit. A suitable thermocouple-potentiometer measuring system may be substituted for the thermometer.

5.3 *Temperature Measurement,* may be conducted in one of two ways: (*a*) a typical glass thermometer with appropriate range and sensitivity (±1°C (±2°F)); or (*b*) a more modern thermocouple or resistive element, electronic temperature measuring system, accurate to ±1°C.

5.4 *Liquid Coolant,* which does not attack the test specimen under the conditions of the test. Methanol cooled with dry ice is satisfactory for most samples. Where methanol-dry ice combination is not appropriate, other cooling media may be used to achieve the prescribed test temperatures. Gaseous media may be employed as the coolant when the design of the

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials and is the direct responsibility of Subcommittee D11.10 on Physical Testing.

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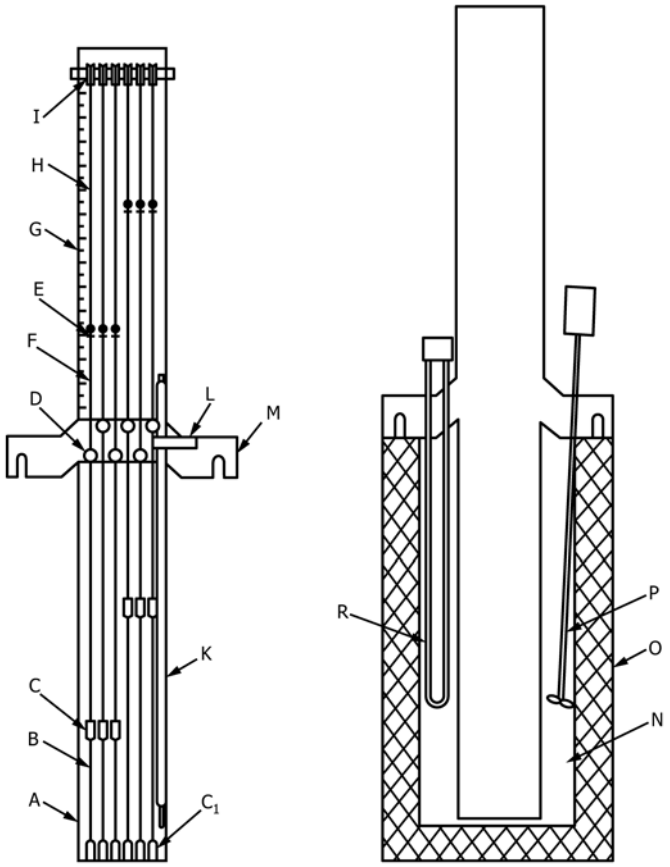
[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



A—Specimen Rack.
B—Test Specimen.
C—Movable Clamp.
C_1—Stationary Clamp.
D—Thumb Nut.
E—Indicator.
F—Connecting Wire.
G—Graduated Scale.
H—Flexible Core.
I—Pulley.
K—Thermometer.
L—Thermometer Holder.
M—Rack Support
N—Bath.
O—Insulating Frame.
P—Stirrer.
R—Heater.

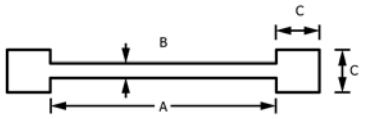
FIG. 1 Retraction Apparatus

apparatus is such that tests using it will duplicate those obtained with the standard liquid media.

5.5 An apparatus specially designed for the TR test[3,4] is schematically illustrated in Fig. 1. The sample rack is shown on the left, and the overall assembly on the right. The bath consists of an unsilvered Dewar flask that is contained in an insulating wooden frame, *0*. The frame contains a wide slot in front, through which the test can be observed and the temperatures read. Other details of the apparatus are given in Section 8.

6. Test Specimens

6.1 The test specimens may be prepared by dieing out with a die of the design shown in Fig. 2. The choice of die length is



	mm	in
A	25, 38 or 51	1, 1.5 or 2
B	2.0 ± 0.2	0.08 ± 0.01
C	6.4 ± 0.4	0.25 ± 0.02

FIG. 2 Die for Preparing Test Specimens

governed by the elongation required and the limitations of the specimen racks. For most work a 38 mm (1.50 in.) die is suitable. Thickness of the specimens shall be 2.0 ± 0.2 mm (0.08 ± 0.01 in.). Any other method of obtaining test specimens of uniform cross section is satisfactory, provided that a suitable clamp is used on the rack.

6.2 Three specimens per material shall be tested.

7. Initial Specimen Extension

7.1 The initial extension (elongation) of specimens to be tested should be chosen with the following considerations:

7.1.1 To study the effect of crystallization at low temperatures use a value of either: (*1*) 250 %, (*2*) half the ultimate elongation if 250 % is unobtainable, or (*3*) 350 % if the ultimate elongation is greater than 600 %.

7.1.2 To avoid the effect of crystallization, use an elongation of 50 %.

7.2 For long exposures, the 50 % elongation may be used in combination with a conditioning procedure, in accordance with Practice D832. In such studies, crystallization of the long- time conditioned specimen is indicated by the displacement of the TR curve toward the higher temperature. Tests conducted at 50 % elongation without previous long-time conditioning have been found to correlate fairly well with stiffness tests.

8. Procedure

8.1 Instruments are now available that may use other procedures to obtain the results listed throughout this method in details that employ techniques and devices that were not available when this method was introduced in 1954 and will produce test data equal to or better than as described in this text.

NOTE 1—Different models of instruments for this test position the thermocouple in relationship to the heater and stirrer at different locations. This will have an effect on actual bath temperature; a separate thermocouple placed in at least three locations will give the user confidence in reported temperature.

8.2 Fill the bath, *N* (Fig. 1) to within about 50 mm (2 in.) of the top with liquid coolant. Start the stirrer, *P*. Reduce the temperature of the liquid coolant by dipping into it, for short intervals, a wire cage filled with chopped dry ice. Care must be employed at the beginning of this operation to prevent excessive frothing. When the temperature drops to –70°C (–94°F) chopped dry ice can be added directly to the liquid coolant.

8.3 Insert one end of the test specimen, *B*, in the stationary clamp, C_1, at the bottom of the sample rack, *A*, and the other end in the movable clamp, *C*. Stretch to the length desired, reading the length by means of the indicator, *E*, attached to the connecting wire, *F*, and moving over the graduated scale, *G*. Anchor the specimen in the elongated position by tightening the thumb nut, *D*. Adjust the flexible cord, *H*, that is attached

[3] A modified Scott T-50 tester has been used by some investigators. See Svetlik, J. F., and Sperberg, L. R., "The T-R (Temperature Retraction) Test Characterizing the Low-Temperature Behavior of Elastomeric Compositions," *India Rubber World*, May, 1951, p. 182.

[4] See Smith, O. H., Hermonat, W. A., Haxo, H. E., and Meyer, A. W., "Retraction Test for Serviceability of Elastomers at Low Temperatures," *Analytical Chemistry*, Vol 23, 1951, p. 322.

to the wire, *F*, at one end and to a counterweight at the other end, so that it moves freely over the pulley, *I*. (The counterweight should be 3 to 5 g heavier than the clamp and wire that it counterbalances.) Repeat this operation for the other specimens in the rack. Insert the thermometer, *K*, in the holder, *L*.

8.4 Place the rack, *A*, in the bath. This must be done slowly to avoid frothing. Tighten the thumb nuts, that anchor the rack support, *M*, to the bath.

8.5 If the temperature of the batch rises above −70°C (−94°F) when the rack is inserted, add a little dry ice to reduce the temperature to between −70 and −73°C.

8.6 Let stand 10 min, then release the thumb nuts, *D*, and allow the specimens to retract freely.

8.7 Turn on the heater, *R*, and maintain a temperature rise of 1°C/min (2°F/min) by adjusting the rheostat.

8.8 Take the first reading at −70°C (−94°F), and continue to read the length at 2 min intervals until retraction is 75 % completed.

NOTE 2—When one standard specimen length and initial elongation are maintained, temperatures at which specific degrees of retraction occur may be read directly.

8.9 If a liquid coolant-dry ice system does not produce temperatures low enough to freeze the specimens to practically a nonelastic state, then other cooling media may be employed.

9. Calculations

9.1 Calculate retraction values at any specific temperature as follows:

$$\text{retraction, \%} = [(L_e - L_t)/(L_e - L_o)] \times 100 \quad (1)$$

where:

L_o = length of specimen in the unstretched condition,
L_e = length of specimen in the stretched condition, and
L_t = length of specimen at the observed temperature.

9.2 Calculate the temperature at any specific retraction as follows:

9.2.1 Determine the length of the test specimen at the desired retraction L_r, by means of the following formula:

$$L_r = L_e - (\%\ \text{retraction}/100)(L_e - L_o) \quad (2)$$

9.2.2 Note the nearest temperature corresponding to the length, L_r, and determine the exact temperature by interpolation.

10. Report

10.1 Report the following information:

10.1.1 The median values of the following:

10.1.1.1 Testing elongation, in percent.

10.1.1.2 Temperatures at which the specimen retracts 10, 30, 50, and 70 %. These temperatures shall be designated, respectively, as TR10, TR30, TR50, and TR70.

10.1.1.3 Difference between TR10 and TR70 in degrees Celsius.

10.1.2 The method or equipment used to measure temperature (glass thermometer, thermocouple, etc.).

10.1.3 Length of the test specimens before elongation.

TABLE 1 Pooling of Within Laboratory S_r and Between Laboratory S_R

Material or S_r: Compound	TR 10	TR 30	TR 50	TR 70	Pooled, S_r	Mean TR Value, °K (°C)
1	0.0	0.0	0.0	0.20	0.10	264.4 (−8.6)
2	0.0	0.20	0.82	0.20	0.437	235.3 (−37.8)
3	0.82	0.0	0.61	0.0	0.511	241.0 (−32.0)
4	0.20	0.0	0.20	0.82	0.434	235.8 (−37.3)
4	0.0	0.61	0.61	0.84	0.602	240.6 (−32.8)
Pooled S_r	0.378	0.287	0.540	0.540	(0.450)	
S_R: Material	TR 10	TR 30	TR 50	TR 70	Pooled, S_R	Mean TR Value, °K (°C)
1	1.01	0.76	0.64	0.18	0.714	264 (−8.6)
2	7.30	5.70	1.04	0.90	4.68	235.3 (−37.8)
3	0.25	2.38	4.26	6.71	4.15	241.0 (−32.0)
4	2.06	0.79	0.88	1.54	1.415	235.8 (−37.3)
5	0.06	0.83	0.73	1.37	0.881	240.6 (−32.8)
Pooled S_R	3.42	2.83	2.05	3.17	(2.914)	

10.1.4 Time and temperature of initial conditioning.

10.1.5 Rate of temperature rise, and

10.1.6 Coolant used.

11. Precision and Bias[5]

11.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical calculations details.

11.2 A Type 1 (interlaboratory) precision was evaluated in 1985. Both repeatability and reproducibility are short term, a period of a few days separates replicate test results. A test result is the mean value, as specified in this test method, obtained on two determination(s) or measurement(s) of the property or parameter in question.

11.3 Five different materials or compounds were used in the interlaboratory program, these were tested in two laboratories on two different days. One of the laboratories had two different operators perform the testing so that a total of three different operators were involved. The statements are based on the testing of five compounds by three operators on two days.

11.4 Standard vulcanized sheets were prepared by the supplying laboratory. Each participant die cut the test specimens. A test result is defined to be the average of two separately prepared specimens. Precision statements were prepared for TR 10, 30, 50, 70, and (70-10) where each operator determined test results in accordance with Section 9.

11.5 Within laboratories, S_r values of zero were obtained for S_r for selected parameters for several of the test compounds. These values are to no variation between the results obtained on two different test days by any of the three operators.

11.6 Due to the occurrence of zero values for S_r, the values of S_r (and S_R) were pooled for TR levels (10 to 70) and for materials. This was done to obtain a better estimate of the true S_r (and S_R) for the expression of precision. A tabulation of the S_r and S_R values and the results of the pooling calculations is given in Table 1. With the exception of Material 1, the values

[5] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1037.

TABLE 2 Type 1 Precision (TR 10 to TR 70)

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).

Material (Compound)	Mean TR Value		Within Laboratories			Between Laboratories		
	°K	°C	S_r	r	r^A	S_r	r	$(R)^A$
1	264.4	(–8.6)	0.10	0.28	0.11	0.713	2.02	0.77
2	235.3	(–37.8)	0.437	1.24	0.53	4.68	13.2	0.56
3	241.0	(–32.0)	0.511	1.45	0.60	4.15	11.7	0.49
4	235.8	(–37.3)	0.434	1.23	0.52	1.42	4.02	1.70
5	240.6	(–32.8)	0.602	1.70	0.71	0.881	2.49	1.03
Pooled (Mean) Value:	243.3	(–29.7)	0.450	1.27	0.52	2.914	8.25	3.40

[A] Mean TR in °K used.

of S_r and S_R are essentially constant for the other four materials. Based upon this the general precision for TR values (10 to 70) is given in Table 2.

11.7 The precision for the difference in TR (70-10) is given in Table 3. The precision of this test method may be expressed in the format of the following statements that use what is called an appropriate value of r, R, (r), 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 the precision tables closest to the mean level under consideration at any given time, for any given material in routine testing operations.

11.8 *Repeatability*—The repeatability r, of this test method has been established as the *appropriate value* tabulated in the precision tables. 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 non-identical sample populations.

11.9 *Reproducibility*—The reproducibility R, of this test method has been established as the *appropriate value* tabulated in the precision tables. 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 non-identical sample populations.

11.10 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. (See, however, the caveat statement, a footnote in Table 3 on (r) and (R) for TR (70-10).)

TABLE 3 Type 1 Precision TR (70-10), °K

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).

Material	Mean Level, TR (70-10), °K	Within Laboratories			Between Laboratories		
		S_r	r	$(r)^A$	S_R	R	$(R)^A$
1	7.9	0.20	0.57	7.1	0.88	2.49	35.1
2	31.3	0.20	0.57	1.8	8.09	22.9	7.3
3	18	0.82	2.32	12.9	6.92	19.6	109.
4	13.2	0.61	1.73	13.1	3.58	10.1	76.5
5	40.5	0.85	2.38	5.9	1.41	4.0	9.9
Pooled (Mean) Value:	22.2	0.605	1.712	7.7	5.08	14.4	64.9

[A] The relative (%), (r) and (R) are given, but these must be interpreted with caution due to the often near zero temperature difference values of TR (70-10).

11.11 *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 crystallization; low temperature retraction; rubber; temperature retraction; testing at subnormal temperatures; TR test; viscoelastic properties