UNCOMPROMISING PRECISION IN DURABILITY TESTING.

DEMATTIA FLEX CRACKING TESTER

ACCELERATED AGING ANALYSIS.

DETERMINE THE FLEX CRACKING RESISTANCE OF POLYMERS AND MATERIALS

PROGRAMMABLE CONTROLS.

PRECISE CONTROL OVER FLEX CYCLES, FREQUENCY, AND INTEGRATED ENVIRONMENTAL OPTIONS.

CLOCK PROGRESS

DETERMINE THE FLEX CRACKING RESISTANCE OF POLYMERS AND MATERIALS

MULTI-STATION EFFICIENCY.

SIMULTANEOUS ANALYSIS OF MULTIPLE TEST SPECIMENS FOR FASTER RESULTS.

PROGRAMMABLE CONTROLS.

DESIGNED FOR LONG-TERM INDUSTRIAL RELIABILITY

ENVI-CHAMBERS

YOUR PARTNER IN PRECISION DURABILITY.

DISCOVER MORE AT **ENVI-CHAMBERS.COM**

Wewon Ozone Test Chambers

UNCOMPROMISING PRECISION IN MATERIALS TESTING

ACHIEVE UNRIVALED ACCURACY WITH INTEGRATED DIGITAL INTERFACE & MULTI-LAYER DATA MONITORING. PRECISE OZONE, TEMPERATURE, & HUMIDITY CONTROL FOR HIGH-END QUALITY ASSURANCE.

WewonTech

Designation: D 1329 – 02

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

This standard is issued under the fixed designation D 1329; 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.

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:*

D 832 Practice for Rubber Conditioning for Low-Temperature Testing[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 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. Gaseous media may be employed as the coolant when the design of the apparatus is such that tests using it will duplicate those obtained with the standard liquid media.

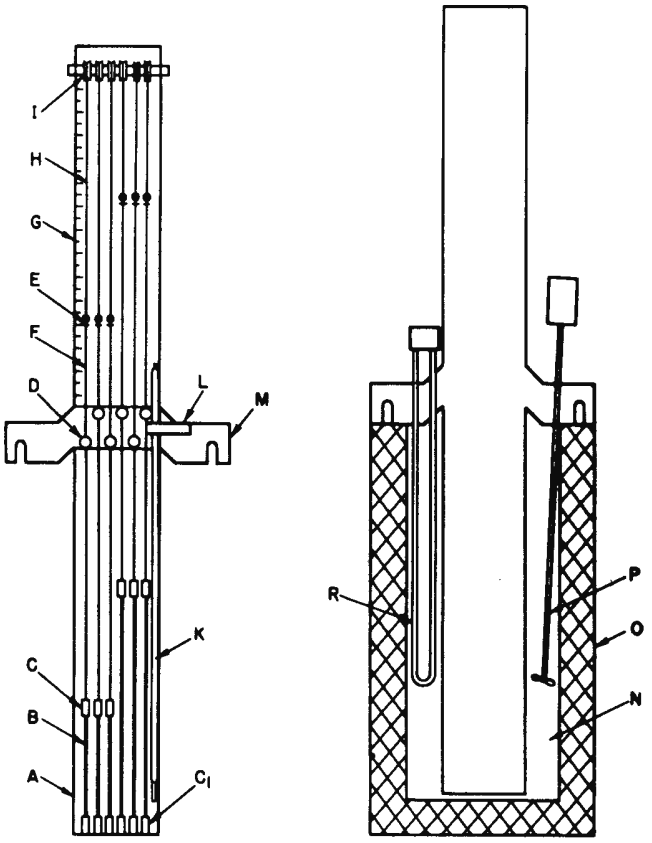
---

[1] This test method is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.14 on Time and Temperature-Dependent Physical Properties.

Current edition approved Dec. 10, 2002. Published January 2003. Originally approved in 1954. Last previous edition approved in 1998 as D 1329 – 88 (1998).

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

Copyright © ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States.



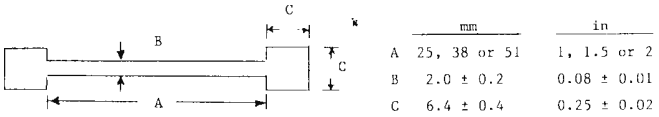
A—Specimen Rack.
B—Test Specimen.
C—Movable Clamp.
C_1—Stationary Clamp.
D—Thumb Nut.
E—Indicator.
F—Connecting Wire.
G—Graduated Scale.
H—Flexible Core.
I—Pulley.
K— Thermometer.
L—Thermometer Holder.
M—Rack Support
N—Bath.
O—Insulating Frame.
P—Stirrer.
R—Heater.

FIG. 1 Retraction Apparatus

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 governed by the elongation required and the limitations of the specimen racks. For most work a 38 mm (1.50 in.) die 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

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 D 832. 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 Fill the bath, *N* (Fig. 1) to within about 50 mm (2 in.) of the top with methanol. Start the stirrer, *P*. Cool the methanol 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 methanol.

8.2 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 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*.

8.3 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.

8.4 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.

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

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

[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.

8.7 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 1—When one standard specimen length and initial elongation are maintained, temperatures at which specific degrees of retraction occur may be read directly.

8.8 If a methanol-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.

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 D 4483. 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.

[5] Supporting data are available from ASTM Headquarters. Request RR: D11-1037.

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)	

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 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

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.

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).

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).)

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

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).