Designation: D1329 – 16 (Reapproved 2021)

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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.

[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.

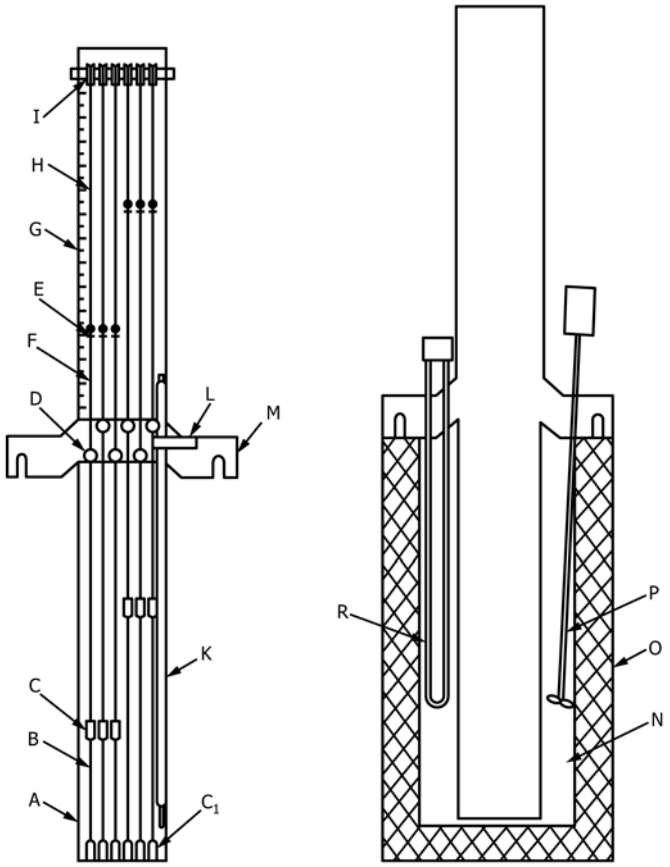
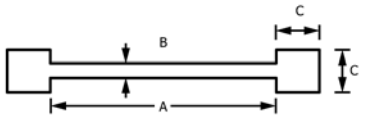


FIG. 1 Retraction Apparatus



	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

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 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 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.

[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.