



Standard Practice for Rubber Conditioning For Low Temperature Testing¹

This standard is issued under the fixed designation D832; 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 practice covers the characteristic mechanical behavior of rubbers at low temperatures, and outlines the conditioning procedure necessary for testing at these temperatures.

1.2 One of the first stages in establishing a satisfactory technique for low temperature testing is the specification of the time and temperature of exposure of the test specimen. It has been demonstrated that any one or more of the following distinct changes, which are detailed in [Table 1](#), may take place on lowering the test temperature:

1.2.1 Simple temperature effects,

1.2.2 Glass transitions, and

1.2.3 First order transitions (crystallization), and solubility and other effects associated with plasticizers.

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

[D471](#) Test Method for Rubber Property—Effect of Liquids

[D1053](#) Test Methods for Rubber Property—Stiffening at Low Temperatures: Flexible Polymers and Coated Fabrics

[D1329](#) Test Method for Evaluating Rubber Property—Retraction at Lower Temperatures (TR Test)

[D1566](#) Terminology Relating to Rubber

[D2136](#) Test Method for Coated Fabrics—Low-Temperature Bend Test

[D5964](#) Practice for Rubber IRM 901, IRM 902, and IRM 903 Replacement Oils for ASTM No. 1, ASTM No. 2, and ASTM No. 3 Oils

¹ This practice is under the jurisdiction of ASTM Committee [D11](#) on Rubber 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.

3. Significance and Use

3.1 Low temperature testing of rubber can yield repeatable results only if the preconditioning of the samples is consistent. Properties such as brittleness and modulus are greatly affected by variations in time/temperature exposures. This practice is intended to provide uniform conditioning for the various low temperature tests conducted on rubbers.

4. General Conditioning

4.1 At least 16 h should elapse between vulcanization and testing of a sample.

4.1.1 If the time between vulcanization and testing is less than 16 h, it shall be agreed upon between customer and supplier and noted in the report section of the test method employed.

5. Simple Temperature Effects (Viscoelasticity)

5.1 Most elastic properties of rubber change as the temperature is changed. As the temperature is reduced toward the glass transition temperature, T_g , the specimen becomes increasingly stiff, loses resilience, and increases in modulus and hardness. At some point, still above T_g , the resilience reaches a minimum. As the temperature is lowered beyond this point, the resilience then increases until a temperature just above T_g is reached.

5.2 Viscoelastic changes are usually complete as soon as the specimen has reached thermal equilibrium. Longer exposure time should be avoided to minimize crystallization or plasticizer-time effects that might influence the test results. The magnitude of these changes depends on the composition of the material and the test temperature.

6. Glass Transition

6.1 Glass transition is a reversible physical change in a material from a viscous or rubbery state to a brittle glassy state (refer to Terminology [D1566](#): transition, glass; transition second order). It does not involve a change in phase and is not a thermodynamic change. It generally occurs over a small temperature range. It is designated as T_g . The T_g of polymers, obtained from measurements of change of modulus with change in temperature, depend upon both the rate of specimen deformation and the rate of temperature change. Primary

TABLE 1 Differentiation Between Crystallization and Glass Transition

Property	Crystallization	Glass Transition
Physical effects (1, 2, 4, 6, 7) ^A	Becomes stiff (hard) but not necessarily brittle	Becomes stiff and brittle
Temperature-volume relation (1, 2, 3, 4, 5, 8)	Significant decrease in volume	No change in volume, but definite change in coefficient of thermal expansion
Latent heat effect (4, 5, 8)	Heat evolved on crystallization	Usually no heat effect, but definite change in specific heat
Rate (2, 4, 6, 7, 8)	Minutes, hours, days, or even months may be required. In general, as temperature is lowered, rate increases to a maximum and then decreases with increase in deformation. Rate also varies with composition, state of cure, and nuclei remaining from previous crystallizations, or from compounding materials such as carbon black. Optimum temperature is specific to the polymer involved.	Usually rapid; takes place within a definite narrow temperature range regardless of thermal history of specimen. May be limited rate effect (2)
Temperature of occurrence (4, 5, 7, 8)		Very wide limits, depending on composition
Effect on molecular structure (1, 2, 5, 6, 8)	Orientation of molecular segments; random if unstrained, approaching parallelism under strain	Change in type of motion of segments of molecule
Materials exhibiting properties (5, 7, 8)	Unstretched polymers including natural rubber (low sulfur vulcanizates), chloroprene, Thiokol A polysulfide rubber, butadiene copolymers with high butadiene content, most silicones, some polyurethanes. Butyl rubbers crystallize when strained. Straining increases rate of crystallization of all of the above materials.	All

^AThe numbers in parentheses refer to the following references:

- (1) Juve, A. E., Whitby, G. S., Davis, C. C., and Dunbrook, R. F., *Synthetic Rubber*, John Wiley & Sons, New York, NY, 1954, pp. 471–484.
- (2) Boyer, R. F., and Spencer, R. S., *Advances in Colloid Sciences*, Vol II, edited by H. Mark and G. S. Whitby, Interscience Publishers, Inc., New York, NY, 1946, pp. 1–55.
- (3) Boyer, R. F., and Spencer, R. S., *High Polymer Physics, A Symposium*, edited by Howard A. Robinson, Chemical Publishing Co., Inc., Brooklyn, NY, 1948, pp. 170–184.
- (4) Wood, L. A., and Bekkedahl, Norman, *High Polymer Physics, loc cit*, 1948, pp. 258–293.
- (5) Schmidt, A. X., and Marlies, C. A., *Principles of High Polymer Theory and Practice*, McGraw-Hill Book Co., New York, NY, 1948, pp. 175–193.
- (6) Treloar, L. R. G., *The Physics of Rubber Elasticity*, Oxford University Press, London, 1949, pp. 152–191.
- (7) Liska, J. W., “Low Temperature Properties of Elastomers,” *Symposium on Effects of Low Temperature on the Properties of Materials*, STP 78, ASTM, 1946, pp. 27–45.
- (8) Turner, Alfrey, Jr., “Mechanical Behavior of High Polymers,” Vol VI of *High Polymer Series*, Interscience Publishers, Inc., New York, NY, 1948, pp. 80–83 and 340–374.

properties, such as hardness and ultimate elongation, and temperature coefficients of properties such as volume and enthalpy, change rapidly near T_g . Thus, thermal expansivity and specific heat appear discontinuous at T_g .

6.2 Some rubbers such as copolymers or polymer blends may show more than a single T_g because of separate contributions by their polymeric components. There may also be damping peaks not directly attributable to glass transitions. A glass transition occurs at a temperature below which the thermal energies of molecular segments are insufficient to free them from the force field of their immediate neighbors within the experimental time scale.

6.3 Values determined for T_g are higher for test methods that require high frequency distortions of the specimen than for those that require low frequency distortions. The latter seem to have the greater resolving power for multiple peaks. For those methods in which the test temperature is changed at a controlled rate, T_g depends upon the rate that is chosen. Therefore, T_g is not a true material property since it depends upon the test method used to obtain it. The method used should always be stated.

7. First Order Transitions (Crystallization)

7.1 A first order transition is a reversible change in phase of a material; in the case of polymers, it is usually crystallization or melting of crystals (refer to Terminology D1566: transition, first order). When a specimen is equilibrated at a temperature at which crystallization is possible, changes in properties resulting from the crystallization may begin immediately or after an induction period of up to several weeks. The time to reach an equilibrium state of crystallization is likewise widely

variable. Both times are dependent on the material being tested and the temperature. Crystallization increases the hardness and modulus. A specimen that has crystallized once may crystallize much more rapidly on subsequent tests, unless, in the meantime, it has been heated sufficiently to destroy the crystal nuclei.

7.2 Examples of materials that crystallize relatively rapidly in certain temperature ranges include Thiokol A³ polysulfide rubber, chloroprenes (excepting the RT types), natural rubber, and some butadiene copolymers cured without sulfur or with low sulfur. Materials that may require much longer times for crystallization effects to become evident include butyl rubber, high sulfur cures of natural rubber, most silicone rubbers, some polyurethane rubbers, RT types of chloroprene, and rubbers containing fluorine.

7.3 The temperature at which crystallization proceeds most rapidly is specific to the polymer involved. For natural rubber, this is near -25°C ; for chloroprenes, -10°C ; for butadiene copolymers, -45°C ; for dimethyl silicones, -55°C ; for polyester-type polyurethanes, -10°C ; and for butyl rubber, -35°C . Both above and below these temperatures, crystallization is slower. Accordingly, any attempt to compare materials (particularly those subject to change in properties resulting from crystallization or plasticizer time effects) on a basis of exposure at a given temperature for a specified time is almost

³ The sole source of supply of this material known to the committee at this time is Thiokol Chemical Corp, Newtown-Yardly Rd., Newtown, PA 18940. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.