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Standard Practice for Rubber Conditioning For Low-Temperature Testing¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice describes 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:

D 1053 Test Methods for Rubber Property—Stiffening at Low Temperatures: Flexible Polymers and Coated Fabrics²

D 1329 Test Method for Evaluating Rubber Property—Retraction at Lower Temperatures (TR Test)²

D 2136 Test Method for Coated Fabrics—Low-Temperature Bend Test²

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.

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 or “logy,” 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. 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 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

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.