Designation: D 3847 – 01

Standard Practice for Rubber—Directions for Achieving Subnormal Test Temperatures[1]

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1. Scope

1.1 This practice covers the general requirements for achieving and maintaining temperatures below 21°C for thermal conditioning and physical testing of rubber.

1.2 This practice describes the acceptable types and construction of low-temperature cabinets for conditioning and testing of rubber, the composition and circulation of heat-transfer media, and the required uniformity and precision of temperature control.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *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 746 Test Method for Brittleness Temperature of Plastics and Elastomers by Impact[2]

D 832 Practice for Rubber Conditioning for LowTemperature Testing[3]

D 945 Test Methods for Rubber Properties in Compression or Shear (Mechanical Oscillograph)[3]

D 1053 Test Methods for Rubber Property—Stiffening at Low Temperatures: Flexible Polymers and Coated Fabrics[3]

D 1229 Test Method for Rubber Property—Compression Set at Low Temperatures[3]

D 1329 Test Method for Evaluating Rubber Property—Retraction at Low Temperatures (TR Test)[3]

D 1415 Test Method for Rubber Property—International Hardness[3]

D 2136 Test Method for Coated Fabrics—Low-Temperature Bend Test[3]

D 2137 Test Methods for Rubber Property—Brittleness Point of Flexible Polymers and Coated Fabrics[3]

D 2240 Test Method for Rubber Property—Durometer Hardness[3]

D 2632 Test Method for Rubber Property—Resilience by Vertical Rebound[3]

E 197 Specification for Enclosures and Servicing Units for Tests Above and Below Room Temperature[4]

3. Significance and Use

3.1 Low temperatures are often needed for conditioning of rubber prior to testing, as well as during the test. Conditioning is required to attain a specific temperature that is uniform throughout the specimen or for producing time-dependent effects. Specimens may be conditioned and tested in the same or different chambers.

3.2 This practice is intended to apply particularly, but not exclusively, to the following ASTM Standards: Test Methods D 746, D 945, D 1053, D 1229, D 1329, D 1415, D 2136, D 2137, D 2240, D 2632, Practice D 832, and Specification E 197.

4. General Equipment Requirements[5]

4.1 The low-temperature cabinet may be refrigerated mechanically, or by dry ice or liquid nitrogen, either directly or indirectly.

4.1.1 The heat-transfer medium in the test chamber should be air or air mixed with carbon dioxide or nitrogen, unless a liquid medium is specified. Although liquids cool the specimens faster than gases, they are more likely to cause property changes in addition to those caused by temperature change. Water, ethyl alcohol, and ethylene glycol are usually acceptable for immersion times that are kept to the minimum for the required tests.

4.1.2 The temperature variation within 250 mm of the test specimen shall be within ±1°C of the specified testing temperature.

4.1.3 The heat-transfer medium should be circulated thoroughly in the test chamber by means of mechanical agitation. A fan or stirrer suitably located in the test chamber can be used for this purpose.

4.1.4 Automatic temperature control should be used.

4.2 The size of the test chamber is optional.

[1] This practice 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.

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[2] *Annual Book of ASTM Standards*, Vol 08.01.

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

[4] Discontinued—see *1982 Annual Book of ASTM Standards*, Parts 40 and 41.

[5] For more detailed information, see Specification E 197.

4.3 The door can be located either in the top of the test cabinet or in the side. A side-opening door is convenient, and is necessary where the equipment involved must be operated from the side. However, it has the disadvantage of allowing the cold air to pour out when the door is opened. In general, a window in the door is desired for observation and the reading of test equipment indicators. This window should have at least five sealed layers of glass, suitably spaced, with dehydrated air between the layers to prevent condensation or frosting.

4.3.1 Return to the test temperature after the introduction of specimens or test apparatus should be as rapid as possible consistent with minimal overshoot, but should not exceed 15 min. Particular care is required for gaseous media.

4.3.2 When the measuring device cannot be located logically within the low-temperature chamber, it should have an extension rod of low thermal conductivity that extends through the insulated wall and contacts the specimen. An example is given in Test Method D 1053.

4.4 Chambers used for conditioning of specimens prior to the test should provide adequate circulation on all sides of the specimens. Suitable supports include racks, metal clips, and wide-mesh wirescreen frames with at least 25 mm between the screen and the cabinet floor. For chambers that are in continuous use, chamber walls having a maximum heat transmission factor of 35 mW/m·K (0.24 Btu·in./h·ft^2·°F) are desirable. For the operation of the equipment involved in Test Methods D 2240 and D 2632, handholes equipped with gloves and insulated sleeves should be installed in the walls of the test cabinet. The interior walls surrounding the chamber should be made of a good thermal conductor to promote uniform temperature and minimum radiant effects. Any heating elements within a gaseous medium chamber should be shielded from the specimen since they could have radiant effects.

4.5 A millivolt-type indicating potentiometer with an electronic control mechanism has been found suitable for measuring the temperature and controlling the relay that opens and closes the coolant valve. Copper-constantan thermocouple wire of 30 gage (0.255 mm) has been found to be most satisfactory. Such instrumentation is desirable when extremely accurate temperature measurement and control are required.

5. Mechanically Refrigerated Units

5.1 In general, the mechanically refrigerated low-temperature cabinets consist of multiple-stage compressor and suitable cooling coils that surround the test chamber. Suitable insulation is provided between the test chamber and the outside walls of the cabinet. Automatic temperature control is obtained by either a thermostatic control located in the test chamber, which turns the compressor on or off, or by a suitable pressure control, which regulates the refrigerant temperature. Air is used as the heat-transfer medium in the test specimen compartment.

5.2 Mechanically refrigerated equipment is well-adapted to continuous operation at a fixed temperature. Except for rather high maintenance and initial costs, this type of equipment is less expensive to operate continuously from a power standpoint than are the dry ice units. Another advantage of mechanical refrigeration is the lower temperatures available. By the installation of electric strip heaters and suitable automatic controls, temperatures up to room temperature and above can be obtained. Heaters should be shielded from test specimens to avoid radiant effects.

6. Dry Ice Units (Direct-Type)

6.1 In the direct type of dry ice low-temperature cabinets, a suitable fan or blower located in the dry ice compartment circulates the carbon dioxide vapor from the dry ice chamber into the test specimen chamber and back. By means of a preset damper between the dry ice compartment and the test chamber, the inlet and outlet openings can be adjusted for maximum efficiency. A bimetallic thermoregulator, located in the test compartment, controls the "on'' and "off'' operation of the fan in the dry ice compartment, thus providing automatic temperature control. To ensure uniformity of temperature in the test compartment, a fan is provided. More accurate temperature control can be obtained by the addition of thermostatically controlled heaters inside the test chamber, making sure that they are shielded from the test specimens.

6.2 Commercial direct dry ice units are available in either 0.028 or 0.227-m^3 (1 or 8-ft^3) capacities and provide a temperature range from −73 to +104°C (−99 to +219°F). These units are particularly adapted for intermittent use where a wide range of testing temperatures is desired. Only a short time is required to cool the test chamber to a low temperature.

7. Dry Ice Units (Indirect-Type)

7.1 In the indirect type of dry ice low-temperature cabinets, air is used as the heat-transfer medium and no carbon dioxide from the dry ice comes in contact with the test specimens. The test chamber is cooled by circulating the carbon dioxide vapor completely around the outside of the test chamber which, in turn, is insulated from the outside of the cold box. In general, this type is a more costly construction than the direct type and is not quite as efficient. The time required to cool the test chamber to a low temperature is somewhat greater and is more comparable in this respect to the mechanically refrigerated units.

7.2 Commercial indirect dry ice units are available in either a 1 or 8-ft^3 test chamber capacity. These units operate over a temperature range from −68 to +104°C (−90 to +219°F). Two fans are provided, one for circulating the carbon dioxide around the outside of the test chamber and the other for circulating the air heat-transfer medium inside the test chamber. An automatic switch on the door shuts down the latter fan when the door is opened, thus tending to prevent frosting.

8. Packaged Air Units

8.1 Temperature-regulated air or carbon dioxide may be circulated through insulated pipes or ducts from a separate unit to the test chamber. This unit may be portable so that it is usable with various test chambers.

8.2 The temperature may be controlled by a thermoswitch that operates either the fan motor or a damper that recirculates part or all of the cold gas so that it bypasses the test chamber. Motor- or solenoid-driven dampers that operate in the dry ice compartment for temperature control may cause trouble due to frosting the mechanism. No entirely satisfactory method has yet been devised for removing sufficient moisture from the heat-transfer medium to eliminate frosting completely. Desiccants, such as calcium chloride and calcium sulfate, have been used.

9. Non-Recirculating Gas

9.1 Liquid nitrogen or liquid carbon dioxide may be introduced into the test chamber in amounts sufficient to maintain the desired low temperature. Any concentrated flow of cold gas directly onto the specimen should be avoided. This method is particularly desirable when very low humidity is required.

10. Keywords

10.1 brittleness point; low temperature; subnormal test temperature; temperature dependent test; thermal conditioning