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Rubber — General directions for achieving elevated or sub-normal temperatures for tests

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 3383 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the Member Bodies in April 1974.

It has been approved by the Member Bodies of the following countries :

Australia	India	Spain
Belgium	Italy	Sweden
Brazil	Mexico	Switzerland
Bulgaria	Netherlands	Turkey
Canada	Poland	United Kingdom
Chile	Portugal	U.S.A.
France	Romania	
Hungary	South Africa, Rep. of	

No Member Body expressed disapproval of the document.

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Rubber — General directions for achieving elevated or sub-normal temperatures for tests

1 SCOPE AND FIELD OF APPLICATION

This International Standard states the general requirements for achieving elevated or sub-normal temperatures in the testing of rubber and other elastomeric materials. It is designed to acquaint the persons making tests at such temperatures with the general principles of the construction and operation of temperature-controlled chambers for conditioning and/or testing. For some tests, for example ageing tests, special procedures, conditions or equipment different from those specified in this International Standard may be required. In such cases, the requirements should be included in the method of test and supersede the requirements of this International Standard.

2 PURPOSE OF CONDITIONING

The purpose of conditioning procedures is to ensure that the test piece is substantially at a uniform temperature throughout its mass and in equilibrium with its environment.

3 HEAT TRANSFER MEDIA

For the control of temperature in the conditioning and/or test chambers, various temperature-control media may be employed, the most common being gaseous or liquid. Liquid media offer the most rapid heat transfer but may have deleterious effects on the rubber if the immersion is prolonged. Fluidized beds using glass ballotini have many of the advantages of liquid heat transfer combined with chemical inertness.

4 GENERAL REQUIREMENTS FOR TEMPERATURE-CONTROLLED CHAMBERS

4.1 The immersion medium in the chamber shall be without significant effects on the rubber properties. Water, ethyl alcohol and ethylene glycol are examples of fluids that have not been found to have significant effects on most solid rubbers, provided that the immersion period is kept to the absolute minimum required in the testing.

4.2 The portion of the chamber in which test pieces may be located shall be controlled within the specified tolerances of the relevant method of test.

4.3 The immersion medium shall be circulated thoroughly in the chamber. A fan or stirrer suitably located in the chamber may be used for this purpose.

4.4 Automatic temperature control is preferred.

4.5 Recovery to the set temperatures after the introduction of test pieces or test apparatus shall be as rapid as possible consistent with minimal overshoot, but in any case shall not exceed 15 min, particular care being required for gaseous medium.

4.6 The size of the chamber is optional, provided that uniform temperature is maintained throughout the space occupied by the test pieces.

4.7 The chamber shall be thermally insulated to prevent condensation on exterior surfaces when testing at sub-normal temperatures and to prevent discomfort to the touch when testing at elevated temperatures.

The thermal insulation shall be stable at the maximum design temperature, shall be protected from condensation at sub-normal temperatures and shall ensure the maximum attainable uniformity of temperature. If a window is needed to observe test equipment indicators, it shall be constructed so as to ensure adequate thermal insulation and to prevent condensation. For example for a chamber operating at -100 °C, five layers of glass, suitably spaced, with dehydrated air between layers, has been found suitable.

4.8 The construction of the chamber depends on the type of immersion medium. For gaseous media, a side entrance for introducing test pieces is convenient, and is necessary where the test equipment is operated from the side. However, the chamber shall be designed to minimize the loss of gaseous media where the test pieces or equipment are introduced. The interior walls surrounding the chamber shall be made of a good thermal conductor, preferably aluminium or tinned copper, to ensure uniform temperature and minimum radiant effects. When manual operation of equipment inside the chamber is necessary, handholes equipped with gloves and insulated sleeves shall be installed in the walls of the chamber. The temperature shall not be controlled by cooling and heating elements within the chamber that can affect the temperature of test equipment or test pieces by radiation.

For liquid media, the temperature may be controlled by elements immersed in the medium or by circulating the medium to a heat-exchange system outside the chamber.

5 CHAMBERS OPERATING AT ELEVATED TEMPERATURES

5.1 Chambers with gaseous heat-transfer media

Such chambers should normally conform to the requirements of clause 4. The gaseous medium, usually air, is heated by means of electric elements, a fan or blower being provided to ensure adequate air circulation. The electric elements shall be shielded to avoid direct radiation on to the test pieces. To obtain the necessary precision of temperature control, the heating system shall

a) use a recirculating gas system and

b) be designed so that most of the heat required is supplied continuously and the remainder intermittently for temperature control or with proportionating devices on the heat supply that present large cyclic variations in temperature.

NOTE — In the case of recirculating gas systems, care shall be taken at higher temperatures to avoid the possibility of cross-contamination by volatile constituents of rubber samples.

5.2 Chambers with liquid heat-transfer media

Such chambers should conform to the requirements of clause 4 and follow the same principles as in 5.1 using an immersion heater in place of the electric elements and a stirrer or pump in place of a fan or blower.

5.3 Fluidized beds

Such chambers should conform to the requirements of clause 4 by utilizing a bed of inert material, such as glass ballotini, which may be "fluidized" (i.e. given the properties of a fluid) by passing a suitable gas through the bed at a suitable velocity. Heaters may be embedded in the bed, or the gas stream itself may be heated.

6 CHAMBERS OPERATING AT SUB-NORMAL TEMPERATURES

Several types of such chambers are available, the chief of which are :

6.1 Mechanically refrigerated units

In general, a mechanically refrigerated low-temperature chamber consists of a multi-stage compressor and suitable cooling coils which surround the test chamber. Suitable insulation is provided between the test piece compartment and the outside walls of the chamber. Automatic temperature control is obtained either by a thermostatic control located in the test piece compartment, 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 piece compartment. This type of 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 solid carbon dioxide units. Another advantage of mechanical refrigeration is that lower temperatures are available. By the installation of electric strip heaters and suitable automatic controls, temperatures up to room temperature and above can be obtained.

6.2 Solid carbon dioxide units (direct type)

In the direct type of solid carbon dioxide low-temperature chamber, a suitable fan or blower, located in the solid carbon dioxide compartment, circulates the carbon dioxide vapour from the solid carbon dioxide compartment into the test piece compartment and back. By means of a preset damper between the solid carbon dioxide compartment and the test piece compartment, the inlet and outlet openings may be adjusted for maximum efficiency. A bimetallic thermoregulator, located in the test piece compartment, controls the "on" and "off" operation of the fan in the solid carbon dioxide compartment, thus providing automatic temperature control.

Alternatively, the test piece compartment may consist of a suitable insulated vessel containing a liquid medium which does not affect the rubber and which remains liquid at the required temperature of test. The temperature of the liquid is adjusted by the addition of small portions of the coolant. In the case of solid carbon dioxide, care is needed to prevent the boiling over of liquid from the vessel. To ensure uniformity of temperature in the test piece compartment, a fan or stirrer is provided. More accurate temperature control may be obtained by the addition of thermostatically controlled heaters inside the test piece compartments.

6.3 Solid carbon dioxide units (indirect type)

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

Alternatively, the test piece compartment may consist of a suitable insulated vessel containing a liquid medium which does not affect the rubber and which remains liquid at the temperature of test. The temperature of the liquid is adjusted by circulating the liquid through a suitable heat-exchanger located externally to the vessel, where the temperature of the liquid is lowered by packing the outside of the heat-exchanger with solid carbon dioxide contained in a suitable insulated compartment.

6.4 Packaged-air units

It is frequently desirable to enclose a piece of test equipment in a separate chamber and circulate temperature-regulated cold air or carbon dioxide from a separate unit through insulated ducts or pipes. This type of unit is somewhat similar to the direct type of solid carbon dioxide unit (see 6.2), but contains an auxiliary blower to force the cold air through the pipes to the separate chamber. It has the advantage of being portable, and can be connected to different pieces of equipment.

In some cases the temperature is controlled by a motor-driven damper which by-passes the cold chamber or test chamber, rather than by control of the fan motor itself. Motor- or solenoid-driven dampers which operate in the compartment for temperature control may cause trouble due to frosting of the machine. No entirely satisfactory method has yet been devised for removing sufficient moisture from the heat-transfer medium to completely eliminate frosting. Desiccants such as calcium chloride and calcium sulphate have been used.

6.5 Liquid nitrogen

Liquid nitrogen is an effective means of maintaining chambers at sub-normal temperatures. The liquid nitrogen may be injected into the chamber as required to control the temperature or the gas in the chamber may be circulated to a liquid-nitrogen vessel outside the chamber in such volume as is required for temperature control. When liquid nitrogen is injected, it shall be completely vaporized and the nitrogen gas shall be at test temperature before contacting the test equipment or test pieces.

ANNEX

CALCULATED CONDITIONING TIME REQUIRED FOR CENTRE OF RUBBER TEST PIECE TO REACH APPROXIMATE TEMPERATURE OF SURROUNDING STILL AIR FOR TEMPERATURE CHANGE OF 10 °C

Temperature differential between air and centre of rubber, °C	Time required, s		
	ISO/R 1432 test piece (Gehman low-temperature test)	Sheet 2,5 mm thick	Cylinder, 12,5 mm thick, 20 mm diameter
1,0	260	520	1 740
0,5	330	680	2 250
0,2	430	890	2 940
0,1	510	980	3 420

NOTE — In a flat thin sheet test piece, the time required for thermal equilibrium may be taken as being directly proportional to the sheet thickness. Thus, for a 25 mm thick slab the times given for a 2,5 mm thick sheet should be multiplied by 10 approximately.

If the air temperature is to be changed by 100 °C, the temperature differentials would be 10, 5, 2 and 1 °C, respectively for the respective time periods.

For any temperature change T, the temperature differential in the table should be multiplied by $T/10$.

For example, if the test piece described in ISO/R 1432 at a room temperature of 20 °C is placed in air at − 70 °C, the temperature change would be 90 °C, and at the end of 510 s, the temperature differential between the centre of the test piece and air would be 0,9 °C, making the temperature of the centre of the test piece − 69,1 °C.

The above times may be reduced at least 50 % by providing an air movement of about 50 m/s past the test piece and by about 85 % by using a circulating-liquid bath.