INTERNATIONAL STANDARD

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Second edition
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Rubber, raw — Determination of the glass transition temperature by differential scanning calorimetry (DSC)

Caoutchouc brut — Détermination de la température de transition vitreuse par analyse calorimétrique différentielle (DSC)

Reference number
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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 22768:2006), which has been technically revised with the following changes:

— additional description on placing an empty pan (crucible) as reference;

— general DSC thermogram inserted to show an inflection point which should be T_g;

— move of the content of the clause on precision data to an informative Annex A.

Rubber, raw — Determination of the glass transition temperature by differential scanning calorimetry (DSC)

WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This document specifies a method using a differential scanning calorimeter to determine the glass transition temperature of raw rubber.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1407, *Rubber — Determination of solvent extract*

ISO 11357-1:2016, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11357-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
glass transition
reversible change in an amorphous polymer, or in amorphous regions of a partially crystalline polymer, from (or to) a rubbery or viscous condition to (or from) a glassy or hard condition

3.2
glass transition temperature
T_g
approximate midpoint of the temperature range over which the *glass transition* (3.1) takes place

Note 1 to entry: For the purposes of this document, the glass transition temperature is defined as the point of inflection of the DSC curve which has been obtained at a heating rate of 20 °C/min (see A.3).

4 Principle

The change in specific heat capacity of the rubber as a function of temperature under a specified inert atmosphere is measured using a differential scanning calorimeter (DSC). The glass transition temperature is determined from the curve thus produced.

5 Apparatus and materials

5.1 Differential scanning calorimeter, in accordance with ISO 11357-1:2016, 5.1.

The calorimeter should be operated in a room held at standard laboratory temperature. It should be protected from draughts, direct sunlight and sudden temperature changes.

5.2 Specimen pans (crucibles), in accordance with ISO 11357-1:2016, 5.2.

5.3 Gas supply, analytical grade, usually nitrogen or helium.

5.4 Balance, capable of measuring the specimen mass to an accuracy of ±0,000 1 g.

6 Test specimen

The test specimen shall be as representative as possible of the sample being examined and shall have a mass between 0,01 g and 0,02 g.

To determine T_g of polymers, extract raw rubber in accordance with ISO 1407.

7 Conditioning

Condition the sample to be examined and the test specimen in accordance with ISO 23529.

8 Calibration

Calibrate the calorimeter according to the manufacturer's instructions.

The use of suitable analytical grade substances is recommended to check the accuracy of the temperature scale. Ideally, substances whose melting points bracket the temperature range of interest should be chosen. *n*-Octane, *n*-heptane and cyclohexane have been found to be useful. Indium should be used if a higher temperature calibrant is required.

9 Procedure

9.1 Gas flow rate

The same inert gas flow rate with a tolerance of ±10 %, shall be used throughout the procedure. Flow rates between 10 ml/min and 100 ml/min have been found to be suitable.

9.2 Loading the test specimen

Determine the mass of the test specimen to an accuracy of ±0,001 g. The same nominal mass shall be used for all determinations. If possible, the specimen shall have a flat surface so as to give good thermal contact with the bottom of the pan.

NOTE 1 Intimate thermal contact between the test specimen and the bottom of the pan is essential for good repeatability.

Place the specimen in the pan, using tweezers and seal with a lid. Place the sealed pan in the calorimeter using tweezers.

Do not handle the test specimen or the pan with bare hands.

NOTE 2 Placing an empty pan with a lid as a reference helps to obtain stable DSC thermograms.

9.3 Temperature scan

9.3.1 Cool the test specimen to a temperature of approximately −140 °C at a rate of 10 °C/min or 20 °C/min and hold at this temperature for 1 min to 10 min until the baseline becomes stable.

A starting temperature of −140 °C is required for the determination of rubbers with very low glass transition temperatures, e.g. high-cis polybutadiene. For rubbers with higher glass transitions, this temperature is not necessary.

A starting temperature should be chosen so that a stable base line is achieved before the glass transition region, e.g. about 30 °C to 40 °C below the expected glass transition temperature.

If the apparatus is not capable of maintaining the specified cooling rate, it should be adjusted to give a rate as close as possible to that specified.

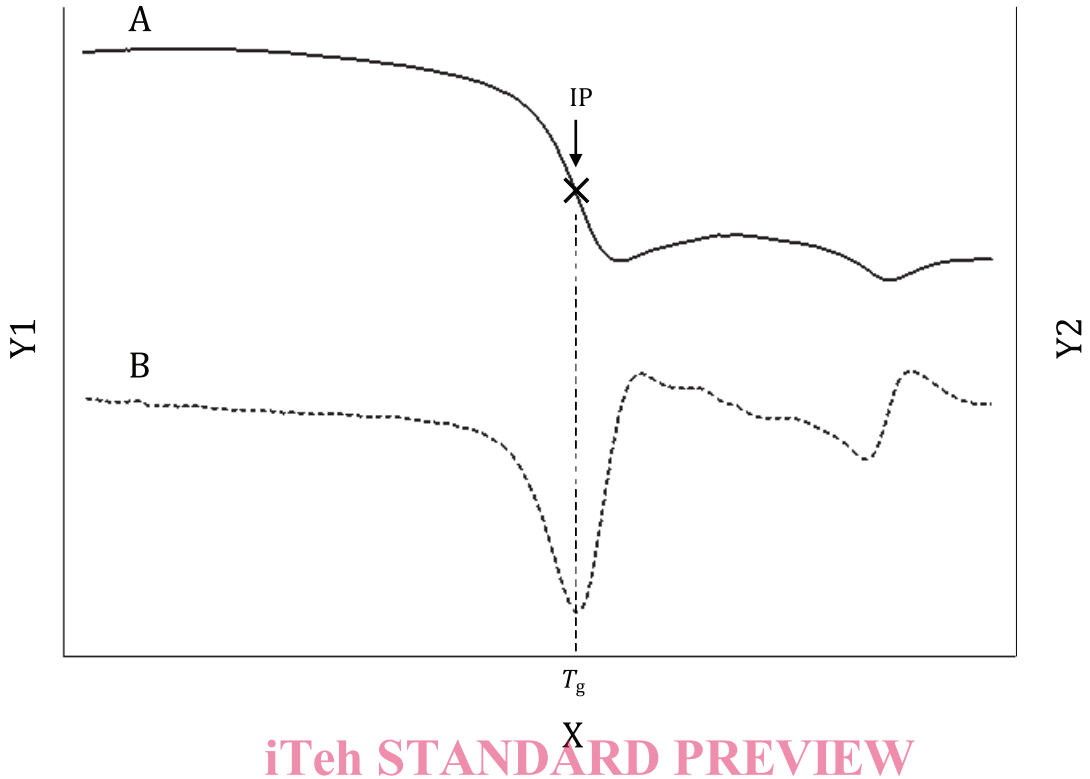
9.3.2 Perform the temperature scan at a heating rate of 20 °C/min, heating until a temperature about 30 °C above the upper limit of the glass transition range is reached.

NOTE Most instruments can be programmed to carry out the required thermal cycle automatically.

10 Expression of results

Determine the glass transition temperature as the inflection point of the transition curve using the instrument software. General DSC endothermogram and inflection point are given in Figure 1.

NOTE If the glass transition temperature has to be determined directly from the curve, a better indication of the position of the inflection point is given by studying the first derivative of the curve (DDSC thermogram). In the case of the representation of the exotherm curve, this is the peak minimum.



Key

A DSC thermogram
B DDSC thermogram
Y1 axis of DSC (exothermic direction)
Y2 axis of DDSC
X temperature °C
IP inflection point

Figure 1 — General thermogram and inflection point

11 Test report

The test report shall include the following:

a) a reference to this document, i.e. ISO 22768;

b) identification of the sample;

c) the mass of the specimen, in grams;

d) the type of DSC instrument used;

e) the type of inert gas and the flow rate;

f) the calibrants used;

g) the thermal cycle used;

h) the T_g value in degrees Celsius, together with the DSC curve;

i) the date of the test.

12 Precision

See Annex A.