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Raw rubber and rubber latex — Determination of the glass transition temperature by differential scanning calorimetry (DSC)

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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of 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 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 third edition cancels and replaces the second edition (ISO 22768:2017), which has been technically revised.

The main changes compared to the previous edition are as follows:

— rubber latex has been added to the scope and to Clause 6 as 6.2;

— a new Annex B on the precision of rubber latex has been added.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Raw rubber and rubber latex — 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.

1 Scope

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

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 124:2014, *Latex, rubber — Determination of total solids content*

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:

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

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

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 *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 test sample 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 ±0,1 mg.

5.5 Oven, capable of being maintained at 105 °C ± 5 °C.

6 Preparation of the test sample

6.1 Raw rubber

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.

6.2 Rubber latex

Dry rubber latex samples at 105 °C± 5 °C in accordance with ISO 124:2014, 6.2. Remove the rubber latex film and cut into pieces about 2 mm × 2 mm.

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.