INTERNATIONAL STANDARD

ISO 28343

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Rubber compounding ingredients — Process oils — Determination of glass transition temperature by DSC

Ingrédients de mélange du caoutchouc — Huiles de mise en œuvre — Détermination de la température de transition vitreuse par DSC

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 28343 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*.

Rubber compounding ingredients — Process oils — Determination of glass transition temperature by DSC

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard 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 International Standard specifies a method for the determination, by differential scanning calorimetry (DSC), of the glass transition temperature, T_g, of process oils used in rubber compounding.

NOTE The same oils are used as extender oils for synthetic rubbers.

2 Normative references

The following referenced documents are indispensable for the application 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 1382, *Rubber — Vocabulary*

ISO 3170, *Petroleum liquids — Manual sampling*

ISO 11357-1:2009, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1382 and ISO 11357-1 and the following apply.

3.1
T_g
glass transition temperature
approximate midpoint of the temperature range over which the glass transition takes place

NOTE For the purposes of this International Standard, the glass transition temperature is defined as the point of inflection of the DSC curve. The point of inflection corresponds to the calculated maximum of the first-derivative curve.

4 Reagents and materials

4.1 Dry helium gas, purity >99,999 % or in accordance with the differential scanning calorimeter manufacturer's instructions.

4.2 If helium is not available, use **dry nitrogen gas** (purity >99,999 % or in accordance with the differential scanning calorimeter manufacturer's instructions).

5 Apparatus

5.1 Differential scanning calorimeter, equipped with a liquid-nitrogen cooling system so that a temperature of around −150 °C can be attained.

Two types of instrument can be used:

— a “heat-flow” analyser[1)], in which the temperature of the test sample is increased or decreased and the variation in the flow of heat emitted or received by the sample is monitored;

— a “power-compensation” analyser[2)], in which the temperature of the test sample is increased or decreased and the energy necessary to keep the sample at the programmed temperature is monitored.

5.2 Sample pans and lids, made of aluminium, with a volume of about 40 µl, suitable for use with the analyser.

5.3 Suitable press, to seal the pans.

5.4 Analytical balance, accurate to 0,1 mg.

6 Storage and laboratory conditions

Store the oil sample in a suitable ventilated laboratory cupboard.

Prepare the sample pan under an extraction hood.

The DSC instrument should be operated in a laboratory kept at standard conditions as specified in the instrument manufacturer's manual.

7 Calibration

Temperature calibration shall be carried out in the desired temperature range in accordance with the equipment manufacturer's instructions or in accordance with ISO 11357-1:2009, Clause 8.

The heating rate, the type of purge gas and the purge gas flow rate need to be the same for calibration as for the measurement of a test sample.

8 Sampling

Carry out sampling in accordance with ISO 3170.

1) Examples of suitable heat-flow analysers are the Mettler Toledo DSC 821 and DSC 822, the TA Instruments DSC 2920 and the Netzsch DSC 204. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these instruments.

2) An example of a suitable power-compensation analyser is the PerkinElmer Pyris®. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this instrument.

9 Procedure

9.1 Principle

The glass transition temperature of the process oil is measured by differential scanning calorimetry. During the glass transition, the specific heat changes. The differential scanning calorimeter is able to detect accurately this change in specific heat, thus allowing the temperature at which the change occurs, T_g, to be determined.

9.2 Experimental conditions

9.2.1 Preparation of the pans

The mass of sample used, the type of pan and the lid-crimping operation are very important parameters. For each instrument, the manufacturer's instructions shall be strictly followed.

Use a test sample mass between 5 mg and 15 mg.

9.2.2 Measurements

Adjust the helium (or nitrogen) gas flow in accordance with the instrument manufacturer's recommendation.

NOTE Typically, a value between 20 ml/min and 50 ml/min is used for either gas.

When results obtained by different parties are going to be compared, it is recommended that the same type of purge gas be used.

Place the test sample in the instrument at room temperature.

Raise the temperature to 60 °C at a rate of 30 °C/min to remove any thermal history of the sample.

Cool to −140 °C at a rate of 10 °C/min and maintain the sample at this temperature for 5 min.

Record the thermogram from −140 °C to 60 °C at a heating rate of 20 °C/min.

9.2.3 Evaluation

The recorded curve is evaluated using equipment-specific software, defining the appropriate limits corresponding to the transition range.

The glass transition temperature, T_g, is determined from the point of inflection of the recorded curve, which corresponds to the maximum in the first-derivative curve (see Example 1 in Annex A).

In cases when the glass transition is followed by a crystallization peak, T_g is also determined from the point of inflection (see Example 2 in Annex A).

By agreement between the interested parties, the so-called midpoint method may be used to determine T_g. In this case, T_g is measured at the point of intersection of the curve with the straight line equidistant from the two straight lines obtained by extrapolating the baselines before and after the transition.

10 Expression of results

Report the temperature corresponding to the point of inflection as the glass transition temperature, T_g, expressed in degrees Celsius.

11 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) all details necessary for the identification of the oil sample(s) tested;

c) the mass of the test sample;

d) the DSC instrument used [name of instrument and type of analyser (“heat flow” or “power compensation”)];

e) the purge gas used (helium or nitrogen) and the gas flow rate, in millilitres per minute;

f) the value of T_g, in degrees Celsius, together with a copy of the recorded DSC curve;

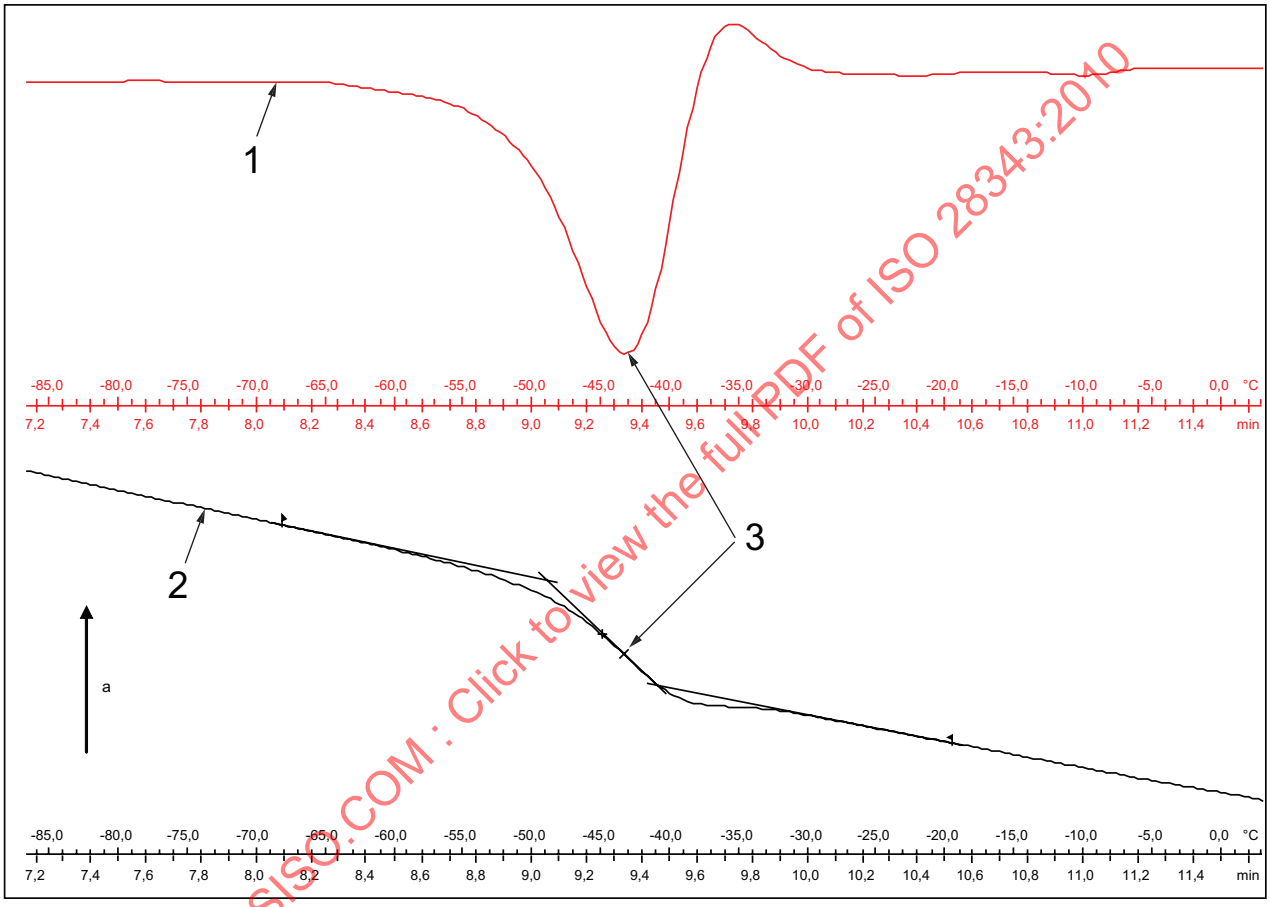
g) details of any operation not included in this International Standard, as well as any observations which could have a bearing on the result;

h) the date of the test.

Annex A
(informative)

Determination of T_g from the point of inflection

EXAMPLE 1 No crystallization after the glass transition



Key

1 first-derivative curve

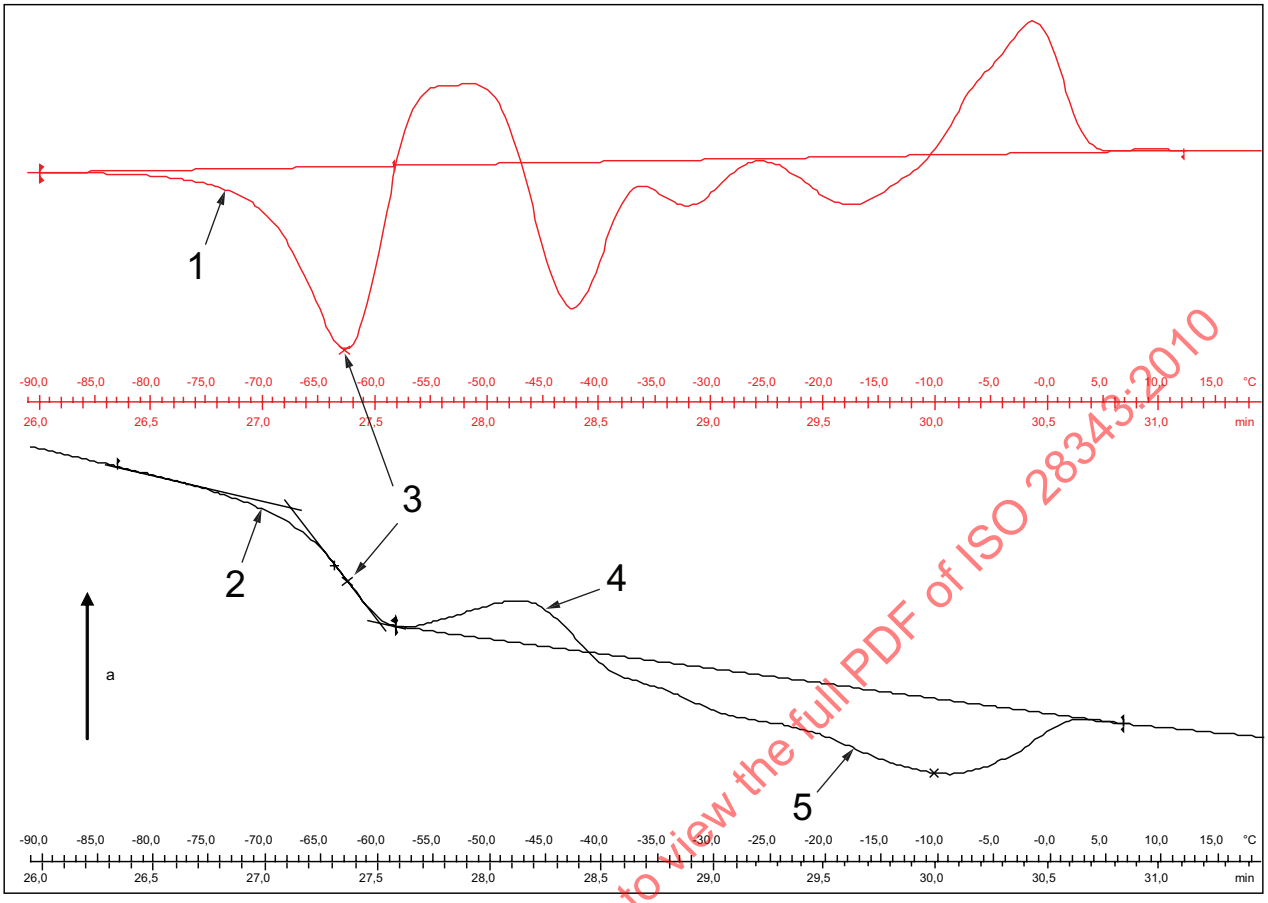
2 recorded curve

3 glass transition temperature (inflection point at –43,41 °C)

a Exothermic.

Figure A.1 — Oil showing no crystallization after the glass transition

EXAMPLE 2 Glass transition followed by crystallization



Key

1 first-derivative curve
2 recorded curve
3 glass transition temperature (inflection point at –62,73 °C)
4 crystallization peak
5 melting

a Exothermic.

Figure A.2 — Oil showing crystallization after the glass transition

Annex B
(informative)

Precision data

B.1 General

The precision of this test method was determined in accordance with ISO/TR 9272. Refer to ISO/TR 9272 for terminology and other statistical details.

The precision results merely give an estimate of the precision to be expected. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific test protocols that include this test method.

An interlaboratory test programme was conducted to determine a type 1 precision. Both the repeatability and the reproducibility determined represent short-term testing conditions. Eleven laboratories tested one process oil (MES oil). The number of within-laboratory replicates was two and the time span for repeatability was 7 days. Each laboratory carried out two replicates per day.

The results of the precision calculations are given in Table B.1.

Table B.1 — T_g measured from inflection point

Material	Mean value of T_g °C	Within-laboratory			Between laboratories		
		s_r	r	(r)	s_R	R	(R)
MES oil	–61,11	0,368	1,04	1,7	0,704	1,99	3,3
Number of laboratories: 11, number of replicates: 2.							
s_r is the within-laboratory standard deviation;							
s_R is the between-laboratory standard deviation;							
r is the repeatability, in measurement units;							
(r) is the repeatability, in percent (this value represents percent relative);							
R is the reproducibility, in measurement units;							
(R) is the reproducibility, in percent (this value represents percent relative).							

B.2 Repeatability

The repeatability (r), in percent relative, has been established as 1,7 %. Two single test results (or determinations) that differ by more than 1,7 % should therefore be considered suspect and dictate that some appropriate investigative action be taken.

B.3 Reproducibility

The reproducibility (R), in percent relative, has been established as 3,3 %. Two single test results (or determinations), obtained in separate laboratories, that differ by more than 3,3 %, should therefore be considered suspect and dictate that some appropriate investigative action be taken.