DRAFT INTERNATIONAL STANDARD ISO/DIS 1432

ISO/TC **45**/SC **2** Secretariat: **JISC**

Voting begins on **2011-04-12**

Voting terminates on **2011-09-12**

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Rubber, vulcanized or thermoplastic — Determination of low temperature stiffening (Gehman test)

Caoutchouc vulcanisé ou thermoplastique — Détermination de la rigidité à basse température (Essai Gehman)

[Revision of third edition (SO 1432:1988) and ISO 1432:1988/Cor.1:2003]

ICS 83.060

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Foreword

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ISO 1432 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 1432:1988), which has been technically revised to allow automatic computerized instruments and it also incorporates Technical Corrigendum 1 from 2003.

Rubber, vulcanized or thermoplastic — Determination of low temperature stiffening (Gehman test)

WARNING 1 — 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.

WARNING 2 — Certain procedures specified in this International Standard might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This International Standard specifies a static procedure, known as the Gehman test, for determining the relative stiffness characteristics of vulcanized or thermoplastic rubbers over a temperature range from room temperature to approximately -150 °C.

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 18899:2004, Rubber — Guide to the calibration of test equipment

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

3 Principle

The torsional stiffness is measured as a function of temperature, starting from a low temperature. The stiffness is measured by connecting the test piece in series with a calibrated spring wire and measuring the angle of twist of the test piece when the top of the wire is turned through 180^0.

4 Apparatus

4.1 Torsion apparatus, consisting of a torsion head, capable of being turned 180° in a plane normal to the torsion wire. The top of the wire is fastened to the torsion head. The bottom of the wire is fastened to the test piece clamp. A device for "friction-free" indication or recording of angle by mechanical or electrical means shall be provided permitting convenient and exact adjustment of the zero point. Indicating or recording system shall allow reading or recording of the angle of twist to the nearest degree. It is advantageous to make the vertical portion of the stand from material of poor thermal conductivity. The base of the stand shall be of stainless steel or other corrosion-resistant material. The principle is shown in figure 1.

4.2 Torsional wires, made of tempered spring wire, of length 65 mm ± 8 mm, and having nominal torsional constants of 0,7 mN.m, 2,8 mN.m and 11,2 mN.m.

In cases of dispute, the 2,8 mN.m wire shall be used.

4.3 Test piece rack, made of material of poor thermal conductivity, for holding the test piece in a vertical position in the heat transfer medium. The rack may be constructed to hold several test pieces. The rack is attached to a stand.

Two clamps shall be provided for holding each test piece. The bottom clamp shall be attached to the test piece rack.

The top clamp acts as an extension of the test piece and shall not touch the rack. The top clamp is connected to the torsion wire by a rod.

4.4 Heat-transfer medium, liquid or gaseous, which remains fluid at the test temperature and which does not appreciably affect the material being tested, as prescribed in ISO 23529.

Gases can be employed as the heat transfer medium provided the design of the apparatus is such that results obtained using them will duplicate those obtained with liquids.

The following fluids have been used satisfactorily:

a) for temperatures down to – 60°C, silicone fluids of kinematic viscosity of about 5 mm2/s at ambient temperature, which are usually suitable owing to their chemical inertness towards rubbers, their non-flammability and their non-toxicity;

b) for temperatures down to – 73 °C, ethanol;

c) for temperatures down to – 120 °C, methylcyclohexane cooled by liquid nitrogen (found to be satisfactory with the use of suitable apparatus).

4.5 Temperature-measuring device, capable of measuring temperature to within 0,5 °C over the whole range of temperature over which the apparatus is to be used.

The temperature sensor shall be positioned near the test pieces.

4.6 Temperature control, capable of maintaining the temperature of the heat-transfer medium to within ±1°C.

4.7 Container for heat-transfer medium, a bath for a liquid medium, or a test chamber for a gaseous medium, with means of heating the coolant

4.8 Stirrer, for liquids, or **fan** or **blower**, for gases, which ensures thorough circulation of the heat-transfer medium. It is important that the stirrer also moves the liquid vertically to ensure a uniform temperature in the liquid.

4.9 Stopwatch, or other timing device, calibrated in seconds.

5 Calibration

The requirements for calibration of the test apparatus are given in Annex A.

6 Test piece

6.1 Preparation of test piece

Test pieces shall be prepared in accordance with ISO 23529.

The dimensions of the test piece shall be 40 mm ± 2,5 mm, 3 mm + 0,2 mm and 2 mm ± 0,2 mm. It shall be moulded or cut with a suitable die from a moulded sheet of suitable thickness.

6.2 Conditioning of test piece

6.2.1 The minimum time between forming and testing shall be 16 h.

For non-product tests, the maximum time between forming and testing shall be 4 weeks and, for evaluations intended to be comparable, the tests should be carried out, as far as possible, after the same time interval.

For product tests, whenever possible, the time between forming and testing should not exceed 3 months. In other cases, tests shall be made within 2 months of the date of receipt by the customer of the product.

6.2.2 Samples and test pieces shall be protected from light as completely as possible during the interval between forming and testing.

6.2.3 Prepared test pieces shall be conditioned in accordance with ISO 23529 immediately before testing for a minimum of 3 h at a Standard laboratory temperature, the same temperature being used throughout any one test or series of tests intended to be comparable.

7 Procedure

7.1 Mounting of test piece

Clamp each test piece used in such a manner that 25 mm ± 3 mm of the test piece is free between the clamps. The test piece clamp shall be located in such a position that the specimen is under zero torque or with a slight pre-load to keep the test piece straight when the temperature changes.

If the absolute modulus is required measure the test length of the test pieces to the nearest 0,5 mm, the width to the nearest 0,1 mm and the thickness to the nearest 0,01 mm.

7.2 Stiffness measurements in liquid media, manual instruments

Place the rack containing the test pieces in the liquid bath with a minimum of 25 mm of liquid covering the test pieces. Then adjust the temperature of the bath to 23 °C ± 2 °C. Connect one of the test pieces to the torsion head by means of the screw connector and the standard torsion wire (2,8 mNm) .

Take care when attaching the screw connector to the test piece clamp stud not to move the stud from the zero torque position. The torsion head shall also remain in the zero position while the connector is being fastened to the stud. The spacer which provides clearance between the test piece rack and the test piece clamp stud need not be used for measurements made at room temperature.

NOTE Clearance between the top of the test piece rack and the test piece clamp stud is ensured by inserting thin spacers between the two. Slotted laminated plastics of thickness about 1,3 mm and width about 12 mm have been found satisfactory. At low temperatures the test pieces stiffen in position and the spacers may be removed without losing the clearance.

Adjust the angle indicating or recording device to zero. Then turn the torsion head quickly but smoothly through 180° and record the torsion angle after 10 s. If the reading at 23 °C does not fall in the range of 120° to 170° the Standard torsion wire is not suitable for testing the test piece. Test pieces producing twists of more than 170° shall be tested with a wire having a torsional constant of 0,7 mNm. Test pieces producing twists of less than 120° shall be tested with a wire having a torsional constant of 11,2 mNm.

Return the torsion head to its initial position and disconnect the test piece.

Then move the test piece rack to bring the next test piece into position for measurement.

Note: Apparatus is now in use in which the rack is stationary while the torsion head is movable and can be positioned over each test piece in turn.

Measure all the test pieces in the rack at 23 °C ± 2 °C.

Insert the spacers between the test piece rack and the test piece clamp studs. Remove the test pieces from the liquid bath and adjust the temperature of the liquid to the lowest temperature desired. Replace the test pieces in the bath and maintain them at this temperature for approximately 15 min. After this, remove one spacer and measure one test piece as was done at 23 °C. Return the spacer to its original position after the test piece has been tested. Measure all the test pieces in the rack in this way, ensuring that the measurements are completed within approximately 2 min.

NOTE Movement of the spacer might alter the setting of the angle indicating or recording device; therefore, adjust this device to zero after removing the spacer

Then increase the bath temperature by one of the two following methods :

a) by stepwise 5 °C intervals, each increase being made after approximately 5 min;

b) increase the temperature continuously with a heating rate of about 1 °C/min.

Make the stiffness measurement in the stepwise case after conditioning of the test piece for 5 min at each temperature and in the continuous case at maximum 5 min intervals. Continue the tests until a temperature is reached at which the angular twist is within 5° to 10° of the twist at 23 °C.

7.3 Stiffness measurements in liquid media, automatic instruments

Make the reference measurement with the standard torsion wire (2,8 mNm) at 23 °C, either in air or in the liquid bath.

If the test is made in the liquid, place the rack containing the test pieces in the liquid bath with a minimum of 25 mm of liquid covering the test pieces.

Adjust the angle measuring device to zero. Then turn all test pieces at the same time quickly but smoothly through 180° and record the torsion angle after 10 s. If the reading at 23 °C does not fall in the range of 120° to 170° the standard torsion wire is not suitable for testing the test piece. Test pieces producing twists of more than 170° shall be tested with a wire having a torsional constant of 0,7 mN·m. Test pieces producing twists of less than 120° shall be tested with a wire having a torsional constant of 11,2 mN·m.

If the reference measurement has been made in the liquid, remove the test pieces from the liquid bath and adjust the temperature of the liquid to the lowest temperature desired. Replace the test pieces in the bath and maintain them at this temperature for approximately 15 min.

Then increase the temperature continuously with a heating rate of about 1 °C/min.

Make the stiffness measurement at regular intervals of 1 min. Continue the tests until a temperature is reached at which the angular twist is within 5° to 10° of the twist at 23 °C.

The relative modulus may be plotted in a graph during the test.

7.4 Stiffness measurement in gaseous media

7.4.1 General

Procedures with air, carbon dioxide or nitrogen differ from those with liquid media only in that cooling is done with the test pieces in the medium and the length of the conditioning period is different.

7.4.2 Increase of temperature in steps

With the test pieces in the test chamber, adjust the temperature of the chamber to the lowest temperature desired in approximately 30 min. After this temperature has been maintained constant for 10 min, make the measurements in a similar way as in the liquid media, ensuring that all the test pieces in the rack are tested within 2 min.

Increase the temperature of the chamber by 5 °C intervals, each increase being made in approximately 10 min, and make stiffness measurements after conditioning of the test pieces for 10 min at each temperature.

7.4.3 Continuous increase of temperature

With the test pieces in the test chamber adjust the temperature of the chamber to the lowest temperature desired, by application of a linear time programme, preferably with a rate of 3 °C/min. After this temperature has been reached, increase the temperature linearly at a rate of about 1 °C/min. Carry out measurements of the twist angle at 5 °C intervals.

7.5 Crystallization

When it is desired to study crystallization or plasticizer effects, the time of conditioning at the desired temperature should be increased.

8 Number of tests

At least three test pieces of each material shall be tested. It is good practice to include a control rubber with known twist temperature characteristics.

9 Expression of results

9.1 Twist versus temperature curve

Plot a graph of the angles of twist of the test piece against the temperature.

9.2 Torsional modulus

The torsional modulus of the test piece at any temperature is proportional to the quantity

$$\frac{180-\alpha}{\alpha}$$

where α is the angle of twist, in degrees, of the test piece.

9.3 Relative modulus

The relative modulus at any temperature is the ratio of the torsional modulus at that temperature to the torsional modulus at 23 °C.