ISO TC 45/SC 2

Date: 2012-06-28

ISO FDIS 13145:2012(E)

ISO TC 45/SC 2/WG

ISO/FDIS 13145:2023(E)

Date: 2023-04-18

ISO TC 45/SC 2/WG

Secretariat: JISCJISC

Rubber — Determination of viscosity and stress relaxation using a rotorless sealed shear rheometer

Caoutchouc — Détermination de la viscosité et de la relaxation de contrainte au moyen d'un rhéomètre à cisaillement sans rotor étanche

Document type:
Document subtype:
Document stage:
Document language:

Document5

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Foreword

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

This second edition cancels and replaces the first edition (ISO 13145:2012), of which it constitutes a minor revision. The changes are as follows:

— the reference ISO 18899 has been revisedupdated with the latest edition.

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

Introduction

The rheological properties of rubbers are related to their structural characteristics and will influence the behaviour of the rubber during processing and the performance of the final product.

For these reasons, the industrial environment requires instruments that can quickly and easily evaluate the rheological properties.

As a consequence, this standard test method was formulated using a rotorless sealed shear rheometer for rheological evaluation under defined conditions.

This test couldcan be an alternative to the Mooney viscometer, still used as standard in many parts of the rubber industry to measure Mooney viscosity (in accordance withaccording to ISO 289-1). The defined conditions have been selected to provide a shear rate range similar to that used for Mooney viscosity and a good repeatability level.

This new test procedure should beis performed over a short time and preferably in the automatic mode to optimize test efficiency.

Rubber — Determination of viscosity and stress relaxation using a rotorless sealed shear rheometer

WARNING — Persons using this ~~International Standard~~document should be familiar with normal laboratory practice. This ~~standard~~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 ~~International Standard~~document describes a method for the determination of the viscosity and stress relaxation of raw or compounded rubber under specified conditions.

The viscosity determination consists of a constant strain, temperature and frequency test in which the elastic and the loss components of the complex shear modulus can be determined.

The determination of stress relaxation consists of a constant static strain and temperature test in which the torque decrease can be determined.

2 Normative references

The following ~~referenced~~ documents are ~~indispensable for~~referred to in the ~~application~~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 1382, *Rubber — Vocabulary*

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 1382 and the following apply.

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

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

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

3.1
rotorless sealed shear rheometer
device consisting of two dies forming a temperature-controlled cavity, one of which is moved relative to the other to apply a stress or strain to the test piece

3.2
sinusoidal strain

$\gamma(t)$
strain produced by the oscillation of the die constituting the test cavity

NOTE Note 1 to entry: It is given by the expression $\gamma(t) = \gamma_0 \sin(\omega t)$, where γ_0 is the maximum amplitude of the applied strain.

3.3
loss angle
δ
phase angle between the stress and the strain

NOTE Note 1 to entry: This is a measure of the presence and extent of viscous behaviour in a material. For viscoelastic materials, the phase angle can assume a value between 0° and 90°. 90° is an ideal Newtonian liquid.

3.4
complex torque
*S**
torque measured by the machine due to application of sinusoidal strain

NOTE Note 1 to entry: The complex torque is a vector which can be represented by a complex number, viz $S^* = S' + iS''$.

3.5
elastic torque
S′
component of torque that is in phase with the imposed sinusoidal strain

NOTE Note 1 to entry: It is given by the equation $S' = |S^*|\cos\delta$.

3.6
loss torque
S″
component of torque that is in quadrature with the imposed sinusoidal strain

NOTE Note 1 to entry: It is given by the equation $S'' = |S^*|\sin\delta$.

3.7
complex shear modulus
*G**
ratio of the shear stress to the shear strain, where each is a vector which can be represented by a complex number

NOTE Note 1— to entry: It is given by the equation $G^* = G' + iG''$.

NOTE 2 Note 2 to entry: The complex shear modulus is determined by dividing the complex torque S^* by the applied strain and multiplying by a geometric factor related to the cavity shape.

3.8
elastic shear modulus
G′
component of the applied shear stress that is in phase with the shear strain, divided by the strain

NOTE Note 1 to entry: It is given by the equation $G' = |G^*|\cos\delta$.

3.9

loss shear modulus
G"
component of the applied shear stress which is in quadrature with the shear strain, divided by the strain

~~NOTE~~ Note 1 to entry: It is given by the equation $G'' = |G^*|\sin\delta$.

3.10
tangent of the loss angle
tan*δ*
ratio of the loss modulus to the elastic modulus

4 Principle

The torque generated in a test piece contained in a heated sealed cavity formed by two dies, one of which can be oscillated through a small amplitude, is measured.

5 Apparatus

5.1 General

A rotorless sealed shear rheometer consists of two dies that are heated and closed, under a specified force, to form a sealed test cavity that contains the test piece. One of the dies oscillates, and a measuring system records the torque required to produce the relative movement. The elastic torque *S'* and the loss torque *S"* produced in a test piece by the strain due to the oscillation of the die can be measured at specified conditions of temperature, frequency and amplitude.

The general arrangement of a rotorless sealed shear rheometer is shown in Figure 1, including typical machine dimensions.

5.2 Die cavity

The dies shall be manufactured from a stiff material. The surface of the dies shall be treated to minimize the effect of test piece contamination if protective or carrying film are not used and shall be hard enough to prevent wear. A minimum Rockwell hardness of 50 HRC, or equivalent, is recommended. The tolerances necessary on the dimensions of the dies depend on the particular design, but as a general guide the dimensions of the cavity should be controlled to ±0,2 %.

The top and bottom surfaces of the cavity shall have a pattern of grooves of dimensions sufficient to prevent slippage of the rubber test piece.

Holes shall be provided in both the upper and lower dies to accommodate temperature sensors. The positions of the sensors relative to the cavity shall be controlled to ensure a reproducible response.

A seal of suitable low, constant friction shall be provided to prevent material leaking from the cavity.

Suitable means shall be employed, by design of the dies or otherwise, to apply pressure to the test piece throughout the test to minimize slippage between the disc and the rubber.

5.3 Die closure

The dies shall be closed and held closed during the test by, for example, a pneumatic cylinder.

The closing force required depends on the clearance area; as a general guide, a minimum of 7 kN is recommended. The contact of the die cavity edges shall be such as to form a perfectly sealed cavity.

5.4 Movement

The moving part in a rotorless instrument is one of the dies. The dies are usually biconical to produce a substantially uniform shear rate, and this shape is useful to make the test piece loading and unloading stages easier. The drive linkage shall be sufficiently stiff to prevent significant deformation.

A torsional oscillating movement shall be applied to one of the dies (typically the lower in the cavity) by means of a motor.

The frequency of oscillation can be varied according to the instrument specification, but in this International Standard a single frequency is selected.

The oscillation amplitude θ may be varied according to the deformation required. The maximum amplitude of the applied strain y_0 is calculated considering the oscillation angle used in the test and the die geometry (conical). It is given by the expression

$$y_0 = \theta / \varphi$$

where φ is the characteristic angle of the conical die.

Generally, greater sensitivity can be obtained with larger amplitudes, but the amplitude that can be used in practice is restricted by the possibility of slippage between the test piece and the die surface.

5.5 Heating and temperature control

The heating and temperature control system shall be capable of producing a reproducible and evenly distributed temperature in the dies and shall permit fast and reproducible temperature recovery after insertion of the test piece.

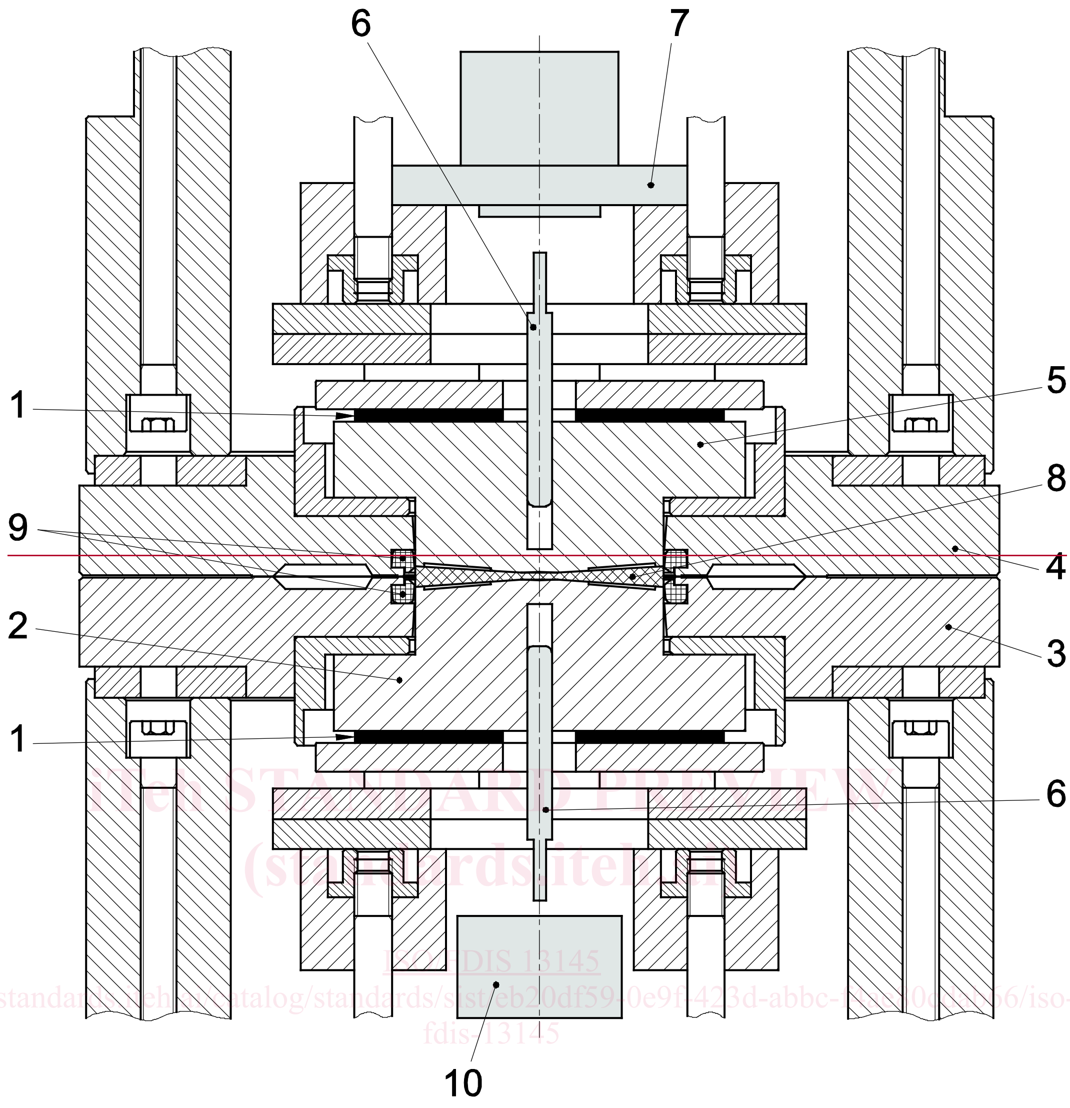
The temperature-measuring system shall enable the temperature to be measured to a resolution of ±0,1 °C over the range from 60 °C to 200 °C. The temperature controllers shall enable the temperature of the dies to be controlled to an accuracy of ±0,3 °C in the steady state.

5.6 Torque-measuring system

A suitable transducer shall be provided which is capable of measuring the force or torque with an accuracy of ±1 %.

A recorder shall be provided to continuously monitor force or torque. It shall have a response time for full-scale deflection equal to or below 1 s.

Dimensions in millimetres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