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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

Reference number
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Contents

Page

Foreword

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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 13145 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

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 could be an alternative to the Mooney viscometer, still used as standard in many parts of the rubber industry to measure Mooney viscosity (in accordance with 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 be 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 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 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 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 18899:2004, *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.

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 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 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 The complex torque is a vector which can be represented by a complex number, viz $S^* = S' + \mathrm{i}S''$.

3.5

elastic torque

S'

component of torque that is in phase with the imposed sinusoidal strain

NOTE 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 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 1 It is given by the equation $G^* = G' + \mathrm{i}G''$.

NOTE 2 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 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 It is given by the equation $G'' = |G^*|\sin\delta$.

3.10

tangent of the loss angle

tanδ

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.

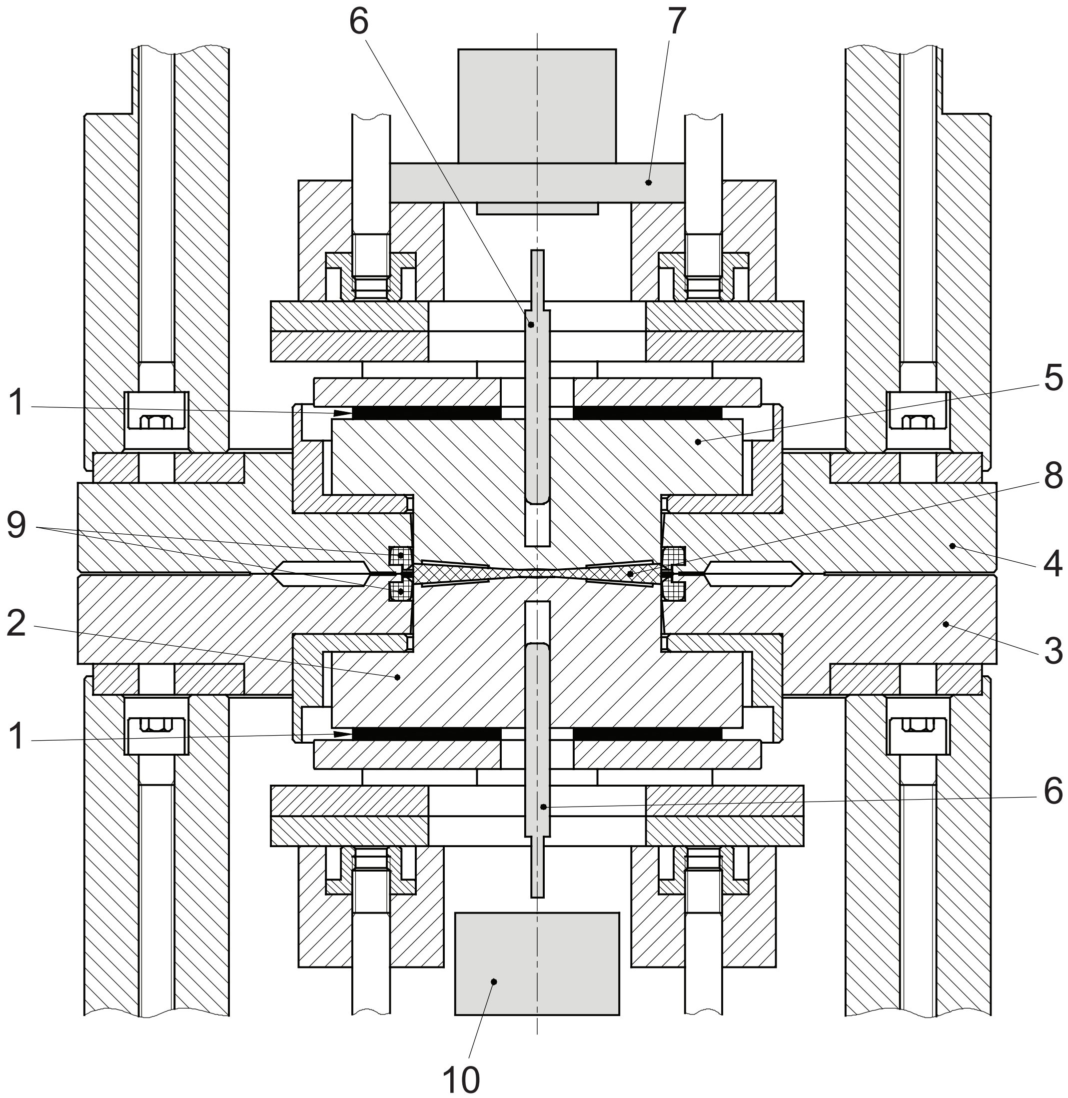
The temperature-measuring system shall enable the temperature to be measured to a resolution of ±0,1 °C over the range from 60 °C to 200 °C. The temperature controllers shall enable the temperature of the dies to be controlled to an accuracy of ±0,3 °C in the steady state.

5.6 Torque-measuring system

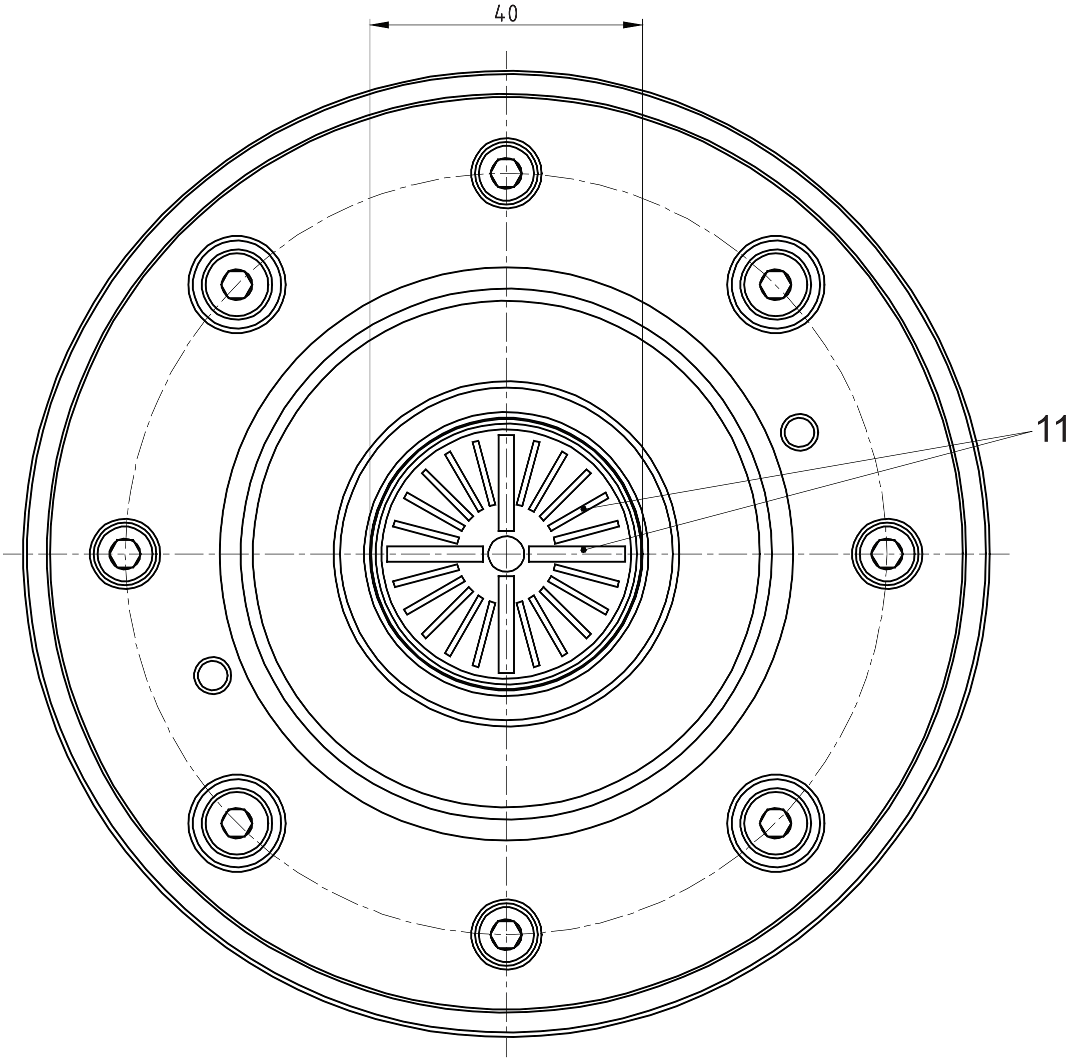
A suitable transducer shall be provided which is capable of measuring the force or torque with an accuracy of ±1 %.

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.

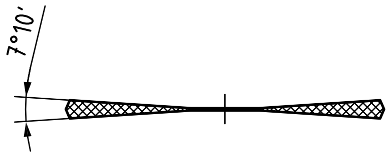
Dimensions in millimetres



a) Measurement principle



b) Die (upper and lower)



c) Test piece

Key

1	heater	7	torque-measuring system
2	lower die	8	test piece
3	lower seal plate	9	seals
4	upper seal plate	10	oscillating-drive system
5	upper die	11	grooves
6	temperature sensor		

Figure 1 — Typical rotorless sealed shear rheometer with biconical-die structure

6 Calibration

The test apparatus shall be calibrated in accordance with the schedule given in Annex A.

Calibration of rheometers shall be carried out in accordance with the manufacturer's instructions. The force or torque shall be determined at several points over the range(s) used but, additionally, it can be useful to have provision to make in-use checks.

Stable rubber reference compounds can also be tested periodically to check for consistent performance.

7 Test piece

7.1 Preparation of the test piece

The test piece shall be homogeneous and, as far as possible, free from trapped air.

The pertinent volume shall be determined by appropriate tests, and test pieces of equal volume shall be used to obtain reproducible results. This volume shall be slightly larger than the die cavity volume (typically 130 % to 150 % larger) so that a small amount of material is extruded between all edges of the dies when they are closed.

This volume corresponds to 5 cm^3 and 6 cm^3 for the typical sealed rheometer shown in Figure 1.

The test piece shall be punched out by an appropriate device which ensures the production of test pieces of constant volume.

For best repeatability, the mass of the test piece shall be controlled to within ±0,5 g to obtain the required volume.

Conditioning the test piece at 23 °C ± 5 °C for a minimum of 3 h before testing is recommended.

7.2 Protective films

One or more layers of protective film (approximately 0,025 mm thick) may be inserted between the test piece and the dies.

Film introduction can be useful to avoid frequent cavity cleaning, since material remaining in the die cavity can influence the test results. Moreover, the use of film can allow tests to be performed in the automatic mode as the biconical shape of the dies allows the films to slide, thus permitting test pieces to be loaded and unloaded automatically.

The film selected shall not react with the test pieces. However, the presence of films could affect the test results so, if they are used, their specification (material, maximum temperature of use, thickness, stiffness) shall be reported.

NOTE Materials that have been found suitable include cellophane, polyester, nylon, high-density polyethylene (at 100 °C only), plain, uncoated fabric, and similar materials.

8 Temperature

The temperature recommended for this test method is 100 °C. However, the temperature may be varied as required within the specified range of the machine.

NOTE Too high a temperature can cause polymer degradation.

9 Procedure

9.1 Testing sequence

The test method specified in this International Standard consists of three different steps applied in sequence on the same test piece:

a) **Preheat**: 1 min at 100 °C with cavity closed and both dies stationary.

b) **Time sweep** at 100 °C: The lower die oscillates with a frequency of 0,1 Hz, applying a strain of 150 % for six oscillations in total.

c) **Conditioning and stress relaxation**: The test piece is kept for 1 min at 100 °C with the dies stationary (this conditioning is necessary for stress recovery after the time sweep). Then a step strain of 150 % is applied and kept constant for 35 s. During this time, the decreasing torque (stress relaxation) is measured. If stress relaxation is not needed, the third stage may be omitted.

Different temperatures may be considered for specific rubbers as agreed between the interested parties.

9.2 Preparation for the test

The temperature of both dies shall be brought to the test temperature with the cavity closed.

Any necessary zeroing, and selection of the range of the force- or torque-measuring device, shall be made before loading the test piece.

9.3 Rheometer loading

The loading of the test piece and the closure of the dies shall be carried out as quickly as possible. The dies shall be closed immediately after insertion of the test piece.

After removal of the tested piece, a further test piece may be inserted immediately if the temperature of the dies remains within ±0,3 °C of the set value. If not, the dies shall be closed and the temperature allowed to recover to the set value.

If film layers between the test piece and the dies are not used, a deposit of material from the tested polymer can build up on the dies, which might affect the final torque values. A reference polymer may be used to detect this occurrence. If such contamination occurs, it shall be removed. Great care shall be taken with cleaning, and the manufacturer’s advice followed.

10 Reporting of results

10.1 Examples of reporting results

Results shall be reported as given in the examples in Tables 1, 2 and 3, including the number of significant figures (for the purpose of the examples, the test piece identification is “AABB” and all reported test results are for reference only).

10.2 Basic expression of results

The elastic modulus G' at 150 % strain obtained by a time sweep test is the representative parameter to evaluate the viscosity behaviour of rubber (see 3.8).

Table 1 — Example of viscosity test report

Material ID code	**Material type**	**Rheometer type**	**Temperature** °C	**G' at 150 %** kPa	**Lower film**	**Upper film**
AABB	Laboratory test piece	Biconical Brand “A” Brand “B”	100	20,00	PET 1 layer 0,025 mm	PET 1 layer 0,025 mm

10.3 Additional results

For a more comprehensive characterization, the loss component G'' of the complex shear modulus and tanδ may be reported in addition to the elastic modulus G' at 150 % strain to characterize the viscous behaviour.

NOTE More details of the methods of calculation of these properties and the precautions necessary to give valid results are given in ISO 4664-1

Table 2 — Advanced viscosity test report

Material ID code	**Material type**	**Rheometer type**	**Temperature** °C	**G' at 150 %** kPa	**G'' at 150 %** kPa	**tanδ**	**Lower film**	**Upper film**
AABB	Laboratory test piece	Biconical Brand “A” Brand “B”	100	20,00	20,00	1,000	PET 1 layer 0,025 mm	PET 1 layer 0,025 mm

10.4 Stress relaxation test results

From stress relaxation, the percent torque reduction (with respect to the maximum value) after 1,0 s and 20,0 s may be reported for additional characterization of the rubber.

Table 3 — Stress relaxation test report

Material ID code	**Material type**	**Rheometer type**	**Temperature** °C	**Torque reduction after 1 s** %	**Torque reduction after 20 s** %	**Lower film**	**Upper film**
AABB	Laboratory test piece	Biconical Brand “A” Brand “B”	100	80,0	90,0	PET 1 layer 0,025 mm	PET 1 layer 0,025 mm

The torque reduction at a different time may be reported, if required.

11 Precision

See Annex B.

12 Test report

The test report shall include the following information:

a) sample details:

   1) a full description of the sample and its origin,

  2) the method of preparation of the test piece from the sample;

b) test method:

  1) a full reference to the test method used, i.e. the number of this International Standard;

c) test details:

  1) the laboratory temperature,

  2) the time and temperature of conditioning prior to the test,

  3) the type of rheometer used,

  4) specification of any film layers used,

  5) details of any procedures not specified in this International Standard;

d) test results:

  1) the number of test pieces tested,

  2) the individual test results,

  3) the mean results;

e) the date(s) of the test.

Annex A
(normative)

Calibration schedule

A.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded on any calibration report or certificate. It shall be reported whether calibration is carried out in the “as-received” condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

A.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this International Standard. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table A.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code-letter. The code-letters used in the calibration schedule are:

N initial verification only;

S standard interval as advised in ISO 18899;

U in use.

Table A.1 — Calibration frequency schedule

Parameter	Requirement	Subclause in ISO 18899:2004	Verification frequency guide	Notes
Surface hardness of the dies	> 50 HRC	15.5	N	
Dimensions of the dies	±0,2 %	15.2	S	
Die closing force	> 7 kN	21.3	S	
Oscillation frequency	±0,1 Hz ± 0,005 Hz	23.3	S	
Temperature resolution	±0,1 °C	18	N	
Temperature accuracy	±0,3 °C	18	S	
Temperature stability at steady state	Within ±0,3 °C	18	S	
Preheat time and stress recovery time	1 min	18	S	
Torque transducer accuracy	±1 %	21.4	S	
Time response of recorder	≤ 1 s	14.6	S	
Test piece volume	5 cm^3 to 6 cm^3	15.8	U	

In addition to the items listed in Table A.1, use of the following is implied, all of which need calibrating in accordance with ISO 18899:

- instruments for determining the dimensions of the dies;
- a load cell for checking the die-closing force;
- a timer;
- an instrument for strain measurement;
- a thermometer for monitoring the conditioning and test temperatures;
- a densimeter.

Annex B
(informative)

Precision data

An interlaboratory test programme was carried out in which 13 laboratories participated.

The following parameters were acquired for three different rubber test pieces:

- the elastic (G') and loss shear modulus (G'') and tanδ, calculated from the time sweep test as the average of the values acquired during the last three oscillations;
- from stress relaxation, the percent torque reduction (with respect to the maximum value) after 1,0 s and 20,0 s.

One layer of PET protective film (0,025 mm thick) was inserted between the test piece and each of the dies.

The number of within-laboratory replicates was two, and the time span for repeatability was 7 days.

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272.

A type 1 (interlaboratory) precision was determined.

The results are given in Tables B.1 to B.5.

The symbols s_r, r, (r), s_R, R and (R) as used in Tables B.1 to B.5 are defined as follows:

s_r	the standard deviation within a laboratory;
r	the absolute repeatability, in measurement units;
(r)	the relative repeatability, as a percentage of the measured values;
s_R	the standard deviation between laboratories;
R	the absolute reproducibility, in measurement units;
(R)	the relative reproducibility, as a percentage of the measured values.

Table B.1 — Precision data for G' at 150 % strain

Material	**Mean level** kPa	**Within lab**			**Between labs**		
		s_r	r	(r)	s_R	R	(R)
Nd BR	21,39	0,342	0,97	4,5	0,642	1,82	8,5
SBR 1723	17,86	0,302	0,86	4,8	0,498	1,41	7,9
TSR 20	52,19	1,424	4,03	7,7	2,110	5,97	11,4
Number of laboratories p = 13; number of materials q = 3; number of replicates n = 2.							

Table B.2 — Precision data for G'' at 150 % strain

Material	Mean level	Within lab			Between labs		
	kPa	s_r	r	(r)	s_R	R	(R)
Nd BR	29,31	0,313	0,89	3,0	0,576	1,63	5,6
SBR 1723	18,59	0,261	0,74	4,0	0,368	1,04	5,6
TSR 20	34,18	0,325	0,92	2,7	0,954	2,70	7,9
Number of laboratories p = 13; number of materials q = 3; number of replicates n = 2.							

Table B.3 — Precision data for tanδ at 150 % strain

Material	Mean level	Within lab			Between labs		
	kPa	s_r	r	(r)	s_R	R	(R)
Nd BR	1,36	0,011	0,03	2,3	0,014	0,04	2,9
SBR 1723	1,04	0,013	0,04	3,4	0,027	0,08	7,2
TSR 20	0,66	0,016	0,05	6,9	0,017	0,05	7,4
Number of laboratories p = 13; number of materials q = 3; number of replicates n = 2.							

Table B.4 — Precision data for percent torque reduction after 1,0 s

Material	Mean level	Within lab			Between labs		
	kPa	s_r	r	(r)	s_R	R	(R)
Nd BR	88,33	0,143	0,40	0,5	2,282	6,46	7,3
SBR 1723	81,83	0,186	0,53	0,6	1,990	5,63	6,9
TSR 20	63,21	0,536	1,52	2,4	2,297	6,50	10,3
Number of laboratories p = 13; number of materials q = 3; number of replicates n = 2.							

Table B.5 — Precision data for percent torque reduction after 20,0 s

Material	Mean level	Within lab			Between labs		
	kPa	s_r	r	(r)	s_R	R	(R)
Nd BR	97,41	0,191	0,54	0,6	0,305	0,86	0,9
SBR 1723	94,53	0,220	0,62	0,7	0,273	0,77	0,8
TSR 20	87,47	0,374	1,06	1,2	1,361	3,85	4,4
Number of laboratories p = 13; number of materials q = 3; number of replicates n = 2.							

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