INTERNATIONAL STANDARD

ISO 289-1

Fourth edition
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Rubber, unvulcanized — Determinations using a shearing-disc viscometer —

Part 1: Determination of Mooney viscosity

Caoutchouc non vulcanisé — Déterminations utilisant un consistomètre à disque de cisaillement —

Partie 1: Détermination de l'indice consistométrique Mooney

Reference number
ISO 289-1:2015(E)

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Foreword

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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).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary Information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 289-1:2014), which has been technically revised to improve the calibration schedule.

ISO 289 consists of the following parts, under the general title *Rubber, unvulcanized — Determinations using a shearing-disc viscometer*:

— *Part 1: Determination of Mooney viscosity*

— *Part 2: Determination of pre-vulcanization characteristics*

— *Part 3: Determination of the Delta Mooney value for non-pigmented, oil-extended emulsion-polymerized SBR*

— *Part 4: Determination of the Mooney stress-relaxation rate*

Rubber, unvulcanized — Determinations using a shearing-disc viscometer —

Part 1: Determination of Mooney viscosity

WARNING — Persons using this part of ISO 289 should be familiar with normal laboratory practice. This part of ISO 289 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 part of ISO 289 specifies a method using a shearing-disc viscometer for measuring the Mooney viscosity of uncompounded or compounded rubbers.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*

ISO 6508-1, *Metallic materials — Rockwell hardness test — Part 1: Test method*

ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*

ISO 18899:2013, *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 torque which has to be applied under specified conditions in order to rotate a metal disc in a cylindrical chamber formed from mating dies filled with rubber is measured. The resistance offered by the rubber to this rotation is expressed in arbitrary units as the Mooney viscosity of the test piece.

4 Apparatus

4.1 Typical shearing-disc viscometer

A typical shearing-disc viscometer (see Figure 1), consisting of

a) two dies to form a cylindrical cavity,

b) a rotor,

c) a means for maintaining the dies at a constant temperature,

d) a means for maintaining a specified closure pressure,

e) a means for rotating the rotor at constant angular velocity, and

f) a means for indicating the torque required to rotate the rotor.

The rotor and die cavity have the dimensions shown in Table 1.

Table 1 — Dimensions of essential parts of the apparatus

Part	Dimension mm
Rotor diameter	38,10 ± 0,03
Rotor thickness	5,54 ± 0,03
Die cavity diameter	50,9 ± 0,1
Die cavity depth	10,59 ± 0,03

NOTE Normally, a rotor with these dimensions is called a large rotor.

It is permissible to use a smaller rotor where high viscosity makes this necessary. This small rotor shall have the same dimensions as the large rotor except that the diameter shall be 30,48 mm ± 0,03 mm. Results obtained with the small rotor are not identical with those obtained using the large rotor.

4.2 Dies

The two dies forming the cavity shall be formed from non-deforming unplated hardened steel of minimum Rockwell hardness 60 HRC (see ISO 6508-1). The dimensions of the cavity are given in Figure 1 and shall be measured from the highest surfaces. For good heat transfer, each die should preferably be made from only one piece of steel. The flat surfaces shall have radial V-grooves to prevent slippage. The grooves shall be spaced radially at 20° intervals and shall extend from an outer circle of diameter 47 mm to an inner circle of diameter 7 mm for the upper die and to within 1,5 mm of the hole in the lower die. Each groove shall form a 90° angle in the die surface with the bisector of the angle perpendicular to the surface and shall be 1,0 mm ± 0,1 mm wide at the surface (see Figure 2).

4.3 Rotor

The rotor shall be fabricated from non-deforming unplated hardened steel of minimum Rockwell hardness 60 HRC. The rotor surfaces shall have rectangular-section grooves 0,80 mm ± 0,02 mm wide, of uniform depth 0,30 mm ± 0,05 mm, and spaced 1,60 mm ± 0,04 mm apart (distance between central axes). The flat surfaces of the rotor shall have two sets of such grooves at right angles to each other (see Figure 3). The edge of the rotor shall have vertical grooves of the same dimensions. The large rotor shall have 75 vertical grooves and the small rotor shall have 60. The rotor is fastened at right angles to a shaft having a diameter of 10 mm ± 1 mm and a length such that, in the closed die cavity, the clearance above the rotor does not differ from that below by more than 0,25 mm. The rotor shaft shall bear on the spindle which turns the rotor shaft not on the wall of the die cavity. The clearance at the point where the rotor shaft enters the cavity shall be small enough to prevent the rubber from leaving the cavity. A grommet, O-ring, or other sealing device can be used as a seal at this point.

The eccentricity or runout of the rotor while turning in the viscometer shall not exceed 0,1 mm.

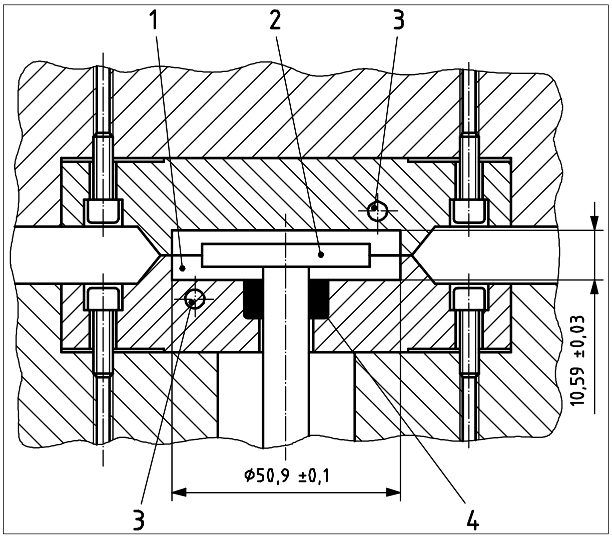
The angular velocity of the rotor shall be 0,209 rad/s ± 0,002 rad/s (2,00 r/min ± 0,02 r/min).

4.4 Heating device

The dies are mounted on, or form part of, platens equipped with a heating device capable of maintaining the temperature of the platens and that of the dies to within ±0,5 °C of the test temperature. After insertion of the test piece, the devices shall be capable of returning the temperature of the dies to within ±0,5 °C of the test temperature within 4 min.

NOTE Older machines might not comply with these requirements and might give less reproducible results.

Dimensions in millimetres



Key

1 die cavity

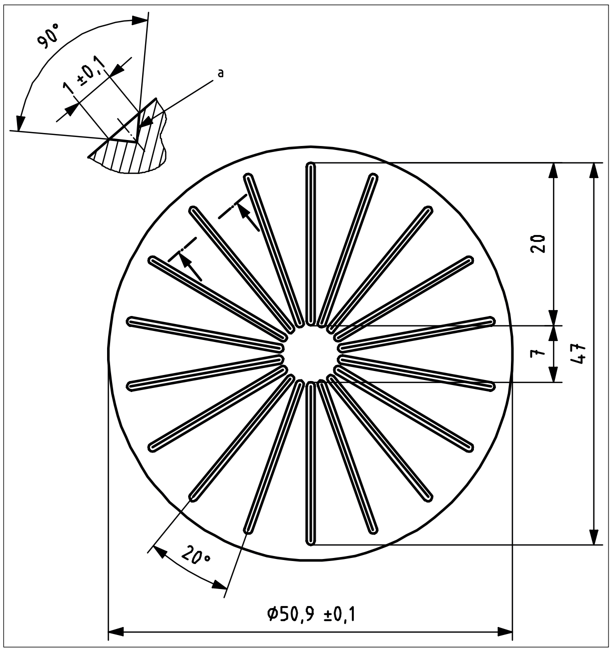
2 rotor

3 temperature sensor

4 sealing device

Figure 1 — Typical shearing-disc viscometer

Dimensions in millimetres

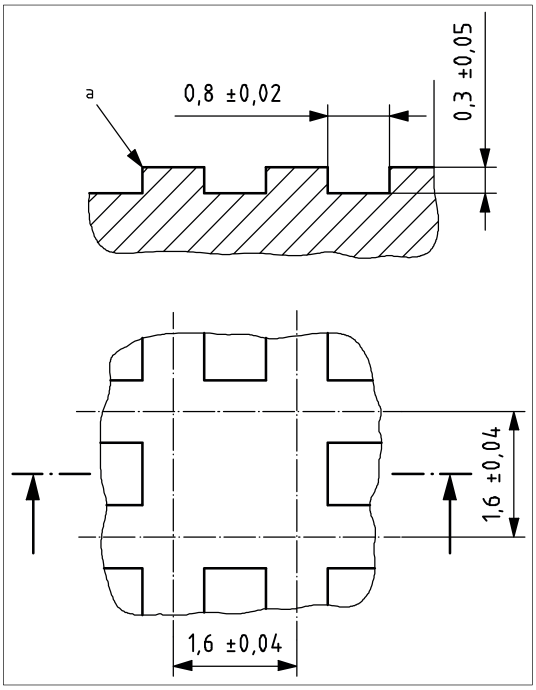


Key

a Section through groove.

Figure 2 — Die with radial V-grooves

Dimensions in millimetres



Key

a *R* at edge of groove ≤0,1 mm.

Figure 3 — Rotor with rectangular-section grooves

4.5 Temperature-measurement system

4.5.1 The test temperature is defined as the steady-state temperature of the closed dies with the rotor in place and the cavity empty. This temperature is measured by two thermocouple measurement probes which can be inserted into the cavity for this purpose as shown in Figure 4. These measurement probes are also used to check the temperature of the test piece as described in 7.2.

4.5.2 In order to control the supply of heat to the dies, a temperature sensor shall be present in each die to measure the die temperature. The sensor shall be located for the best possible heat contact with the dies, i.e. heat gaps and other heat resistance shall be excluded. The axes of the sensors shall be at a distance of 3 mm to 5 mm from the working surface of the dies and 15 mm to 20 mm from the rotational axis of the rotor (see Figure 1).

4.5.3 Both the thermocouple measurement probes and the temperature sensors shall be capable of indicating temperature to an accuracy of ±0,25 °C.

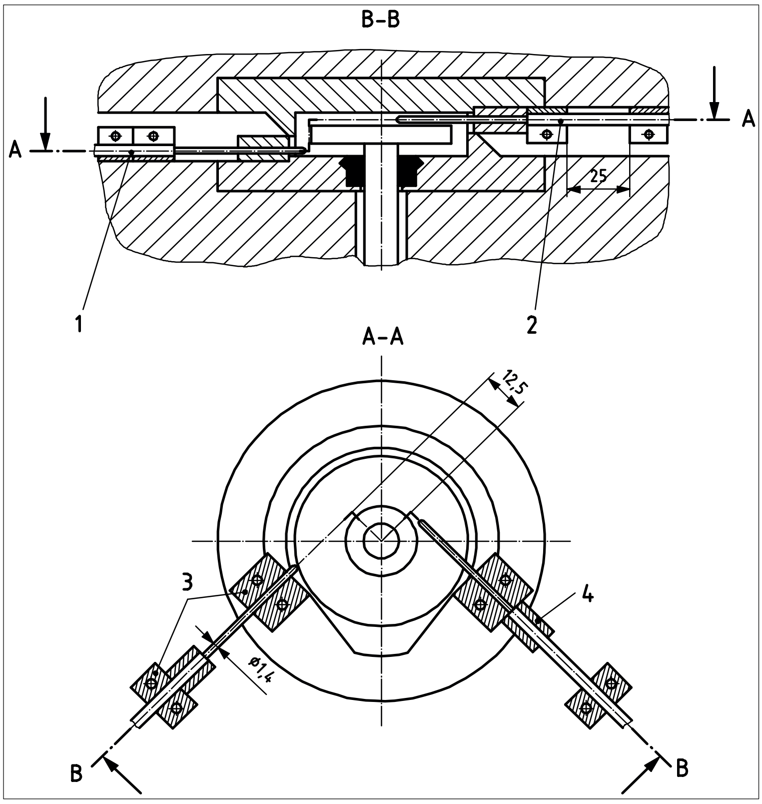
4.6 Die-closure system

The dies could be closed and held closed by hydraulic, pneumatic, or mechanical means. A force of 11,5 kN ± 0,5 kN shall be maintained on the dies during the test.

A greater force can be required to close the dies when rubbers of high viscosity are tested. At least 10 s before starting the viscometer, the force shall be reduced in such cases to 11,5 kN ± 0,5 kN and maintained at this level throughout the test.

For all types of closing device, a piece of soft tissue paper not thicker than 0,04 mm placed between the mating surfaces shall show a continuous pattern of uniform intensity when the dies are closed. A non-uniform pattern indicates incorrect adjustment of the die closure, worn or faulty mating surfaces, or distortion of the dies. Any of these conditions might result in leakage and erroneous results.

Dimensions in millimetres



Key

1 measurement probe, withdrawn

2 measurement probe, inserted

3 stop connected to apparatus

4 stop connected to measurement probe

Figure 4 — Measurement-probe design

4.7 Torque-measurement device and calibration of the device

The torque required to turn the rotor is recorded or indicated on a linear scale graduated in Mooney units. The reading shall be zero when the machine is run empty and 100 ± 0,5 when a torque of 8,30 N·m ± 0,02 N·m is applied to the rotor shaft. Therefore, a torque of 0,083 N·m is equivalent to one Mooney unit. The scale shall be capable of being read to 0,5 Mooney units. Variation from zero shall be less than ±0,5 Mooney units when the machine is running with the rotor in place and the dies closed and empty.

If the viscometer is equipped with a rotor-ejection spring, the zero calibration shall be made with the dies open so that the rotor is not pressing against the upper die.

The viscometer shall be calibrated while the machine is running at the test temperature. A suitable method for the calibration of most machines is as follows.

The scale is calibrated to a reading of 100 by applying certified masses fastened with flexible wire to an appropriate rotor. During calibration, the rotor is turned at 0,209 rad/s and the platens are at the specified test temperature.

NOTE In order to check linearity, intermediate masses can be used to give scale readings of 25 Mooney units, 50 Mooney units, and 75 Mooney units, respectively. In addition, a sample of butyl rubber of certified Mooney viscosity can be used to check whether or not the machine is working correctly. Measurement can be carried out at 100 °C or 125 °C for 8 min.

5 Preparation of test piece

For uncompounded rubbers, the test piece shall be prepared in accordance with ISO 1795 and the material standard relevant to the rubber. For compounded rubbers which are to be tested for referee purposes, the test piece shall be taken from a compound prepared in accordance with ISO 2393 and the material standard relevant to the rubber.

The test piece shall be allowed to rest at standard laboratory temperature (see ISO 23529) for at least 30 min before testing is carried out. Testing shall be commenced not later than 24 h after homogenization.

The Mooney viscosity is affected by the manner in which the rubber is prepared and the conditions of storage. Accordingly, the prescribed procedure in methods for evaluating a particular rubber shall be followed rigorously.

The test piece shall consist of two discs of rubber, of diameter about 50 mm, and of thickness approximately 6 mm sufficient to fill completely the die cavity of the viscometer. The rubber discs shall be as free as possible from air and from pockets that could trap air against the rotor and die surfaces. A hole is pierced or cut through the centre of one disc to permit the insertion of the rotor shaft.

6 Temperature and duration of test

Carry out the test at 100 °C ± 0,5 °C for 4 min unless otherwise specified in the appropriate material standard.

7 Procedure

7.1 Heat the dies and rotor to the test temperature and allow them to reach a steady-state. Open the dies, insert the rotor shaft through the hole in the pierced disc of the test piece, and place the rotor in the viscometer. Place the unpierced disc of the test piece centrally on the rotor and close the dies as quickly as possible.

NOTE A heat-stable film, for example, of polyester of thickness between 0,02 mm and 0,03 mm, can be inserted between the rubber and die surfaces to facilitate removal after test of low-viscosity or sticky materials. The use of such film could affect the test results (see Annex B).

7.2 Note the time at which the dies are closed and allow the rubber to preheat for 1 min. Start the rotor; the running time shall be as indicated in Clause 6. If the viscosity is not recorded continuously, observe the scale during the 30 s interval preceding the specified reading time and report the minimum value to the nearest 0,5 units as the viscosity. For reference purposes, take readings at 5 s intervals from 1 min before to 1 min after the specified time. Draw a smooth curve through the minimum points of the periodic fluctuations or through all the points if there are no fluctuations. Take the viscosity as the point where the curve passes through the time specified. If a recorder is used, take the viscosity from the curve in the same manner as specified for the plotted curve.

To check if the temperature of the test piece is at the test temperature at the test time, two thermocouple measurement probes can be inserted into the test piece as shown in Figure 4. In a preliminary test with the test piece, the rotor is stopped after a running time of 3,5 min and immediately after the resulting standstill, the two measurement probes are inserted and after 4 min, the two mean test piece temperatures are read off. The temperature tolerance shall be between +1,0 °C and −1,0 °C.

The temperature gradients in the test piece and the rate of heat transfer vary between viscometers, particularly if different types of heating are employed. Therefore, the values obtained with different viscometers can be expected to be more comparable after the rubber has attained the test temperature. Usually, this condition is reached within 10 min after the die cavity is closed.

8 Expression of results

Report the results of a typical test in the following format:

50 ML (1 + 4) 100 °C (1)

where

50 M	is the viscosity, in Mooney units;
L	indicates that the large rotor was used (S would indicate use of the small rotor);
1	is the preheating time, in minutes, before starting the rotor;
4	is the running time, in minutes, after starting the rotor at the end of which the final reading was taken;
100 °C	is the temperature of the test.

9 Precision

The precision calculation to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. The results of the three interlaboratory test programmes (ITPs) are given in Annex A.

10 Test report

The test report shall include the following information:

a) sample details:

   1) a full description of the samples and its origin;

   2) the methods of preparation of the test piece from the sample, for example, cut or milled;

b) test methods:

   1) a full reference to the test method used, that is, a reference to this part of ISO 289, i.e. ISO 289-1;

   2) the test procedure used;

   3) the type of test piece used;

c) test details:

   1) the time and temperature of conditioning prior to the test;

   2) the rotor size (large or small);

   3) the temperature of test and the relative humidity if necessary;

   4) the preheat time if other than 1 min;

   5) the running time;

   6) the die-closing force if other than 11,5 kN;

   7) the heat-stable film used including film thickness;

   8) the details of any procedures not specified in this part of ISO 289;

d) test results:

   1) the value of the Mooney viscosity (see Clause 8);

   2) if more than one test piece is tested:

      i) the number of test pieces used;

      ii) the individual test results;

      iii) the mean results (if more than one test piece is tested);

e) the date(s) of test.

Annex A
(informative)

Precision statement

A.1 General

The precision calculations to provide repeatability and reproducibility values were performed in accordance with ISO/TR 9272, the guidance document for ISO/TC 45 test methods. Consult this for precision concepts and nomenclature.

A.2 Programme details

A.2.1 An interlaboratory test programme (ITP) was organized in 1987. Duplicate test pieces of the following raw rubbers were sent to all participating laboratories: butyl (IIR), chloroprene (CR), EPDM, fluorocarbon rubber (FKM), and SBR 1500.

Mooney viscosity tests (single measurements) were made on two separate days (one week apart) in June 1987. A total of 24 laboratories participated.

This ITP corresponds to a type 1 precision evaluation with no preparation or processing steps in the participating laboratories.

A.2.2 Another ITP was carried out in 1988 to determine the influence of mill massing on Mooney viscosity. Three rubbers were used: chloroprene (CR), SBR 1507, and SBR 1712. NIST butyl (IIR) was also included, but it was tested only in the (normal) unmassed form.

Test samples of each rubber were sent to all participating laboratories. The test pieces of massed and unmassed materials were prepared by each laboratory in accordance with ISO 1795.

Mooney viscosity tests (single measurements) were made on two separate days (one week apart) in May 1988. All tests were made at a running time of 4 min and at 100 °C. A total of 15 laboratories participated.

This ITP corresponds to a type 2 precision evaluation.

A.2.3 A third ITP was carried out in 2012. The Mooney viscosity tests [single measurements were made on two separate days (one week apart)] in May 2012.

This ITP corresponds to a type 1 precision evaluation with no preparation or processing steps in the participating laboratories. All tests were made at a running time of 4 min and at 125 °C. A total of 18 laboratories participated.

A.3 Precision results

A.3.1 The precision results of the first ITP are given in Table A.1, those of the second ITP in Table A.2, and those of the third ITP in Table A.3.

A.3.2 The symbols used in Table A.1, Table A.2, and Table A.3 are defined as follows:

s_r within-laboratory standard deviation (in measurements units);

r repeatability (in measurements units);

(r) repeatability (in per cent of mean level);

s_R between-laboratory standard deviation (for total between-laboratory variation, in measurement units);

R reproducibility (in measurements units);

(R) reproducibility (in per cent of mean level).

Table A.1 — Precision of Mooney viscosity determinations (1987)

Rubber material	Average	Within laboratory		Between laboratories	
		r	(*r*)	*R*	(*R*)
SBR 1500[a]	48,0	2,25	4,67	4,43	9,23
CR[a]	48,5	1,82	3,75	4,39	9,06
FKM[b]	56,5	5,00	8,85	8,77	15,50
IIR[a]	69,7	2,15	3,08	3,81	5,47
EPDM[c]	73,1	2,18	2,98	6,61	9,05
Pooled values	**58,9**	**2,93**	**4,98**	**5,85**	**9,93**

[a] At 100 °C, 4 min.

[b] At 100 °C, 10 min.

[c] At 120 °C, 4 min.

Table A.2 — Precision of Mooney viscosity determinations — Influence of mill massing (1988)

Rubber material	Average	Within laboratory		Between laboratories	
		r	(*r*)	*R*	(*R*)
Massed samples					
SBR 1507	33,3	1,66	4,98	2,26	6,80
SBR 1712	51,7	2,37	4,59	5,86	11,30
CR	80,5	2,56	3,19	6,21	7,71
Pooled values	**55,2**	**2,23**	**4,04**	**5,10**	**9,24**
Unmassed samples					
SBR 1507	33,0	1,53	4,63	2,35	7,12
SBR 1712	52,3	1,79	3,42	3,18	6,08
CR	75,3	2,30	3,06	3,72	4,94
Pooled values	**53,5**	**1,90**	**3,55**	**3,13**	**5,86**
NIST IIR (reference rubber)	71,3	1,77	2,49	2,91	4,09

Table A.3 — Precision of Mooney viscosity determinations (2012)

Rubber material	Average	Within laboratory			Between laboratories			Number of laboratories[a]
		s_r	r	(r)	s_R	R	(R)	
material 1 = EPDM 1	22,1	0,28	0,81	3,67	0,43	1,22	5,50	15
material 2 = EPDM 2	33,9	0,27	0,79	2,33	0,37	1,04	3,06	15
material 3 = IIR 1	55,5	0,54	1,55	2,80	0,87	2,43	4,37	15
material 4 = IIR 2	52,1	0,50	1,48	2,84	0,70	1,95	3,74	16
material 5 = EPDM 3	64,5	0,33	0,95	1,48	0,49	1,37	2,12	14
material 6 = EPDM 4	62,8	0,33	0,95	1,51	0,41	1,15	1,83	14
material 7 = EPDM 5	80,2	0,34	0,98	1,22	0,60	1,69	2,11	14
material 8 = EPDM 6	74,8	0,65	1,87	2,50	1,27	3,55	4,75	16

[a] The final number of laboratories in the revised database after deletion of outliers (option 1 of ISO/TR 9272).

Annex B
(informative)

Heat-stable film for Mooney viscosity measurements

It is common practice to insert a heat-stable film between the rubber and the die surfaces to facilitate removal after test of low-viscosity or sticky materials. The use of these films can affect the test result. In this study, Mooney viscosity measurements were carried out on test pieces without a heat-stable film and with three different heat-stable films. The polymers and heat-stable films used are listed in Table B.1. The measurements were carried out on an MV2000E machine.

Table B.1 — Polymers and heat-stable film used

Polymers					
Natural rubber (NR):	SMR CV 60		EPDM:	EDPM material 1	
Butyl rubber (BR):	IRM 241		EPDM:	EDPM material 2	
Heat-stable film					
Polypropylene (PP)		Polyester (PET)		Cellophane	
d = 0,020 mm	Tm = 165 °C	d = 0,025 mm	Tm = 250 °C	d = 0,028	

The Mooney viscosity of the test pieces with or without heat-stable films was measured in duplicate on four different days within one week by the same operator and on the same equipment. The conditions and the original measurement values are given in Table B.2. The measurement values were evaluated with a Q-test for outliers and F-test for the choice of the *t*-test and finally, the *t*-test.

An example of an evaluation is given in Figure B.1 and the overall results are in Table B.3. In general, lower average values were found when a heat-stable film was used compared to the test pieces without film and in some cases, the differences were significant. Also, in three cases, significant differences were found between the measurements with the test pieces with the two different heat-stable films. This investigation clearly shows that the use of a heat-stable film can result in different values which could result in a dispute for borderline values.

Table B.2 — Original measurement values for the different polymers and heat-stable film

		without film			PP film			PET film			Cellophane film		
Day 1		No. 1	No. 2	avg	No. 1	No. 2	avg	No. 1	No. 2	avg	No. 1	No. 2	avg
Butyl rubber	ML (1 + 4) 125 °C	54,1	53,1	53,60	52,2	53,0	52,60	53,3	51,2	52,25	52,4	52,8	52,60
EDPM material 1	ML (1 + 4) 125 °C	34,2	34,2	34,20	34,7	34,2	34,45	33,9	33,6	33,75	34,0	34,1	34,05
EDPM material 2	ML (1 + 4) 125 °C	79,2	78,2	78,70	79,1	79,8	79,45	79,0	78,8	78,90	78,3	78,8	78,55
Natural rubber	ML (1 + 4) 100 °C	70,1	68,4	69,25	69,6	69,4	69,50	69,2	69,1	69,15	68,7	60,8	64,75
Day 2		No. 1	No. 2	avg	No. 1	No. 2	avg	No. 1	No. 2	avg	No. 1	No. 2	avg
Butyl rubber	ML (1 + 4) 125 °C	52,7	52,6	52,65	52,7	52,6	52,65	51,3	51,7	51,50	51,5	51,9	51,70
EDPM material 1	ML (1 + 4) 125 °C	34,1	34,3	34,20	34,1	34,3	34,20	33,8	34,1	33,95	33,9	34,2	34,05
EDPM material 2	ML (1 + 4) 125 °C	79,2	79,3	79,25	79,2	79,3	79,25	78,7	79,2	78,95	79,3	79,0	79,15
Natural rubber	ML (1 + 4) 100 °C	70,4	70,1	70,25	69,0	69,7	69,35	70,2	69,2	69,70	68,9	69,1	69,00
Day 3		No. 1	No. 2	avg	No. 1	No. 2	avg	No. 1	No. 2	avg	No. 1	No. 2	avg
Butyl rubber	ML (1 + 4) 125 °C	52,5	52,5	52,50	52,2	52,5	52,35	51,2	51,6	51,40	51,6	51,5	51,55
EDPM material 1	ML (1 + 4) 125 °C	34,0	34,1	34,05	33,8	34,4	34,10	33,4	33,5	33,45	33,4	33,6	33,50
EDPM material 2	ML (1 + 4) 125 °C	79,2	79,2	79,20	78,5	79,3	78,90	77,2	78,7	77,95	78,2	78,9	78,55
Natural rubber	ML (1 + 4) 100 °C	69,1	68,1	68,60	68,3	68,8	68,55	69,6	68,8	69,20	69,2	68,0	68,60
Day 4		No. 1	No. 2	avg	No. 1	No. 2	avg	No. 1	No. 2	avg	No. 1	No. 2	avg
Butyl rubber	ML (1 + 4) 125 °C	52,4	52,4	52,40	52,3	52,3	52,30	51,4	51,5	51,45	51,7	51,9	51,80
EDPM material 1	ML (1 + 4) 125 °C	34,1	33,9	34,00	33,9	34,2	34,05	33,6	33,7	33,65	33,6	33,8	33,70
EDPM material 2	ML (1 + 4) 125 °C	78,6	79,5	79,05	79,1	79,0	79,05	79,0	78,3	78,65	78,2	78,1	78,15
Natural rubber	ML (1 + 4) 100 °C	69,8	69,0	69,40	70,5	69,2	69,85	69,3	69,0	69,15	69,6	69,0	69,30

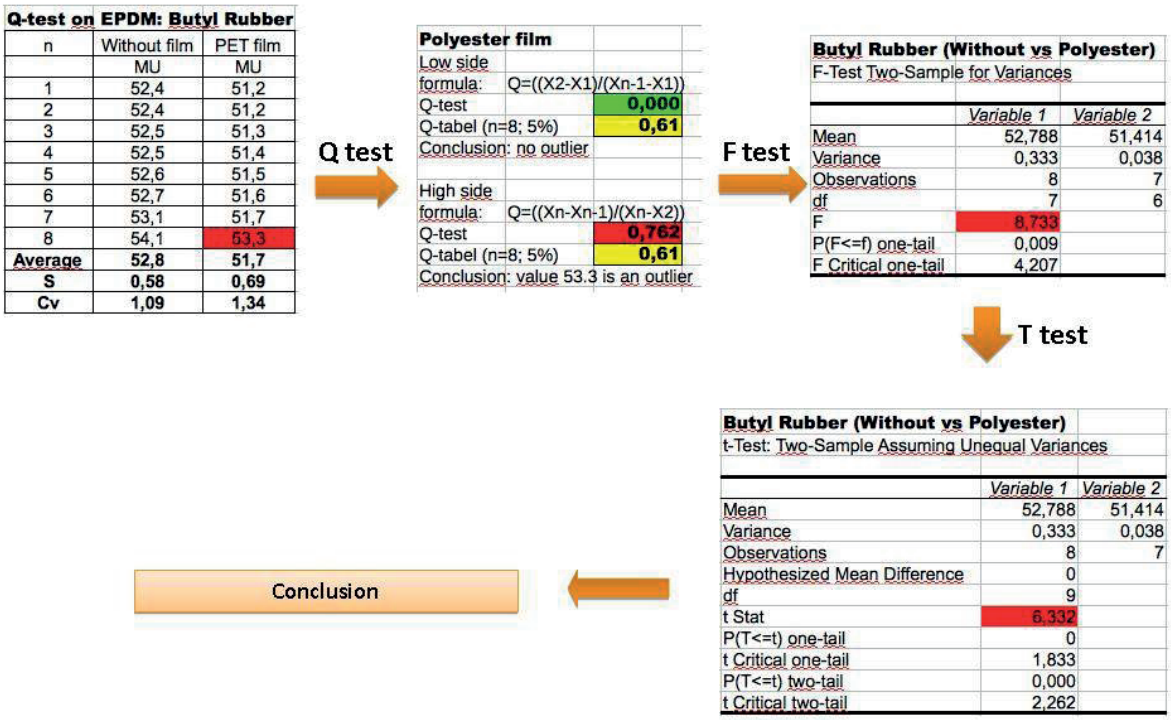


Figure B.1 — Example of data evaluation

Table B.3 — Differences between the average Mooney viscosity values of different test piece without or with heat-stable film

	Natural rubber	EDPM material 1	EPDM material 2	Butyl rubber
Without film vs. polypropylene	0,06[a]	−0,10[a]	−0,05[a]	0,31[a]
Without film vs. polyester	0,08[a]	0,41[b]	0,13[a]	1,37[b]
Without film vs. cellophane	0,36[a]	0,31[b]	0,51[b]	0,88[b]
Polypropylene vs. polyester	0,01[a]	0,51[b]	0,18[a]	1,06[b]
Polypropylene vs. cellophane	0,30[a]	0,41[b]	0,57[b]	0,56[b]
Polyester vs. cellophane	0,29[a]	−0,10[a]	0,39[a]	−0,50[b]

[a] No significant change.

[b] Significant differences.

Annex C
(normative)

Calibration schedule

C.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration was 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.

C.2 Schedule

Verification/calibration of the test apparatus is a mandatory part of this part of ISO 289. However, the frequency of calibration and procedures used are, unless otherwise stated, at the discretion of the individual laboratory using ISO 18899 for guidance.

The calibration schedule given in Table C.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 the following:

- N initial verification only;
- S standard interval as given in ISO 18899;
- U in use.

Table C.1 — Calibration frequency schedule

Parameter	Requirement(s)	Subclause in ISO 18899:2013	Verification frequency schedule
Surface hardness of the dies	≥60 HRC	15.5	N
Dimensions of the dies	See 4.2	15.2	N
Die grooves	See 4.2	15.2	N
Surface hardness rotor	≥60 HRC	15.5	N
Dimensions rotor	See 4.3	15.2	N
Rotor grooves	See 4.3	15.2	N
Angular velocity	0,209 rad/s ± 0,002 rad/s	23.2	S

Table C.1 *(continued)*

Parameter	Requirement(s)	Subclause in ISO 18899:2013	Verification frequency schedule
Temperature accuracy	±0,25 °C	18	S
Temperature stability at steady-state	±0,5 °C	18	S
Die closure	11,5 kN ± 0,5 kN	21.3	S
Torque	See 4.7	21.4	S

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

— instruments for determining dimensions of the dies;

— load cell for die closure check;

— thermometer for monitoring the condition and test temperatures.

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