Ovulkat gummi – Bestämning med hjälp av rotationsviskometer –
Del 1: Bestämning av Mooney-viskositet
(ISO 289-1:2005, IDT)

Rubber, unvulcanized – Determinations using a shearing-disc viscometer –
Part 1: Determination of Mooney viscosity
(ISO 289-1:2005, IDT)

Den internationella standarden ISO 289-1:2005 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av ISO 289-1:2005.

Denna standard ersätter SS-ISO 289-1, utgåva 1.

The International Standard ISO 289-1:2005 has the status of a Swedish Standard. This document contains the official English version of ISO 289-1:2005.

This standard supersedes the Swedish Standard SS-ISO 289-1, edition 1.

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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 289-1 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 289-1:1994), which has been revised to update the normative references.

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 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 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 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 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 (scales A, B, C, D, E, F, G, H, K, N, T)*

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

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

The essential parts of the apparatus (see Figure 1) are:

a)   two dies to form a cyclindrical 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;

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	$38,10 \pm 0,03$
Rotor thickness	$5,54 \pm 0,03$
Die cavity diameter	$50,9 \pm 0,1$
Die cavity depth	$10,59 \pm 0,03$

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.

4.1   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).

4.2   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 rubber leaving the cavity. A grommet, O-ring or other sealing device may 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).

4.3 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 may not comply with these requirements and may give less reproducible results.

Dimensions in millimetres
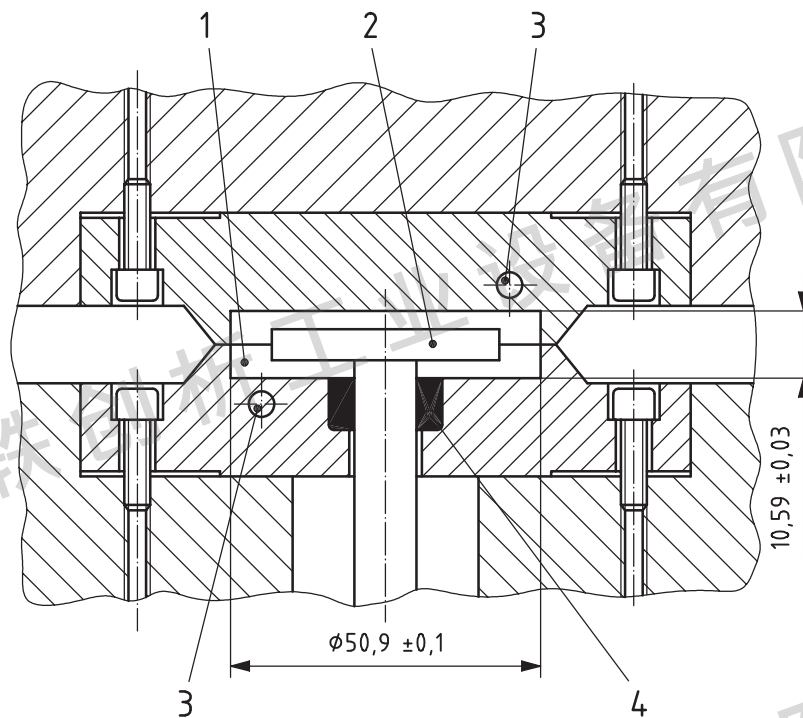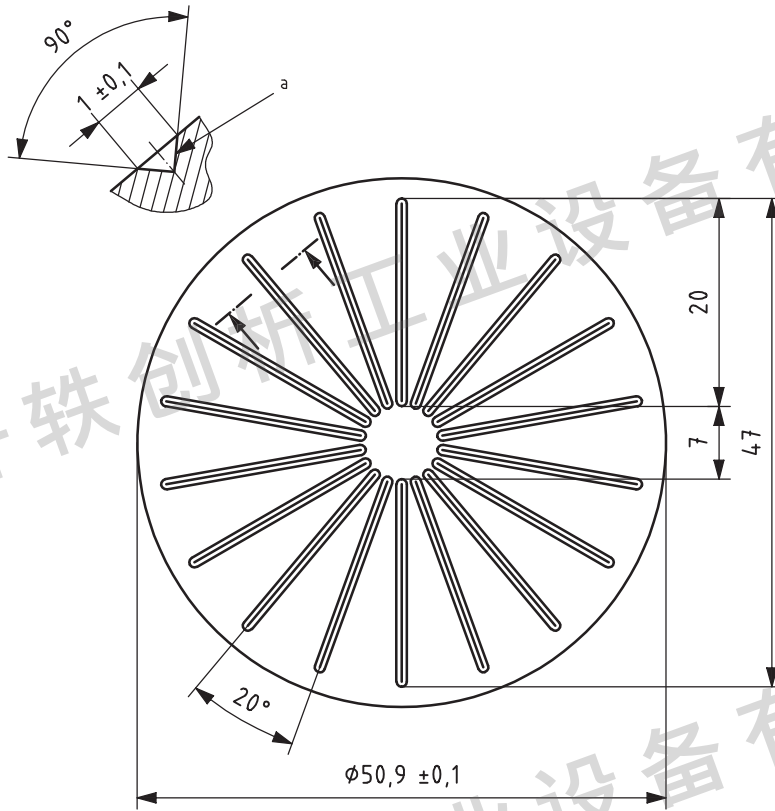

Key
1   die cavity
2   rotor
3   temperature sensor
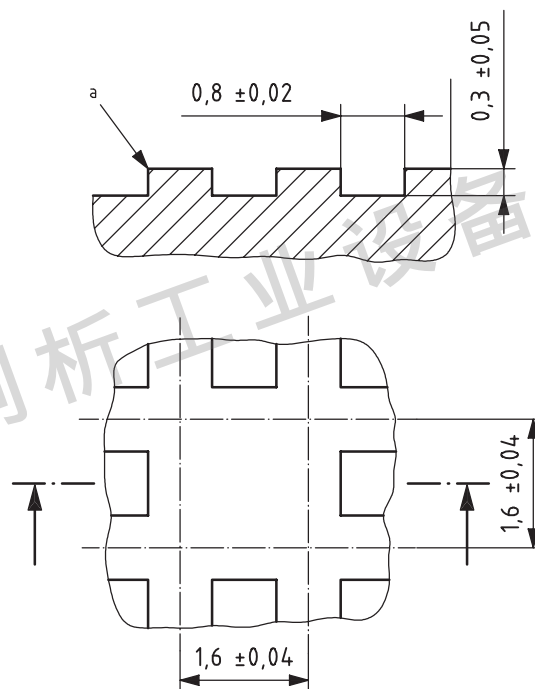4   sealing device

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 $\leqslant$ 0,1 mm.

Figure 3 — Rotor with rectangular-section grooves

4.4   Temperature-measurement system

4.4.1   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.

4.4.2   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).

4.4.3   Both the thermocouple measurement probes and the temperature sensors shall be capable of indicating temperature to an accuracy of $\pm$ 0,25 °C.

4.5   Die-closure system

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

A greater force may 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 $\pm$ 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 may result in leakage and erroneous results.

Dimensions in millimetres

B – B

A – A
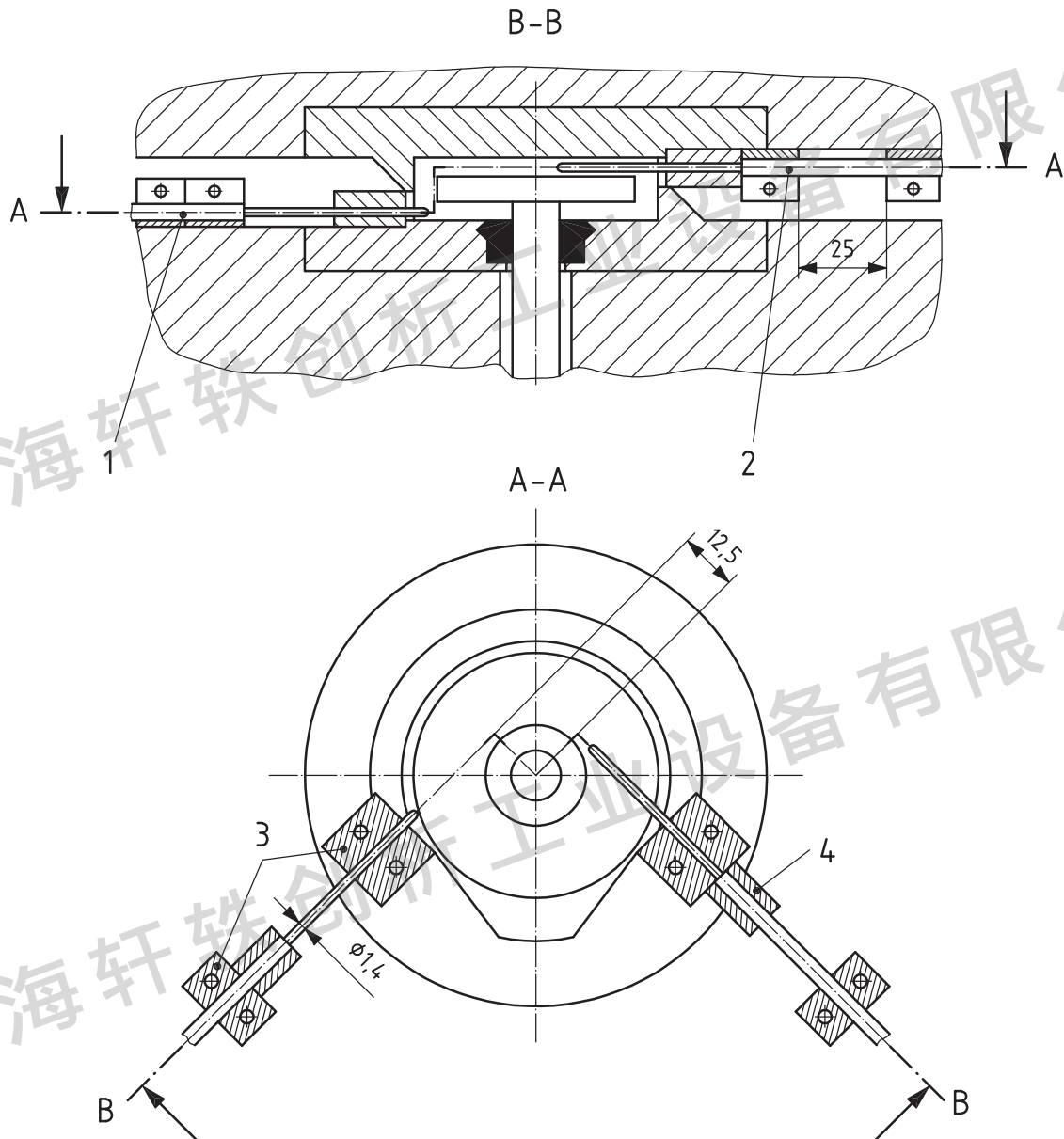
Key

1   measurement probe, withdrawn
2   measurement probe, inserted
3   stop connected to apparatus
4   stop connected to measurement probe

Figure 4 — Measurement-probe design

4.6 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 $100 \pm 0{,}5$ when a torque of $8{,}30$ N·m $\pm 0{,}02$ N·m is applied to the rotor shaft. Therefore a torque of $0{,}083$ N·m is equivalent to 1 Mooney unit. The scale shall be capable of being read to $0{,}5$ Mooney units. Variation from zero shall be less than $\pm 0{,}5$ 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 of diameter $0{,}45$ mm 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 may be used to give scale readings of 25, 50 and 75 Mooney units, respectively. In addition, a sample of butyl rubber of certified Mooney viscosity can be used to check whether the machine is working correctly. Measurement may 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 may 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 $\pm 0{,}5$ °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 approximately $0{,}03$ mm may be inserted between the rubber and die surfaces to facilitate removal after test of low-viscosity or sticky materials. The use of such film may affect the test results.

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 referee 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 may 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 0,0 °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 may 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

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

9.1   General

The precision calculations to express repeatability and reproducibility were performed in accordance with ISO/TR 9272. Consult this for precision concepts and nomenclature. Annex A gives guidance on the use of repeatability and reproducibility.

9.2   Programme details

9.2.1   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.

9.2.2   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.

9.3   Precision results

9.3.1   The precision results of the first ITP are given in Table 2 and those of the second ITP in Table 3.

9.3.2   The symbols used in Tables 2 and 3 are defined as follows:

r = repeatability, in Mooney units

(r) = repeatability, in percent (relative)

R = reproducibility, in Mooney units

(R) = reproducibility, in percent (relative)

Table 2 — Precision of Mooney viscosity determinations

Rubber material	Average	Within lab		Between labs	
		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 3 — Precision of Mooney viscosity determinations — Influence of mill massing

Rubber material	Average	Within lab		Between labs	
		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

10 Test report

The test report shall include the following information:

a) a full description and identification of the sample tested, including

   1) its origin,

   2) details of compounded rubbers, if applicable;

b) details of the preparation of the test pieces;

c) a reference to this part of ISO 289;

d) a description of the apparatus used, including the model used, the manufacturer of the apparatus and the rotor size (large or small);

e) details of the conditions of test, including

   1) the test temperature,

   2) the preheat time, if other than 1 min,

   3) the running time,

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

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

g) any operation not included in this part of ISO 289 or regarded as optional;

h) the date of the test.

Annex A
(informative)

Guidance for using precision results

A.1   The general procedure for using precision results is as follows, with the symbol $|x_1 - x_2|$ designating a positive difference in any two measurement values (i.e. without regard to sign).

A.2   Enter the appropriate precision table (for whatever test parameter is being considered) at an average value (of the measured parameter) nearest to the "test" data average under consideration. This line will give the applicable r, (r), R or (R) for use in the decision process.

A.3   With these r and (r) values, the following general repeatability statements may be used to make decisions.

A.3.1   For an absolute difference: The difference $|x_1 - x_2|$ between two test (value) averages, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability r on average not more than once in twenty cases.

A.3.2   For a percentage difference between two test (value) averages: The percentage difference

$$\frac{|x_1 - x_2|}{0,5(x_1 + x_2)} \times 100$$

between two test values, found on nominally identical material samples under normal and correct operation of the test procedure, will exceed the tabulated repeatability (r) on average not more than once in twenty cases.

A.4   With these R and (R) values, the following general reproducibility statements may be used to make decisions.

A.4.1   For an absolute difference: The absolute difference $|x_1 - x_2|$ between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility R not more than once in twenty cases.

A.4.2   For a percentage difference between two test (value) averages: The percentage difference

$$\frac{|x_1 - x_2|}{0,5(x_1 + x_2)} \times 100$$

between two independently measured test (value) averages, found in two laboratories using normal and correct test procedures on nominally identical material samples, will exceed the tabulated reproducibility (R) not more than once in twenty cases.