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ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 289

DETERMINATION OF VISCOSITY OF NATURAL AND SYNTHETIC RUBBERS BY THE SHEARING DISK VISCOMETER

1st EDITION
January 1963

For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 289, *Determination of Viscosity of Natural and Synthetic Rubbers by the Shearing Disk Viscometer*, was drawn up by Technical Committee ISO/TC 45, *Rubber*, the Secretariat of which is held by the British Standards Institution (B.S.I.).

Work on this question by the Technical Committee began in 1959 and led, in 1960, to the adoption of a Draft ISO Recommendation.

In May 1960, this Draft ISO Recommendation (No. 378) was circulated to all the ISO Member Bodies for enquiry. It was approved by the following Member Bodies:

Australia	Germany	Portugal
Austria	Hungary	Republic of South Africa
Brazil	India	Spain
Canada	Israel	Sweden
Chile	Italy	Switzerland
Colombia	Japan	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
Denmark	New Zealand	U.S.S.R.
France	Poland	Yugoslavia

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in January 1963, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 289 January 1963

DETERMINATION OF VISCOSITY OF NATURAL AND SYNTHETIC RUBBERS BY THE SHEARING DISK VISCOMETER

1. SCOPE

The method given in the present ISO Recommendation describes the procedure for determining the viscosity, expressed in Mooney viscosity units, of uncompounded or compounded unvulcanized natural or synthetic rubbers or reclaimed rubbers, by means of the shearing disk viscometer.

Caution should be exercised in interpreting viscosity values obtained by this method as a measure of molecular mass of the higher molecular mass rubbers. For example, as the molecular mass increases, the viscosity values for butyl rubbers reach an upper limit of about 80 at 100 °C, using the specified rotor at a speed of 2 revolutions/minute, and may then decrease to considerably lower values. For these higher molecular mass rubbers, better correlation between viscosity and molecular mass is obtained if the rotor speed is reduced or the test temperature increased.

2. PRINCIPLE OF TEST

The test involves the determination of the torque which should be applied under specified conditions in order to rotate a metal disk in a cylindrical chamber filled with rubber. A number proportional to this torque is taken as an index of the viscosity of the rubber.

3. PREPARATION OF TEST PIECES

Two disks of rubber, about 45 mm in diameter and of sufficient thickness to fill completely the die cavity of the viscometer, are prepared. The test piece should be cut with a die slightly smaller than the die cavity, but of a thickness to give an excess volume, i.e. about 25 cm^3. The rubber disks should 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 disk to permit the insertion of the rotor stem.

NOTE. – The viscosity is affected by the manner in which the rubber is prepared and the conditions of storage prior to test. Accordingly, the prescribed procedure in methods for evaluating the particular rubber should be followed rigorously.

4. APPARATUS

The essential parts of the apparatus are:

a rotor,
a hollow cylindrical die,
a means for rotating the rotor,
a means for indicating the torque required to rotate the rotor, and
controls for maintaining the die at a constant temperature.

The rotor and die cavity have the dimensions shown in *either* column (*a*) *or* column (*b*) of the following table, but in order to ensure the greatest accuracy of results, the metric dimensions should in future be as shown in column (*a*).

TABLE. – **Dimensions of essential parts of the apparatus**

	(a)		(b)
	millimetres	inches	millimetres
Rotor diameter	38.10 ±0.03	1.500 ±0.001	38.10 ±0.05
Rotor thickness	5.54 ±0.03	0.218 ±0.001	5.50 ±0.05
Die cavity diameter	50.93 ±0.13	2.005 ±0.005	50.80 ±0.05
Die cavity depth	10.59 ±0.03	0.417 ±0.001	10.60 ±0.05

It is permissible to use a smaller rotor where high viscosity makes this desirable. This small rotor should have the same dimensions as the large rotor except that the diameter is 30.48 ±0.03 mm (1.200 ±0.001 in).

Results obtained with the small rotor are not identical with those obtained with the large rotor. However, for the purposes of comparing rubbers or compounds, they lead to the same conclusions.

The die cavity should preferably be formed from only two pieces of unplated hardened steel for improved heat transfer, and have radial V-grooves on the flat surfaces to retard slipping. The grooves are spaced at 20° intervals, and extend from at least the 7 mm circle to the 47 mm diameter circle; each groove forms a 90° angle in the die surface, with the bisector of the angle perpendicular to the surface, and is 1.00 ±0.25 mm wide at the surface.

The die cavity may alternatively be formed from four pieces of steel with rectangular-section grooves on the cavity surfaces to retard slipping. The grooves are 0.80 ±0.02 mm wide, of uniform depth between 0.25 and 0.38 mm, and spaced on 1.60 ± 0.04 mm centres. The flat surfaces of the cavity have two sets of these grooves at right angles to each other.

The rotor surfaces are grooved as described for the die cavity formed from four pieces of steel. The hardened rotor is fastened to a shaft not exceeding 11 mm in diameter and positioned securely, so that in the closed die cavity the clearance above the rotor does not differ from the clearance below the rotor by more than 0.25 mm. The eccentricity or runout of the rotor while turning in the viscometer should not exceed 0.013 mm. The rotor shaft bears on the spindle which turns the rotor, and not on the wall of the die cavity. The clearance at the point where the rotor enters the cavity should be small enough to prevent rubber leaving the cavity. A grommet may be used as a seal at this point.

The dies forming the die cavity are mounted on/or form part of platens equipped with a heating device capable of maintaining the die cavity within ±0.5 °C of the test temperature (see section 6).

NOTE. – The test temperature is defined as the steady-state temperature of the closed cavity, with rotor in place but without rubber. Since a temperature difference may exist between the platens and the die cavity, it may be necessary to adjust the platen temperatures to obtain the correct cavity temperature. In making such adjustments, it is important that the temperatures of the two platens be within 0.5 °C of each other. The cavity temperature is determined within 0.3 °C and may be measured with calibrated thermocouples or thermistors, using wires about 0.3 mm in diameter to minimize thermal conduction to the exterior.

The die cavity may be closed by hydraulic, pneumatic or mechanical means. If fluid pressure is used, a force of 1 400 kgf may be required for the initial closure, when rubbers of very high viscosity are tested. At least 10 seconds before starting the viscometer, the force is reduced to 350 ± 20 kgf and maintained at this value during the test. If mechanical closure is used, the platens are adjusted, preferably by means of a gauge block, so that the total deformation of the parts is between 0.10 and 0.15 mm when the die cavity is closed at the test temperature.

For all types of closing devices, a piece of thin soft tissue paper not thicker than 0.04 mm placed between the meeting surfaces should show a continuous pattern of uniform intensity, when the die cavity is closed. A non-uniform pattern indicates improper adjustment of die closure, worn or faulty meeting surfaces or distortion of die cavity parts; any of these conditions results in excessive leakage and erroneous results.

The torque required to turn the rotor is recorded or indicated on a linear scale graduated in Mooney viscosity units so that a torque of 84.6 $\pm$0.2 kgf·cm or 73.5 $\pm$0.2 lbf·in on the rotor shaft equals 100 on the scale. The scale is capable of being read easily to 0.5 unit and is calibrated by means of weights fastened to a special rotor with flexible wire not over 0.5 mm in diameter, passing over pulleys free of friction. During the calibration, the rotor is turned at 2 revolutions/minute and the platens are at the temperature required to produce the test temperature in the cavity. The reading should be zero with the torque removed and 100 $\pm$0.5 when a torque of 84.6 kgf·cm is applied.

After calibration, the scale reading is adjusted to zero with the test rotor turning in the empty, closed die cavity at 2 revolutions/minute. The fluctuations in readings during a period of a half minute or more should be less than 0.5 unit.

5. PROCEDURE

The die cavity and rotor are heated to the test temperature and allowed to reach a steady state. The die cavity is opened, the rotor stem inserted through the hole in the test disk, the rotor is placed in the viscometer, the solid test disk is placed centrally on the rotor, and the die cavity is closed as quickly as possible.

A film of transparent cellulosic material approximately 0.03 mm in thickness may be inserted between the rubber and metal surfaces to facilitate removal after test when testing low-viscosity or sticky materials.

The time of closing the die cavity is noted and the rubber is allowed to heat for the specified time; a minimum of one minute is recommended.

NOTE. – The temperature gradients and rate of heat transfer vary among 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 about 10 minutes after the die cavity is closed. For most rubbers, the reading is not altered appreciably by permitting the rubber to heat in the viscometer for different times, provided the viscosity is read at a specified time. The running time should never be less than 2 minutes.

The rotor is turned at 2.00 $\pm$ 0.02 revolutions/minute, unless otherwise specified, after the specified time of preheat. If the viscosity is not recorded continuously, the indicator and scale are observed continuously during the 30-second interval preceding the specified time of reading, and the minimum value to the nearest 0.5 unit during this interval is taken as the viscosity. For reference purposes, readings are taken at 5-second intervals from 1 minute before to 1 minute after the time specified. A smooth curve is drawn through the minimum points of the periodic fluctuations or through all points if there are no fluctuations. The viscosity is taken as the point where the curve intersects the time specified. If a recorder is used, the viscosity is taken from the recorded curve in the same manner as specified for the plotted curve.

6. TEMPERATURE OF TEST

The test is normally made at a temperature of 100 °C.

7. TEST REPORT

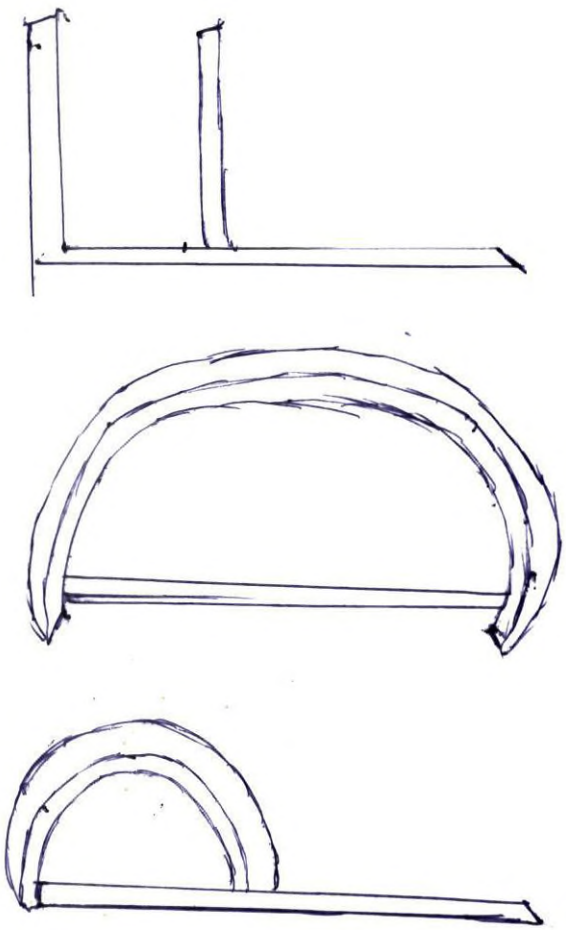
The report should include the following:

(1) viscosity in Mooney units,
(2) temperature of test,
(3) time interval of preheat,
(4) time of reading after preheat,
(5) rotor speed if other than 2 revolutions/minute, and
(6) method of sample preparation.

Note on reproducibility of results.

The standard deviation for measurements on the same instrument of a uniform sample of rubber is about 0.2 unit. Variation in sample preparation results in standard deviations of about 1 unit; recorded figures from laboratories working on natural rubber range from 0.65 to 2 units, and those from laboratories concerned with synthetic rubber production from 0.2 to 0.75 unit. The standard deviation is substantially independent of the viscosity, at least within the range of 40 to 90 units.

Variability among laboratories may cause even larger variations in results. Part of the inter-laboratory variability is due to sample preparation, and part to errors in calibrating or adjusting the viscometer.



INTERNATIONAL STANDARD

ISO 498

Second edition
1992-03-15

Natural rubber latex concentrate — Preparation of dry films

Latex concentré de caoutchouc naturel — Préparation de pellicules sèches

Reference number
ISO 498:1992(E)

S1

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.

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.

International Standard ISO 498 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Sub-Committee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 498:1974), of which it constitutes a minor technical revision.

International Organization for Standardization
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Natural rubber latex concentrate — Preparation of dry films

1 Scope

This International Standard specifies a method for preparing dry, homogeneous films, substantially free of air bubbles, from natural rubber latex concentrate.

The procedure is not necessarily suitable for latices from natural sources other than *Hevea brasiliensis* or for compounded latex, vulcanized latex or artificial dispersions of rubber or synthetic rubber latices.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 123:1985, *Rubber latex — Sampling.*

ISO 124:1992, *Rubber latices — Determination of total solids content.*

3 Apparatus

3.1 Suitable mould, in which the film can be cast, prepared by cementing strips of glass or a rigid plastic material 6 mm wide and 1,5 mm thick on a flat piece of glass plate. The cavity so formed shall be of an adequate size to provide suitable specimens for testing, e.g. with sides of 100 mm to 150 mm.

NOTE 1 As a result of the effect of surface tension, areas of the film around the edges may be thicker than at the centre.

Adhesives suitable for affixing the strips to the glass are epoxide resin adhesives, and poly(vinyl acetate) dissolved in methyl ethyl ketone. Such a mould will give dry films about 1 mm thick when filled with latex of 62 % (m/m) total solids content.

3.2 Square-mesh gauze, of polyamide or stainless steel, with an average aperture width of 180 µm ± 10 µm, for straining the latex.

3.3 Straightedge, wooden, plastic or stainless steel, with which to scrape the surface of the latex in the mould free of air bubbles.

3.4 Cabinet or **covered space**, clean, dry and dust-free, with a level surface on which to place the mould.

3.5 Oven, capable of maintaining a temperature of 35 °C ± 2 °C.

3.6 Cellulosic-film sheets, thin, clear and transparent, to cover and protect the dry film.

3.7 Desiccator or **airtight container**, for storing the dry film.

3.8 Beaker, of suitable capacity, e.g. 50 cm^3.

4 Sampling

Carry out the sampling in accordance with one of the methods specified in ISO 123.

5 Procedure

Determine the total solids content of the latex in accordance with ISO 124. If the total solids content is less than or equal to 62 % (m/m), prepare the film without dilution of the latex. If the total solids content is greater than 62 % (m/m), add distilled water to bring it to 61,5 % (m/m) solids content.

Mix the latex sample gently to ensure homogeneity and allow to stand for 5 min. Strain 35 cm^3 to

40 cm^3 carefully through the gauze (3.2) into the beaker (3.8). Allow to stand for 5 min in the beaker. During this period, keep the beaker covered in order to minimize surface drying. Remove any bubbles from the surface of the latex in the beaker with a piece of filter paper.

Place the mould in the position in which the film will be left to dry (see 3.4). Then pour the latex into the mould in a continuous stream while moving the beaker to and fro over the surface and close to the plate to avoid the formation of air bubbles. Pour a slight excess of latex over that required to fill the mould completely. Allow the latex in the mould to stand for 1 min, then scrape off the excess with the clean straightedge (3.3) by moving it evenly across the mould at a speed of up to 25 mm/s once only.

Allow the cast film to dry in a normal, dust-free atmosphere for not less than 16 h (i.e. overnight). After drying at room temperature, continue to dry the film in the oven (3.5) at a temperature of 35 °C ± 2 °C. When sufficiently dry to handle, strip the film from the mould, taking care to handle the surface of the film as little as possible. Turn the film over and place it flat on a piece of thin, clear, transparent cellulosic sheet (3.6). Allow to stand for at least another 24 h at a temperature of 35 °C ± 2 °C and, when dry, cover the remaining side of the film with a similar cellulosic sheet.

In some cases, the dryness of the film can be judged by its clarity. Clarity of the film generally increases as it becomes dry. If it is not possible to judge the dryness visually, dry the film to constant mass at a temperature of 35 °C ± 2 °C in a dry atmosphere.

Store the dry film in the desiccator or airtight container (3.7) to prevent absorption of moisture, and keep in a cool place in the dark until required.