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METHODS OF TEST FOR RAW ACRYLONITRILE BUTADIENE RUBBER

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METHODS OF TEST FOR RAW ACRYLONITRILE BUTADIENE RUBBER

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Indian Standard

METHODS OF TEST FOR RAW ACRYLONITRILE BUTADIENE RUBBER

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 24 November 1977, after the draft finalized by the Rubber Sectional Committee had been approved by the Chemical Division Council.

0.2 In the preparation of this standard, assistance has been derived from the following publications:

ISO 247-1976 Rubber — Determination of ash. International Organization for Standardization.

ISO 289-1963 Determination of viscosity of natural and synthetic rubbers by shearing disk viscometer. International Organization for Standardization.

ISO 1407-1976 Rubber — Determination of solvent extract. International Organization for Standardization.

BS 1673: Part 7 : 1969 Methods of testing raw rubber and unvulcanized compounded rubbers, Part 7 : Chemical analysis of acrylonitrile butadiene rubbers (NBR). British Standards Institution.

0.3 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard prescribes the methods of test for raw emulsion acrylonitrile butadiene rubbers for volatile matter (NBR : 1), solvent extract (NBR : 2), organic acid content (NBR : 3), soap content (NBR : 4), total ash content (NBR : 5), combined acrylonitrile content (NBR : 6), solubility (NBR : 7), and Mooney viscosity (NBR : 8).

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

NOTE — 'Pure Chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

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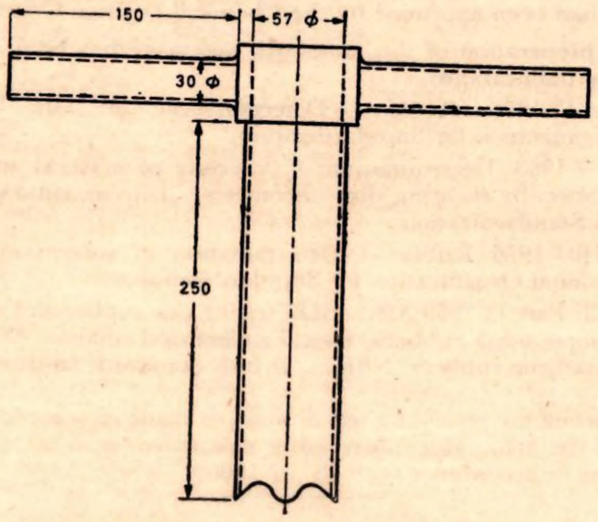
*Rules for rounding off numerical values (*revised*).

†Specification for water for general laboratory use (*second revision*).

3. METHOD OF TAKING OUT TEST PORTION FROM SAMPLE BALES

3.1 Take out a piece or pieces of rubber from the selected bale by cuts through the entire bale perpendicular to the surface of largest area of the bale. The total mass of the material shall be between 600 g and 1 500 g depending on the tests to be done. Using a tool similar to that shown in Fig. 1, obtain plugs of rubber by cutting through the bale. Do not use any lubricant with the tool to obtain piece or pieces of the material. If the material is not tested immediately, place it in a narrow airtight container of not more than twice the volume of the material or keep it wrapped in two layers of aluminium foil until required.



All dimensions in millimetres.

FIG. 1 SAMPLING TOOL

DETERMINATION OF VOLATILE MATTER

(NBR : 1)

0. GENERAL

0.1 Details of three methods, namely, Method A, Method B and Method C, are given. In Method A, weighed test portion is passed through a laboratory mill having heated rolls till a constant mass is obtained. In Method B, the test portion is heated above phosphorus pentoxide for 2 hours under vacuum and loss in mass determined. In Method C, a weighed sample of polymer is sheeted out on a laboratory mill, then placed in an oven and dried to constant mass. For polymers which are too tacky to be handled satisfactorily on a heated mill, Method C is most appropriate.

1. METHOD A

1.1 Apparatus

1.1.1 *Laboratory Mill*

1.2 Procedure— Weigh a test portion of about 500 g of the sample to the nearest 0·1 g. Pass the weighed material through a laboratory mill, with the rolls of the mill maintained at 100 to 105°C, and the distance between the rolls set at 0·50 ± 0·05 mm. Repeatedly pass the rubber between the rolls for 4 minutes without allowing it to form a band and then cool and reweigh to the nearest 0·1 g. Pass the rubber between the rolls for further 2 minutes and again weigh. If the masses at the end of 4- and 6-minute periods are not within 0·1 g, continue passing for further periods of 2 minutes until constant mass is obtained.

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1.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass in g of the material before drying, and
M_2 = mass in g of the material after drying.

2. METHOD B

2.1 Apparatus

2.1.1 *Laboratory Mill*

2.1.2 *Vacuum Oven*

2.2 Procedure — Weigh a test portion of about 100 g to the nearest 0·1 g and pass it through a mill with a nip of 0·6 mm (friction ratio 1 : 1·11) having a roll temperature of 20°C. The sheet is cut into small cubes, which are placed above phosphorus pentoxide for 2 hours at 60°C and a pressure of 2·6 kPa (approximately 20 mmHg). Cool and weigh to the nearest 0·01 g.

2.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass in g of the material before drying, and
M_2 = mass in g of the material after drying.

3. METHOD C

3.1 Apparatus

3.1.1 *Laboratory Mill*

3.1.2 *Forced-Circulation Oven*

3.2 Procedure — Roll a sample of material through a laboratory mill with the distance between the rolls set at 0·25 ± 0·05 mm. The temperature of the rolls should not exceed 30°C. Weigh a test portion of about 250 g of the sample to the nearest 0·1 g. Place the sheets in a forced-circulation oven set at 100 ± 5°C so that both surfaces of the sheet are exposed to the draft. Allow the sample to remain in the oven until the mass is constant to within 0·1 g. Usually 1 hour is sufficient for polymers containing not more than 1·0 percent moisture.

3.3 Calculation

$$\text{Volatile matter, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass in g of the material before drying, and
M_2 = mass in g of the material after drying.

DETERMINATION OF SOLVENT EXTRACT

(NBR : 2)

0. GENERAL

0.1 This method is intended to determine the amount of various organic materials used or formed in the production process of the rubber. By this method the residual amounts of organic acid and soap as well as antioxidant or stabilizer added are measured.

1. OUTLINE OF THE METHOD

1.1 A weighed test portion of the rubber is extracted with *iso*propyl alcohol in a suitable extraction apparatus. The solvent is distilled off and the residue is dried and weighed.

2. APPARATUS

2.1 Extraction Apparatus — The extraction apparatus is of the reflux type with the condenser placed immediately above the cup which holds the sample. The cup is situated in the vapour of the boiling solvent and is emptied by a siphon. The apparatus is of glass except in patterns where an extraction cup is suspended from the end of condenser and, in that case, platinum wire is used for suspension. The apparatus fits together without the use of cork, rubber or metal and in such a manner that loss of vapour during extraction does not exceed 20 percent of the extracting liquid.

2.1.1 Suitable types of apparatus are shown in Fig. 1.

2.2 Filter Paper or Cloth — Acid washed hardened filter paper or nylon cloth of open weave made from continuous filaments.

3. REAGENT

3.1 *Iso*propyl Alcohol — *See* IS : 2631-1976*.

4. PROCEDURE

4.1 Heat the rolls of the laboratory mill to 95 ± 5°C and set the nip at 0·25 ± 0·05 mm. Pass a sample portion of rubber between the rolls at least six times. Cut sufficient rubber from the sheet to give a strip weighing 3 to 5 g. Weigh the test portion to the nearest 0·01 g and roll in filter paper to form a loose cylinder from which the rubber cannot fall and such that the surfaces of the rubber are not in contact.

4.2 Place the roll in the extraction cup of the extraction apparatus. Assemble the extraction apparatus and pour *iso*propyl alcohol into the extraction flask, a quantity sufficient to fill the extraction cup two or three times. Extract the test portion with hot solvent for a continuous period of 16 ± 0·5 hours at a rate of 10 to 20 changes of solvent per hour.

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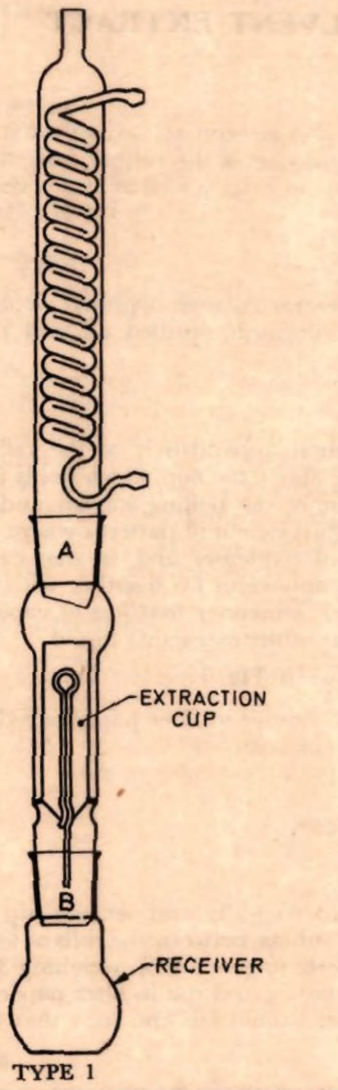
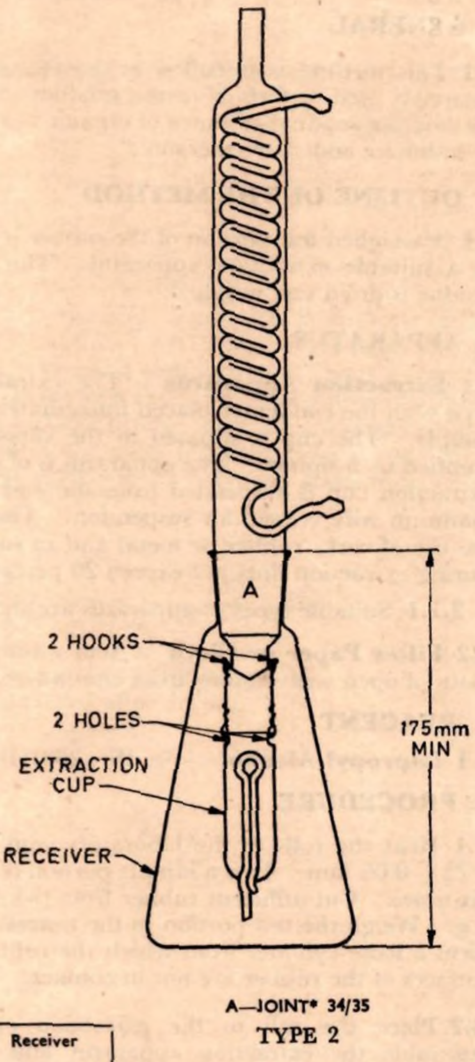
*Specification for *iso*propyl alcohol (*first revision*).



TYPE 1



TYPE 2

Extraction Cap	Joints*		Receiver
	A	B	
ml			ml
20 — 30	34/35	34/35	150
50 — 60	45/50	34/35	250

FIG. 1 EXTRACTION APPARATUS

4.3 Distil the solvent from the extract in a weighed vessel preferably in an extraction flask, dry the extract at 100±2°C, cool and weigh to the nearest 0·001 g.

5. CALCULATION

$$\text{Solvent extract, percent by mass} = \frac{M_1 - M_2}{M_3} \times 100$$

where

M_1 = mass in g of extraction flask and extract,
M_2 = mass in g of extraction flask, and
M_3 = mass in g of test portion.

DETERMINATION OF ORGANIC ACID CONTENT

(NBR : 3)

1. *OUTLINE OF THE METHOD*

1.1 Accurately weighed sample is extracted with *iso*propyl alcohol. The solvent used for extraction as well as the rinsings of the sample are transferred to a volumetric flask and made to a known volume.

An aliquot of the solution is titrated to the first colour change with sodium hydroxide using *m*-cresol purple as indicator.

2. APPARATUS

2.1 Chemical Balance

2.2 Conical Flask — 250 ml capacity.

2.3 Burette — 25 ml capacity.

2.4 Measuring Cylinder — 100 ml capacity.

2.5 Volumetric Flask — 250 ml capacity.

2.6 Extraction Apparatus

3. REAGENTS

3.1 *Iso*propyl Alcohol — *See* IS : 2631-1976*.

3.2 *m*-Cresol Purple Indicator — Dissolve 0·1 g of *m*-cresol purple in 100 ml of ethyl alcohol or water and add 2·6 ml of 0·1 N sodium hydroxide solution.

3.3 Standard Sodium Hydroxide Solution — 0·01 N.

4. PROCEDURE

4.1 Prepare a test portion and extract with 2-propanol as described in (NBR : 2). At the end of the extraction period pour the solution into a 250-ml volumetric flask; rinse the extraction flask with two or three 10-ml portions of warm *iso*propyl alcohol and add to the main bulk of the solution. Make up to the graduation mark with *iso*propyl alcohol, cool to room temperature. Mix thoroughly.

4.2 Measure 100 ml of this solution into a conical flask, add six drops of *m*-cresol purple indicator and titrate with 0·01 N sodium hydroxide solution to the first colour change from yellow to purple-blue. Titrate also 100 ml of *iso*propyl alcohol as a blank determination.

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*Specification for *iso*propyl alcohol (*first revision*).

5. CALCULATION

$$\text{Organic acid, percent by mass} = \frac{(V_1 - V_2) \times N \times F \times 2{\cdot}5}{M}$$

where

V_1 = volume in ml of sodium hydroxide solution required for test portion,

V_2 = volume in ml of sodium hydroxide solution required for the blank,

N = normality of sodium hydroxide solution,

M = mass in g of the test portion, and

F = 28·4 when organic acid is calculated as stearic acid, and
= 34·6 when acid is to be calculated as rosin acid.

DETERMINATION OF SOAP CONTENT

(NBR : 4)

1. OUTLINE OF THE METHOD

1.1 Rubber is extracted with *iso*propyl alcohol and a known portion of the extract is titrated with 0·05 N hydrochloric acid, using *m*-cresol purple as indicator, to the first colour change. From the mass of the test portion and the titration, the percentage of soap is calculated.

2. APPARATUS

2.1 Extraction Apparatus

2.2 Volumetric Flask — 250 ml capacity.

2.3 Burette — 25 ml capacity.

2.4 Conical Flask — 250 ml capacity.

2.5 Measuring Cylinder — 100 ml capacity.

3. REAGENTS

3.1 *Iso*propyl Alcohol — *See* IS : 2631-1976*.

3.2 Hydrochloric Acid — 0·05 N.

3.3 *m*-Cresol Purple Indicator — Dissolve 0·1 g of *m*-cresol purple in 100 ml of ethyl alcohol or water and add 2·6 ml of 0·1 N sodium hydroxide solution.

4. PROCEDURE

4.1 Prepare *iso*propyl alcohol extract as per the procedure given in **4.1** of NBR : 3. Measure exactly 100 ml of the solution and transfer into a 250-ml conical flask. Add six drops of *m*-cresol purple indicator and titrate with 0·05 N hydrochloric acid to the first colour change from purple blue to yellow. Titrate also 100 ml of the *iso*propyl alcohol as a 'blank' determination.

5. CALCULATION

$$\text{Soap, percent by mass} = \frac{(V_1 - V_2) \times N \times F \times 2{\cdot}5}{M}$$

where

V_1 = volume in ml of hydrochloric acid required for the test portion;

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*Specification for *iso*propyl alcohol (*first revision*).

DETERMINATION OF TOTAL ASH CONTENT
(NBR : 5)

0. GENERAL

0.1 Details of two methods, namely, Method A and Method B, have been given.

1. METHOD A

1.1 Outline of the Method — A sample of the rubber is wrapped in filter paper and placed in a crucible which is then put directly into muffle furnace at 550±25°C, till all the carbonaceous matter is oxidized. The ash is weighed and calculated as a percentage of the sample taken for test.

1.2 Apparatus

1.2.1 *Crucible* — silica or porcelain of capacity 85 ml.

1.2.2 *Filter Paper* — ashless, 150 mm diameter.

1.2.3 *Muffle Furnace* — with temperature controlling device.

1.3 Procedure

1.3.1 Heat the crucible to a temperature of 550±25°C for about 30 minutes, cool and weigh to the nearest 1 mg. Weigh about 3 g of dried rubber to the nearest 1 mg. Roll the sample tightly in filter paper, place in the crucible and put the crucible in the muffle furnace at 550±25°C and immediately close the doors and vent.

1.3.2 Continue heating until the carbon is completely oxidized and a white ash is obtained. Cool and weigh to the nearest 1 mg.

1.4 Calculation

$$\text{Total ash, percent by mass} = \frac{M_1 - M_2}{M_3 - M_2} \times 100$$

where

M_1 = mass in g of crucible plus ash,
M_2 = mass in g of crucible, and
M_3 = mass in g of crucible plus dry rubber.

2. METHOD B

2.1 Outline of the Method — The rubber is first heated in a crucible over a low burner flame. When the volatile decomposition products have been expelled the crucible is transferred to a muffle furnace where it is heated until all carbonaceous matter is oxidized.

V_2 = volume in ml of hydrochloric acid required for the blank;

N = normality of hydrochloric acid;

M = mass in g of the test portion; and

F = 30·6 when soap is to be calculated as sodium stearate,
= 36·8 when the soap is to be calculated as sodium rosinate, and
= 38·4 when the soap is to be calculated as potassium rosinate.

NOTE — Unless the exact composition of the soap is known, the value of F will be purely arbitrary, and the figure obtained for the soap content only approximate.

2.2 Apparatus

2.2.1 *Crucible* — silica or porcelain of capacity 85 ml.

2.2.2 *Muffle Furnace* — with temperature controlling device.

2.2.3 *Asbestos Board* — with a central hole to accommodate the crucible.

2.3 Procedure

2.3.1 Heat the crucible in muffle furnace to a temperature of 550±25°C for about 30 minutes, cool and weigh to the nearest 1 mg. Weigh about 3 g of dried rubber to the nearest 1 mg and place in the crucible. Rest the crucible in the hole of the asbestos board, and heat with a small bunsen flame so that the rubber does not inflame and no spurting or undue frothing occurs.

2.3.2 When the rubber has decomposed to a charred mass, increase the heat from the burner until the volatile decomposition products have been substantially expelled, and then transfer the crucible to a muffle furnace at a temperature of 550±25°C. Continue heating until the carbon is completely oxidized and a clean ash is obtained. Cool and weigh to the nearest 1 mg.

2.4 Calculation

$$\text{Total ash, percent by mass} = \frac{M_1 - M_2}{M_3 - M_2} \times 100$$

where

M_1 = mass in g of crucible plus ash,
M_2 = mass in g of crucible, and
M_3 = mass in g of crucible plus dry rubber.

DETERMINATION OF COMBINED ACRYLONITRILE CONTENT

(NBR : 6)

1. OUTLINE OF THE METHOD

1.1 The rubber is oxidized by heating with a mixture of potassium sulphate and concentrated sulphuric acid together with a catalyst, thereby converting nitrogen compounds into ammonium hydrogen sulphate, from which the ammonia is removed by distillation after making the solution alkaline. The liberted ammonia is absorbed in boric acid solution and titrated with standard acid. From this nitrogen determination the amount of combined acrylonitrile content in rubber is calculated.

2. APPARATUS

2.1 Semi-micro Kjeldahl Digestion Flasks — 30 ml capacity.

2.2 Semi-micro Kjeldahl Digestion Unit — with condenser tube of borosilicate glass. The capacity of distillation flask should be 80 to 100 ml.

2.3 Burette — 10 ml, graduated in 0·02 ml.

2.4 Conical Flask — 100 ml capacity, as receiver for Kjeldahl distillate.

3. REAGENTS

3.1 Catalyst Mixture — Prepare a finely divided and intimate mixture of 30 parts by mass of anhydrous potassium sulphate, 4 parts of copper sulphate pentahydrate ($CuSO_4 . 5H_2O$) and 1 part of selenium or 5 parts of sodium selenate decahydrate ($Na_2SeO_4 . 10H_2O$).

3.2 Sodium Hydroxide — 10 M solution.

3.3 Concentrated Sulphuric Acid — 98 percent *m/m* (36 N) solution.

3.4 Boric Acid — 0·5 M solution.

3.5 Sulphuric Acid — 0·1 N solution.

3.6 Indicator Solution — Dissolve 0·1 g of methyl red and 0·05 g of methylene blue in 100 ml of 96 percent ethanol. This indicator may deteriorate on storage and should be freshly prepared as required.

4. PROCEDURE

4.1 Weigh accurately about 0·1 g of dry rubber and place it in a clean dry Kjeldahl flask. Add 0·65 g of catalyst mixture and 3 ml of concentrated sulphuric acid. Boil gently and continue boiling for about half an hour after the digest has become a clear green liquid with no yellow tint.

Commence at the same time a blank determination using same quantities of reagents taken from the same containers as those used in testing the sample, and thereafter treat the test portion and blank determination similarly. Excess boiling, as indicated by a tendency for the digest to solidify on cooling, should be avoided, since this may lead to loss of nitrogen.

4.2 Boil water in the steam generator of the distillation unit and pass steam through the apparatus, including the receiver for 2 minutes.

4.3 Meanwhile, cool the Kjeldahl flask and its contents to room temperature, dilute with 10 ml of water and immediately transfer to the distillation flask at the conclusion of the steaming-out process. Rinse with three 3-ml portions of water to complete the transfer and drain as thoroughly as possible after each transfer.

4.4 Discard any distillate from the receiver of the distillation unit, add 10 ml of boric acid solution, two drops of indicator solution, and place the receiver under the condenser so that the end of the condenser dips below the surface of the acid.

4.5 From a measuring cylinder add 15 ml of 10 M sodium hydroxide solution to the distillation flask and drain well. Pass steam from the generator through the distillation flask for 10 to 12 minutes at such a rate that the final volume of the liquid in the receiver is about 70 ml. At the conclusion of the distillation, lower the receiver until the tip of the condenser is above the level of the acid, continue distilling for 1 minute longer and then rinse the end of the condenser into the distillate with a few millilitres of water. Immediately titrate the contents of the receiver with 0·1 N sulphuric acid.

5. CALCULATION

$$\text{Combined acrylonitrile content, percent by mass} = \frac{(V_1 - V_2) \times N \times 0{\cdot}053\,06}{M} \times 100$$

where

V_1 = volume in ml of titrant used for test portion,
V_2 = volume in ml of titrant used for blank,
N = normality of titrant, and
M = mass in g of the sample taken.

DETERMINATION OF SOLUBILITY

(NBR : 7)

1. OUTLINE OF THE METHOD

1.1 A weighed amount of unmilled rubber is shaken with appropriate solvent for 24 hours at room temperature. The solution is poured through a weighed standard sieve of 150 micron aperture size, the insoluble material thoroughly washed with solvent, and the screen dried and reweighed.

2. REAGENT

2.1 Methyl Ethyl Ketone

3. APPARATUS

3.1 Bottle with Screw Cap — approximately 250 ml to 300 ml capacity.

3.2 Sieve — 150-micron IS sieve made from non-corrodible wire.

3.3 Dispersion Rolls or Mechanical Shaker

4. PROCEDURE

4.1 Weigh accurately about 0·1 g of raw unmilled rubber cut into small pieces, put it into the bottle and add 100 ml of methyl ethyl ketone.

4.2 Close the bottle tightly and allow to roll on dispersion rolls or shake slowly on mechanical shaker for 24 hours at room temperature. Decant the solution through 150-micron IS sieve which has been weighed to 0·1 mg and rinse the jar and the sieve with several portions of cold solvent making certain that all the insoluble gel is transferred to the sieve. When the soluble material has been completely washed from the sieve, place it in a vacuum oven at 120±5°C for 30 minutes, cool in a desiccator and weigh to 0·1 mg.

5. CALCULATION

$$\text{Solubility, percent by mass} = \frac{M_1 - M_2}{M_1} \times 100$$

where

M_1 = mass in g of the material used for the test, and
M_2 = mass in g of the insoluble gel collected on sieve.

DETERMINATION OF MOONEY VISCOSITY
(NBR : 8)

1. OUTLINE OF THE METHOD

1.1 The test involves the application of a torque to a metal disk in a cylindrical chamber filled with rubber under specified conditions. A number proportional to the torque necessary to rotate the disk is taken as an index of the viscosity of the rubber.

2. APPARATUS

2.1 The essential parts of the Shearing Disc Viscometer 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 (*see* Fig. 1). The rotor and die cavity have the dimensions given below:

	mm
Rotor diameter	38·10 ± 0·03
Rotor thickness	5·55 ± 0·03
Die cavity diameter	50·90 ± 0·15
Die cavity depth	10·60 ± 0·03

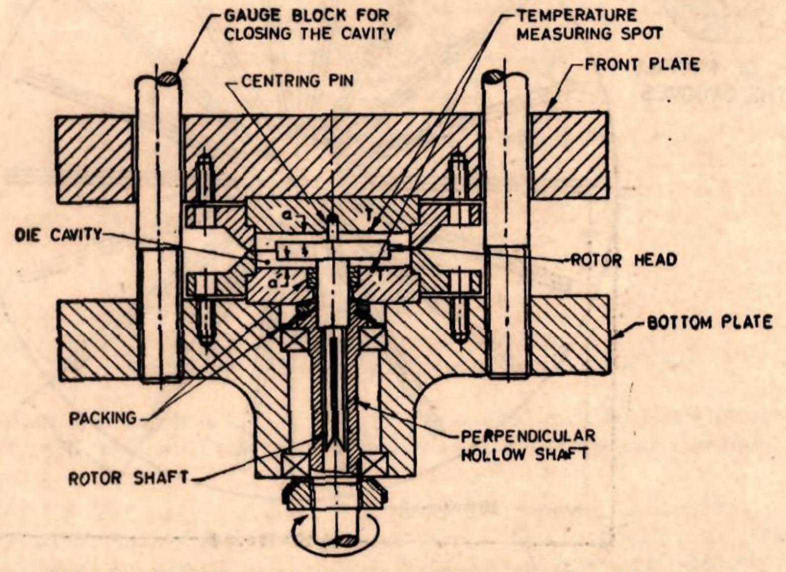


FIG. 1 ESSENTIAL PARTS OF THE SHEARING DISC VISCOMETER

2.1.1 It is permissible to use a smaller rotor where high viscosity makes this desirable. This small rotor should have the same thickness as the large rotor but the diameter should be 30·48 ± 0·03 mm.

2.1.2 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.

2.1.3 The die cavity (*see* Fig. 2) 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 circle; each groove forms a 90° angle in the die surface with the disector of the angle perpendicular to the surface and is 1·00 ± 0·25 mm wide at the surface.

2.1.4 The die cavity (*see* Fig. 3) may alternatively be formed from four pieces of steel with rectangular-section grooves (*see* Fig. 3A and 3B) on the cavity surfaces to retard slipping. The grooves are 0·80 ± 0·02 mm wide,

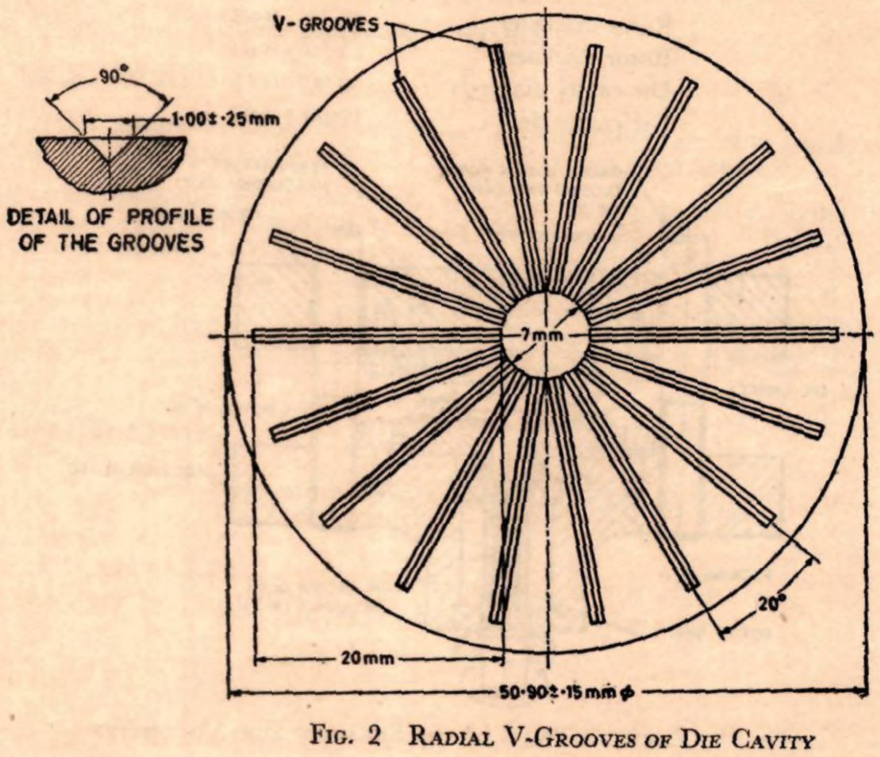
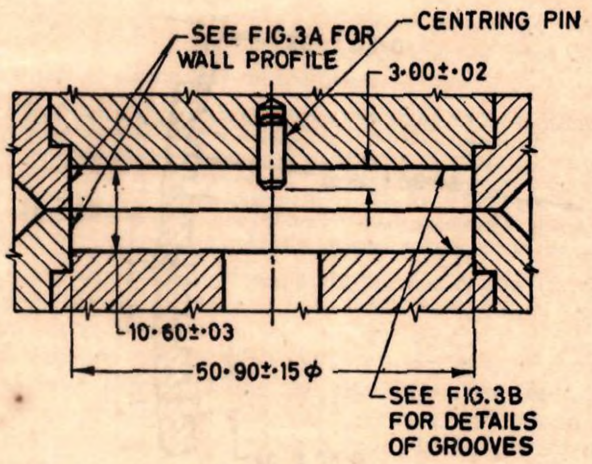
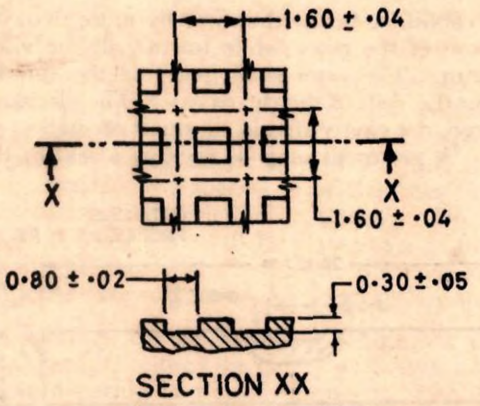


FIG. 2 RADIAL V-GROOVES OF DIE CAVITY



All dimensions in millimetres.

FIG. 3 DIE CAVITY

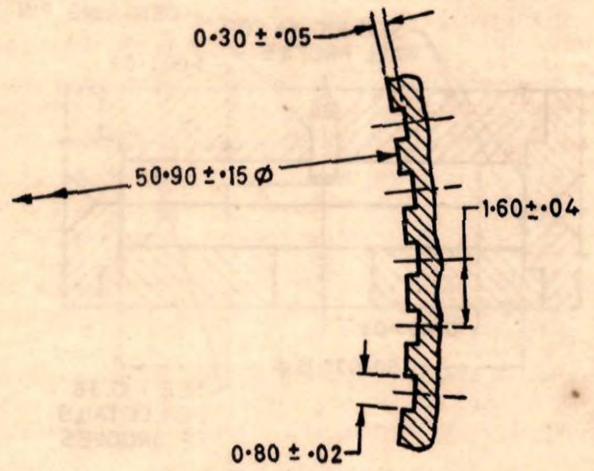


All dimensions in millimetres.

FIG. 3A WALL PROFILE OF THE DIE CAVITY

of uniform depth of 0·3±0·05 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.

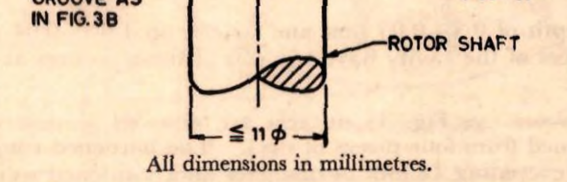
2.1.5 The rotor (*see* Fig. 4) surfaces are grooved as described for the die cavity formed from four pieces of steel. The hardened rotor is fastened to a shaft not exceeding 11 mm in diameter and positioned securely so that in the closed die cavity the clearance above the rotor (*see a* in Fig. 1) does



All dimensions in millimetres.

FIG. 3B RECTANGULAR SECTION GROOVES

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.



All dimensions in millimetres.

FIG. 4 ROTOR

2.1.6 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.

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 differential exists between the platens and the die cavity, it is necessary to adjust the platen temperatures to obtain the correct cavity temperature. In making such adjustments it is important that the temperature of the two platens be within 0·5°C of each other. The cavity temperature may be measured with calibrated thermocouples or thermistors using lead wires about 0·3 mm in diameter to minimize thermal conduction to the exterior.

2.1.7 The die cavity may be closed by hydraulic, pneumatic or mechanical means. If fluid pressure is used, a force of 11500±500 N (approximately 1 150±50 kgf) is maintained on the dies during the test. A greater force may be needed to close the die cavity when rubber of high viscosity is tested. At least 10 seconds before starting the viscometer, the force is reduced to 3 500±200 N (approximately 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 mm and 0·15 mm when the die cavity is closed at the test temperature. For all types of closing devices the closure is adjusted so that a piece of thin soft tissue paper not thicker than 0·04 mm, placed between the meeting surfaces shows a continuous pattern of uniform intensity when the die cavity is closed.

2.1.8 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 8·28 joule (approximately 84·6±0·2 kgf. cm) on the rotor 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 frictionless pulleys. During the calibration the rotor is turned at 2 rev/min and the platens are at the test temperature. The reading should be zero with the torque removed and 100±0·5 when a torque of 8·28 joule (approximately 84·6 kgf.cm) is applied.

2.1.9 After calibration, the scale reading is adjusted to zero with the test rotor turning in the empty, closed die cavity at 2 rev/min. The fluctuations in readings during a period of a half minute or more should be less than 0·5 unit.

3. PROCEDURE

3.1 Preparation of Rubber Test Piece — Take a test portion of about, but not more than 250 g and mix it by passing 3 times between the rolls of the laboratory mill with rolls having 150±0·5 mm diameter with a working distance between the guides at the nip of 250 to 280 mm. The speed of the slow roll shall be 24±0·5 rev/min and the gear ratio between 1 : 1 and 1·5 : 1. Set the distance between the rolls at 1·30±0·15 mm as determined by a lead strip and maintain the roll surface temperature at 50±5°C.

Do not allow the test portion to band on the mill rolls and return any solid matter parting from the rubber to it. Cut out disks 45 mm in diameter from the thickest part of the sheet and allow them to rest at room temperature for at least 30 minutes. Prepare two disks of rubber each about 45 to 50 mm in diameter and 6 to 8 mm in thickness to fill completely the cavity of the viscometer. The rubber disks should be as free as possible from air and die surfaces. A hole is pierced or cut through the centre of one disk to permit the insertion of the rotor stem.

3.2 Heat the die cavity and rotor to the test temperature of 100°C and allow to reach a steady-state. Open the die cavity, insert the rotor stem through the hole in test disk, place the rotor in the viscometer, place the solid test disk centrally on the rotor and close the die cavity as quickly as possible. Insert, if necessary, a film of transparent cellulosic material approximately 0·03 mm in thickness between the rubber and metal surfaces to facilitate removal after test when testing low-viscosity or sticky materials. Note the time of closing the die cavity and allow the rubber to heat for one minute. Turn the rotor at 2·00±0·02 rev/min unless otherwise specified after the specified time of preheat. If the viscosity is not recorded continuously, observe the indicator and scale continuously during the 30 seconds interval preceding the specified time of reading and the minimum value to the nearest 0·5 unit during this interval as the viscosity. For reference purposes, take readings at 5-second intervals from 1 minute before to 1 minute after a 4-minute test period. Draw smooth curve through the minimum points of the periodic fluctuations or through all points if there are no fluctuations. Take the viscosity as the point where the curve intersects the time specified. If a recorder is used, take the viscosity from the recorded curve in the same manner as specified for the plotted curve. The time of reading is generally 4 minutes after preheat.

4. REPORT

4.1 The report shall include the following :

a) Mooney viscosity number (M);
b) Temperature of test;
c) Rotor size, large or small (L or S);
d) Time interval of preheat; and
e) Time of reading after preheat.