Indian Standard

METHODS OF TEST FOR NATURAL RUBBER

PART 7 DETERMINATION OF MOONEY VISCOSITY

[NR : 8]

(Second Revision)

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0. FOREWORD

0.1 This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards on 14 April 1988, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Methods of test for natural rubber had been originally covered in the follwing four parts of IS : 3660:

IS : 3660 (Part 1)-1972 Determination of dirt, volatile matter, ash, total copper, manganese, rubber hydrocarbon, viscosity (shearing disk viscometer), and mixing and vulcanizing of rubber in a standard compound (*first revision*)

IS : 3660 (Part 2)-1968 Determination of solvent extract and nitrogen content

IS : 3660 (Part 3)-1971 Plasticity and plasticity retention index

IS : 3660 (Part 4)-1979 Determination of colour, accelerated storage-hardening test and vulcanization characteristics (MOD test)

0.2.1 While reviewing various test methods for natural rubber, the Committee decided to align them with corresponding international standards. No unification of test methods for natural and synthetic rubber has been considered necessary. However, in revising test methods for natural rubber, the Committee had decided to revise and spli tthe standard into further parts and publish individual test methods under natural rubber (NR) series. For proper referencing of the existing test methods and the new methods under revision, a table showing correspondence of the various methods of test covered in the earlier 4 parts of IS : 3660 with the presently split parts retaining the original NR number has been given in Appendix A.

0.2.2 In order to facilitate cross-reference, it has been decided to retain the original discrete NR series numbers assigned to various test methods in original IS : 3660.

0.3 The test method given in this revised standard will supersede the test method as given in NR : 8 of IS : 3660 (Part 1)-1972. All the four parts of the original IS : 3660 shall be withdrawn when it is completely revised.

0.4 The earlier test method was designated as determination of viscosity by shearing disk viscometer. The designation has now been modified.

0.4.1 In this revision, relevant drawings of shearing disk viscometer and its parts have been included. Minor deviations in the dimensions of the various parts of viscometer have been made. Other changes in the apparatus and procedure have been made in order to update the standard.

0.5 In the preparation of this standard, assistance has been derived from ISO 289-1985 'Rubber, unvulcanized — Determination of Mooney viscosity', issued by the International Organization for Standardization (ISO).

0.6 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*.

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

1. SCOPE

1.1 This standard (Part 7) specifies a test method for determining Mooney viscosity of raw natural rubber by means of shearing disk viscometer.

2. OUTLINE OF THE METHOD

2.1 The torque which has to be applied under specified conditions in order to rotate a metal disk in a cylindrical chamber formed from mating dies filled with rubber is measured. The

resistance to this rotation, offered by the rubber, is expressed in arbitrary units as the Mooney viscosity of the test piece.

3. APPARATUS

3.1 The essential parts of the apparatus (*see* Fig. 1) are:

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 of 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.

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

TABLE 1 DIMENSIONS OF ESSENTIAL PARTS OF THE APPARATUS

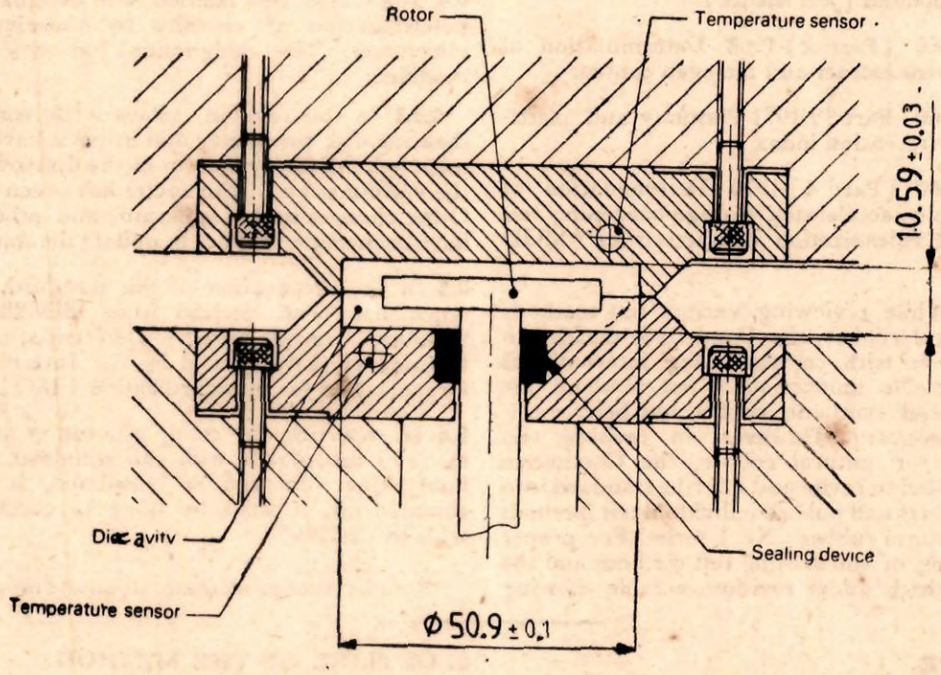
PART	DIMENSION, mm
Rotor diameter	38·10 ± 0·03
Rotor thickness	5·54 ± 0·03
Die cavity diameter	50·9 ± 0·1
Die cavity depth	10·59 ± 0·03

NOTE — Normally this rotor is called the large rotor.

3.1.2 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 should be 30·48 ± 0·03 mm. Results obtained with the small rotor are not identical with those obtained using the large rotor.

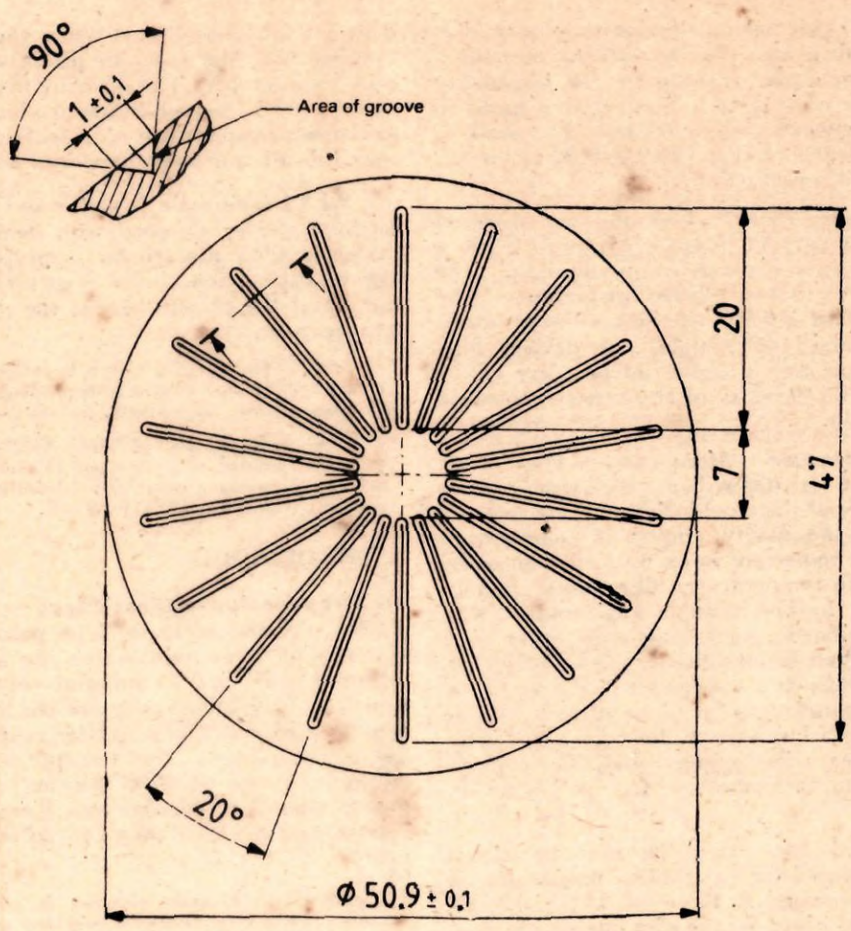
3.1.3 *Dies* — The two dies forming the cavity shall be formed from non-deforming unplated hardened steel of minimum Rockwell hardness HRC 60. The dimensions of the cavity are given in Fig. 1 and shall be measured from the highest surfaces. For good heat transfer, each die shall preferably be made from only one piece of steel. The surfaces shall have radial V-grooves on the flat surfaces to prevent slipping. 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 dies and to within 1·5 mm of the hole in the lower die; each groove shall form 90° angle in the die surface with the bisector of the angle perpendicular to the surface and shall be 1·0 ± 0·1 mm wide at the surface (*see* Fig. 2).

3.1.3.1 Alternatively, each die may be formed from two pieces of steel with rectangular section grooves on the surfaces to prevent



All dimensions in millimetres.

FIG. 1 TYPICAL SHEARING DISK VISCOMETER



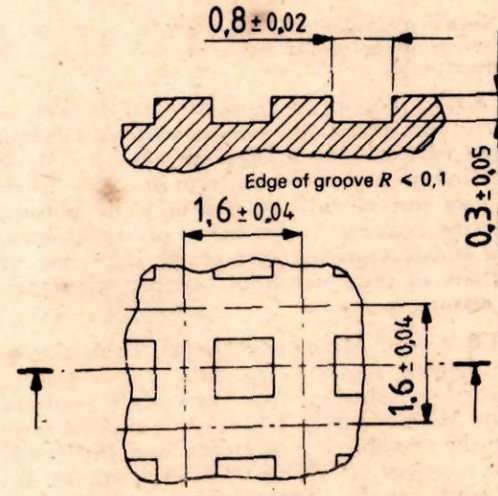
All dimensions in millimetres.

FIG. 2 DIE WITH RADIAL V-GROOVES

slipping. The grooves shall be 0·80 ± 0·02 mm wide, of uniform depth 0·30 ± 0·05 mm and spaced on 1·60 ± 0·04 mm centres. The flat surfaces of the dies have two sets of these grooves at right angles to each other (*see* Fig. 3).

NOTE — The two types of dies may not give the same results.

3.1.4 *Rotor* — The rotor shall be fabricated from a non-deforming unplated hardened steel of minimum Rockwell hardness HRC 60. The rotor surfaces shall be grooved as in the manner shown in Fig. 3. The rotor is fastened at right angles to a shaft having a diameter 10 ± 1 mm, and of 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 enters the cavity shall be small enough to prevent rubber leaving the cavity. A grommet,



All dimensions in millimetres.

FIG. 3 GROOVES WITH RECTANGULAR SECTION OF DIE AND ROTOR

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 relative speed of rotation between the rotor and dies shall be 0·209 ± 0·000 2 rad/s (2·00 ± 0·02 rev/min), unless otherwise stated.

3.1.5 *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 sample, the devices shall be capable of returning the temperature of the dies to within ± 0·5°C of the test temperature within 5 min.

3.1.6 *Temperature Measurement* — The test temperature is defined as the steady state temperature of the closed dies with the rotor in place and the cavity empty. A temperature sensor shall be present in each die for measurement of die temperature. The sensor shall be located for the best possible heat contact with the dies, that is, heat gaps and other heat resistance shall be excluded. The axes of the sensors shall be at a distance of 3 to 5 mm from the working surface of the dies and 15 to 20 mm from the rotation axis of the rotor. The temperature measuring system shall be capable of indicating temperature to an accuracy of 0·25°C.

3.1.7 *Die Closure* — The dies may be closed and held closed by hydraulic, pneumatic or mechanical means. A force of 11·5 ± 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 from 11·5 to 0·5 kN and maintained at this level throughout the test.

NOTE — A closing and holding force of 8·0 kN is allowable if the rubber has a viscosity less than 50 Mooney units.

3.1.7.1 For all types of closing devices, 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 die closure, worn or faulty mating surfaces or distortion of dies; any of these conditions results in leakage and erroneous results.

3.1.8 *Torque Measurement and Calibration* — 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 ± 0·02 Nm is applied to the rotor shaft. Therefore, a torque of 0·083 Nm 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 ± 0·5 Mooney units when the machine is running with the rotor in place, and the dies closed and empty. The shearing disk 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.

3.1.8.1 The scale is calibrated 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 1 — In order to check linearity, intermediate masses may be used to give scale readings of 25, 50 and 75 Mooney units, respectively.

NOTE 2 — A sample of butyl rubber of certified Mooney viscosity may be used to check whether the machine is working correctly. Measurement may be carried out at 100 or 125°C for 8 min.

4. PROCEDURE

4.1 Preparation of Test Piece — For natural rubber, the test piece shall be passed through the nip of a roll mill where the nip width is adjusted to 1·3 ± 0·15 mm and rolls are maintained at 70 ± 5°C. Carry out the homogenization of rubber by passing the rubber through the nip 10 times. The test portion shall be allowed to rest at 27°C for at least 30 min before testing is carried out. Testing shall be commenced not later than 24 h after homogenization.

NOTE — The Mooney 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 evaluation of the particular rubber should be followed rigorously.

4.1.1 The test piece shall consist of two disks 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 disks 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 disk to permit the insertion of the rotor stem.

4.2 Temperature and Duration of Test — The test shall be made at 100 ± 0·5°C for 4 min.

4.3 Testing

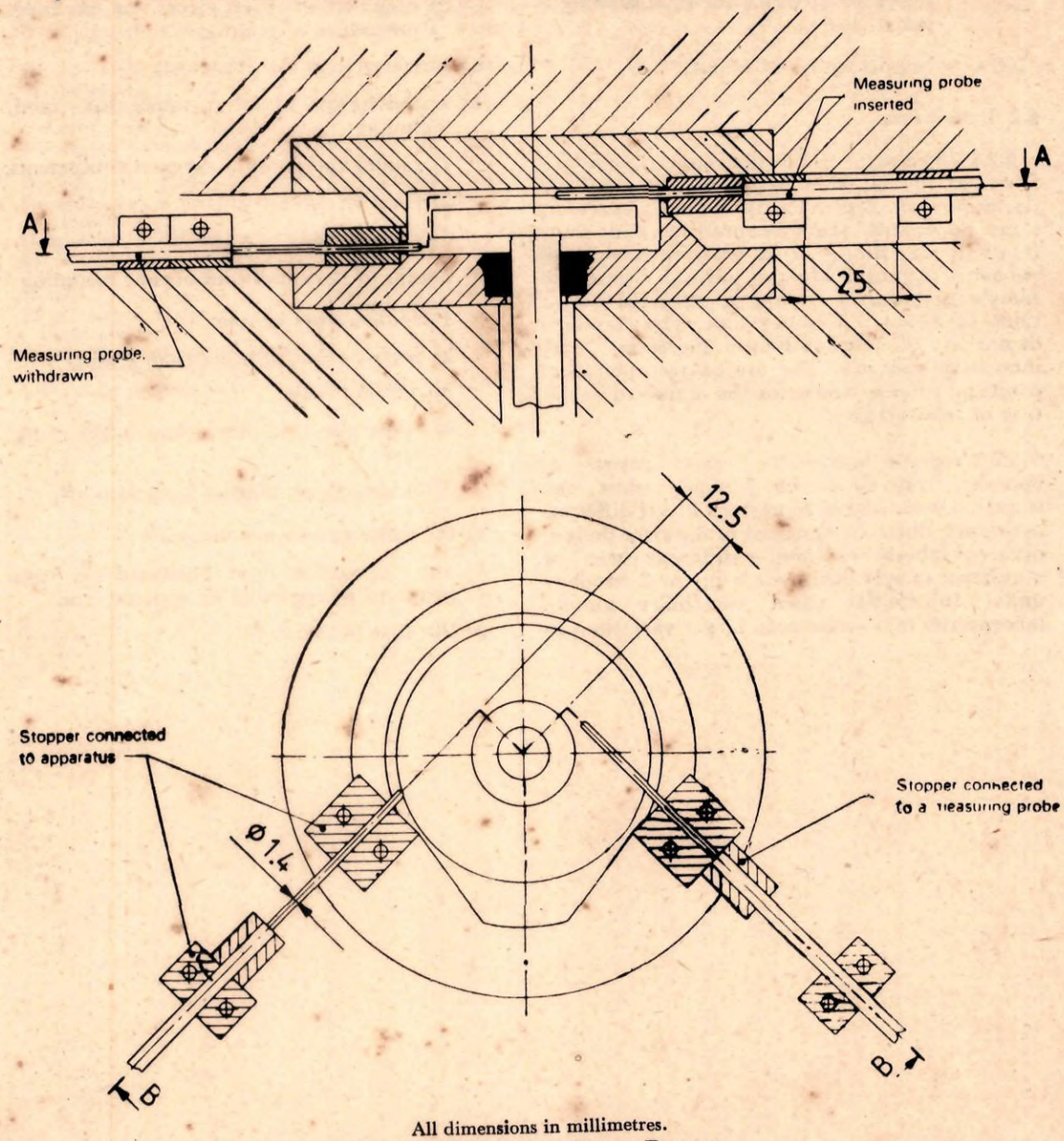
4.3.1 Heat the dies and rotor to the test temperature and allow them to reach a steady state. Open the dies, insert the rotor stem through the hole in the test piece and place the rotor in the viscometer. Place the second test piece centrally on the rotor and close the dies as quickly as possible.

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.

4.3.2 Note the time at which the dies are closed and allow the rubber to preheat for 1 min. Start the rotor and run for 4 min. 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 unit 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 point of the periodic fluctuations or through all the 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 curve in the same manner as specified for the plotted curve.

NOTE — To check if the temperature of the test piece is at the test temperature at the preferred test-time (4 min), two thermo-couple measurement probes may be inserted into the sample as shown in Fig. 4. In a preliminary test with the test piece to be measured, the rotor is stopped after a running time of 3 and 5 min, and immediately after the resulting standstill, the two measurement probes are inserted so that after 4 min the two mean test piece temperatures can be read off. The temperature tolerance should be between 0·0 and −2°C. The temperature gradients in the test piece 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 conditions is reached within 10 min after the die cavity is closed.



All dimensions in millimetres.

FIG. 4 MEASURING PROBE DESIGN

5. EXPRESSION OF RESULTS

5.1 Results of a typical test shall be reported as follows:

50 *ML* (1 + 4) 100°C

where

50 *M* = viscosity, in Mooney units;

L = large rotor used (*S* would indicate use of the small rotor);

1 = preheating time, in minutes, before starting the rotor;

4 = running time, in minutes, after starting rotor at which the final reading is taken; and

100°C = temperature of the test.

5.2 Precision

5.2.1 *Repeatability* — For the range of viscosity from 40 to 60 Mooney units, the standard deviation of repeatability (same operator, same apparatus, same laboratory and short intervals of time) of a uniform sample of rubber is about 0·2 Mooney units. Variation in sample preparation results in standard deviations of about 1 Mooney unit. The standard deviation of repeatability increases with increasing viscosity. The use of test pieces of constant volume decreases the standard deviation of repeatability.

5.2.2 *Reproducibility* — For the range of viscosity from 40 to 60 Mooney units, the standard deviation of reproducibility (different operators, different appartus of the same design, different laboratories and/or different times) of a uniform sample of rubber is up to 2 Mooney units. In special cases, variability among laboratories may cause even larger variations in results. Part of the inter laboratory variability is due to sample preparation, and part to differences in calibrating or adjusting the viscometer. The standard deviation of reproducibility increases with increasing viscosity. The use of test pieces of constant volume decreases the standard deviation of reproducibility.

6. TEST REPORT

6.1 The test report shall include the following information:

a) a full description and identification of the sample tested, including
   1) its origin; and
   2) preparation of test pieces, for example, procedure of milling;
b) a reference to this standard;
c) a description of the apparatus used, including
   1) model and manufacturer of equipments;
   2) rotor sizes; and
   3) type of dies;
d) details of the conditions of test, including
   1) temperature of test;
   2) preheat time, if other than 1 min;
   3) running time;
   4) rotor speed, if other than 0·209 rad/s; and
   5) closing force, if other than 11·5 kN;
e) the value of Mooney viscosity;
f) any operation not included in this standard or regarded as optional; and
g) the date of test.

APPENDIX A

(*Clause* 0.2.1)

TABLE SHOWING CORRESPONDENCE OF VARIOUS METHODS OF TEST COVERED IN THE EXISTING IS : 3660 (PART 1)-1972, IS : 3660 (PART 2)-1968, IS : 3660 (PART 3)-1971, AND IS : 3660 (PART 4)-1979 WITH THE REVISION/PROPOSED REVISION OF ALL THE FOUR PARTS OF IS : 3660

Existing Test Methods			Proposed Revision		Remarks
Test Methods	IS : No.	Part (Series)	IS : No.	(Series)	
(1)	(2)	(3)	(4)	(5)	(6)
NR Series					
Determination of dirt	IS : 3660-1972	Part 1 (NR : 1)	IS : 3660 (Part 1)-1985	(NR : 1)	
Determination of volatile matter	IS : 3660-1972	Part 1 (NR : 2)	IS : 3660 (Part 2)-1985	(NR : 2)	
Determination of ash	IS : 3660-1972	Part 1 (NR : 3)	IS : 3660 (Part 3)-1988	(NR : 3)	
Determination of total copper	IS : 3660-1972	Part 1 (NR : 4)	IS : 3660 (Part 4)-1988	(NR : 4)	Under revision
Determination of manganese	IS : 3660-1972	Part 1 (NR : 5)	IS : 3660 (Part 5)	(NR : 5)	Under revision
Determination of iron	IS : 3660-1972	Part 1 (NR : 6)	Deleted since this test is no longer being done		
Determination of rubber hydrocarbon	IS : 3660-1972	Part 1 (NR : 7)	IS : 3660 (Part 6)-1988	(NR : 7)	
Determination of viscosity by shearing disk viscometer	IS : 3660-1972	Part 1 (NR : 8)	IS : 3660 (Part 7)-1988	(NR : 8)	
Mixing and vulcanizing in a standard compound	IS : 3660-1972	Part 1 (NR : 9)	IS : 3660 (Part 8)	(NR : 9)	Under revision
Determination of solvent extract	IS : 3660-1968	Part 2 (NR : 10)	IS : 3660 (Part 9)	(NR : 10)	Under revision
Determination of nitrogen content	IS : 3660-1968	Part 2 (NR : 11)	IS : 3660 (Part 10)	(NR : 11)	Under revision
Determination of plasticity	IS : 3660-1971	Part 3 (NR : 12)	IS : 3660 (Part 11)	(NR : 12)	Under revision
Determination of plasticity retention index (PRI)	IS : 3660-1971	Part 3 (NR : 13)	IS : 3660 (Part 12)	(NR : 13)	Under revision
Determination of colour	IS : 3660-1979	Part 4 (NR : 14)	IS : 3660 (Part 13)	(NR : 14)	Under revision
Determination of storage-hardening test	IS : 3660-1979	Part 4 (NR : 15)	IS : 3660 (Part 14)	(NR : 15)	Under revision
Determination of vulcanization characteristics (MOD test)	IS : 3660-1979	Part 4 (NR : 16)	IS : 3660 (Part 15)	(NR : 16)	Under revision
Method for preparation of test samples	IS : 3660-1972	Part 1 (Clause 3)	IS : 3660 (Part 16)	(NR : 17)	Under revision