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Indian Standard

METHODS OF TEST FOR VULCANIZED RUBBERS

PART VI RESISTANCE TO LIQUIDS

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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Indian Standard
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PART VI RESISTANCE TO LIQUIDS

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 13 September 1967, after the draft finalized by the Rubber Products Sectional Committee had been approved by the Chemical Division Council.

0.2 This method of test is intended to furnish the rubber industry with standard procedure and terminology in the field of testing rubber and rubber products.

0.3 The action of a liquid on a vulcanized rubber generally results in (a) absorption of liquid by the rubber, and (b) extraction of soluble constituents from the rubber. Usually (a) is greater than (b) so that the net result is an increase in volume, commonly termed 'swelling'. The absorption of liquid can profoundly alter the physical and chemical properties of the rubber, such as tensile strength, extensibility and hardness, so that it is important to measure these properties of the rubber after treatment. The extraction of soluble constituents, especially plasticizers, can likewise alter the physical and chemical properties shown by the rubber after drying out the liquid (assuming this to be volatile); mechanical tests on the rubber after immersion and drying are therefore required.

The methods described below accordingly comprise determination of:

a) change in volume or dimensions,
b) soluble matter extracted,
c) tensile stresss-train properties of the rubber after treatment,
d) hardness of the rubber after treatment,
e) tensile stress-strain properties of the rubber after drying out the immersion liquid, and
f) hardness after drying out the immersion liquid.

Although in some respects these tests may simulate service conditions closely, no direct correlation with service behaviour is implied; thus, the rubber giving the lowest change in volume is not necessarily the best in service. It is known moreover, that the action of a liquid on rubber, especially at high temperature can be markedly effected by the presence of atmospheric oxygen. The test described here, however, can provide valuable information on the suitability of a rubber for use with a given liquid, and, in particular, constitute a useful control when used comparatively for developing rubbers resistant to oils, fuels, or other liquids.

0.4 This standard is based on the following standards:

ISO/TC 45(Sect-858) 1140 Second draft ISO proposal for the methods of tests for the resistance of vulcanized rubbers to liquids. International Organization for Standardization.

B.S. 903 : Part A16 : 1956 Methods of testing vulcanized rubber, determination of swelling in liquids. British Standards Institution.

ASTM D 471-59T Determination of swelling in liquids. American Society for Testing and Materials.

0.5 In reporting the results 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 test procedures for determination of change in volume or dimensions, soluble matter extracted by liquid, mechanical tests on swollen rubber and mechanical properties after immersion and drying in Section 1, Section 2, Section 3, and Section 4 respectively.

2. TERMINOLOGY

2.0 For the purpose of this standard, the following definitions shall apply.

2.1 Change in Volume — Change in volume is the percentage change in volume which a test piece of given initial dimensions undergoes when immersed in or exposed to the vapour of a liquid for a given time and at a given temperature.

2.2 Change in Dimensions — The percentage change in linear dimensions which a test piece of given initial dimensions undergoes when immersed in or exposed to the vapour of a liquid for a given time and at a given temperature.

SECTION I DETERMINATION OF CHANGE IN VOLUME OR CHANGE IN DIMENSIONS

3. OUTLINE OF THE METHOD

3.1 The tests described in **8.1** and **8.2** consist in immersing a test piece of rubber in a liquid for a given time, at a constant temperature, and determining the change in volume or the change in linear dimensions. In some

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

instances the liquid may extract a proportion of the plasticizer or other soluble ingredients of the rubber, and this possibility should be borne in mind when interpreting the results. The curve relating swelling to time of immersion for natural rubber is of the form shown in Fig. 1, if there is no substantial extraction of soluble matter by the liquid. It consists of a markedly curved part *OA* representing the initially rapid absorption of liquid, and the final part *AB* which is almost straight, with an upward slope representing a slow continuous absorption of liquid, which is termed as the swelling increment and appears to be due to oxidation. With synthetic rubbers, the increment may be so small as to be negligible.

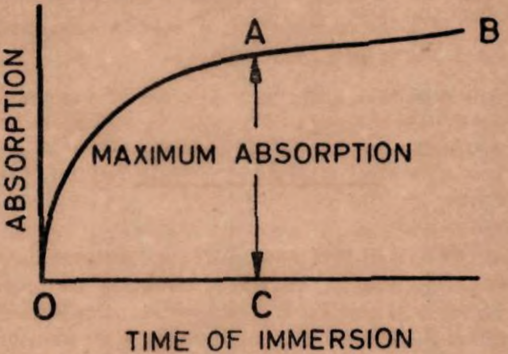

FIG. 1 TIME-ABSORPTION CURVE OF SOFT VULCANIZED RUBBERS

3.2 The two methods described in this standard for studying swelling characteristics of rubbers are as follows:

a) Volumetric method, and
b) Dimensional-change method.

3.2.1 Volumetric method is more precise, and is the preferred method when it is required to know the change in volume due to immersion. Dimensional-change method is not suitable for absolute measurements but is useful for detecting grain (anisotropy) in the rubber, and it shall be used when it is required to determine changes in linear dimensions, because these cannot always be calculated from the volume change owing to grain in the rubber. Further a method for examining the effect of exposing a thin sheet of rubber or a rubber coated fabric to liquid on one side only is also described.

4. QUALITY OF REAGENTS

4.1 Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS : 1070-1960*) 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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*Specification for water, distilled quality (*revised*).

5. TEST LIQUIDS

5.0 The selection of the test liquid depends on the purpose of the tests.

5.1 When information is required on the probable service behaviour of a rubber in contact with a liquid, this liquid shall preferably be used in the test. Commercial liquids, however, are not always constant in composition, and the test shall, therefore, whenever practicable, include a control rubber of known change in volume characteristics; abnormal results due to unsuspected variations in the commercial liquid shall thus be made apparent. It may be found essential to set aside a bulk supply of the liquid for a particular series of tests.

5.2 Mineral lubricating oils and fuels are liable to vary appreciably in aromatic content even when supplied to a recognized specification. The aniline point of a mineral oil gives some indication of its aromatic content and helps to characterize the action of the oil on rubber; other things being equal, the lower the aniline point the more severe is the action. The report shall therefore include the density, refractive index, and the aniline point or aromatic content of the oil or fuel, if this is used as the immersion liquid.

5.3 As commercial liquids may not have an entirely constant composition, the test shall, whenever possible, be made in well-defined chemical products, used alone or as mixtures. These shall be as representative as possible of the effect of the commercial products on the rubber. Examples of such chemicals are toluene, alcohols, esters, cyclohexane, acetone.

5.4 For use in systems of classification of vulcanized rubbers or for quality control, a standard test liquid for immersion shall be used. Suitable test liquids for immersion have been recommended in Appendix A.

6. CONDITIONING OF TEST PIECES

6.1 The properties of vulcanized rubbers change continuously with time, these changes being particularly rapid during the first 24 hours after vulcanization. No tests shall, therefore, be carried out within this period and, for accurate comparisons between different rubbers, it may be necessary to ensure that these are tested at substantially the same interval after vulcanization. Samples after any necessary preparation, shall be conditioned at 27° ± 1°C for at least 12 hours before the test pieces are cut. These test pieces may be tested immediately, but, if not tested immediately, they shall be kept at 27° ± 1°C until tested. If the preparation involves buffing, the interval between buffing and testing shall not exceed 72 hours.

7. TEMPERATURE OF TEST

7.1 The immersion test should preferably be made at one or more of the following standard temperatures:

$-55 \pm 1, -40 \pm 1, -25 \pm 1, -10 \pm 1, 0 \pm 1$°C
$20 \pm 1, 23 \pm 1, 27 \pm 1$°C
$50 \pm 1, 70 \pm 1, 100 \pm 1, 125 \pm 1$°C
$150 \pm 2, 175 \pm 2, 200 \pm 2, 225 \pm 2, 250 \pm 2$°C

Whenever possible the temperature equal to or immediately above that at which the rubber will be used, shall be selected. In other case the standard laboratory temperature as given in **6.1** shall be adopted.

8. TEST METHODS

8.1 Volumetric Method

8.1.1 *Apparatus* — The apparatus shall be either (a) or (b) depending on the temperature of the test and the volatility of the test liquid, used for immersion.

a) For tests at temperatures appreciably below the boiling point of the test liquid, a stoppered glass bottle or tube is used, of such dimensions that the test pieces remain completely immersed in the specified volume of the test liquid, are freely exposed at all surfaces without restraint.

b) For tests at temperatures near the boiling point of the test liquid, the bottle or tube is fitted with a reflux condenser capable of minimizing evaporation of the test liquid.

8.1.2 *Test Pieces*—The test pieces are 1 to 3 mm^3 in volume and of uniform thickness of $2 \pm 0{\cdot}2$ mm. Test pieces cut from sheet shall be rectangular, about 50 mm long and 25 mm wide.

8.1.3 *Duration of Test* (*Immersion Period*) — The large variation in the rate of change in volume or dimensions of rubbers in different liquids and in the same liquid at different temperatures precludes the adoption of one standard period of immersion. For general purpose it is recommended that determinations be made and recorded after several periods of immersion, so as to indicate the change of volume or dimensions with time; the total period shall, if possible, extend well beyond the point of maximum absorption (A in Fig. 1). However, in the case of liquids which are absorbed very slowly by the vulcanizate, such as water or high viscosity oils, an immersion period of 70 ± 2 hours may be insufficient.

8.1.4 *Procedure* — Use three test pieces. Weigh each test piece in air to the nearest mg (W_1) and then in water at 27° ± 1°C (W_2) (*see* Note 1), care being taken to ensure that all air bubbles are removed (*see* Note 2). Dry the test pieces by blotting with filter paper and then place suitably separated, in a glass container with a volume of the test liquid at least 15 times the total volume of the test pieces and sufficient to keep them totally immersed. If the conditions of test do not necessitate a reflux condenser, stopper the container. Keep the container at the required temperature, and shield the rubber from light during the test. At the end of the immersion period, cool the test pieces, if necessary, to 27° ± 1°C by quickly transferring them to a fresh portion of the test liquid at this temperature for a period of not less than five minutes and not more than ten minutes. Remove surplus test liquid from the surfaces of each test piece (*see* Note 3), then place immediately in a tared and a stoppered weighing bottle and determine its weight in air (W_3) to the nearest mg. Then remove it from the bottle and immediately weigh in water (W_4) at 27° ± 1°C. If the test liquid is appreciably volatile at room temperature, the time for each transference of the test piece, after removal from the test liquid shall not exceed 30 seconds. If the test is being continued, replace the test piece at once in the test liquid and return them to the thermostatically controlled oven or bath.

NOTE 1 — The above procedure may not be suitable if the test liquid (other than water) is readily miscible with water or reacts with it. For such a liquid, if it is not too viscous or volatile at room temperature, weighings W_2 or W_4 may be made in the test liquid instead of water and these weights used in the formula for Δv given. If this is not practicable the same procedure is used for the water displacement method except that the final weighing in water is omitted and Δv calculated from the formula:

$$\Delta v = \frac{(W_3 - W_1)}{d\,(W_1 - W_2)} \times 100$$

where d is the density of the test liquid at 27° ± 1°C; this formula may be only approximate if the test liquid is a mixture because the density of the absorbed liquid may differ that of the bulk. Also the density of any matter extracted from the rubber may differ from that of the immersion liquid.

NOTE 2 — Formation of bubbles can be avoided by adding a trace of a surface-active material, for example, detergent to the water.

NOTE 3 — The method of removing the surplus liquid from the surface of the test piece will vary with the nature of the liquid. When mobile volatile liquids, such as *isooctane* and benzene are used, a quick wipe with filter paper is adequate. Some difficulty may be experienced in completely removing viscous nonvolatile oils by this method and it may be necessary to dip the test piece quickly in a suitable volatile liquid, such as acetone and again quickly wipe with filter paper.

8.1.5 The change in volume Δv is calculated as follows:

$$\Delta v = \frac{[(W_3 - W_4) - (W_1 - W_2)]}{(W_1 - W_2)} \times 100$$

where

W_1 = the initial weight in air,
W_2 = the initial weight in water,
W_3 = the weight of the treated rubber in air, and
W_4 = the weight of the treated rubber in water.

8.1.5.1 The results for the three test pieces shall be averaged.

8.1.6 *Report* — The report shall state the following:

a) The values for change in volume $\triangle v$ calculated as described in **8.1.5** and the corresponding periods of immersion (*see* **8.1.3**);

b) Temperature of test (*see* **7.1**);

c) Description of the test liquid, used for immersion including (in the case of mineral oils other than standard test liquids recommended in Table 1 in Appendix A) the density, refractive index, and the aniline point or aromatic content;

d) Initial thickness and dimensions of the test piece (*see* **8.1.2**);

e) Temperature of conditioning (*see* **6.1**); and

f) Any discolouration of the test liquid used for immersion or formation of sediment at the conclusion of the test.

8.2 Dimensional-Change Method

8.2.1 *Apparatus*

8.2.1.1 *Immersion apparatus* — This apparatus for immersion of the test pieces shall be as described in **8.1.1.**

8.2.1.2 *Thickness measuring device* — The instrument for measuring the thickness of the test pieces shall consist of a micrometer dial gauge of adequate accuracy firmly held in a rigid stand over a flat base plate. The gauge shall have a scale graduated in unit divisions of 0·01 mm. The plunger shall be fitted with a flat circular contact of diameter approximately 4 mm which shall be square to the plunger and parallel to the base plate. The dial gauge shall be operated under a dead-weight load to give a pressure on the rubber of approximately 200 g/cm².

NOTE — For most accurate results, the instrument is used as a comparator.

8.2.2 *Test Piece* — The test piece shall be rectangular, 50 mm long and 25 mm wide. The thickness shall be uniform preferably 2 ± 0·2 mm. The sides shall be cut cleanly and at right angles to the top and bottom surfaces. When the direction of calender grain is known, the test piece shall be cut with long axis parallel to the grain.

8.2.3 *Temperature of Test* — The temperatures of test shall be as prescribed in **7.1**.

8.2.4 *Duration of Test* — The duration of immersion shall be as prescribed in **8.1.3**.

8.2.5 *Procedure* — Use three test pieces. Take the initial length of each test piece along its centre line to the nearest 0·5 mm; take measurements along the top and bottom surfaces and average the two results. Measure initial width similarly but take four measurements in all, one top and one bottom near each end of the test piece. Measure the initial thickness with the thickness gauge at four different points along the test piece and average the results. Make all measurements with the test piece at 27° ± 1°C. Then place the test piece suitably separated, in the glass container with a volume of the test liquid at least 15 times the total volume of the test piece, and sufficient to keep them totally immersed. If the conditions of the test do not necessitate a reflux condenser, stopper the container. Keep the container at the required temperature, and shield the rubber from light during the test. At the end of the immersion period, cool the test pieces, if necessary, to 27° ± 1°C by quickly transferring them to a fresh portion of the test liquid at this temperature for a period of not less than 30 minutes and not more than 60 minutes and remove surplus test liquid from the surface of the test pieces by wiping with filter paper or a textile fabric which does not deposit lint. Re-measure the length, width and thickness of each test piece as described above at 27° ± 1°C. If the test liquid is appreciably volatile at room temperature, complete the measurements within one minute of removing the test piece from the test liquid.

8.2.6 *Calculation* — Calculate the percentage change in length $\triangle L$ as follows:

$$\triangle L = \frac{l - l_o}{l_o} \times 100$$

where

l = the length after immersion, and

l_o = the initial length.

8.2.6.1 Change in width $\triangle w$ and change in thickness $\triangle T$ are calculated in a similar manner.

8.2.6.2 The results for the three test pieces shall be averaged.

8.2.7 *Report* — The report shall state the following:

a) The value for change in length $\triangle L$ change in width $\triangle W$ and change in thickness $\triangle T$ calculated as described in **8.2.6** at each period of immersion;

b) Temperature of test (*see* **7.1**);

c) Description of the test liquid, used for immersion including (in the case of mineral oils other than standard test liquids recommended in Table 1 in Appendix A) the density, refractive index, and the aniline point or aromatic content;

d) Initial thickness and dimensions of test piece (*see* **8.1.2**);

e) Temperature of conditioning (*see* **6.1**); and

f) Any discolouration of the test liquids, used for immersion or formation of sediment at the conclusion of the test.

8.3 Test With Liquid on One Surface Only

8.3.1 *Scope* — This test is applicable to relatively thin sheet materials like rubber-coated fabrics diaphragms that are exposed to the test liquid (*see* **5**) only on one surface.

8.3.2 *Outline of the Method* — Test pieces are made to contact with the test liquid only on one surface for a specified period at a specified temperature and the change in the thickness (Method A) and change in weight per unit surface (Method B) is measured.

8.3.3 *Apparatus*

8.3.3.1 The apparatus shall consist of a base plate *A* and an open-end cylindrical chamber *B* which shall be held tightly against the test piece *C* by the wing nuts *D*, *D* being mounted on the bolts *E* (*see* Fig. 2). During the test, the opening in the top of the chamber shall be closed by a close fitting plug *F*.

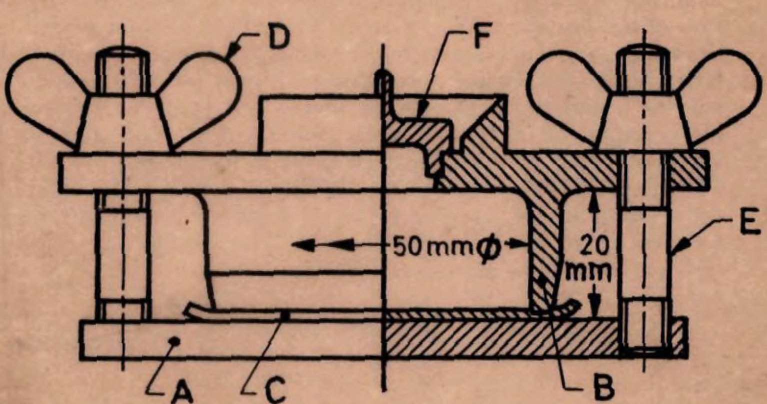

FIG. 2 APPARATUS FOR TEST WITH LIQUID ON ONE SURFACE

8.3.4 *Test Piece* — A disc 60 mm in diameter; thickness shall be that of the sample.

8.3.5 *Temperature of Test* — The temperature of test shall be as prescribed in **7.1**.

8.3.6 *Duration of Test* — The duration of exposure of the test piece liquid shall be as prescribed in **8.1.3.**

8.3.7 *Procedure*

a) *Method A* (*change in thickness*) — Measure the thic test piece at five points distributed over its area, us meter dial gauge. Mount the test piece in the apr in Fig. 2, fill the chamber *B* to a depth of 1·5 cm with the test liquid, and insert the closure *F*. At the end of the test period, remove the test piece and remeasure its thickness at five places as quickly as possible. Average each set of five thickness measurement. Calculate the percentage change in thickness, using the formula:

$$\text{Percentage change in thickness } \triangle t = \frac{t_2 - t_1}{t_1} \times 100$$

where

t_2 = the average thickness after exposure to liquid, and
t_1 = the average thickness before exposure to liquid.

b) *Method B (change in weight per unit surface)* — Weigh the test piece in air to the nearest mg (W_1). Place the test piece in the apparatus as indicated in Method A. Fill the chamber of the apparatus with the test liquid to a depth of 1·5 cm and insert the plug *F*. Maintain the apparatus at the required temperature for the duration of the liquid contact. At the end of the contact period, cool the apparatus, if necessary, to the standard laboratory temperature. Remove the test liquid and release the test piece. Remove any surplus liquid from the surface of the test piece by wiping with filter paper or a textile fabric which does not deposit lint. Determine the weight of the test piece to the nearest mg (W_2) at $27° \pm 1°C$. If the immersion liquid is appreciably volatile at room temperature, complete the measurement within 2 minutes from the removal of the test liquid. The change in weight per unit surface is calculated as follows:

$$\text{Change in weight per unit surface } \frac{\triangle W}{S} = \frac{W_2 - W_1}{0{\cdot}001\ 963}$$

where

W_2 = final weight of test piece,
W_1 = initial weight of test piece, and
0·001 963 = the area in m^2 of the circle 50 mm in diameter which corresponds to the area of contact of the test liquid on the test piece.

8.3.8 *Report* — The report shall state the following:

a) The value of $\triangle t$ (change in thickness) at the corresponding period of immersion in Method A or the value of $\frac{\triangle W}{S}$ (change in weight) er unit surface in Method B and the corresponding period of mersion;

erature of test (*see* **7.1**);

c) Description of the test liquid, used for immersion including (in the case of mineral oils other than standard test liquids recommended in Table 1 in Appendix A) the density, refractive index, and the aniline point oraromatic content;

d) Initial thickness of the test piece;

e) Temperature of conditioning (*see* **6.1**); and

f) Any discolouration of the test liquid, used for immersion or formation of sediment, at the conclusion of the test.

SECTION 2 DETERMINATION OF SOLUBLE MATTER EXTRACTED BY LIQUID

9. OUTLINE OF THE METHOD

9.1 If the test liquid used for immersion is readily volatile the amount of matter which it extracts from the rubber can be determined either (a) by drying the treated rubber and comparing its weight with the weight before immersion or (b) evaporating to dryness the liquid used in the test and weighing the nonvolatile residue. Both methods are liable to error. In Method (a) the rubber may have oxidized if air is present during immersion, especially if this is at high temperature; in method (b) there may be some loss of volatile extracted material, especially plasticizers. Both methods are described in this section and the choice between them should depend on the nature of the rubber and the conditions of test. It is difficult to define precisely what is meant by a 'readily volatile' liquid, but it may be suggested that the procedures described are not suitable for liquids less volatile than test liquids A and B given in **A-1.1** that is, for liquids boiling above 110°C.

10. TEST METHODS

10.1 Test Piece — The determination of soluble matter is made after the determination of change in volume or dimensions by any one of the methods described in Section 1 and hence same test pieces described, therein shall be used.

10.2 Procedure

10.2.1 *By Weighing Dried Rubber* — Dry the treated test piece in a vacuum of less than 40 mm of mercury at approximately 40°C to constant weight.

10.2.2 *By Evaporating the Test Liquid* — Transfer the liquid in which the rubber has been immersed to a suitable vessel; wash the rubber with 25 ml of fresh liquid; and then transfer to the same vessel. Evaporate the liquid, dry the residue to constant weight in vacuum and weigh.

10.3 Expression of Results — The weight of matter extracted is given by (a) the difference between the original weight of the test piece and its weight after treatment and drying in **10.2.1** or (b) the weight of the dried residue in **10.2.2**. Express the weight in either cases as percentage of the original weight of the test piece.

SECTION 3 MECHANICAL PROPERTIES ON SWOLLEN RUBBER

11. TENSILE STRESS-STRAIN TESTS

11.1 Apparatus — The apparatus for immersion shall be as in **8.1.1**. Tensile test apparatus shall be as prescribed in IS : *3400* (Part I)-*1965**.

11.2 Test Piece — The test piece shall be either a ring or a dumbbell as prescribed in IS : *3400* (Part I)-*1965**.

11.3 Procedure — Measure the cross-section of the test piece as in IS: 3400 (Part I)-1965*. Then immerse the test piece in the test liquid as described in **8.1.4**, select the temperature in accordance with the considerations given in **7.1** and the period of immersion sufficient to reach the maximum absorption, point *A* in Fig. 1 (*see* **8.1.3**). If immersion is at an elevated temperature, cool the test piece to a laboratory temperature 27° ± 1°C by immersing for 30 to 60 minutes in a fresh portion of the liquid at that temperature. Remove any surplus test liquid from the surface of the test piece. The method of removing the surplus liquid shall vary with the nature of the liquid. When mobile volatile liquids, such as *iso*octane and benzene are used, remove and quickly wipe the test piece with a filter paper or piece of fabric, which does not deposit lint. Some difficulty may be experienced in completely removing viscous nonvolatile oils by this method and it may be necessary to dip the test piece quickly in a suitable volatile liquid, such as acetone and again quickly wipe with filter paper or a piece of textile which does not deposit lint. In the case of dumbbells apply gauge marks as in IS : 3400 (Part I)-1965*. Carry out the tensile test at the standard laboratory temperature within an interval of two or three minutes after final removal from the test liquid, used for immersion.

11.4 Expression of Results — Record the following:

a) Type of test piece.

b) Description of the test liquid, used for immersion including (in the case of mineral oils other than standard test liquids No. 1, 2 and 3 as recommended in Table 1 in Appendix A) the density, refractive index, and the aniline point or aromatic content.

c) Period and temperature of immersion.

d) Tensile strength expressed per unit area of original cross-section before immersion of the test piece.

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*Methods of tests for vulcanized rubbers : Part I Tensile stress-strain properties.

e) Breaking elongation, expressed as percentage of gauge length (for dumbbell test piece) or percentage increase in the internal circumference of the treating ring test piece.

f) The modulus calculated on the original cross-section before immersion of the test piece.

NOTE — The elongation at which modulus is measured is that of the treated rubber.

g) Temperature at which tensile strength is determined.

12. HARDNESS

12.1 Apparatus — Apparatus for immersion shall be as in **8.1.1.** Apparatus for measuring thickness shall be as in **8.2.1.2.** Apparatus for measuring hardness shall be as prescribed in IS : 3400 (Part II)-1965*.

12.2 Test Piece — The test piece shall be either (a) or (b) as follows:

a) For use with the normal hardness test described in IS : 3400 (Part II)-1965* two pieces each 2·5 to 3 mm thick and of lateral dimensions at least 20 mm, for example, as described in **8.1.2** or **8.2.2** to form the test piece.

b) For use with the microtest a piece with flat parallel faces between 1·5 and 2·5 mm thick and not less than 8 mm lateral dimensions.

12.3 Procedure — Measure the thickness of the test piece at four point and take average of the results. Measure the hardness of the test piece in IS : 3400 (Part II)-1965*. Immerse the rubber in the test liquid as described in **8.1.4**. Select the temperature in accordance with the considerations given in **7.1**, and the period of immersion sufficient to reach the maximum absorption, point A in Fig. 1 (*see* **8.1.3**). If immersion is carried out at an elevated temperature, cool the test piece to 27°±1°C by immersion in a fresh portion of the liquid for not less than 30 minutes and not more than 60 minutes at that temperature. Remove any surplus test liquid from the surface of the test piece. The method of removing the surplus liquid from the surface of the test piece will vary with the nature of the liquid. When mobile volatile liquids, such as *iso*octane and benzene are used, remove and quickly wipe the test piece with a filter paper or piece of fabric which does not deposit lint. Some difficulty may be experienced in completely removing viscous nonvolatile oils by this method and it may be necessary to dip the test piece quickly in a suitable volatile liquid, such as acetone and again quickly wipe with filter paper or a piece of fabric which does not deposit lint. Then measure the hardness as described in IS : 3400 (Part II)-1965* at 27° ± 1°C within an interval of 2-3 minutes after final removal from the test liquid.

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*Methods of test for vulcanized rubbers : Part II Hardness.

12.4 Expression of Results — Record the following:

a) Nature of test piece (**12.2**);
b) *Description of the test liquid, used for immersion, including* (in the case of mineral oils other than standard test liquids No. 1, 2 and 3 recommended in Table 1 in Appendix A) the density, refractive index, and the aniline point or aromatic content;
c) *Period and temperature of immersion;*
d) The hardness in IRHD of the original test piece and of the treated test piece; and
e) Temperature at which hardness is measured.

NOTE — It will not usually be possible to have the same thickness of test piece before and after immersion since the immersion will change the thickness. An increase will make the hardness reading slightly lower and hence not strictly comparable with the reading taken before immersion. However, with *test piece of the thickness recommended provided the change in volume does* not exceed a 200 percent increase, the resulting error will not exceed about 2 IRHD.

SECTION 4 MECHANICAL PROPERTIES AFTER IMMERSION AND DRYING

13. TENSILE STRESS-STRAIN TEST

13.1 Apparatus — Apparatus for immersion shall be as in Section **8.1.1**. Tensile test apparatus shall be as described in IS : 3400 (Part I)-1965*.

13.2 Test Piece — The test piece shall be either a ring or a dumbbell as prescribed in *IS : 3400* (Part I)*-1965**.

13.3 Procedure — Follow the procedure as in **11.3** up to and including the immersion treatment. Dry the treated test piece in vacuum of less than 150 mm mercury at approximately 40°C to constant weight, cool to room temperature and condition by keeping at 27° ± 1°C for not less than 12 hours. Then make the test piece (in the case of dumbbells) and test at 27° ± 1°C as described in IS : 3400 (Part I)-1965*.

13.4 Expression of Results — Record the following:

a) The type of test piece (ring or dumbbell);
b) Description of the test liquid, used for immersion, including (*in the case of mineral oils other than standard test liquids No. 1,* 2 and 3 as recommended in Table 1) the density, refractive index, and the aniline point or aromatic content;
c) Period and temperature of immersion;
d) Tensile strength expressed per unit area of the original cross-section before immersion;

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*Methods of test for vulcanized rubbers : Part I Tensile stress-strain properties.

e) Breaking elongation expressed as percentage increase of gauge length (for dumbbell test piece) or percentage increase in the internal circumference of the dried ring test piece;

f) Modulus calculated on the original cross-section before immersion in liquid; and

g) Temperature at which tensile strength is determined.

> NOTE — The elongation at which modulus is measured is that of the dried rubber.

14. HARDNESS

14.1 Apparatus — Apparatus for immersion shall be as in **8.1.1**. Apparatus for measuring thickness shall be as prescribed in **8.2.1.2**. Apparatus for hardness test shall be as prescribed in IS : 3400 (Part II)-1965*.

14.2 Test Piece — Test piece shall be as prescribed in **11.2**.

14.3 Procedure — Follow the procedure as in **12.3** up to and including the immersion treatment. Then dry treated test piece in a vacuum of less than 150 mm mercury at approximately 40°C to constant weight, cool to room temperature and condition by keeping at 27° ± 1°C for at least 12 hours. Re-measure the thickness of the test piece and measure its hardness as in IS : 3400 (Part II)-1965*.

14.4 Expression of Results — Record the following:

a) Thickness of test piece (1) before immersion, and (2) after drying;

b) Description of the test liquid, used for immersion including (in the case of mineral oils other than standard test liquids No. 1, 2 and 3 as recommended, in Table 1 in Appendix A) the density, refractive index, and the aniline point or aromatic content;

c) Period and temperature of immersion;

d) Hardness in IRHD (1) before immersion, and (2) after drying;

> NOTE — Since the thickness after immersion and drying will usually be less than the original thickness, owing to extraction of soluble matter, this will tend to increase the apparent hardness of the dried rubber. However, except when, large proportions of plasticizer are extracted this effect will probably be negligible.

e) Form of hardness test used; and

f) Temperature at which hardness is measured.

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*Methods of test for vulcanized rubbers : Part II Hardness.

APPENDIX A

(*Clause* 5.4)

RECOMMENDED STANDARD TEST LIQUID FOR IMMERSION OF TEST PIECE

A-1. STANDARD TEST LIQUIDS

A-1.1 The following liquids may be used as standard test liquids for classification of vulcanized rubber or for quality control:

a) Liquid A — 2, 2, 4-trimethylpentane (*iso*octane)

b) Liquid B — 2, 2, 4-trimethylpentane, 70 percent by volume; toluene, 30 percent by volume

c) Liquid C — 2, 2, 4-trimethylpentane, 50 percent by volume; toluene, 50 percent by volume

d) Oil No. 1 — Low volume increase oil as defined in Table 1

e) Oil No. 2 — Medium volume increase oil as defined in Table 1

f) Oil No. 3 — High volume increase oil as defined in Table 1

TABLE 1 STANDARD TEST LIQUIDS (OILS)

CHARACTERISTIC (1)	OIL No. 1 (2)	OIL No. 2 (3)	OIL No. 3 (4)
Aniline point, °C	123·9 ± 1	93 ± 3	69·5 ± 1
Kinematic viscosity, m^2/sec	$19{\cdot}5\times10^{-6}\pm2\times10^{-6}$*	$20\times10^{-6}\pm2\times10^{-6}$*	$32\times10^{-6}\pm10^{-6}$†
Flash point, °C	244 *Min*	246 ± 6	166 ± 3

*Measured at 99°C

†Measured at 38°C