

International Standard



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Rubber, vulcanized — Determination of the effect of liquids

Caoutchouc vulcanisé — Détermination de l'action des liquides

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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Rubber, vulcanized — Determination of the effect of liquids

0 Introduction

The action of a liquid on a vulcanized rubber may generally result in

- a) absorption of liquid by the rubber;
- b) extraction of soluble constituents from the rubber;
- c) chemical reaction with the rubber.

The amount of absorption [a)] is usually greater than that of extraction [b)] so that the net result is an increase in volume, commonly termed "swelling". The absorption of liquid can profoundly alter physical and chemical properties and hence change tensile strength, extensibility and hardness, of the rubber, so that it is important to measure these properties of the rubber after treatment. The extraction of soluble constituents, especially plasticizers and antidegradants, can likewise alter the physical properties and chemical resistance shown by the rubber after drying out the liquid (assuming this to be volatile); physical tests on the rubber after immersion and drying are therefore required. The methods described in this International Standard accordingly comprise determinations of:

- change in volume, dimensions and mass;
- extracted soluble matter;
- tensile stress-strain properties of the rubber after immersion;
- hardness of the rubber after immersion;
- tensile stress-strain properties of the rubber after drying out the test liquid;
- hardness of the rubber after drying out the test liquid.

Although, in some respects, these tests may closely simulate service conditions, no direct correlation with service behaviour is implied; thus, the rubber giving the lowest change in volume is not necessarily the best in service. The thickness of the rubber must be taken into account since the rate of penetration of liquid is time-dependent and the bulk of a very thick rubber product may remain unaffected for the whole of the projected service life. It is known, moreover, that the action of a liquid on rubber, especially at high temperature, can be markedly affected by the presence of atmospheric oxygen. The tests described in this International Standard, 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.

1 Scope and field of application

This International Standard describes methods of evaluating the resistance of vulcanized rubbers to the action of liquids by measurement of properties of the rubbers before and after immersion in selected test liquids. The liquids considered include petroleum derivatives, organic solvents and chemical reagents.

2 References

ISO 37, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 48, *Vulcanized rubbers — Determination of hardness (Hardness between 30 and 85 IRHD).*

ISO 175, *Plastics — Determination of the effects of liquid chemicals, including water.*

ISO 471, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1826, *Rubber, vulcanized — Time-interval between vulcanization and testing — Specification.*

ISO 2592, *Petroleum products — Determination of flash and fire points — Cleveland open cup method.*

ISO 2977, *Petroleum products and hydrocarbon solvents — Determination of aniline point and mixed aniline point.*

ISO 3104, *Petroleum products — Transparent and opaque liquids — Determination of kinematic viscosity and calculation of dynamic viscosity.*

ISO 3675, *Crude petroleum and liquid petroleum products — Laboratory determination of density or relative density — Hydrometer method.*

ISO 4661, *Rubber — Preparation of test pieces.*

ISO 5282, *Aromatic hydrocarbons — Determination of sulfur content — Pitt-Ruprecht reduction and spectrophotometric method.*

ISO 5661, *Petroleum products — Hydrocarbon liquids — Determination of refractive index.*

3 Test liquids

3.1 The selection of the test liquid depends on the purpose of the test.

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

Mineral oils and fuels are liable to vary appreciably in chemical composition 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, but aniline point alone is not sufficient to characterize a mineral oil; other things being equal, the lower the aniline point the more severe is the action. If a mineral oil is used as the test liquid, therefore, the test report should include the density, refractive index, viscosity and the aniline point or aromatic content of the oil. For the standard mineral oils described in the annex, mineral oil raffinates are employed. Service oils, having similar fluid characteristics to the standard oils (see the annex, clauses A.1 to A.3), will not necessarily have the same effect on the material as the latter. Some fuels, particularly gasoline, vary widely in composition and, in the case of some possible constituents, minor variations can have a large influence on the effect on rubber. Full details of the composition of the fuel used should therefore be included in the test report.

3.3 As commercial liquids may not have an entirely constant composition, a standard immersion liquid consisting of well defined chemical compounds or mixtures of compounds should be used for the purposes of classification of vulcanized rubbers or quality control; some suitable liquids are recommended in the annex.

When testing to determine the effect of chemical solutions, the concentration of the solutions should be appropriate to the proposed application.

4 Time-interval between vulcanization and testing

Unless otherwise specified for technical reasons, the following requirements, in accordance with ISO 1826 for time-intervals, shall be observed.

4.1 For all test purposes, the minimum time between vulcanization and testing shall be 16 h.

4.2 For non-product tests, the maximum time between vulcanization and testing shall be four weeks and, for evaluations intended to be comparable, the tests, as far as possible, should be carried out after the same time-interval.

4.3 For product tests, whenever possible, the time between vulcanization and testing shall not exceed three months. In other cases, tests shall be made within two months of the date of receipt of the product by the customer.

5 Conditioning of test pieces

Test pieces of rubber required for test in the "as received" condition shall be conditioned for not less than 3 h in one of the standard laboratory temperatures specified in ISO 471. The same temperatures shall be used throughout any one test or any series of tests intended to be comparable.

6 Temperature of immersion

The immersion should preferably be carried out at one or more of the following standard temperatures listed in ISO 471:

-70 ± 1 , -55 ± 1 , -40 ± 1 , -25 ± 1 , -10 ± 1 , 0 ± 1 °C
 20 ± 2 , 23 ± 2 , 27 ± 2 °C
 40 ± 1 , 55 ± 1 , 70 ± 1 , 85 ± 1 , 100 ± 1 °C
 125 ± 2 , 150 ± 2 , 175 ± 2 , 200 ± 2 , 225 ± 2 , 250 ± 2 °C

As elevated temperatures greatly increase oxidation of the rubber, volatilization or decomposition of the immersion liquid, and also the effects of any chemically active additives in the liquid (for example in "service liquids"), appropriate selection of the test temperature and of the degree of access of air is very important.

In tests intended to simulate service conditions, and using the actual liquid with which the rubber will be used, the test conditions should approximate to those found in service using the lowest standard temperature equal to or higher than the service temperature.

7 Period of immersion

Since the rate of penetration of liquids into rubbers depends on the particular material, liquid and temperature, the adoption of one standard period of immersion is precluded. For acceptance purposes, it is recommended that determinations should be made and recorded after several periods of immersion, so as to indicate the change of volume, mass or dimensions with time; the total period should, if possible, extend well beyond the point of maximum absorption. For control purposes, it may not be necessary to reach maximum absorption; in such cases, a single period of immersion may suffice and one of the following should then be used:

$22 \pm 0,25$ h $72 \pm \frac{0}{2}$ h
 7 days ± 2 h multiples of 7 days ± 2 h.

In tests for change in volume, mass or dimensions, in which the period of immersion used is insufficient for maximum absorption to be reached, the test piece shall be of substantially constant thickness (see note 2 to 8.2.2).

In tests for change in physical properties, it is preferable to use a period of immersion sufficient to allow equilibrium to be reached.

NOTE — Since the amount of liquid absorbed is initially proportional to the square root of time rather than time itself, it is advisable to assess the time to "maximum absorption" by plotting the amount absorbed against the square root of time.

8 Determination of change in volume, change in mass or change in dimensions

8.1 General

Three methods for studying the resistance of rubber to liquids are described, i.e.

- a) volumetric method;
- b) gravimetric method;
- c) dimensional change methods.

The volumetric method is more precise than the dimensional change method and is the preferred method when it is required to know the change in volume due to immersion. The dimensional change method is useful for detecting anisotropy in the rubber, and should be used when it is required to determine changes in linear dimensions because these cannot always be calculated from the volume change owing to anisotropy in the rubber. The gravimetric method can be used as a quality control test and may be combined with the volumetric method, much of the procedure being common to both methods.

8.2 Volumetric and gravimetric methods

8.2.1 Apparatus

The apparatus to be used is determined by the temperature of immersion and the volatility of the test liquid. For tests at temperatures appreciably below the boiling point of the test liquid, a stoppered glass bottle or tube should be used, of such dimensions that the test pieces remain completely immersed in the specified volume of test liquid and are freely exposed on all surfaces without restraint. For tests at temperatures near the boiling point of the test liquid, the bottle or tube should be fitted with a reflux condenser or other suitable means of minimizing evaporation of the test liquid.

The balance to be used for weighing the test pieces shall be accurate to 1 mg.

8.2.2 Test piece

The test piece should be 1 to 3 cm³ in volume with a uniform thickness of $2 \pm 0,2$ mm. Test pieces cut from sheet or products may be any convenient shape.

NOTES

1 Test pieces cut from finished products may be used, including test pieces from finished products thinner than 1,8 mm; products, thicker than 2,2 mm should, however, be reduced to a thickness of $2 \pm 0,2$ mm.

The method of preparation should be in accordance with ISO 4661.

2 In tests where the maximum absorption is not reached, a smaller thickness tolerance of $\pm 0,1$ mm should be used as the percentage volume change during the early stages of absorption is inversely proportional to the test piece thickness.

8.2.3 Procedure

8.2.3.1 Use three test pieces. Weigh each test piece in air to the nearest milligram (mass m_1), and then reweigh each test piece in distilled water at the standard laboratory temperature (mass m_2) (see note 1 to 8.2.4), taking care to ensure that all air bubbles are removed (see note 2 to 8.2.4). If the density of the material is less than 1 g/cm³, it will be necessary to use a sinker when weighing in water to ensure that the test pieces are completely immersed. If a sinker is used, the mass of the sinker alone in distilled water shall be determined separately (mass m_5). Blot the test pieces dry with filter paper or a textile fabric that does not deposit lint.

8.2.3.2 Place the test pieces, suitably separated, in a glass container with a volume of the test liquid that is at least 15 times the combined volume of the test pieces and sufficient to keep them totally immersed. If the conditions of test do not necessitate the use of a reflux condenser, the container should be stoppered. The test liquid should be kept at the test temperature, and the rubber test pieces should be shielded from light during the test.

Only test pieces of the same vulcanizate should be placed in any one container. If the density of the rubber is less than that of the liquid, means should be provided for holding the test pieces completely below the surface of the liquid.

At the end of the period of immersion, bring the test pieces, if necessary, to the standard laboratory temperature, preferably by quickly transferring them to a fresh portion of the test liquid at this temperature for a period of not less than 10 min and not more than 30 min. Surplus test liquid should be removed from the surfaces of each test piece (see note 3 to 8.2.4).

8.2.3.3 Immediately determine the mass of each test piece in air (mass m_3) to the nearest milligram, and then weigh them in distilled water (mass m_4) at the standard laboratory temperature. If the test liquid is appreciably volatile at room temperature, an evaporation curve should be plotted for the test pieces freely suspended on the balance arm. A plot of mass versus the square root of time will give a straight line. This should be extrapolated to zero time which shall be taken as the time at which the test piece is removed from the liquid or is free of surplus liquid. The mass of the test piece should be determined at 20 or 30 s intervals for not more than 2 min; this is easily possible with an automatic balance. The test piece should be returned immediately to the test liquid for 30 to 60 min before carrying out the weighing in distilled water. Alternatively, a weighing bottle method may be used in which the time for transfer of the test piece shall not exceed 30 s after removal from the liquid.

The weighings in distilled water (m_2 and m_4) and of the sinker (m_5) may be omitted if change in mass only is to be determined.

If the test is being continued, replace the test pieces immediately in the test liquid and return to the thermostatically controlled oven or bath.

8.2.4 Expression of results

Calculate the percentage change in volume ΔV_{100} and/or percentage change in mass Δm_{100} by means of the formulae

$$\Delta V_{100} = \frac{(m_3 - m_4 + m_5) - (m_1 - m_2 + m_5)}{(m_1 - m_2 + m_5)} \times 100$$

$$\Delta m_{100} = \frac{m_3 - m_1}{m_1} \times 100$$

where

m_1 is the initial mass of the test piece in air;

m_2 is the initial apparent mass of the test piece (plus the sinker if used) in water;

m_3 is the mass of the test piece in air after immersion;

m_4 is the apparent mass of the test piece (plus the sinker if used) in water after immersion;

m_5 is the apparent mass of the sinker in water.

Take as the result the average of the values obtained for the three test pieces.

NOTES

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, the masses m_2 , m_4 and m_5 may be determined in the test liquid instead of water and these values used in the formula for calculating the percentage change in volume; masses m_1 and m_3 should, in this case, be determined in a fresh portion of the immersion liquid.

If this is not practicable, the same procedure should be used as for the water displacement method, except that the final weighing in water is omitted and the percentage change in volume is calculated from the formula:

$$\Delta V_{100} = \frac{(m_3 - m_1)}{\rho(m_1 - m_2 + m_5)} \times 100$$

where ρ is the density of the test liquid at the standard laboratory temperature; this formula may be only approximate if the test liquid is a mixture, because the density of the absorbed liquid may differ from that of the bulk. Also the density of any matter extracted from the rubber may differ from that of the test liquid.

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

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 iso-octane and toluene, are used, remove and quickly wipe the test piece with a filter paper or piece of textile fabric which does not deposit lint. Some difficulty may be experienced in completely removing viscous non-volatile oils by this method and it may be necessary to dip the test piece quickly in a suitable volatile liquid, such as methanol or petroleum ether, and again quickly wipe with filter paper or a piece of textile fabric which does not deposit lint.

8.3 Dimensional change methods

8.3.1 Methods for determining changes in length, width and thickness

8.3.1.1 Apparatus

The apparatus for immersion of the test pieces shall be as described in 8.2.1.

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 area approximately 100 mm² which should be normal to the plunger and parallel to the base-plate. The dial-gauge shall operate to give a pressure on the rubber of approximately 2 kPa.

The instrument for measuring the length and width of the test pieces shall have a scale graduated in unit divisions of 0.01 mm and should preferably operate without contact with the test piece, for example an optical system.

8.3.1.2 Test piece

The test piece shall be rectangular, 50 mm long and 25 mm wide. The thickness should 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 should be cut with its long axis parallel to the grain.

NOTE — Test pieces cut from finished products may be used, including test pieces from finished products thinner than 1.8 mm; products thicker than 2.2 mm should, however, be reduced to a thickness of 2 ± 0.2 mm.

8.3.1.3 Procedure

Use three test pieces. Measure the initial length of each test piece along its centre line to the nearest 0.5 mm (measurements shall be taken along the top and bottom surfaces and the two results averaged). Similarly measure the initial width but taking four measurements in all, one on top and one on the 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. All measurements shall be made with the test piece at the standard laboratory temperature.

Follow the procedure described in 8.2.3.2.

Re-measure the length, width and thickness of each test piece as described above, with the test pieces at the standard laboratory temperature.

If the test liquid is appreciably volatile at room temperature, the measurements should be completed within 1 min of removing the test pieces from the test liquid.

If the test is being continued, replace the test pieces immediately in the liquid and return to the thermostatically controlled oven or bath.

8.3.1.4 Expression of results

Calculate the percentage change in length Δl_{100} by means of the formula

$$\Delta l_{100} = \frac{l - l_0}{l_0} \times 100$$

where

l_0 is the initial length;

l is the length after immersion.

Calculate the percentage changes in width and thickness similarly.

Take as the results the average of the values obtained for the three test pieces.

8.3.2 Method for determining change in surface area

8.3.2.1 Apparatus

The apparatus for immersion of the test pieces shall be as described in 8.2.1.

The instrument for measuring the lengths of the diagonal of the test pieces shall have a scale graduated in unit divisions of 0,01 mm and should preferably operate without contact with the test piece, for example an optical system.

8.3.2.2 Test piece

The test piece shall be rhomboid, with the sides cut cleanly and at right angles to the top and bottom surfaces. This may be achieved by two consecutive cuts at approximately right angles to each other, with a cutter consisting of two parallel blades suitably spaced. The thickness of the test piece shall normally be $2 \pm 0,2$ mm and the length of the sides 8 mm. It may be convenient to use thinner test pieces, for example when cut from products or when rapid attainment of equilibrium is required, but the results may not be comparable with those obtained using the specified thickness. Smaller test pieces will reduce the precision of the results.

8.3.2.3 Procedure

Use three test pieces. Measure the initial lengths of the diagonals of each test piece to the nearest 0,01 mm at the standard laboratory temperature.

Follow the procedure described in 8.2.3.2.

Remeasure the lengths of the diagonals as described above. If an optical measuring system is used, this may be done in a suitable glass container without removing the test pieces from the test liquid.

If the test is being continued, replace the test pieces immediately in the liquid and return to the thermostatically controlled oven or bath.

8.3.2.4 Expression of results

Calculate the percentage change in area ΔA_{100} by means of the formula

$$\Delta A_{100} = 100 \left[\frac{l_A l_B}{l_a l_b} - 1 \right]$$

where

l_A and l_B are the lengths of the diagonals after immersion;

l_a and l_b are the lengths of the diagonals before immersion.

If required, the percentage volume change ΔV_{100} may be calculated from the formula

$$\Delta V_{100} = 100 \left[\left(\frac{l_A l_B}{l_a l_b} \right)^{3/2} - 1 \right]$$

NOTE — The formula for percentage volume change assumes isotropic swelling. If doubt exists, the percentage volume change should be determined by the volumetric method which is the preferred method.

9 Determination of extracted soluble matter

9.1 Introduction

If the test liquid is readily volatile, the amount of matter which it extracts from the test piece can be determined either:

- by drying the treated test piece and comparing its mass with the mass before immersion; or
- evaporating the test liquid to dryness and weighing the non-volatile residue.

Both methods are liable to errors. In the method by weighing the dried test piece, the material may have oxidized if air is present during immersion, especially if this is at high temperature. In the method by evaporating the test liquid, there may be some loss of volatile extracted material, especially plasticizers. Both methods are described in this International Standard and the choice between them must depend on the nature of the material 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 standard liquids A, B, C, D and E in the annex, i.e. for liquids boiling above 110 °C.

9.2 Test piece

The determination of soluble matter may be made after the determination of change in volume or dimensions by the methods described in 8.2 and 8.3 using the appropriate test piece described in 8.2.2 or 8.3.2.2.

9.3 Procedure

9.3.1 Method by weighing the dried test piece

Dry the test piece, after immersion, under an absolute air pressure of approximately 20 kPa at approximately 40 °C, to constant mass, that is, until the difference between successive weighings at 30 min intervals does not exceed 1 mg.

9.3.2 Method by evaporating the test liquid

Transfer the liquid in which the test piece has been immersed to a suitable vessel and wash the test piece with 25 ml of fresh liquid which should then be transferred to the same vessel. Evaporate the liquid and dry the residue to constant mass under an absolute air pressure of approximately 20 kPa at approximately 40 °C.

Carry out a blank test to estimate the solids content of a volume of the test liquid equal to that used for the immersion and washing.

9.4 Expression of results

According to the method used, the mass of extracted soluble matter is equal to

- a) in the case of the method by weighing the dried test piece: the difference between the original mass of the test piece and its mass after immersion and drying;
- b) in the case of the method by evaporating the test liquid: the mass of the dried residue, corrected for the result of the blank test on the test liquid.

In both cases, express the mass as a percentage of the original mass of the test piece.

10 Determination of change in physical properties after immersion

10.1 Tensile stress-strain tests

10.1.1 Apparatus

10.1.1.1 Apparatus for immersion, as described in 8.2.1.

10.1.1.2 Tensile test apparatus, as described in ISO 37.

10.1.2 Test piece

The test piece shall be either a ring or a dumb-bell as described in ISO 37.

10.1.3 Procedure

Measure the cross section of the test piece as described in ISO 37. Follow the procedure described in 8.2.3.2. (The immersion temperature should be selected in accordance with the considerations given in clause 6 and the period of immersion should preferably be sufficient to reach maximum absorption.)

In the case of dumb-bells, gauge marks should be applied on the swollen test piece as described in ISO 37. In the case of rings, measure the internal diameters by means of a suitable cone gauge. Carry out the tensile test at the standard laboratory temperature within 3 min of final removal from the immersion liquid. Tests should be carried out on unimmersed test pieces for comparison.

10.1.4 Expression of results

Calculate the tensile strength per unit area of the original cross-section of the test piece before immersion and report the result as the change expressed as a percentage of the value for unimmersed material.

Calculate the elongation at break as a percentage of the gauge length for dumb-bell test pieces after immersion, or as a percentage increase in the internal circumference of the ring test piece after immersion, and report the result as the change expressed as a percentage of the value for unimmersed material.

Calculate the stress at a given elongation on the original cross-section before immersion of the test piece, and report the result as the change expressed as a percentage of the value for unimmersed material (note, however, that the elongation at which modulus is measured is that of the rubber after immersion).

10.2 Hardness tests

10.2.1 General

The hardness shall be measured using the micro-test described in ISO 48. Alternatively, the normal hardness test described in ISO 48 may be used with two plied-up test pieces (see 10.2.3) in which case the result shall be expressed as apparent hardness.

10.2.2 Apparatus

10.2.2.1 Apparatus for immersion, as described in 8.2.1.

10.2.2.2 Apparatus for measuring hardness, as described in ISO 48.

10.2.3 Test piece

The test piece shall have flat parallel faces, and shall be of uniform thickness of $2,0 \pm 0,2$ mm with other dimensions of not less than 8 mm.

10.2.4 Procedure

Measure the hardness of the test piece at a standard laboratory temperature as described in ISO 48. Then follow the procedure described in 8.2.3.2. (The immersion temperature should be selected in accordance with the considerations given in clause 6 and the period of immersion should preferably be sufficient to reach maximum absorption.)

Remeasure the hardness as described in ISO 48 at the standard laboratory temperature within 3 min of final removal from the test liquid.

10.2.5 Expression of results

Report the hardness before immersion in International Rubber Hardness Degrees (IRHD) as described in ISO 48. Report also the change in hardness as the difference between the value obtained after immersion and the value obtained before immersion.

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

11 Determination of change in physical properties after immersion and drying

11.1 Tensile stress-strain tests

11.1.1 Apparatus

11.1.1.1 Apparatus for immersion, as described in 8.2.1.

11.1.1.2 Tensile test apparatus, as described in ISO 37.

11.1.2 Test piece

The test piece shall be one of those described in ISO 37.

11.1.3 Procedure

Follow the procedure specified in 10.1.3 up to and including the immersion treatment. Then dry the test piece under an absolute air pressure of approximately 20 kPa at approximately 40 °C to constant mass, that is until the difference between successive weighings at 30 min intervals does not exceed 1 mg. Cool to room temperature and condition by keeping at the standard laboratory temperature for not less than 3 h. Determine the tensile properties by the procedure specified in ISO 37.

11.1.4 Expression of results

Calculate the tensile strength per unit area of the original cross-section of the test piece before immersion, and report the result as the change expressed as a percentage of the value for un-immersed material.

Calculate the elongation at break as a percentage of the gauge length for dumb-bell test pieces after immersion and drying, or as a percentage increase in the internal circumference of the ring test piece after immersion and drying, and report the result as the change expressed as a percentage of the value for un-immersed material.

Calculate the stress at a given elongation on the original cross-section before immersion of the test piece, and report the result as the change expressed as a percentage of the value for un-immersed material (note, however, that the elongation at which modulus is measured is that of the rubber after immersion and drying).

11.2 Hardness tests

11.2.1 General

The hardness shall be measured using the micro-test described in ISO 48. Alternatively, the normal hardness test described in ISO 48 may be used with two plied-up test pieces (see 11.2.3) in which case the result shall be expressed as apparent hardness.

11.2.2 Apparatus

11.2.2.1 Apparatus for immersion, as described in 8.2.1.

11.2.2.2 Apparatus for measuring hardness, as described in ISO 48.

11.2.3 Test piece

The test piece shall have flat parallel faces, and shall be of uniform thickness of $2,0 \pm 0,2$ mm with other dimensions of not less than 8 mm.

11.2.4 Procedure

Follow the procedure specified in 10.2.3 up to and including the immersion treatment. Then dry the test piece under an air pressure of approximately 20 kPa at approximately 40 °C to constant mass, that is until the difference between successive weighings at 30 min intervals does not exceed 1 mg. Cool to room temperature and condition by keeping at the standard laboratory temperature for not less than 3 h.

Remeasure the hardness at the standard laboratory temperature as described in ISO 48.

11.2.5 Expression of results

Report the hardness before immersion in International Rubber Hardness Degree (IRHD) as described in ISO 48. Report also the change in hardness as the difference between the value obtained after immersion and drying and the value obtained before immersion.

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.

12 Test with liquid on one surface only

12.1 Applicability

This test is applicable to relatively thin sheet materials, for example rubber diaphragms, that are exposed to liquid on one surface only.

12.2 Apparatus

The apparatus shall be suitable for holding the test piece. Suitable apparatus is illustrated in the figure and comprises a base-plate (A) and an open-ended cylindrical chamber (B) which is held tightly against the test piece (C) by the wing nuts (D) mounted on the bolts (E). A hole of diameter approximately 30 mm may be made in the base-plate for the examination of the surface not in contact with the liquid. During the test, the opening in the top of the chamber should be closed by a close-fitting plug (F).

12.3 Test piece

The test piece shall consist of a disc of diameter about 60 mm and of the thickness of the material under test.

12.4 Procedure

Measure the nominal thickness of the test piece then weigh it in air to the nearest milligram (mass m_1). Then place the test piece in the apparatus as indicated in the figure. Fill the chamber of the apparatus with the test liquid to a depth of approximately 15 mm and insert the plug (F). Maintain the apparatus at the required temperature for the duration of the test.

At the end of the contact period, bring the apparatus, if necessary, to the standard laboratory temperature. Remove the

liquid and release the test piece. Any surplus liquid should be removed from the surface of the test piece by wiping with filter paper or a textile fabric which does not deposit lint (see note 3 to 8.2.4). Then weigh the test piece to the nearest milligram (mass m_2) at the standard laboratory temperature.

If the test liquid is appreciably volatile at room temperature, the measurement should be completed within 2 min of removal of the liquid (see note 3 of 8.2.4).

12.5 Expression of results

The change in mass per unit surface area Δm_A , expressed in grams per square metre, is given by the formula

$$\Delta m_A = \frac{m_2 - m_1}{A}$$

where

m_1 is the initial mass, in grams, of the test piece;

m_2 is the final mass, in grams, of the test piece;

A is the area, in square metres, of the circular surface of the test piece in contact with the test liquid.

Take as the result the average value for at least three test pieces.

Dimensions in millimetres

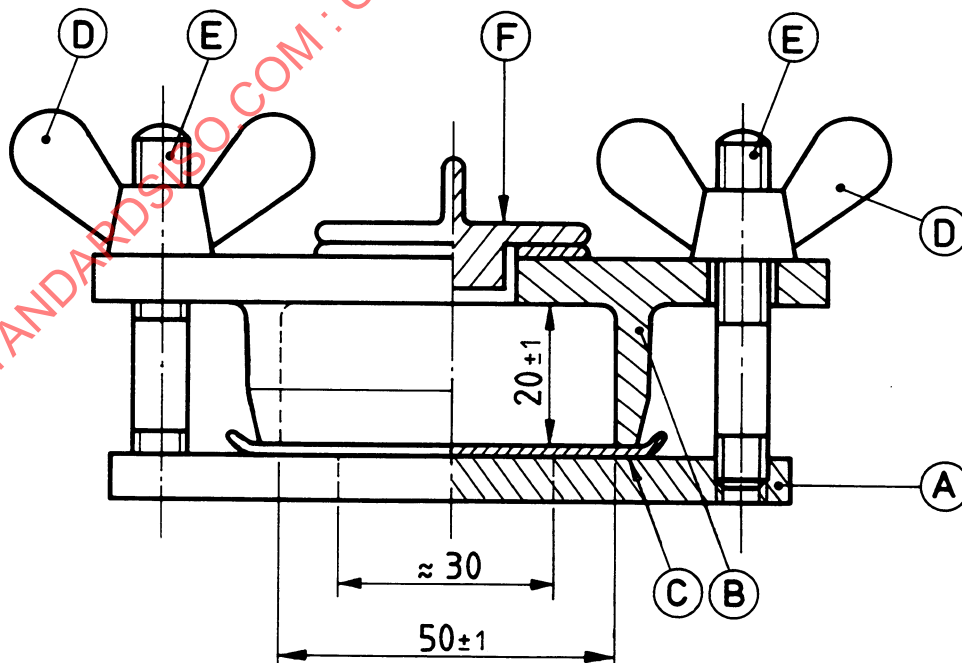


Figure — Apparatus for tests on one surface only

13 Test report

The test report shall include the following information :

a) sample details :

- 1) a full description of the sample and its origin,
- 2) compound details, cure time and temperature where appropriate,
- 3) the method of preparation of the test pieces from the sample, for example whether moulded or cut,
- 4) a full description of the test liquid; in the case of mineral oils (other than standard oils 1, 2 and 3), this should include the density, viscosity, refractive index and the aniline point or aromatic content ;

b) test method and test details :

- 1) the number of this International Standard,

- 2) the method(s) used,

- 3) the type of test piece used (dimensions),

- 4) the standard laboratory temperature adopted,

- 5) the period and temperature of immersion,

- 6) any deviation from the procedure specified ;

c) test results :

- 1) results expressed in the form stated in the relevant clauses,

- 2) note of the appearance of the test piece (for example cracking, delamination) if appropriate,

- 3) note of the appearance of the test liquid (for example discolouration, sedimentation) if appropriate;

d) date of test.

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Annex

Test liquids

(Forms an integral part of the standard.)

WARNING — Appropriate safety precautions should be taken when preparing and handling test liquids, especially those known to be toxic, corrosive or flammable. Products giving off fumes should be handled only under an efficiently ventilated hood, corrosive products should not be allowed to come into contact with the skin or ordinary clothing, and flammable products should be kept away from any source of ignition.

A.1 Standard simulated fuels

The liquids listed in table 1 should be used as standard fuels for the classification of vulcanized rubber or for quality control in respect of the behaviour of rubbers in hydrocarbon based liquids, for example petroleum-derived fuels.

Table 1

Liquid	Constituents	Content % (V/V)
A	2,2,4-trimethylpentane	100
B	2,2,4-trimethylpentane	70
	toluene	30
C	2,2,4-trimethylpentane	50
	toluene	50
D	2,2,4-trimethylpentane	60
	toluene	40
E	toluene	100
F	straight-chain paraffin (C ₁₂ to C ₁₈)	80
	1-methylnaphthalene	20

NOTES

1 Liquids B, C and D will afford a reasonable simulation of the effect on rubbers of the majority of motor gasolines currently available. The latter vary widely in composition, even within the same grade (i.e. knock-rating) and within the same location, and the use of all three liquids is necessary to cover this range of compositions.

2 Motor gasolines containing alcohol are currently available in a few geographical areas and the availability of such fuels is expected to become more widespread. The presence of a relatively small quantity of alcohol produces a disproportionate effect on some conventionally fuel-resistant rubbers. Hence, the use of a suitable test liquid containing alcohol is recommended to simulate such fuels. However, at present, no single standard test liquid can simulate the properties of this rapidly changing group of fuels. The liquids shown in table 2 have been used as test liquids and may serve as guide to the formulation of suitable test liquids.

Table 2

Liquid	Constituents	Content % (V/V)
1	2,2,4-trimethylpentane	30
	toluene	50
	di-isobutylene	15
	ethanol	5
2	2,2,4-trimethylpentane	25,35
	toluene	42,25
	di-isobutylene	12,68
	ethanol	4,22
	methanol	15,00
	water	0,50
3	2,2,4-trimethylpentane	45
	toluene	45
	ethanol	7
	methanol	3
4	2,2,4-trimethylpentane	42,5
	toluene	42,5
	methanol	15

Test liquids containing alcohol such as those above should not be used if the fuels involved are known to be free of alcohol.

3 Liquid F is intended to simulate diesel fuel, domestic heating oils and similar light furnace oils. A suitable test liquid is available commercially. Details may be obtained from the Secretariat of ISO/TC 45 (BSI) or ISO Central Secretariat.

4 Analytical reagent quality materials should be used in making up the various test liquids.

A.2 Standard oils

A.2.1 General descriptions

A.2.1.1 Oil No. 1

This is a "low volume increase" oil consisting of a closely controlled blend of mineral oils comprising a solvent extracted, chemically treated dewaxed paraffinic residuum and neutral oil.

A.2.1.2 Oil No. 2

This is a "medium volume increase" oil obtained by solvent extraction, acid and clay treatment of a high viscosity distillate from selected naphthenic (Gulf Coastal) crudes.

A.2.1.3 Oil No. 3

This is a "high volume increase" oil consisting of a closely controlled blend of two lubricating oil fractions obtained by vacuum distillation of selected naphthenic (Gulf Coastal) crudes.

NOTE — These oils are identical to ASTM Reference Oils No. 1, No. 2 and No. 3.