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VULCANIZED RUBBERS

METHODS OF TEST FOR RESISTANCE TO LIQUIDS

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BRIEF HISTORY

The ISO Recommendation R 1817, *Vulcanized rubbers – Methods of test for resistance to liquids,* was drawn up by Technical Committee ISO/TC 45, *Rubber,* the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1817, which was circulated to all the ISO Member Bodies for enquiry in March 1969. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Spain
Austria	India	Sweden
Brazil	Israel	Switzerland
Canada	Italy	Turkey
Ceylon	Netherlands	U.A.R.
Czechoslovakia	New Zealand	United Kingdom
France	Poland	U.S.A.
Germany	Romania	U.S.S.R.
Greece	South Africa, Rep. of	Yugoslavia

No Member Body opposed the approval of the Draft.

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

ISO Recommendation R 1817 March 1971

VULCANIZED RUBBERS

METHODS OF TEST FOR RESISTANCE TO LIQUIDS

INTRODUCTION

The action of a liquid on a vulcanized rubber generally results in

(*a*) absorption of liquid by the rubber;

(*b*) extraction of soluble constituents from the rubber;

(*c*) chemical reaction with 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) : physical tests on the rubber after immersion and drying are therefore required. The methods described below accordingly comprise determinations of

- change in volume or dimensions;
- soluble matter extracted;
- 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 immersion liquid;
- 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 temperatures, can be markedly affected by the presence of atmospheric oxygen. The tests 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.

1. SCOPE

This ISO Recommendation describes methods of test for 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.

2. TEST LIQUIDS

The selection of the test liquid depends on the purpose of the test :

2.1 When information is required on the *probable service behaviour* of a 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 rubber 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 oil based fluids 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, though 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. The report should 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. For the standardized mineral oils given in the Appendix, mineral oil raffinates are employed. Service liquids having similar fluid characteristics to standard liquids Nos.1 to 3 (see Appendix) will not necessarily have the same effect on rubber as the latter.

2.2 As commercial liquids may not have an entirely constant composition the test should, whenever possible, be made in well-defined chemical products, used alone or as mixtures. These should 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.

2.3 For use in systems of *classification of vulcanized rubbers*, or for quality control, a standard immersion liquid should be used; suitable liquids are recommended in the Appendix.

3. TIME LAPSE BETWEEN VULCANIZATION AND TESTING

Unless otherwise specified for technical reasons the following requirements for time lapses should be observed.

3.1 For all test purposes the minimum time between vulcanization and testing should be 16 hours.

3.2 For non-product tests the maximum time between vulcanization and testing should be 4 weeks and for evaluations intended to be comparable, the tests, as far as possible, should be carried out after the same time interval.

3.3 For product tests, whenever possible, the time between vulcanization and testing should not exceed 3 months. In other cases tests should be made within 2 months of the date of receipt by the customer of the product.

4. CONDITIONING OF TEST PIECES

Test pieces required for test in the "as received" condition should be conditioned for not less than 3 hours in one of the standard laboratory atmospheres of ISO Recommendation R 471, *Standard atmospheres for the conditioning and testing of rubber test pieces,* namely : 20 ± 2 °C, 65 ± 5 % relative humidity; 23 ± 2 °C, 50 ± 5 % relative humidity; or 27 ± 2 °C, 60 ± 5 % relative humidity. The same temperature should be used throughout any one test or any series of tests intended to be comparable.

5. TEMPERATURE OF TEST IMMERSION

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

− 75 ± 1,	− 55 ± 1,	− 40 ± 1,	− 25 ± 1,	− 10 ± 1,	0 ± 1 °C
20 ± 2,	23 ± 2,	27 ± 2 °C,			
40 ± 1,	50 ± 1,	70 ± 1,	85 ± 1,	100 ± 1,	125 ± 1 °C
150 ± 2,	175 ± 2,	200 ± 2,	225 ± 2,	250 ± 2 °C	

Whenever possible the temperature equal to or next more severe than that at which the rubber will be used should be selected. In other cases one of the standard laboratory temperatures given in section 4 should be adopted.

6. DURATION OF TEST IMMERSION

The dependence of the rate of penetration of liquids into rubbers on the particular rubber and liquid and the temperature, precludes the adoption of one standard period of immersion. For acceptance purposes it is recommended that determination should be made and recorded after several periods of immersion, so as to indicate the change of volume 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 immersion period may suffice and one of the following periods should then be used :

22 ± 0.25 hours,	70^{+2}_{0} hours
7 days ± 2 hours,	multiples of 7 days.

In the volumetric method described in clause 7.3, if the immersion period is insufficient to reach maximum absorption, the test piece should be of substantially constant thickness (see Note 2 to clause 7.3.2).

7. DETERMINATION OF CHANGE IN VOLUME OR CHANGE IN DIMENSIONS

7.1 Principle

The tests described in clauses 7.3 and 7.4 consist in immersing the test piece of rubber in a liquid for a given time, at a constant temperature, and determining the change in volume, or the changes in linear dimensions, respectively. In some instances the liquid may extract a proportion of the plasticizer or other soluble ingredient of the rubber, and this possibility must be borne in mind when interpreting the results.

The two methods described for studying resistance to liquid characteristics of rubber are as follows :

(*a*) volumetric method;

(*b*) dimensional-change method.

Method (*a*) is the more precise, and is the preferred method when it is required to know the change in volume due to immersion. Method (*b*) is not suitable for absolute measurement but is useful for detecting grain (anisotropy) in the rubber, and it 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 grain in the rubber.

7.2 Definitions

7.2.1 *Change in volume.* 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.

7.2.2 *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.

7.3 Volumetric method

7.3.1 *Apparatus.* This is determined by the temperature of the test and the volatility of the immersion liquid. For tests at temperatures appreciably below the boiling point of the 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 immersion liquid and are freely exposed at all surfaces without restraint. For tests at temperatures near the boiling point of the immersion liquid, the bottle or tube should be fitted with a reflux condenser or other suitable means of minimizing evaporation of the test liquid.

7.3.2 *Test piece.* The test piece should be 1 to 3 cm^3 in volume and of uniform thickness of 2 ± 0.2 mm. Test pieces cut from sheet may be of any convenient rectangular shape but should in no case be greater than 50 mm in length or breadth.

NOTES

1. Test pieces cut from finished products may be used. Test pieces from finished products thinner than 1.8 mm may be used; products thicker than 2.2 mm should be buffed to a thickness of 2 ± 0.2 mm.

2. In tests where the maximum absorption is not reached, a smaller thickness tolerance of ± 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.

7.3.3 *Procedure.* Use three test pieces. Weigh each test piece in air to the nearest milligramme (m_1), and then reweigh each test piece in distilled water at the standard laboratory temperature (m_2) (see Note 1 to clause 7.3.4), care being taken to ensure that all air bubbles are removed (see Note 2 to clause 7.3.4).

Blot the test pieces dry with filter paper or a textile fabric that does not deposit lint and then place them, suitably separated, in a glass container with a volume of the immersion liquid 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 a reflux condenser, the container should be stoppered. The container should be kept at the required temperature, and the rubber 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 immersion period, bring the test pieces, if necessary, to the standard laboratory temperature, preferably by quickly transferring them to a fresh portion of the immersion liquid at this temperature for a period of not less than 30 minutes and not more than 60 minutes. Surplus immersion liquid should be removed from the surfaces of each test piece (see Note 3 to clause 7.3.4). Then immediately place the test piece in a tared and stoppered weighing bottle and determine its mass in air (m_3) to the nearest milligramme. Remove the test piece from the bottle and immediately weigh in distilled water (m_4) at the standard laboratory temperature.

If the immersion liquid is appreciably volatile at room temperature, the time for each transference of the test piece, after removal from the immersion liquid, should not exceed 30 seconds.

7.3.4 *Expression of results.* The percentage change in volume should be calculated as follows :

$$\text{Percentage change in volume} = \frac{(m_3 - m_4) - (m_1 - m_2)}{(m_1 - m_2)} \times 100$$

where

m_1 is the initial mass of the rubber in air;

m_2 is the initial apparent mass of the rubber in water;

m_3 is the mass in air of the rubber after immersion;

m_4 is the apparent mass in water of the rubber after immersion.

If the test is being continued, the test pieces should at once be replaced in the immersion liquid and returned to the thermostatically controlled oven or bath.

The results for the three test pieces should be averaged.

NOTES

1. The above procedure may not be suitable if the immersion 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 m_2 and m_4 may be made in the immersion liquid instead of water and these values used in the formula for percentage change in volume given above; weighing m_4 should in this case be made 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 calculated from the formula :

$$\text{Percentage change in volume} = \frac{(m_3 - m_1)}{d\,(m_1 - m_2)} \times 100$$

   where d is the density of the immersion liquid at the standard laboratory temperature; this formula may be only approximate if the immersion 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 immersion 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 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 and again quickly wipe with filter paper or a piece of textile which does not deposit lint.

7.3.5 *Test report.* The test report should include the following information :

(*a*) the value(s) for the percentage change in volume, calculated as described in clause 7.3.4, and the corresponding period(s) of immersion;

(*b*) temperature of test;

(*c*) description of the immersion liquid; in the case of mineral oils (other than standard liquids Nos. 1, 2 and 3) this should include the density, refractive index, and the aniline point or aromatic content;

(*d*) initial thickness and dimensions of the test piece;

(*e*) temperature of conditioning;

(*f*) note of any discolouration of the immersion liquid, or formation of sediment, at the conclusion of the test;

(*g*) note of the appearance of the test piece (cracks, delamination, etc.).

7.4 Dimensional-change method

7.4.1 *Apparatus.* The apparatus for immersion of the test pieces should be as described in clause 7.3.1.

The instrument for measuring the thickness of the test pieces should consist of a micrometer dial-gauge of adequate accuracy firmly held in a rigid stand over a flat base-plate. The gauge should have a scale graduated in unit divisions of 0.01 mm. The plunger should be fitted with a flat circular contact of area of approximately 100 mm^2 which should be normal to the plunger and parallel to the base-plate. The dial-gauge should operate to give a pressure on the rubber of approximately 2 kN/m^2.

7.4.2 *Test piece.* The test piece should be rectangular, 50 mm long and 25 mm wide. The thickness should be uniform, preferably 2 ± 0.2 mm. The sides should 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. Test pieces from finished products thinner than 1.8 mm may be used; products thicker than 2.2 mm should be buffed to a thickness of 2 ± 0.2 mm.

7.4.3 *Procedure.* Use three test pieces. Measure the initial length of each test piece along its centre line to the nearest 0.5 mm; measurements should 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 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. All measurements should be made with the test piece at the standard laboratory temperature.

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

Only test pieces of the same vulcanizates 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 immersion period, bring the test pieces, if necessary, to the standard laboratory temperature, preferably by quickly transferring them to a fresh portion of the immersion liquid at this temperature for a period of not less than 30 minutes and not more than 60 minutes. Surplus immersion liquid should be removed from the surface of the test pieces by wiping with filter paper or a textile fabric which does not deposit lint. Then remeasure the length, width and thickness of each test piece as described above, with the test pieces at the standard laboratory temperature.

If the immersion liquid is appreciably volatile at room temperature the measurements should be completed within 1 minute of removing the test piece from the immersion liquid.

7.4.4 *Expression of results.* The percentage changes should be calculated as follows :

$$\text{Percentage change in length} = \frac{L - L_0}{L_0} \times 100$$

where

L_0 is the initial length;

L is the length after immersion.

Percentage change in width and percentage change in thickness should be calculated in a similar manner.

If the test is being continued, the test pieces should at once be replaced in the liquid and returned to the thermostatically controlled oven or bath.

The results for the three test pieces should be averaged.

7.4.5 *Test report.* The test report should include the following information :

(*a*) the values for percentage change in length, percentage change in width, and percentage change in thickness, calculated as described in clause 7.4.4, and the corresponding periods of immersion;

(*b*) temperature of test;

(*c*) description of the immersion liquid; in the case of mineral oils (other than standard liquids Nos. 1, 2 and 3) this should include the density, refractive index, and the aniline point or aromatic content;

(*d*) initial thickness and dimensions of test piece;

(*e*) temperature of conditioning;

(*f*) note of any discolouration of the immersion liquid, or formation of sediment, at the conclusion of the test;

(*g*) note of the appearance of the test piece (cracks, delamination, etc.).

8. TEST WITH LIQUID ON ONE SURFACE ONLY

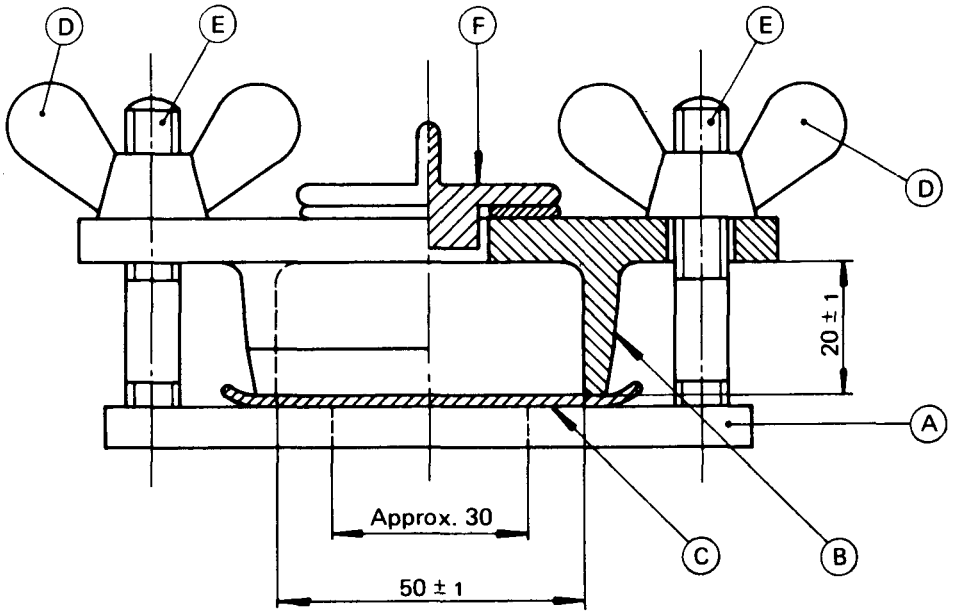
8.1 Scope

This test is applicable to relatively thin sheet materials (rubber-coated fabrics, diaphragms, etc.) that are exposed to the immersion liquid only on one surface. In consequence of possible absorption of fluid by the fabric this method may be less accurate than the volumetric method described in clause 7.3.

8.2 Apparatus

The apparatus should be suitable for holding the standard 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 fluid. During the test the opening in the top of the chamber should be closed by a close-fitting plug (F).

Dimensions in millimetres



FIGURE