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Rubber, vulcanized — Determination of the effect of liquids

Caoutchouc, vulcanisé — Détermination de l'action des liquides

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Contents

Page

1	Scope	1
2	Normative references	1
3	Apparatus	2
4	Test liquids	3
5	Test pieces	4
5.1	Preparation	4
5.2	Dimensions	4
5.3	Time interval between vulcanization and testing	4
5.4	Conditioning	4
6	Immersion in the test liquid	5
6.1	Temperature	5
6.2	Duration	5
7	Procedure	5
7.1	General	5
7.2	Change in mass	6
7.3	Change in volume	6
7.4	Change in dimensions	7
7.5	Change in surface area	8
7.6	Change in hardness	8
7.7	Change in tensile stress-strain properties	9
7.8	Testing with liquid on one surface only	9
7.9	Determination of extractable matter	10
8	Test report	10
Annex A	(normative) Reference liquids	12
A.1	Standard simulated fuels	12
A.2	Reference oils	13
A.3	Simulated service liquids	15
A.4	Chemical reagents	15

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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1817 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 1817:1999), of which it constitutes a minor revision the main purpose of which was to update the normative references clause.

In this corrected version of ISO 1817:2005, Equation (2) on page 7 has been corrected to include “–1” in the brackets on the right-hand side.

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Introduction

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

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

The amount of absorption [a)] is usually larger 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 it is important to measure these properties after treatment of the rubber. The extraction of soluble constituents, especially plasticizers and antidegradants, can likewise alter the rubber's physical properties and chemical resistance after drying (assuming the liquid to be volatile). Therefore, it is necessary to test these properties following immersion or drying of the rubber. This International Standard describes the methods necessary for determining the changes in the following properties:

- change in mass, volume and dimensions;
- extractable matter;
- change in hardness and tensile stress-strain properties after immersion and after immersion and drying.

Although in some respects these tests may 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 one 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, especially with viscous liquids. Moreover, it is known that the action of a liquid on rubber, especially at high temperatures, can be affected by the presence of atmospheric oxygen. The tests described in this International Standard can, however, provide valuable information on the suitability of a rubber for use with a given liquid and, in particular, constitute a useful control when used for developing rubbers resistant to oils, fuels, or other service liquids.

The effect of a liquid may depend on the nature and magnitude of any stress within the rubber. In this International Standard, test pieces are tested in an unstressed condition.

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Rubber, vulcanized — Determination of the effect of liquids

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

CAUTION — Certain procedures specified in this International Standard may involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

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 test liquids. The liquids concerned include current service liquids, such as petroleum derivatives, organic solvents and chemical reagents, as well as reference test liquids.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

ISO 48, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 175, *Plastics — Methods of test for the determination of the effects of immersion in liquid chemicals*

ISO 2592, *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 3016, *Petroleum products — Determination of pour 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 — Hydrometer method*

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

ISO 23529:2004, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Apparatus

3.1 Total immersion apparatus, designed to take account of the volatility of the test liquid and of the immersion temperature in order to prevent and minimize evaporation of the test liquid and the ingress of air.

For tests at temperatures considerably below the boiling point of the test liquid, a stoppered glass bottle or tube shall be used. For tests at temperatures near the boiling point of the test liquid, the bottle or tube shall be fitted with a reflux condenser or other suitable means of minimizing the evaporation of liquid.

The bottle or tube shall be so dimensioned that the test pieces remain completely immersed and all surfaces are completely exposed to the liquid without any restriction. The volume of liquid shall be at least 15 times the combined volume of the test pieces and the volume of air above the liquid shall be kept to a minimum.

The test pieces shall be mounted in jigs, preferably hanging on a rod or wire, and separated from any adjacent test piece, for instance by glass rings or other non-reactive spacers.

The materials of the apparatus shall be inert to the test liquid and to the rubber; for example, materials containing copper shall not be used.

3.2 Apparatus for testing one surface only, which holds the test piece in contact with the liquid on only one of its surfaces.

A suitable apparatus is illustrated in Figure 1. It comprises a base-plate (A) and an open-ended cylindrical chamber (B), which is held tightly against the test piece (C) by wing nuts (D) mounted on bolts (E). A hole of approximately 30 mm diameter is allowed in the base-plate for examination of the surface not in contact with the liquid. During the test, the opening on the top of the chamber shall be closed by a close-fitting plug (F).

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Dimensions in millimetres

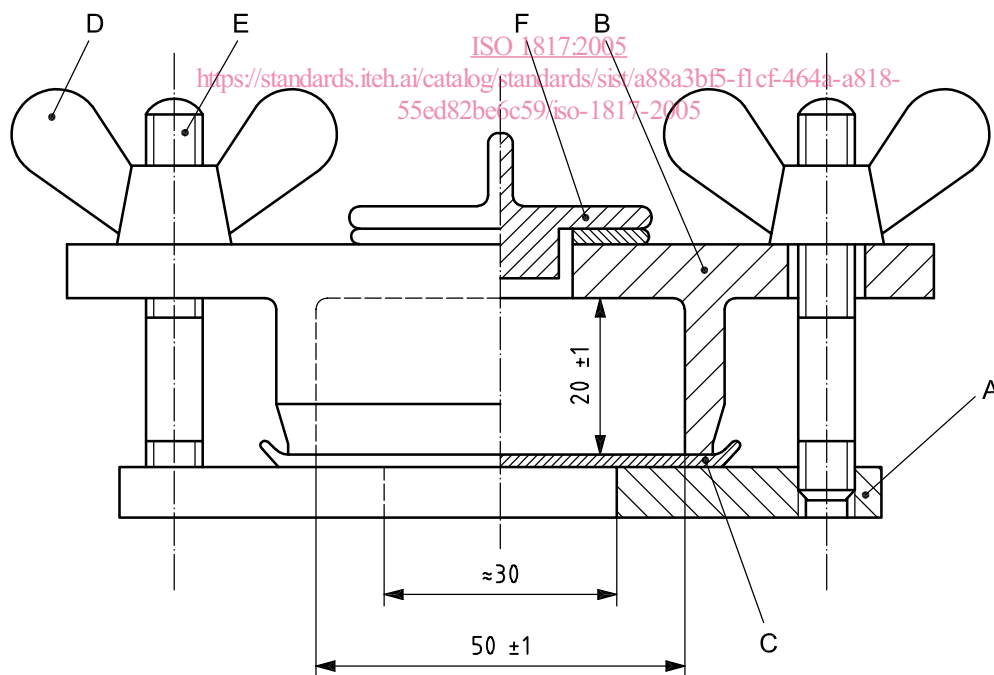


Figure 1 — Apparatus for testing one surface only

3.3 Balance, accurate to 1 mg.

3.4 Instrument for measuring the thickness of the test piece, consisting 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 divisions of 0,01 mm. The plunger shall be fitted with a flat circular contact having a surface area of approximately 100 mm². The contact shall be perpendicular 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.

3.5 Instrument for measuring the length and width of the test piece, having a scale graduated in divisions of 0,01 mm and preferably operating without contact with the test piece, for example using an optical system.

3.6 Instrument for measuring the change in surface area, capable of measuring the lengths of the diagonals of the test pieces.

It shall have a scale graduated in divisions of 0,01 mm and should preferably operate without contact with the test piece, for example using an optical system.

4 Test liquids

The choice of the test liquid shall depend on the purpose of the test.

When information is required on the service behaviour of a vulcanized rubber in contact with a particular liquid, then this liquid shall, if possible, be chosen for the test. Commercial liquids are not always constant in composition and the test shall, whenever practicable, include a reference material of known characteristics. Any abnormal results due to unexpected variations in the composition of the commercial liquid will thus become apparent. It may be necessary to set aside a bulk supply of the liquid for a particular series of tests.

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Mineral oils and fuels are liable to vary considerably in chemical composition even when supplied at 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 the aniline point alone is not sufficient to characterize a mineral oil; other things being equal, the lower the aniline point, the more pronounced the action. If a mineral oil is used as test liquid, the test report shall include the density, refractive index, viscosity and aniline point or aromatic content of the oil. For the reference mineral oils described in Annex A, mineral oil raffinates are employed.

Service oils having similar fluid characteristics to the reference liquids (see Annex A, Clauses A.1 to A.3) will not necessarily have the same effect on the material as the reference liquids. Some fuels, particularly gasoline, vary widely in composition and, for some possible constituents, minor variations can have a large influence on the effect on rubber. Complete details of the composition of the fuel used shall therefore be included in the test report.

As commercial liquids do not always have a constant composition, a standard liquid consisting of well-defined chemical compounds or mixtures of compounds shall be used as reference liquid for the purpose of classification of vulcanized rubbers or quality control. Some suitable liquids are listed in Annex A.

When testing to determine the effect of chemical solutions, the concentration of the solution shall be appropriate to the intended use.

Ensure that the composition of the test liquid does not change significantly during immersion. The ageing of the test liquid and any interaction with the test pieces shall be taken into consideration. If there are chemically active additives in the liquid, or if there is a significant change in composition by extraction, absorption or reaction with the rubber, either the volume shall be increased, or the liquid shall be replaced with fresh liquid at specified intervals.

5 Test pieces

5.1 Preparation

Test pieces shall be prepared in accordance with ISO 23529.

5.2 Dimensions

Data obtained on test pieces having different original thicknesses may not be comparable. Therefore, where possible, test pieces shall be of uniform thickness of $(2 \pm 0,2)$ mm.

Test pieces cut from commercial articles may be used. For products thinner than 1,8 mm, use the original thickness. If the material is thicker than 2,2 mm, reduce the thickness to $(2 \pm 0,2)$ mm.

Test pieces for the determination of the change in volume and mass shall have a volume of 1 cm^3 to 3 cm^3 .

Test pieces for the determination of the change in hardness shall have lateral dimensions of no less than 8 mm.

Test pieces for the determination of the change in dimensions shall be quadrilateral with sides between 25 mm and 50 mm in length, or circular with a diameter of 44,6 mm (internal diameter of type B test piece in ISO 37). This type of test piece can also be used for the determination of mass and volume.

Test pieces for the determination of the change in surface area shall be rhomboid, with the sides cut cleanly and at right angles to the top and bottom surfaces. This can be achieved by two consecutive cuts at approximately right angles to each other, with a cutter consisting of two parallel blades, suitably spaced. The length of the sides shall be nominally 8 mm.

NOTE For the determination of the change in surface area, it may be convenient to use smaller or thinner test pieces, for example when cut from products or when rapid attainment of equilibrium is required. However, the results may not be comparable with those obtained using the specified thickness. Smaller test pieces will reduce the precision of the results.

Test pieces for the determination of tensile properties shall be in accordance with ISO 37, preferably type 2 dumb-bells. This type of test piece can also be used when determining the change in mass, volume or hardness.

For tests with liquid contact on one surface only, the test piece shall consist of a disc with a diameter of about 60 mm.

5.3 Time interval between vulcanization and testing

Unless otherwise specified for technical reasons, the following requirements, in accordance with ISO 23529 for time intervals, shall be observed.

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

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

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

5.4 Conditioning

Test pieces for test in the "as received" condition shall be conditioned for not less than 3 h at one of the standard laboratory temperatures specified in ISO 23529. The same temperature shall be used throughout any test or any series of tests intended to be comparable.

6 Immersion in the test liquid

6.1 Temperature

The immersion should preferably be carried out at one or more of the temperatures listed in 8.2.2 of ISO 23529:2004.

As elevated temperatures may greatly increase the oxidation of the rubber, volatilization or decomposition of the immersion liquid and the effect of any chemically active additives in the liquid (for example in service liquids), appropriate selection of the test temperatures 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 shall approximate those found in service, using the closest standard temperature equal to or higher than the service temperature.

6.2 Duration

Since the rate of penetration of liquids into rubbers depends on the temperature, the type of rubber material and the type of liquid, the use of only one standard period of immersion is precluded. For acceptance purposes, it is recommended that repeated determinations be made and recorded after successive periods of immersion so as to indicate the change in properties with time. The total immersion time shall, if possible, extend well beyond the point of maximum absorption.

For control purposes, a single period of immersion may suffice but maximum absorption should preferably be reached. For such purposes one of the following periods shall be used:

24 ± 2 h; 72 ± 2 h; 7 days \pm 2 h; multiples of 7 days \pm 2 h.

NOTE 1 Since the amount of liquid absorbed is initially proportional to the square root of time rather than time itself, it is helpful to assess the "time to maximum absorption" by plotting the amount absorbed against the square root of time.

NOTE 2 The percentage change during the early stages of immersion is inversely proportional to the test piece thickness. Therefore, lower tolerances for thickness are advisable to obtain consistent results when maximum absorption is not reached.

7 Procedure

7.1 General

Use three test pieces for each set of measurements and make any identification marks required before immersion.

Immerse the test pieces in the appropriate apparatus described in 3.1 or 3.2, using the liquid selected (see Clause 4) and the temperature selected (see 6.1).

For total immersion, place the test pieces at a distance of at least 5 mm from the sides of the container and at least 10 mm from the bottom and top surfaces. If the density of the rubber is less than that of the liquid, means shall be provided for holding the test pieces completely below the surface of the liquid.

The ingress of air shall be avoided. If the influence of air is to be tested, the degree of access of air may be determined by agreement between the interested parties.

At the end of the period of immersion, bring the test pieces, if necessary, to the standard laboratory temperature within 30 min. This can be done by quickly transferring the test pieces to a fresh portion of the test liquid at this temperature and allowing to stand for a period of 10 min to 30 min.