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Rubber, vulcanized or thermoplastic — Determination of the effect of liquids

Caoutchouc vulcanisé ou thermoplastique — Détermination de l'action des liquides

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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

This seventh edition cancels and replaces the sixth edition (ISO 1817:2015), which has been technically revised.

The main changes are as follows:

— Clause 2, normative references, has been updated;

— methods A to E have been defined and the apparatus to be used has been updated in Clause 4;

— the question about liquid replacement or not has been clarified in 9.2.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The action of a liquid on vulcanized or thermoplastic rubber can 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 and drying of the rubber. This document 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 needs to 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 document 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 document, test pieces are tested in an unstressed condition.

Rubber, vulcanized or thermoplastic — Determination of the effect of liquids

WARNING 1 — Persons using this document should be familiar with normal laboratory practice. This document 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 determine the applicability of any other restrictions.

WARNING 2 — Certain procedures specified in this document 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 document describes methods of evaluating the resistance of vulcanized and thermoplastic 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 48-2, *Rubber, vulcanized or thermoplastic — Determination of hardness — Part 2: Hardness between 10 IRHD and 100 IRHD*

ISO 18899:2013, *Rubber — Guide to the calibration of test equipment*

ISO 23529:2016, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

ASTM D5964, *Standard Practice for Rubber IRM 901, IRM 902, and IRM 903 Replacement Oils for ASTM No. 1, ASTM No. 2, and ASTM No. 3 Oils*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

4 Apparatus

4.1 General

Five different methods are defined, all with different equipment.

Method A – Glass vessel with glass lid. Suitable for test with non-volatile and volatile liquids, below the boiling point. Examples of non-volatile liquids are different type of oils (mineral or synthetic), e.g. engine, gearbox and hydraulic oils.

Method B – Glass vessel with stopper or lid, in order to prevent and minimize evaporation of the test liquid and the ingress of air. Suitable for test with non-volatile and volatile liquids, below the boiling point.

Method C – Glass vessel with reflux condenser. For test with high volatile liquids, near the boiling point.

Method D – Pressure vessel with hermetically closed lid, allowing for test at overpressure. For test e.g. above the boiling point, with flammable liquids, or where evaporation of the liquid and ingress of air shall be completely hindered.

Method E – Apparatus for testing one surface only.

In method A, the liquid to test piece volume ratio shall be (80 ± 10):1 and the amount of air above the liquid shall be 10 % ± 2 % of the total vessel volume. If the test deviates from this, it shall clearly be stated in the report. This means that the size of the vessel depends on the size and the amount of test pieces to be tested.

In method B to D, 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 in methods A to D shall be mounted hanging on a rod or wire and separated from any adjacent test piece.

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

Stirring of the liquid is not permitted except in method C.

Heating of the vessels shall be done by storing the vessel in an air heated oven, except for method C where a heating mantle is recommended.

Example on apparatus for methods A to D are shown in Figure 1.

4.2 Apparatus for method A

The glass vessel shall be of flange type with ground surface. The lid shall have a ground surface where it seals to the vessel flange, and shall be clamped by suitable means, e.g. by a wire/steel bracket. The size of the vessel shall be so that the liquid to test piece volume ratio is (80 ± 10):1. Greasing or use of rubber sealant on the flanges is not permitted.

4.3 Apparatus for method B

A glass vessel with stopper or lid shall be used.

4.4 Apparatus for method C

A glass vessel fitted with a reflux condenser shall be used.

4.5 Apparatus for method D

A pressure vessel of suitable quality for the liquid to be tested shall be used. The lid shall be hermetically closed by suitable type of sealing and clamping. The vessel shall be designed to withstand the temperature and pressure it will be exposed to.

Key

1 apparatus for method A
2 apparatus for method B
3 apparatus for method C
4 apparatus for method D

Figure 1 — Apparatus for methods A to D

4.6 Apparatus for method E

An apparatus for testing one surface only, which holds the test piece in contact with the liquid on only one of its surfaces shall be used.

A suitable apparatus is illustrated in Figure 2. 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).

Dimensions in millimetres

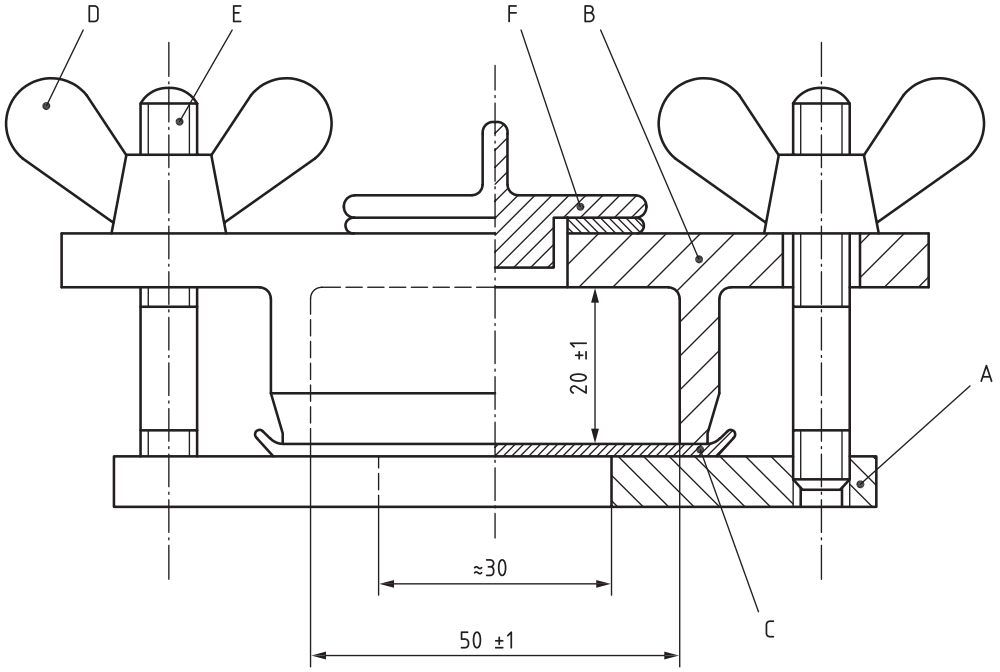

Figure 2 — Apparatus for method E (testing one surface only)

4.7 Additional equipment

4.7.1 Balance, accurate to 1 mg.

4.7.2 Instrument for measuring the thickness of the test piece, consisting of a micrometre dial gauge, of adequate accuracy, firmly held in a rigid stand over a flat base-plate. The instrument shall comply with the requirements given for such apparatus in ISO 23529:2016, method A.

4.7.3 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 complying with the requirements given for such apparatus in ISO 23529:2016, method D.

4.7.4 Instrument for measuring the change in surface area, capable of measuring the lengths of the diagonals of the test piece. 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 complying with the requirements given for such apparatus in ISO 23529:2016, method D.

5 Calibration

The requirements for calibration of the test apparatus are given in Annex B.

6 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 or thermoplastic 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 can then be necessary to set aside a bulk supply of the liquid for a particular series of tests.

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.

Service oils having similar fluid characteristics to the reference liquids (see 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, according to Annex A, shall be used as reference liquid for the purpose of classification of vulcanized or thermoplastic rubbers or quality control.

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

Depending on the temperature, the composition of the test liquid can 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, see 9.2 for more information.

Some liquids are sensitive to oxidation. Thus, they will change significantly when exposed to air at elevated temperature, which may be the case in method C.

NOTE It has been shown that when using commercial oils containing additives, it is not necessarily the entry of air that is important to prevent, but also the escape of volatiles from the immersion vessel (degradation products of the oil or its additives can be very aggressive).

7 Test pieces

7.1 Preparation

Test pieces shall be prepared in accordance with ISO 23529.

7.2 Dimensions

Data obtained on test pieces having different original thicknesses are not always comparable. Therefore, where possible, test pieces shall be of uniform thickness of (2 ± 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 ± 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 A 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 can 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 can differ from 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. Type 2 dumb-bells are preferred because their size makes them more convenient to immerse in liquid than type 1. The type 2 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.

7.3 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 four 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 three months. In other cases, tests shall be made within two months of the date of receipt of the product by the customer.

7.4 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.

8 Immersion in the test liquid

8.1 Temperature

Unless otherwise specified, the immersion shall be carried out at one or more of the temperatures listed in ISO 23529:2016, 10.2.2.

As elevated temperatures can 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.

If necessary, heating cabinets with explosion protection shall be used.

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

8.2 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