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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 document 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 eighth edition cancels and replaces the seventh edition (ISO 1817:2022), which has been technically revised.

The main changes are as follows:

— Clause 4: examples for methods A and C added;

— Clause 6: text added for metalworking fluids;

— 9.6: a method added to enable very short periods between taking the samples out of a liquid and registration of the surface change by the use of photography.

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 following properties after immersion and after immersion and drying:

— change in mass, volume and dimensions;

— extractable matter;

— change in hardness and tensile stress-strain properties.

Although in some respects these tests can 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 can 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 can 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 can 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), for example 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, for example, 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 be clearly 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 methods 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. For a greater number of test pieces, use of a test piece holder can be useful. A possible test piece holder preventing the contact and the floating of samples with lower density is shown in Figures 1 and 2.

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 to 7.

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, for example by a wire or 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.

Measurements are needed to avoid sticking of test pieces in a vessel (shown in Figure 3).

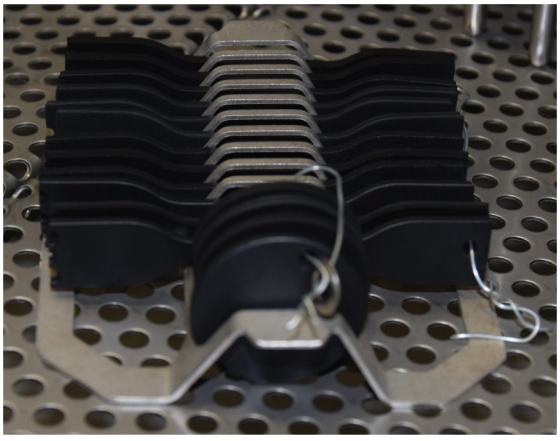


Figure 1 — Example of test piece rack with trays and test pieces (S2 dumbbells and discs)

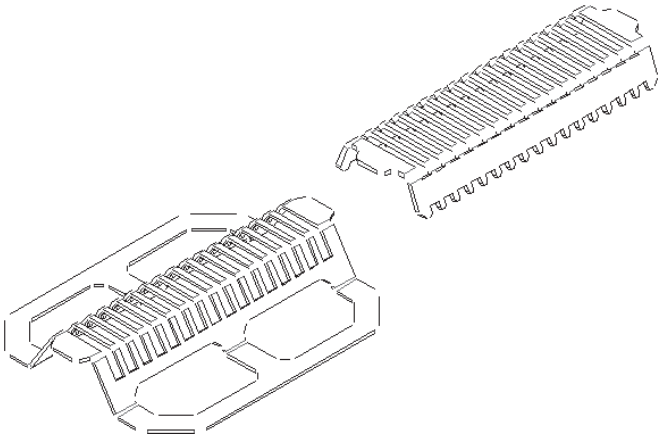


Figure 2 — Example of tray and tray-weight for test pieces with low density

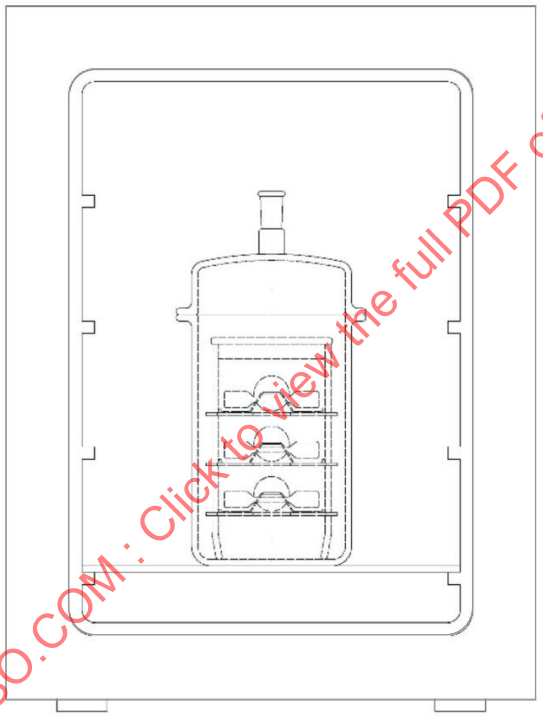


Figure 3 — Example of glass vessel with lid and inside oven

For pressure relief, if the vessel is closed with a glass stopper it could be necessary to open this shortly after the desired temperature is stable inside the oven (vessel).

4.3 Apparatus for method B

A glass vessel with stopper or lid shall be used to prevent the ingress of air.

4.4 Apparatus for method C

4.4.1 General

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

Vessel usually heated up on a heating plate with a magnetic stirrer.

4.4.2 With air contact

Figure 4 gives an example configuration of a reflux condenser and, if necessary, additional openings to have contact to the atmosphere so that a contact of air or oxygen is possible via the surface of the test liquid.

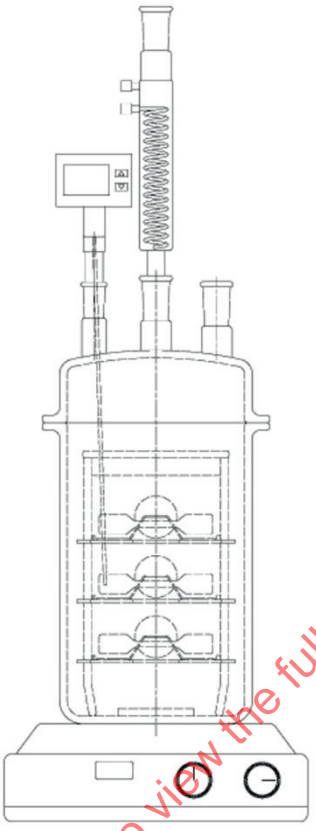


Figure 4 — Example configuration of reflux condenser with air contact

4.4.3 Without air contact

To prevent air (oxygen) contact with the atmosphere, it is possible to connect the reflux condenser with two bottles with a separation liquid. The liquid should not react or generate volatiles which can react with the test liquid. This is shown in Figure 5.

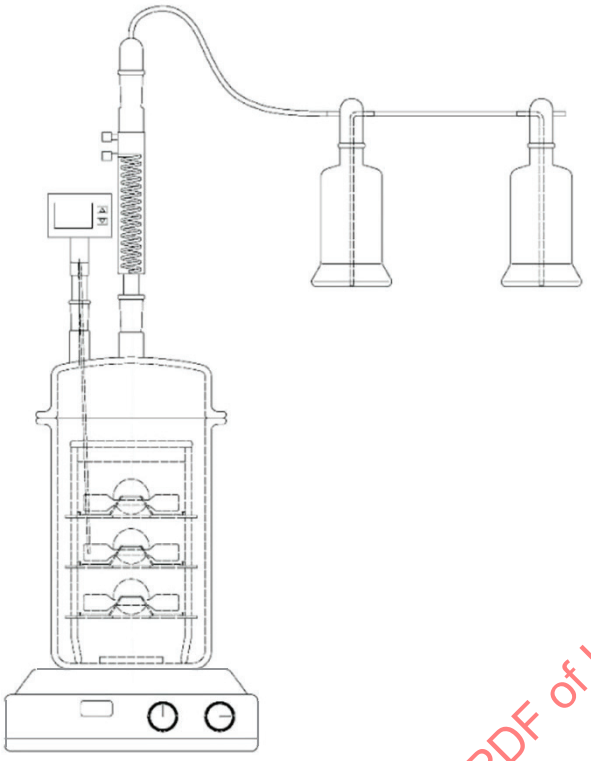


Figure 5 — Example reflux condenser to prevent air (oxygen) contact with the atmosphere.

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 method A
2 method B
3 method C
4 method D

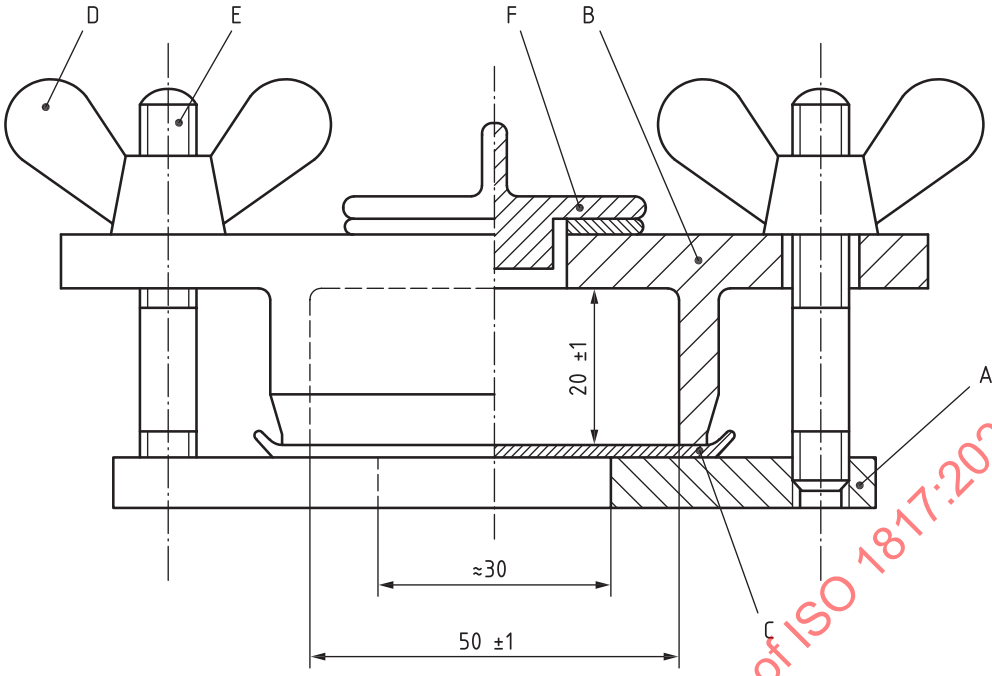
Figure 6 — Possible apparatus for methods A to D

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 7. 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



Key

A base plate
B open-ended cylindrical chamber
C test piece
D wing nuts
E bolts
F close fitting plug

Figure 7 — 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 conform to 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 conforming to 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 conforming to the requirements given for such apparatus in ISO 23529:2016, method D.

5 Calibration

The test apparatus shall be calibrated in accordance with the requirements 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.

Metalworking fluids (MWFs) are used in workshops worldwide for the cutting and forming of metals. Their main purposes are to cool and lubricate tools, work pieces and machines, inhibit corrosion and remove swarf. MWFs are available as non-water-miscible oils, water-miscible or emulsifiable concentrates or fully synthetic, oil-free products. Non-water-miscible MWFs mainly consist of base oil (usually over 95 %). This can be a mineral oil, ester oil (e.g. unrefined or chemically modified rapeseed oil) or synthetic oil (e.g. poly-alpha-olefin). For testing, they can be used as they are shipped.

Water-miscible MWFs are mixed with water before use, in concentrations of 2 % to 25 %, depending on the product and type of machining. This type of MWF is based on oil and shares of 20 % to 80 % are possible. To combine this oil with water to yield an oil-in-water emulsion, an emulsifier is necessary. Fully and semi-synthetic fluids types of MWFs are water-miscible and do not require emulsifiers. They can be based on water-miscible glycol compounds, for example. Mixing in water yields a transparent water-mixed MWF. Seal compatibility tests can be performed either with the delivered concentrate or fresh prepared emulsion or solution. For mixing a test emulsion or solution, de-ionised water at ambient temperature should be used. The standard concentration is a mass fraction of 10 %, but other concentrations can be used if necessary. The metalworking fluid concentrate should be added slowly to water with agitation (e.g. by employing a magnetic stirrer), to avoid gelling or splitting of the emulsion.

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 only important to prevent the entry of air, 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 (see ISO 23529).

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. Circular test pieces with a diameter of 44,6 mm can also be used.

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 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.

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 (e.g. 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 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 acceptance purposes, it is recommended that repeated determinations be made and recorded after successive periods of immersion 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 can suffice, preferably chosen such that maximum absorption is reached. For such purposes, one of the following periods shall be used: $24^{\ 0}_{-2}$ h; $72^{\ 0}_{-2}$ h; 7 days ± 2 h; multiples of 7 days ± 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 will give more consistent results when maximum absorption is not reached.

9 Procedure

9.1 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 4.2, 4.3, 4.4, 4.5 or 4.6, using the liquid selected (see Clause 6) and the temperature selected (see 8.1).

For methods A to D, 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.

At the end of the period of immersion, bring the test pieces 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.

Remove surplus test liquid from the surface. When volatile liquids are used, remove and quickly wipe the test pieces with a filter paper or a piece of lint-free fabric. Viscous non-volatile liquids can be removed by filter paper and, if necessary, by quickly immersing the test pieces in a volatile liquid, such as ethanol or petroleum ether, then quickly wiping them.

Following removal of the test pieces from volatile test liquids, it is important that each subsequent manipulation takes place as soon as possible. Carry out the tests immediately after the removal of surplus liquid or, for change in mass or volume, by placing the test piece immediately in a weighing bottle.

If, after the measurement of mass or dimensions, the same test pieces are used for the measurement of other properties, immerse the test pieces in the volatile liquid again. The total immersion time shall be in accordance with 8.2. For a volatile liquid, the maximum time between removal from the test liquid and the end of the measurement shall be:

— change in dimensions: 1 min;

— change in hardness: 2 min;

— tensile test: 2 min.

If the immersion is to be continued, put the test pieces back in the liquid immediately and return them to the temperature-controlled oven or bath.

The changes in properties can also be determined after drying. For this purpose, dry the test pieces under an absolute air pressure of approximately 20 kPa at approximately 40 °C to constant mass, i.e. until the difference between successive weighing 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.

9.2 Replacement of test liquid

No replacement of the test liquid is permitted, unless otherwise stated.

If liquid replacement is prescribed, it shall be clearly stated in the test description and the test report. The intervals for liquid replacement shall be according to Table 1.

Table 1 — Intervals for replacement of test liquid

Liquid	Temperature °C	Recommended replacement interval h
Engine type of oils	125 to 140	504
	141 to 150	336
Hypoid (gearbox type) oils	125 to 140	336
	141 to 150	168

For other type of fluids, replacement intervals shall be agreed between interested parties.

9.3 Change in mass

NOTE The rubber industry uses the term equation for the relationships herein termed formula.

Weigh each test piece to the nearest milligram at the standard laboratory temperature before and after immersion.

Calculate the percentage change in mass Δm_{100} using Formula (1):

$$\Delta m_{100} = \frac{m_i - m_0}{m_0} \times 100 \quad (1)$$

where

m_0 is the initial mass of the test piece;

m_i is the mass of the test piece after immersion.

Report the result as the median value for the three test pieces.

9.4 Change in volume

The water displacement method is used for test liquids which are immiscible with water.

Weigh each test piece in air to the nearest milligram (mass m_0), and then reweigh each test piece in distilled water at the standard laboratory temperature (mass $m_{0,w}$), taking care to ensure that all air bubbles are removed (a detergent can be used). If the density of the material is less than 1 g/cm^3, 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, determine the mass of the sinker alone in distilled water separately (mass $m_{s,w}$). Blot the test pieces dry with filter paper or lint-free fabric.

Immerse each test piece in the test liquid. At the end of the period of immersion, weigh each test piece in air (mass m_i) to the nearest milligram, and then reweigh each test piece in distilled water (mass $m_{i,w}$), also at the standard laboratory temperature.

Calculate the percentage change in volume ΔV_{100} using Formula (2):

$$\Delta V_{100} = \left(\frac{m_i - m_{i,w} + m_{s,w}}{m_0 - m_{0,w} + m_{s,w}} - 1 \right) \times 100 \quad (2)$$

where

m_0 is the initial mass of the test piece;

m_i is the mass of the test piece after immersion;

$m_{0,w}$ is the initial mass of the test piece (plus sinker if used) in water;

$m_{i,w}$ is the mass of the test piece (plus sinker if used) after immersion in water;

$m_{s,w}$ is the mass of the sinker, if used, in water.

Report the result as the median value for the three test pieces.

If the test liquid is readily miscible with water or reacts with it, water cannot be used after immersion. If the test liquid is not too viscous or volatile at room temperature, a fresh portion of the test liquid can be used. If the test liquid is not suitable, use another liquid after immersion and calculate using Formula (3):

$$\Delta V_{100} = \left[\frac{1}{\rho} \left(\frac{m_i - m_{i,liq} + m_{s,liq}}{m_0 - m_{0,w} + m_{s,w}} \right) - 1 \right] \times 100 \quad (3)$$

where

ρ is the density of the liquid;

$m_{i,liq}$ is the mass of the test piece (plus sinker, if used) in the liquid;

$m_{s,liq}$ is the mass of the sinker, if used, in the liquid.

9.5 Change in dimensions

Measure the initial length of each test piece along its centre line to the nearest 0,5 mm at the standard laboratory temperature (taking measurements along the top and bottom surfaces and averaging the two results). Similarly, measure the initial width by taking four measurements in all (top and bottom, both sides) near each end of the test piece.

Measure the initial thickness with the thickness gauge at four different points along the test piece and calculate the average of the results.

After immersion, re-measure the length, width and thickness of each test piece.

Make all measurements with the test piece at the standard laboratory temperature.

Calculate the percentage change in length Δl_{100} using Formula (4):

$$\Delta l_{100} = \frac{l_i - l_0}{l_0} \times 100 \tag{4}$$

where

l_0 is the initial length;

l_i is the length after immersion.

Similarly, calculate the percentage changes in width and thickness.

Report the results as the median values for the three test pieces. The change in surface area can be calculated from the values obtained for the length and the width.

9.6 Change in surface area

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

After immersion, re-measure the lengths of the diagonals. 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.

For method D, an alternative to an optical measuring system is taking a photo of the test piece and an accurate calibrated ruler together (see example in Figure 8). Especially for high evaporating media, this saves time and the dimensions are frozen shortly after taking the test pieces out of the medium. It is easiest to take a photo from the bottom through a glass surface. By fixing the camera with a set focus it is possible to take a photo within 10 seconds of removal from the liquified gas. The dimensions can be measured afterwards on the photo taken, without hurry. Use of circular test pieces can make it easier.

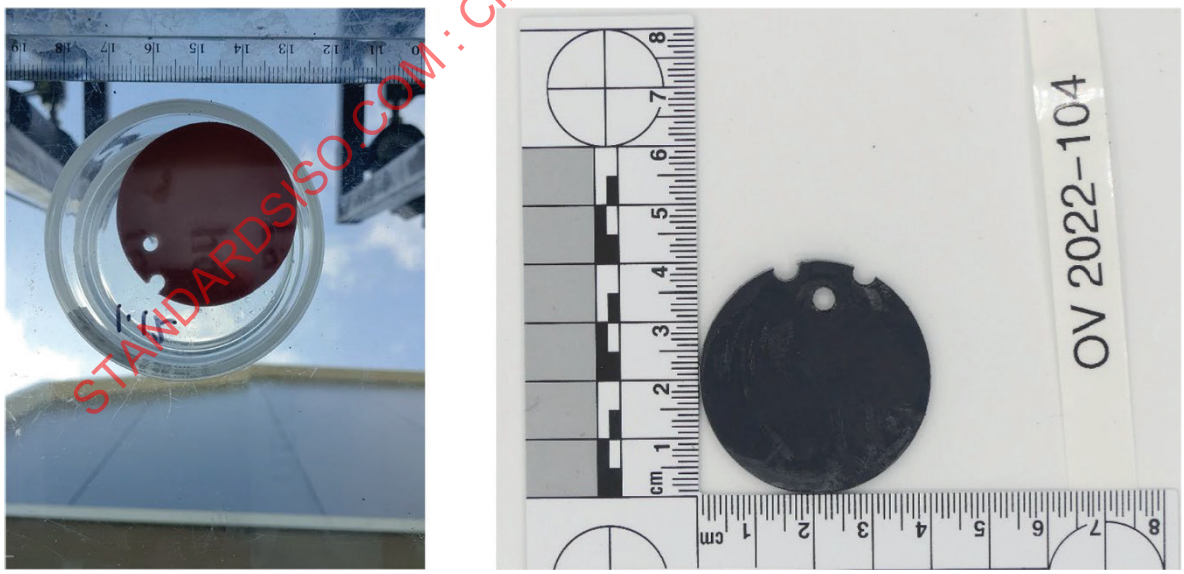


Figure 8 — Photos of test pieces after immersion from below with a ruler

Calculate the percentage change in area ΔA_{100} using Formula (5):

$$\Delta A_{100} = \left(\frac{l_A l_B}{l_a l_b} - 1 \right) \times 100 \quad (5)$$

where

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

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

If required, the percentage volume change ΔV_{100} can be calculated using Formula (6):

$$\Delta V_{100} = \left[\left(\frac{l_A l_B}{l_a l_b} \right)^{3/2} - 1 \right] \times 100 \quad (6)$$

Formula (6) assumes isotropic swelling. If any doubt exists, determine the percentage volume change as specified in 9.4, which is the preferred method.

For circular test pieces use Formula (7)

$$\Delta V_{100} = \left[\left(\frac{A_{\text{after immersion}}}{A_{\text{before immersion}}} \right)^{3/2} - 1 \right] \times 100 \quad (7)$$

where $A = \pi \times \left(\frac{d}{2} \right)^2$

Report the result as the median value for the three test pieces.

9.7 Change in hardness

Measure the IRHD hardness using the micro test described in ISO 48-2 on each test piece before and after immersion.

As an alternative, the normal IRHD hardness may be used with three plied-up test pieces, but in this case express the result as the apparent hardness.

Calculate the change in IRHD hardness ΔH, before and after immersion, using Formula (8):

$$\Delta H = H_i - H_0 \quad (8)$$

where

H_0 is the initial hardness;

H_i is the hardness after immersion.

Report the result as the median value for the three test pieces.

Alternatively, the method specified in ISO 48-4 may be used.

9.8 Change in tensile stress-strain properties

Measure tensile stress-strain properties before and after immersion in accordance with ISO 37.

Calculate the selected tensile properties using the initial cross-section of the test piece for stress calculations. Calculate the change in the property ΔX_{100} as a percentage of the value for un-immersed material using Formula (9):

$$\Delta X_{100} = \frac{X_i - X_0}{X_0} \times 100 \tag{9}$$

where

X_0 is the initial value of the property;

X_i is the value of the property after immersion.

Report the result as the median value for the three test pieces.

9.9 Testing with liquid on one surface only

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

Measure the nominal thickness of the test piece and then weigh it in air to the nearest milligram (mass m_0).

Next, place the test piece in the apparatus as indicated in Figure 7. 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. Remove any surplus liquid from the surface of the test piece by wiping with filter paper or a lint-free fabric. Then weigh the test piece to the nearest milligram (mass m_i) and measure the thickness at the standard laboratory temperature.

If the test liquid is volatile at room temperature, make the measurement within 2 min of removal from the liquid.

Express the change in mass per unit surface area Δm_A, in grams per square metre, using Formula (10):

$$\Delta m_A = \frac{m_i - m_0}{A} \tag{10}$$

where

m_0 is the initial mass, in grams, of the test piece;

m_i 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.

Report the result as the median for the three test pieces.

Calculate the change in thickness as specified in 9.5.

9.10 Determination of extractable matter

9.10.1 General

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

a) drying the treated test piece and comparing its mass with the mass before immersion;

b) evaporating the test liquid to dryness and weighing the non-volatile residue.

Both methods are susceptible to error. In the method in which the dried test piece is weighed, the material can be oxidized if air is present during immersion, especially at high temperatures. In the method in which the test liquid is evaporated, there can be some loss of volatile extracted matter, especially plasticizers. Both methods are described in this document and the choice between them depends 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 is suggested that the procedures described are not suitable for liquids less volatile than standard liquids A, B, C, D and E in Annex A, i.e. for liquids boiling at above 110 °C.

The determination of extractable matter shall be made after having determined the change in mass (see 9.3), the change in volume (see 9.4) and the change in dimensions (see 9.5).

Report the result as the median for the three test pieces.

9.10.2 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, i.e. until the difference between successive weighing at 30 min intervals does not exceed 1 mg.

The extractable-matter content is taken as the difference between the original mass of the test piece and its mass after immersion and drying, expressed as a percentage of the original mass of the test piece.

9.10.3 By evaporating the test liquid

Transfer the liquid in which the test piece was immersed to a suitable container and wash the test piece with 25 ml of fresh liquid, collecting the washings in the same container. 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 in a volume of the test liquid equal to that used for the immersion plus that used for washing.

The extractable-matter content is taken as the mass of the dried residue, corrected for the result of the blank test, expressed as a percentage of the original mass of the test piece.

10 Precision

Precision results of an interlaboratory test programme (ITP) are given in Annex C.

11 Test report

The test report shall include the following information:

a) sample details:

   1) a full description of the sample and its origin;

2) the method of preparation of test pieces from the sample, for example whether moulded or cut;

b) a reference to this document (i.e. ISO 1817:2024);

c) test method and test details:

1) the method used;

2) the type(s) of test piece used (dimensions);

3) the test liquid used (detailed enough to enable repeating to test);

4) the standard laboratory temperature used;

5) details of conditioning;

6) the period and temperature of immersion;

7) liquid replacement or not during the exposure;

8) any deviation from the specified procedure;

d) test results:

1) the results, expressed in the form stated in the relevant subclause;

2) the appearance of the test piece (e.g. cracking, delamination), if appropriate;

3) the appearance of the test liquid (e.g. discoloration, sedimentation), if appropriate;

e) the date of the test.

Annex A
(normative)

Reference liquids

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

Commercial fuels vary widely in composition even within the same grade (i.e. knock-rating) and from the same source. There are hydrocarbon-based fuels with and without oxygen compounds as well as alcohol-based fuels. The grade of gasoline is improved by adding aromatic or oxygen-containing compounds, but these additives increase the effect of fuels on normally fuel-resistant rubbers. The composition varies with the situation on the gasoline market and with the geographical area and can change rapidly. Hence, several test liquids which are used in practice are recommended in Tables A.1 and A.2 to cover the range of different compositions. They can also serve as guidelines for the formulation of other suitable test liquids. Analytical reagent quality materials shall be used in making up the test liquids. Test liquids containing alcohol shall not be used if the fuels involved are known to be free of alcohol.

Table A.1 — Standard simulated fuels without oxygen compounds

Liquid	Constituents	CAS Registry Number®[a]	Content % (by volume)
A	2,2,4-Trimethylpentane	540–84–1	100
B	2,2,4-Trimethylpentane Toluene	540–84–1 108–88–3	70 30
C	2,2,4-Trimethylpentane Toluene	540–84–1 108–88–3	50 50
D	2,2,4-Trimethylpentane Toluene	540–84–1 108–88–3	60 40
E	Toluene	108–88–3	100
F	Straight-chain paraffins (C_{12} to C_{18}) 1-Methylnaphthalene	68476–34–6 90–12–0	80 20

NOTE Liquids B, C and D simulate petroleum-derived fuels without oxygen compounds. Liquid F is intended to simulate diesel fuel, domestic heating oils and similar light furnace oils.

[a] Chemical Abstracts Service (CAS) Registry Number® is a trademark of the American Chemical Society (ACS). This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Table A.2 — Standard simulated fuels containing oxygen compounds (alcohols)

Liquid	Constituents	CAS Registry Number	Content % (by volume)
1	2,2,4-Trimethylpentane Toluene Di-isobutylene Ethanol	540–84–1 108–88–3 25167–70–8 64–17–5	30 50 15 5
2	2,2,4-Trimethylpentane Toluene Di-isobutylene Ethanol Methanol Water	540–84–1 108–88–3 25167–70–8 64–17–5 67–56–1 7732–18–5	25,35[a] 42,25[a] 12,68[a] 4,22[a] 15,00 0,50
3	2,2,4-Trimethylpentane Toluene Ethanol Methanol	540–84–1 108–88–3 64–17–5 67–56–1	45 45 7 3
4	2,2,4-Trimethylpentane Toluene Methanol	540–84–1 108–88–3 67–56–1	42,5 42,5 15

[a] Together, these four constituents are equivalent to 84,5 % (by volume) of liquid 1.

A.2 Reference oils

A.2.1 General descriptions

Oil no. 1 (IRM 901) is a “low volume increase” oil, oil no. 2 (IRM 902) is a “medium volume increase” oil and oil no. 3 (IRM 903) is a “high volume increase” oil.

These reference oils are representative of low-additive mineral oils.

A.2.2 Requirements

The oils shall have the properties specified in ASTM D5964 and shown in Table A.3. The properties given in Table A.4 are typical of the oils but cannot be guaranteed by suppliers.

When these reference oils are required as test liquids, only those obtained from recognized suppliers shall be used for referee purposes and they shall be available for general use. However, in the event that they are not available, alternative oils may be used, but for routine testing only, provided that they conform to the requirements of Table A.3 and also have been shown to give results similar to those obtained with the reference oils when testing rubbers of the same type of composition as those on which the routine tests are to be carried out.

Table A.3 — Specifications of reference oils

Property	Requirements		
	Oil no. 1	Oil no. 2	Oil no. 3
Aniline point, °C	124 ± 1	93 ± 3	70 ± 1
Kinematic viscosity, m^2/s ($\times 10^{-6}$)	18,12 to 20,34[a]	19,2 to 21,5[a]	31,9 to 34,1[b]
Flash point, °C, min.	243	232	163
API gravity at 16 °C	27,8 to 29,8	19,0 to 21,0	21,0 to 23,0
Viscosity-gravity constant	0,790 to 0,805	0,860 to 0,870	0,875 to 0,885
Naphthenics content, c_N, %	27 (average)	35, min.	40, min.
Paraffinics content, c_P, %	65, min.	50, max.	45, max.

[a] Measured at 99 °C.

[b] Measured at 37,8 °C.

Table A.4 — Typical properties of reference oils

Property	Requirements		
	Oil no. 1	Oil no. 2	Oil no. 3
Pour point, °C	−12	−12	−31
ASTM colour	L3.5	L2.5	L0.5
Refractive index at 20 °C	1,484 8	1,510 5	1,502 6
UV absorbance at 260 nm	0,8	4,0	2,2
Aromatics content, c_A, %	3	12	14

A.3 Simulated service liquid

A.3.1 General

Liquid 102 simulates certain high-pressure hydraulic oils. It is a blend comprising 95 % (mass fraction) of oil no. 1 and 5 % (mass fraction) of a hydrocarbon-compound oil additive containing 29,5 % (mass fraction) to 33 % (by mass) of sulfur, 1,5 % to 2 % (mass fraction) of phosphorus and 0,7 % (mass fraction) of nitrogen.

NOTE Liquid 102 has been removed from this document because no supplier can be found.

A.3.2 Liquid 101

Liquid 101 is intended to simulate synthetic diester-type lubricating oils. It is a blend comprising 99,5 % (mass fraction) of di-2-ethylhexyl sebacate (CAS Registry Number: 122-62-3) and 0,5 % (mass fraction) of phenothiazine (CAS Registry Number: 92-84-2).

A.3.3 Liquid 103

Liquid 103 is intended to simulate phosphate-ester hydraulic oils used in aircraft. It is tri-*n*-butyl phosphate (CAS Registry Number: 126-73-8).

A.4 Cooling lubricants

MWFs contain between 1 to 20 (and more) different substances. Some of them can cause adverse health effects, so it is highly recommended to have a close look to the safety data sheets. Some substances can react during storage, namely at elevated temperatures. Therefore, soluble MWF concentrates should not be used for testing if they are older than one year or if the concentrate got separated into different phases. There are numerous formulations available on the market and frequent formulation changes are necessary due to

health and safety legislation acts and new technical requirements. However, standard formulations can be used that cover a wide variety of formulation concepts. These standard water miscible fluid formulations are:

— "VSI 14": formulation based on polyalkylenglycole, boron-free and elevated pH (high amine content) in Table A.5;[1)]

— "VSI 22": formulation based on esters, boron-free in Table A.6;[2)]

— "VSI 34": formulation based on mineral oil, boron-free and elevated pH (high amine content) in Table A.7.[3)]

Table A.5 — Typical composition of "VSI 14"

Key components VSI 14	CAS Registry Number	Mass fraction %
Cyclohexylamin and triethanlolamine reaction products with carboxylic acids	Based on 101–83–7, 102–71–6 and 141–43–5	20 to 40
Triethanolamine	102–71–6	5 to 10
Polyglycole, Phenylglycole	122–99–6	2 to 10
Di-Cyclohexylamine	101–83–7	1 to 5
Benzisothialozinone	2634–33–5	1,5
Others		rest

Table A.6 — Typical composition of "VSI 22"

Key components VSI 22	CAS Registry Number	Mass fraction %
Monoethanol and triethanlolamin reaction products with carboxylic acids	Based on 141–43–5 and 68603–39–4	20 to 40
Triethanolamin	102–71–6	1 to 3
Synthetic Esters	68920–66–1	20 to 40
Ethoxylates	61791–12–6	1,5
Butyldiglycole	112–34–5	5 to 10
Alkylophosphates	192268–65–8	1 to 5
Fettyalcoholes	67762–25–8	1 to 5
Others		Rest

1) VSI 14 can be obtained commercially.

2) VSI 22 can be obtained commercially.

3) VSI 34 can be obtained commercially.

Table A.7 — Typical composition of "VSI 34"

Key components VSI 34	CAS Registry Number	Mass fraction %
Mineral oil, naphthenic	64742–52–5	30 to 50
Fatty acids	8002–26–4, 227310–69–2	5 to 10
Dicyclohexylamine, monoethanol and triethanlolamin neutralization products	Based on 101–83–7, 102–71–6 and 141–43–5	10 to 20
Fatty alcohol ethoxylates	68920–66–1	5 to 10
Polysulfides	68425–15–0	1 to 5
Alcohols	2425–77–6	1 to 5
MEA	141–43–5	1 to 5
Biocide	55406–53–6	0,1 to 1
Others	—	Rest

A.5 Chemical reagents

Tests with chemical reagents shall be carried out using the same chemicals at the same concentrations as those to be encountered in the intended use of the product. For general purposes, where no specification is known, the list of chemical reagents given in ISO 175 can be useful.

Annex B
(normative)

Calibration schedule

B.1 Inspection

Before any calibration is undertaken, the condition of the items to be calibrated shall be ascertained by inspection and recorded in any calibration report or certificate. It shall be reported whether calibration is carried out in the "as-received" condition or after rectification of any abnormality or fault.

It shall be ascertained that the apparatus is generally fit for the intended purpose, including any parameters specified as approximate and for which the apparatus does not therefore need to be formally calibrated. If such parameters are liable to change, then the need for periodic checks shall be written into the detailed calibration procedures.

B.2 Schedule

Verification or calibration of the test apparatus is a mandatory part of this document. However, the frequency of calibration and the procedures used are, unless otherwise stated, at the discretion of the individual laboratory, using ISO 18899 for guidance.

The calibration schedule given in Table B.1 has been compiled by listing all of the parameters specified in the test method, together with the specified requirement. A parameter and requirement can relate to the main test apparatus, to part of that apparatus or to an ancillary apparatus necessary for the test.

For each parameter, a calibration procedure is indicated by reference to ISO 18899, to another publication or to a procedure particular to the test method which is detailed (whenever a calibration procedure which is more specific or detailed than that in ISO 18899 is available, it shall be used in preference).

The verification frequency for each parameter is given by a code-letter. The code-letters used in the calibration schedule are:

R use of certified reference material;

C requirement to be confirmed, but no measurement;

N initial verification only;

S standard interval as given in ISO 18899;

U in use.

Table B.1 — Calibration frequency schedule

Parameter	Requirement(s)	Subclause in ISO 18899:2013	Verification frequency guide	Notes
Total immersion apparatus	Volume such that test pieces remain completely immersed and all surfaces are exposed to liquid	15.8, 19.1	U	
	Volume of liquid 80 ± 10 times that of the test pieces	15.8, 19.1	U	Method A
	Volume of liquid at least 15 times that of test pieces	15.8, 19.1	U	Method B to D
	Amount of air above the liquid 10 % ± 2 % of the total vessel volume	15.8, 19.1	U	Method A
	Inert to test liquid and rubber	15.8, 19.1	U	
	Stoppered bottle or tube to be used	19.1	U	Method B
	Bottle or tube to be fitted with reflux condenser or equivalent	19.1	U	Method C
Apparatus for testing one surface only	As shown in Figure 7	—	N	
Balance	Accurate to 1 mg	22.1	S	
Dial gauge	See ISO 23529:2016, method A	15.1, 16.6	S	
Instrument for measuring length and width	Scale graduated in divisions of 0,01 mm	15.1	S	Preferably non-contacting
Instrument for measuring change in surface area	Scale graduated in divisions of 0,01 mm	15.3	S	Preferably non-contacting
Test liquid	As specified in Annex A	—	N	
Materials — distilled water — lint-free blotting medium	To be used in 9.4	—	U	

In addition to the items listed in Table B.1, use of the following is implied, all of which shall be calibrated in accordance with ISO 18899:

— a timer;

— a thermometer for monitoring the conditioning and test temperatures;

— a hygrometer for monitoring the conditioning and test humidities;

— apparatus for measuring selected physical properties.