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Rubber, vulcanized — Determination of the effect of liquids

Caoutchouc, vulcanisé — Détermination de l'action des liquides

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Foreword

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

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.

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).

Dimensions in millimetres

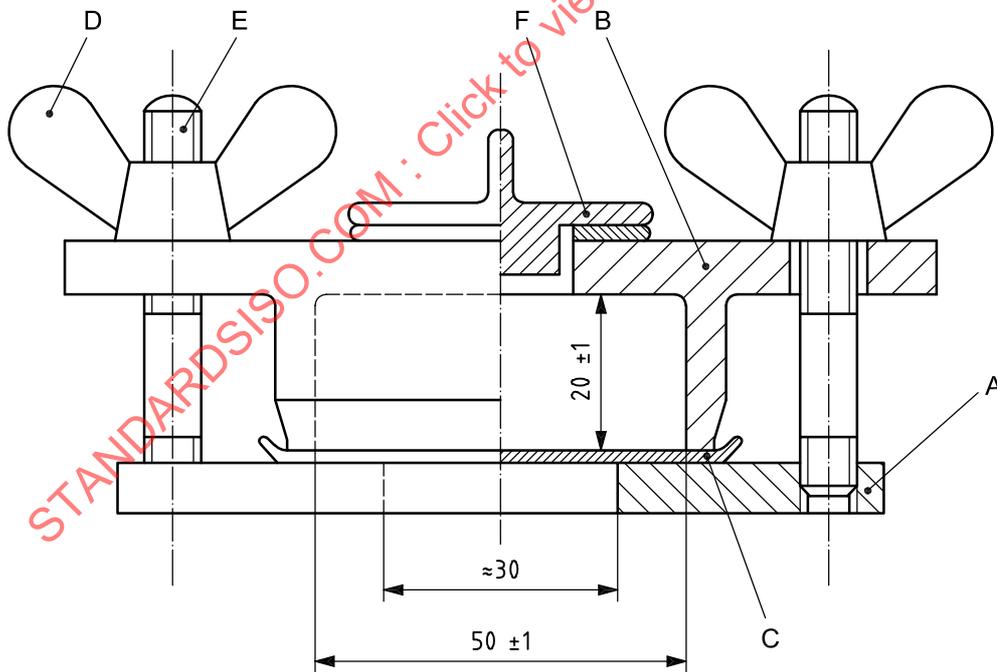


Figure 1 — Apparatus for testing one surface only

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.

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.

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 methanol 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 6.2. 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: 1 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 weighings at 30 min intervals does not exceed 1 mg. Cool to room temperature and condition by keeping at the standard laboratory temperature for not less than 3 h.

7.2 Change in mass

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} as follows:

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

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.

7.3 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/cm³, it will be necessary to use a sinker when weighing in water to ensure that the test pieces are completely immersed. If a sinker is used, 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 the following equation:

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

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 as follows:

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

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;

the other symbols are as defined for Equation (2).

7.4 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 as described above.

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

Calculate the percentage change in length Δl_{100} using the following equation:

$$\Delta l_{100} = \frac{l_i - l_0}{l_0} \times 100 \quad (4)$$

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.

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

Calculate the percentage change in area ΔA_{100} using the following equation:

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

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} may be calculated from the following equation:

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

The equation for percentage volume change assumes isotropic swelling. If any doubt exists, determine the percentage volume change as specified in 7.3, which is the preferred method.

7.6 Change in hardness

Measure the IRHD hardness using the microtest described in ISO 48 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 the following equation:

$$\Delta H = H_i - H_0 \quad (7)$$

where

H_0 is the initial hardness;

H_i is the hardness after immersion.

7.7 Change in tensile stress-strain properties

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

Calculate the tensile strength, the elongation at break and the stress at a given elongation using the initial cross-section of the test piece. Calculate the change in the property ΔX_{100} as a percentage of the value for un-immersed material using the following equation:

$$\Delta X_{100} = \frac{X_i - X_0}{X_0} \times 100 \quad (8)$$

where

X_0 is the initial value of the property;

X_i is the value of the property after immersion.

7.8 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).

Then place the test piece in the apparatus as indicated in Figure 1. 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 following removal from the liquid.

Express the change in mass per unit surface area Δm_A , in grams per square metre, using the following equation:

$$\Delta m_A = \frac{m_i - m_0}{A} \quad (9)$$

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 7.4.

7.9 Determination of extractable matter

7.9.1 General

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

- a) either by drying the treated test piece and comparing its mass with the mass before immersion;
- b) or by 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 International Standard 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 7.2), the change in volume (see 7.3) and the change in dimensions (see 7.4).

7.9.2 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 weighings 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.

7.9.3 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.

8 Test report

The test report shall include the following information:

- a) a reference to this International Standard;
- b) sample details:
 - 1) a full description of the sample and its origin,

- 2) compound details, cure time and temperature where appropriate, and time interval between vulcanization and testing,
 - 3) the method of preparation of the test pieces from the sample, for example whether moulded or cut,
 - 4) a full description of the test liquid; in the case of mineral oils (other than reference oils 1, 2 and 3), this shall include the density, viscosity, refractive index and aniline point or aromatic content;
- c) test method and test details:
- 1) the method(s) used,
 - 2) the type of test piece used (dimensions),
 - 3) the standard laboratory temperature used,
 - 4) details of conditioning,
 - 5) the period and temperature of immersion,
 - 6) 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 (for example cracking, delamination), if appropriate,
 - 3) the appearance of the test liquid (for example discoloration, sedimentation), if appropriate;
- e) the date of the test.

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