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Rubber — Determination of solvent extract

Caoutchouc — Détermination de l'extrait par les solvants

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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.

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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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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*.

The fifth edition cancels and replace the fourth edition (ISO 1407:2011), which has been technically revised.

The main changes are as follows:

- corrections to clarify mass measurement accuracy have been made;
- warnings to ensure safety in operations have been added;
- the CAS numbers have been added for all chemicals listed in this document

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.

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Rubber — Determination of solvent extract

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 specifies four methods for the quantitative determination of the material extractable from raw rubbers, both natural and synthetic; two of the methods are also applicable to the unvulcanized and vulcanized rubber compounds.

Method A measures the mass of the solvent extract, after evaporation of the solvent, relative to the mass of the original test portion.

Method B measures the difference in the mass of the test portion before and after extraction.

Method C, which is for raw rubbers only, measures the difference in the mass of the test portion before and after extraction using boiling solvent.

Method D, which is for raw rubbers only, measures the difference in the mass of the test portion before and after extraction relative to the mass of the original test portion.

NOTE 1 Depending on the test method used, the conditioning of the test portion and the solvent used, the test result is not necessarily the same.

NOTE 2 Method C generally gives results which are lower than those obtained with methods A and B due to an equilibrium which is set up, particularly if large test portions are used, depending on the content and the nature of the extractable matter. Method C is, however, a quicker method than method A or method B.

NOTE 3 Methods C and D are not suitable if the test portion disintegrates during the extraction.

NOTE 4 Method D is normally used for production controls.

Recommendations as to the solvent most appropriate for each type of rubber are given in [Annex A](#).

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 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

ISO 4661-2, *Rubber, vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests*

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>

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

4 Principle

A rubber test piece is extracted with an appropriate solvent in suitable equipment.

The amount extracted is obtained by measuring either the mass of the extract or the mass of the test piece after extraction and comparing it to the mass of the original test piece.

5 Solvent

During the analysis, use only solvents of recognized analytical grade.

One of the solvents recommended in [Table A.1](#) should preferably be used, unless otherwise specified or agreed between the interested parties.

IMPORTANT — Persons using this document should consult the safety data sheet for the solvent before its use and take appropriate measures.

6 Apparatus

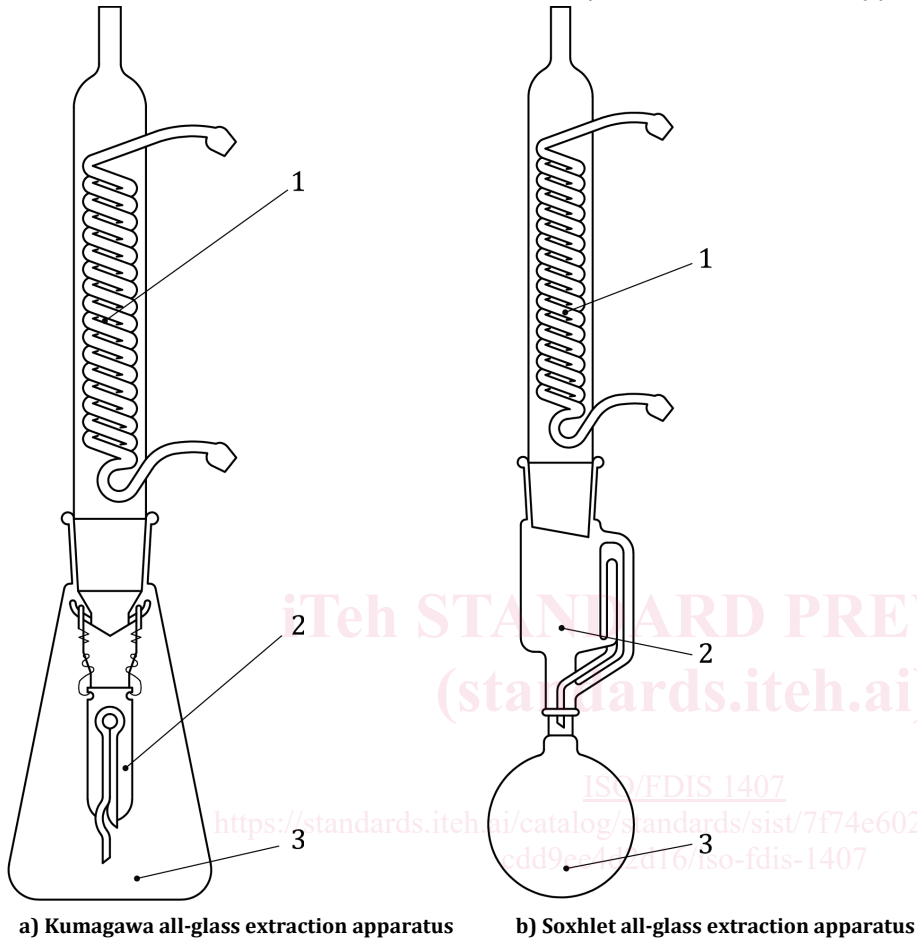
6.1 Balance, precision $\pm 0,1$ mg.

6.2 Extraction apparatus: Examples of suitable types of extraction apparatus are shown in [Figure 1](#). Any other type of apparatus which performs the same extraction function may be used, provided it can be demonstrated to give results which are the same as those obtained using the types of apparatus shown in [Figure 1](#). It is recommended that the ground joints be ungreased.

NOTE The apparatus used for method C is the same as that used for methods A, B and D, but does not have an extraction chamber.

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- Key**
- 1 condenser
 - 2 extraction chamber
 - 3 receiver flask

Figure 1 — Suitable types of extraction apparatus

- 6.3 **Regulated heating system**, with abilities of refluxing for the solvents.
- 6.4 **Rotary evaporator**, or any other suitable type of evaporation equipment.
- 6.5 **Drying equipment**, of the oven or vacuum desiccator type.
- 6.6 **Press**, available setting temperature and pressure.

7 Preparation of test pieces

7.1 Methods A and B

For raw rubber and unvulcanized compounds, select a laboratory sample in accordance with ISO 1795 and pass it at ambient temperature through a two-roll laboratory mill or a press to obtain sheets about 0,5 mm in thickness.

For vulcanized rubber, select a laboratory sample in accordance with ISO 4661-2. Since the efficiency of the extraction is a function of the solid-solvent contact area, comminute the laboratory sample, if necessary, to give fragments of maximum surface area 2 mm².

7.2 Method C (raw rubbers only)

7.2.1 Select a laboratory sample in accordance with ISO 1795 and prepare it by either the procedure specified in 7.2.2 or 7.2.3.

7.2.2 Pass the laboratory sample at ambient temperature through a two-roll laboratory mill or a press to obtain sheets of 0,5 mm or less in thickness.

The thickness of the sheets is important for the efficiency of the extraction. If it is difficult to obtain sheets of thickness 0,5 mm or less, the roll or press temperature may be increased to a maximum of 100 °C.

7.2.3 Comminute the laboratory sample to give fragments of maximum surface area 2 mm².

7.3 Method D (raw rubbers only)

Select a laboratory sample in accordance with ISO 1795. Press a test piece into chromium-nickel wire gauze and roll the gauze into a tube.

8 Procedure

8.1 General

Carry out the determination in duplicate.

8.2 Method A

8.2.1 Dry the empty receiver flask to constant mass and weigh it to the nearest 0,1 mg (m_1).

8.2.2 Weigh, to the nearest 0,1 mg, a test piece of 2 g to 5 g (m_0), depending on the amount of extract expected.

8.2.3 Place the weighed test piece in a cellulose extraction thimble or wrap it in a filter paper or in a woven wire cloth with a mesh suited to the nature and size of the fragments in the test piece (e.g. 150 µm or 100 mesh). These wrapping materials shall have been previously cleaned in the solvent to be used. Place the wrapped test portion in the extraction chamber of the apparatus.

8.2.4 Introduce a quantity of solvent equal to two or three times the extraction chamber volume into the receiver flask. Then assemble the extraction apparatus.

8.2.5 Carry out the extraction for 16 h ± 0,5 h (or for a shorter time if it can be demonstrated that the extraction is complete). Adjust the heating conditions during the extraction so that the solvent distils at a rate at which the extraction chamber is filled at least five times per hour.

8.2.6 At the end of the heating period, turn off the heater, allow the apparatus to cool, remove the extraction chamber from the apparatus and discard the rubber test portion unless it is required for further testing.

8.2.7 Using a rotary evaporator and/or other suitable equipment, remove the solvent from the extract until a constant mass is obtained (i.e. until the difference between two successive weighings at 30 min intervals is 0,2 % or less). Weigh the mass of the receiver flask plus extract to the nearest 0,1 mg (m_2), and record it.

The solvent may also be evaporated from the open flask by gentle heating on the heater used for the extraction.

CAUTION — This may be done only where local health and safety regulations permit and only in a well-ventilated fume cupboard.

8.2.8 Carry out a blank test, going through the entire procedure using the same type of apparatus and same quantity of solvent as for the test piece, but omitting the test piece.

8.3 Method B

8.3.1 Weigh, to the nearest 0,1 mg, a test piece of 0,5 g to 5 g (m_0), depending on the amount of extract expected.

8.3.2 Place the weighed test piece in a cellulose (CAS 9004-34-6) extraction thimble or wrap it in a filter paper or in a woven wire cloth with a mesh suited to the nature and size of the fragments in the test piece (e.g. 150 μm or 100 mesh). These wrapping materials shall have been previously cleaned in the solvent to be used. Place the wrapped test piece in the extraction chamber of the apparatus.

8.3.3 Introduce a quantity of solvent equal to two or three times the extraction chamber volume into the receiver flask. Then assemble the extraction apparatus.

8.3.4 Carry out the extraction for 16 h \pm 0,5 h (or for a shorter time if it can be demonstrated that the extraction is complete). Adjust the heating conditions during the extraction so that the solvent distils at a rate at which the extraction chamber is filled at least five times per hour.

8.3.5 At the end of the heating period, turn off the heater, allow the apparatus to cool, remove the extraction chamber from the apparatus and take out the test piece. Discard the solvent in an appropriate manner.

8.3.6 Dry the test piece in an oven to constant mass (i.e. until the difference between two successive weighings at 30 min intervals is 0,2 % or less) at a temperature higher than the boiling point of the solvent used. A drying time of about 1 h at 100 °C in a ventilated oven is usually sufficient.

CAUTION — For safety reasons, gently blot the test piece with absorbent tissue to remove excess solvent or air dry it in a drying chamber before placing it in the oven.

Allow the dried test piece to cool in a desiccator and weigh to the nearest 0,1 mg (m_3).

It is recommended that the dried test piece be checked to verify that oxidation has not occurred.

When the test piece cannot be weighed due to the collapsing of the test piece, try again with a new test piece or apply Method A.

NOTE In the presence of air, too high a drying temperature can cause degradation of the test piece and influence the result. The use of a vacuum lowers the boiling point of the solvent.

8.4 Method C

8.4.1 Cut the laboratory sample of raw rubber into sheets weighing between 90 mg and 110 mg.

8.4.2 Weigh, to the nearest 0,1 mg, a test piece of 200 mg to 600 mg (m_0), depending on the amount of extract expected.

8.4.3 Place the weighed test piece in a 150-ml to 300-ml receiver flask.

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8.4.4 Add 25 ml of solvent for each 100 mg of test piece. Connect the condenser to the receiver flask and reflux for 30 min.

8.4.5 At the end of the reflux period, allow the apparatus to cool and remove the receiver flask from the condenser. Decant the solvent and add the same amount of fresh solvent as was added in 8.4.4. Connect the condenser to the receiver flask and reflux for another 30 min.

8.4.6 At the end of the second reflux period, allow the apparatus to cool and remove the receiver flask from the condenser. Decant off the solvent and add the same amount of fresh solvent as was added in 8.4.4. Connect the condenser to the receiver flask and reflux for another 30 min.

8.4.7 At the end of the third reflux period, turn off the heater, allow the apparatus to cool and remove the receiver flask from the condenser. Pour the contents of the receiver flask into a clean-woven cloth with a mesh suited to the nature and size of the fragments in the test piece in order to recover the extracted test piece. Discard the solvent in an appropriate manner.

If particles are observed to be passing through the cloth, either repeat the whole procedure from 8.4.1 to 8.4.7 using a more suitable cloth, or use method A or B.

If the mass of the cloth is known, the following drying and weighing operations can be carried out with the test piece on the cloth.

8.4.8 Dry the test piece in an oven to constant mass (i.e. until the difference between two successive weighings at 30 min intervals is 0,2 % or less) at a temperature higher than the boiling point of the solvent used. A drying time of about 1 h at 100 °C in a ventilated oven is usually sufficient.

CAUTION — For safety reasons, gently blot the test piece with absorbent tissue to remove excess solvent or air-dry it in a drying chamber before placing it in the oven.

Allow the dried test piece to cool in a desiccator and weigh to the nearest 0,1 mg (m_4).

It is recommended that the dried test piece be checked to verify that oxidation has not occurred.

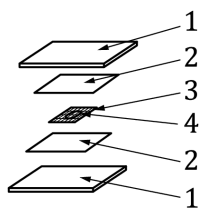
NOTE In the presence of air, too high a drying temperature can cause degradation of the test piece and influence the result. The use of a vacuum lowers the boiling point of the solvent.

8.5 Method D

8.5.1 Weigh a test piece of 0,5 g \pm 0,05 g to the nearest 0,1 mg.

8.5.2 Prepare the chromium nickel gauze¹ (CAS 11106-97-1) tube in the following way.

a) Clean the gauzes in the chosen solvent for 8 h and dry them in an oven maintained at 105 °C \pm 5 °C for 1 h. Weigh the gauze to the nearest 0,1 mg (m_7) and prepare a press assembly as shown in Figure 2, taking care that the test piece is in the centre gauze.



Key

- 1 press plate
- 2 polyester (CAS 23038-59-9) film or polytetrafluoroethylene (CAS 9002-84-0) film

¹ Chromium nickel gauze (woven wire cloth), dimensions: 80 mm \times 80 mm; meshes per 25,4 mm: 100; nominal aperture size: 0,14 mm; wire diameter: 0,11 mm.

- 3 chromium nickel gauze
4 test piece

Figure 2 — Press assembly for method D

- b) Press at 150 °C ± 5 °C for 10 s at 130 kN.
c) Remove from the press and cool to room temperature.
d) Weigh the gauze and test piece to the nearest 0,1 mg (m_5).
e) Carefully roll the gauze into a tube form.
f) Close one end of the tube by folding it, so it does not unroll.

8.5.3 Place the gauze tube in the Soxhlet extractor.

8.5.4 Introduce 125 ml of solvent into the receiver flask. Then assemble the system.

8.5.5 Carry out the extraction for a length of time that allows a sufficient number of extraction cycles to take place. The number of extraction cycles and/or extraction time can be different for each raw rubber plus solvent combination and the heating equipment used and shall be determined beforehand. See the example given in [Annex C](#) for ethylene-propylene-diene (EPDM) [CAS 25038-36-2] in 2-butanone (CAS 78-93-3).

Extraction starts the moment the extraction liquid starts to boil.

8.5.6 At the end of the extraction, turn off the heating system and allow the apparatus to cool. Discard the solvent in an appropriate manner.

8.5.7 Take out the gauze tube from the extractor and dry it in an oven to constant mass at a temperature higher than the boiling point of the solvent used, i.e. until the difference between two successive weighings at 30 min intervals is 0,2 % or less. A drying time of about 1 h at 100 °C in a ventilated oven is usually sufficient. Cool the gauze tube in a desiccator at room temperature for 15 min ± 1 min and weigh to the nearest 0,1 mg (m_6).

CAUTION — For safety reasons, gently blot the test piece with absorbent tissue to remove excess solvent or air-dry it in a drying chamber before placing it in the oven.

NOTE In the presence of air, too high a drying temperature can cause degradation of the test piece and influence the result. The use of a vacuum lowers the boiling point of the solvent.

9 Calculation and expression of results

9.1 Method A

The amount of solvent-extractable material extracted, w_{ex1} , expressed as a percentage mass fraction, is given by Formula (1):

$$w_{ex1} = \frac{(m_2 - m_1) - (m'_2 - m'_1)}{m_0} \times 100 \quad (1)$$

where

- m_0 is the mass, in grams, of the test piece;
 m_1 is the mass, in grams, of the empty receiver flask;
 m_2 is the mass, in grams, of the receiver flask plus extract.