
**Rubber — Determination of solvent
extract**

Caoutchouc — Détermination de l'extrait par les solvants

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

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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 1407 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 1407:2009), which has been technically revised with the addition of a Method D for raw rubbers only.

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Rubber — Determination of solvent extract

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.

IMPORTANT — 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 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 compounds of raw rubbers.

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 the fact that an equilibrium 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 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 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 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.

4 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 International Standard should consult the safety data sheet for the solvent before its use and take appropriate measures.

5 Apparatus

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

5.2 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.

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

5.3 Regulated heating system.

5.4 Rotary evaporator or any other suitable type of evaporation equipment.

5.5 Drying equipment, of the oven or vacuum desiccator type.

6 Preparation of test pieces

6.1 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.

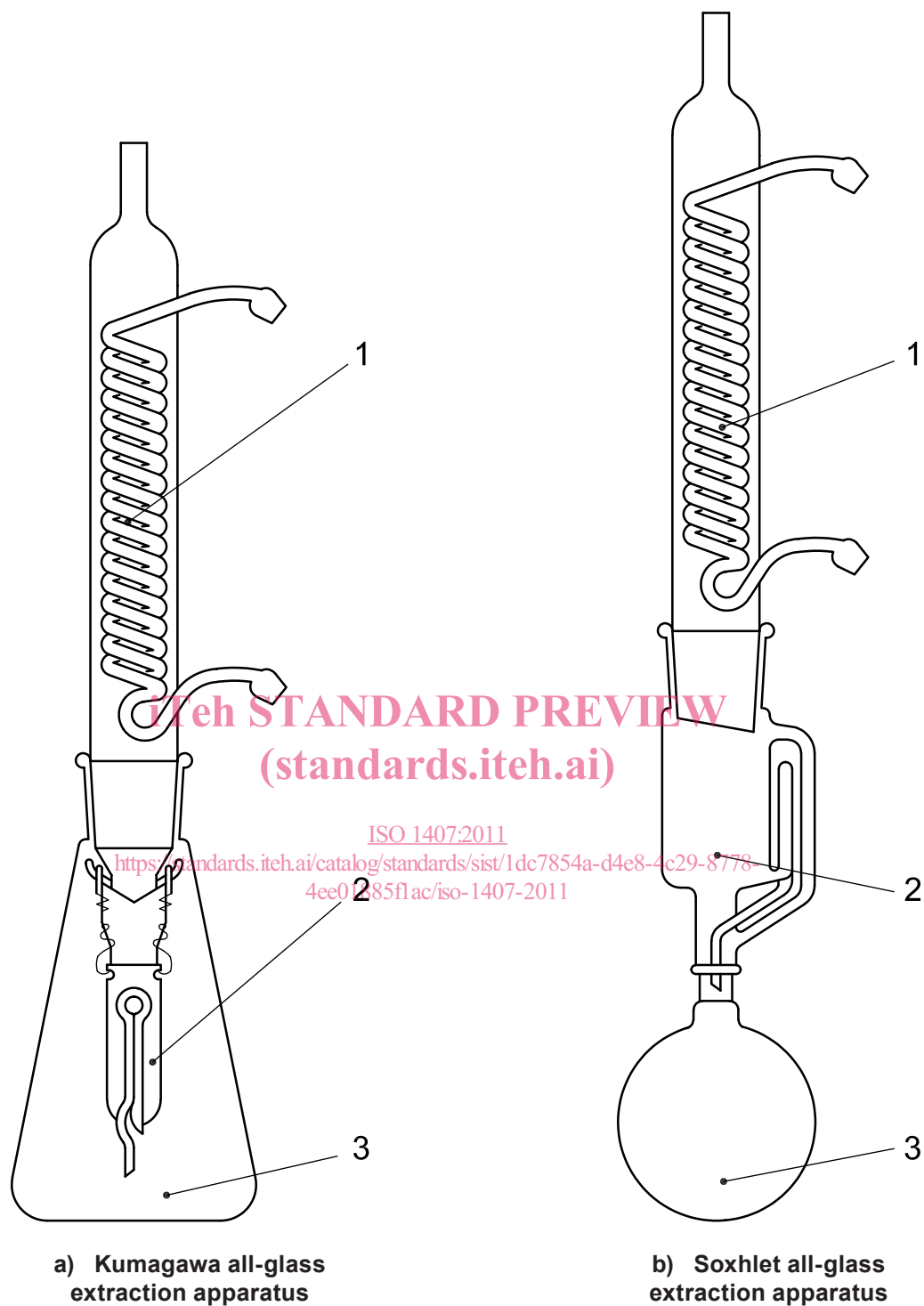
6.2 Method C (raw rubbers only)

6.2.1 Select a laboratory sample in accordance with ISO 1795 and prepare it by either procedure 6.2.2 or procedure 6.2.3.

6.2.2 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.

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

**Key**

- 1 condenser
- 2 extraction chamber
- 3 receiver flask

It is recommended that the ground joints be ungreased.

Figure 1 — Suitable types of extraction apparatus

6.3 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.

7 Procedure

7.1 General

Carry out the determination in duplicate.

7.2 Method A

7.2.1 Dry the empty receiver flask to constant mass and weigh it (m_1).

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

7.2.3 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.

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

7.2.5 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 cup is filled at least five times per hour.

7.2.6 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.

7.2.7 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). Record the mass of the receiver flask plus extract (m_2).

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.

7.2.8 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.

7.3 Method B

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

7.3.2 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 piece in the extraction chamber of the apparatus.

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

7.3.4 Carry out the extraction for $16 \text{ h} \pm 0,5 \text{ h}$ (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 cup is filled at least five times per hour.

7.3.5 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.

7.3.6 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 $100 \text{ }^\circ\text{C}$ 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.

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.

7.4 Method C

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

7.4.2 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.

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

7.4.4 Add 25 cm^3 of solvent for each 100 mg of test piece. Connect the condenser to the receiver flask and reflux for 30 min.

7.4.5 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 7.4.4. Connect the condenser to the receiver flask and reflux for another 30 min.

7.4.6 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 7.4.4. Connect the condenser to the receiver flask and reflux for another 30 min.

7.4.7 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 7.4.1 to 7.4.7 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.

7.4.8 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.

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

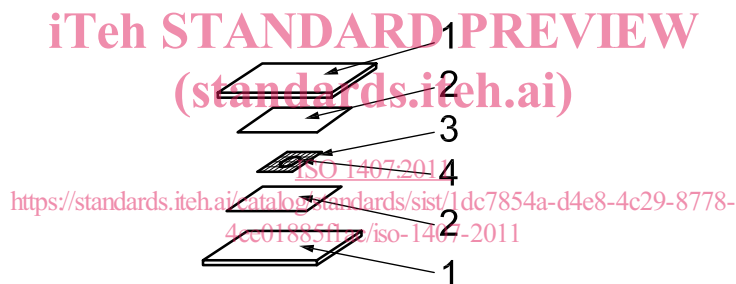
NOTE In the presence of air, too high a drying temperature can cause degradation of the test piece and influence the result. The use of a vacuum lowers the boiling point of the solvent.

7.5 Method D

7.5.1 Weigh a test piece of 0,5 g ± 0,05 g to the nearest 1 mg.

7.5.2 Prepare the chromium nickel gauze¹⁾ 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 ± 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 film
 - 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.

7.5.3 Place the gauze tube in the Soxhlet extractor.

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

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

7.5.5 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 may 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) in 2-butanone.

Extraction starts the moment the extraction liquid starts to boil.

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

7.5.7 Take out the gauze tube from the extractor and dry it in an oven to constant mass (m_6) 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 \pm 1 min.

8 Calculation and expression of results

8.1 Method A

The amount of solvent-extractable material extracted, w_{ex1} , expressed as a percentage mass fraction, is given by the equation:

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

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;

m'_1 is the mass, in grams, of the empty receiver flask in the blank test;

m'_2 is the mass, in grams, of the receiver flask in the blank test after the extraction.

8.2 Method B

The amount of solvent-extractable material extracted, w_{ex2} , expressed as a percentage mass fraction, is given by the equation:

$$w_{\text{ex2}} = \frac{m_0 - m_3}{m_0} \times 100$$

where

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

m_3 is the mass, in grams, of the test piece after the extraction.