INTERNATIONAL
STANDARD

ISO
1407

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Rubber — Determination of solvent extract

Caoutchouc — Détermination de l'extrait par les solvants

Reference number
ISO 1407:1992(E)

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.

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.

International Standard ISO 1407 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This second edition cancels and replaces the first edition (ISO 1407:1976), of which it constitutes a technical revision.

Annex A of this International Standard is for information only.

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Rubber — Determination of solvent extract

1 Scope

This International Standard specifies two methods for the quantitative determination of extractable material from raw rubbers, both natural and synthetic, and their unvulcanized and vulcanized compounds.

Method B is a rapid extraction method, while method A is for use in cases of dispute.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 383:1976, *Laboratory glassware — Interchangeable conical ground joints.*

ISO 1629:1987, *Rubber and latices — Nomenclature.*

ISO 1795:—[1], *Rubber, raw, natural and synthetic — Sampling and further preparative procedures (Combination and revision of ISO 1795:1974 and ISO 1796:1982, incorporating ISO/TR 2630:1978).*

ISO 4661-2:1987, *Rubber, vulcanized — Preparation of samples and test pieces — Part 2: Chemical tests.*

3 Principle

3.1 Method A

Extraction of an individual weighed test portion of the rubber is carried out in a Soxhlet-type apparatus with the appropriate solvent. The solvent is then removed from the extract solution by distillation or evaporation, followed by drying and weighing of the residue.

3.2 Method B

Extraction of a series of weighed test portions of a set of rubbers is carried out by boiling in the appropriate solvent. The solvent is then removed from the extracted rubbers by blotting and heating in an oven, followed by cooling and weighing of the separated test portions.

NOTE 1 For a complete description of method B, which is beyond the scope of this International Standard, the user is referred to reference [1] in annex A.

4 Limitations

4.1 These methods are applicable to the rubbers given in table 1.

4.2 The solvents recommended for individual rubbers are listed in table 1, although other combinations may be desirable in certain cases. For instance, alcohols are particularly useful for extraction of vulcanized IR, NR and SBR when the extract will be used for thin-layer chromatography and not primarily for quantitative determination of the amount of extractables.

4.3 The same quantitative results may not necessarily be obtained with either method A or method B, nor with the different solvents.

Method B generally gives results which are lower than those obtained with method A due to an equilibrium being set up, particularly if large test portions are used.

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[1] To be published.

Table 1 — Recommended solvents

Rubber[1]	Raw rubbers and unvulcanized compounds	Vulcanizates
IR, NR	acetone[2]	acetone[2]
SBR	ETA	ETA
CR	propan-2-ol	methanol
NBR	propan-2-ol	propan-2-ol
IIR	acetone[2]	acetone[2]

[1] For the meanings of the rubber abbreviations, see ISO 1629.

[2] When acetone is used for the extraction, it may be partially converted into high-boiling diacetone alcohol. If a difficult evaporation of the solvent is observed during the drying step, due to the presence of diacetone alcohol, repeat the extraction using a different solvent or use method B.

5 Reagents

WARNING — All recognized health and safety precautions should be taken when using the reagents and procedures specified in this International Standard.

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

5.1 Acetone.

5.2 Ethanol/toluene azeotropic mixture (ETA), a mixture of 70 volumes of ethanol and 30 volumes of toluene. Reflux for 4 h over freshly calcined calcium oxide. Distill and collect a middle fraction with a boiling range of not more than 1 °C. If absolute ethanol is used, the drying over calcium oxide may be omitted.

5.3 Propan-2-ol (Isopropanol).

5.4 Methanol.

6 Apparatus

6.1 Extraction apparatus.

NOTE 2 Three types of extraction apparatus are described. Any other type of apparatus which performs the same function may be used.

a) The type 1 apparatus comprises a receiver flask, a jacketed Soxhlet extractor and a condenser as shown in figure 1.

b) The type 2 apparatus comprises a $250\ \text{cm}^3$ receiver flask, a condenser and an extraction cup suspended by clean wire as shown in figure 2.

c) The type 3 apparatus comprises a $250\ \text{cm}^3$ receiver flask, a condenser and a Soxhlet extractor with a side arm as shown in figure 3. Typical Soxhlet capacity is $100\ \text{cm}^3$ to $200\ \text{cm}^3$. There is no extraction cup.

6.2 Distillation head and **condenser** (not shown in the figures), appropriate for the apparatus, to distil off the solvent after extraction.

6.3 Oven, operating at 100 °C ± 2 °C.

6.4 Filter paper or **nylon filter cloth**, extracted before use with the same solvent as that used for the extraction.

6.5 150 µm **sieve** (100 mesh).

7 Sampling

For both methods A and B, select a sample of raw rubber in accordance with ISO 1795 and pass it through the cold rolls of a laboratory mill set to a nip not exceeding 0,5 mm. Select a sample of vulanized rubber in accordance with ISO 4661-2.

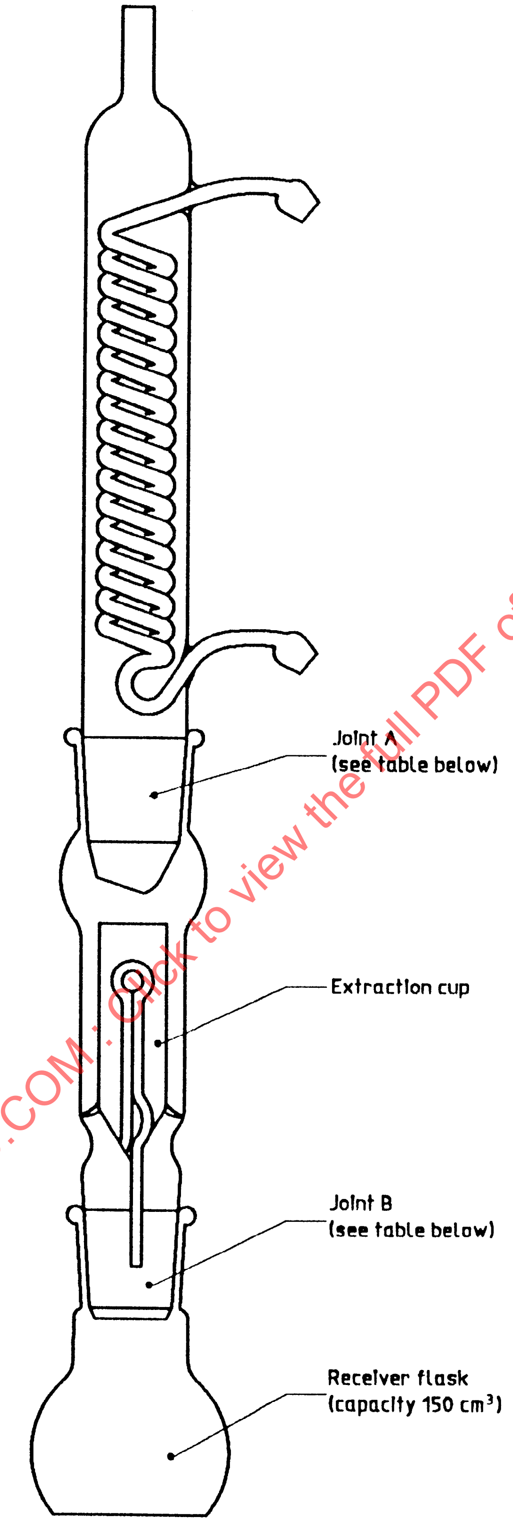
NOTE 3 Results may be different depending on the sample preparation, which influences the ability of the solvent to swell the rubber and dissolve out the extractables.

8 Procedure

8.1 Method A

8.1.1 Take duplicate test portions from the sample prepared in accordance with clause 7. Take a test portion of about 3 g to 5 g and weigh it to the nearest 0,1 mg (mass m_0).

8.1.2 Dry the chosen flask (see 6.1) in the oven (6.3) at 100 ° C ± 2 °C. Remove the flask from the oven and allow it to cool to room temperature in a desiccator. Weigh to the nearest 0,1 mg (mass m_1).



Extraction cup capacity	Joint[1]		Receiver capacity
cm^3	A	B	cm^3
15 to 30 50 to 60	34/35 45/40	24/29 34/35	150 250

[1] See ISO 383.

Figure 1 — Type 1 all-glass extraction apparatus

Dimensions in millimetres

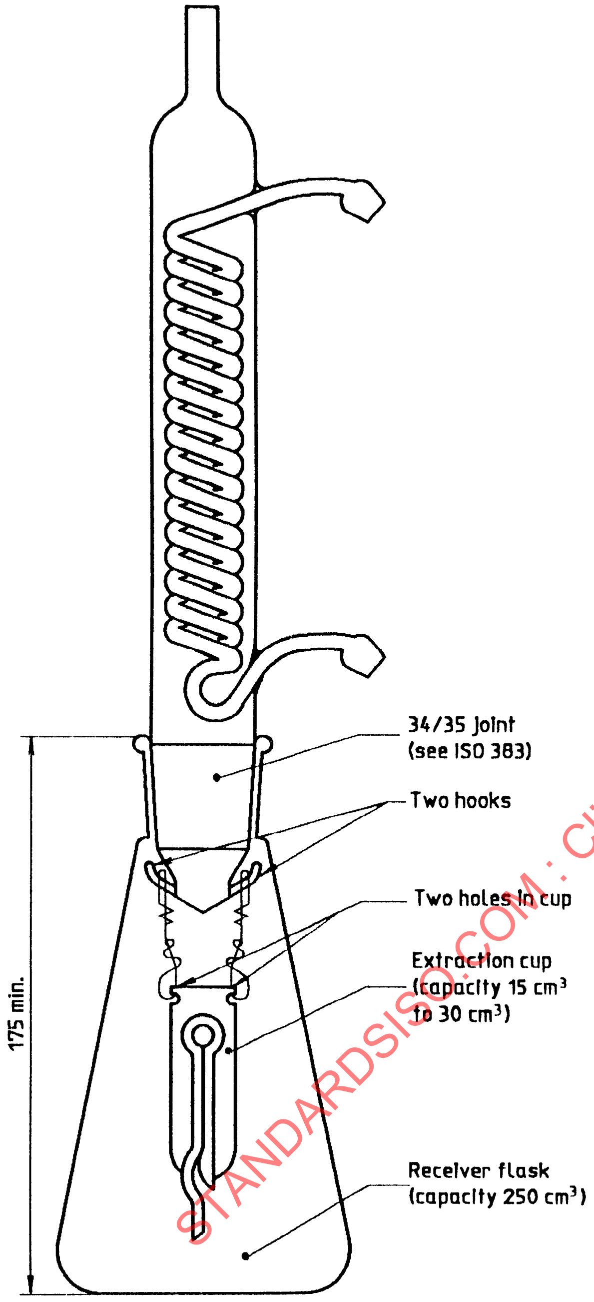


Figure 2 — Type 2 all-glass extraction apparatus

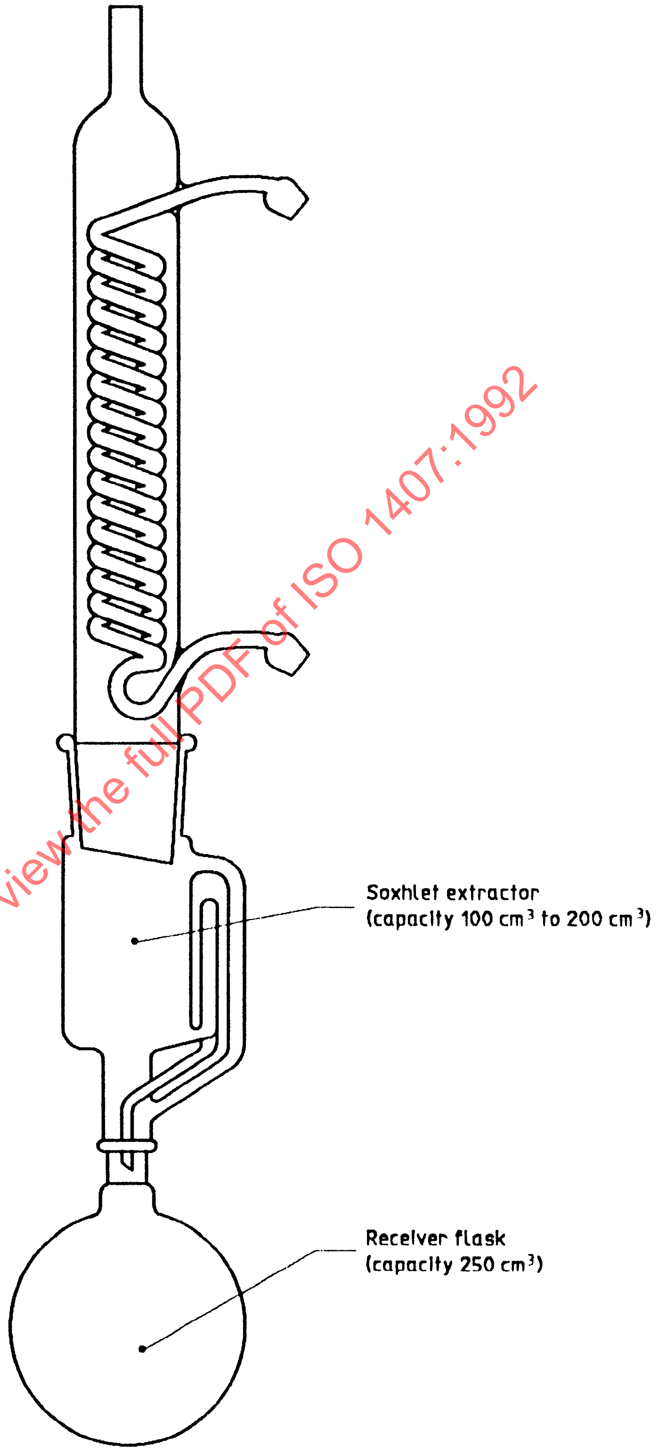


Figure 3 — Type 3 all-glass extraction apparatus

8.1.3 Roll the weighed test portion in filter paper or nylon filter cloth (6.4) to form a loose roll from which the rubber cannot fall and so that no part of the rubber is anywhere in contact with any other part of the rubber. If the test portion is in the form of small pieces, make a loose packet of the pieces in filter paper or nylon filter cloth. Fasten each test packet with clean wire. Place the packet in the appropriate extraction apparatus (see figures 1 to 3), adjust the heating rate so that the quantity of distilled solvent fills the extraction cup 10 to 20 times per hour and extract for 16 h ± 0,5 h.

8.1.4 At the end of the heating period, turn off the heating device, allow the apparatus to cool, remove the extractor or siphon cup and discard the rubber test portion unless it is required for further testing.

8.1.5 Remove the receiver flask, fit it with the distillation head and condenser (6.2) and distil off the bulk of the solvent into a suitable flask, retaining about 0,5 cm height of solvent in the flask. Alternatively, a rotary evaporator may be used. Discard the distillate unless it is required for further testing.

CAUTION — The solvent may also be evaporated from the open flask by gentle heating with the heating device. This may, however, be done only where local health and safety regulations permit and only in a well ventilated fume cupboard.

8.1.6 Dry the flask and contents for 2 h at 100 °C± 2 °C in the oven and, at the end of this time, remove the flask from the oven, cool in a desiccator and weigh to the nearest 0,1 mg (mass m_2).

8.1.7 Carry a blank through the entire procedure, using the same type of apparatus and quantity of solvent as for the test portion, but omitting the test portion. (Increase in mass, m_3.)

8.2 Method B

8.2.1 Take duplicate test portions from the sample prepared in accordance with clause 7. Cut test portions weighing between 90 mg and 110 mg into identifying shapes. Weigh to the nearest 0,1 mg (mass m_4).

8.2.2 Assemble the extraction apparatus (see figures 1 to 3) but connect only the condenser to the flask. No Soxhlet, extractor cup or siphon cup is necessary — only the receiving flask and the condenser. The test portion is placed directly in the solvent.

Alternatively, any arrangement for boiling solvent and test portions can be used, e.g. a beaker covered with a watch glass, a beaker covered with another beaker containing cold water, etc. The solvent volume shall be held constant, and may be replenished with fresh solvent should evaporation occur.

8.2.3 Add about 25 cm^3 of solvent for each test portion. Use a maximum of six test portions in a 250 cm^3 receiver flask and four test portions in a 150 cm^3 flask. Reflux for 60 min.

8.2.4 At the end of the heating period, turn off the heating device, allow the apparatus to cool, then remove the receiver flask from the condenser. Pour the contents of the flask into a clean 150 µm sieve (6.5) to recover the extracted test pieces. Discard the solvent in an appropriate manner.

The appearance of small particles of rubber on the sieve indicates loss of part of the test portion. In this case, fresh test portions shall be taken and the analysis repeated.

8.2.5 Gently blot the extracted test portions between absorbent tissue to remove excess solvent and dry them separately at 100 °C ± 2 °C for 30 min or until the mass does not change by more than ± 0,1 mg over a period of 10 min.

8.2.6 Cool the test portions for about 10 min in a desiccator and weigh each to the nearest 0,1 mg (mass m_5).

9 Expression of results

9.1 Method A

The solvent-extractable material, expressed as a percentage by mass, is given by the formula

$$\frac{m_2 - m_1 - m_3}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion (8.1.1);

m_1 is the mass, in grams, of the empty receiver flask (8.1.2);

m_2 is the mass, in grams, of the receiver flask plus the extract after drying (8.1.6);

m_3 is the increase in mass, in grams, of a receiver flask during the blank test (8.1.7).

9.2 Method B

The solvent-extractable material, expressed as a percentage by mass, is given by the formula

$$\frac{m_4 - m_5}{m_4} \times 100$$

where

m_4 is the mass, in milligrams, of the test portion before extraction (8.2.1);

m_5 is the mass, in milligrams, of the test portion after extraction (8.2.6).

9.3 Test result

A test result is the average of two determinations for either method A or method B.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard;

b) full identification of the rubber tested;

c) the method of sample preparation;

d) whether method A or method B was used;

e) which solvent was used;

f) which apparatus was used for extraction;

g) the mean of two determinations.

Annex A
(informative)

Bibliography

[1] Kress, K.E., *Rubber World,* August 1956, pp. 709-717, "Semimicro rapid reflux method for solvent extraction of rubber products".