

## INTERNATIONAL STANDARD



1407

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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

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**Rubber — Determination of solvent extract***Caoutchouc — Détermination de l'extrait par les solvants*

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**Descriptors** : rubber, natural rubber, synthetic elastomers, chemical analysis, measurement, solubility.

## FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

Prior to 1972, the results of the work of the technical committees were published as ISO Recommendations; these documents are in the process of being transformed into International Standards. As part of this process, Technical Committee ISO/TC 45, *Rubber and rubber products*, has reviewed ISO Recommendation R 1407-1971 and found it technically suitable for transformation. International Standard ISO 1407 therefore replaces ISO Recommendation R 1407-1971, to which it is technically identical.

ISO Recommendation R 1407 had been approved by the member bodies of the following countries :

|                     |             |                |
|---------------------|-------------|----------------|
| Australia           | Hungary     | Spain          |
| Austria             | India       | Sweden         |
| Brazil              | Iran        | Switzerland    |
| Cuba                | Israel      | Thailand       |
| Czechoslovakia      | Italy       | Turkey         |
| Egypt, Arab Rep. of | Netherlands | United Kingdom |
| France              | New Zealand | U.S.A.         |
| Germany             | Poland      | U.S.S.R.       |

The member body of the following country had expressed disapproval of the Recommendation on technical grounds :

Canada\*

- \* Subsequently, this member body approved the Recommendation.

The member body of the following country disapproved the transformation of the Recommendation into an International Standard :

Germany

# Rubber — Determination of solvent extract

## 1 SCOPE AND FIELD OF APPLICATION

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

The method is applicable only to those rubbers listed in the table.

## 2 PRINCIPLE

Extraction of a weighed test portion of the rubber with the appropriate solvent (stated in the table), in a suitable apparatus. Distillation of the solvent, followed by drying and weighing of the residue.

## 3 REAGENTS

Solvents of recognized analytical quality shall be used.

TABLE — Recommended solvents

| Elastomer                      | Solvent for raw rubbers and unvulcanized compounds | Solvent for vulcanizate |
|--------------------------------|----------------------------------------------------|-------------------------|
| Natural rubber                 | Acetone <sup>1)</sup>                              | Acetone <sup>1)</sup>   |
| SBR <sup>3)</sup>              | ETA <sup>2)</sup>                                  | Acetone <sup>1)</sup>   |
| Oil-extended SBR               | ETA <sup>2)</sup>                                  | Acetone <sup>1)</sup>   |
| Chloroprene rubber             | Isopropanol                                        | Methanol                |
| Butadiene acrylonitrile rubber | Isopropanol                                        | Isopropanol             |
| Butyl rubber                   | Butanone (MEK)                                     | Butanone (MEK)          |

1) Acetone (boiling point 56 to 57 °C) freshly distilled from dry sodium carbonate (Na<sub>2</sub>CO<sub>3</sub>) or potassium carbonate (K<sub>2</sub>CO<sub>3</sub>).

2) Mixture of 70 volumes of ethanol and 30 volumes of toluene. Reflux for 4 h over freshly calcined calcium oxide. Distil 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.

3) With the exception of unvulcanized alum-coagulated rubbers.

## 4 APPARATUS

4.1 All-glass extraction apparatus : to be preferred (see figures 1 and 2), or

4.2 Metal condenser extraction apparatus (see figure 3).

## 5 PROCEDURE

5.1 Pass the rubber six times between the rolls of a laboratory mill set to a nip not exceeding 0,5 mm. Cut from the sheet a test portion having an estimated mass of 2 to 5 g, depending on the material to be tested, and weigh to the nearest 0,01 g. If it is not possible to pass the sample through the mill, the sample may be cut into pieces less than 1 mm per side. The results obtained may be different depending on the method of sample preparation.

5.2 Roll the weighed test portion in filter paper or nylon cloth (previously extracted with the solvent used) 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.

5.3 Place the roll in the extraction cup of the appropriate extraction apparatus and pour into the extraction flask sufficient solvent to fill the extraction cup two or three times.

5.4 Assemble the apparatus and adjust the rate of heating so that the distilled quantity of solvent will fill the extraction cup ten to twenty times per hour. The extraction time shall be 16 ± 0,5 h.

5.5 Evaporate off the solvent in a vessel, preferably in the extraction flask, weighed to the nearest 0,001 g, at about 100 °C.

5.6 Dry the flask for 2 h at 100 ± 2 °C in an oven. Cool in a desiccator and weigh to the nearest 0,001 g.

6 EXPRESSION OF RESULTS

The solvent extract is given, as a percentage by mass, by the formula

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

where

- $m_0$  is the mass, in grams, of the test portion;
- $m_1$  is the mass, in grams, of the weighed vessel or extraction flask and extract;
- $m_2$  is the mass, in grams, of the weighed vessel or extraction flask.

7 TEST REPORT

The test report shall include the following particulars :

- a) reference to this International Standard;
- b) identification of sample;
- c) method of sample preparation;
- d) solvent used;
- e) type of extraction apparatus;
- f) result obtained.

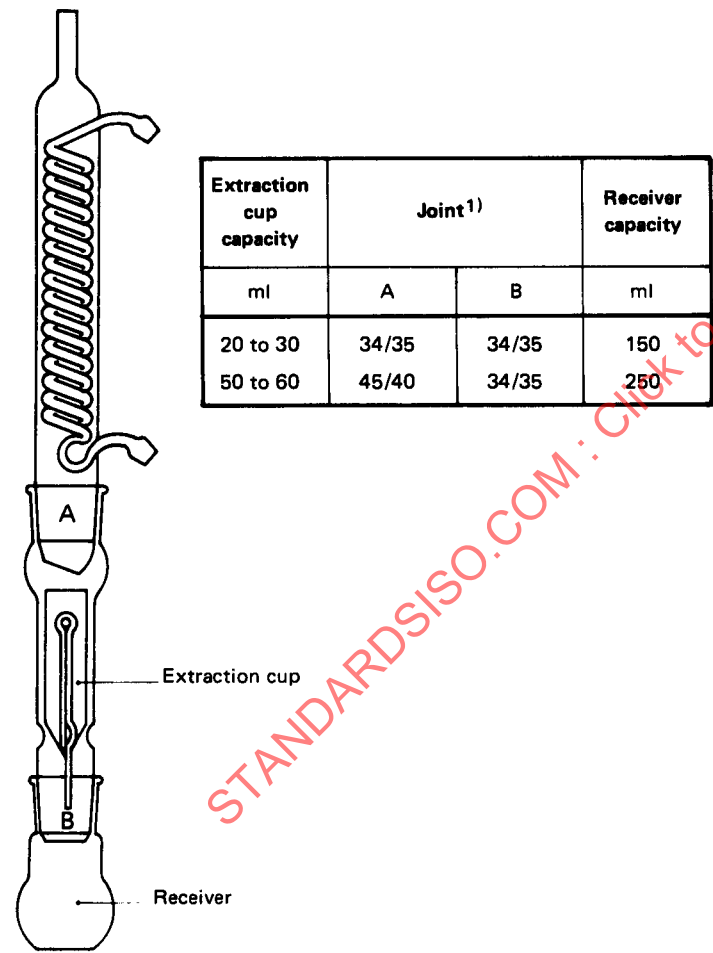


FIGURE 1 – All-glass extraction apparatus

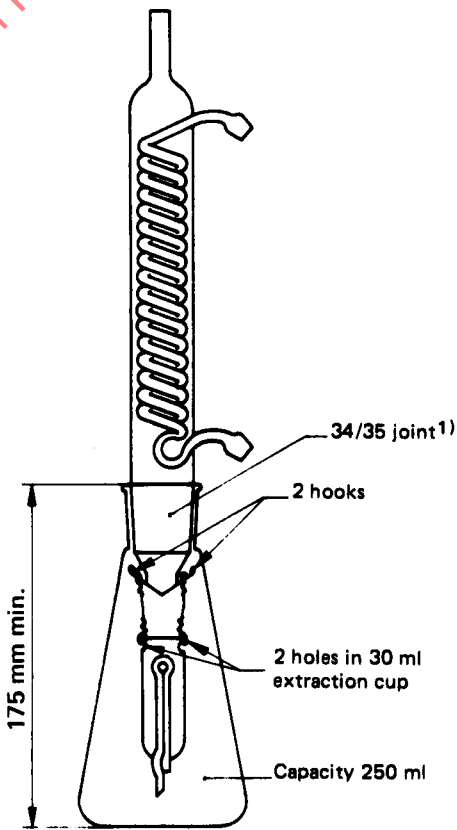


FIGURE 2 – All-glass extraction apparatus

1) See ISO 383, Laboratory glassware – Interchangeable conical ground joints.

Dimensions in millimetres

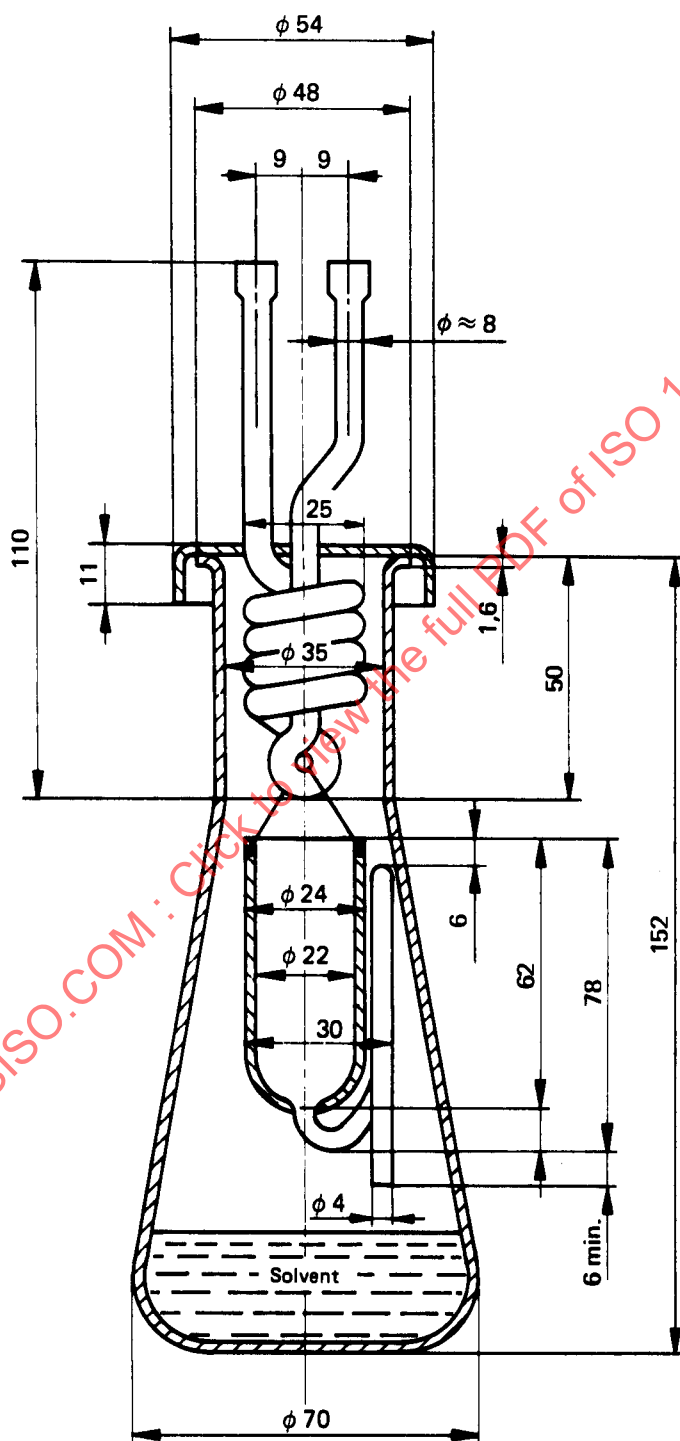


FIGURE 3 – Metal condenser extraction apparatus

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