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R 1407

RUBBER

DETERMINATION OF SOLVENT EXTRACT

1st EDITION

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For each individual country the only valid standard is the national standard of that country.

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BRIEF HISTORY

The ISO Recommendation R 1407, *Rubber – Determination of solvent extract*, was drawn up by Technical Committee ISO/TC 45, *Rubber*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of Draft ISO Recommendation No. 1407, which was circulated to all the ISO Member Bodies for enquiry in July 1968.

The Draft was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

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

The following Member Body opposed the approval of the Draft :

Canada

This Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided to accept it as an ISO RECOMMENDATION.

ISO Recommendation | R 1407 | November 1971

RUBBER

DETERMINATION OF SOLVENT EXTRACT

1. SCOPE

This ISO Recommendation describes 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

A weighed test portion of the rubber is extracted with the appropriate solvent (stated in the Table), in a suitable apparatus. The solvent is distilled off and the residue is dried and weighed.

3. REAGENTS

Solvents of recognized analytical quality should be used.

TABLE – Recommended solvents

Elastomer	Solvent for raw rubbers and unvulcanized compounds	Solvent for vulcanizate
Natural rubber	Acetone*	Acetone*
SBR***	ETA**	Acetone*
Oil-extended SBR	ETA**	Acetone*
Chloroprene rubber	Isopropanol	Methanol
Butadiene acrylonitrile rubber	Isopropanol	Isopropanol
Butyl rubber	Butanone (MEK)	Butanone (MEK)

* Acetone (boiling point 56 to 57 °C) freshly distilled from dry sodium carbonate (Na_2CO_3) or potassium carbonate (K_2CO_3).

** Mixture of 70 volumes of ethanol and 30 volumes of toluene. Reflux for 4 hours 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.

*** With the exception of unvulcanized alum coagulated rubbers.

4. APPARATUS

One of the following may be used :

4.1 *All glass extraction apparatus* : to be preferred (see Figures 1 and 2).

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 with 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 10 to 20 times per hour. The extraction time should be 16 ± 0.5 hours.

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 hours at 100 ± 2 °C in an oven. Cool in a desiccator and weigh to the nearest 0.001 g.

6. EXPRESSION OF RESULTS

Calculate the percentage of extract from the following formula :

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

where

m_0 is the mass, in grammes, of the test portion;

m_1 is the mass, in grammes, of the weighed vessel or extraction flask and extract;

m_2 is the mass, in grammes, of the weighed vessel or extraction flask.

7. TEST REPORT

The test report should include the following particulars :

(*a*) identification of sample;

(*b*) sample preparation;

(*c*) solvent used;

(*d*) type of extraction apparatus;

(*e*) result obtained.