IS : 4518 (Part II) - 1971

Indian Standard

METHODS OF TEST FOR STYRENE-BUTADIENE RUBBERS

PART II DETERMINATION OF SOLVENT EXTRACT AND OIL CONTENT

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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METHODS OF TEST FOR STYRENE-BUTADIENE RUBBERS

PART II DETERMINATION OF SOLVENT EXTRACT AND OIL CONTENT

0. FOREWORD

0.1 This Indian Standard (Part II) was adopted by the Indian Standards Institution on 29 October 1971, after the draft finalized by the Rubber Sectional Committee had been approved by the Chemical Division Council.

0.2 Part I of this standard covers the methods of test for determination of the following:

a) Volatile matter by mill method (SBR : 1) and mill oven method (SBR : 2),
b) Total ash (SBR : 3),
c) Organic acid content (SBR : 4),
d) Soap content (SBR : 5),
e) Antioxidants (SBR : 6),
f) Bound styrene (SBR : 7), and
g) Mooney viscosity (SBR : 8).

0.3 The methods prescribed in this standard take into consideration the recommendations of International Synthetic Rubber Producers Association. Besides, assistance has also been derived from ASTM Designation D 1416-67 T ' Chemical analysis of synthetic elastomers (solids tyrene-butadiene copolymers) ' issued by the American Society for Testing and Materials and Draft ISO Recommendation No. 1407 ' Determination of solvent extract in natural and synthetic rubber ' issued by International Organization for Standardization.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

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*Rules for rounding off numerical values (*revised*).

1. SCOPE

1.1 This standard prescribes the methods of test for determining solvent extract (SBR : 9) and oil content (SBR : 10) of raw styrene-butadiene rubbers.

2. QUALITY OF REAGENTS

2.1 Unless otherwise, specified 'pure chemicals' and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the result of analysis.

3. METHOD FOR TAKING OUT SAMPLES

3.1 The method for taking out samples shall be in accordance with the method prescribed in **3** of IS : 4518 (Part I)-1967†.

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*Specification for water, distilled quality (*revised*).

†Methods of tests for styrene-butadiene rubbers (SBR): Part I.

DETERMINATION OF SOLVENT EXTRACT
(SBR : 9)

0. GENERAL

0.1 Two methods are prescribed here. Method A is based on the method being considered by ISO/TC 45 Rubber, and is recommended as a reference method. Method B is an alternate method and is recommended for routine analysis.

1. SCOPE

1.1 This method is intended to determine the various organic constituents in the rubber as solvent extract. It will give the combined amount of rosin and fatty acids, soaps, oil extenders, defoamer tars, antioxidants and other uncombined organic constituents.

NOTE — The rubber hydrocarbon can also be estimated by subtracting the sum of the ethanol-toluene azeotrope (ETA) extract, the total ash, and the volatile matter from 100.

METHOD A

2. OUTLINE OF THE METHOD

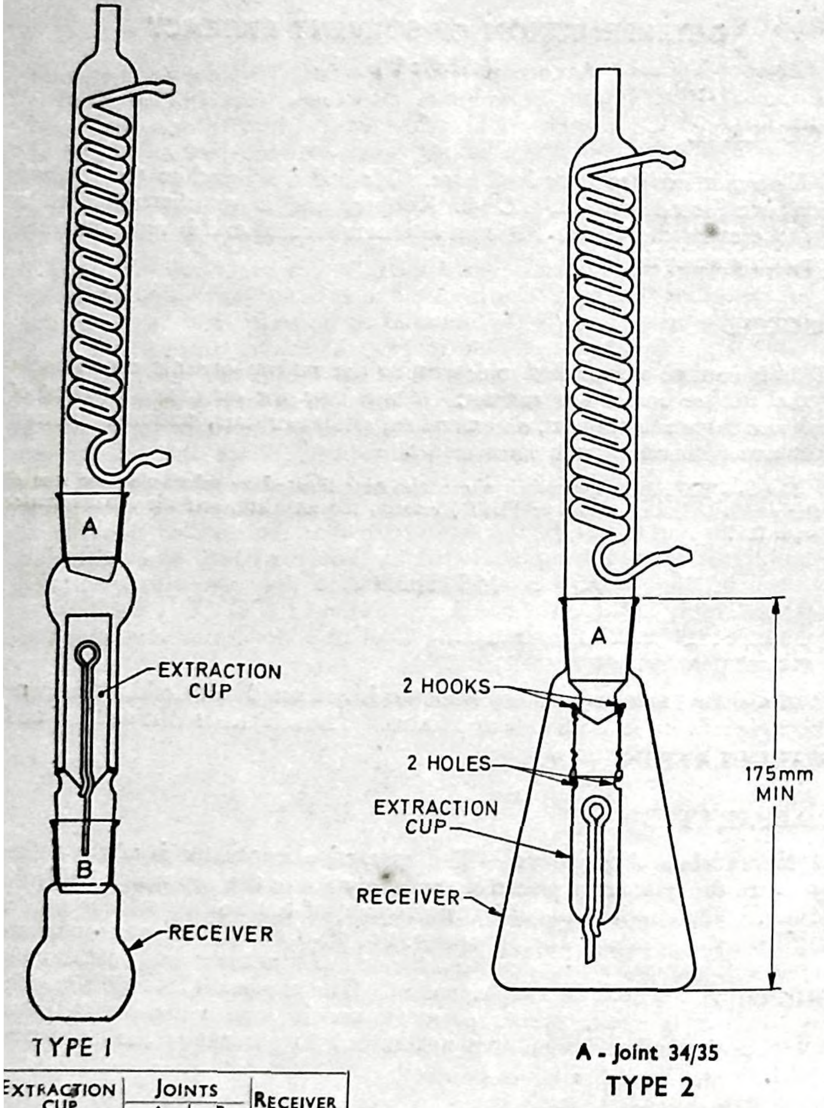
2.1 A weighed test portion of the rubber is extracted with ethanol-toluene azeotrope solvent in a suitable apparatus. The solvent is distilled off and the residue is dried and weighed.

3. APPARATUS

3.1 Extraction Apparatus — The extraction apparatus is of the reflux type with the condenser placed immediately above the cup which holds the rubber. The cup is situated in the vapour of the boiling solvent and is emptied by a siphon. The apparatus is of glass except in patterns where an extraction cup is suspended from the end of condenser and, in that case, platinum wire is used for the suspension. The apparatus fits together without the use of cork, rubber or metal and in such a manner that loss of vapour during extraction does not exceed 20 percent of the extracting liquid.

3.1.1 Suitable types of apparatus are shown in Fig. 1.

3.2 Filter Paper or Cloth — Acid washed hardened filter paper or nylon cloth of open weave and continuous filaments.



Extraction Cup	Joints		Receiver
	A	B	
ml			ml
20 - 30	34/35	34/35	150
50 - 60	45/50	34/35	250

Fig. 1 Extraction Apparatus

4. REAGENTS

4.1 Ethanol-Toluene Azeotrope (ETA) — Mix 70 volumes of ethanol (*see* IS : 321-1964*) and 30 volumes of toluene (*see* IS : 537-1967†). Reflux for four hours over freshly calcined calcium oxide. Distil and collect a middle fraction with a boiling range between 76·2 to 77·2°C at atmospheric pressure.

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 estimated to weigh 3 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. Roll the weighed test portion in filter paper or nylon cloth 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. Place the roll in the extraction cup of the extraction apparatus and pour into the extraction flask sufficient solvent to fill the extraction cup two or three times. Assemble the apparatus and adjust the temperature so that the distilled quantity of solvent fills the extraction cup ten to twenty times per hour. The extraction time shall be 16·0 ± 0·5 hours. Evaporate off the solvent in a weighed vessel, preferably in the extraction flask, at about 100°C. Dry the flask for two hours at 100 ± 2°C in an oven. Cool in a desiccator and weigh to the nearest 0·001 g.

NOTE — The results obtained may be different depending on the method of sample preparation.

6. CALCULATION

6.1 Solvent extract, percent by mass $= \frac{M_1 - M_2}{M_3} \times 100$

where

M_1 = mass in grams of the extraction flask and extract,
M_2 = mass in grams of the extraction flask, and
M_3 = mass in grams of the test portion.

7. REPORT

7.1 The report shall include the following:

a) Proper identification of sample,
b) Sample preparation,

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*Specification for absolute alcohol (*revised*).

†Specification for toluene, pure, nitration grade (*first revision*).

c) Type of extraction apparatus, and
d) Result obtained.

METHOD B

8. OUTLINE OF THE METHOD

8.1 Thin, narrow strips of the dried rubber are extracted three times with 100 ml of hot ETA (*see* **10.1**) solvent for one hour each and rinsed three times with 10 ml portions of ETA solvent. Although two extractions are sufficient for non-pigmented rubbers, three extractions are necessary for oil-masterbatch rubbers. The extracted rubber is boiled with 25 ml of acetone, decanted, and dried. The difference in weight between the original sample and the extracted sample is the weight of ETA extract.

9. APPARATUS

9.1 Asbestos Mat

9.2 Filter Paper

9.3 Hot Plate

9.4 Wide-Mouth Flask — 400 to 500 ml capacity.

9.5 Wire Gauze

10. REAGENTS

10.1 Ethanol-Toluene Azeotrope (ETA) — Prepare by mixing 70 volumes of ethyl alcohol and 30 volumes of toluene, refluxing the mixture for four hours over calcium oxide and distilling. Discard the first and last portion, keeping only that distillate coming over within a range of 1°C.

NOTE — Distilling may be avoided by using absolute alcohol.

10.2 Acetone — conforming to IS : 170-1966*.

11. PROCEDURE

11.1 Dry the sample in accordance with the method prescribed in (SBR : 1) or (SBR : 2) of IS : 4518 (Part I)-1967†. Sheet it to a thickness of 0·5 mm or less. Cut approximately 6 g of the dried, sheeted material into strips not wider than 10 mm nor longer than 50 mm.

NOTE — Ensure that the strips are 0·5 mm or less in thickness in order to secure complete extraction.

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*Specification for acetone (*first revision*).

†Methods of tests for styrene-butadiene rubbers (SBR): Part I.

11.2 Accurately weigh 6 g sample of the dried strips. Add 100 ml of ETA (*see* **10.1**) solvent to the wide-mouth flask. Add each strip of the weighed sample separately to the flask, swirling the flask after each addition so that each strip is thoroughly wetted with the solvent to avoid sticking. To prevent the sample from sticking to the wide-mouth flask, place a filter paper in the bottom of the flask and use a wire gauze or asbestos mat between the flask and the hot plate. Reflux the contents of the wide-mouth flask on the hot plate for one hour. After refluxing, decant the ETA extract and discard it. Add a second 100 ml portion of the ETA solvent to the rubber sample and reflux again for one hour. Again decant the ETA extract and discard it. Add a third 100 ml portion of the ETA solvent to the rubber sample and reflux again for one hour. For the third time, decant the ETA extract and discard it. Rinse the sample with three successive 10 ml portions of the ETA solvent. Add approximately 25 ml of acetone to the rubber sample remaining in the wide-mouth flask. Heat the acetone to gentle boiling and boil it for approximately 5 minutes. Decant the acetone and transfer the extracted sample to a tared watch glass. Dry it to constant weight at 105°C.

NOTE — This can be done in 1 hour at 105°C and 725 mm of vacuum in a vacuum oven.

13. CALCULATION

13.1 ETA extract, percent $= 100 \frac{(M_1 - M_2)}{M_1}$

where

M_1 = mass in grams of the original dry sample, and
M_2 = mass in grams of the extracted sample.

DETERMINATION OF OIL CONTENT
(SBR : 10)

1. SCOPE

1.1 This method covers the estimation of the oil content of oil extended (SBR)-rubbers and carbon black masterbatches. It is intended for use in checking for uniformity having loading up to 75 parts per hundred of rubber (phr).

METHOD A

2. OUTLINE OF THE METHOD

2.1 The ETA extract of the dried rubber is determined. The oil content of the masterbatch ise stimated by subtracting from the ETA extract the total of the ETA soluble constituents other than oil. This difference is an estimate of the oil content.

3. PROCEDURE

3.1 Determine ETA extract in styrene butadiene rubbers as percent by mass of original dry sample (A) in accordance with (SBR : 9).

3.2 Determine organic acid in styrene butadiene rubbers as percent by mass of original dry sample (B) in accordance with (SBR : 4) [*see* IS : 4518 (Part I)-1967*].

3.3 Determine soap content in styrene butadiene rubbers as percent by mass of original dry sample (C) in accordance with (SBR : 5) [*see* IS : 4518 (Part I)-1967*].

3.4 Determine antioxidants in styrene butadiene rubbers as percent by mass of original dry sample (D) in accordance with (SBR : 6) [*see* IS : 4518 (Part I)-1967*].

NOTE — If antioxidant/antiozonants (D) cannot be determined, the following value may be calculated:

$$D = \frac{E}{F} \times 100$$

where

E = nominal antioxidant added to rubber phr†; and

F = masterbatch recipe, in parts = 100 + phr† of oil + phr† of carbon black.

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*Methods of tests for styrene-butadiene rubbers (SBR): Part I.

†Parts per hundred of rubber.

4. CALCULATION

4.1 Oil content percent by mass = $A - (B + C + D)$

where

A = ETA extract, percent by mass of the original dry sample;
B = organic acid, percent by mass of the original dry sample;
C = soap, percent by mass of the original dry sample; and
D = antioxidant/antiozonant, percent by mass of the original dry sample.

METHOD B

5. OUTLINE OF THE METHOD

5.1 The acetone extract of the dried rubber is determined. The oil content of the masterbatch is estimated by subtracting from the acetone extract the total of the acetone soluble constituents other than the oil. The difference is an estimate of the oil content.

6. APPARATUS

6.1 Extraction Apparatus — The extraction flask and condenser is of the general type prescribed in (SBR : 9).

6.2 Hot Plate — Surface to maintain a temperature of at least 100°C.

6.3 Vacuum Oven

7. PROCEDURE

7.1 Dry the sample in accordance with the method prescribed in SBR : 1 or (SBR : 2) of IS : 4518 (Part I)-1967*. Sheet it to a thickness of 0·5 mm or less. Cut approximately 6 g of the dried, sheeted material into strips not wider than 10 mm and not longer than 50 mm.

NOTE — Ensure that the strips are 0·5 mm or less in thickness in order to secure complete extraction.

7.2 Accurately weigh a 2 g sample of the dried strips. Add 200 ml of acetone to a 400 to 500-ml flask. Add each strip of the weighed sample separately to the flask, swirling the flask after each addition so that each strip is thoroughly wetted with solvent to avoid sticking. To prevent the sample from sticking to the flask, place a filter paper in the bottom of the flask, and use a wire gauze or an asbestos mat between the flask and the hot plate. Reflux the contents of the flask on the hot plate for 30 minutes.

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*Methods of tests for styrene-butadiene rubbers (SBR) : Part I.

Decant the acetone extract and discard it, but make sure that all the rubber remains in the flask. Add a second 200 ml portion of acetone to the rubber sample and reflux again for 30 minutes. Decant the acetone extract and discard it, but make sure that all the rubber remains in the flask. Add a third 200 ml portion of acetone to the rubber sample and reflux again for 30 minutes. For the third time, decant the acetone extract and discard it. Rinse the sample with 10 to 20 ml of acetone. Decant the acetone and transfer the extracted sample to a tared watch glass. Dry the rubber to constant weight in a vacuum oven at 105°C and minimum 685 mm of mercury vacuum. Weigh the dry rubber.

8. CALCULATION

8.1 Oil content, percent by mass $= \frac{100\ (A - X)}{A} - (B + C + D)$

where

A = mass of the original dry sample;
X = mass of the extracted sample;
B = organic acid, percent by mass (SBR : 4);
C = soap, percent by mass (SBR : 5); and
D = antioxidant, percent by mass (SBR : 6).

NOTE — If antioxidant cannot be determined, the following value may be calculated:

$$D = \frac{E}{F} \times 100$$

where

E = nominal antioxidant added to rubber, phr*; and
F = masterbatch recipe, in parts = 100 + phr* of oil + phr* of carbon black.

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*Parts per hundred of rubber.