IS : 10016 (Part 4) - 1984

Indian Standard

METHODS OF TEST FOR POLYBUTADIENE RUBBERS

PART 4 DETERMINATION OF CIS, TRANS AND VINYL STRUCTURE

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METHODS OF TEST FOR POLYBUTADIENE RUBBERS

PART 4 DETERMINATION OF CIS, TRANS AND VINYL STRUCTURE

0. FOREWORD

0.1 This Indian Standard (Part 4) was adopted by the Indian Standards Institution on 10 September 1984, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 In this series, methods of test for polybutadiene rubbers are given. Various other methods of test for polybutadiene rubbers are covered in the following parts:

Part 1 Method of taking out test portions from sample bales

Part 2 Determination of ash

Part 3 Determination of antioxidants

Part 5 Determination of gel content

0.3 Test methods for the following characteristics are similar to those for styrene butadiene rubbers and the methods indicated below shall be used in these cases:

Volatile matter	SBR : 1 and SBR : 2 of IS : 4518 (Part 1)-1967*;
Mooney viscosity	SBR : 8 of IS 4518 (Part 1)-1967*;
Solvent extract	SBR : 9 of IS : 4518 (Part 2)-1971*;
Oil content	SBR : 10 of IS : 4518 (Part 2)-1971*.

*Methods of tests for styrene-butadiene rubbers (SBR) :
Part 1 Determination of volatile matter, total ash. organic acid, soap, antioxidants, bound styrene and mooney viscosity (*Reaffirmed* 1979).
Part 2 Determination of solvent extract and oil content.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard (Part 4) prescribes the method of determining the relative amounts of various unsaturated forms in polybutadiene rubbers.

2. QUALITY OF REAGENTS

2.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977†) shall be employed in tests.

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

3. OUTLINE OF METHOD

3.1 A film of rubber is cast from dichloromethane or benzene or carbon tetrachloride (CCl_4) solution on to a potassium bromide prism and an infrared spectrum obtained from the film in the region from 9 to 16 microns (1200 to 600 cm^{-1}). The cis, trans and vinyl concentration are calculated from peaks appearing at 13·6 microns to 13·8 microns (793·6 to 735 cm^{-1}) for cis, at 10·35 microns (965 cm^{-1}) for trans and at 10·95 microns (910 cm^{-1}) for vinyl.

4. APPARATUS

4.1 *Infrared Spectrophotometer*

4.2 Conical Flask 50 ml

4.3 Potassium Bromide Prism

4.4 Mechanical Shaker

4.5 Analytical Balance

4.6 Prism Holder

4.7 Desiccator — Containing suitable drying agent.

5. REAGENTS

5.1 Dichloromethane, Benzene or Carbon Tetrachloride — Spectroscopic grade.

*Rules for rounding off numerical values (*revised*).
†Specification for water for general laboratory use (*second revision*).

6. PROCEDURE

6.1 Weigh accurately about 2·5 g of dry finished rubber taken in accordance with 2 of IS : 10016 (Part 1)-1981*. Cut the rubber into small cubes of about 0·3 mm size. Place the rubber in the conical flask; add 28 ml of dichloromethane or benzene or carbon tetrachloride. Place the flask on a mechanical shaker for 6 to 8 hours. Flask should be tightly closed to avoid evaporation.

6.2 Adjust zero and hundred percent transmittance on the infrared spectrophotometer.

6.3 Remove the potassium bromide prism from the dessicator. Add several drops of cement from the conical flask to the centre of the prism. Tilt the prism to 45° angle and let excess cement run off. Let the prism dry for about 10 to 15 minutes at 45°angle. The thickness of the film so formed should be uniform. Carefully place the prism into the aluminium holder with film outward. With the function switch set at stop, manually move the pen carriage slowly past the 13 microns to 14 micron lines on the chart and observe whether the cis peak is recorded between 0·7 to 1·0 in the absorption graph. Move the pen carriage slowly back to approximately the 8 micron line, lower the pen and move the function switch to start. The instrument will now make the desired scan from 8 micron to 16 micron when it will shut itself off.

6.4 Remove the prism from the holder and carefully remove the rubber film by rubbing gently with tissue paper. Remove the chart paper from the bed and use it for the calculation.

7. CALCULATION

7.1 Draw a perpendicular line through the trans peak appearing at 10·35 microns (965 cm^{-1}), another line through vinyl peak 10·95 microns (910 cm^{-1}) and a third line through the cis peak appearing at 13·6 microns to 13·8 microns (793·6 to 735 cm^{-1}).

7.2 The base line for vinyl peak 10·95 microns (910 cm^{-1}) and for cis peak 13·6 microns to 13·8 microns (793·6 to 735 cm^{-1}) will be the same.

7.3 With a french curve, carefully draw the curve line which represents the reference base line correction for the trans absorption at about 10·35 microns (965 cm^{-1}) and at about 10·95 microns (910 cm^{-1}) for vinyl absorption. The point where the curve line crosses the perpendicular line serves as the reference base line for calculating the trans absorption and vinyl absorption.

*Methods of test for polybutadiene rubber : Part 1 Method of taking out test portions from sample bales.

7.3.1 For information a representative curve obtained from the standard infrared spectrophotometer is shown in Fig. 1.

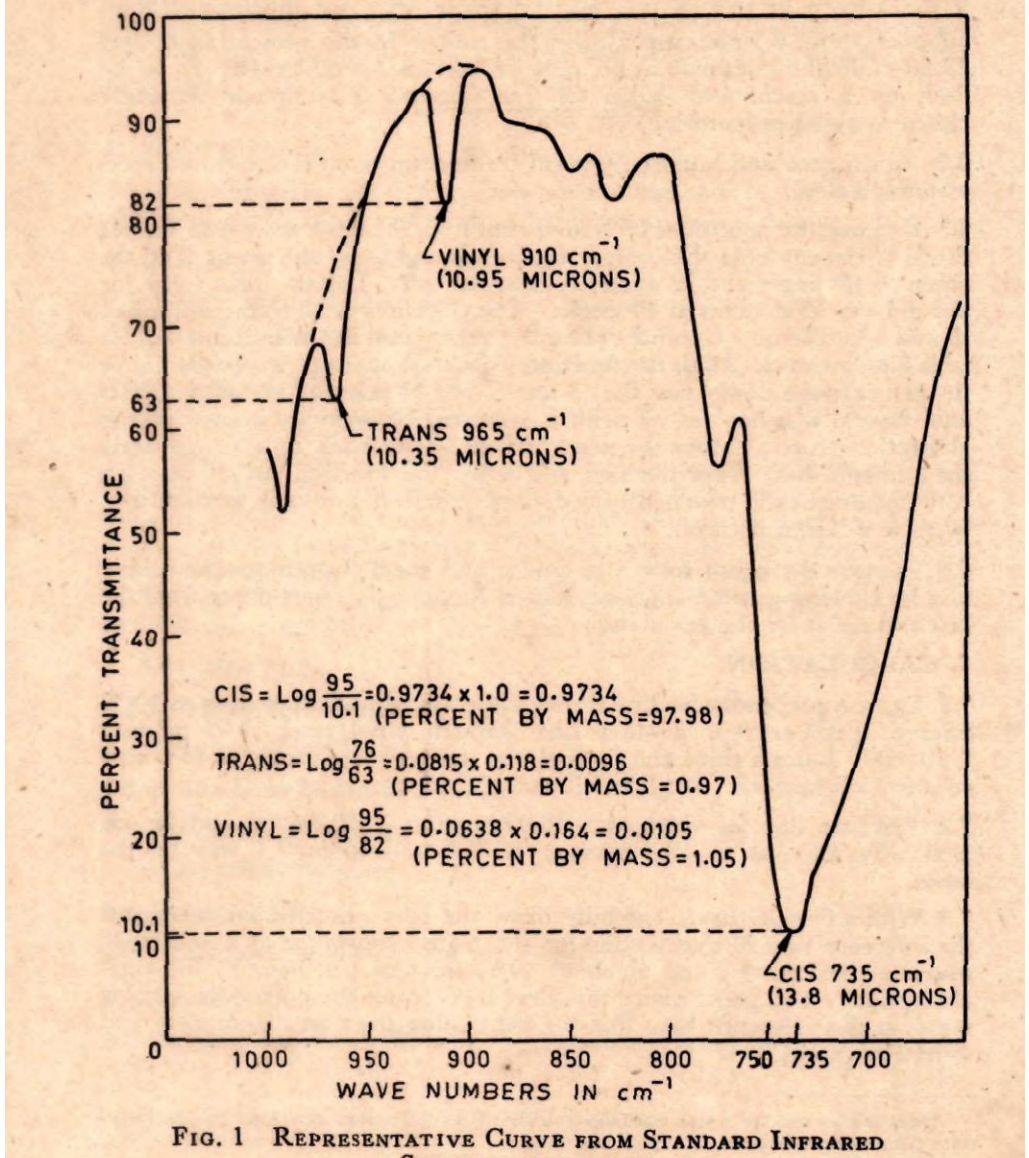

FIG. 1 REPRESENTATIVE CURVE FROM STANDARD INFRARED SPECTROPHOTOMETER

7.4 Calculate the relative concentration of the three components as follows:

$C_c = 1 \times$ cis peak

$C_t = 0{\cdot}118 \times$ trans peak

$C_v = 0{\cdot}164 \times$ vinyl peak

$$\text{Cis percent by mass} = \frac{C_c \times 100}{C_c + C_t + C_v}$$

$$\text{Trans percent by mass} = \frac{C_t \times 100}{C_c + C_t + C_v}$$

$$\text{Vinyl percent by mass} = \frac{C_v \times 100}{C_c + C_t + C_v}$$

NOTE — Factors 0·118 and 0·164. These factors are derived from a certified standard of high Cis-Polybutadiene.

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