Indian Standard

METHODS OF TEST FOR NATURAL RUBBER

PART 6 DETERMINATION OF RUBBER HYDROCARBON

[NR : 7]

(Second Revision)

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Gr 3

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0. FOREWORD

0.1 This Indian Standard (Part 6) (Second Revision) was adopted by the Bureau of Indian Standards on 16 May 1988, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 'Methods of test for natural rubber' had been originally covered in the following four parts of IS : 3660:

IS : 3660 (Part 1)-1972 Determination of dirt, volatile matter, ash, total copper, manganese, rubber hydrocarbon, viscosity (shearing disk viscometer), and mixing and vulcanizing of rubber in a standard compound (*first revision*)

IS : 3660 (Part 2)-1968 Determination of solvent extract and nitrogen content

IS : 3660 (Part 3)-1971 Plasticity and plasticity retention index

IS : 3660 (Part 4)-1979 Determination of colour, accelerated storage-hardening test and vulcanization characteristics (MOD test)

0.2.1 While reviewing various test methods for natural rubber, the Committee decided to align them with the corresponding international standards. No unification of test methods for natural and synthetic rubber has been considered necessary. However, in revising test methods for natural rubber, the Committee had decided to revise and split the standard (IS : 3660) into further parts and publish individual test methods under natural rubber (NR) series. For proper referencing of the existing test methods and the new methods under revision, a table showing correspondence of the various methods of test covered in the earlier 4 parts of IS : 3660 with the presently split parts retaining the original NR number have been given in Appendix A.

0.2.2 In order to facilitate cross-reference, it has been decided to retain the original discrete NR series numbers assigned to various test methods in original IS : 3660 (Part 1, Part 2, Part 3 and Part 4), in the revised parts of IS : 3660.

0.3 The test method given in this revised standard will supersede the test methods as given under NR : 7 of IS : 3660 (Part 1)-1972. All the four parts of the original IS : 3660 shall be withdrawn upon its complete revision.

0.4 In the preparation of this standard, assistance has been derived from ISO 5945-1982 (E) 'Rubber — Determination of polyisoprene content', issued by the International Organization for Standardization (ISO).

0.5 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*.

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

1. SCOPE

1.1 This standard (Part 6) specifies a method for determining the rubber hydrocarbon content of raw natural rubber.

2. QUALITY OF REAGENTS

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

*Specification for water for general laboratory use (*second revision*).

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

3. OUTLINE OF THE METHOD

3.1 Acetone extracted rubber is oxidized by a mixture of chromic and sulphuric acids. The acetic acid formed is removed by steam distillation and estimated by titration with sodium hydroxide solution. The calculation is based on the observation that a 75 percent yield of acetic acid is always obtained in oxidation of the isoprene unit under the specified test conditions.

4. APPARATUS

4.1 Digestion and Distillation Assembly — A suitable assembly for the digestion and distillation apparatus is shown in Fig. 1. Heating is carried out by heating mantles. Any apparatus which will perform the same functions in the same sequence as the items shown may be substituted.

4.2 Aeration Assembly — Containing a capillary tube which, when connected to a reduced pressure line, will maintain, through the receiving flask, an air flow of approximately 33 ml/s (*see* Fig. 2). If the absolute pressure is less than 4 kPa (30 mmHg), a capillary tube of length approximately 100 mm and of 1 mm bore will maintain the required air flow. Since it is essential to maintain the aeration at a rate within 20 percent of 33 ml/s, each capillary tube shall be tested before use. The following method may be used.

4.2.1 Invert a graduted tube over a beaker filled with water and evacuate the air through the capillary by means of a tube extending into the graduated tube. The rate of air flow will be the same as the rate at which water fills the graduated tube.

4.3 Extraction Apparatus — The extraction apparatus 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 the condenser in which 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 vapours during extraction does not exceed 20 percent of the extracting liquid.

4.4 Absorption Device — to prevent carbon dioxide from entering the aeration assembly.

5. REAGENTS

5.1 Phenolphthalein Indicator — *See* IS : 2263-1979*.

5.2 Chromic Acid Digestion Mixture — Dissolve 200 g of chromium trioxide (CrO_3) in 500 ml of water. Carefully add, while stirring, 150 ml of sulphuric acid (rd 1·84).

5.3 Standard Sodium Hydroxide Solution — aqueous solution, approximately 0·1 N, accurately standardized.

*Methods of preparation of indicator solutions (*first revision*).

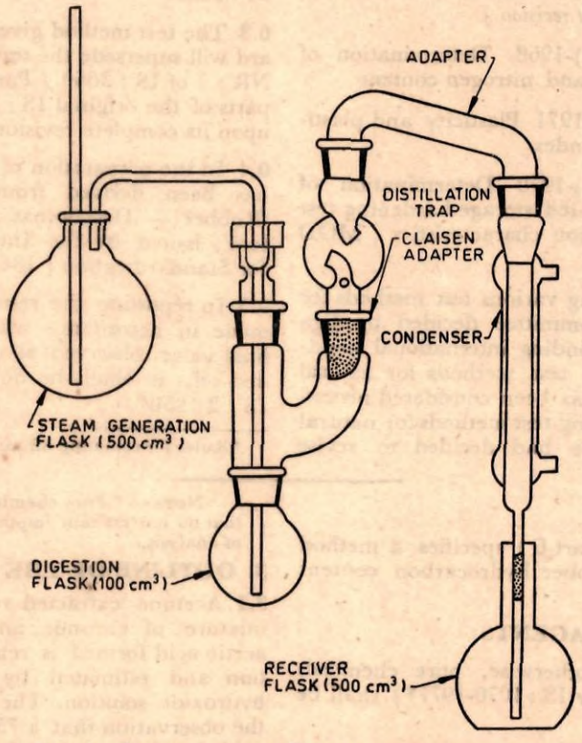

FIG. 1 DIGESTION AND DISTILLATION ASSEMBLY

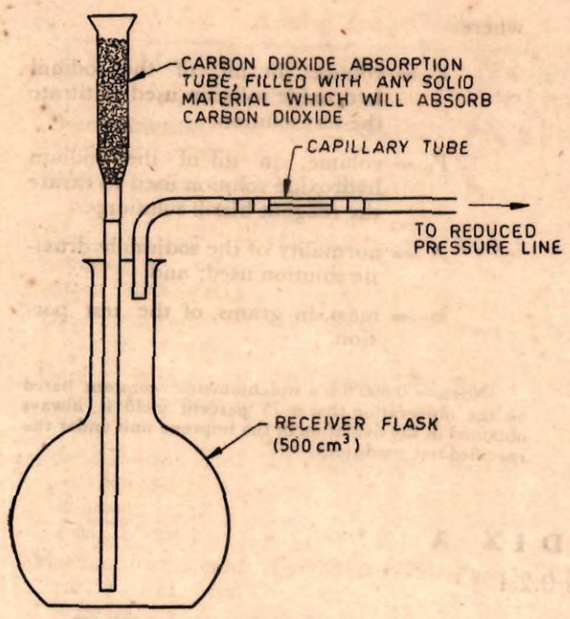

FIG. 2 AERATION ASSEMBLY

5.4 Acetone

6. PROCEDURE

6.1 Preparation of Test Sample — From the homogenized test piece prepared according to **3.1.1** of IS : 3660 (Part 1)-1972*, cut a portion of the rubber weighing about 5 to 10 g. Pass the rubber once or twice between the rolls of a laboratory mill, then reset the nip to 0·5 mm and mill out the rubber between hot rolls maintained at 95 ± 5°C. It is convenient to run the milled sheet into polyethylene or glazed linen to prevent sticking as it leaves the mill.

6.2 Test Portion — Cut from the homogenized sheet, avoiding the edges, a test portion to contain 0·3 g of rubber hydrocarbon into small pieces or thin sheets and weigh to the nearest 0·000 1 g.

6.3 Extraction — If the sheeted test portion is coherent, place it in the extraction cup (**4.3**). If it is unmanageable or sticky, wrap it in filter paper before placing in the cup. Extract the test portion for 16 h or overnight with the acetone extract.

6.3.1 All test portions shall be thoroughly dried before proceeding to the digestion.

6.4 Digestion — If necessary, unwrap the test portion; bits of paper adhering to the test portion need not be completely removed; small amounts of cellulose do not interfere with the determination. Place the test portion in the digestion flask and add an appropriate volume of the digestion mixture. For the apparatus shown in Fig. 1, 25 ml should be used. Greater volumes may be used according to the type of apparatus. Place the receiver flask, containing 100 ml of water, under the condenser and adapter of the distillation assembly. Heat the digestion mixture to 100 ± 5°C for a period sufficient to ensure complete digestion of the polyisoprene. Determine the correct heating mantle setting by heating the digestion mixture, without the sample, measuring the temperature of the digestion mixture with a thermometer. For the apparatus shown in Fig. 1, a digestion time of 30 minutes is sufficient. Other types of apparatus may need longer digestion times up to 1 h, for example.

6.5 Distillation

6.5.1 During the digestion step, the steam generation flask should be heated with stoppers removed so that steam is available at the end of the digestion period. If the laboratory is equipped with a steam line, this is unnecessary.

6.5.2 After 30 min, replace the stopper and delivery tube in the mouth of the steam generation flask, and allow steam to enter the digestion flask. Continue heating the digestion flask to maintain the total volume at about 75 ml. The rate of distillation and cooling shall be controlled so that the temperature of the condensate is not more than 30°C.

6.5.3 Collect 150 ml of distillate for the apparatus shown in Fig. 1 and appropriately larger volumes for larger apparatus.

6.5.4 At the end of distillation (usually 20 min for the apparatus shown in Fig. 1), remove the delivery tube from the receiver flask, carefully wash the tube with distilled water into the receiver flask and place on the aeration assembly.

6.5.5 Disconnect the stem delivery tube from the digestion flask as soon as the receiver flask is removed from the apparatus. This is to ensure that chromic acid mixture will not be sucked back into the steam generator when the heat source is removed.

6.6 Aeration — Adjust the temperature of the distillate to room temperature and aerate for 30 min for all types of digestion and distillation apparatus.

NOTE — The rates of loss of carbon dioxide and acetic acid during aeration have been investigated for temperature range, type of aeration apparatus and recommended rate of air flow. Variation of any of these factors may lead to erroneous analytical results.

*Method of test for natural rubber: Part 1 Determination of ash, total copper, manganese, rubber hydrocarbon, viscosity (shearing disc viscometer), and mixing and vulcanizing of rubber in a standard compound (*first revision*).

6.7 Titration

6.7.1 Remove the receiver flask from the aeration assembly, washing the delivery tube with distilled water and collecting the washings in the receiver flask. Add 5 drops of the phenolphthalein solution. Titrate with 0·1 N sodium hydroxide soultion.

6.7.2 Carry out a blank test of the digestion mixture reagents as often as necessary, or when a new supply of chromic acid is used.

7. CALCULATION

7.1 Calculate the rubber hydrocarbon content as follows:

$$\text{Rubber hydrocarbon, percent by mass} = \frac{0{\cdot}0908 \times (V_1 - V_0) \times N}{m} \times 100$$

where

V_1 = volume, in ml, of the sodium hydroxide solution used to titrate the test solution;

V_0 = volume, in ml of the sodium hydroxide solution used to titrate the reagent blank solution;

N = normality of the sodium hydroxide solution used; and

m = mass, in grams, of the test portion.

NOTE — 0·0908 is a stoichiometric constant based on the observation that a 75 percent yield is always obtained in the oxidation of the isoprene unit under the specified test conditions.

APPENDIX A

(*Clause* 0.2.1)

TABLE SHOWING CORRESPONDENCE OF THE VARIOUS METHODS OF TEST COVERED IN THE EXISTING IS : 3660 (PART 1)-1972, IS : 3660 (PART 2)-1968, IS : 3660 (PART 3)-1971, AND IS : 3660 (PART 4)-1979 WITH THE REVISION/ PROPOSED REVISION OF ALL THE FOUR PARTS OF IS : 3660

	Existing Test Methods		*Proposed Revision*	
Test Method	IS No.	Part (Series)	IS No.	Series
(1)	(2)	(3)	(4)	(5)
NR SERIES:				
Determination of dirt	IS : 3660-1972	Part 1 (NR : 1)	IS : 3660 (Part 1)-1985	(NR : 1)
Determination of volatile matter	IS : 3660-1972	Part 1 (NR : 2)	IS : 3660 (Part 2)-1985	(NR : 2)
Determination of ash	IS : 3660-1972	Part 1 (NR : 3)	IS : 3660 (Part 3)-1988	(NR : 3)
Determination of total copper	IS : 3660-1972	Part 1 (NR : 4)	IS : 3660 (Part 4)-1988	(NR : 4)
Determination of manganese	IS : 3660-1972	Part 1 (NR : 5)	IS : 3660 (Part 5)	(NR : 5)*
Determination of iron	IS : 3660-1972	Part 1 (NR : 6)	Deleted since this test is no longer being done	
Determination of rubber hydrocarbon	IS : 3660-1972	Part 1 (NR : 7)	IS : 3660 (Part 6)-1988	(NR : 7)
Determination of viscosity by shearing disc viscometer	IS : 3660-1972	Part 1 (NR : 8)	IS : 3660 (Part 7)-1988	(NR : 8)
Mixing and vulcanizing in a standard compound	IS : 3660-1972	Part 1 (NR : 9)	IS : 3660 (Part 8)	(NR : 9)*
Determination of solvent extract	IS : 3660-1968	Part 2 (NR : 10)	IS : 3660 (Part 9)	(NR : 10)*

*Under revision.

Existing Test Methods			*Proposed Revision*	
Test Method	IS No.	Part (Series)	IS No.	Series
(1)	(2)	(3)	(4)	(5)
Determination of nitrogen content	IS : 3660-1968	Part 2 (NR : 11)	IS : 3660 (Part 10)	(NR : 11)*
Determination of plasticity	IS : 3660-1971	Part 3 (NR : 12)	IS : 3660 (Part 11)	(NR : 12)*
Determination of plasticity retention index (PRI)	IS : 3660-1971	Part 3 (NR : 13)	IS : 3660 (Part 12)	(NR : 13)*
Determination of colour	IS : 3660-1979	Part 4 (NR : 14)	IS : 3660 (Part 13)	(NR : 14)*
Determination of storage-hardening test	IS : 3660-1979	Part 4 (NR . 15)	IS : 3660 (Part 14)	(NR : 15)*
Determination of vulcanization characteristics (MOD test)	IS : 3660-1979	Part 4 (NR : 16)	IS : 3660 (Part 15)	(NR : 16)*
Method for preparation of test samples	IS : 3660-1972	Part 1 (Clause 3)	IS : 3660 (Part 16)	(NR : 17)*

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*Under revision.