भारतीय मानक

प्राकृतिक रबड़ की परीक्षण विधियाँ

भाग 1 मैल ज्ञात करना

[एन आर : 1]

(तीसरा पुनरीक्षण)

Indian Standard

METHODS OF TEST FOR NATURAL RUBBER

PART 1 DETERMINATION OF DIRT

[NR : 1]

(Third Revision)

ICS 83.060; 19.020

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

June 1999

Price Group 2

Indian Standard

METHODS OF TEST FOR NATURAL RUBBER

PART 1 DETERMINATION OF DIRT

[NR : 1]

(Third Revision)

1 SCOPE

This standard (Part 1) prescribes the method of test for the determination of dirt content of raw natural rubber.

2 NORMATIVE REFERENCES

The following Indian Standards contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
460 (Part 1) : 1985	Test sieves: Part 1 Wire cloth test sieves (*third revision*)
1381 (Part 2) : 1977	Boiling flasks : Part 2 Flasks with conical ground socket (*first revision*)
2480 (Part 1) : 1983	General purpose glass thermometers : Part 1 Solid stem thermometers (*first revision*)
5599 : 1970	Methods of sampling of raw rubber

3 APPARATUS

3.1 Chemical/Electronic Balance

3.2 Conical Flask

500 ml capacity [*see* IS 1381 (Part 2)].

3.3 Desiccator with Efficient Desiccant

3.4 Thermometer

To read atleast 200°C [*see* IS 2480 (Part 1)].

3.5 Heater

For heating conical flask and its contents. Hot plates which provide uniform heating surfaces, or heater with infra red lamp or a sand bath may be used.

3.6 Wire Sieve

Of nominal size of opening 45 micron IS Sieve [*see* IS 460 (Part 1)].

3.6.1 The wire sieve shall be mounted across the bottom surface of a stainless steel tube of internal diameter 25 mm and outer diameter 30 mm. The height of the sieve frame shall not be less than 15 mm.

3.7 Ultrasonic Equipment, for Cleaning Sieves

4 REAGENTS

4.1 High aromatic hydrocarbon solvent known as white spirit or low aromatic hydrocarbon solvent such as turpentine or kerosene with boiling range above 150°C.

4.2 Mixed Xylenes, Boiling Range Above 140°C

4.3 Petroleum hydrocarbon solvent, such as petroleum ether with boiling range 60-80°C.

4.4 Toluene

4.5 Rubber Peptizing Agents

4.5.1 Toyl mercaptan solution, 20 percent (*m/m*) to 40 percent (*m/m*) in high aromatic hydrocarbon solvent.

4.5.2 *Copper Oleate*

NOTE — *Preparation of copper oleate* — Dissolve 6.58 g of sodium hydroxide in about 65 ml water and 46.38 g oleic acid with stirring. Filter the solution through Whatman filter paper (No. 41). Wash the precipitate 2 or 3 times with distilled water so as to remove the excess sodium hydroxide. Take out the precipitate in clean beaker and dissolve it in about 500 ml water by heating. To this add concentrated solution of copper sulphate, prepared by dissolving 25 g of copper sulphate in water and heat at 90 to 95°C. Stir the mixture for one hour. Separate the top layer of copper oleate, wash with water and purify in a separating funnel using diethyl ether as solvent. Keep the copper oleate as 25 percent solution in pure mineral turpentine or benzene.

4.5.3 Other fully soluble rubber peptizing agent.

5 PROCEDURE

5.1 To the conical flask add around 200 ml rubber solvent and 1 ml of the rubber peptizing agent solution.

5.2 Weigh 10 g of rubber from the test portion, as specified in IS 5599 to the nearest 0.1 g. Cut into strips and place in the conical flask.

5.3 Heat the flask and its contents at 125 to 130°C until a smooth solution is obtained or stand for several hours at room temperature in closed condition before heating to 125 to 130°C.

5.4 Shake the flask occasionally during the standing and heating periods by hand. Boiling or overheating of the rubber solution may result in the formation of a gel like substance which renders subsequent filtration difficult and may result in a higher apparent dirt content. Hence avoid apparatus and conditions which can cause local overheating.

5.5 When the rubber is completely dissolved, decant the hot solution through the sieve which has been weighed to the nearest 0.1 mg, retaining the bulk of the dirt in the flask.

5.6 Wash the dirt in the flask 3 or 4 times with 20 to 30 ml each of hot solvent and transfer completely to the sieve.

5.7 Wash the sieve twice with petroleum hydrocarbon solvent (4.3) and dry at 100°C for 30 minutes or with solvent (4.1 and 4.2) and dry at 100°C for 1 h.

5.8 The dirt on the sieve after drying should be loose and free-flowing. If this is not so, treat the sieve with boiling toluene (4.4).

5.9 Cool the sieve and residue in a desiccator and weigh to the nearest 0.1 mg.

5.10 Carry out three determinations.

6 CARE OF SIEVES

6.1 At all stages handle the sieve carefully. Inspect it after each determination to check for damage or block of perforations with the help of a slide projector. If noticeable distortion or block of holes are seen, replace it by a new sieve.

6.2 Sieve may be stored in warm toluene to lessen build up of rubber.

7 EXPRESSION OF RESULTS

The dirt content expressed as a percentage by mass of the test portion is given by the formula:

$$\text{Dirt content, percent by mass} = \frac{M_1}{M_2} \times 100$$

where

M_1 = mass in g of the dirt, and

M_2 = mass in g of the test portion.

ANNEX A

(Foreword)

TABLE SHOWING CORRESPONDENCE OF THE VARIOUS METHODS OF TEST COVERED IN THE ORIGINAL IS 3660 (Part 1) : 1972 (NOW DESIGNATED AS Part 51), IS 3660 (Part 2) : 1968 (NOW DESIGNATED AS Part 52), IS 3660 (Part 3) : 1971 (SINCE WITHDRAWN ON ITS COMPLETE REVISION) , AND IS 3660 (Part 4) : 1979 (NOW DESIGNATED AS Part 54) WITH THE NEW REVISED STANDARDS

Test Method	*IS No.*	*Original Part (Series)*	*IS No.*	*New Parts Series*	*Remarks/ Reaffirmed*
(1)	(2)	(3)	(4)	(5)	(6)
NR Series					
Determination of dirt	3660 : 1972	Part 1 (NR : 1)	3660 (Part 1) : 1999	(NR : 1)	—
Determination of volatile matter	3660 : 1972	Part 1 (NR : 2)	3660 (Part 2) : 1985	(NR : 2)	December 1995
Determination of ash	3660 : 1972	Part 1 (NR : 3)	3660 (Part 3) : 1988	(NR : 3)	January 1995
Determination of total copper	3660 : 1972	Part 1 (NR : 4)	3660 (Part 4) : 1988	(NR : 4)	January 1995

Test Method	*IS No.*	*Original Part (Series)*	*IS No.*	*New Parts Series*	*Remarks/ Reaffirmed*
(1)	(2)	(3)	(4)	(5)	(6)
Determination of manganese	3660 : 1972	Part 1 (NR : 5)	3660 (Part 5) : 1989	(NR : 5)	January 1995
Determination of iron	3660 : 1972	Part 1 (NR : 6)	Deleted since this test is no longer being done		
Determination of rubber hydrocarbon	3660 : 1972	Part 1 (NR : 7)	3660 (Part 6) : 1988	(NR : 7)	January 1995
Determination of Viscosity by shearing disk viscometer	3660 : 1972	Part 1 (NR : 8)	3660 (Part 7) : 1988	(NR : 8)	January 1995
Mixing and vulcanizing in a standard compound	3660 : 1972	Part 1 (NR : 9)	3660 (Part 8) : 1999	(NR : 9)	—
Determination of solvent extract	3660 : 1968	Part 2 (NR : 10)	3660 (Part 9) : 1989	(NR : 10)	January 1995
Determination of nitrogen	3660 : 1968	Part 2 (NR : 11)	3660 (Part 10)	(NR : 11)	—
Determination of plasticity	3660 : 1971	Part 3 (NR : 12)	3660 (Part 11) : 1989	(NR : 12)	January 1995
Determination of plasticity retention index (PRI)	3660 : 1972	Part 3 (NR : 13)	3660 (Part 12) : 1989	(NR : 13)	January 1995
Determination of colour	3660 : 1979	Part 4 (NR : 14)	3660 (Part 13) : 1997	(NR : 14)	—
Determination of storage hardening test	3660 : 1979	Part 4 (NR : 15)	3660 (Part 14)	(NR : 15)	Revision still to be published
Determination of vulcanization characteristics (MOD test)	3660 : 1979	Part 4 (NR : 16)	3660 (Part 15)	(NR : 16)	do
Method of preparation of test samples	3660 : 1972	Part 1 (Clause 3)	5599 : 1999		

NOTES

1 Original IS 3660 (Part I) : 1972 now redesignated as IS 3660 (Part 51) : 1972.

2 Original IS 3660 (Part II) : 1968 now redesignated as IS 3660 (Part 52) : 1968.

3 Subsequent to its complete revision, IS 3660 (Part III) : 1971 has since been withdrawn.

4 Original IS 3660 (Part IV) : 1979 now re-designated as IS 3660 (Part 54) : 1979.

Rubber Sectional Committee, PCD 14

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Rubber Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

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

IS 3660 (Part I) : 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 [subsequently designated as IS 3660 (Part 51) : 1972].

IS 3660 (Part II) : 1968 Determination of solvent extract and nitrogen content [subsequently designated as IS 3660 (Part 52) : 1968].

IS 3660 (Part III) : 1971 Plasticity and plasticity retention index [subsequently withdrawn with the publication of Parts 11 and 12 of IS 3660 in 1989].

IS 3660 (Part IV) : 1979 Determination of colour, accelerated storage-hardening test and vulcanization characteristics (MOD test) [subsequently designated as IS 3660 (Part 54) : 1979].

While reviewing various test methods for natural rubber, the Committee decided to align them with the corresponding International Standards. Unification of test methods for natural and synthetic rubber has not been considered necessary. However, in revising test methods for natural rubber, the Committee had decided to revise and split the standards into further parts and publish test methods for individual characteristics under natural rubber (NR) series. For proper referencing of the original test methods and the new methods revised/under revision, a table showing correspondence of the various methods of test covered in the previous 4 parts of IS 3660, namely (Part I, II, III and IV) with the presently split parts have been given in Annex A. 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 I, II, III and IV) in the revised part of IS 3660.

In the preparation of this standard, assistance has been derived from ISO 249 : 1995 (E) 'Rubber, raw natural — Determination of dirt content', published by the International Organization for Standardization (ISO).

In this revision toyl mercaptan has been included as an additional peptizing agent and xylyl mercaptan has been deleted. Method for determination of dirt content has been elaborated.

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*)'.