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भारतीय मानक

कच्चे प्राकृतिक और संश्लेषित रबड़ के नमूने तैयार करने और नमूने लेने की पद्धतियाँ

(पहला पुनरीक्षण)

Indian Standard

RUBBER — RAW, NATURAL AND SYNTHETIC — METHODS FOR SAMPLING AND SAMPLE PREPARATION

(First Revision)

ICS 83.040.10

BUREAU OF INDIAN STANDARDS
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FOREWORD

This Indian Standard (First 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.

This standard was first published in 1970 and covered only sampling of raw rubber. In this version, scope has been enlarged so as to cover all raw rubbers. Scale of sampling with AQL 2.5 percent, method of preparation of test sample to cover NBR, BR, IIR, CR and EPDM have been modified. Alternate procedure for individual sample, a diagram showing sequence of operation in sampling and further preparation have been included. Criteria of conformity for composite sample has been updated by incorporating single limit and double limit requirements.

In the preparation of this standard assistance has been derived from ISO 1795 'Rubber, raw natural and synthetic — Sampling and further preparative procedures', issued by the International Organization for Standardization (ISO).

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*)'

Indian Standard

RUBBER — RAW, NATURAL AND SYNTHETIC — METHODS FOR SAMPLING AND SAMPLE PREPARATION

(First Revision)

1 SCOPE

This standard prescribes the method for the sampling of raw rubber in bales, blocks or packages and further procedure carried out on those samples to prepare test portions for chemical and physical tests. It specifies the number of tests that should be made for each characteristic and lays down the criteria for ascertaining the conformity of the material in a lot to the requirements specified for the characteristics.

A diagram showing the sequence of operations in sampling and further preparation is given in Annex A.

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*
3660	Methods of test for natural rubber:
(Part 1) : 1985	Determination of dirt (NR:1) (*second revision*)
(Part 2) : 1985	Determination of volatile matter (NR:2) (*second revision*)
(Part 3) : 1988	Determination of ash (NR:3)(*second revision*)
(Part 4) : 1988	Determination of total copper (NR:4) (*second revision*)
(Part 5) : 1989	Determination of manganese (NR:5) (*second revision*)
(Part 6) : 1988	Determination of rubber hydrocarbon (NR:7) (*second revision*)
(Part 7) : 1988	Determination of Mooney viscosity (NR:8) (*second revision*)
(Part 9) : 1989	Determination of solvent extract (NR:10) (*first revision*)
(Part 11) : 1989	Determination of plasticity (NR:12) (*first revision*)
(Part 12) : 1989	Determination of plasticity retention index (PRI) (NR:13) (*first revision*)
4518	Methods of test for styrene-butadiene rubber (SBR):
(Part 1) : 1967	Determination of volatile matter, total ash, organic acid, soap, antioxidants, bound styrene and Mooney viscosity
(Part 2) : 1971	Determination of solvent extract and oil content
4905 : 1968	Methods for random sampling
8683 : 1977	Methods of test for raw acrylonitrile-butadiene rubber
10016	Methods of test for polybutadiene rubbers:
(Part 4) : 1984	Determination of CIS, trans and vinyl structure
(Part 5) : 1981	Determination of gel content
11720	Methods of test for synthetic rubber:
(Part 1) : 1986	Determination of antioxidants (SR:1)
(Part 3) : 1993	Determination of Mooney viscosity
(Part 4) : 1993	Determination of volatile matter
(Part 5) : 1993	Determination of ash

3 TERMINOLOGY

For the purpose of this standard, the following definitions shall apply.

3.1 Bale

A unit package in a form convenient for handling.

3.2 Lot

An assembly of bales of rubber bearing the same grade and lot marks.

3.3 Sample

A group of bales selected to represent the lot.

3.4 Individual Sample

The rubber taken from a bale of the sample to represent the bale.

3.5 Composite Sample

A quantity of rubber which will represent the sample prepared by collecting equal parts of the individual samples for specified physical, chemical and vulcanization tests.

3.6 Test Portion

The rubber taken from the individual sample or the composite sample for testing.

4 SCALE OF SAMPLING

4.1 The bales shall be selected from each lot separately for ascertaining the conformity of the lot to the specified requirements.

4.2 The number of bales to be selected from each lot shall depend on the size of the lot and unless agreed to otherwise between the purchaser and the supplier, shall be in accordance with Table 1 or Table 2.

Table 1 Scale of Sampling by Attributes

No. of Bales in the Lot	No. of Bales in the Sample
(1)	(2)
Up to 40	4
41 to 100	7
101 and above	10

Table 2 Scale of Sampling by Variables

No. of Bales in the Lot	No. of Bales in the Sample	*K* Value	*Max* Allowable Percent Defective
(1)	(2)	(3)	(4)
3 to 120	3	1.12	7.6
121 to 195	4	1.17	10.9
196 to 330	5	1.24	9.8
331 to 540	7	1.33	8.4
541 to 900	10	1.41	7.3
901 to 1 500	15	1.47	6.6
1 501 to 2 400	20	1.51	6.2

NOTES

1 The sampling plan is based on an acceptable quality level of 2.5 percent at a median inspection level.

2 Minimum value of *K* for quality characteristic having single specification limits.

3 Maximum allowable percent defective for quality characteristic having both maximum and minimum specification limits.

4.3 The bales shall be selected at random from the lot. In order to ensure randomness of selection, the procedures as specified in IS 4905 shall be adopted.

5 PREPARATION OF INDIVIDUAL AND COMPOSITE SAMPLES

5.1 Individual Sample

An individual sample shall be taken from each of the selected bales by the following preferred method. Make two cuts, without the use of lubricant, through the entire bale normal to the surfaces of the largest area of the bale so that a slice is removed from the middle of the bale. The outer wrapping sheets, polyethylene wrapping or other surface material shall be removed. Alternatively, an individual sample may be cut from any convenient part of the bale. However, for reference purposes, the preferred method shall be used. The total mass of the individual sample shall be between 600 g and 1 500 g depending on the tests to be carried out.

If the rubber is in chip or powder form, a similar quantity shall be taken at random from the package. Unless the individual sample is to be immediately used, it shall be placed in a moisture proof container or package of not more than twice its volume, until it is required.

5.2 Composite Sample

Pieces of equal mass, each not less than 150 g, shall be taken from each individual sample, and all the pieces so obtained from all the bales in the sample shall together constitute the composite sample. Homogenization or blending of the pieces is carried out to prepare test portions or as the initial part of preparation of rubber test mixes from the composite sample.

5.3 Sets of Samples

Each individual sample, as also the composite sample shall be divided into 3 equal parts. Each part shall be transferred to a suitable airtight container or wrapped with two layers of aluminium foil and marked properly with all identification marks. The three identical sets of individual and composite samples thus obtained shall be meant for the purchaser, the supplier and the referee. The total mass of the piece or pieces be between 600 g and 1 500 g depending on the tests to be carried out.

5.4 Sampling Report

The sampling report shall include the following information:

a) Type and grade of rubber;
b) The number and kind of bales or package forming the lot;
c) The number of bales or packages forming the sample; and
d) All details required for full identification of the sample.

6 METHODS OF PREPARATION OF TEST PORTIONS

6.1 Apparatus

A two roll mill having the following characteristics shall be used for homogenization.

6.1.1 The mill shall preferably have rolls 150 ± 5 mm in outside diameter and shall be equipped with guides spaced 250 to 280 mm apart to retain the rubber at the nip.

NOTES

1 If mills of other sizes are used, adjustments to batch masses and mixing cycles may be required to obtain comparable results.

2 Preferably the mill should be capable of operating at friction and at even speed.

6.1.2 The speed of the slow roll (front roll) shall be 24 ± 1 rev/min and the ratio between the fast and slow roll shall be preferably 1.4:1. Other ratios may be used, but modifications in mixing procedure may be required to obtain comparable results.

6.1.3 Means shall be provided for controlling the mill roll temperatures to the specified temperature within a tolerance of ± 5°C unless otherwise specified in the appropriate Indian Standard.

6.1.4 *Determination of the Clearance Between the Rolls*

The clearance between the rolls shall be adjustable at least from 0.2 to 3.0 mm. Roll clearance shall be determined by means of two lead strips 10 ± 3 mm in width, at least 50 mm long and 0.25 to 0.50 mm thicker than the roll clearance to be measured. The lead strips shall be inserted, one at each end of the rolls, approximately 25 mm from the guides, while a piece of compounded rubber, with a Mooney viscosity greater than 50 and measuring approximately 75 mm × 75 mm × 6 mm, is passing through the centre portion of the nip. The rolls shall be at the temperature specified for mixing. After passing between the rolls, the thickness of the lead strip shall be measured with a micrometer to an accuracy of 0.01 mm. Tolerance on roll clearance shall be ± 10 percent or ± 0.05 mm, whichever is the larger.

6.2 Natural Rubber

6.2.1 *Homogenization*

The piece is passed six times through the gap between the rolls of a 150 mm × 300 mm laboratory mill having the rolls rotating at uneven speeds with friction ratio 1 : 1.4 ± 0.1 with the back roll rotating at 31 rpm. The rolls are cooled with running water at room temperature and the gap is set at 1.65 ± 0.16 mm.

After each pass the rubber is rolled into a cylinder and introduced endwise for the next pass. A clean, stainless steel tray below the rolls should be available to catch any rubber or dirt from the piece. Any such rubber or dirt is returned to the rubber at the next pass. The rubber is not rolled after the sixth pass and test pieces are cut out for the various tests.

6.2.1.1 The homogenization could also be carried out by the R.R.I.M. method, that is, with six passes between the surfaces of the mill rolls set at a nip of 1.65 ± 0.15 mm and with the rolls at room temperature. This method, however, may give slightly different initial plasticity values compared to the method given above.

6.2.2 *Allocation of Portions for Chemical and Physical Tests and for Determination of Vulcanization Characteristics*

Cut test portions from the homogenized individual or composite sample and allocate them to such of the specified tests as may be required from those indicated in Annex A under 'Natural Rubber'. The tests shall be performed in accordance with the following Indian Standards:

IS 3660 (Part 1)
IS 3660 (Part 2)
IS 3660 (Part 3)
IS 3660 (Part 4)
IS 3660 (Part 5)
IS 3660 (Part 6)
IS 3660 (Part 7)
IS 3660 (Part 9)
IS 3660 (Part 11)
IS 3660 (Part 12)

6.3 Synthetic Rubbers

6.3.1 *Chemical Tests*

Cut a test portion of at least 250 g (or, if the product is in chip or powder form, a similar sample taken at random) from the laboratory sample and use for the determination of volatile matter content in accordance with the hot mill method of IS 11720 (Part 4), where specified. Certain rubber tend to stick to the rolls during the hot mill method; if sticking occurs, the oven method of IS 11720 (Part 4) at 100 ± 5°C shall be used. Take portions from the material remaining from the determination of volatile matter content in such amounts as required for such other chemical tests as may be required; perform the tests in accordance with the Indian Standards listed below. If the oven method is used for determination of volatile matter content, the rubber shall be dried by the hot mill method prior to carrying out chemical tests. If this is not possible then the test portions may be taken directly from the laboratory sample.

IS 4518 (Part 1)
IS 4518 (Part 2)
IS 8683 : 1977
IS 10016 (Part 4)
IS 10016 (Part 5)
IS 11720 (Part 1)
IS 11720 (Part 4)
IS 11720 (Part 5)

NOTE — A composite sample may be prepared by blending together material remaining from each determination of volatile

matter content so that a composite sample of about 250 ± 5 g is formed. Blend the individual pieces together using the procedure described in **6.3.2.1**.

6.3.2 *Mooney Viscosity*

6.3.2.1 Synthetic rubbers (except isoprene — IR butyl rubber — IIR and halogenated butyl rubber — BIIR, CIIR). Take a test portion of rubber of about 250 ± 5 g for determination of Mooney viscosity. Pass this test portion ten times between the surfaces of the mill rolls with the nip set at 1.4 ± 0.1 mm and with the mill roll surface temperature maintained at 50 ± 5°C. In passes 2 to 9 inclusive, double the rubber upon itself. On the tenth pass sheet the rubber without doubling for testing in accordance with IS 11720 (Part 3).

NOTES

1 For butadiene rubber (BR) and ethylene-propylene-diene rubber (EPDM), the mill roll surface temperature shall be 35 ± 5°C.

2 For chloroprene rubber (CR), the mill roll surface temperature shall be 20 ± 5°C. Set the nip at 0.40 ± 0.05 mm and make only two passes.

3 For some types of butadiene acrylonitrile rubber (NBR), the nip shall be set at 1.0 ± 0.1 mm and the mill roll surface temperature shall be 50 ± 5°C.

4 Crumb samples tested during production should be massed according to the procedure specified in **6.3.2.1**.

6.3.2.2 *Isoprene rubber (IR), butyl rubber (IIR) and halogenated butyl rubber (BIIR, CIIR)*

Cut a test portion from the laboratory sample avoiding areas which contain many bubbles for testing Mooney viscosity.

NOTE — Butyl rubber crumb tested during production shall be massed according to the procedure in **6.3.2.1**.

6.3.2.3 *Vulcanization characteristics*

Cut a test portion (or physically select, if the rubber is in chip or powder form) from the composite sample for the determination of vulcanization characteristics in accordance with IS 11720 (Part 2).

Collect approximately equal portions from each individual sample to form a composite laboratory sample of the correct size. The blending operation takes place in the initial part of the mixing procedure.

7 NUMBER OF TESTS

7.1 Each individual sample shall be tested separately for volatile matter (mill stage), dirt, initial plasticity and PRI and Mooney viscosity.

7.2 The composite sample shall be tested for chemical properties and vulcanization characteristics.

8 CRITERIA FOR CONFORMITY

8.1 Sampling by Attributes

See Table 1.

8.1.1 *Individual Samples*

In respect of each characteristics tested on sample bales individually the lot shall be declared to conform if the test results on all samples bales are found to satisfy the specified requirement.

8.1.2 *Composite Sample*

In respect of the characteristics tested on the composite sample, the lot shall be declared to conform, if all the test results on the composite sample are found to satisfy the specified requirements.

8.2 Sampling by Variables

See Table 2.

8.2.1 *Q Value*

a) For a requirement having a maximum limit, calculate the Q value as follows:

$$Q = \frac{X_{Max} - X}{s}$$

where

X_{Max} = maximum value permitted by the specification;

X = mean of the values obtained for the sample; and

s = standard deviation of the sample values.

b) For a requirement having a minimum limit, calculate the Q value as follows:

$$Q = \frac{X - X_{Min}}{s}$$

where

X_{Min} = minimum value permitted by the specification;

X = mean of the values obtained for the sample; and

s = standard deviation of the sample values.

8.2.1.1 *Single limit*

For a quality characteristic having a single specification limit, a lot is acceptable if the Q value equals or exceeds the K value in Table 3 for the applicable lot and sample size.

8.2.1.2 *Double limit*

For a quality characteristic having both a maximum and minimum specification limit, estimate the percentages of the lot above the maximum limit and below the minimum limit from the Table 2 using the appropriate sample size and Q values calculated in **8.2.1**. A lot is acceptable if the sum of the two percentages does not exceed the percent defective given in the last column of Table 2 for the applicable lot and sample size.

8.2.2 *Composite Sample*

In respect of the characteristics tested on the composite sample, the lot shall be declared to conform if all the

test results of the composite sample are found to satisfy the specified requirements.

Table 3 Estimate of Lot Percent Defective
(*Clause* 8.2.1.1)

Q Value	Sample Sizes						
	3	4	5	7	10	15	20
0.95	19.3	18.3	17.9	17.5	17.3	17.2	17.2
1.00	16.7	16.7	16.4	16.1	16.0	15.9	15.9
1.05	13.7	15.0	14.9	14.8	14.7	14.7	14.7
1.10	9.8	13.3	13.5	13.5	13.5	13.5	13.5
1.15	0.3	11.7	12.1	12.3	12.3	12.4	12.4
1.20	—	10.0	10.8	11.1	11.2	11.3	11.4
1.25	—	8.3	9.5	10.0	10.2	10.3	10.4
1.30	—	6.7	8.2	8.9	9.2	9.4	9.5
1.35	—	5.0	7.0	7.9	8.3	8.5	8.6
1.40	—	3.3	5.8	7.0	7.4	7.7	7.5
1.45	—	1.7	4.8	6.1	6.6	6.9	7.0
1.50	—	—	3.8	5.3	5.9	8.2	6.3
1.55	—	—	2.9	4.5	5.2	5.5	5.7
1.60	—	—	2.0	3.8	4.5	4.9	5.1
1.65	—	—	1.3	3.2	4.0	4.4	4.5
1.70	—	—	0.7	2.6	3.4	3.8	4.0
1.75	—	—	0.2	2.1	2.9	3.4	3.5
1.80	—	—	—	1.7	2.5	2.9	3.1
1.85	—	—	—	1.3	2.1	2.6	2.8
1.90	—	—	—	0.9	1.8	2.2	2.4
1.95	—	—	—	0.6	1.4	1.9	2.1
2.00	—	—	—	0.4	1.2	1.6	1.8
2.10	—	—	—	0.1	0.7	1.2	1.3
2.20	—	—	—	—	0.4	0.8	1.0
2.30	—	—	—	—	0.2	0.5	0.7
2.40	—	—	—	—	0.1	0.3	0.5
2.50	—	—	—	—	—	0.2	0.3
2.60	—	—	—	—	—	0.1	0.2
2.70	—	—	—	—	—	0.1	0.1
2.80	—	—	—	—	—	—	0.1
2.90	—	—	—	—	—	—	—

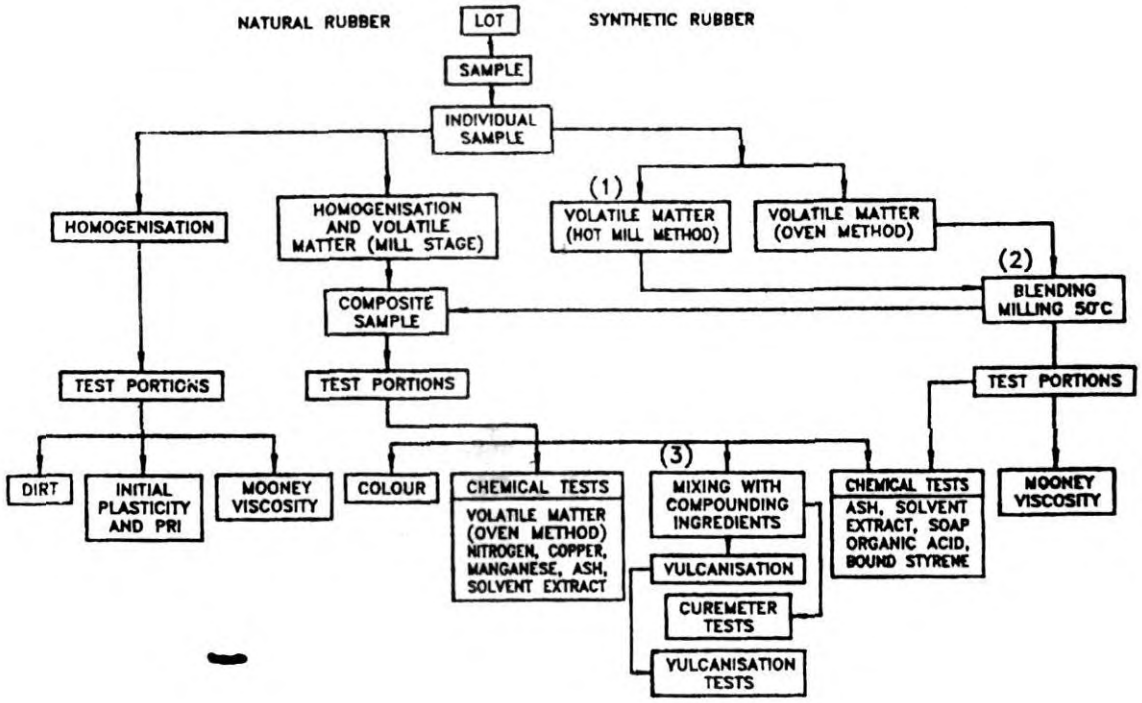
Weigh the piece to the nearest 0.1 g and then homogenize it by passing ten times between the surfaces of the mill rolls with the nip set at 1.3 ± 0.15 mm and with the rolls maintained at 70 ± 5°C. In passes 2 to 9 inclusive, rolls, the rubber after passing through the nip and present the roll endwise to the nip for the next pass. Return to the rubber any solid matter separating from it. On the tenth pass, sheet the rubber, allow it to cool in a desiccator and weigh it again to the nearest 0.1 g.

NOTE — The initial and final masses are used in the calculation of volatile matter (mill stage) since some of the volatile are lost during homogenization [*see* the procedure given in IS 3660 (Part 2)]. If the volatile matter is not to be determined immediately, store the homogenized rubber in an airtight container of not more than twice its volume, or wrap it tightly in two layers of aluminium foil until required for test.

ANNEX A

(*Clauses* 1 *and* 6.2.2)

SEQUENCE OF OPERATIONS IN SAMPLING AND FURTHER PREPARATION



A detailed sequence of operations in the sampling and further preparation of raw rubber is given in the figure given above. Attention is drawn to be two routes available for determining the volatile matter and chemical properties of synthetic rubbers. The route chosen depends on the suitability of the material for processing on a hot mill.

(1) Preferred method.

(2) Omit for IIR (butyl) and halobutyl, and if rubber is very difficult to hot mill. The test portion can be taken directly from the individual sample for chemical tests in such cases.

(3) Blending/homogenization of individual samples may be done in the initial part of the mixing procedure.

Bureau of Indian Standards

BIS is a statutory institution established under the *Bureau of Indian Standards Act*, 1986 to promote harmonious development of the activities of standardization, marking and quality certification of goods and attending to connected matters in the country.

Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

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