IS : 3660 ( Part 3 ) - 1988

# *Indian Standard*

# METHODS OF TEST FOR NATURAL RUBBER

## PART 3 DETERMINATION OF ASH

## NR : 3

## *( Second Revision )*

UDC 678·4 : 543·822



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## 0. FOREWORD

**0.1** This Indian Standard ( Part 3 ) ( Second Revision ) was adopted by the Bureau of Indian Standards on 14 March 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:

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

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 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 previous 4 parts of IS : 3660, Part 1, 2, 3 and 4, 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, 2, 3 and 4 ), in the revised parts of IS : 3660.

**0.3** The test method given in this revised standard will supersede the test method as given under NR : 3 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 All the four parts of the original IS : 3660 shall be withdrawn upon its complete revision.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 247-1978 'Rubber—Determination of ash' issued by the International Organization for Standardization (ISO) which prescribes two methods for raw rubbers, namely, Method A of heating the sample in a crucible outside over a gas burnner and then placing the crucible inside muffle furnace, and Method B by placing the sample in muffle furnace directly until constant mass is attained. Lately the above ISO is being revised in which Method B is proposed to be deleted. However, the concerned Committee after detailed consideration has decided to retain both the test methods in the revision of this Indian Standard since the results by both the test methods are fairly accurate and Method B is helpful particularly when a large number of samples are to be analysed.

**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*.

## 1. SCOPE

**1.1** This standard ( Part 3 ) prescribes method for determination of ash in raw natural rubber.

## 2. OUTLINE OF THE METHOD

**2.1 Method A** — Heating of a weighed test portion in a crucible over a gas burner. After expulsion of the volatile decomposition products,

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

transfer of the crucible to a muffle furnace where it is heated until all the carbonaceous matter has been burnt off and constant mass is attained.

**2.2 Method B** — Heating of a weighed test portion wrapped in ashless filter paper and placed in a crucible, in a muffle furnace until volatile decomposition products have been expelled, all the carbonaceous matter has been burnt off and constant mass is attained.

## 3. APPARATUS

**3.1 Crucible** — Silica or porcelain crucible of 50 ml capacity.

NOTE — If copper or manganese is to be determined in the ash subsequently, use only silica crucible.

**3.2 Desiccator with Efficient Desiccant**

**3.3 Asbestos Board** — 100-mm square and of the thickness approximately 5 mm, with a central hole to accommodate the crucible. About two thirds of the crucible should project below the asbestos board.

**3.4 Bunsen Burner** — or similar type of gas burner.

**3.5 Filter Paper** — ashless, of diameter 150 mm.

**3.6 Muffle Furnace** — fitted with a flue and with provision for controlling the air flow through the furnace ( This may be achieved by adjusting the door opening ). A temperature controlling device is required to maintain a temperature of 550 ± 25°C.

## 4. PROCEDURE

**4.1** For determination of ash content, cut test portions of raw natural rubber prepared according to **3.1.1** of IS : 3660 ( Part 1 )-1972*. Take a test portion of about 5 g to the nearest 0·000 1 g. Then ash and weigh as described under Method A or Method B.

**4.1.1** *Method A* — Heat the clean empty crucible for about 30 min in the muffle furnace, maintained at 550 ± 25°C, allow to cool to ambient temperature in desiccator and weigh to the nearest 0·000 1 g. Place the weighed test portion in the crucible mounted in the hole in asbestos board. Heat the crucible gently with the burner, taking care that the rubber does not ignite. If any material is lost due to spurting

*Methods of test for natural rubber: Part 1 [ Method for preparation of test samples under revision as IS : 3660 ( Part 16 ) ( NR : 17 ) ].

or frothing, repeat the above procedure with a new test portion. When the rubber has decomposed to a charred mass, gradually increase the heat from the burner until the volatile decomposition products have been substantially expelled and a dry carbonaceous residue remains. Transfer the crucible and its contents to the muffle furnace, maintained at 550 ± 25°C, leaving the door of the furnace slightly open to provide sufficient air to oxidize the carbon.

**4.1.1.1** Continue heating until the carbon is completely oxidized and a clean ash is obtained. Remove the crucible and its contents from the furnace, allow to cool to ambient temperature in the desiccator and weigh to the nearest 0·000 1 g. Then heat the crucible and its contents again for about 30 min in the muffle furnace, maintained at 550 ± 25°C, allow to cool to ambient temperature in the desiccator and re-weigh to the nearest 0·000 1 g This mass should not differ from the previous mass by more than 0·001 g. If this requirement is not fulfilled, repeat the heating, cooling and weighing procedure until the difference between two successive weighings meets this requirement.

**4.1.2** *Method B* — Heat the clean empty crucible of appropriate size for about 30 min in the muffle furnace, maintained at 550 ± 25°C. Allow to cool to ambient temperature in a desiccator and weigh to the nearest 0·000 1 g. Wrap the weighed test portion in ashless filter paper and place in the crucible. Transfer the crucible and its contents to the muffle furnace, maintained at 550 ± 25°C, and close the door rapidly. The furnace door must not be opened during the first hour because of the risk of igniting combustible gases.

**4.1.2.1** After 1 h, open the door of the furnace slightly to provide sufficient air to oxidize the carbon. Continue heating until the carbon has been compeletely oxidized and a clean ash is obtained. Remove the crucible and its contents from the furnace, allow to cool to ambient temperature in a desiccator and weigh to the nearest 0·000 1 g. Then heat the crucible and its contents again for about 30 min in the muffle furnace, maintained at 550 ± 25°C. Allow to cool to ambient temperature in the desiccator and re-weigh to the nearest 0·000 1 g. This mass should not differ from the previous mass by more than 0·00 1 g. If this requirement is not fulfilled, repeat the heating, cooling and weighing procedure until the difference between two successive weighings meets this requirement.

NOTE 1 — Since the furnace door must be closed rapidly and kept closed after the insertion of a crucible, if more than one determination is being made, it is convenient to place the crucibles together on a suitable rack or tray. All the crucibles can be introduced into the furnace in one operation.

NOTE 2 — If the ash line is within 3 mm of the rim of the crucible, the determination shall be abandoned. The test shall then be repeated using either a

smaller test portion or a larger crucible, Alternatively, method A may be used in place of method B.

## 5. EXPRESSION OF RESULTS

**5.1** Calculate the ash content of the rubber, as a percentage by mass, by the following formula:

$$\text{Ash, percent by mass} = \frac{m_2 - m_1}{m_0} \times 100$$

where

$m_0$ = mass in g of test portion,

$m_1$ = mass in g of empty crucible, and

$m_2$ = mass in g of crucible and ash.

# APPENDIX A

( *Clause* 0.2.1 )

**TABLE SHOWING CORRESPONDENCE OF 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 | | Remarks |
|---|---|---|---|---|---|
| Test Methods | IS : No. | Part ( Series ) | IS : No. | ( Series ) | |
| (1) | (2) | (3) | (4) | (5) | (6) |
| *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 ) | ( NR : 4 ) | Under revision |
| Determination of manganese | IS : 3660-1972 | Part 1 ( NR : 5 ) | IS : 3660 ( Part 5 ) | ( NR : 5 ) | Under revision |
| 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 disk 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 ) | |
| Determination of nitrogen content | IS : 3660-1968 | Part 2 ( NR : 11 ) | IS : 3960 ( 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 ) | Under revision |
| 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 ) | |