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Indian Standard

METHODS OF TEST FOR POLYBUTADIENE RUBBERS

PART 2 DETERMINATION OF ASH

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(*Continued on page* 2)

(*Continued from page* 1)

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(*Continued on page* 6)

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METHODS OF TEST FOR POLYBUTADIENE RUBBERS

PART 2 DETERMINATION OF ASH

0. FOREWORD

0.1 This Indian Standard (Part 2) was adopted by the Indian Standards Institution on 21 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 3 Determination of antioxidants
Part 4 Determination of cis, trans and vinyl structure
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*

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.

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*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.
Part 2 Determination of solvent extract and oil content.

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

1. SCOPE

1.1 This standard (Part 2) prescribes the method for the determination of total ash in polybutadiene rubbers.

2. OUTLINE OF THE METHOD

2.1 Ashing process takes place while heating a dry weighed test portion, wrapped in ashless filter paper and placed in a crucible in a muffle furnace. Process is continued until volatile decomposition products have been expelled, all carbonaceous matter has been burnt off and constant mass is obtained.

3. APPARATUS

3.1 Muffle Furnace — Fitted with provision for controlling air flow, pyrometer and thermostatic control required to maintain a temperature of 550±25°C.

3.2 Asbestos Board — Approximately 10 cm square and 0·5 cm thick with a hole in the centre to support the crucible such that approximately two thirds of its depth project below the board.

3.3 Crucible — Silica, porcelain or platinum of capacity approximately 50 ml.

3.4 Desiccator

3.5 Chemical Balance

3.6 Filter Paper — Ashless type of approximately 150 cm diameter.

4. SAMPLE PREPARATION

4.1 Test sample shall be dried in accordance with IS : 4518 (Part 1)-1967* for determination of volatile matter. Test portions shall be cut from dried rubber.

5. PROCEDURE

5.1 Heat the crucible to 550±25°C for 30 minutes. Allow to cool in a desiccator and weigh accurately. Weigh accurately about 3 to 5 g of the dried material and wrap it in ashless filter paper and place it in the crucible. Heat the crucible gently in order that the rubber does not ignite and no spurting occurs. When the rubber completely decomposes to a charred mass transfer the crucible to a muffle furnace maintained at a temperature of 550±25°C. Continue heating until the carbon has been

*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.

completely oxidised and a clean ash is obtained. Remove the crucible from the furnace and cover it in open air by placing on asbestos board for 3 to 4 minutes. Then keep the crucible in a desiccator for cooling and weigh accurately.

5.2 Heat the crucible and contents again for about 30 minutes in the muffle furnace, maintained at 550±25°C. Allow to cool to ambient temperature in the desiccator and re-weigh. This mass should not differ from previous mass by more than 0·000 1 g. If this requirement is not met, repeat the heating, cooling and weighing until the difference between two successive weighings meet this requirement.

NOTE — If the ash line is within 3 mm of the rim of the crucible, discard the ash and repeat the analysis.

6. CALCULATION

6.1 Calculate the ash as a percentage of mass by the following formula:

$$\text{Total ash, percent by mass} = \frac{M_1 - M_2}{M_3} \times 100$$

where

M_1 = mass of crucible and ash,

M_2 = mass of empty crucible, and

M_3 = mass of test portion.

(*Continued from page* 2)

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