

Rubber latices —

Part 3: Methods of test —

Section 3.19 Determination of total bound styrene content of reinforced styrene-butadiene rubber latices

[ISO title: Rubber — Reinforced styrene-butadiene latex — Determination of total bound styrene content]

NOTE Attention is drawn to BS 6057-0 “*General introduction*”, issued separately.

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This British Standard, having been prepared under the direction of the Rubber Standards Committee, was published under the authority of the Board of BSI and comes into effect on 31 March 1986

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The Committees responsible for this British Standard are shown in Part 0.

The following BSI references relate to the work on this standard:
Committee reference RUM/38
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Amendments issued since publication

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National foreword

This Section of BS 6057 is identical with ISO 4655:1985 “*Rubber — Reinforced styrene-butadiene latex — Determination of total bound styrene content*”, published by the International Organization for Standardization (ISO). It supersedes method 8.3 of BS 3397:1976 which is withdrawn by amendment.

Terminology and conventions. The text of the International Standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Wherever the words “International Standard” appear, referring to this standard, they should be read as “British Standard”.

Cross-references

International Standard	Corresponding British Standard
ISO 2453:1975	BS 1673 <i>Methods for testing raw rubber and unvulcanized compounded rubber</i> Part 5/5.1:1963 <i>Analysis of styrene-butadiene copolymers (SBR), bound styrene content</i> (Technically equivalent)

The Technical Committee has reviewed the provisions of ISO 123:1974, to which reference is made in clauses 2, 4.3 and 5.3, and has decided that they are acceptable for use in conjunction with this standard. Method 2 of BS 1672:1972 is related to ISO 123:1974, and will eventually be replaced by BS 6057-2 “*Sampling*” (in preparation); it is intended that BS 6057-2 will be identical with a revision of ISO 123 (also in preparation).

Additional information. Water complying with BS 3978 “*Water for laboratory use*” is suitable for any dilution or washing of samples that may be required during the preparation of the sample (see clauses 4 and 5).

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 10, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope and field of application

This International Standard specifies two methods for determining the total bound styrene content of styrene-butadiene rubber latices which are reinforced with polystyrene or a copolymer of butadiene and styrene.¹⁾

The two methods, the carbon/hydrogen method and the nitration method, give comparable results although they differ in principle. Either method may be used, according to preference.

NOTE Any auxiliary materials present in the test sample may affect the accuracy of the determination.

2 References

ISO 123, *Rubber latex — Sampling*.

ISO 2453, *Styrene-butadiene copolymers — Determination of bound styrene content*.

3 Principles

In both methods, the latex is coagulated with 2-propanol and the coagulum is thoroughly washed, shredded and dried under vacuum.

In the carbon/hydrogen method, the dry polymer is subjected to controlled combustion and the carbon dioxide and water produced are quantitatively absorbed. The total bound styrene content is calculated from the masses of absorbed carbon dioxide and water. The method depends on the fact that the proportion of carbon in butadiene (88,82 %) differs from that in styrene (92,26 %).

In the nitration method, the dry polymer is nitrated and oxidized to convert its total bound styrene content to *p*-nitrobenzoic acid, which is separated by extraction and determined quantitatively by measuring its ultraviolet absorption at 265, 274 and 285 nm.

4 Carbon/hydrogen method

4.1 Reagents and materials

All recognized health and safety precautions shall be taken when carrying out the procedure specified in this International Standard.

During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.1.1 Sulfuric acid, 96 % (m/m).

4.1.2 Soda asbestos granules, 710 to 1 000 µm, which change colour on absorption of carbon dioxide.

4.1.3 Magnesium perchlorate granules, 710 to 1 000 µm.

WARNING — This material is explosive.

Handle in accordance with the manufacturer's instructions.

4.1.4 Copper(II) oxide, in wire form.

4.1.5 Copper(II) oxide/cobalt oxide (Co₃O₄) catalyst

Ignite 98 g of copper(II) oxide at 700 ± 25 °C in a furnace for 1 h, cool, mix thoroughly with 8 g of cobalt(II) nitrate dissolved in the minimum quantity of water, heat at 120 °C with occasional stirring for 1 h, and ignite in a silica dish at 700 ± 25 °C in a furnace for 1 h.

4.1.6 Platinized asbestos, 5 % platinum content.

4.1.7 Silver wire cloth, having an average aperture width of 250 ± 15 µm, 70 mm square, rolled into a solid cylinder of 13 mm diameter.

The roll shall be degreased with ether, then immersed in nitric acid, $c(\text{HNO}_3) = 3 \text{ mol/dm}^3$ for a few seconds to remove any oxide or sulfide, washed copiously with water and dried for 1 h at 105 °C and 1 h at 700 ± 25 °C in a furnace.

4.1.8 Silver wire

4.1.9 2-Propanol

4.1.10 Benzoic acid

4.1.11 Naphthalene

4.1.12 Standard SBR, of accurately known bound styrene content (approximately 23,5 %), determined in accordance with ISO 2453.

4.2 Apparatus

Figure 1 shows the arrangement of a suitable combustion apparatus. Before being mixed, oxygen and air pass separately through a pressure-release vessel containing sulfuric acid (4.1.1), a guard tube (4.2.1) to remove carbon dioxide and moisture, a needle valve to control the rate of flow and a calibrated rotameter (4.2.2). The gas mixture flows through the purification tube (4.2.3) and another guard tube (4.2.1) into the combustion tube (4.2.4). The products of combustion pass into two absorption tubes (4.2.5), where the water and carbon dioxide are collected. Most of the purification tube and about half of the combustion tube are within the furnace (4.2.6).

All glass-to-glass joints shall be gas-tight and shall be either close-butted within heavy-walled silicone rubber tubing or of the cone and socket type. All joints between the purification tube and the absorption tubes shall remain gas-tight at 700 ± 25 °C.

¹⁾ Both methods are also suitable for the determination of the bound styrene content of non-reinforced styrene-butadiene rubber latices.