



BS 6057 : Part 3 : Section 3.19 : 1986 ISO 4655-1985

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British Standard

Rubber latices

Part 3. Methods of test

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

98/年10月

98年7月2

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

99年7月20

Latex de caoutchouc

Partie 3. Méthodes d'essai

Section 3.19 Détermination de la teneur totale en styrène lié des latex de styrène-butadiène renforcé

2001年9月28日

2002年6月2日

2004年6月14日

Kautschuklatex

Teil 3. Prüfverfahren

Abschnitt 3.19 Bestimmung des Gesamtgehaltes an gebundenem Styrol in mit Styrol-Butadien verstärktem Kautschuklatex

NOTE. Attention is drawn to BS 6057 : Part 0 'General introduction', issued separately.

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 It supersedes method 8.3 of BS 3397 : 1976 which is withdrawn by amendment.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
ISO 2453-1975

Corresponding British Standard
BS 1673 Methods for testing raw rubber and unvulcanized compounded rubber
Part 5/5.1 : 1963 Analysis of styrene-butadiene copolymers (SBR), bound styrene content (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 : Part 2 'Sampling' (in preparation); it is intended that BS 6057 : Part 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).

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

2006年7月18日

2005年7月13日



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 (Co_3O_4) 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, $c(\text{HNO}_3) = 3$ mol/dm³ 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

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

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.

4.2.1 Guard tubes, each of capacity approximately 35 cm^3 , half-filled with the soda asbestos (4.1.2) at the gas entry end and half-filled with the magnesium perchlorate (4.1.3) at the gas exit end.

4.2.2 Rotameters, calibrated over the range 2 to $25 \text{ cm}^3/\text{min}$, one for oxygen and the other for air.

4.2.3 Purification tube, containing copper(II) oxide in wire form (4.1.4) and having the dimensions and contents specified in figure 2.

4.2.4 Combustion tube, of clear silica, having the dimensions, contents and fittings specified in figure 3.

The silver wire in the beak of the tube, in contact with the silver cloth (4.1.7), serves to prevent condensation of water. The copper(II) oxide/cobalt oxide catalyst (4.1.5) shall be packed into the tube carefully so that there is neither channelling nor restriction of gas flow.

The pyrolysis zone of the tube is encircled by 30 separate heaters, each with a power of approximately 50 W. Each heater consists of about 500 mm of nickel-chromium wire (diameter 0,5 mm) arranged in an open pattern, to ensure visibility into the tube, and occupying approximately 5 mm out of 8 mm of tube length. The whole of the pyrolysis zone of the tube is covered with a hinged, concentric stainless steel shield to conserve heat and protect from draughts.

The cone of the tube is closed by means of a 19/26 cap held in position by lugs and springs.

4.2.5 Absorption tubes of soda glass, of wall thickness 0,25 to 0,50 mm, having the dimensions and packing specified in figure 4.

The mass when empty of either tube a), for absorption of water, or tube b), for absorption of carbon dioxide, shall not exceed 7,5 g.

Each tube shall be carefully packed as rapidly as possible, to prevent ingress of moisture or carbon dioxide, avoiding tight packing or channelling, and both taps shall then be closed.

Tube a) shall be connected to the beak of the combustion tube, as shown in figure 1. The other end of the tube a) shall be connected to the end of tube b) which contains soda asbestos.

4.2.6 Furnace, as specified in figure 5.

The nickel-chromium wire wound around the smaller diameter silica tube develops a power of approximately 80 W. The wire wound around the larger diameter silica tube develops a power of approximately 135 W. Both tubes are individually maintained at 700 ± 25 °C, as indicated by the thermocouples, by means of variable transformers.

4.2.7 Platinum combustion boats, 16 mm × 5,5 mm, with platinum sheath.

4.2.8 Microbalance, accurate to 2 µg.

4.2.9 Timer, accurate to 1 s.

4.3 Sampling

Sampling shall be carried out in accordance with one of the methods specified in ISO 123.

4.4 Preparation of test sample

Dilute 5 g of latex with 2 to 3 cm^3 of water. Using a dropping tube, add the diluted latex to 100 cm^3 of the vigorously stirred 2-propanol (4.1.9) at approximately 23 °C, allow to settle and decant the supernatant liquid. Stir vigorously with water to wash the coagulum and drain thoroughly on a Büchner funnel. Wash copiously with water. Steep overnight in cold water, drain off the water, rinse well with 2-propanol, shred and then dry in an open dish under vacuum at approximately 50 °C to constant mass. All handling of the test sample shall be carried out in such a way as to avoid contamination.

If the test sample cannot be used immediately, store it under nitrogen in a cool, dark place.

4.5 Procedure

Insert the freshly packed purification tube (4.2.3) and combustion tube (4.2.4) into the furnace (4.2.6) and assemble the apparatus as indicated in figure 1. Pass oxygen and air, each at a rate of $20 \text{ cm}^3/\text{min}$, and raise the temperature of the furnace to 700 ± 25 °C.

Maintain the temperature of the furnace at 700 ± 25 °C for approximately 16 h to remove occluded water and traces of organic matter. Check the stability and performance of the combustion system by repeated assays of the benzoic acid (4.1.10) until replicates fall within $68,84 \pm 0,08$ % carbon and $4,95 \pm 0,03$ % hydrogen. Use 20 mg samples of the benzoic acid and the first heating schedule specified in the table.

Maintain the flow rates of oxygen and air at $20 \pm 1 \text{ cm}^3/\text{min}$ and the temperature of the furnace at 700 ± 25 °C, until replacement of the tube packings becomes necessary.

* Glassware with either cm^3 or ml marking is satisfactory for use with the procedure described in this International Standard.