
International Standard



4655

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

**Rubber — Reinforced styrene-butadiene latex —
Determination of total bound styrene content**

Caoutchouc — Latex de styrène-butadiène renforcé — Détermination de la teneur en styrène lié

Second edition — 1985-05-15

UDC 678.031 : 678.746.22 : 543.8

Ref. No. ISO 4655-1985 (E)

Descriptors : rubber, synthetic rubber, styrene-butadiene rubber, latex, chemical analysis, determination of content, styrene, test equipment.

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4655 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

ISO 4655 was first published in 1977. This second edition cancels and replaces the first edition, of which it constitutes a minor revision.

Rubber — Reinforced styrene-butadiene latex — Determination of total bound styrene content

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.