
**Rubber, raw styrene-butadiene,
emulsion-polymerized —
Determination of bound styrene
content — Refractive index method**

*Caoutchouc butadiène-styrène brut polymérisé en émulsion —
Détermination de la teneur en styrène lié — Méthode par l'indice de
réfraction*

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Published in Switzerland

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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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This third edition cancels and replaces the second edition (ISO 2453:1991) which has been technically revised. It also incorporates the Technical Corrigendum ISO 2453:1991/Cor.1:2003.

The main changes compared to the previous edition are as follows:

- deletion of spiders and introduction of the extraction procedure of ISO 7781 into the preparation procedure (7.1);
- addition of wide-mouthed conical flask (6.1);
- correction of Table 1;
- move of precision data to an informative Annex A with addition of precision data evaluated in 2019.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Rubber, raw styrene-butadiene, emulsion-polymerized — Determination of bound styrene content — Refractive index method

1 Scope

This document specifies a method for determining the bound styrene content of emulsion-polymerized styrene-butadiene rubbers (SBR) by correlation with the measured refractive index of an extracted sample according to a table of refractive indices versus percentage mass fractions styrene.

The method is also applicable to extracted oil-extended emulsion-polymerized SBR as long as it produces a film suitable for refractive index measurements. It is not applicable to solution-polymerized SBR.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at <https://www.iso.org/obp>

— IEC Electropedia: available at <http://www.electropedia.org/>

4 Principle

The bound styrene test is a measure of the bound monomeric composition of the rubber. It is used to check the accuracy of monomer charging and also as a guide to the uniformity of the product, since the bound styrene content affects the physical properties.

The methods consist in the extraction of a test piece with ethanol-toluene azeotrope (ETA), followed by drying and pressing between sheets of aluminium foil to provide sheeted rubber having a thickness of not more than 0,50 mm.

The bound styrene content is calculated from the refractive index obtained at 25 °C on this thinly sheeted rubber.

5 Reagents

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

5.1 Ethanol-toluene azeotrope (ETA), by mixing 7 volumes of absolute ethanol with 3 volumes of toluene.

Alternatively, mix 7 volumes of commercial grade ethanol with 3 volumes of toluene, and boil the mixture with anhydrous calcium oxide (quicklime) under reflux for 4 h.

Then distil the azeotrope and collect the fraction with a boiling range not exceeding 1 °C, for use in the test.

5.2 Acidified ETA, by adding 10 cm³ of concentrated hydrochloric acid (approximately 35 % mass fraction) to a portion of ETA (5.1) and make the solution up to 1 000 cm³ with ETA.

NOTE Acidified ETA is used for alum-coagulated polymers.

5.3 α -Bromonaphthalene.

6 Apparatus

Ordinary laboratory equipment and the following.

6.1 Wide-mouthed conical flask, of capacity 400 cm³ to 500 cm³.

6.2 Reflux condenser.

NOTE Alternatively, a Soxhlet extractor can be used instead of a reflux condenser and a conical flask.

6.3 Abbe-type refractometer, having fourth decimal-place accuracy and whose refracting prism can be placed in a nearly horizontal position for measurement of the refractive index of solids. An Amici-type compensating prism for achromatization is necessary unless a sodium-vapour lamp is used as a light source.

The refractometer shall be maintained at a temperature of 25 °C \pm 0,1 °C, obtained by the use of a constant temperature room or by circulating constant temperature water through the instrument.

6.4 Vacuum oven, capable of being evacuated to a pressure of 1 300 Pa¹⁾ and of maintaining a temperature of 100 °C \pm 5 °C.

6.5 Aluminium foil, between 0,025 mm and 0,08 mm thick, having good tear strength.

6.6 Glass reference, for checking adjustment of the refractometer. The glass reference shall be calibrated for use at 25 °C.

6.7 Hydraulic press, that can be maintained at a temperature of 100 °C and can attain a total force of up to 100 kN on the platens.

6.8 Pressing plates (optional apparatus), measuring 210 mm \times 210 mm \times 3 mm, with a wooden handle. One of the plates shall have a 150 mm square area in the centre milled out to a depth not to exceed 0,65 mm.

6.9 Scissors, small and sharp.

1) 1 Pa = 1 N/m².