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**Styrene-butadiene rubber, raw —  
Determination of soap and organic-  
acid content**

*Caoutchouc butadiène-styrène brut — Détermination de la teneur en  
savons et acides organiques*

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ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
[copyright@iso.org](mailto:copyright@iso.org)  
[www.iso.org](http://www.iso.org)

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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](http://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](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on 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 the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html). (standards.iteh.ai)

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 fifth edition cancels and replaces the fourth edition (ISO 7781:2008), which has been technically revised.

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

- addition of a new Method C (8.4) for the back titration using automatic potentiometric titrator;
- update of Method B (8.3) for the titration method using automatic potentiometric titrator;
- deletion of buffer solutions of pH 7, pH 4 and pH 9 (former 4.6 to 4.8);
- addition of pipette (6.9) and automatic potentiometric titrator (6.11);
- update of the expression of results (Clause 9);
- addition of precision data in informative Annex B;
- addition of a bibliography.

# Styrene-butadiene rubber, raw — Determination of soap and organic-acid content

## 1 Scope

This document specifies three methods for the determination of the soap and organic-acid content of raw styrene-butadiene rubber (SBR).

- Method A is the titration method using indicator reagent.
- Method B is the titration method using an automatic potentiometric titrator.
- Method C is the back titration method using an automatic potentiometric titrator.

Since the soaps and organic acids present in the rubber are not single chemical compounds, the method gives only an approximate value for the soap and organic-acid content.

## 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 385, *Laboratory glassware — Burettes*

ISO 648, *Laboratory glassware — Single-volume pipettes*

ISO 1042, *Laboratory glassware — One-mark volumetric flasks*

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

ISO 4799, *Laboratory glassware — Condensers*

## 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:

- IEC Electropedia: available at <http://www.electropedia.org/>
- ISO Online browsing platform: available at <https://www.iso.org/obp>

## 4 Principle

A weighed test portion of the rubber, in the form of thin strips, is extracted using an ethanol-toluene azeotrope, or, for alum-coagulated rubber, using an ethanol-toluene-water mixture. After making up to a standard volume, an aliquot portion of the extract is withdrawn and titrated with acid-base titration.

- Method A: Titrate with standard acid for the determination of soap and with standard alkali for the determination of organic acid using indicator reagent.
- Method B: Titrate with standard acid for the determination of soap and with standard alkali for the determination of organic acid using automatic potentiometric titrator. The titration equivalence point is determined by inflection point.

- Method C: Back titrate with standard alkali for the determination of soap and organic acid after adding sufficient acid. The titration equivalence point is determined by inflection point.

For methods A and B, there are no precision data available. Therefore their precision is not known. For method C, if the quantity of soap is very low, the standard deviation of the measurement of soap content should be looked at carefully.

## 5 Reagents

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

### 5.1 Ethanol-toluene azeotrope (ETA).

Mix seven volumes of absolute ethanol with three volumes of toluene. Alternatively, mix seven volumes of commercial-grade ethanol with three volumes of toluene, and boil the mixture with anhydrous calcium oxide (quicklime) under reflux for 4 h. Cool to room temperature and decant through No. 42 filter paper.

### 5.2 Ethanol-toluene-water mixture.

Mix 95 cm<sup>3</sup> of ETA (5.1) and 5 cm<sup>3</sup> of water.

### 5.3 Sodium hydroxide solution, $c(\text{NaOH}) = 0,1 \text{ mol/dm}^3$ , accurately standardized.

### 5.4 Thymol blue indicator.

Dissolve 0,06 g of thymol blue in 6,45 cm<sup>3</sup> of 0,02 mol/dm<sup>3</sup> sodium hydroxide solution and dilute to 50 cm<sup>3</sup> with water.

### 5.5 Hydrochloric acid, $c(\text{HCl}) = 0,05 \text{ mol/dm}^3$ , accurately standardized.

## 6 Apparatus

Use ordinary laboratory apparatus and the following.

### 6.1 Balance, accurate to 1 mg.

### 6.2 Hotplate.

### 6.3 Wide-mouthed conical flask, of capacity 400 cm<sup>3</sup> to 500 cm<sup>3</sup>.

### 6.4 Volumetric flask, of capacity 250 cm<sup>3</sup>, complying with the requirements of ISO 1042.

### 6.5 Reflux condenser, complying with the requirements of ISO 4799.

### 6.6 Conical flask, of capacity 250 cm<sup>3</sup>.

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

### 6.7 Burette, of capacity 25 cm<sup>3</sup>, complying with the requirements of ISO 385.

### 6.8 Pipette, of capacity 100 cm<sup>3</sup>, complying with the requirements of ISO 648.

**6.9 Pipette**, of capacity 2 cm<sup>3</sup>, complying with the requirements of ISO 648.

**6.10 Magnetic stirrer**, with a polytetrafluoroethylene-coated stirrer bar.

**6.11 Automatic potentiometric titrator**, including an electrode which is applicable to test the pH of organic solution.

## 7 Sampling and sample preparation

Sheet out 2 g to 6 g of rubber, selected and prepared in accordance with ISO 1795. Cut into pieces no larger than 2 mm × 2 mm or strips no longer than 10 mm and no wider than 5 mm. Weigh a test portion of approximately 2 g to the nearest 0,001 g.

## 8 Procedure

### 8.1 Preparation of the test solution

Place a circular filter paper in the bottom of the wide-mouthed conical flask (6.3), and add 100 cm<sup>3</sup> of ETA extraction solvent (5.1) for all rubbers except alum-coagulated rubbers. For alum-coagulated rubbers, use an ethanol-toluene-water mixture (5.2).

Introduce the strips of rubber separately into the flask, swirling after each addition so that the strips are thoroughly wetted with solvent and sticking is minimized.

Fit the reflux condenser (6.5) to the flask and boil the solvent very gently under reflux for 1 h.

Transfer the extract to the volumetric flask (6.4), and treat the rubber with a second 100 cm<sup>3</sup> portion of extraction solvent under reflux for 1 h. Add this extract also to the volumetric flask. Rinse the strips with three successive 10 cm<sup>3</sup> portions of extraction solvent, add these washings to the volumetric flask and, after cooling to room temperature, adjust the final volume to 250 cm<sup>3</sup> with appropriate extraction solvent.

Alternatively, the weighed strips of sample can be wrapped in filter paper and placed in a Soxhlet extractor (see Note to 6.6) and extracted with an ETA (5.1) or ethanol-toluene-water mixture (5.2) under reflux for a minimum of 4 h.

### 8.2 Method A — Titration method using indicator reagent

#### 8.2.1 Procedure for determination of soap content

After thorough mixing, pipette 100 cm<sup>3</sup> of the diluted extract into the 250 cm<sup>3</sup> conical flask (6.6), add six drops of thymol blue indicator (5.4) and titrate the solution with hydrochloric acid solution (5.5) to the first colour change ( $V_1$ ).

Carry out a blank titration on 100 cm<sup>3</sup> of extraction solvent taken from the same stock as was used for the test portion and using the same indicator as used for titration of the test portion ( $V_2$ ).

#### 8.2.2 Procedure for determination of organic-acid content

Proceed exactly as in 8.2.1, but titrate the aliquot portion with sodium hydroxide solution (5.3), again using thymol blue indicator (5.4) ( $V_3$ ).

Carry out a blank titration on 100 cm<sup>3</sup> of extraction solvent using the same method ( $V_4$ ).

### 8.3 Method B — Titration method using automatic potentiometric titrator

#### 8.3.1 Procedure for determination of soap content

Turn on the automatic potentiometric titrator (6.11), and allow the electronic circuit to stabilize.

Pipette 100 cm<sup>3</sup> of the test solution into a 250 cm<sup>3</sup> beaker containing a stirrer bar, then place the beaker on the magnetic stirrer (6.10). Insert the electrode into the test solution in the beaker.

While stirring, titrate the test solution with hydrochloric acid solution (5.5). Record the volume of hydrochloric acid solution used at the equivalence point that is given as the inflection point of the titration curve ( $V_1$ ).

NOTE The x-axis value of the inflection point of the titration curve is shown as the x-axis value of maximal value point of the derivative of the titration curve. This x-axis value is the volume of hydrochloric acid solution used at the equivalence point.

Carry out a blank titration on 100 cm<sup>3</sup> of extraction solvent using the same method ( $V_2$ ).

#### 8.3.2 Procedure for determination of organic-acid content

Proceed exactly as in 8.3.1, but titrate the aliquot portion with sodium hydroxide solution (5.3). Record the volume of sodium hydroxide solution used at the equivalence point that is given as the inflection point of the titration curve ( $V_3$ ).

NOTE The x-axis value of the inflection point of the titration curve is shown as the x-axis value of the maximal value point of the derivative of the titration curve. This x-axis value is the volume of sodium hydroxide solution used at the equivalence point.

Carry out a blank titration on 100 cm<sup>3</sup> of extraction solvent using the same method ( $V_4$ ).

### 8.4 Method C — Back titration method using automatic potentiometric titrator

#### 8.4.1 Preparation

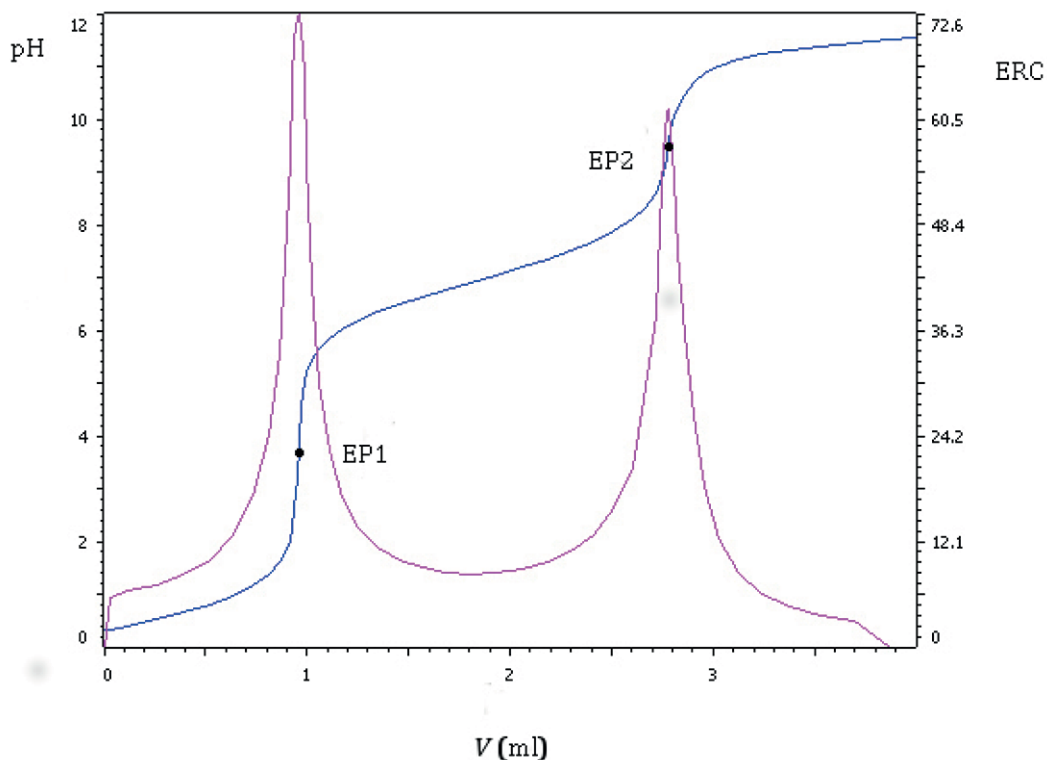
Turn on the automatic potentiometric titrator (6.11), expel the air from the dosing tube and titration tube of the titrator. Set the main parameters referring to Table 1 which gives some recommended values.

NOTE The experimenter can also set the parameters based on his/her own experience, but he/she is supposed to obtain a sample titration curve similar to Figure 1 and a blank titration curve similar to Figure 2.

**Table 1 — Parameters of automatic potentiometric titrator**

Parameters	Set value
Test mode	pH dynamic titration
Volume of dosing tube	5 cm <sup>3</sup>
Stirring rate	A small vortex should be formed but no splash
Regulation of signal drift	50 mV
Minimum waiting time	0 s
Maximum waiting time	25 s
Minimum dosing increment	0,01 cm <sup>3</sup>
Maximum dosing increment	Close
Stop condition	After adding 4 cm <sup>3</sup> of titrant





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Figure 1 — Typical titration curve of sample  
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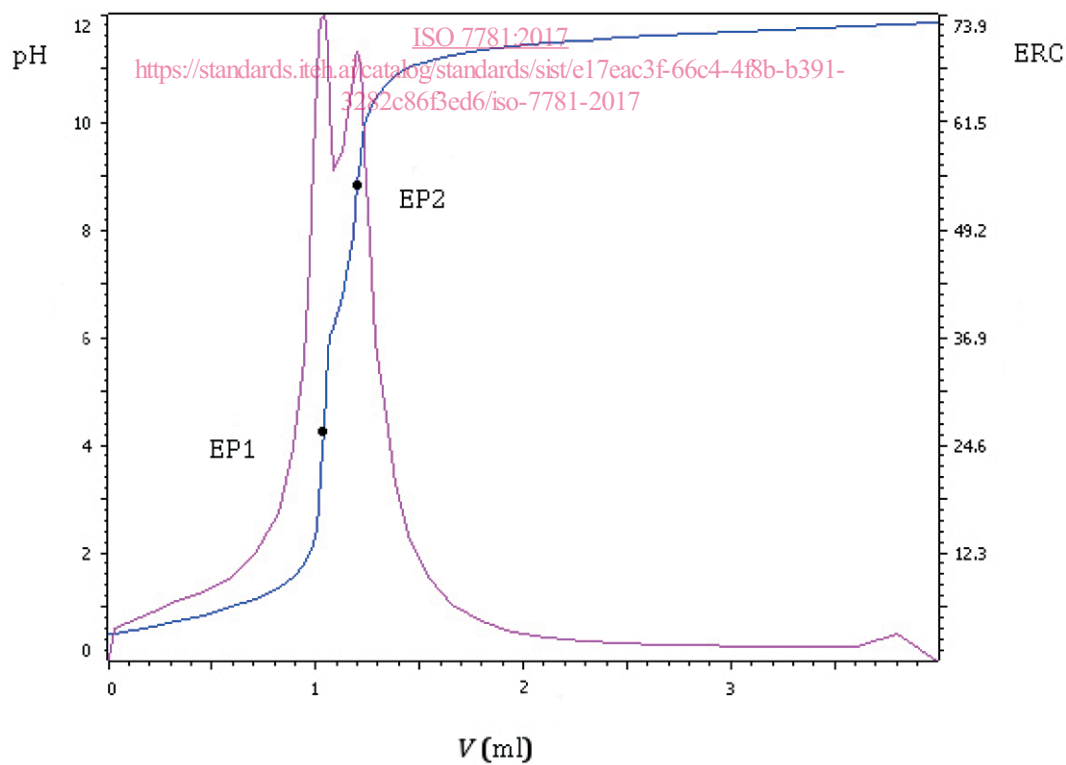


Figure 2 — Typical titration curve of blank