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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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Foreword

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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).

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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 cm^3 of concentrated hydrochloric acid (approximately 35 % mass fraction) to a portion of ETA (5.1) and make the solution up to 1 000 cm^3 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 cm^3 to 500 cm^3.

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 ± 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 Pa[1)] and of maintaining a temperature of 100 °C ± 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 × 210 mm × 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/m^2.

6.10 Light source, which shall be collimated to provide a beam at grazing incidence to the prism.

If an incandescent light source is used, such as a flashlight bulb, it shall be of low intensity. A sodium-vapour lamp may also be used.

The light source requirement is that a clear, sharp line with good contrast can be observed in the telescope of the refractometer. Attenuation or diffusion of the light for better viewing may be accomplished by placing crumpled tissue paper in the light path.

7 Preparation of test pieces

7.1 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 not wider than 5 mm. Weigh a test portion of approximately 2 g to the nearest 0,001 g.

Place a circular filter paper in the bottom of the wide-mouthed conical flask (6.1), and add 100 cm^3 of ETA extraction solvent (5.1) for all rubbers except alum-coagulated rubbers. For alum-coagulated rubbers, use acidified ETA (5.2).

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

Fit the reflux condenser (6.2) to the flask and boil the solvent very gently under reflux for 1 h. Replace the solution with another 100 cm^3 of ETA or acidified ETA and extract for an additional hour. Rinse the rubber pieces with three successive 10 cm^3 portions of extraction solvent.

NOTE As an alternative, the weighed rubber pieces can be wrapped in filter paper and placed in a Soxhlet extractor and extracted with ETA or acidified ETA under reflux for a minimum of 4 h.

Remove the rubber pieces from the flask and dry them to constant mass in the vacuum oven (6.4), maintained at a pressure of about 1 300 Pa[1)] and a temperature of 100 °C ± 5 °C.

It is important that the rubber pieces be extracted and dried thoroughly since either residual solvent or incompletely extracted materials will result in erroneous readings of the refractive index.

Avoid plasticizing of the sample by overheating.

7.2 After the test pieces have been thoroughly dried, more than one technique is suitable for pressing the test piece. The method of pressing may be modified to suit the type of rubber and the type of equipment available. The pressure and the time of pressing at 100 °C may be varied. The test piece may be cooled to room temperature under pressure, or removed from the press while hot. The time of hot pressing shall never exceed 10 min, and should preferably be 5 min.

The conditions shall be chosen so that the pressed test piece is homogeneous and so that a distinct line can be observed dividing the light and dark fields of the telescope field when the refractive index is determined.

The two following techniques are given for the pressing operation.

a) If pressing plates (6.8) are used, proceed as follows.

   Place approximately 0,3 g of the dry extracted polymer between two sheets of aluminium foil about 50 mm square and fold the corners over once. Place this test piece between the pressing plates and place the plates in the press held at 100 °C. Close the platens without applying pressure and preheat for 1 min. Several test pieces may be pressed at one time. Apply a force of about 100 kN for 3 min. Release the pressure, remove the test pieces from the press and allow them to cool.

b) If the pressing is to be done between flat platens without a cavity, proceed as follows, modifying the details of the procedure to suit the sample.

Prepare an aluminium foil about 25 mm wide and 50 mm long. Place approximately 0,2 g of the dry extracted polymer on the foil, then fold the foil along midline. Press the test piece between the foil squares with a force of between 2,2 kN and 6,6 kN at 100 °C for from 3 min to 10 min (preferably 3 min to 5 min). If several test pieces are pressed at one time, increase the applied force proportionally so that the pressure on each test piece is between about 3,45 MPa[1)] and 10,35 MPa[1)]. Forces lower than the usual limit may be necessary for some polymers. It may also be necessary, with some polymers, to allow the pressed test pieces to cool under pressure while cooling the platens with cold water.

7.3 The thickness of the final test piece to be measured shall not exceed 0,5 mm and may be much thinner. The ability to handle the pressed test piece and the ability to obtain a good refractive index reading are the only requirements with respect to test piece thickness.

7.4 Cut the prepared test piece in half with sharp scissors (6.9) and peel off one piece of the foil. Cut off a strip about 6 mm wide by 12 mm long with the scissors, in such a way that one of the narrower ends is freshly cut. The second piece of foil may be removed but it is frequently easier to handle the test piece with one foil piece left on the rubber.

8 Procedure

8.1 Check that the temperature of the refractometer has stabilized at 25 °C.

8.2 Check the adjustment of the refractometer by means of the glass reference (6.6) pressed firmly against the prism, using a drop of α-bromonaphthalene (5.3) as contact liquid. The small light source (6.10) shall be collimated and the best readings are obtained with the glass reference if the light is diffused through crumpled tissue paper.

Move the hand control until the boundary line just reaches the cross-hairs (always moving from the light into the dark field).

Make at least three readings. Adjust the instrument to give the reading of the glass reference.

After this adjustment, clean the prism well with ethanol and a lens paper.

8.3 Place the test piece on the prism with the cut edge toward the light source approximately where the glass reference was positioned. Remove the tissue paper from the light source. Press the test piece firmly on the prism with a finger and wait 1 min for temperature equilibrium. It is also permissible to close the upper prism on the test piece lightly if adequate light can still be focused on the end of the test piece. However, unless the test piece is very thin, this operation can damage the prism or its mounting.

Adjust the compensating prism until a sharp dividing line between the light and dark fields, with minimum colour, is obtained. Test the contact between rubber and prism by pressing the test piece against the prism. There shall be no change in the position of the boundary line during the test.

8.4 Make at least three readings. If the readings differ by more than 0,000 1, further readings are necessary.

8.5 Repeat the process of obtaining readings with another portion of the test piece having a freshly cut edge. Average the mean values of the two sets of readings thus obtained.

If the two mean values do not differ by more than 0,000 2, use this average for the calculation in accordance with Clause 7.

If the difference is more than 0,000 2, the procedure shall be repeated.

If necessary, correct the refractive index measurement to 25 °C using Formula (1):

$$n_{25} = n_{\theta} + 0{,}000\,37\,(\theta - 25) \tag{1}$$

where

n_{25} is the refractive index at 25 °C;

n_{θ} is the refractive index at temperature of measurement θ;

θ is the temperature of measurement, in °C.

9 Expression of results

The bound styrene content, w_s, of the styrene-butadiene rubber, expressed as a percentage mass fraction, is determined from the refractive index, corrected to 25 °C, by using Table 1 or Formula (2):

$$w_s = 23{,}50 + 1\,164\,(n_{25} - 1{,}534\,56) - 3\,497\,(n_{25} - 1{,}534\,56)^2 \tag{2}$$

Table 1 — Values of refractive index at 25 °C and percentage mass fraction of bound styrene

Refractive index n_{25}	0	1	2	3	4	5	6	7	8	9
1,515						0,04	0,17	0,30	0,43	0,56
1,516	0,69	0,82	0,95	1,08	1,21	1,34	1,47	1,60	1,72	1,85
1,517	1,98	2,11	2,24	2,37	2,50	2,62	2,75	2,88	3,01	3,14
1,518	3,27	3,39	3,52	3,65	3,78	3,90	4,03	4,16	4,29	4,41
1,519	4,54	4,67	4,80	4,92	5,05	5,18	5,30	5,43	5,56	5,68
1,520	5,81	5,94	6,06	6,19	6,32	6,44	6,57	6,70	6,82	6,95
1,521	7,07	7,20	7,32	7,45	7,58	7,70	7,83	7,95	8,08	8,20
1,522	8,33	8,45	8,58	8,70	8,83	8,95	9,08	9,20	9,33	9,45
1,523	9,58	9,70	9,83	9,95	10,07	10,20	10,32	10,45	10,57	10,69
1,524	10,82	10,94	11,07	11,19	11,31	11,44	11,56	11,68	11,81	11,93
1,525	12,05	12,18	12,30	12,42	12,54	12,67	12,79	12,91	13,04	13,16
1,526	13,28	13,40	13,52	13,65	13,77	13,89	14,01	14,13	14,26	14,38
1,527	14,50	14,62	14,74	14,87	14,99	15,11	15,23	15,35	15,47	15,59
1,528	15,71	15,83	15,96	16,08	16,20	16,32	16,44	16,56	16,68	16,80
1,529	16,92	17,04	17,16	17,28	17,40	17,52	17,64	17,76	17,88	18,00
1,530	18,12	18,24	18,36	18,48	18,60	18,72	18,84	18,95	19,07	19,19
1,531	19,31	19,43	19,55	19,67	19,79	19,91	20,02	20,14	20,26	20,38
1,532	20,50	20,62	20,73	20,85	20,97	21,09	21,21	21,32	21,44	21,56
1,533	21,68	21,79	21,91	22,03	22,15	22,26	22,38	22,50	22,61	22,73
1,534	22,85	22,96	23,08	23,20	23,31	23,43	23,55	23,66	23,78	23,90
1,535	24,01	24,13	24,24	24,36	24,48	24,59	24,71	24,82	24,94	25,05
1,536	25,17	25,28	25,40	25,51	25,63	25,74	25,86	25,97	26,09	26,20