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Styrene-butadiene rubber (SBR) — Determination of the microstructure of solution-polymerized SBR

Caoutchouc styrène-butadiène (SBR) — Détermination de la microstructure du SBR polymérisé en solution

Reference number
ISO 21561:2005(E)

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Fax + 41 22 749 09 47
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Foreword

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The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 21561 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Styrene-butadiene rubber (SBR) — Determination of the microstructure of solution-polymerized SBR

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard specifies procedures for the quantitative determination of the microstructure of the butadiene units and the content of styrene units in solution-polymerized SBR (S-SBR) by ^{1}H-NMR spectrometry as an absolute method and by IR spectrometry as a relative method. The styrene content is expressed in mass % relative to the whole polymer. The 1,4-trans, 1,4-cis and 1,2-vinyl contents are expressed in mol % relative to the butadiene units.

NOTE IR spectrometry can also give absolute values of microstructure by calibration with S-SBRs of known absolute microstructure obtained by ^{1}H-NMR spectrometry.

2 Normative references

The following referenced documents are indispensable for the application 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 1407, *Rubber — Determination of solvent extract*

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

3 NMR method (absolute method)

3.1 Principle

3.1.1 A small quantity of an extracted S-SBR is dissolved in deuterated-chloroform.

3.1.2 A ^{1}H-NMR spectrum of the sample solution is measured at a 15 ppm sweep width. The peak areas of the 1,4-bond (the sum of the 1,4-trans bond and 1,4-cis bond) and the 1,2-vinyl bond of the butadiene portion are determined along with the peak area of styrene. The microstructure of the butadiene portion and the styrene content are then calculated using theoretical formulae.

3.2 Reagents

3.2.1 **Deuterated chloroform**, $CDCl_3$, containing 0,03 % of tetramethyl silane (TMS) as internal standard. The purity of the $CDCl_3$ itself shall be > 99,8 %.

3.2.2 **Anhydrous ethanol-toluene azeotrope (ETA)**.

3.2.3 Acetone.

3.3 Apparatus

3.3.1 ^{1}H-NMR spectrometer: Fourier transform nuclear magnetic resonance (FT-NMR) spectrometer with a resonance frequency of 150 MHz or higher.

3.3.2 Extraction apparatus, as described in ISO 1407.

3.3.3 Vacuum oven, operated at 50 °C to 60 °C.

3.3.4 Analytical balance, accurate to 0,1 mg.

3.4 Sampling

The raw rubber shall be sampled in accordance with ISO 1795.

3.5 Procedure

3.5.1 Extract rubber additives such as extender oil and antioxidant in accordance with ISO 1407, using anhydrous ETA or acetone as the extraction solvent. Dry the extracted S-SBR under vacuum in the oven at 50 °C to 60 °C.

3.5.2 Take 15 mg to 50 mg of the extracted S-SBR and dissolve it completely in 0,5 ml of deuterated chloroform containing 0,03 % of TMS. The concentration of this sample solution shall be selected according to the resolution of the spectrometer used.

3.5.3 Transfer 0,5 ml of the S-SBR sample solution into an NMR tube for measurement.

3.5.4 Measure the ^{1}H-NMR spectrum of the S-SBR solution under the following conditions:

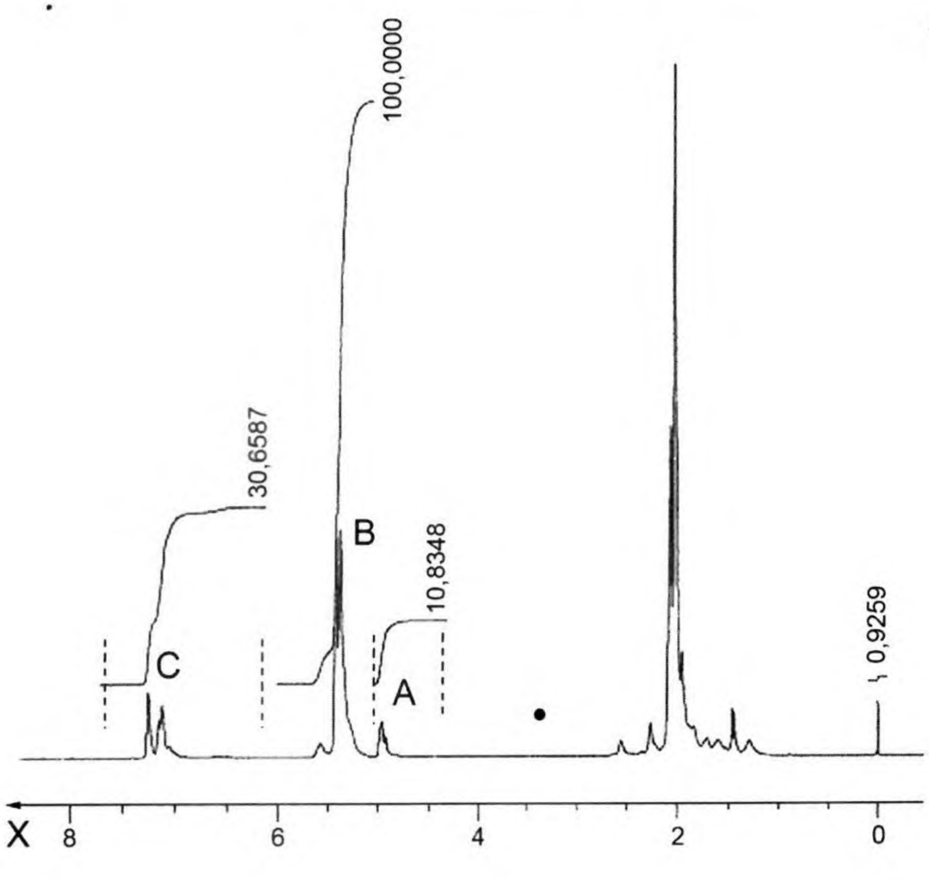
Mode	single-pulse mode
Measurement temperature	room temperature to 50 °C
No. of data points	**32 k**
Offset	5 ppm
Sweep width	15 ppm or wider
Flip angle	30° pulse
Pulse repetition	4 s to 30 s
Accumulation	32 times to 256 times

3.6 Determination of the microstructure

3.6.1 Integrate the signal intensities of the ^{1}H-NMR spectrum over each of the areas A, B and C defined in Table 1. Figure 1 gives an example of a ^{1}H-NMR spectrum showing the areas A, B and C.

Table 1 — Definition of signal integration areas

Area	Signal integration range
A	From 4,3 ppm to minimum intensity point around 5,0 ppm
B	From minimum intensity point around 5,0 ppm to minimum intensity point around 6,1 ppm
C	From minimum intensity point around 6,1 ppm to 7,7 ppm
TMS_{blank}	Integrated signal intensity of TMS in $CDCl_3$ containing TMS
CD_{blank}	From 6,1 ppm to 7,7 ppm in $CDCl_3$ containing TMS
TMS	Integrated signal intensity of TMS in S-SBR sample solution



Key

X shift (ppm)

A, B, C signal integration areas

Figure 1 — Example of a ^{1}H-NMR spectrum for an S-SBR

3.6.2 Measure as a solvent blank the ^{1}H-NMR spectrum of the deuterated chloroform containing 0,03 % of TMS. The TMS blank is normalized using Equation (1) and subtracted from the signal intensity of the solvent determined from the integrated signal intensity of area C:

$$C_{calib} = C - CD_{blank} \times (TMS/TMS_{blank}) \quad (1)$$

3.6.3 Calculate the content of each microstructure component (1,4-bond and 1,2-vinyl bond) of the butadiene portion and the styrene content, using Equations (2) to (4):

$$\% \mathrm{St} = \frac{(C_{\mathrm{calib}}/5) \times 104}{(C_{\mathrm{calib}}/5) \times 104 + (B/2 + A/4) \times 54} \times 100 \qquad (2)$$

$$\% \mathrm{Bd}_{1,2} = \frac{A/2}{B/2 + A/4} \times 100 \qquad (3)$$

$$\% \mathrm{Bd}_{1,4} = \frac{B/2 - A/4}{B/2 + A/4} \times 100 \qquad (4)$$

where

C_{calib}	is the integrated signal intensity of area C compensated for the effect of $CHCl_3$ in $CDCl_3$;
% St	is the styrene content of the SBR, in mass %;
% $\mathrm{Bd}_{1,2}$	is the 1,2-vinyl bond content of the butadiene portion of the SBR, in mol %;
% $\mathrm{Bd}_{1,4}$	is the 1,4-bond content of the butadiene portion of the SBR, in mol %.

4 IR method (relative method)

4.1 Principle

4.1.1 A small quantity of extracted S-SBR is dissolved in cyclohexane and coated on a KBr plate.

4.1.2 The IR spectrum of the S-SBR sample on the KBr plate is measured over the range 1 200 cm^{-1} to 600 cm^{-1}. From the absorbances at four specified wavelengths, the contents of the 1,4-trans bond, the 1,4-cis bond, the 1,2-vinyl bond and the styrene unit are calculated using Hampton's method (see Reference [1] in the Bibliography).

4.1.3 IR spectrometry can also give absolute values of microstructures by calibration with S-SBRs of known absolute microstructure obtained by ^{1}H-NMR spectrometry.

4.2 Reagents

4.2.1 Anhydrous ethanol-toluene azeotrope (ETA).

4.2.2 Acetone.

4.2.3 Cyclohexane.

4.3 Apparatus

4.3.1 ***IR spectrophotometer****: Fourier transform infrared (FT-IR) spectrophotometer or double-beam IR* spectrophotometer.

4.3.2 Extraction apparatus, as described in ISO 1407.

4.3.3 Vacuum oven, operated at 50 °C to 60 °C.

4.3.4 Analytical balance, accurate to 0,1 mg.

4.3.5 **20 ml sample vial**, with cap.

4.3.6 **Pasteur pipette** and **10 ml pipette**.

4.3.7 **KBr plate**, for IR spectrophotometry.

4.3.8 **Holder**, for KBr plate.

4.3.9 **Metal spacer**, of 0,1 mm thickness.

4.4 Sampling

The raw rubber shall be sampled in accordance with ISO 1795.

4.5 Procedure

4.5.1 Preparation of sample solution

4.5.1.1 Extract extender oil and antioxidant from the S-SBR in accordance with ISO 1407, using anhydrous ETA or acetone. Dry the extracted S-SBR in a vacuum oven at 50 °C to 60 °C.

4.5.1.2 Put 0,2 g of extracted S-SBR sample in a 20 ml sample vial.

4.5.1.3 Add 10 ml of cyclohexane to the sample vial using the 10 ml pipette. Place the cap on the vial and shake to dissolve the S-SBR completely.

4.5.2 Preparation of film coating

4.5.2.1 Place the 0,1 mm thick metal spacer on the KBr plate and spread the sample solution uniformly in the spacer hole using the Pasteur pipette.

4.5.2.2 Remove any excess sample solution from the spacer, leaving a film of uniform thickness. As film thickness affects absorbance, adjust the volume of sample solution accordingly.

4.5.2.3 Dry the sample solution on the KBr plate.

4.5.2.4 Remove the spacer from the KBr plate and place the KBr plate in the holder ready for insertion in the IR spectrophotometer.

NOTE Details of the equipment and procedure for preparing the film coating are given in Annex A.

4.5.3 Measurement of IR spectrum

4.5.3.1 Measurement by Fourier transform IR spectrophotometer

4.5.3.1.1 Measure the background spectrum of a blank KBr plate from 1 200 cm^{-1} to 600 cm^{-1}.

4.5.3.1.2 Measure the spectrum of the sample KBr plate from 1 200 cm^{-1} to 600 cm^{-1}.

4.5.3.1.3 In order to ensure good reproducibility, the maximum absorbance for the sample spectrum shall be in the range 0,10 to 0,15.

4.5.3.1.4 An example of an FT-IR spectrum is shown in Figure 2.

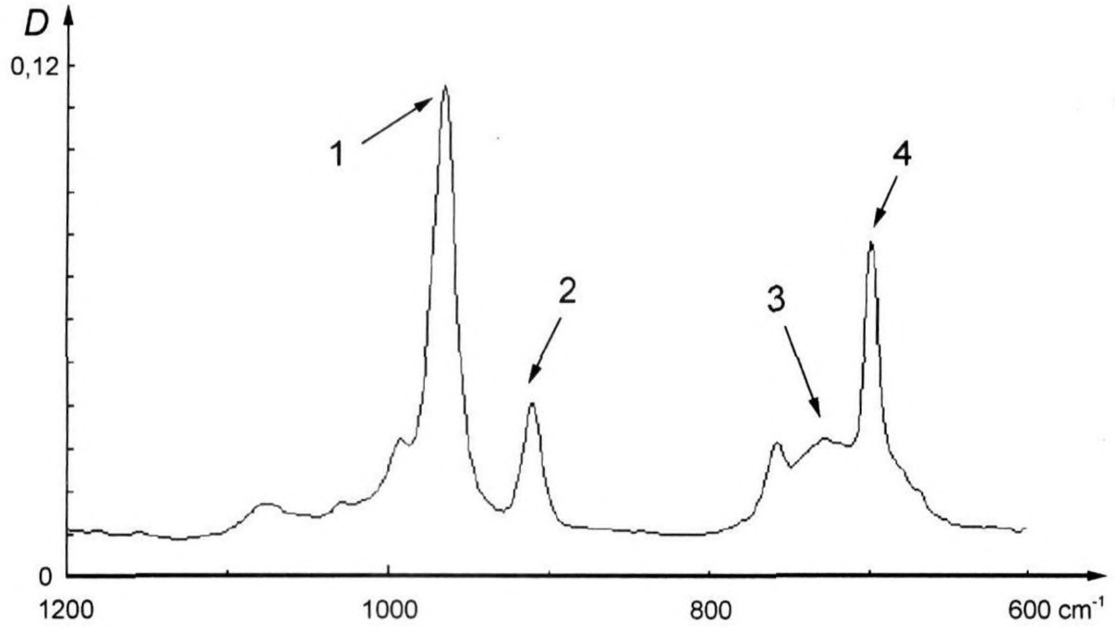
4.5.3.2 Measurement by double-beam IR spectrophotometer

4.5.3.2.1 Place blank KBr plates in both the sample and reference paths, and measure the background spectrum from 1 200 cm^{-1} to 600 cm^{-1} in the absorbance mode.

4.5.3.2.2 Place the sample KBr plate in the sample path and a blank KBr plate in the reference path. Measure the sample spectrum from 1 200 cm^{-1} to 600 cm^{-1} in the absorbance mode.

4.5.3.2.3 In order to ensure good reproducibility, the maximum absorbance for the sample spectrum shall be in the range 0,10 to 0,15.

4.5.3.2.4 Produce a difference spectrum for the calculation of the microstructure by subtracting the background spectrum from the sample spectrum.



Key

1 1,4-trans
2 1,2-vinyl
3 1,4-cis
4 styrene

Figure 2 — An example of an FT-IR spectrum for an S-SBR

4.6 Determination of microstructure

4.6.1 Measurement of absorbance corresponding to each microstructure component

Measure the absorbance values at the wave numbers corresponding to the microstructure components as specified in Table 2. For "1,4-cis", the absorption peak is weak and the wave number of the peak is affected by the styrene content of the polymer.

Table 2 — Measurement of absorbances for each microstructure component of S-SBR

Notation for absorbance	Microstructure component	Wave number cm^{-1}	Remarks
D_{trans}	1,4-trans bond	967	Measure the absorbance at the peak maximum.
D_{vinyl}	1,2-vinyl bond	911	Measure the absorbance at the peak maximum.
D_{cis}	1,4-cis bond	724	The wave number at this peak maximum is affected by the nature of the polymer, such as the styrene content. When the peak maximum is visible, read off the absorbance at the peak maximum from 720 cm^{-1} to 740 cm^{-1}. The absorbance at 724 cm^{-1} is not necessary in this case. If the styrene content is over 30 %, the peak of the 1,4-cis bond is hidden between the two large styrene absorptions at 755 cm^{-1} and 699 cm^{-1}. In this case, measure the absorbance value at 724 cm^{-1}.
$D_{styrene}$	Styrene	699	Measure the absorbance at the peak maximum.

4.6.2 Calculation

4.6.2.1 General

The content of each microstructure component in the S-SBR is calculated from the absorbances (D_{trans}, D_{vinyl}, D_{cis} and $D_{styrene}$) of the sample at the corresponding wave numbers by the equation given by Hampton [1].

4.6.2.2 Normalization

Normalize the measured absorbances using the following equations:

$$\% D_{trans} = \frac{D_{trans}}{D_{trans} + D_{vinyl} + D_{cis} + D_{styrene}} \times 100 \tag{5}$$

$$\% D_{vinyl} = \frac{D_{vinyl}}{D_{trans} + D_{vinyl} + D_{cis} + D_{styrene}} \times 100 \tag{6}$$

$$\% D_{cis} = \frac{D_{cis}}{D_{trans} + D_{vinyl} + D_{cis} + D_{styrene}} \times 100 \tag{7}$$

$$\% D_{styrene} = \frac{D_{styrene}}{D_{trans} + D_{vinyl} + D_{cis} + D_{styrene}} \times 100 \tag{8}$$

4.6.2.3 Calculation of microstructure component concentration

The concentration, in g/l, of each microstructure component (c_{trans}, c_{vinyl}, c_{cis} and c_{styrene}) is expressed by the following equations:

$$c_{\text{trans}} = 0{,}393\,7 \times \%\, D_{\text{trans}} - 0{,}011\,2 \times \%\, D_{\text{vinyl}} - 0{,}036\,1 \times \%\, D_{\text{cis}} - 0{,}006\,5 \times \%\, D_{\text{styrene}} \qquad (9)$$

$$c_{\text{vinyl}} = -0{,}006\,7 \times \%\, D_{\text{trans}} + 0{,}314\,0 \times \%\, D_{\text{vinyl}} - 0{,}015\,4 \times \%\, D_{\text{cis}} - 0{,}007\,1 \times \%\, D_{\text{styrene}} \qquad (10)$$

$$c_{\text{cis}} = -0{,}004\,4 \times \%\, D_{\text{trans}} - 0{,}027\,4 \times \%\, D_{\text{vinyl}} + 1{,}834\,7 \times \%\, D_{\text{cis}} - 0{,}025\,1 \times \%\, D_{\text{styrene}} \qquad (11)$$

$$c_{\text{styrene}} = -0{,}013\,8 \times \%\, D_{\text{vinyl}} - 0{,}260\,4 \times \%\, D_{\text{cis}} + 0{,}373\,9 \times \%\, D_{\text{styrene}} \qquad (12)$$

4.6.2.4 Calculation of microstructure

The content (mol % in butadiene portion or mass % of styrene) of each microstructure component is expressed by the following equations:

$$\%\,\text{trans} = \frac{c_{\text{trans}}}{c_{\text{trans}} + c_{\text{vinyl}} + c_{\text{cis}}} \times 100 \qquad (13)$$

$$\%\,\text{vinyl} = \frac{c_{\text{vinyl}}}{c_{\text{trans}} + c_{\text{vinyl}} + c_{\text{cis}}} \times 100 \qquad (14)$$

$$\%\,\text{cis} = \frac{c_{\text{cis}}}{c_{\text{trans}} + c_{\text{vinyl}} + c_{\text{cis}}} \times 100 \qquad (15)$$

$$\%\,\text{styrene} = \frac{c_{\text{styrene}}}{c_{\text{trans}} + c_{\text{vinyl}} + c_{\text{cis}} + c_{\text{styrene}}} \times 100 \qquad (16)$$

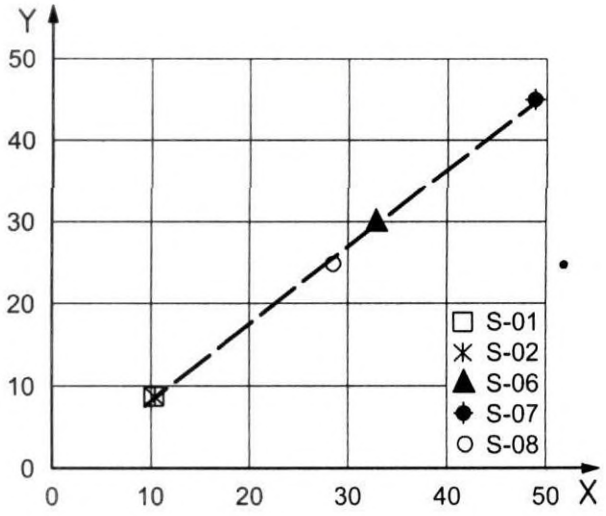
where

% trans	is the 1,4-trans bond content in the butadiene portion of the SBR, in mol %;
% vinyl	is the 1,2-vinyl bond content in the butadiene portion of the SBR, in mol %;
% cis	is the 1,4-cis bond content in the butadiene portion of the SBR, in mol %;
% styrene	is the styrene content in the SBR, in mass %.

4.7 Calibration of IR method to give absolute results

4.7.1 Absolute values of the styrene and vinyl contents can be obtained from IR spectrum measurements by calibration of the IR method using SBR with known styrene and vinyl contents determined by ^{1}H-NMR. Figures 3 and 4 show examples of such a calibration.

4.7.2 The measurement conditions (resolution, apodization, scan time, etc.) used to produce the IR spectrum of the unknown sample shall be the same as the measurement conditions for the standard samples used to make the calibration curve.

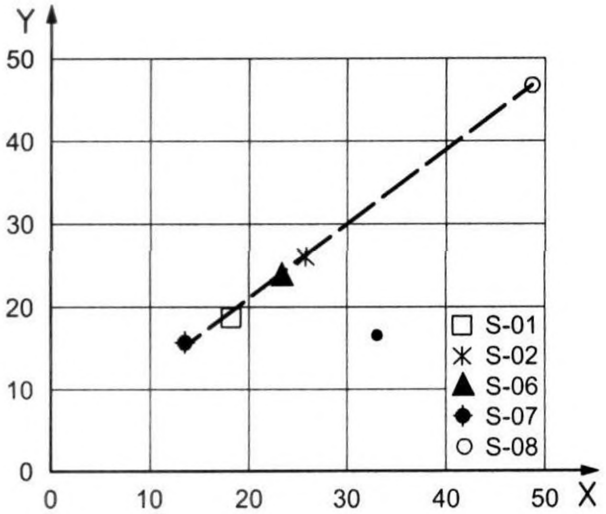


Key

X 1,2-vinyl content by NMR (mol % in butadiene portion)

Y 1,2-vinyl content by IR (mol % in butadiene portion)

Figure 3 — Calibration curve for vinyl content between NMR method and IR method



Key

X styrene content by NMR (mass % in SBR)

Y styrene content by IR (mass % in SBR)

Figure 4 — Calibration curve for styrene content between NMR method and IR method

5 Precision

5.1 The precision of these two methods was determined in accordance with ISO/TR 9272. The results obtained with the NMR method are given in Tables 3 and 4 and the results obtained with the IR method are given in Tables 5 and 6. The precision parameters should not be used for acceptance or rejection of any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific test protocols of the test method.

5.2 The results contained in Tables 3 to 6 are average values and give an estimate of the precision of these test methods as determined in two interlaboratory test programmes involving seven laboratories for the NMR method and eight laboratories for the IR method, the laboratories performing duplicate analyses on two different S-SBRs. The symbols used in the tables are defined as follows:

s_r is the repeatability standard deviation;

r is the repeatability, in measurement units;

(r) is the repeatability, in percent (relative);

s_R is the reproducibility standard deviation;

R is the reproducibility, in measurement units;

(R) is the reproducibility, in percent (relative).

5.3 Repeatability: The repeatability r of the test method has been established as the appropriate value tabulated in Tables 3 to 6. Two single test results that differ by more than the value must be considered suspect and suggest that some appropriate investigative action be taken.

5.4 Reproducibility: The reproducibility R of the test method has been established as the appropriate value tabulated in Tables 3 to 6. Two single test results that differ by more than the value must be considered suspect and suggest that some appropriate investigative action be taken.

Table 3 — Styrene content of S-SBR by NMR method

Sample	Mean value	Within lab			Between labs		
	%	s_r	r	(r)	s_R	R	(R)
SBR-1	25,2	0,04	0,12	0,47	0,13	0,37	1,46
SBR-2	24,7	0,07	0,19	0,75	0,13	0,36	1,44

Table 4 — Vinyl content in butadiene portion of S-SBR by NMR method

Sample	Mean value	Within lab			Between labs		
	%	s_r	r	(r)	s_R	R	(R)
SBR-1	10,4	0,08	0,22	2,15	0,25	0,72	6,88
SBR-2	32,6	0,25	0,71	2,18	0,29	0,81	2,50

Table 5 — Styrene content of S-SBR by IR method

Sample	Mean value	Within lab			Between labs		
	%	s_r	r	(r)	s_R	R	(R)
SBR-1	24,9	0,45	1,27	5,09	0,60	1,71	6,87
SBR-2	25,4	0,41	1,15	4,53	0,67	1,89	7,45

Table 6 — Vinyl content in butadiene portion of S-SBR by IR method

Sample	Mean value	Within lab			Between labs		
	%	s_r	r	(r)	s_R	R	(R)
SBR-1	10,4	0,17	0,48	4,60	0,24	0,69	6,61
SBR-2	32,5	0,14	0,40	1,24	0,36	1,01	3,10

6 Test report

The test report shall include the following:

a) a reference to this International Standard;

b) full identification of the rubber used;

c) the method used (^{1}H-NMR or IR);

d) the results of the determination, expressed in % and rounded to one place of decimals;

e) details of any deviations from the method;

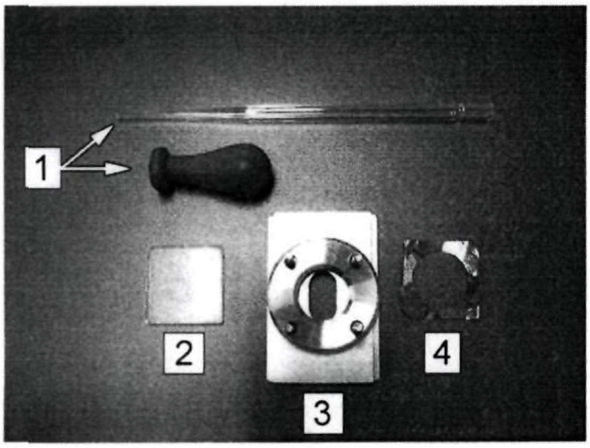
f) the date of the test.

Annex A
(informative)

Procedure for sample-film preparation and measurement of microstructure by FT IR spectrophotometry

A.1 Apparatus

The apparatus used for sample-film preparation and measurement of the microstructure of S-SBR by FT-IR spectrophotometry is shown in Figure A.1.



Key

1	Pasteur pipette
2	35 mm × 35 mm × 5 mm KBr plate for IR spectrophotometry
3	holder for KBr plate
4	0,1 mm thick metal (lead) spacer

Figure A.1 — Apparatus for preparing coated KBr plate

A.2 Procedure

A.2.1 Place the spacer on the KBr plate (see Figure A.2).

A.2.2 Using the Pasteur pipette, put several drops of sample solution in the centre of the hole in the spacer (see Figure A.3).

A.2.3 Remove the excess sample solution by placing another KBr plate over the spacer, lifting it off and wiping the excess solution off with a soft cloth. Repeat this procedure two or three times (see Figure A.4).

A.2.4 Evaporate the sample solution on the KBr plate (see Figure A.5).

A.2.5 Remove the metal spacer from the KBr plate and insert the coated plate in its holder (see Figure A.6).

A.2.6 Mount the plate holder in the optical path of the spectrophotometer (see Figure A.7).

A.2.7 If the maximum absorbance for the sample spectrum is not in the range 0,10 to 0,15, adjust the position of the IR beam passing through the sample.

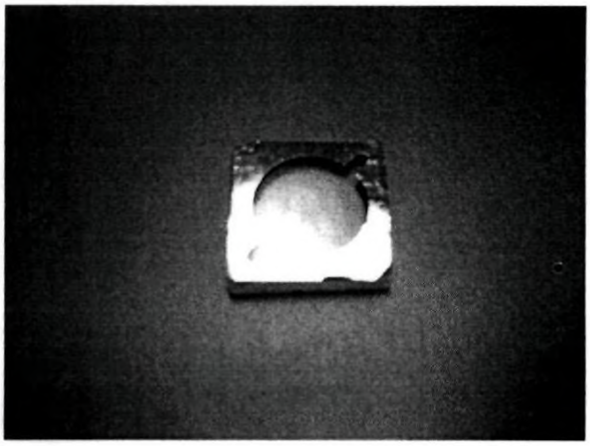


Figure A.2 — Spacer placed on KBr plate

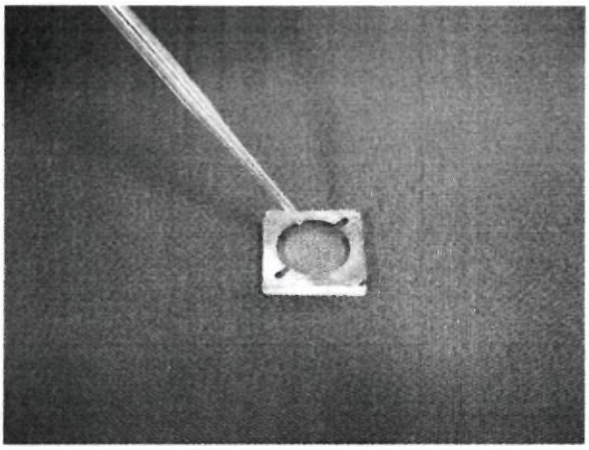


Figure A.3 — Placing drops of sample solution in the hole in the spacer, using a Pasteur pipette

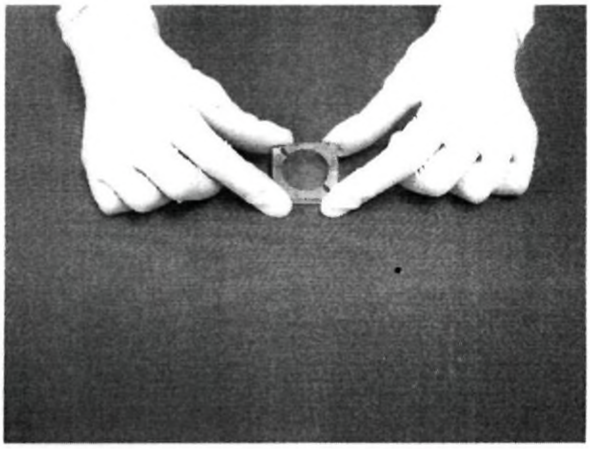


Figure A.4 — Removing excess sample solution

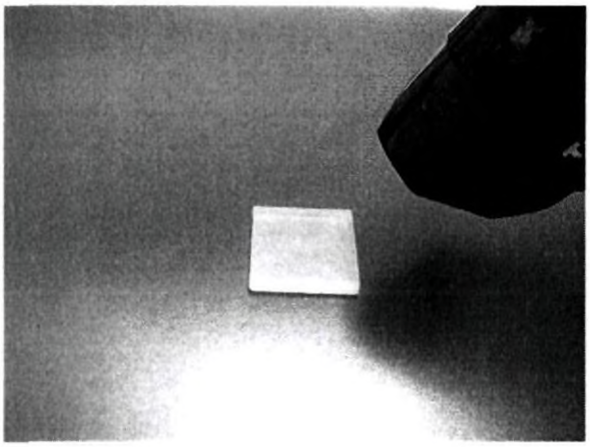


Figure A.5 — Evaporating the sample solution

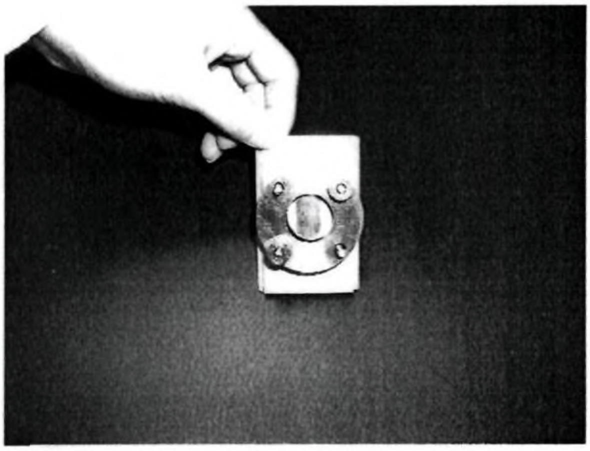


Figure A.6 — Coated KBr plate inserted in the plate holder

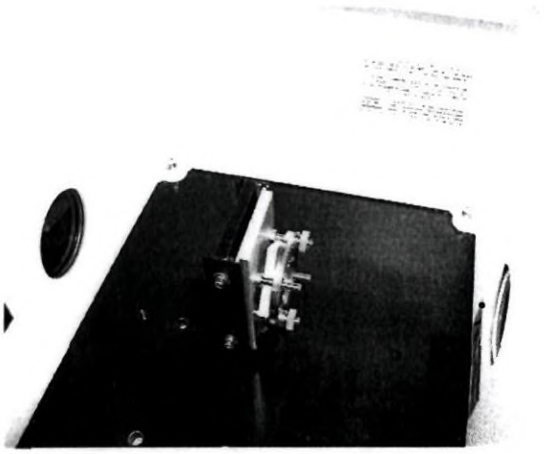


Figure A.7 — Plate holder mounted in the IR spectrometer

Bibliography

[1] HAMPTON, R.R.: *Analyt. Chem.*, **21** (1949), p. 923

[2] ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards* [replaced since the interlaboratory test programme described in Clause 5 by ISO/TR 9272:2005]

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AMENDEMENT 1

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Web www.iso.org

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Amendment 1 to ISO 21561:2005 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

Styrene-butadiene rubber (SBR) — Determination of the microstructure of solution-polymerized SBR

AMENDMENT 1

Page 2, Subclause 3.5.4

In the first line, between "solution" and "under", insert "and a solvent blank (deuterated chloroform containing 0,03 % of TMS)".

Page 3, Subclause 3.6.2

Delete the first sentence.

At the beginning of the second sentence, replace "The TMS blank is normalized using Equation (1) and subtracted from" by "Normalize the solvent blank measured in 3.5.4 using Equation (1) and subtract it from".

Page 7, Table 2

Replace the current Table 2 with the following table, in which the third column (wave number) has been deleted and information on the relevant wave number range given in the last column:

Notation for absorbance	**Microstructure component**	**Remarks**
D_{trans}	1,4-trans bond	Measure the absorbance at the peak maximum from 960 cm^{-1} to 980 cm^{-1}.
D_{vinyl}	1,2-vinyl bond	Measure the absorbance at the peak maximum from 900 cm^{-1} to 920 cm^{-1}.
D_{cis}	1,4-cis bond	The wave number at this peak maximum is affected by the nature of the polymer, such as the styrene content. When the peak maximum is visible, read off the absorbance at the peak maximum from 720 cm^{-1} to 740 cm^{-1}. If the styrene content is over 30 %, the peak of the 1,4-cis bond is hidden between the two large styrene absorptions at 755 cm^{-1} and 699 cm^{-1}. In this case, measure the absorbance value at 724 cm^{-1}.
$D_{styrene}$	Styrene	Measure the absorbance at the peak maximum from 690 cm^{-1} to 710 cm^{-1}.

Page 8, Subclause 4.7

Change the subclause heading to "Conversion of the IR method results to absolute results".

ICS 83.060

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