

BS ISO 21561-1:2015



BSI Standards Publication

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

Part 1:  $^1\text{H}$ -NMR and IR with cast-film  
method

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**National foreword**

This British Standard is the UK implementation of ISO 21561-1:2015. Together with BS ISO 21561-2, it supersedes BS ISO 21561:2005+A1:2010 which will be withdrawn upon publication of BS ISO 21561-2.

The UK participation in its preparation was entrusted to Technical Committee PRI/23, Test methods for rubber and non-black compounding ingredients.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Date	Text affected
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**Styrene-butadiene rubber (SBR) —  
Determination of the microstructure  
of solution-polymerized SBR —**

**Part 1:  
<sup>1</sup>H-NMR and IR with cast-film method**

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

*Partie 1: Méthode <sup>1</sup>H-NMR et IR avec film moulé*





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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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analyses*.

This first edition of ISO 21561-1, together with ISO 21561-2, cancels and replaces ISO 21561:2005, which has been technically and nominally revised with the following changes:

- the descriptions of D's were modified in [4.6.2](#);
- some terms and expressions were revised to be aligned with those in the ISO 21561-2 to be (ATR method);
- it also incorporates the Amendment, ISO 21561:2005/Amd 1:2010.

ISO 21561 consists of the following parts, under the general title *Styrene-butadiene rubber (SBR)*:

- *Part 1: <sup>1</sup>H-NMR and IR with cast-film method*
- *Part 2: FTIR with ATR method*

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

## Part 1:

### <sup>1</sup>H-NMR and IR with cast-film method

**WARNING** — Persons using this International Standard should be familiar with normal laboratory practice. This International 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.

**CAUTION** — Certain procedures specified in this International Standard may involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

## 1 Scope

This part of ISO 21561 specifies procedures for the quantitative determination of the microstructure of the butadiene and the content of styrene in solution-polymerized SBR (S-SBR) by <sup>1</sup>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 trans, cis and vinyl contents are expressed in mol % relative to the butadiene. These methods are applicable only for raw rubber.

NOTE 1 IR spectrometry can also give absolute values of microstructure by calibration with S-SBRs of known absolute microstructure obtained by <sup>1</sup>H-NMR spectrometry.

NOTE 2 Only “vinyl”, “trans” and “cis” are used in this part of ISO 21561. However, the expression of vinyl, trans and cis mean as follows in general:

- vinyl: vinyl unit, vinyl bond, 1,2-unit, 1,2-bond, 1,2-vinyl-unit or 1,2-vinyl-bond;
- trans: 1,4-trans unit, 1,4-trans bond, trans-1,4 unit or trans1,4 bond;
- cis: 1,4-cis unit, 1,4-cis bond, cis-1,4 unit or cis-1,4 bond.

## 2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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  $^1\text{H}$ -NMR spectrum of the sample solution is measured at a 15 ppm sweep width.

With the peak areas obtained from  $^1\text{H}$ -NMR, calculate the microstructure of the butadiene portion and the styrene content by the theoretical formulae.

## 3.2 Reagents

**3.2.1 Deuterated chloroform**,  $\text{CDCl}_3$ , containing 0,03 % of tetramethyl silane (TMS) as internal standard. The purity of the  $\text{CDCl}_3$  itself shall be >99,8 %.

**3.2.2 Ethanol-toluene azeotrope (ETA).**

**3.2.3 Acetone.**

## 3.3 Apparatus

**3.3.1  $^1\text{H}$ -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 antioxidants in accordance with ISO 1407, using 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 (3.2.1). The concentration of this sample solution shall be selected according to the resolution of the spectrometer used.

**3.5.3** Measure the  $^1\text{H}$ -NMR spectrum of the S-SBR solution and a solvent blank [deuterated chloroform (3.2.1)] 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



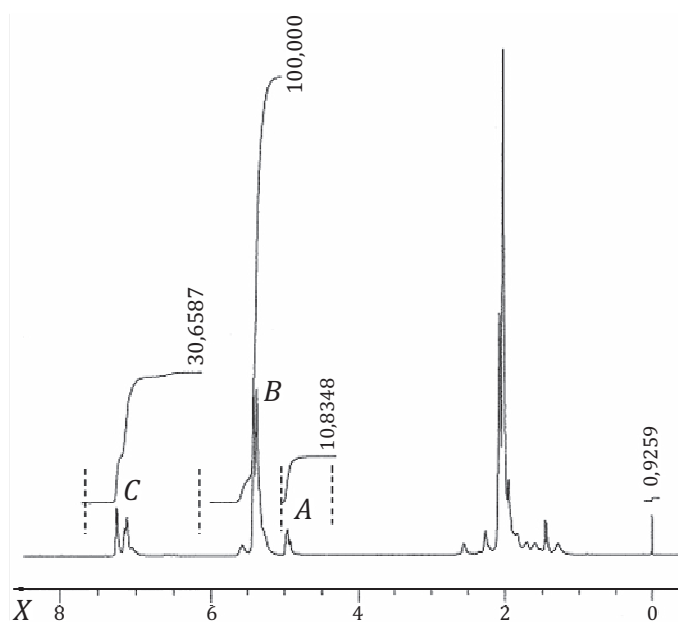
Pulse repetition	4 s to 30 s
Accumulation	32 times to 256 times

### 3.6 Determination of the microstructure

**3.6.1** Integrate the signal intensities of the  $^1\text{H}$ -NMR spectrum over each of the areas *A*, *B* and *C* defined in [Table 1](#). [Figure 1](#) gives an example of a  $^1\text{H}$ -NMR spectrum showing the areas *A*, *B* and *C*.

**Table 1 — Definition of signal integration areas**

Area	Signal integration range
<i>A</i>	From 4,3 ppm to minimum intensity point around 5,0 ppm
<i>B</i>	From minimum intensity point around 5,0 ppm to minimum intensity point around 6,1 ppm
<i>C</i>	From minimum intensity point around 6,1 ppm to 7,7 ppm
$TMS_{\text{blank}}$	Integrated signal intensity of TMS in $\text{CDCl}_3$ containing TMS
$CD_{\text{blank}}$	From 6,1 ppm to 7,7 ppm in $\text{CDCl}_3$ containing TMS
<i>TMS</i>	Integrated signal intensity of TMS in S-SBR sample solution



**Key**

<i>X</i>	shift (ppm)
<i>A, B, C</i>	signal integration areas

**Figure 1 — Example of a Text  $^1\text{H}$ -NMR spectrum for an S-SBR**

**3.6.2** Normalize the solvent blank measured in [3.5.3](#) using Formula (1) and subtract it from the signal intensity of the solvent determined from the integrated signal intensity of area *C*:

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

where

$C_{\text{calib}}$  is the integrated signal intensity of area  $C$  compensated for the effect of  $\text{CHCl}_3$  in  $\text{CDCl}_3$ .

**3.6.3** Calculate the content of each microstructure component (trans and cis, and vinyl) of the butadiene portion and the styrene content, using Formulae (2) to (4):

$$S_m = \frac{(C_{\text{calib}}/5) \times 104}{(C_{\text{calib}}/5) \times 104 + (B/2 + A/4) \times 54} \times 100 \quad (2)$$

$$V = \frac{A/2}{B/2 + A/4} \times 100 \quad (3)$$

$$TC = \frac{B/2 - A/4}{B/2 + A/4} \times 100 \quad (4)$$

where

$S_m$  is the styrene content of the S-SBR, in mass %;

$V$  is the vinyl content of the butadiene portion of the S-SBR, in mol %;

$TC$  is the trans and the cis content of the butadiene portion of the S-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 from 1 200  $\text{cm}^{-1}$  to 600  $\text{cm}^{-1}$ . From the absorbances at four specified wavelengths, the contents of the trans, the cis, the vinyl and the styrene 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  $^1\text{H-NMR}$  spectrometry.

### 4.2 Reagents

**4.2.1 Ethanol-toluene azeotrope (ETA).**

**4.2.2 Acetone.**

**4.2.3 Cyclohexane.**

### 4.3 Apparatus

**4.3.1 IR spectrophotometer,** Fourier transform infrared (FTIR) 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 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<sup>-1</sup> to 600 cm<sup>-1</sup>.

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

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

4.5.3.1.4 An example of an FTIR spectrum is shown in [Figure 2](#).

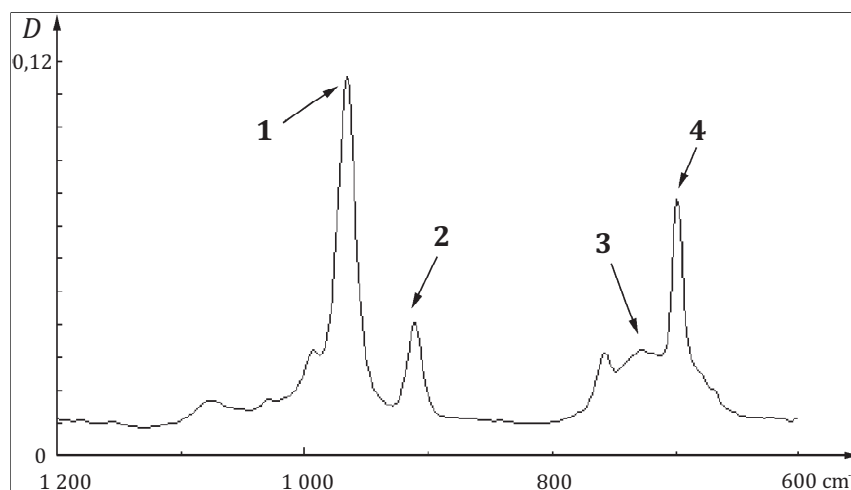
#### 4.5.3.2 Measurement by double-beam IR spectrophotometer

4.5.3.2.1 Place blank KBr plates in both the sample and reference paths, and measure the background spectrum from  $1\,200\text{ cm}^{-1}$  to  $600\text{ cm}^{-1}$  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  $1\,200\text{ cm}^{-1}$  to  $600\text{ cm}^{-1}$  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 from 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 trans
- 2 vinyl
- 3 cis
- 4 styrene

Figure 2 — An example of an FTIR 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 “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	Remarks
$D_t$	trans	Measure the absorbance at the peak maximum from 960 cm <sup>-1</sup> to 980 cm <sup>-1</sup> .
$D_v$	vinyl	Measure the absorbance at the peak maximum from 900 cm <sup>-1</sup> to 920 cm <sup>-1</sup> .
$D_c$	cis	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 <sup>-1</sup> to 740 cm <sup>-1</sup> . If the styrene content is over 30 %, the peak of the cis is hidden between the two large styrene absorptions at 755 cm <sup>-1</sup> and 699 cm <sup>-1</sup> . In this case, measure the absorbance value at 724 cm <sup>-1</sup> .
$D_s$	styrene	Measure the absorbance at the peak maximum from 690 cm <sup>-1</sup> to 710 cm <sup>-1</sup> .

## 4.6.2 Calculation

### 4.6.2.1 General

The content of each microstructure component in the S-SBR is calculated from the absorbances ( $D_t$ ,  $D_v$ ,  $D_c$  and  $D_s$ ) of the sample at the corresponding wave numbers by the formula given by Hampton.<sup>[1]</sup>

### 4.6.2.2 Calculation of microstructure component concentration

The concentration of each microstructure component ( $C_s$ ,  $C_v$ ,  $C_t$ , and  $C_c$ ) is expressed by the following formulae:

$$C_s = -0,013\ 8 \times D_v - 0,260\ 4 \times D_c + 0,373\ 9 \times D_s \quad (5)$$

$$C_v = -0,006\ 7 \times D_t + 0,314\ 0 \times D_v - 0,015\ 4 \times D_c - 0,007\ 1 \times D_s \quad (6)$$

$$C_t = 0,393\ 7 \times D_t - 0,011\ 2 \times D_v - 0,036\ 1 \times D_c - 0,006\ 5 \times D_s \quad (7)$$

$$C_c = -0,004\ 4 \times D_t - 0,027\ 4 \times D_v + 1,834\ 7 \times D_c - 0,025\ 1 \times D_s \quad (8)$$

### 4.6.2.3 Calculation of microstructure

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

$$S_m = \frac{C_s}{C_s + C_v + C_t + C_c} \times 100 \quad (9)$$

$$V = \frac{C_v}{C_v + C_t + C_c} \times 100 \quad (10)$$

$$T = \frac{C_t}{C_v + C_t + C_c} \times 100 \quad (11)$$

$$C = \frac{C_c}{C_v + C_t + C_c} \times 100 \quad (12)$$

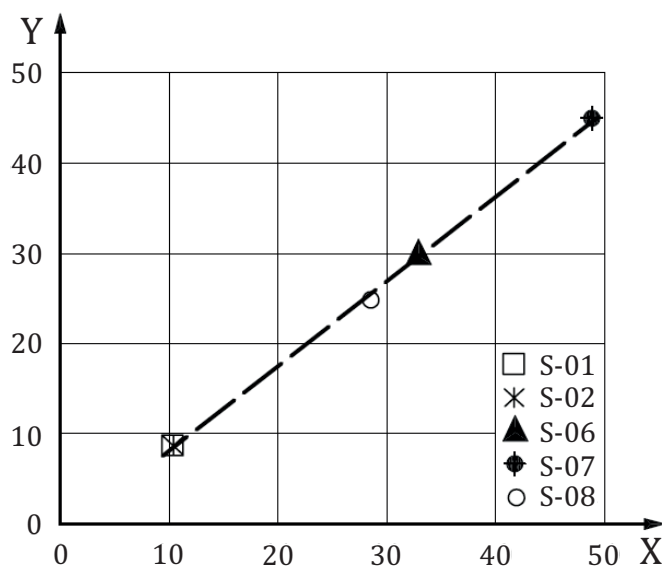
where

- $S_m$  is the styrene content in the S-SBR, in mass %;
- $V$  is the vinyl content in the butadiene portion of the S-SBR, in mol %;
- $T$  is the trans content in the butadiene portion of the S-SBR, in mol %;
- $C$  is the cis content in the butadiene portion of the S-SBR, in mol %.

#### 4.7 Conversion of the IR method results to 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 S-SBR with known styrene and vinyl contents determined by <sup>1</sup>H-NMR. Figures 3 and 4 show examples of such a calibration.

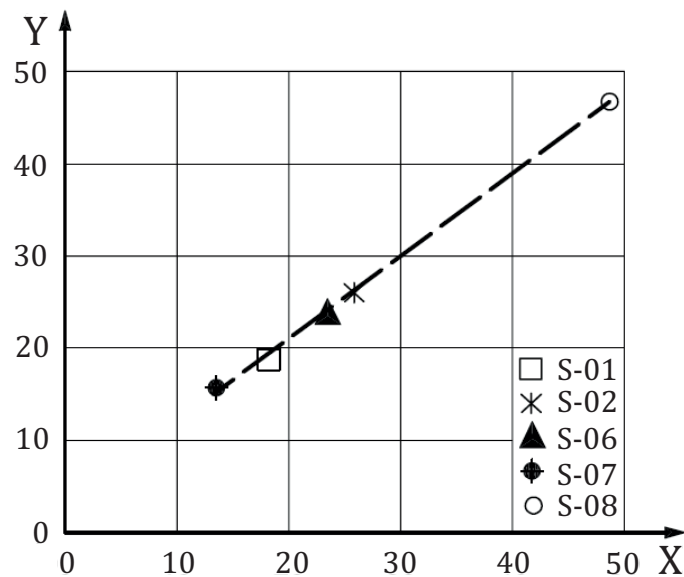
**4.7.2** The measurement conditions (resolution, apodization, scan time, etc.) used to produce the IR spectrum of the unknown sample shall be the same as the measurement conditions for the standard samples used to make the calibration curve.



**Key**

- X vinyl content by NMR (mol % in butadiene portion)
- Y 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 S-SBR)  
Y styrene content by IR (mass % in S-SBR)

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

## 5 Precision

See [Annex B](#).

## 6 Test report

The test report shall include the following information:

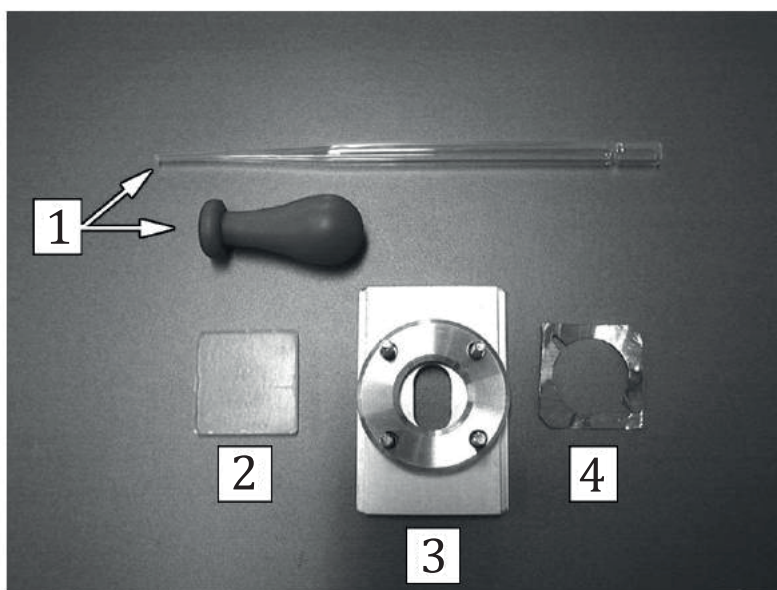
- a) sample details:
  - 1) a full description of the sample and its origin,
  - 2) if appropriate, method of preparation of test piece from the sample;
- b) a reference to this part of ISO 21561, i.e. ISO 21561-1;
- c) test details including any details of any procedures not specified in this part of ISO 21561;
- d) test results:
  - 1) the number of test pieces used,
  - 2) the results of the determination, expressed in % and rounded to one place of decimals;
- e) date(s) of test.

## Annex A (informative)

### Procedure for sample-film preparation and measurement of microstructure by FTIR spectrophotometry

#### A.1 Apparatus

The apparatus used for sample-film preparation and measurement of the microstructure of S-SBR by FTIR 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](#)).



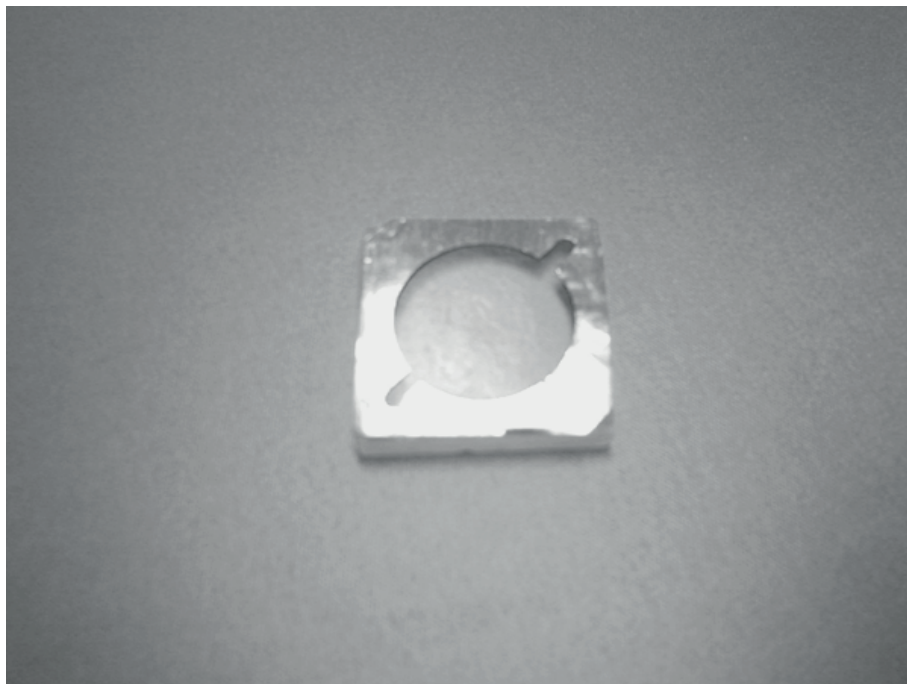
**A.2.3** Remove the excess sample solution by placing another KBr plate over the spacer, lifting it off and wiping the excess solution off with a soft cloth. Repeat this procedure two or three times (see [Figure A.4](#)).

**A.2.4** Evaporate the sample solution on the KBr plate (see [Figure A.5](#)).

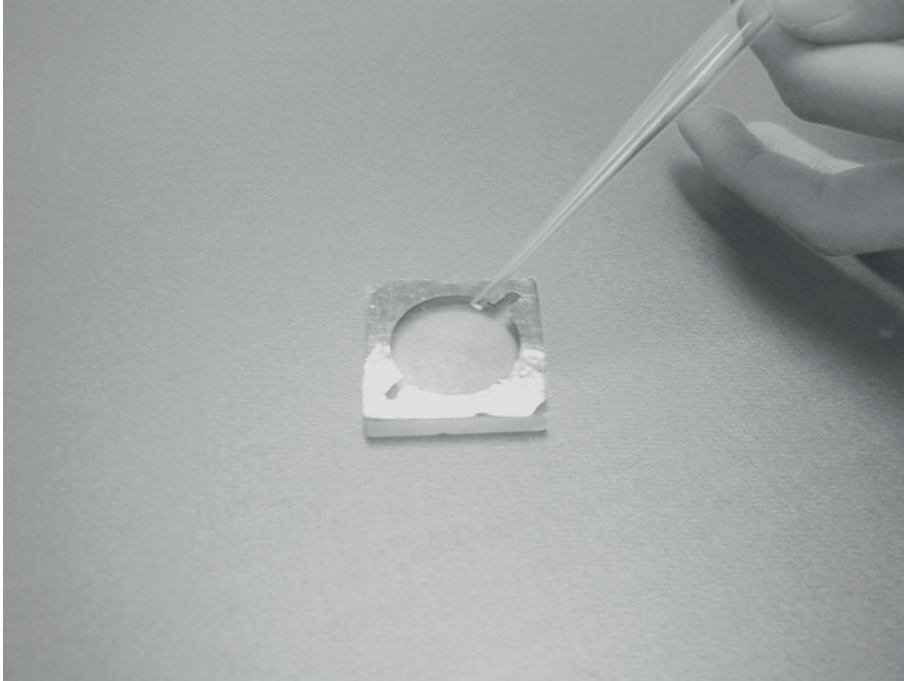
**A.2.5** Remove the metal spacer from the KBr plate and insert the coated plate in its holder (see [Figure A.6](#)).

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

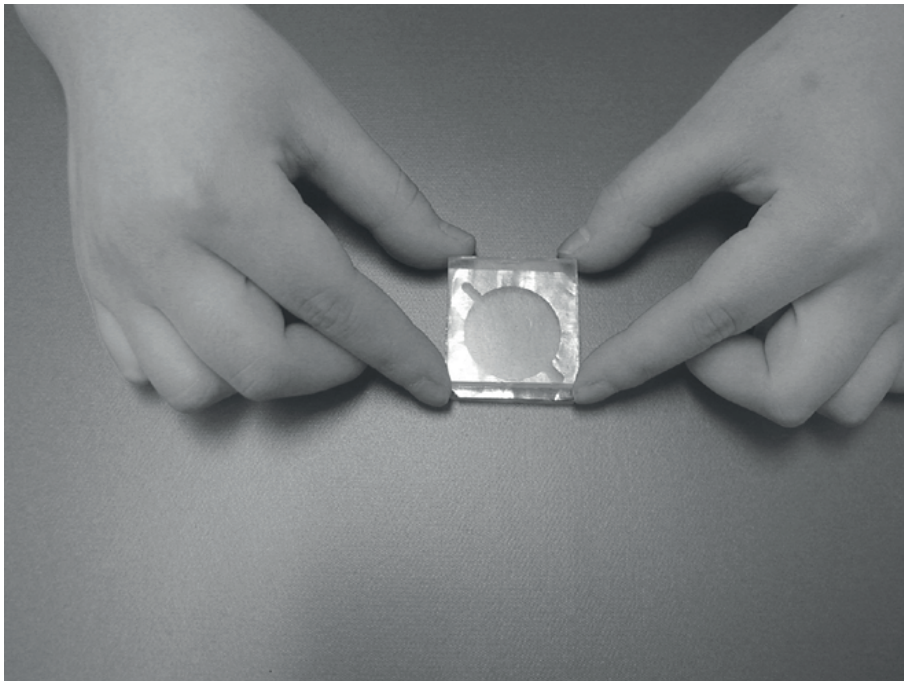
**A.2.7** If the maximum absorbance for the sample spectrum is not in the range from 0,10 to 0,15, adjust the position of the IR beam passing through the sample.



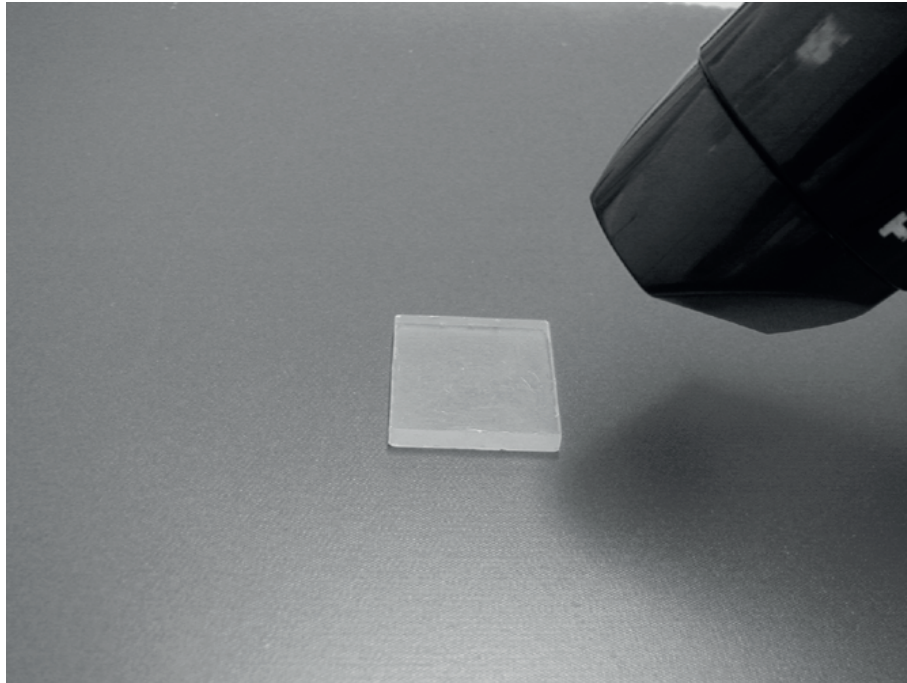
**Figure A.2 — Spacer placed on KBr plate**



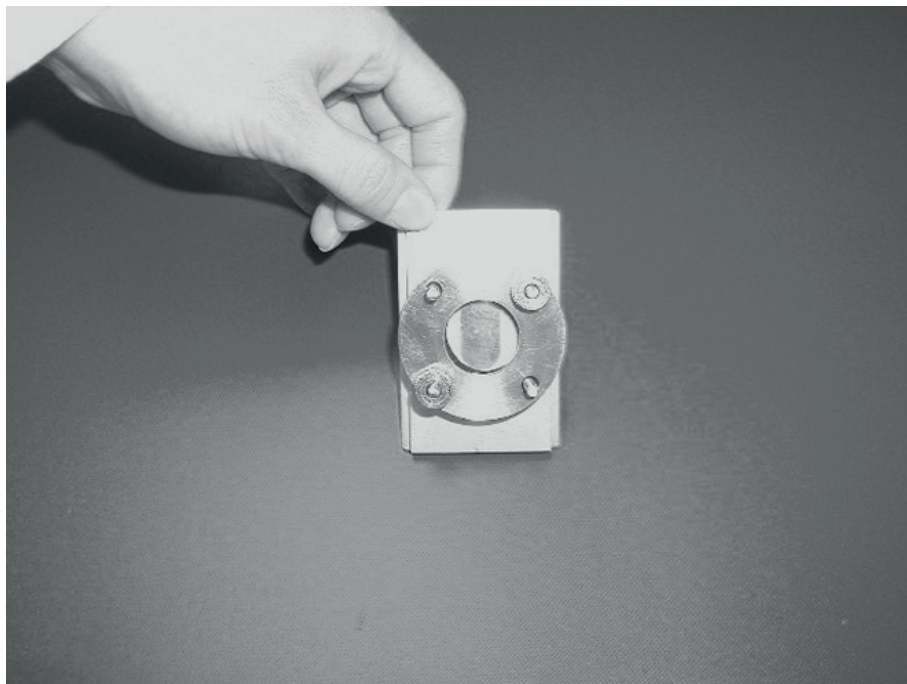
**Figure A.3 — Placing drops of sample solution in the hole in the spacer,  
using a Pasteur pipette**



**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**

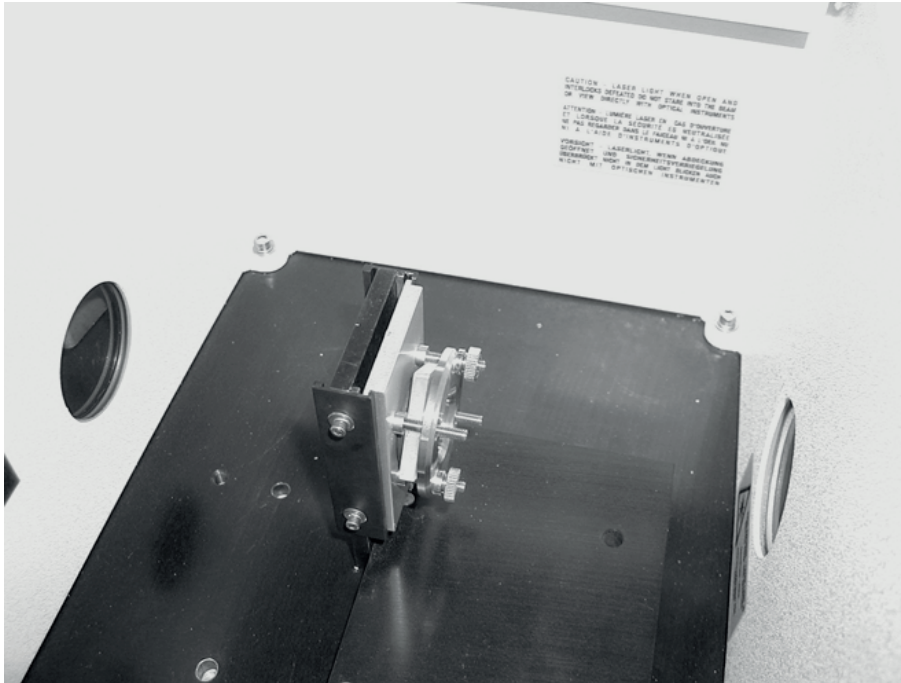


Figure A.7 — Plate holder mounted in the IR spectrometer

## Annex B (informative)

### Precision

**B.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 [Table B.1](#) and [Table B.2](#) and the results obtained with the IR method are given in [Table B.3](#) and [Table B.4](#). 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.

**B.2** The results contained in [Table B.1](#) to [Table B.4](#) are average values and give an estimate of the precision of these test methods as determined in two inter-laboratory 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).

**B.3 Repeatability:** The repeatability  $r$  of the test method has been established as the appropriate value tabulated in [Table B.1](#) to [Table B.4](#). Two single test results that differ by more than the value must be considered suspect and suggest that some appropriate investigative action be taken.

**B.4 Reproducibility:** The reproducibility  $R$  of the test method has been established as the appropriate value tabulated in [Table B.1](#) to [Table B.4](#). 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 B.1 — Styrene content of S-SBR by NMR method**

Sample	Mean value	Within lab			Between laboratories		
	%	$s_r$	$r$	$(r)$	$s_R$	$R$	$(R)$
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 B.2 — Vinyl content in butadiene portion of S-SBR by NMR method**

Sample	Mean value	Within lab			Between laboratories		
	%	$s_r$	$r$	$(r)$	$s_R$	$R$	$(R)$
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 B.3 — Styrene content of S-SBR by IR method**

Sample	Mean value	Within lab			Between laboratories		
	%	$s_r$	$r$	$(r)$	$s_R$	$R$	$(R)$
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 B.4 — Vinyl content in butadiene portion of S-SBR by IR method**

Samples	Mean value	Within lab			Between laboratories		
	%	$s_r$	$r$	$(r)$	$s_R$	$R$	$(R)$
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

## Bibliography

- [1] HAMPTON R.R. Anal. Chem. 1949, **21** p. 923
- [2] ISO/TR 9272, *Rubber and rubber products — Determination of precision for test method standards*







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