# INTERNATIONAL STANDARD

ISO 13226

Second edition 2005-11-01

# Rubber — Standard reference elastomers (SREs) for characterizing the effect of liquids on vulcanized rubbers

Caoutchouc — Élastomères de référence normalisés (SRE) pour la caractérisation de l'effet des liquides sur les caoutchoucs vulcanisés vulcanisés vulcanisés con control de l'effet des liquides sur les caoutchoucs vulcanisés vulcanis



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#### **Foreword**

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

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.

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.

ISO 13226 was prepared by Technical Committee ISO/TC 45, Rubber and rubber products, Subcommittee SC 2, Testing and analysis.

This second edition cancels and replaces the first edition (180 13226:1999), in which the normative references have been updated.

# Introduction

- SRE-MQ/1

SRE-VMQ/1 and SRE-VMQ/1X

The materials covered by this International Standard are specified in Annexes A to N. The following standard elastomers are included:

a) Acrylic rubbers: SRE-NBR 28/P and SRE-NBR 28/PX

SRE-NBR 28/S, SRE-NBR 28/SX, SRE-NBR 34/S and SRE-NBR 34/SX

SRE-NBR/M

SRE-NBR/L

Chlorobutyl rubbers:

SRE-CIIR/1

Chloroprene rubbers:

SRE-CR/1

thylene-propylene rubbers:

SRE-EPM/1

Joropolymer rubbers:

SRE-FKM/1 b) Acrylonitrile-butadiene rubbers: c) Chlorobutyl rubbers: d) Chloroprene rubbers: e) Ethylene-propylene rubbers: — SRE-FKM/2X g) Hydrogenated acrylonitrile-butadiene rubbers: SRE-HNBR/1 and SRE-HNBR/1X h) Natural rubbers: — SRE-NR/1 Silicone rubbers:

# Rubber — Standard reference elastomers (SREs) for characterizing the effect of liquids on vulcanized rubbers

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This standard does not purport to address all of 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 requirements for vulcanized rubbers in sheet form for use as standards in characterizing the effect of test liquids and service fluids. Details of the individual standard reference elastomers are listed in the annexes.

The compounding and preparation ensure that the property profile agrees sufficiently with that of the material group represented, while the simple formulation ensures reliable reproducibility.

The specified property changes of the SRE when in contact with a fluid under specified conditions may be included as supplementary data in specifications for the fluid concerned.

This International Standard is not designed to provide formulations of elastomeric-product compositions for actual service.

#### 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 37, Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties

ISO 48, Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD)

ISO 1817, Rubber, vulcanized — Determination of the effect of liquids

ISO 2230, Rubber products — Guidelines for storage

ISO 2393, Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures

ISO 2781:1988, Rubber, vulcanized — Determination of density

ISO 7619-1, Rubber, vulcanized or thermoplastic — Determination of indentation hardness — Part 1: Durometer method (Shore hardness)

ISO 23529, Rubber — General procedures for preparing and conditioning test pieces for physical test methods

# 3 Composition

The materials shall be homogeneous mixes of the ingredients shown in the relevant annex, in the proportions shown, weighed to the accuracy required in ISO 2393.

All ingredients shall be of recognized rubber quality.

The identification of proprietary materials as suitable ingredients does not exclude the use of other materials that can be shown to meet the requirements of the standard.

If necessary, the quantity of carbon black or silica may be adjusted from one consignment of rubber to another to give properties within the limits specified in the annexes. If the filler content is adjusted, the details shall be declared.

# 4 Preparation

The mixing of compounds and the vulcanization of test sheets shall be carried out in accordance with ISO 2393 as modified by detailed conditions and procedures given in the annexes. If a mould release agent is needed, dry PTFE mould lubricant shall be used.

# 5 Description

The vulcanized test sheets shall be 2 mm  $\pm$  0,2 mm thick when measured in accordance with ISO 23529, free from surface defects and from internal voids when viewed with normally corrected vision.

A "lot" of sheets is those sheets made from a single batch of rubber mix vulcanized under the same conditions. A "pressing" is the quantity of sheets produced at the same time in a single moulding operation.

# 6 Test sheet properties

The test sheets shall be characterized by one or more physical properties in accordance with the relevant ISO standard test method. Properties and tolerances for individual materials are given in the annexes.

All sheets shall be tested for compliance with the requirements of Clause 5. A sample sheet from each lot shall be tested for the properties of interest for compliance with the tolerances given in the appropriate annex. The actual values obtained shall be reported.

The test pieces necessary for determining material properties shall be taken from the test sheets in such a way that a minimum distance from the edge of the sheets is maintained. Bar-shaped test pieces shall be taken with their longitudinal axis parallel to the direction of milling of the moulding blank.

#### 7 Designation

Each elastomer shall be designated as ISO 13226 SRE-A/B where ISO 13226 is this International Standard, SRE is the abbreviation for standard reference elastomer and, after the hyphen, A is a set of code-letters designating the polymer type and B is a descriptor identifying the particular compound. Some designations end with the letter X to signify that additional test requirements apply.

#### 8 Storage

Test sheets shall be stored in accordance with ISO 2230. After one year, they shall be retested or disposed of. If the sheets still meet the test requirements, they may be stored and used for a further year.

# Annex A

(normative)

# Acrylic rubbers: SRE-ACM/1 and SRE-ACM/1X

### A.1 Purpose

These SREs are representative of ACM materials such as are used, for instance, for parts in contact with petroleum products in the mechanical-engineering and automobile sectors.

They are used for the characterization of service fluids such as mineral oils, fuels, lubricants, hydraulic fluids, coolants and refrigerants with regard to their effect on vulcanized acrylic rubbers.

The changes in mass, volume, hardness, tensile strength and elongation at break of the SRE when in contact with the service fluid under specified conditions may be included as supplementary data in specifications for the fluid concerned.

# A.2 Composition

Table A.1 — Composition of the SRE

Ingredients	Parts by mass
Acrylic rubber <sup>a</sup>	100,0
Stearic acid	1,0
Pentaerythrite stearate <sup>b</sup>	2,0
Octylated diphenylamine <sup>c</sup> (ODPA)	2,0
Carbon black, N550	65,0
Sodium stearate	4,0
Quaternary ammonium salts <sup>d</sup>	2,0
Total	176,0

Vulkanox OCD/SG® from Bayer AG, or equivalent.

HyTemp NPC-50® from Zeon Chemicals L.P., or equivalent.

# A.3 Recommended mixing procedure

Mix on a two-roll mill (see Clause A.6).

# A.4 Vulcanization

Condition the sheeted compound at ambient temperature for 12 h to 48 h.

Press-cure at 180 °C  $\pm$  2 °C for 10 min  $\pm$  1 min.

Post-cure at 175 °C  $\pm$  2 °C for 4 h  $\pm$  0,5 h.

Struktol WB 222® from Schill & Seilacher, or equivalent.

# A.5 Test sheet properties

Material SRE-ACM/1 shall fulfil the basic property given in Table A.2. Additional properties may be specified (see Table A.3).

Material SRE-ACM/1X shall fulfil both the basic and the additional properties.

### Table A.2 — Basic property

Property	Unit	Requirement	Test method
Increase in mass in test liquid B for fuels, as in ISO 1817	%	26 to 29	ISO 1817, three type 2 dumb-bell test pieces $^a$ immersed for 22 h $\pm$ 0,5 h at 23 °C $\pm$ 2 °C Test piece/test liquid volume ratio: $1/(30 \pm 5)$
a As specified in ISO 37.			

#### Table A.3 — Additional properties

Property	Unit	Requirement	Test method
Tensile strength	MPa	12 to 16	ISO 37, five type 2 dumb-bell test pieces
Elongation at break	%	140 to 220	O.
Hardness	Shore A	69 to 74	ISO 7619-1, three type 2 dumb-bell test pieces <sup>a</sup> , three plies
	IRHD	69 to 74	SO 48, three type 2 dumb-bell test pieces <sup>a</sup> , three plies
Density	Mg/m <sup>3</sup>	1,30 to 1,34	ISO 2781:1988, method A, three test pieces
a As specified in ISO 37.		×O	

# A.6 Mixing procedures

The following mixing procedure can be used to produce SRE-ACM/1X material:

Roll diameter: 200 mm Working width: 395 mm Speed of rolls: 18/22 min<sup>-1</sup>

Surface temperature of rolls: 70 °C ± 5 °C

Mass of rubber: 650 g

min	Nip opening mm
0	$2.0 \pm 0.5$
1	
11	
13	$\textbf{2,2} \pm \textbf{0,5}$
17	
19	
21	
	0 1 11 13 17 19

Final temperature of sheet: approx. 75 °C

<sup>&</sup>lt;sup>a</sup> It is recommended that some of the final cuts are made during the addition of the sodium stearate to finalize the mixing procedure so that the sheet is taken off not later than 6 min  $\pm$  1 min after the accelerator has been added.

# Annex B

(normative)

# Acrylonitrile-butadiene rubbers: SRE-NBR 28/P and SRE-NBR 28/PX

### **B.1 Purpose**

These SREs are representative of peroxide-cured NBR materials such as are used, for instance, for parts in contact with petroleum products in the mechanical-engineering and automobile sectors.

They are used for the characterization of service fluids such as mineral oils, fuels, lubricants, hydraulic fluids, coolants and refrigerants with regard to their effect on vulcanized nitrile rubbers.

The changes in mass, volume, hardness, tensile strength and elongation at break of the SRE when in contact with the service fluid under specified conditions may be included as supplementary data in specifications for the fluid concerned.

# **B.2 Composition**

Table B.1 — Composition of the SRE

Ingredients	Parts by mass
NBR with (28 $\pm$ 0,5) % by mass of acrylonitrile $^{3}$	100,0
N-(1,3-dimethylbutyl)-N-phenyl-p-phenylenediamine (6PPD) <sup>b</sup>	0,5
Zinc oxide, precipitated <sup>c</sup>	5,0
Carbon black, N550	70,0
Dicumyl peroxide (40 % by mass) <sup>d</sup>	3,0
Total	178,5

- <sup>a</sup> Perbunan NT 2845® from Bayer AG, or equivalent.
- b Vulkanox 4020® from Bayer AG, or equivalent.
- C Zinkoxyd aktiv<sup>®</sup> from Bayer AG, or equivalent.
- d Perkadox BC 40<sup>®</sup> from Akzo-Nobel Chemicals BV, or equivalent.

# B.3 Recommended mixing procedure

Prepare a masterbatch without the peroxide in an internal mixer followed by homogenizing and addition of peroxide on a two-roll mill (see B.6.1).

Alternatively, mix completely on a two-roll mill (see B.6.2).

#### **B.4 Vulcanization**

Condition the sheeted compound at ambient temperature for 20 h to 24 h.

Press-cure at 170 °C  $\pm$  2 °C for 20 min  $\pm$  1 min.

# **B.5 Test sheet properties**

SRE-NBR 28/P material shall fulfil the basic property given in Table B.2. Additional properties may be specified (see Table B.3).

SRE-NBR 28/PX material shall fulfil both the basic and the additional properties.

Table B.2 — Basic property

Property	Unit	Requirement	Test method
Increase in mass in test liquid B for fuels, as in ISO 1817	%	25 to 28	ISO 1817, three type 2 dumb-bell test pieces <sup>a</sup> immersed for 22 h $\pm$ 0,5 h at 23 °C $\pm$ 2 °C  Test piece/test liquid volume ratio: 1/(30 $\pm$ 5)
a As specified in ISO 37.			376.

#### Table B.3 — Additional properties

Property	Unit	Requirement	Test method
Tensile strength	MPa	20 to 25	ISO 37, five type 2 dumb-bell test pieces
Elongation at break	%	170 to 220	
Hardness	Shore A	79 to 84	ISO 7619-1, three type 2 dumb-bell test pieces <sup>a</sup> , three plies
	IRHD	79 to 84	ISO 48, three type 2 dumb-bell test pieces <sup>a</sup> , three plies
Density	Mg/m <sup>3</sup>	1,21 to 1,25	ISO 2781:1988, method A, three test pieces
a As specified in ISO 37.		7/2	

# **B.6 Mixing procedures**

The following mixing procedures can be used to produce SRE-NBR 28/PX material:

#### B.6.1 Internal mixer and subsequent two-roll mill

With cooling fully operative.

Example of a mixing procedure for preparation of a masterbatch without peroxide, using an internal mixer fitted with intermeshing blades:

Mixing-chamber volume <sup>a</sup> : 4,6 dm <sup>3</sup>	
Chamber filled to: (65 $\pm$ 5) % <sup>b</sup>	
Speed <sup>c</sup> : 30 min <sup>-1</sup>	
Mass of rubber: 2 000 g	
Mixing step	Elapsed time min
Add rubber	0
Add zinc oxide and 6PPD	2
Add carbon black	3
Dump	8
Final temperature of masterbatch: approx. 120 °C	
Determined by means of wheat grains.	
b When using a mixer fitted with non-intermeshing blades, fill the chamber	to (80 + 5) %.

Example of a mixing procedure for homogenization of masterbatch and addition of peroxide using a two-roll mill:

Roll diameter: 250 mm Working width: 410 mm

Speed of rolls:

0 min to 13 min: 12/18 min<sup>-1</sup> 14 min to 25 min: 12/12 min<sup>-1</sup> Surface temperature of rolls: 50 °C  $\pm$  5 °C

Mixing step	Elapsed time min	Nip opening mm
Band hot masterbatch	0	000
Make 3/4 cuts (14 from each side)	1	S. V
Turn the rolled sheet (eight times)	8	
Make 3/4 cuts (two from each side)	13	
Add peroxide	14	$3,0\pm0,5$
Make 3/4 cuts (five from each side)	16	
Turn the rolled sheet (four times)	19	
Make 3/4 cuts (five from each side)	21	
Turn the rolled sheet (four times)	23	
Sheet off	25	1,5 ± 0,5
Final temperature of sheet: approx. 75 °C	<u>e</u>	

# B.6.2 Mixing using a two-roll mill

Roll diameter: 200 mm Working width: 395 mm

Speed of rolls: 20/24 min<sup>-1</sup>

Surface temperature of rolls: 50 °C+5

Mass of rubber: 1 000 g

Mixing step	Elapsed time min	Nip opening mm
Band rubber	0	
Add zinc oxide and 6PPD	3	$2.0\pm0.5$
Make 3/4 cuts (three from each side)	4	
Add carbon black	6	Set stepwise to 3,2 ± 0,5
Add peroxide	17	
Make 3/4 cuts (six from each side)	19	3 2 + 0 5
Turn the rolled sheet (four times)	23	$3,2\pm0,5$
Sheet off	26	1,5 ± 0,5
Final temperature of sheet: approx. 90 °C		

# Annex C

(normative)

# Acrylonitrile-butadiene rubbers: SRE-NBR 28/S, SRE-NBR 28/SX, SRE-NBR 34/S and SRE-NBR 34/SX

# **C.1 Purpose**

These SREs are representative of low-sulfur-cured NBR materials such as are used, for instance, for parts in contact with petroleum products in the mechanical-engineering and automobile sectors.

They are used for the characterization of service fluids such as mineral oils, fuels, lubricants, hydraulic fluids, coolants and refrigerants with regard to their effect on vulcanized nitrile rubbers.

In order to cover a wide range of service fluids, two different acrylonitrile (ACN) contents and thus a different swelling behaviour of the SREs are specified as follows:

- ISO 13226 SRE-NBR 28/S and SRE-NBR 28/SX (ACN content 28 %);
- ISO 13226 SRE-NBR 34/S and SRE-NBR 34/SX (ACN content 34%).

The changes in mass, volume, hardness, tensile strength and elongation at break of the SRE when in contact with the service fluid under specified conditions may be included as supplementary data in specifications for the fluid concerned.

#### **C.2 Composition**

Table CM - Composition of the SRE

Ingredients	Parts by mass		
	28/S 28/SX	34/S 34/SX	
NBR (ACN content 28 %) <sup>a</sup>	100,0	_	
NBR (ACN content 34 %) <sup>b</sup>	_	100,0	
Polymerized 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ) <sup>c</sup>	2,0	2,0	
Zinc oxide, precipitated <sup>d</sup>	5,0	5,0	
Steario acid	1,0	1,0	
Carbon black, N550	65,0	65,0	
Tetramethylthiuram disulfide (TMTD) <sup>e</sup>	2,5	2,5	
N-Cyclohexylbenzothiazyl-2-sulfenamide (CBS) <sup>f</sup>	1,5	1,5	
Sulfur	0,2	0,2	
Total	177,2	177,2	

Perbunan NT 2845<sup>®</sup> from Bayer AG, or equivalent.

b Perbunan NT 3445<sup>®</sup> from Bayer AG, or equivalent.

Vulkanox HS/LG® from Bayer AG, or equivalent.

<sup>&</sup>lt;sup>d</sup> Zinkoxyd aktiv<sup>®</sup> from Bayer AG, or equivalent.

e Vulkacit Thiuram/C<sup>®</sup> from Bayer AG, or equivalent.

f Vulkacit CZ/C® from Bayer AG, or equivalent.

# C.3 Recommended mixing procedures

Prepare a masterbatch without the TMTD, CBS and sulfur in an internal mixer followed by homogenizing and addition of curing agents on a two-roll mill (see C.6.1).

To obtain good sulfur dispersion, use about 10 % of the rubber to make a sulfur masterbatch.

Alternatively, mix completely on a two-roll mill. Prepare a sulfur masterbatch and store it for between 30 min and 24 h before proceeding with the main mix (see C.6.2).

#### C.4 Vulcanization

Condition the sheeted compound at ambient temperature for 2 h to 24 h.

Press-cure at 160 °C  $\pm$  2 °C for 20 min  $\pm$  1 min.

# C.5 Test sheet properties

SRE-NBR 28/S and SRE-NBR 34/S materials shall fulfil the basic property given in Table C.2. Additional properties may be specified (see Table C.3).

SRE-NBR 28/SX and SRE-NBR 34/SX materials shall fulfil both the basic and the additional properties.

Table C.2 — Basic property

Property	Unit	Requir	rement	Test method
		28/S 28/SX	34/S 34/SX	
Increase in mass in test liquid B for fuels, as in ISO 1817	() ()	26 to 29	17 to 20	ISO 1817, three type 2 dumb-bell test pieces <sup>a</sup> immersed for 22 h ± 0,5 h at 23 °C ± 2 °C
a As specified in ISO 37.	7.			Test piece/test liquid volume ratio: $1/(30 \pm 5)$

Table C.3 — Additional properties

Property	Unit	Requirement		Test method
ATI		28/SX	34/SX	
Tensile strength	MPa	20 to 25	20 to 25	ISO 37, five type 2 dumb-bell test pieces
Elongation at break	%	300 to 400	300 to 400	
Hardness	Shore A	76 to 81	77 to 82	ISO 7619-1, three type 2 dumb-bell test pieces <sup>a</sup> , three plies
	IRHD	76 to 81	77 to 82	ISO 48, three type 2 dumb-bell test pieces <sup>a</sup> , three plies
Density	Mg/m <sup>3</sup>	1,19 to 1,23	1,20 to 1,24	ISO 2781:1988, method A, three test pieces
a As specified in ISO 37	7.			

# C.6 Mixing procedures

The following mixing procedures can be used to produce SRE-NBR 28/SX and SRE-NBR 34/SX materials:

#### C.6.1 Internal mixer and subsequent two-roll mill

Use an internal mixer to prepare a masterbatch, without the vulcanizing ingredients, e.g. as specified in Table C.4.

Homogenize using a two-roll mill and add the vulcanizing ingredients, e.g. as specified in Table C.5.

To achieve good sulfur dispersion, which is essential for good tensile characteristics, take about 10 % of the rubber and mix a sulfur batch in accordance with Table C.6 (with properly adjusted working width and/or nip opening). Add this batch on the two-roll mill after step 2, as specified in Table C.5.

Table C.4 — Mixing procedure for preparation of a masterbatch without vulcanizing ingredients, using an internal mixer fitted with intermeshing blades (example)

_	
Mixing-chamber volume <sup>a</sup> : 4,6 dm <sup>3</sup>	<u> </u>
Chamber filled to: $(65 \pm 5) \%^b$	
Speed <sup>c</sup> : 30 min <sup>-1</sup>	
Mass of rubber: 2 000 g	
Mixing step	Elapsed time min
Add rubber	0
Add zinc oxide and TMQ	2
Add carbon black and stearic acid	3
Dump	8
Final temperature of masterbatch: approx. 120 °C	
a Determined by means of wheat grains.	
$^{\mbox{\scriptsize b}}$ When using a mixer fitted with non-intermeshing blades, fill the chamber to (80 $\pm$	5) %.
With cooling fully operative.	

Table C.5 — Mixing procedure for homogenization of masterbatch and addition of vulcanizing ingredients, using a two-roll mill (example)

Roll diameter: 250 mm
Working width: 410 mm
Speed of rolls:
0 min to 13 min: 12/18 min-1
14 min to 25 min: 12/12 min-1
Surface temperature of rolls: 50 °C ± 5 °C

Mixing step	Elapsed time min	Nip opening mm
Band hot masterbatch	0	
Make 3/4 cuts (14 from each side)	1	
Turn the rolled sheet (eight times)	8	
Make 3/4 cuts (two from each side)	13	
Add sulfur, TMDT and CBS	14	$3,0\pm0,5$
Make 3/4 cuts (five from each side)	16	
Turn the rolled sheet (four times)	19	
Make 3/4 cuts (five from each side)	21	
Turn the rolled sheet (four times)	23	
Sheet off	25	$1,5 \pm 0,5$

Final temperature of sheet: approx. 90 °C. To ensure homogenous sulfur distribution, the final temperature shall not be less than 90 °C. Increase the surface temperature of the rolls if necessary.

#### C.6.2 Mixing using a two-roll mill

To ensure good sulfur distribution, prepare a sulfur batch (masterbatch) at a roll surface temperature of 80  $^{\circ}$ C  $\pm$  5  $^{\circ}$ C, e.g. as specified in Table C.6.

Store the sulfur batch for at least 30 min, but no longer than 24 h.

Continue the preparation of the mix at a roll surface temperature of 50  $^{\circ}$ C  $\pm$  5  $^{\circ}$ C, e.g. as specified in Table C.7.

Table C.6 — Mixing procedure for preparation of a sulfur batch (example)

Roll diameter: 200 mm
Working width: 395 mm
Speed of rolls: 20/24 min<sup>-1</sup>

Surface temperature of rolls: 80 °C  $\pm$  5 °C

Rubber mass: 1 000 g

rabber made. 1 ddd g				
Mixing step	Elapsed time min	Nip opening mm		
Pass the rubber twice	<b>♦</b>	≤ 1		
Band rubber	1			
Add sulfur	2	$2.5 \pm 0.5$		
Make 3/4 cuts (three from each side)	4	2,3 ± 0,3		
Sheet off	7			

Final temperature of sheet: approx. 90 °C. To ensure homogenous sulfur distribution, the final temperature shall not be less than 90 °C. Increase the surface temperature of the oils if necessary.

Table C.7 — Mixing procedure for preparation of mix with sulfur batch (see Table C.6) (example)

Roll diameter: 200 mm

Working width: 395 mm

Speed of rolls: 20/24 min<sup>-1</sup>

Surface temperature of rolls: 50 °C ± 5 °C

Mixing step	Elapsed time	Nip opening mm
Pass sulfur batch between rolls	0	
Add antioxidant	1	
Make 3/4 cuts (two from each side)	2,5	$2,5\pm0,5$
Add zinc oxide	3	
Make 3/4 cuts (two from each side)	5	
Add carbon black and stearic acid	6	Set stepwise to $3.2 \pm 0.5$
Make 3/4 cuts (four from each side)	24	
Add TMDT and CBS	26	22.40.5
Make 3/4 cuts (six from each side)	28	3,2 ± 0,5
Turn the rolled sheet (four times)	30	
Sheet off	31	1,5 ± 0,5
Final temperature of sheet: approx. 90 °C	•	•

# Annex D

(normative)

# Acrylonitrile-butadiene rubbers: SRE-NBR/M

# **D.1 Purpose**

Representative of a medium-nitrile-content acrylonitrile-butadiene rubber.

# **D.2 Composition**

Table D.1 — Composition of the SRE

Ingredients	Parts by mass
Medium acrylonitrile-butadiene rubber (ACN content 33 %) <sup>a</sup>	100
Stearic acid	1,5
Zinc oxide	5,0
Carbon black, N550	55 (nominal)
Sulfur masterbatch <sup>b</sup>	1,9
Dibenzothiazyl disulfide masterbatch (MBTS) <sup>c</sup>	2,0
Total	165,4

<sup>&</sup>lt;sup>a</sup> Medium-nitrile-content, hot-polymerized Breon N33H80 from Zeon Chemicals Europe Ltd. has been found to be suitable.

# **D.3 Vulcanization**

Press-cure for 20 min  $\pm$  15 s at 165 °C  $\pm$  2 °C.

# D.4 Test sheet properties

SRE-NBR/M material shall fulfil the basic property given in Table D.2.

Table D.2 — Basic property

Property	Unit	Requirement	Test method
Increase in mass in test liquid 101, as in ISO 1817	%	19 to 22	ISO 1817, three test pieces immersed for (48 $_{-1}^{0}$ ) h at 100 °C $\pm$ 1 °C

b Masterbatch containing 80 % sulfur and 20 % of an ethylene-propylene rubber.

Masterbatch containing 75 % active accelerator and 25 % of an ethylene-propylene rubber.

# Annex E

(normative)

# Acrylonitrile-butadiene rubbers: SRE-NBR/L

# **E.1 Purpose**

Representative of a low-nitrile-content acrylonitrile-butadiene rubber.

# **E.2** Composition

Table E.1 — Composition of the SRE

Ingredients	Parts by mass
Low-acrylonitrile-butadiene rubber (ACN content 18 %) <sup>a</sup>	100
Stearic acid	1,5
Zinc oxide	5,0
Carbon black, N550	67 (nominal)
Sulfur masterbatch <sup>b</sup>	1,9
Dibenzothiazyl disulfide masterbatch (MBTS)	2,0
Total	177,4

a Low-nitrile-content Paracril 18.80 from Uniroyal Chemical has been found to be suitable.

# E.3 Vulcanization

Press-cure for 30 min  $\pm$  15 s at 155 °C  $\pm$  2 °C.

# E.4 Test sheet properties

SRE-NBR/L material shall fulfil the basic property given in Table E.2.

Table E.2 — Basic property

Property	Unit	Requirement	Test method
Increase in mass in test liquid B, as in ISO 1817	%		ISO 1817, three test pieces immersed for (48 $_{-1}^{0}$ ) h at 40 °C $\pm$ 1 °C

b Masterbatch containing 80 % sulfur and 20 % of an ethylene-propylene rubber.

Masterbatch containing 75% active accelerator and 25 % of an ethylene-propylene rubber.

# Annex F

(normative)

# Chlorobutyl rubbers: SRE-CIIR/1

# F.1 Purpose

Representative of a chlorobutyl rubber.

# F.2 Composition

Table F.1 — Composition of the SRE

Ingredients	Parts by mass
Chlorobutyl rubber <sup>a</sup>	√ 100
Stearic acid	1,5
Zinc oxide	5,0
Carbon black, N550 Carbon black, N990 <sup>b</sup> Sulfur masterbatch <sup>c</sup> Benzothiazyl disulfide masterbatch <sup>d</sup>	25 (nominal)
Carbon black, N990 <sup>b</sup>	120 (nominal)
Sulfur masterbatch <sup>c</sup>	0,94
Benzothiazyl disulfide masterbatchd	0,67
Petroleum jelly	3,0
Mineral oil (DEF STAN 91-44)	3,0
Dibenzothiazyl disulfide masterbatch (MBTS)d	1,33
Total	260,44

a Chlorobutyl 1066 from Exxon has been found to be suitable.

# F.3 Vulcanization

Press-cure for 25 min 15 s at 155 °C  $\pm$  2 °C.

# F.4 Test sheet properties

SRE-CIIR/1 material shall fulfil the basic property given in Table F.2.

Table F.2 — Basic property

Property	Unit	Requirement	Test method	
Increase in mass in test liquid B, as in ISO 1817	%		ISO 1817, three test pieces immersed for (48 $_{-1}^{0}$ ) h at 40 °C $\pm$ 1 °C	

b Thermax MT N990® from Degussa AG, or equivalent.

Masterbatch containing 80 % sulfur and 20 % of an ethylene-propylene rubber.

Masterbatch containing 75 % active accelerator and 25 % of an ethylene-propylene rubber.

# Annex G

(normative)

Chloroprene rubbers: SRE-CR/1

# **G.1 Purpose**

Representative of a general-purpose chloroprene rubber.

# **G.2 Composition**

Table G.1 — Composition of the SRE

	Ingredients	Parts by mass
Chloroprene rubbera	40	100
Stearic acid		0,5
Lightly calcined magnesia	X	4,0
Zinc oxide	Est.	5,0
Carbon black, N550	the	20 (nominal)
Organic acceleratorb	ient.	0,75 to 1,0
Antioxidant <sup>c</sup>	je	2,0
	Total	132,5

A slow-crystallizing form of a non-sulfur-modified polymer is required. Neoprene WRT from DuPont has been found to be suitable.

# G.3 Vulcanization

Press-cure for 25 min  $\pm$  15 s at 160 °C  $\pm$  2 °C.

# G.4 Test sheet properties

SRE-CR/1 material shall fulfil the basic property given in Table G.2.

Table G.2 — Basic property

Property	Unit	Requirement	Test method
Increase in mass in test liquid B, as in ISO 1817	%		ISO 1817, three test pieces immersed for (48 $_{-1}^{0}$ ) h at 40 °C $\pm$ 1 °C

b Vulkacit CRV from Bayer AG has been found to be suitable.

Octamine from Uniroyal Chemical has been found to be suitable.

# Annex H

(normative)

# Ethylene-propylene rubbers: SRE-EPM/1

# **H.1 Purpose**

Representative of a general-purpose ethylene-propylene rubber.

# **H.2 Composition**

Table H.1 — Composition of the SRE

Ingredients	Rarts by mass			
Ethylene-propylene copolymer rubber <sup>a</sup>	100			
Stearic acid	0,5			
Carbon black, N550	55 (nominal)			
Sulfur masterbatch <sup>b</sup>	0,44			
1,1-dibutylperoxy-3,3,5-trimethylcyclohexane (40 % active powder) <sup>c</sup>	7,5			
Total	163,44			
<sup>a</sup> Keltan 3300 A ethylene-propylene copolymer from DMS Elastomers has been found to be suitable.				
b Masterbatch containing 80 % sulfur and 20 % of an ethylene-propylene rubber.				

# **H.3 Vulcanization**

Press-cure for 10 min  $\pm$  15 s at 140 °C  $\pm$  2 °C

# H.4 Test sheet properties

SRE-EPM/1 material shall fulfil the basic property given in Table H.2.

Trigonox 29-40 BPD from Akzo has been found to be suitable.

Table H.2 — Basic property

Property	Unit	Requirement	Test method
Increase in mass in test liquid 103 (tri- <i>n</i> -butyl phosphate: density at 20 °C 0,975 Mg/m <sup>3</sup> to 0,976 Mg/m <sup>3</sup> ), as in ISO 1817	%	20 to 23	ISO 1817, three test pieces immersed for 22 h $\pm$ 0,25 h at 100 °C $\pm$ 1 °C

# Annex I

(normative)

# Fluoropolymer rubbers: SRE-FKM/1

# I.1 Purpose

Representative of a fluoropolymer rubber.

# **I.2 Composition**

Table I.1 — Composition of the SRE

Ingredients	Parts by mass
Fluoropolymer rubber <sup>a</sup>	100
Lightly calcined magnesia <sup>b</sup>	3,0
Carbon black, N990 <sup>c</sup>	30 (nominal)
2,5-di( <i>t</i> -butylperoxy)-2,5-dimethylhexane (50 % active powder)	3,0
Coagent <sup>d</sup>	3,0
Total	139,0
a Viton type GF from DuPont Dow Elastomers has been found to be suitable	

b Maglite D from Elastochem has been found to be suitable.

# I.3 Vulcanization

Press-cure for 8 min ± 15 s at 175 °C ± 2 °C.

Post-cure for  $(24\binom{2}{2})$  h at 230 °C ± 5 °C.

# I.4 Test sheet properties

SRE-FKM/1 material shall fulfil the basic property given in Table I.2.

Table I.2 — Basic property

Property	Unit	Requirement	Test method
Increase in mass in test liquid E (toluene), as in ISO 1817	%	8 to 11	ISO 1817, three test pieces immersed for 168 h $\pm$ 2 h at 40 °C $\pm$ 1 °C

<sup>&</sup>lt;sup>c</sup> Corax N990<sup>®</sup> from Degussa AG, or equivalent.

d Triallyl isocyanurate has been found to be suitable for use with Viton type GF.

# Annex J (normative)

# Fluoropolymer rubbers: SRE-FKM/2X

### J.1 Purpose

This SRE is representative of FKM materials such as are used, for instance, for parts in contact with petroleum products in the mechanical-engineering and automobile sectors.

It is used for the characterization of service fluids such as mineral oils, fuels, lubricants, hydraulic fluids, coolants and refrigerants with regard to their effect on vulcanized fluoropolymer rubbers.

The changes in mass, volume, hardness, tensile strength and elongation at break of the SRE when in contact with the service fluid under specified conditions may be included as supplementary data in specifications for the fluid concerned.

# J.2 Composition

Table J.1 — Composition of the SRE

Ingredients	Parts by mass
Vinylidene fluoride-hexafluoropropylene copolymer <sup>a</sup>	100,0
Magnesium oxide, high activity <sup>b</sup>	3,0
Calcium hydroxide, small particle size <sup>c</sup>	2,0
Carbon black, N990 <sup>d</sup>	25,0
Accelerator: organic phosphonium salt, e.g. triphenylbenzyl-phosphonium chloride <sup>e</sup>	0,44
Crosslinking agent: 2,2-bis(4-hydroxyphenyl)-hexafluoropropane (bisphenol AF) <sup>f</sup>	1,35
Total	131,79

The following brands have been found to be suitable:

- Tecnoflon N935<sup>®</sup> from Solvay, or Viton A 500<sup>®</sup> from Du Pont Dow Elastomers or Fluorel FC 2230<sup>®</sup> from Dyneon.
- b Elastomag 170<sup>®</sup> from Nordmann, Rassmann GmbH & Co.
- <sup>c</sup> Calcium hydroxide from Boehringer AG.
- d Thermax MT N990® from Degussa AG.
- e Intercure 2<sup>®</sup> from Interbusiness, Milan, or an equivalent accelerator such as Tecnoflon M2, Ausimont. Take 1,45 phr of this accelerator, and reduce the amount of rubber by 1 g per 100 g of rubber.
- f 2,2-Bis(4-hydroxyphenyl)-hexafluoropropane from Hoechst, or an equivalent crosslinking agent such as Tecnoflon M1, Ausimont or Viton Curative No. 30, DuPont Dow Elastomers. The bisphenol content of both agents is 50 %, so take 2,76 phr and reduce the amount of rubber by 1 g per 100 g of rubber.

#### J.3 Recommended mixing procedure

A two-roll mill or an internal mixer can be used (see Clause J.6).

#### J.4 Vulcanization

Condition the sheeted compound at ambient temperature for about 24 h. Prepare samples for cure. The direction of milling shall be marked on the slabs.

Press-cure at 180 °C  $\pm$  2 °C for 7 min  $\pm$  1 min.

Post-cure at 220 °C  $\pm$  5 °C for 16 h  $\pm$  2 h in an air-circulating oven with air exchange.

### J.5 Test sheet properties

SRE-FKM/2X material shall fulfil both the basic and the additional properties given in Tables 2 and J.3.

Table J.2 — Basic property

Property	Unit	Requirement	Test method
Increase in mass in test liquid C for fuels, as in ISO 1817	%		ISO 1817, three type 2 dumb-bell test pieces a immersed for (24 $_2^0$ ) h at 60 °C $\pm$ 2 °C Test piece/test liquid volume ratio: 1/(30 $\pm$ 5)
a As specified in ISO 37.			8

Table J.3 — Additional properties

Property	Unit	Requirement	Test method
Tensile strength	MPa	12 to 18	ISO 37, five type 2 dumb-bell test pieces
Elongation at break	%	250 to 330	
Hardness	IRHO	67 to 73	ISO 48, microtest, three test pieces
Density	Mg/m <sup>3</sup>	1,83 to 1,87	ISO 2781:1988, method A, three test pieces

# J.6 Mixing procedures

The following procedure can be used to produce SRE-FKM/2X material:

Roll diameter: 150 mm
Working width: 350 mm
Speed of rolls: 18/22 min-1

Surface temperature: 50 °C  $\pm$  5 °C

Mass of rubber: 1 000 g

Mixing step	Elapsed time min	Nip opening mm
Band rubber (first ingredient in Table J.1)	0	$2,0\pm0,5$
Blend the other ingredients (2 to 6) together and add evenly across the rolls at a constant rate	2	3,0 ± 1,0
Make 3/4 cuts (four from each side)		
Turn the rolled sheet (six times)	12	$0.5\pm0.2$
Sheet off	18	$2,2\pm0,5$
Final temperature of sheet: 60 °C to 70 °C		

Refine mixed stock after about 24 h storage on the mill under the same conditions, as follows:

Mixing step	Elapsed time min	Nip opening mm
Band batch	0	3,0 ± 1,0
Make 3/4 cuts (three from each side) Turn the rolled sheet (six times)	2	$0.5\pm0.2$
Sheet off	8	2,2 ± 0,5

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# Annex K

(normative)

# Hydrogenated acrylonitrile-butadiene rubbers: SRE-HNBR/1 and SRE-HNBR/1X

# **K.1 Purpose**

These SREs are representative of saturated HNBR materials such as are used, for instance, for parts in contact with petroleum products in the mechanical-engineering and automobile sectors.

They are used for the characterization of service fluids such as mineral oils, fuels dubricants, hydraulic fluids, coolants and refrigerants with regard to their effect on vulcanized fully hydrogenated nitrile rubbers.

The changes in mass, volume, hardness, tensile strength and elongation at break of the SRE when in contact with the service fluid under specified conditions may be included as supplementary data in specifications for the fluid concerned.

# **K.2 Composition**

Table K.1 — Composition of the SRE

Ingredients 1	Parts by mass		
HNBR (ACN content 39 %) <sup>a</sup>	100,0		
Zinc oxide, precipitated <sup>b</sup>	2,0		
Magnesium oxide <sup>c</sup>	2,0		
Diphenylamine, styrenated (SDPA) <sup>d</sup>	1,0		
Carbon black, N550	50,0		
Triallyl isocyanurate (TAIC) <sup>e</sup>	1,5		
1,3-bis( <i>t</i> -butylperoxyisopropyl)-benzene, 40 % by mass <sup>f</sup>	7,5		
Total	164,0		
a Therban A 3907 <sup>®</sup> from Bayer AG, or equivalent.			
Zinkoxyd aktiv <sup>®</sup> from Bayer AG, or equivalent.			
<sup>C</sup> Maglite DE <sup>®</sup> from Merck & Co, or equivalent.			
<sup>d</sup> Vulkanox DDA <sup>®</sup> from Bayer AG, or equivalent.			
<sup>e</sup> Perkalink 301 <sup>®</sup> from Akzo-Nobel BV, or equivalent.			
f Perkadox 14/40® from Akzo-Nobel BV, or equivalent.			

#### K.3 Recommended mixing procedure

Prepare a masterbatch without the peroxide in an internal mixer followed by homogenizing and addition of peroxide on a two-roll mill (see K.6.1).

Alternatively, sheet off the masterbatch on a two-roll mill, cut into strips, cool and re-introduce into the internal mixer and add peroxide (see K.6.2).