INTERNATIONAL STANDARD

ISO 5794-3

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Rubber compounding ingredients — Silica, precipitated, hydrated —

Part 3: Evaluation procedures in a blend of solution styrene-butadiene rubber (S-SBR) and butadiene rubber (BR)

Ingrédients de mélange du caoutchouc — Silices hydratées précipitées — Partie 3: Méthodes d'évaluation dans un mélange de solution de caoutchouc styrène-butadiène (S-SBR) et butadiène (BR)

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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 5794-3 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*.

ISO 5794 consists of the following parts, under the general title *Rubber compounding ingredients — Silica, precipitated, hydrated*:

— *Part 1: Non-rubber tests*

— *Part 2: Evaluation procedures in styrene-butadiene rubber*

— *Part 3: Evaluation procedures in a blend of solution styrene-butadiene rubber (S-SBR) and butadiene rubber (BR)*

Rubber compounding ingredients — Silica, precipitated, hydrated —

Part 3:
Evaluation procedures in a blend of solution styrene-butadiene rubber (S-SBR) and butadiene rubber (BR)

WARNING — Persons using this part of ISO 5794 should be familiar with normal laboratory practice. This International 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 part of ISO 5794 specifies the test formulation, equipment, procedure and test methods for determining the physical properties of precipitated hydrated silica in a compound based on a blend of solution styrene-butadiene and butadiene rubber. The formulation can be regarded as a model compound for silica-based passenger car tyre treads.

NOTE 1 ISO 5794-2[2] specifies the test formulation, equipment, procedure and test methods for determining the physical properties of precipitated hydrated silica in a styrene-butadiene rubber mix.

NOTE 2 ISO 5794-1[1] specifies methods for chemical analysis of precipitated hydrated silica, describes its physical and chemical properties, and classifies silicas with respect to their specific surface area obtained by nitrogen adsorption.

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 289-1, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

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

ISO 3417, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter*

ISO 6502, *Rubber — Guide to the use of curemeters*

3 Test formulation

The standard formulation is given in Table 1. The formulation shall be used for any type of silica precipitated for its evaluation.

International or national standard chemicals shall be used if available. Materials used shall be chemically equivalent to those indicated in Table 1.

For interlaboratory comparisons, the same type of S-SBR and BR shall be used.

4 Procedure

4.1 Equipment and procedure

4.1.1 General

Equipment and procedure for preparation, mixing and vulcanization shall be in accordance with ISO 2393. The following procedure is an example which has been found suitable for a 1,5 l internal mixer of the Banbury[1)] type.

4.1.2 First stage

— Filling factor: 0,74.

— Rotational speed: 70 r/min.

— Starting temperature: 80 °C.

— Friction: 1:1,11.

— Ram pressure: 0,55 MPa.

The first stage procedure is given in a) to j) herafter.

		Duration min
a)	Add polymers.	0,0 to 0,5
b)	Add half of the silica, carbon black/silane, ZnO, stearic acid, oil.	0,5 to 1,5
c)	Clean.	1,5
d)	Add half of the silica, antioxidant, wax.	1,5 to 3,5
e)	Clean.	3,5
f)	Mix and adjust rotational speed if required to reach discharge temperature.	3,5 to 5,0
g)	Discharge batch (batch temperature: 155 °C to 165 °C).	5,0
h)	Transfer to mill.	
i)	Band for 45 s using a clearance of 4 mm between the rolls.	
j)	Store compound for 24 h at room temperature before proceeding to second stage.	

1) Banbury is the trade name of a product supplied by Farrel Corp. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Table 1 — Standard test formulation

Material	Parts by mass
1st stage	
S-SBR, extended with TDAE[a]	96,25
BR[b]	30
Silica, precipitated	80
Carbon black N 330	6,4
Organosilane TESPT[c]	6,4
ZnO[d]	3
Stearic acid[e]	2
Oil TDAE[f]	11,25
Antioxidant (6PPD)[g]	1,5
Wax[h]	1
2nd stage (no additional ingredient)	
3rd stage	
DPG[i]	2
CBS[j]	1,5
TBzTD[k]	0,2
Sulfur[l]	2,1
Total	243,6

a Solution styrene-butadiene rubber, extended with treated distilled aromatic extract (TDAE) oil, vinyl content 50 % mass fraction, styrene content 25 % mass fraction. Oil content is 37,5 % mass fraction referring to 100 parts of rubber or 27,3 % mass fraction referring to the oil-extended material, respectively.

b Solution butadiene rubber, manufactured with neodymium catalyst, 96 % *cis* 1,4 content.

c For convenience, TESPT [*bis*(3-triethoxysilylpropyl)tetrasulfide] may be added in the form of a 50:50 admixture with carbon black N 330. If such an admixture is used, no additional carbon black is used in the compound.

d Zinc oxide, indirect type, class B1a according to ISO 9298:1995[5], Table D.1.

e Stearic acid, stearic/palmitic 65/30, class B according to ISO 8312[3].

f Treated distillate aromatic extract.

g *N*-(1,3-Dimethylbutyl)-*N′*-phenyl-*p*-phenylenediamine.

h Wax, mixture of refined hydrocarbons.

i *N*,*N′*-Diphenylguanidine.

j *N*-Cyclohexyl-2-benzothiazylsulfenamide.

k Tetrabenzylthiuram disulfide.

l Sulfur, soluble (rhombic), class W according to ISO 8332:2006[4], Table A.1.

4.1.3 Second stage

— Filling factor: 0,71.

— Rotational speed: 80 r/min.

— Starting temperature: 80 °C.

— Friction: 1:1,11.

— Ram pressure: 0,55 MPa.

The second stage procedure is given in a) to f) herafter.

		Duration min
a)	Plasticize batch from first stage.	0,0 to 2,0
b)	Maintain batch temperature of 160 °C by adjusting the rotational speed.	2,0 to 5,0
c)	Discharge batch (batch temperature: 155 °C to 165 °C).	5,0
d)	Transfer to mill.	
e)	Form a sheet for 45 s using a clearance of 4 mm between the rolls.	
f)	Store compound for 4 h at room temperature before proceeding to third stage.	

4.1.4 Third stage

— Filling factor: 0,68.

— Rotational speed: 40 r/min.

— Starting temperature: 50 °C.

— Friction: 1:1,11.

— Ram pressure: 0,55 MPa.

The third stage procedure is given in a) to h) herafter.

		Duration min
a)	Add batch of second stage.	0,0 to 0,5
b)	Add DPG, CBS, TBzTD and sulfur.	0,5 to 2,0
c)	Discharge batch (batch temperature: 90 °C to 110 °C).	2,0
d)	Transfer to mill.	
e)	Form a sheet for 20 s using a clearance of 3 mm to 4 mm between the rolls.	
f)	During the next 40 s, cut three times left, three times right, nip and pass the rolled batch twice endwise through the rolls using a mill opening of 3 mm.	
g)	From the freshly prepared batch, form one sheet of the desired thickness.	
h)	Store the sheet for at least 12 h at room temperature before cure.	

4.2 Testing of the uncured mix

Determine the viscosity at 100 °C using the shearing disc viscometer in accordance with ISO 289-1.

5 Evaluation of vulcanization characteristics

5.1 Evaluation according to oscillating disc curemeter test

Measure the following standard test parameters:

M_L, M_H, t_{s1}, $t'_c(50)$ and $t'_c(90)$

in accordance with ISO 3417, using the following test conditions:

— oscillation frequency: 1,7 Hz (100 cycles per minute);

— amplitude of oscillation: 3° arc; 1° arc may be used if required;

— selectivity: to be chosen to give at least 75 % full scale deflection at M_H;

— die temperature: 160 °C;

— pre-heat time: none.

5.2 Evaluation according to rotorless curemeter test

Measure the following standard test parameters:

F_L, F_{max} at a specified time, t_{s1}, $t_c(50)$ and $t_c(90)$

in accordance with ISO 6502, using the following test conditions:

— oscillation frequency: 1,7 Hz (100 cycles per minute);

— amplitude of oscillation: 0,5° arc;

— selectivity: to be chosen to give at least 75 % full scale deflection at F_{max};

— die temperature: 160 °C;

— pre-heat time: none.

5.3 Evaluation according to stress-strain properties

Vulcanize the test slabs at 160 °C for 15 min.

Determine the tensile stress-strain properties [stress at 300 % strain, stress at 500 % strain (if elongation at break exceeds 600 %), tensile strength and elongation at break] in accordance with ISO 37 using type 2 test pieces.

5.4 Hardness

Determine the hardness in accordance with ISO 48.

6 Precision

The test precision may vary depending on the type of silica and on the rubber properties determined.