
**Rubber compounding ingredients —
Silica, precipitated, hydrated —**

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

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*Ingédients de mélange du caoutchouc — Silices hydratées
précipitées —
Partie 2: Méthodes d'évaluation dans le caoutchouc styrène-butadiène*

ISO 5794-2:2014

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Contents

	Page
Foreword	iv
1 Scope	1
2 Normative references	1
3 Sampling	1
4 Test formulations	2
5 Procedure	2
5.1 Equipment and procedure	2
5.2 Mill mixing procedure	2
5.3 Testing of the uncured mix	3
6 Evaluation of vulcanization characteristics	3
6.1 Evaluation according to oscillating disc curemeter test	3
6.2 Evaluation according to rotorless curemeter test	4
6.3 Evaluation according to stress-strain properties	4
6.4 Hardness	4
7 Precision	4
8 Test report	4
Bibliography	5

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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).

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).

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 3, *Raw materials (including latex) for use in the rubber industry*.

This third edition cancels and replaces the second edition (ISO 5794-2:1998), which has been technically revised with the following changes:

- ISO 5794-3 is now mentioned in the scope;
- normative references have been updated;
- those standards which refer to the ingredients of the standard test formulations have been moved to the new section "Bibliography";
- footnote 1 in [Table 1](#) has been deleted because SBR 1500 EST is no longer available.

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 2: Evaluation procedures in styrene-butadiene rubber

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 styrene-butadiene rubber mix.

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

ISO 5794-3 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 rubber and butadiene rubber.

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 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*

ISO 15528, *Paints, varnishes and raw materials for paints and varnishes — Sampling*

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

3 Sampling

The sampling shall be carried out in accordance with ISO 15528.

4 Test formulations

Two standard formulations are given in [Table 1](#), differing in the amount of activator.

Silica with high specific surface area requires more activator than silica with lower specific surface area. International or national standard chemicals shall be used, if available. Materials used shall be chemically identical with those indicated in [Table 1](#).

Table 1 — Standard test formulations

Material	Formulation	
	1 Parts by mass	2 Parts by mass
SBR 1500	100	100
Silica (type A, B, C, D)	50	—
Silica (type E, F)	—	50
Zinc oxide ^a	5	5
Stearic acid ^b	1	1
PEG 4000 ^c	3	1,5
MBTS	1,2	1,2
MBT	0,7	0,7
DPG	0,5	0,5
Sulfur ^d	2	2
Total	163,4	161,9
^a ISO 9298:1995, Table D.1, class B1a. ^b ISO 8312:1999, Table L.1, class A, very low iodine value. ^c Poly(ethylene glycol) (relative molecular mass 4 000). ^d ISO 8332:2011, Table A.1, grade W.		

5 Procedure

5.1 Equipment and procedure

Equipment and procedure for preparation, mixing, and vulcanization shall be in accordance with ISO 2393.

5.2 Mill mixing procedure

The standard laboratory mill batch mass, in grams, shall be based on four times the test formulation mass. The surface temperature of the rolls shall commence at 25 °C ± 5 °C with proper cooling. The mass of the mixed batch shall not differ from the total mass of materials by more than +0,5 % or -1,5 %.

	Time min	Cumulative time min
5.2.1 Band the rubber with the mill opening set at 1 mm. Add zinc oxide and stearic acid. Make 3/4 cuts every 30 s from alternate sides.	3	3
5.2.2 Add 1/3 of the silica and make two 3/4 cuts from each side.	5	8
5.2.3 Add 1/3 of the silica and make two 3/4 cuts from each side.	5	13
5.2.4 Add 1/3 of the silica and the activator. Make two 3/4 cuts from each side	6	19
5.2.5 Add sulfur and accelerator slowly and evenly across the rubber. When all substances have been incorporated, make two 3/4 cuts from each side.	3	22
5.2.6 Cut the batch from the mill, set the mill opening to 0,8 mm to 1 mm nip and pass the rolled batch three times endwise through the rolls.	1,5	23,5
5.2.7 Cut the batch from the mill, set the mill opening to 3 mm to 3,5 mm nip and pass the rolled batch three times endwise through the rolls.	1,5	25
	Total time:	25
5.2.8 From the freshly prepared batch form one 6 mm sheet for samples for the determination of vulcanization characteristics and one 2,2 mm sheet for the preparation of tensile test pieces.		
5.2.9 Condition the batch for 18 h to 24 h before vulcanization, if possible, at standard temperature and humidity as defined in ISO 23529.		

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5.3 Testing of the uncured mix

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

6 Evaluation of vulcanization characteristics

6.1 Evaluation according to oscillating disc curemeter test

Measure the following standard test parameters in accordance with ISO 3417:

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

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.

6.2 Evaluation according to rotorless curemeter test

Measure the following standard test parameters in accordance with ISO 6502:

F_L , F_{max} at a specified time, t_{s1} , $t'_c(50)$ and $t'_c(90)$

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.

6.3 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.

6.4 Hardness

Determine the hardness in accordance with ISO 48.

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7 Precision

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

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8 Test report

The test report shall include the following information:

- a) a reference to this part of ISO 5794, i.e. ISO 5794:2014;
- b) all details necessary for the identification of the sample;
- c) standard test formulation used (1 or 2);
- d) viscosity of the uncured mix (see 5.3);
- e) cure characteristics (see 6.1 or 6.2);
- f) stress-strain properties (see 6.3);
- g) hardness (see 6.4);
- h) any operations not included in this part of ISO 5794 or in the International Standards to which reference is made, as well as any operation regarded as optional;
- i) date of the test.

Bibliography

- [1] ISO 5794-1:2010, *Rubber compounding ingredients — Silica, precipitated, hydrated — Part 1: Non-rubber tests*
- [2] ISO 8312:1999, *Rubber compounding ingredients — Stearic acid — Definition and test methods*
- [3] ISO 8332:2011, *Rubber compounding ingredients — Sulfur — Methods of test*
- [4] ISO 9298:1995, *Rubber compounding ingredients — Zinc oxide — Test methods*

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