INTERNATIONAL STANDARD

ISO 5794-2

Second edition
1998-11-01

Rubber compounding ingredients — Silica, precipitated, hydrated —

Part 2:
Evaluation procedures in styrene-butadiene rubber

Ingrédients de mélange du caoutchouc — Silices hydratées précipitées —

Partie 2: Méthodes d'évaluation dans le caoutchouc styrène-butadiène

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Foreword

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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.

International Standard ISO 5794-2 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*.

This second edition cancels and replaces the first edition (ISO 5794-2:1982), which has been technically revised.

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*

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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 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 silicas with respect to their specific surface area obtained by nitrogen adsorption.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 5794. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 5794 are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties.*

ISO 48:1994, *Rubber, vulcanized or thermoplastic — Determination of hardness (hardness between 10 IRHD and 100 IRHD).*

ISO 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity.*

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing.*

ISO 842:1984, *Raw materials for paints and varnishes — Sampling.*

ISO 2393:1994, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter.*

ISO 5794-1:1994, *Rubber compounding ingredients — Silica, precipitated, hydrated — Part 1: Non-rubber tests.*

ISO 6502:1991, *Rubber — Measurement of vulcanization characteristics with rotorless curemeters.*

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ISO 8312:—[1], *Rubber compounding ingredients — Stearic acid — Definition and test methods.*

ISO 8332:1997, Rubber compounding ingredients — Sulfur — Methods of test.

ISO 9298:1995, *Rubber compounding ingredients — Zinc oxide — Test methods.*

3 Sampling

The sampling shall be carried out in accordance with ISO 842.

4 Test formulations

Two standard formulations are given in table 1, differing in the amount of activator.

Silicas with high specific surface area require more activator than silicas 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	Formulation 2 Parts by mass
SBR 1500[1)]	100	100
Silica (type A, B, C, D)	50	—
Silica (type E, F)	—	50
Zinc oxide[2)]	5	5
Stearic acid[3)]	1	1
PEG 4000[4)]	3	1,5
MBTS	1,2	1,2
MBT	0,7	0,7
DPG	0,5	0,5
Sulfur[5)]	2	2
Total	163,4	161,9

1) SBR 1500 EST (Enichem Elastomers; Milan Italy) is an example of a suitable commercial product. This information is given for the convenience of users of this part of ISO 5794 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.

2) ISO 9298:1995, table D.1, class B1a.

3) ISO 8312:—, table L.1, class A, very low iodine value.

4) Poly(ethylene glycol) (relative molecular mass 4000).

5) ISO 8332:1997, table A.1, grade W.

1) To be published. (Revision of ISO 8312-1:1988)

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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 3 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 3 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 471.

5.3 Testing of the uncured mix

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

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6 Evaluation of vulcanization characteristics

6.1 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

6.2 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

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.

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;

b) all details necessary for the identification of the sample;

c) the standard test formulation used (1 or 2);

d) the viscosity of the uncured mix (see 5.3);

e) the cure characteristics (see 6.1 or 6.2);

f) the stress-strain properties (see 6.3);

g) the 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) the date of the test.

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