BSI

BS 6449 : Part 2 : 1984
ISO 5794/2-1982

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British Standard

Rubber compounding ingredients : precipitated, hydrated silica

Part 2. Method for determination of physical properties in rubber (including test recipe)

[ISO title: Rubber compounding ingredients — Silica, precipitated, hydrated — Part 2 : Test recipe and determination of physical properties in rubber]

Ingrédients de mélange du caoutchouc : silices hydratés précipités
Partie 2. Méthode de détermination des propriétés physiques dans le caoutchouc (y inclus la formule d'essai)

Zusätze zu Gummimischungen : Siliziumdioxyd-Präzipitat, hydriert
Teil 2. Verfahren zur Bestimmung der physikalischen Eigenschaften in Gummi (einschließlich Prüfrezept)

National foreword

This Part of this British Standard has been prepared under the direction of the Rubber Standards Committee and is identical with ISO 5794/2 'Rubber compounding ingredients — Silica, precipitated, hydrated — Part 2 : Test recipe and determination of physical properties in rubber', published in 1982 by the International Organization for Standardization (ISO).

Attention of users of this Part of this British Standard is drawn to note 2) under the table in clause **3.** It is there acknowledged that ingredients may be in accordance with equivalent national standards if the NBS (National Bureau of Standards of the USA) standard reference material is not used. In the UK the equivalent national standard is BS 4398 'Compounding ingredients for rubber test mixes'.

It is anticipated that Parts 1 and 3 of this British Standard will be published at a later date and will correspond to ISO 5794/1 and ISO 5794/3 respectively, when these international standards are published (see clauses **1** and **2**).

Terminology and conventions. The text of the international standard has been approved as suitable for publication as a British Standard without deviation. Some terminology and certain conventions are not identical with those used in British Standards; attention is drawn especially to the following.

The comma has been used as a decimal marker. In British Standards it is current practice to use a full point on the baseline as the decimal marker.

Where the words 'part of ISO 5794' appear, referring to this standard, they should be read as 'Part of BS 6449'.

Cross-references

International standard	Corresponding British Standard
	BS 903 Methods of testing vulcanized rubber
ISO 34-1979	Part A3 : 1982 Determination of tear strength (trouser, angle and crescent test pieces) (Identical)
ISO 37-1977	Part A2 : 1971 Determination of tensile stress-strain properties (Technically equivalent)
ISO 48-1979	Part A26 : 1969 Determination of hardness (Technically equivalent)
ISO 2393-1973	BS 1674 : 1976 Specification for equipment and general procedure for mixing and vulcanizing rubber test mixes (Technically equivalent)

British Standards Institution

BS 6449 : Part 2 : 1984
ISO 5794/2-1982

This British Standard, having been prepared under the direction of the Rubber Standards Committee, was published under the authority of the Board of BSI and comes into effect on **31 January 1984.**

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The following BSI references relate to the work on this standard:
Committee reference RUM/25 Draft for comment 81/50832 DC

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Rubber Standards Committee (RUM/-) to Technical Committee RUM/25 upon which the following bodies were represented:

British Aggregate Construction Materials Industries
British Rubber Manufacturers' Association
China Clay Association
Malaysian Rubber Producers' Association
Ministry of Defence
Zinc Pigment Development Association

Amendments issued since publication

Amd. No.	Date of issue	Text affected

International standard	Corresponding British Standard
ISO 3417-1977	BS 1673 Methods for testing raw rubber and unvulcanized compounded rubber Part 10 : 1977 Measurement of prevulcanizing and curing characteristics by means of curemeters (Technically equivalent)

The Technical Committee has reviewed the provisions of ISO 3257, to which reference is made in the text and for which there is no corresponding British Standard, and has decided that they are acceptable for use in conjunction with this standard. ISO 289, referred to in clause **2** and **4.3**, is currently at the stage of draft. The British Standard technically equivalent to ISO/R 289 is BS 1673 'Methods of testing raw rubber and unvulcanized compounded rubber' Part 3 : 1969 'Methods of physical testing'.

1 Scope and field of application

This part of ISO 5794 specifies the test recipe, 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, and ISO 5794/3 specifies its physical and chemical properties and properties in the rubber mix.

2 References

ISO 34, *Rubber, vulcanized — Determination of tear strength (trouser, angle and crescent test pieces).*

ISO 37, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

ISO 48, *Vulcanized rubbers — Determination of hardness (Hardness between* 30 *and* 85 IRHD).

ISO 289, *Rubber, unvulcanized — Determination of Mooney viscosity.*[1)]

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

ISO 3257, *Rubber compounding ingredients — Carbon black — Test recipe and method of evaluation in styrene-butadiene rubbers.*

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

ISO 5794, *Rubber compounding ingredients — Silica, precipitated, hydrated —*

Part 1 : Non-rubber tests.[2)]
Part 3 : Specification.[2)]

1) At present at the stage of draft. (Revision of ISO/R 289-1963.)

2) At present at the stage of draft.

3 Test recipe

The standard test recipe is given in the following table.

Material	Reference material number	Parts by mass
SBR 1 500	EST[1)]	100,0
Silica		40,0
Zinc oxide	NBS 370d[2)]	3,0
Stearic acid	NBS 372g[2)]	1,5
TMTD[3)]	NBS 374c[2)]	2,0
TBBS[4)]	NBS 384[2)]	2,0
Sulphur	NBS 371f[2)]	0,4
Total		148,9

1) See ISO 3257. A European equivalent to NBS standard reference material 386 has been developed by ANIC. This EST (European Standard Type) rubber is an SBR 1 500 type using a rosin acid emulsifier and a staining stabilizer.

2) NBS standard reference material number (National Bureau of Standards of the USA). Alternatively the ingredients shall be in accordance with equivalent national standards.

3) Tetramethylthiuramdisulphide.

4) *N-tert*-butyl-2-benzothiazole sulphenamide.

4 Procedure

4.1 Equipment and procedure

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

4.2 Mill mixing procedure

The standard laboratory mill batch mass, in grams, shall be based on four times the test recipe mass. The surface temperature of the rolls shall commence at 30 ± 5 °C with proper cooling. The mass of the mixed batch shall not differ from the total mass of materials by more than 1,0 %.

	Duration (min)
4.2.1 Band the rubber with the mill opening set at 1,1 mm and make 3/4 cuts every 30 s from alternate sides	2
4.2.2 Add the sulphur slowly and evenly across the rubber. When the sulphur has been incorporated, make one 3/4 cut from each side.	2
4.2.3 Add the zinc oxide and approximately 10 % of the silica. No cuts shall be made at this stage.	4
4.2.4 Add the stearic acid and a further 10 % of the silica, again without cutting the batch	4
4.2.5 Add the rest of the silica slowly. Adjust the mill opening so that the rolling bank has a diameter of approximately 15 mm. Do not cut during incorporation of the silica. Add the material from the pan and when all the silica is incorporated, cut once from each side	10
4.2.6 Add the accelerator and make three 3/4 cuts from each side	4
4.2.7 Cut the batch from the mill, set the mill opening to 0,8 mm and pass the rolled batch endwise through the rolls three times	2
4.2.8 Allow the compound to run for 5 min on the mill with a suitable mill opening so that the rolling bank has a diameter of approximately 15 mm	5
4.2.9 Sheet the batch to approximately 5 mm and check the mass of the batch.	
Total time	33

4.2.10 Condition the batch for 18 to 24 h.

4.2.11 Remilling shall be performed in accordance with the following procedure.

With the surface temperature of the rolls maintained at 30 ± 5 °C, set the mill opening to 0,2 mm and pass the batch once (without banding) through the rolls.

Set the mill opening to approximately 3 mm. Band the mix and allow it to work with a good rolling bank for 5 min without cutting.

Open the mill to give a minimum mix thickness of 6 mm and pass the mix through the mill four times, folding it back on itself each time.

Take samples for the determination of vulcanization characteristics.

Sheet the mix from the mill at such a setting as to obtain a finished thickness of approximately 2,2 mm for the preparation of the dumb-bell specimens (or another appropriate thickness for the preparation of the ring specimens).

Allow to stand for 2 h before vulcanizing.

4.3 Testing of the uncured mix

Determine the viscosity using the shearing disk viscometer in accordance with ISO 289.

5 Evaluation of vulcanization characteristics

5.1 Evaluation according to stress-strain properties

Vulcanize the test slabs at 145 °C or alternatively at 150 °C to optimum cure. Condition the vulcanized test slabs for 16 to 72 h.

Determine the tensile stress-strain properties (stress-strain at 500 %, tensile strength and elongation at break) in accordance with ISO 37.

Determine the hardness in accordance with ISO 48, and the tear strength in accordance with ISO 34.

5.2 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 :	1° arc
selectivity :	to be chosen to give at least 75 % full scale deflection at M_H
die temperature :	160 °C
pre-heat time	none

NOTE — Alternatively, macro-dies may be used in which case a pre-heat of 1 min is necessary.

6 Precision

To be added later.

Publications referred to

See national foreword.