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Vulcanized crumb rubber — Evaluation procedures

Poudrettes de caoutchouc vulcanisées — Méthodes d'évaluation

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Foreword

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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).

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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

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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*.

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Vulcanized crumb rubber — Evaluation procedures

1 Scope

This Technical Specification specifies physical and chemical tests, standard test formulations, equipment and processing methods for the vulcanization-characteristics evaluation and the mechanical properties of vulcanized crumb rubber.

It is not aimed to provide specifications or limitations, or whether these vulcanized crumb rubbers may be used.

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 34-1:2010, *Rubber, vulcanized or thermoplastic — Determination of tear strength — Part 1: Trouser, angle and crescent test pieces*

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

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ISO 247:2006, *Rubber — Determination of ash*

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

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ISO 1407:2011, *Rubber — Determination of solvent extract*

ISO 1408, *Rubber — Determination of carbon black content — Pyrolytic and chemical degradation methods*

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

CEN/TS 14243:2010, *Materials produced from end of life tyres — Specification of categories based on their dimension(s) and impurities and methods for determining their dimension(s) and impurities*

3 Sampling and sample preparation

Take a laboratory sample of approximately 1,5 kg.

4 Physical and chemical tests on raw vulcanized crumb rubber

4.1 Classification

Classify raw vulcanized crumb rubber, based mainly on their dimensions, in accordance with CEN/TS 14243:2010, Clause 4.

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4.2 Determination of particle size distribution

Determine the particle size distribution in accordance with CEN/TS 14243:2010, Clauses 5 and 6.

4.3 Determination of free steel content

Determine the free steel content in accordance with CEN/TS 14243:2010, Annex B.

4.4 Determination of free textile content

Determine the free textile content in accordance with CEN/TS 14243:2010, Annex C.

4.5 Acetone extract

Determine the acetone extract in accordance with ISO 1407:2011, methods A or B.

4.6 Ash

Determine the ash in accordance with ISO 247:2006, method A.

4.7 Carbon black

Determine the carbon black content in accordance with ISO 1408.

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4.8 Rubber content

Determine the rubber content, R, in percent (%), with the following equation:

$$R = 100 - (a + b + c)$$

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where

a is the acetone extract, in percent (%);

b is the ash content, in percent (%);

c is the carbon black content, in percent (%).

5 Preparation of test mixes for evaluation

5.1 Standard test formulation

The standard test formulation is given in Table 1.

The materials used shall be national or international standard reference materials. If no standard reference material is available, the materials to be used shall be agreed between the interested parties.

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Table 1 — Standard test formulation

Material	Parts by mass
Natural rubber STR 20 [a]	100,00
Vulcanized crumb rubber	30,00 [e]
Industry reference black [b]	5,00
Stearic acid [c]	2,00
Zinc oxide [c]	3,00
Sulfur [c]	2,25
TBBS [d]	1,00
Total	**143,25**

[a] Standard Thai Rubber 20 was used in the formula standard test for the drafting of this Technical Specification. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

[b] The current industry reference black shall be used.

[c] Powder materials shall be used (standard curing ingredients used in the industry).

[d] N-tert-butyl-benzothiazole-2-sulfenamide. This is supplied in powder form having an initial insoluble-matter content, determined in accordance with ISO 11235, of less than 0,3 %. The material shall be stored at room temperature in a closed container and the insoluble matter checked every 6 months. If this is found to exceed 0,75 % the TBBS shall be discarded or recrystallized.

[e] 30 parts by mass of vulcanized crumb rubber is the suitable ratio to show the result of crumb rubber, while the short duration for mixing procedure is acquired. And it allows stabilizing the particle size distribution of the crumb.

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5.2 Procedure

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5.2.1 Equipment and procedure

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

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5.2.2 Mixing procedure - Two stage mixing using an internal mixer for initial mixing and a mill for final mixing

5.2.2.1 Stage 1 - Initial mixing procedure

	Duration min	Cumulative time min
a) Adjust the temperature of the internal mixer to a starting temperature of 50 °C ± 3 °C. Close the discharge door, start the rotor and raise the ram.	0	0
b) Load the rubber, lower the ram and allow the rubber to be masticated.	1,0	1,0
c) Raise the ram and add the zinc oxide and the stearic acid. Lower the ram and allow the batch to be masticated.	0,5	1,5
d) Raise the ram and add the carbon black and the crumb rubber. Lower the ram and allow the batch to be mixed.	0,75	2,25
e) Raise the ram, sweep the mouth of the mixing chamber and lower the ram. Allow the batch to be mixed.	0,5	2,75
Total time (max.)	**2,75**	

f) Turn off the rotor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature.

g) Immediately pass the batch four times through a laboratory mill with its mill opening set at 2,5 mm and at a temperature of 50 °C ± 5 °C. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than + 0,5 % or - 1,5 %, discard the batch and re-mix.

h) Leave the batch for 30 min to 24 h after mixing.

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NOTE Only one mixing procedure is used during initial mixing to get similar result in particle size distribution in various mixes.

5.2.2.2 Stage 2 - Final mill mixing procedure

The standard laboratory-mill batch mass, in grams, shall be based on the recorded batch mass.

Set the mill temperature at 50 °C ± 5 °C and the mill opening to 1,5 mm.

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A good rolling bank at the nip of the rolls shall be maintained during mixing. If this is not obtained with the nip settings specified hereunder, small adjustments to the mill openings may be necessary.

		Duration min	Cumulative time min
a)	Band the master-batch on the slow roll.	1,0	1,0
b)	Add the TBBS. Do no cut the band until the accelerator is completely dispersed.	0,5	1,5
c)	Make a 3/4 cut from each side, allowing 15 s between each cut.	0,5	2,0
d)	Add the sulfur. Do not cut the band until the powder is completely dispersed.	0,5	2,5
e)	Make a 3/4 cut from each side, allowing 15 s between each cut.	0,5	3,0
f)	Make three alternating 3/4 cuts from each side, allowing 15 s between each cut.	2,0	5,0
g)	Cut the batch from the mill. Set the mill opening at 0,8 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternately.	2,0	7,0
Total time (max.)		**7,0**	

h) Sheet the batch to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than + 0,5 % or - 1,5 %, discard the batch and remix. Remove sufficient material for curemeter testing.

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i) Sheet the batch to approximately 2,2 mm in order to prepare test sheets or to the appropriate thickness in order to prepare ISO rings or dumbbell test pieces in accordance with ISO 37.

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j) After mixing and prior to vulcanization, condition the batch for at least 2 h at room temperature, but not more than 24 h.

6 Evaluation of vulcanization characteristics by a curemeter test

6.1 Using an oscillating-disc curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, t_{s1}, $t'_c(50)$, $t'_c(90)$, $t'_c(95)$ and $t'_c(99)$

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

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

— amplitude of oscillation: 1° of arc;

An amplitude of oscillation of 3° of arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1};

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

— die temperature: 150 °C ± 0,3 °C;

— pre-heat time: none.

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