INTERNATIONAL STANDARD

ISO 4097

Fifth edition
2007-07-15

Rubber, ethylene-propylene-diene (EPDM) — Evaluation procedure

Caoutchouc éthylène-propylène-diène (EPDM) — Méthode d'évaluation

Reference number
ISO 4097:2007(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

COPYRIGHT PROTECTED DOCUMENT

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

Published in Switzerland

Contents

Page

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.

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 4097 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 fifth edition cancels and replaces the fourth edition (ISO 4097:2000), which has been technically revised.

Rubber, ethylene-propylene-diene (EPDM) — Evaluation procedure

WARNING — Persons using this International Standard 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 International Standard specifies

— physical and chemical tests on raw rubbers;

— standard materials, standard test formulations, equipment and processing methods for evaluating the vulcanization characteristics of ethene-propene-diene (EPDM) rubbers, commonly termed ethylene-propylene-diene rubbers, including oil-extended types.

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 247:2006, *Rubber — Determination of ash*

ISO 248, *Rubber, raw — Determination of volatile-matter content*

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

ISO 1795, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*

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

3 Sampling and sample preparation

3.1 Take a laboratory sample of approximately 1,5 kg by the method described in ISO 1795.

3.2 Prepare the test portion in accordance with ISO 1795.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1, on a test portion prepared as indicated in 3.2 (without massing).

If massing is necessary, maintain the mill roll surface temperature at 50 °C ± 5 °C (for rubbers with a low Mooney viscosity, a temperature of 35 °C ± 5 °C can be used). Massing, if used, shall be mentioned in the test report.

Record the result as ML(1+4) at 125 °C unless another test temperature (100 °C or 150 °C) and/or test time (1+8) min has been agreed by the interested parties.

4.2 Volatile matter

Determine the volatile-matter content in accordance with ISO 248.

4.3 Ash

Determine the ash in accordance with method A or method B of ISO 247:2006.

5 Preparation of test mixes for evaluation

5.1 Standard test formulations

The standard test formulations are given in Table 1, in which:

— formulation 1 is applicable to non-oil-extended EPDMs with a nominal ethylene content not higher than 67 % by mass;

— formulation 2 is applicable to non-oil-extended EPDMs with a nominal ethylene content equal to or higher than 67 % by mass;

— formulation 3 is applicable to non-oil-extended low Mooney viscosity EPDMs;

— formulation 4 is applicable to oil-extended EPDMs containing 50 or less parts by mass of oil per 100 parts of rubber;

— formulation 5 is applicable to oil-extended EPDMs containing more than 50 but less than 80 parts by mass of oil per 100 parts of rubber;

— formulation 6 is applicable to oil-extended EPDMs containing 80 or more parts by mass of oil per 100 parts of rubber.

The materials used shall be national or international standard reference materials, unless no standard reference materials are available, in which case the materials to be used shall be agreed between the interested parties.

Table 1 — Standard test formulations for evaluation of EPDM rubbers

Material	Test formulation					
	1	2	3	4	5	6
	Parts by mass					
EPDM	100,00	100,00	100,00	100,00 + x [a]	100,00 + y [b]	100,00 + z [c]
Stearic acid	1,00	1,00	1,00	1,00	1,00	1,00
Industry reference black [d]	80,00	100,00	40,00	80,00	80,00	150,00
ASTM 103 oil [e]	50,00	75,00	—	50,00 − x [a]	—	—
Zinc oxide	5,00	5,00	5,00	5,00	5,00	5,00
Sulfur	1,50	1,50	1,50	1,50	1,50	1,50
Tetramethyl thiuram disuffide (TMTD) [f]	1,00	1,00	1,00	1,00	1,00	1,00
Mercaptobenzothiazole (MBT)	0,50	0,50	0,50	0,50	0,50	0,50
Total	239,00	284,00	149,00	239,00	189,00 + y [b]	259,00 + z [c]

[a] x is the number of parts by mass of oil per 100 parts of base rubber for types having an oil content of 50 or less.

[b] y is the number of parts by mass of oil per 100 parts of base rubber for types having an oil content more than 50 but less than 80.

[c] z is the number of parts by mass of oil per 100 parts of base rubber for types having a minimum oil content of 80.

[d] The current industry reference black (IRB) shall be used.

[e] This oil, density 0,92 g/cm^3, is produced by the Sun Refining and Marketing Company and distributed by R.E. Carroll Inc., 1570 North Olden Avenue Ext, Trenton, NJ 08638, USA. Overseas requests should be directed to Sunoco Overseas Inc., 1801 Market Street, Philadelphia, PA 19103-1699, USA. Alternative oils can be used but may give slightly different results.

[f] A standard reference material for TMTD is available as IRM 1 from Forcoven Products Inc., 22010 E. Martin Dr., Porter, TX 77365, USA.

5.2 Procedure

5.2.1 Equipment and procedure

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

5.2.2 Mixing procedures

5.2.2.1 General

Three alternative mixing procedures are specified:

— method A: internal mixing;

— method B: mill mixing;

— method C: use of internal mixer for initial and mill for final mixing.

NOTE Mixing of ethylene-propylene-diene rubbers in the standard test formulations using a mill is more difficult than for other rubbers and the use of an internal mixer allows better results to be obtained. Because of the difficulty of mill mixing EPDM rubbers, it is recommended that method B be used only if an internal mixer is not available.

5.2.2.2 Method A — Internal mixing

5.2.2.2.1 Initial mixing procedure

		Duration (min)	Cumulative time (min)
a)	Adjust the temperature of the internal mixer to achieve a final mix temperature of 150° C in about 5 min. Close the discharge door, set the rotor at 8 rad/s (77 r/min), start the rotor and raise the ram.	0	0
b)	Load the rubber, the zinc oxide, the carbon black, the oil and the stearic acid. Lower the ram.	0,5	0,5
c)	Allow the batch to mix.	2,5	3,0
d)	Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0,5	3,5
e)	Discharge the batch when the temperature reaches 150 °C or after 5 min, whichever occurs first.	max. 1,5	5,0
	Total time (max.)	5,0	

f) Immediately pass the batch three 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 $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

g) Leave the batch for 30 min to 24 h after mixing, if possible at standard temperature and humidity as defined in ISO 23529.

5.2.2.2.2 Final mixing procedure

		Duration (min)	Cumulative time (min)
a)	Adjust the chamber and rotors to 40 °C ± 5 °C. Close the discharge gate, start the rotor at 8 rad/s (77 r/min) and raise the ram.	0	0
b)	Charge one-half of the batch prepared in 5.2.2.2.1, the accelerators and the sulfur, and then the rest of the batch. Lower the ram.	0,5	0,5
c)	Allow the batch to mix until a temperature of 110 °C or a total mixing time of 2 min is reached, whichever occurs first. Discharge the batch.	max. 1,5	2,0
	Total time (max.)	2,0	

d) Immediately pass the batch through a laboratory mill with its mill opening set at 0,8 mm and at a temperature of 50 °C ± 5 °C.

e) Pass the roiled batch endwise through the rolls six times.

f) Sheet the batch to approximately 6 mm. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

g) Remove sufficient material for curemeter testing.

h) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens in accordance with ISO 37.

i) Leave the batch for 30 min to 24 h after mixing, if possible at standard temperature and humidity as defined in ISO 23529.

5.2.2.3 Method B — Mill mixing

The standard laboratory mill batch mass, in grams, shall be based on twice the formulation mass. The surface temperature of the rolls shall be maintained at 50 °C ± 5 °C throughout the mixing. Mix the zinc oxide, stearic acid, oil and carbon black together in a suitable container before starting to mix (however, see the Note below).

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 below, small adjustments to the mill openings may be necessary.

		Duration (min)	Cumulative time (min)
a)	Band the rubber on the fast roll with the mill set at 50 °C ± 5 °C and a 0,7 mm opening.	1,0	1,0
b)	Add the mixture of oil, carbon black, zinc oxide and stearic acid evenly across the mill with a spatula. NOTE In formulations 2, 4 and 5, some of the oil may be withheld for addition c).	13,0 [steps b) + c)]	14,0
c)	When about half of the mixture is incorporated, open the mill to 1,3 mm and make one 3/4 cut from each side. Then add the remainder of the mixture, opening the mill to 1,8 mm. When all the mixture has been incorporated, make two 3/4 cuts from each side. Be sure to add any material that has fallen into the mill-pan.		
d)	Add the accelerators and sulfur evenly across the rolls, still at an opening of 1,8 mm.	3,0	17,0
e)	Make three 3/4 cuts from each side, allowing 15 s between each cut.	2,0	19.0
f)	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	21,0
	Total time	21,0	

g) Sheet the batch to approximately 6 mm. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

h) Remove sufficient material for curemeter testing.

i) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

j) Leave the batch for 2 h to 24 h after mixing, if possible at standard temperature and humidity as defined in ISO 23529.

5.2.2.4 Method C — Internal mixer for initial and mill for final mixing

5.2.2.4.1 Stage 1 — Initial mixing procedure

		Duration (min)	Cumulative time (min)
a)	Adjust the temperature of the internal mixer to achieve a final mix temperature of 150 °C in about 5 min. Close the discharge door, set the rotor at 8 rad/s (77 r/min), start the rotor and raise the ram.	0	0
b)	Load the rubber, the zinc oxide, the carbon black, the oil and the stearic acid. Lower the ram.	0,5	0,5
c)	Allow the batch to mix.	2,5	3,0
d)	Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0,5	3,5
e)	Discharge the batch when the temperature reaches 150 °C or after 5 min, whichever occurs first.	max. 1,5	5,0
	Total time (max.)	5,0	

f) Immediately pass the batch three 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 $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

g) Leave the batch for 30 min to 24 h after mixing, if possible at standard temperature and humidity as defined in ISO 23529.

5.2.2.4.2 Stage 2 — Final mill mixing procedure

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.

The standard laboratory mill batch mass, in grams, shall be based on twice the formulation mass.

		Duration (min)	Cumulative time (min)
a)	Set the mill temperature at 50 °C ± 5 °C and the mill opening to 1,5 mm. Band the masterbatch on the fast roll and add the sulfur and accelerators. Do not cut the band until the sulfur and accelerators are completely dispersed. Be sure to add any material that has fallen into the mill-pan.	1,0	1,0
b)	Make three 3/4 cuts from each side, allowing 15 s between each cut.	2,0	3,0
c)	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	5,0
	Total time	5,0	

d) Sheet the batch to approximately 6 mm. Check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than $^{+0,5}_{-1,5}$ %, discard the batch and re-mix.

e) Remove sufficient material for curemeter testing.

f) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring specimens.

g) Leave the batch for 2 h to 24 h after mixing, if possible at standard temperature and humidity as defined in ISO 23529.

6 Evaluation of vulcanization characteristics by a curemeter test

WARNING — Formation of nitrosamines is possible during the cure.

6.1 Using an oscillating-disc curemeter

Measure the following standard test parameters:

M_L, M_H at defined time, t_{s1}, $t'_c(50)$ 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 % of full-scale deflection

NOTE With some rubbers, 75 % may not be attainable.

— die temperature: 160 °C ± 0,3 °C

— pre-heat time: none

6.2 Using a rotorless curemeter

Measure the following standard test parameters:

F_L, F_{max} at defined 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 % of full scale deflection at F_{max}

NOTE With some rubbers, 75 % may not be attainable.

— die temperature: 160 °C ± 0,3 °C

— pre-heat time: none

7 Evaluation of tensile stress-strain properties of vulcanized test mixes

WARNING — Formation of nitrosamines is possible during the cure.

Vulcanize sheets at 160 °C for three periods chosen from a cure series of 10 min, 20 min, 30 min, 40 min and 50 min. The middle vulcanization time shall be chosen from the above list to be nearest to $t'_c(90)$. The three periods of cure shall be chosen to cover undercure, optimum cure and overcure of the material under test.

Condition the vulcanized sheets for 16 h to 96 h at a standard temperature, and if possible a standard humidity, defined in ISO 23529.

Measure the stress-strain properties in accordance with ISO 37.

8 Precision

8.1 General

The precision results for the internal-mixer procedure are based on data from ASTM D 3568-03 [6]. The repeatability and reproducibility were calculated in accordance with ISO/TR 9272:1986 [3].

8.2 Precision details

8.2.1 Internal mixing

A type 2 (interlaboratory) precision was determined. Three different materials (EPDM rubbers) were used in the interlaboratory programme. These were tested in eight laboratories on two different days following the internal-mixing procedure (method A) and using international reference black No. 6 (IRB No. 6). Only curemeter (oscillating-disc) tests were performed.

8.2.2 Mill mixing

A type 2 (interlaboratory) precision was determined for cure characteristics measured using an oscillating-disc curemeter. One material (EPDM rubber) was used in the interlaboratory programme. This was tested in eight laboratories on three different days following the mill mixing procedure (method B).

8.3 Precision results

The calculated repeatability and reproducibility values are given in Tables 2 and 3.

The symbols used in Tables 2 and 3 are defined as follows:

r repeatability, in measurement units. This is the value below which the absolute difference between two "within-laboratory" test results may be expected to lie, with a specified probability.

(r) repeatability, in per cent (relative).

In the case of repeatability, the two test results are obtained with the same method on nominally identical test materials under the same conditions (same operator, apparatus and laboratory) and within a specified time period. Unless stated otherwise, the probability is 95 %.

R reproducibility, in measurement units. This is the value below which the absolute difference between two "between-laboratory" test results may be expected to lie, with a specified probability.

(R) reproducibility, in percent (relative).

In the case of reproducibility, the two test results are obtained with the same method on nominally identical test materials under different conditions (different operators, apparatus and laboratories) and within a specified time period. Unless stated otherwise, the probability is 95 %.

Table 2 — Internal-mixing procedure type 2 precision for various test parameters

Property	Units	Range of values [a]	Within laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
M_L	dN·m	6,7 to 12,4	0,50	1,42	14,8	1,24	3,51	36,6
M_H	dN·m	32,7 to 46,9	1,29	3,65	9,2	3,66	10,4	26,1
t_{S1}	min	2,2 to 2,7	0,11	0,31	12,4	0,38	1,08	43,2
$t'_C(90)$	min	12,6 to 15,6	0,64	1,81	12,8	1,20	3,40	24,1
NOTE s_r = Repeatability standard deviation, in measurement units. s_R = Reproducibility standard deviation, in measurement units.								
[a] Measured at 160 °C, 1,7 Hz, 1° amplitude arc. The midpoint of the range was used for calculations of (r) and (R).								

Table 3 — Mill mixing procedure type 2 precision for various test parameters

Property	Units	Mean level [a]	Within laboratory			Between laboratories		
			s_r	r	(r)	s_R	R	(R)
M_L	dN·m	7,00	0,54	1,51	21,57	1,49	4,19	59,86
M_H	dN·m	46,09	1,06	2,96	6,42	2,41	6,74	14,62
t_{S1}	min	2,23	0,05	0,14	6,72	0,25	0,69	30,94
$t'_C(50)$	min	4,43	0,18	0,49	11,06	0,27	0,75	16,93
$t'_C(90)$	min	13,47	0,45	1,25	9,28	0,95	2,67	19,82
NOTE s_r = Repeatability standard deviation, in measurement units. s_R = Reproducibility standard deviation, in measurement units.								
[a] Measured at 160 °C, 1,7 Hz, 1° amplitude arc. The mean level was used for calculations of (r) and (R).								

9 Test report

The test report shall include the following:

a) a reference to this International Standard;

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

c) the time and temperature used for the Mooney viscosity determination, and whether a massing procedure was used (and, if so, the parameters);

d) the method used for the ash determination (method A or B of ISO 247:2006);

e) the standard test formulation used;

f) the reference materials used;

g) the mixing procedure used in 5.2.2;

h) the conditioning times used in 5.2.2.2.1 g) or 5.2.2.2.2 i) or 5.2.2.3 j) or 5.2.2.4.1 g) and 5.2.2.4.2 g);

i) for Clause 6:

— the type of curemeter used,

— the reference standard,

— the time for M_H or F_{max},

— the amplitude of oscillation used for the curemeter test;

j) the vulcanization periods used in Clause 7;

k) any unusual features noted during the determination;

l) any operation not included in this International Standard or in the International Standards to which reference is made, as well as any operation regarded as optional;

m) the results and the units in which they have been expressed;

n) the date of the test.

Bibliography

[1] ISO 1382, *Rubber — Vocabulary*

[2] ISO 6472, *Rubber compounding ingredients — Abbreviations*

[3] ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards* (now withdrawn)

[4] ASTM D 88, *Standard Test Method for Saybolt Viscosity*

[5] ASTM D 2161, *Standard Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity*

[6] ASTM D 3568-03, *Standard Test Methods for Rubber — Evaluation of EPDM (Ethylene Propylene Diene Terpolymers) Including Mixtures With Oil*

ICS 83.060

Price based on 11 pages