

BS ISO 4097:2014



BSI Standards Publication

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

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**National foreword**

This British Standard is the UK implementation of ISO 4097:2014. It supersedes BS ISO 4097:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Rubber - Raw, natural and synthetic, including latex and carbon black.

A list of organizations represented on this committee can be obtained on request to its secretary.

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

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



Reference number  
ISO 4097:2014(E)



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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](http://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](http://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 sixth edition cancels and replaces the fifth edition (ISO 4097:2007), which has been technically revised with the following changes:

- addition of new standard test formulations for the evaluation of EPDM rubbers ([Table 1](#));
- removal of tetramethyl thiuram disulfide (TMTD) from the standard test formulations;
- changes in mixing procedures ([5.3](#)): inclusion of two-stage mixing and laboratory internal mixer;
- addition of a statement that single-stage mixing is the preferred method ([5.3.1](#));
- addition of new precision data in a new informative [Annex A](#);
- the standard test formulations and the precision data of the fifth edition (ISO 4097:2007) were moved to a new informative [Annex B](#);
- addition of a statement in [Annex B](#) to explain that the standard formulations of the fifth edition (ISO 4097:2007) are retained until the next systematic review with a limitation of five years.

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

**WARNING** — Persons using this International Standard 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 International Standard specifies the following:

- physical and chemical tests on raw rubbers;
- standard materials, standard test formulations, equipment, and processing methods for evaluating the vulcanization characteristics of ethylene-propylene-diene rubbers (EPDM), including oil-extended types.

## 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 247, *Rubber — Determination of ash*

ISO 248-1, *Rubber, raw — Determination of volatile-matter content — Part 1: Hot-mill method and oven method*

ISO 248-2, *Rubber, raw — Determination of volatile-matter content — Part 2: Thermogravimetric methods using an automatic analyser with an infrared drying unit*

ISO 289-1, *Rubber, unvulcanised — 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  $50\text{ °C} \pm 5\text{ °C}$  (for rubbers with a low Mooney viscosity, a temperature of  $35\text{ °C} \pm 5\text{ °C}$  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 248-1 or ISO 248-2.

### 4.3 Ash

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

## 5 Preparation of test mixes for evaluation

### 5.1 Standard test formulations

The standard test formulations are given in [Table 1](#), in which:

- a) formulation 1 is applicable to non-oil-extended EPDMs with a nominal ethylene content not higher than 67 % by mass;
- b) formulation 2 is applicable to non-oil-extended EPDMs with a nominal ethylene content equal to or higher than 67 % by mass;
- c) formulation 3 is applicable to non-oil-extended low Mooney viscosity EPDMs;
- d) formulation 4 is applicable to oil-extended EPDMs containing 50 or less parts by mass of oil per 100 parts of rubber;
- e) 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;
- f) 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 material is 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
	<b>Parts by mass</b>					
EPDM	100,00	100,00	100,00	100,00 + x <sup>a</sup>	100,00 + y <sup>b</sup>	100,00 + z <sup>c</sup>
Stearic acid	1,00	1,00	1,00	1,00	1,00	1,00
Industry reference black <sup>d</sup>	80,00	100,00	40,00	80,00	80,00	150,00
ASTM 103 oil <sup>e</sup>	50,00	75,00	—	50,00 – x <sup>a</sup>	—	—
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
N-Cyclohexyl-2-Mercaptobenzothiazilesulphenamide(CBS)	3,5	3,5	3,5	3,5	3,5	3,5
Mercaptobenzothiazole (MBT)	1,0	1,0	1,0	1,0	1,0	1,0
Total	242,00	287,00	152,00	242,00	192,00 + y <sup>b</sup>	262,00 + z <sup>c</sup>
<p><sup>a</sup> 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.</p> <p><sup>b</sup> 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.</p> <p><sup>c</sup> 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.</p> <p><sup>d</sup> The current industry reference black (IRB) is used.</p> <p><sup>e</sup> This oil density is 0,92 g/cm<sup>3</sup>. Alternative oils can be used but might give slightly different results. ASTM 103 oil is an example of a suitable product available commercially. It 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. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.</p>						

## 5.2 Equipment and procedure

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

## 5.3 Mixing procedures

### 5.3.1 General

Four alternative mixing procedures are specified.

- Method A1: single stage mixing with LIM (laboratory internal mixer) which is the preferred method.
- Method A2: two-stage mixing with LIM.
- Method A3: two-stage mixing using a LIM for initial mixing and mill mixing for final mixing.
- Method B: mill mixing.

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 a LIM allows better results. Because of the difficulty of mill mixing EPDM rubbers, it is recommended that method B be used only if a LIM is not available.

### 5.3.2 LIM mixing for methods A1, A2, and A3

#### 5.3.2.1 General

The mixing technique in each method can be modified to achieve a good dispersion of all the ingredients. The LIM conditions shall be the same during the preparation of a series of identical mixes for each batch mixed. At the beginning of each series of test mixes, a machine-conditioning batch shall be mixed using the same formulation as the mixes under test. The LIM shall be allowed to cool down to 60 °C between the end of one test batch and the start of the next. Temperature control condition shall not be altered during the mixing of a series of test.

#### 5.3.2.2 Method A1 — Single stage mixing with LIM

The final temperature of the batch discharged after mixing shall not exceed 120 °C. If necessary, adjust the batch mass, head temperature, or rotors speed, so that this condition is met.

Compounding materials other than rubber, carbon black, and oil can be added to LIM batches more precisely and with greater ease if they are previously blended together in the proportions required by the formulation. Such blends can be made using a mortar and pestle, by mixing for 10 min in a biconical blender with intensifier bar turning, or by mixing in a blender for five 3 s-periods and scraping the inside of the blender to dislodge materials stuck to the sides after each 3 s-mix. A Waring<sup>1)</sup> blender has been found suitable for this method.

**Caution — If mixed longer than 3 s, the stearic acid can melt and prevent good dispersion.**

NOTE An example of mixing procedure for LIM is as follows.

	Duration (min)	Cumulative time (min)
a) Load the rubber, lower the ram and allow the rubber to be masticated.	1,0	1,0
b) Raise the ram and add the pre-blended zinc oxide, sulfur, stearic acid, and accelerators, taking care to avoid any loss. Then add the carbon black and oil, sweep the orifice, and lower the ram.	1,0	2,0
c) Allow the batch to mix.	7,0	9,0
	-----	
Total time (max)	9,0	
d) Turn off the motor, raise the ram, remove the mixing chamber, and discharge the batch. Record the maximum batch temperature.		
e) After discharging the mixed batch, immediately pass it through a laboratory mill with its mill opening set at 0,8 mm and at a temperature of 50 °C ± 5 °C.		
f) Pass the rolled batch endwise through the rolls six times.		
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 -1,5 % to +0,5 %, discard the batch and re-mix.		
h) Remove sufficient material for cure testing.		
i) Sheet the batch to approximately 2,2 mm for preparing test sheets or to the appropriate thickness for preparing ISO ring test pieces in accordance with ISO 37.		

1) This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

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

### 5.3.2.3 Method A2 — Two-stage mixing with LIM

#### 5.3.2.3.1 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 -1,5 % to +0,5 %, 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.		

#### 5.3.2.3.2 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 <a href="#">5.3.2.3.1</a> , 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 rolled 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 -1,5 to +0,5 %, 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.

**5.3.2.4 Method A3 — Two-stage mixing using a LIM for initial mixing and a mill for final mixing**

**5.3.2.4.1 Stage 1 — Initial mixing procedure**

	<b>Duration (min)</b>	<b>Cumulative time (min)</b>
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 -1,5 % to +0,5 %, 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.		

**5.3.2.4.2 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 might be necessary.

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

	<b>Duration (min)</b>	<b>Cumulative time (min)</b>
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

	<b>Duration (min)</b>	<b>Cumulative time (min)</b>
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 -1,5 % to +0,5 %, 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.		

### 5.3.3 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.

NOTE 1 In formulations 1, 2, and 4, some of the oil can be withheld to be added at step 5.3.3 c).

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 might be necessary.

	<b>Duration (min)</b>	<b>Cumulative time (min)</b>
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 2 In formulations 1, 2, and 4, some of the oil can be withheld to be added at step c).	13,0	
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.	[steps b) and c)]	14,0
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

	<b>Duration (min)</b>	<b>Cumulative time (min)</b>
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 -1,5 % to +0,5 %, discard the batch and re-mix.		
h) Remove sufficient material for cure 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.		

## 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,  $t_{s1}$ ,  $t'_c(50)$ , 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 % might 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 % might 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

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

See [Annex A](#).

## 9 Test report

The test report shall include the following:

- a) a reference to this International Standard, i.e. ISO 4097;
- 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 content determination (method A or B of ISO 247);
- e) the standard test formulation used;
- f) the reference materials used;
- g) the mixing procedure used in [5.3](#);
- h) the conditioning times used in [5.3.2.2 j](#)), or [5.3.2.3.1 g](#)) and [5.3.2.3.2 i](#)), or [5.3.2.4.1 g](#)) and [5.3.2.4.2 g](#)), or [5.3.3 j](#));
- i) for [Clause 6](#):
  - the type of curemeter used,
  - the reference standard, and
  - 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.

## Annex A (informative)

### Precision

#### A.1 General

An interlaboratory test program (ITP) for the precision evaluation of both the single mixing (Method A1 – LIM mixing) and the two-stage mixing (Method A3 – LIM/mill-mixing) procedure was conducted in August 2011. The calculation was carried out in accordance with ISO/TR 9272[4]. Five laboratories participated in this ITP.

Two types of EPDM samples as shown in [Table A.1](#) were used for this ITP. The test formulation 1 (according to [Table 1](#)) is applied for EPDM-2 and the test formulation 5 for EPDM-1. The carbon black used was IRB No.8. Each compound was prepared twice and tested on each of the two different days at one week interval.

**Table A.1 — Type of EPDM**

Sample	EPDM-1	EPDM-2
ML(1+4) at 125 °C	69	66
Ethylene content (by mass %)	66	65
Diene content (by mass %)	4,5	4,0
Extender oil content (phr)	75	0

The precision results as determined by this ITP should not be used for acceptance or rejection testing of any group of materials or products without documentation that they are applicable to those particular materials or products and the specific test protocol of the test method.

NOTE The precision results of the two stage mixing, method A2, and mill mixing, method B, can be referred to ASTM D3568-03.

#### A.2 Results

##### A.2.1 General

The precision results of methods A1 and A3 from the two EPDM samples for curemeter and stress-strain testing are given [Tables A.2](#) through [A.5](#).

Two single test results obtained in the same laboratory (by the proper use of this International Standard) that differ by more than the tabulated values for  $r$ , in measurement units, or  $(r)$ , in percent, should be suspected of being from different populations and some appropriate action taken.

The precision results can be summarized as follows.

##### A.2.2 Repeatability

###### A.2.2.1 Curemeter properties

The values of repeatability  $(r)$  from methods A1 and A3 (see [Tables A.2](#) and [A.3](#)) for two rubbers are reasonably consistent. On an overall basis (all properties for two rubbers), an average  $(r)$  of 10,9 % and 6,3 % has been respectively found.



### A.2.2.2 Stress-strain properties

The values of repeatability ( $r$ ) from methods A1 and A3 (see [Tables A.4](#) and [A.5](#)) for two rubbers are reasonably consistent. On an overall basis (all properties for two rubbers), an average ( $r$ ) of 10,6 % and 8,8 % has been respectively found.

### A.2.3 Reproducibility

#### A.2.3.1 Curemeter properties

The values of reproducibility ( $R$ ) from methods A1 and A3 for two rubbers are less consistent than within laboratory results, but the level is acceptable. On an overall basis (all properties for two rubbers), an average ( $R$ ) of 33,1 % and 23,9 % has been respectively found.

#### A.2.3.2 Stress-strain properties

The values of reproducibility ( $R$ ) from methods A1 and A3 for two rubbers are less consistent than within laboratory results, but the level is acceptable. On an overall basis (all properties for two rubbers), an average ( $R$ ) of 30,2 % and 20,5 % has been respectively found.

### A.2.4 Bias

In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method. Therefore bias cannot be determined.

**Table A.2 — Precision of curemeter properties for method A1 procedure**

Property unit	Material	Mean	Within laboratory			Between laboratories			Number of laboratories <sup>a</sup>
			$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$	
$M_L$ dN-m	EPDM-1	2,81	0,16	0,45	15,9	0,36	1,02	36,4	5
	EPDM-2	2,22	0,06	0,18	8,1	0,20	0,57	25,6	5
$M_H$ dN-m	EPDM-1	14,69	0,41	1,16	7,9	2,59	7,33	49,9	5
	EPDM-2	17,09	0,58	1,63	9,5	2,00	5,66	33,1	4
$t_{s1}$ min.	EPDM-1	2,01	0,13	0,37	18,4	0,29	0,82	40,8	5
	EPDM-2	2,00	0,04	0,13	6,3	0,33	0,94	47,0	5
$t'_c(50)$ min.	EPDM-1	4,80	0,12	0,35	7,2	0,20	0,57	12,0	4
	EPDM-2	4,40	0,11	0,31	7,1	0,46	1,30	29,6	5
$t'_c(90)$ min.	EPDM-1	13,38	0,64	1,80	13,5	1,55	4,38	32,7	5
	EPDM-2	12,76	0,69	1,96	15,4	1,09	3,09	24,2	4
Average					10,9			33,1	

$S_r$  within-laboratory standard deviation (in measurement units)  
 $r$  repeatability (in measurement units)  
 $(r)$  repeatability (in percent of mean level)  
 $S_R$  between-laboratories standard deviation (for total between-laboratories variation) (in measurement units)  
 $R$  reproducibility (in measurement units)  
 $(R)$  reproducibility (in percent of mean level)  
<sup>a</sup> The number of laboratories is the final number of laboratories in the ITP after deletion of outliers (using option 1).

**Table A.3 — Precision of curemeter properties for method A3 procedure**

Property unit	Material	Mean	Within laboratory			Between laboratories			Number of laboratories <sup>a</sup>
			$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$	
$M_L$ dN-m	EPDM-1	2,52	0,04	0,13	5,0	0,32	0,92	36,4	5
	EPDM-2	2,07	0,07	0,20	9,7	0,26	0,73	35,1	5
$M_H$ dN-m	EPDM-1	13,86	0,30	0,85	6,1	2,40	6,80	49,1	5
	EPDM-2	16,58	0,36	1,01	6,1	1,89	5,34	32,2	4
$t_{s1}$ min.	EPDM-1	2,35	0,05	0,14	6,0	0,08	0,23	10,0	4
	EPDM-2	2,23	0,05	0,14	6,4	0,08	0,22	9,9	4
$t'_c(50)$ min.	EPDM-1	5,08	0,06	0,18	3,5	0,22	0,62	12,2	5
	EPDM-2	4,93	0,05	0,14	2,9	0,14	0,39	8,0	4
$t'_c(90)$ min.	EPDM-1	13,16	0,40	1,12	8,6	1,48	4,18	31,8	5
	EPDM-2	12,38	0,40	1,12	9,1	0,63	1,77	14,3	4
Average					6,3			23,9	

NOTE For the meanings of the symbols used in this Table, see [Table A.2](#).

<sup>a</sup> The number of laboratories is the final number of laboratories in the ITP after deletion of outliers (using option 1).

**Table A.4 — Precision of stress-strain properties for method A1 procedure**

Property unit	Material	Mean	Within laboratory			Between laboratories			Number of laboratories <sup>a</sup>
			$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$	
$S_{300}$ MPa	EPDM-1	8,51	0,37	1,06	12,4	0,63	1,77	20,8	4
	EPDM-2	7,97	0,54	1,53	19,2	0,58	1,65	20,7	4
$TS_b$ MPa	EPDM-1	20,73	0,54	1,52	7,3	3,23	9,15	44,2	5
	EPDM-2	15,96	0,60	1,69	10,6	1,83	5,19	32,5	5
$E_b$ %	EPDM-1	491,0	13,78	39,0	7,9	71,6	202,5	41,2	5
	EPDM-2	476,0	10,00	28,3	6,0	36,4	102,9	21,6	5
Average					10,6			30,2	

NOTE For the meanings of the symbols used in this Table, see [Table A.2](#).

<sup>a</sup> The number of laboratories is the final number of laboratories in the ITP after deletion of outliers (using option 1).

**Table A.5 — Precision of stress-strain properties for method A3 procedure**

Property unit	Material	Mean	Within laboratory			Between laboratories			Number of laboratories <sup>a</sup>
			$S_r$	$r$	$(r)$	$S_R$	$R$	$(R)$	
$S_{300}$ MPa	EPDM-1	8,33	0,37	1,06	12,7	0,42	1,18	14,2	4
	EPDM-2	9,42	0,37	1,05	11,2	1,14	3,22	34,2	5
$TS_b$ MPa	EPDM-1	21,99	0,65	1,84	8,4	1,95	5,52	25,1	5
	EPDM-2	18,25	0,67	1,88	10,3	0,64	1,81	9,9	4
$E_b$ %	EPDM-1	520,0	7,75	21,9	4,2	34,4	97,4	18,7	5
	EPDM-2	483,0	10,49	29,7	6,2	35,3	99,9	20,7	5
Average					8,8			20,5	

NOTE For the meanings of the symbols used in this Table, see [Table A.2](#).

<sup>a</sup> The number of laboratories is the final number of laboratories in the ITP after deletion of outliers (using option 1).

## Annex B (informative)

### Standard test formulations of ISO 4097:2007

#### B.1 General

Tetramethyl thiuram disulfide (TMTD) has been recognized as a nitrosoamine generator. Therefore, the revision of ISO 4097:2007 is aimed to remove the material from the standard test formulations.

The standard formulations given in ISO 4097:2007 are retained until the next systematic review with a limitation of five years, to allow time for users of this International standard to adapt to the new standard test formulations.

#### B.2 Standard test formulation of ISO 4097:2007

The standard test formulations are given in [Table B.1](#). They are described in [5.1](#) a) to f).

The materials used shall be national or International Standard reference materials unless no standard reference material is available, in which case the materials to be used shall be agreed between the interested parties.

**Table B.1 — ISO 4097:2007 test formulation for evaluation of EPDM rubbers**

Material	Test formulation					
	B1	B2	B3	B4	B5	B6
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 <sup>d</sup>	80,00	100,00	40,00	80,00	80,00	150,00
ASTM 103 oil <sup>e</sup>	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 disulphide (TMTD)	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 + $z^c$

<sup>a</sup>  $x$  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.

<sup>b</sup>  $y$  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.

<sup>c</sup>  $z$  is the number of parts by mass of oil per 100 parts of base rubber for types having a minimum oil content of 80.

<sup>d</sup> The current industry reference black (IRB) is used.

<sup>e</sup> This oil density is 0,92 g/cm<sup>3</sup>. Alternative oils can be used but might give slightly different results. ASTM 103 oil is an example of a suitable product available commercially. It 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. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

## B.3 Precision

### B.3.1 General

The precision results for the internal-mixer procedure are based on data from ASTM D 3568-03[5]. The repeatability and reproducibility were calculated in accordance with ISO/TR 9272:1986[3].

### B.3.2 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.

### B.3.3 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).

### B.3.4 Precision results

The calculated repeatability and reproducibility values are given in [Tables B.2](#) and [B.3](#). The symbols used in [Tables B.2](#) and [B.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 can be expected to lie, with a specified probability.
- $(r)$  repeatability, in percent (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 can 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 B.2 — Internal-mixing procedure type 2 precision for various test parameters**

Property	Units	Range of values <sup>a</sup>	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
$s_r$ repeatability standard deviation, in measurement units								
$s_R$ reproducibility standard deviation, in measurement units								
<sup>a</sup> Measured at 160 °C, 1,7 Hz, 1° amplitude arc. The midpoint of the range was used for calculations of $(r)$ and $(R)$ .								

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

Property	Units	Mean level <sup>a</sup>	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

$s_r$  repeatability standard deviation, in measurement units  
 $s_R$  reproducibility standard deviation, in measurement units  
<sup>a</sup> Measured at 160 °C, 1,7 Hz, 1° amplitude arc. The mean level was used for calculations of  $(r)$  and  $(R)$ .

## Bibliography

- [1] ISO 1382, *Rubber — Vocabulary*
- [2] ISO 6472, *Rubber compounding ingredients — Symbols and abbreviated terms*
- [3] ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards*
- [4] ISO/TR 9272:2005, *Rubber and rubber products — Determination of precision for test method standards*
- [5] ASTM D3568-03, *Standard Test Methods for Rubber — Evaluation of EPDM (Ethylene Propylene Diene Terpolymers) Including Mixtures With Oil*









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