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

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

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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).

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document 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 seventh edition cancels and replaces the sixth edition (ISO 4097:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

— normative references have been updated in Clauses 2 and 10 d) and subclauses 5.3, and 7.1, in particular replacing ISO 247 by ISO 247-1 and ISO 247-2;

— the standard formulations given in ISO 4097:2007 have been retained in ISO 4097:2014, to allow time for users to adapt to the new standard test formulations; they have been removed by deleting Annex B of the previous edition.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

WARNING — Users of this document should be familiar with the normal laboratory practice. This document 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.

1 Scope

This document specifies:

— the physical and chemical tests on raw rubbers;

— the 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 are referred to in the text in such a way that some or all of their content constitutes requirements 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 247-1:2018, *Rubber — Determination of ash — Part 1: Combustion method*

ISO 247-2:2018, *Rubber — Determination of ash — Part 2: Thermogravimetric analysis (TGA)*

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, 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 6502-1, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 1: Introduction*

ISO 6502-2, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 2: Oscillating disc curemeter*

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

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Sampling and sample preparation

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

4.2 Prepare the test portion in accordance with ISO 1795.

5 Physical and chemical tests on raw rubber

5.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289-1, on a test portion prepared as indicated in 4.2 (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.

5.2 Volatile matter

Determine the volatile matter content in accordance with ISO 248-1 or ISO 248-2.

5.3 Ash

Determine the ash in accordance with either method A, or method B, or method C of ISO 247-1:2018, or method A of ISO 247-2:2018.

6 Preparation of test mixes for evaluation

6.1 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
	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
N-Cyclohexyl-2-Mercapto-benzothiazilesulphenamide (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[b]	262,00 + 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) is used.

[e] This oil density is 0,92 g/cm³. 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 document and does not constitute an endorsement by ISO of this product.

6.2 Equipment and procedure

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

6.3 Mixing procedures

6.3.1 General

Four alternative mixing procedures are specified.

— Method A1: single stage mixing with laboratory internal mixer (LIM) 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.

6.3.2 LIM mixing for methods A1, A2, and A3

6.3.2.1 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.

6.3.2.2 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[1)] 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 (maximum)	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.

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

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

6.3.2.3 Method A2 — Two-stage mixing with LIM

6.3.2.3.1 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.	maximum 1,5	5,0
	Total time (maximum)	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.

6.3.2.3.2 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 6.3.2.3.1, 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.	maximum 1,5	2,0
	Total time (maximum)	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.