INTERNATIONAL STANDARD

ISO 4097

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

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

Reference number
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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard 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 fourth edition cancels and replaces the third edition (ISO 4097:1991), which has been technically revised.

Annex A of this International Standard is for information only.

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

WARNING — Persons using this International Standard should be familiar with normal laboratory practices. 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 normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*.

ISO 247:1990, *Rubber — Determination of ash*.

ISO 248:1991, *Rubbers, raw — Determination of volatile-matter content*.

ISO 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*.

ISO 471:1995, *Rubber — Temperatures, humidities and times for conditioning and testing*.

ISO 1795:2000, *Rubber, raw natural and raw synthetic — Sampling and further preparative procedures*.

ISO 2393:1994, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*.

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter*.

ISO 6502:1999, *Rubber — Guide to the use of curemeters*.

ISO/TR 9272:1986, *Rubber and rubber products — Determination of precision for test method standards*.

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 B of ISO 247:1990.

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 oil-extended EPDMs containing less than 80 parts by mass of oil per 100 parts of rubber;

— formulation 4 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.

An alternative formulation for non-oil-extended low Mooney viscosity EPDMs is given in annex A.

Table 1 — Standard test formulations for evaluation of EPDM rubbers

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

[a] y is the number of parts by mass of oil per 100 parts of base rubber in the oil-extended rubber. If y is greater than 50 but less than 80, do not add oil to formulation 3. In this case, the total mass of the formulation will be higher than 239.

[b] 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.

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

[d] This oil, density 0,92 g/cm^3, is produced by the Sun Refining and Marketing Company and distributed by R.E. Carroll Inc., P.O. Box 139, Trenton, NJ 08601, USA. Overseas requests should be directed to Sunoco Overseas Inc., 1801 Market Street, Philadelphia, PA 19103, USA. Alternative oils, such as Shellflex 724, are suitable but may give slightly different results.

ASTM 103 oil has the following characteristics:

kinematic viscosity at 100 °C: 16,8 mm^2/s ± 1,2 mm^2/s;

viscosity gravity constant: 0,889 ± 0,002.

The viscosity gravity constant (VGC) is calculated from the Saybolt universal viscosity at 37,8 °C and the relative density (specific gravity) at 15,5 °C/15,5 °C. Use the following equation to calculate the VGC from the measured properties:

$$\mathrm{VGC} = \frac{10d - 1{,}0752 \log_{10}(v - 38)}{10 - \log_{10}(v - 38)}$$

where

d is the relative density (specific gravity) at 15,5 °C/15,5 °C;

v is the Saybolt universal viscosity at 37,8 °C (ASTM D 88/ASTM D 2161).

[e] A standard reference material for TMTD is available as IRM 1 from Forcoven Products Inc., P.O. Box 1556, Humble, Texas 77338, 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 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 471.

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 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 +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 471.

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 (see, however, 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.	13,0 [steps b) + c)]	14,0

NOTE In formulations 2 and 3, some of the oil may be withheld for addition c).