INTERNATIONAL STANDARD

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

Caoutchouc éthylène-propylène-diène (EPDM) — Types à usage général — Méthode d'évaluation

INTERNATIONAL ISO

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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.

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.

International Standard ISO 4097 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*.

This third edition cancels and replaces the second edition (ISO 4097:1988), of which clause 1, subclause 5.1, table 1 and clause A.3 have been technically revised. The scope of the standard has been widened to include oil-extended general purpose EPDM rubbers, and as a result three new standard test formulae (2, 3 and 4) have been added.

Annex A forms an integral part of this International Standard.

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

1 Scope

This International Standard specifies

— physical and chemical tests on raw rubbers;

— standard materials, standard test formulae, equipment and processing methods for evaluating the vulcanization characteristics of general purpose ethylene-propylene-diene (EPDM) rubbers, including oil-extended types.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1977, *Rubber, vulcanized — Determination of tensile stress-strain properties.*

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

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

ISO 289:1985, *Rubber, unvulcanized — Determination of Mooney viscosity.*

ISO 471:1983, *Rubber — Standard temperatures, humidities and times for the conditioning and testing of test pieces.*

ISO 1795:1974, *Raw rubber in bales — Sampling.*

ISO 1796:1982, *Rubber, raw — Sample preparation.*

ISO 2393:1973, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures.*

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

ISO 6502:1991, *Rubber — Measurement of vulcanization characteristics with rotorless curemeters.*

3 Sampling and sample preparation

3.1 A sample of mass approximately 1 500 g shall be taken by the method described in ISO 1795.

3.2 Preparation of the test portion shall be in accordance with ISO 1796.

4 Physical and chemical tests on raw rubber

4.1 Mooney viscosity

Determine the Mooney viscosity in accordance with ISO 289 on a test portion prepared as indicated in ISO 1796. If a massing process is necessary, maintain the mill roll surface temperature at 35 °C ± 5 °C. Record the result as ML (1 + 4) at 125 °C.

Other test conditions, 100 °C or 150 °C instead of 125 °C, and 8 min instead of 4 min, may be used by agreement between the interested parties.

4.2 Volatile matter

Determine the volatile matter content by the oven method as specified in ISO 248.

4.3 Ash

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

5 Preparation of the test mixes for evaluation of EPDM rubbers

5.1 Standard test formulae

The standard test formulae are given in table 1, in which

— formula 1 is applicable to non-oil-extended EPDM with an ethylene content no higher than 67 % by mass;

— formula 2 is applicable to non-oil-extended EPDM with an ethylene content higher than 67 % by mass;

— formula 3 is applicable to oil-extended EPDM containing less than 80 parts by mass of oil per 100 parts of rubber;

— formula 4 is applicable to oil-extended EPDM containing 80 or more parts by mass of oil per 100 parts of rubber.

The materials shall be NIST*) standard reference materials as indicated in table 1, or other equivalent national or international standard reference materials.

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.

Details of a suitable internal mixer are given in annex A.

5.2.2 Mixing procedures

Two alternative mixing procedures are specified.

Method A — Mill mixing.

Method B — Internal mixer for initial and mill for final mixing.

NOTE 1 Mixing of ethylene-propylene-diene rubbers in the standard test formulae 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 mixing EPDM rubbers, it is recommended that method B be used whenever such apparatus is available.

*) National Institute of Standards and Technology (formerly the National Bureau of Standards) of the USA.

Table 1 — Standard test formulae for evaluation of EPDM rubbers

Material	NIST standard reference material number	Test formula 1	2	3	4
		Parts by mass			
EPDM	-	100,00	100,00	100,00 + Y [1]	100,00 + Z [2]
Stearic acid	372	1,00	1,00	1,00	1,00
Oil furnace black (HAF)[3]	378	80,00	100,00	80,00	150,00
ASTM 103 oil[4]	-	50,00	75,00	50,00 − Y [1]	—
Zinc oxide	370	5,00	5,00	5,00	5,00
Sulfur	371	1,50	1,50	1,50	1,50
Tetramethyl thiuram disulfide (TMTD)[5]	-	1,00	1,00	1,00	1,00
Mercaptobenzothiazole (MBT)	383	0,50	0,50	0,50	0,50
Total		239,00	284,00	239,00 + (Y − 50) if $Y > 50$	259,00 + Z

1) "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, do not add oil to formula 3. In this case, the total mass of the formula will be higher than 239.

2) "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.

3) The current Industry Reference Black may be used in place of NIST 378, but this may give slightly different results.

4) This oil, density 0,92 g/cm³, 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 Circosol 4240 or Shellflex 724, are suitable but may give slightly different results.

ASTM 103 oil has the following characteristics:

— Kinematic viscosity at 100 °C: 16,8 mm²/s ± 1,2 mm²/s;

— Viscosity gravity constant: 0,889 ± 0,002.

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

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

where

d is the density at 15,5/15,5 °C;

v is the Saybolt Universal viscosity at 37,8 °C.

5) A standard reference material for TMTD is available as IRM 1 from Forcoven Products Inc., P.O. Box 1556, Humble, Texas 77338, USA.

5.2.2.1 Method A — Mill mixing

a) The standard laboratory mill batch mass, in grams, shall be based on twice the formula mass. The surface temperatures of the rolls shall be maintained at 35 °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.

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)
b) Band the rubber on the fast roll with the mill set at 35 °C and 0,7 mm opening.	1,0	1,0
c) Add the mixture of oil, carbon black, zinc oxide and stearic acid, with a spatula, evenly across the mill. 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.	13,0	14,0
d) Add accelerators and sulfur, evenly across the rolls still at 1,8 mm opening.	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 %, discard the batch and re-mix. 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.

i) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 471.

5.2.2.2 Method B — Internal mixer for initial and mill for final mixing

5.2.2.2.1 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.	—	
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.	1,5 (max.)	5,0
Total time (max.)	5,0	

f) Immediately pass the batch three times through a laboratory mill with a mill opening of 2,5 mm and 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 %, discard the batch and re-mix.

g) Leave the batch for at least 30 min and up to 24 h, if possible at standard temperature and humidity as defined in ISO 471.

5.2.2.2.2 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.

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

	Duration (min)	Cumulative time (min)
b) Set the mill temperature at 50 °C ± 5 °C and the mill opening to 1,5 mm. Band the masterbatch on the slow roll and add the sulfur and accelerators. Do not cut the band until the sulfur and accelerators are completely dispersed.	1,0	1,0
c) Make three 3/4 cuts from each side, allowing 15 s between each cut.	2,0	3,0
d) 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	

e) 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 %, discard the batch and re-mix. 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) Condition the batch for 2 h to 24 h after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 471.

6 Evaluation of vulcanization characteristics with a curemeter test

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 or ISO 6502, using the following test conditions:

oscillation frequency:	1,7 Hz (100 cycles per minute)
amplitude of oscillation:	1° arc An amplitude of oscillation of 3° arc is permitted as an alternative.
selectivity:	to be chosen to give at least 75 % of full scale deflection at M_H
	NOTE 2 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

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 three periods of cure shall be chosen to cover the 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 471.

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

8 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 conditions used for the Mooney viscosity determination, and whether a massing process was used;

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

e) the standard test formula used;

f) the reference materials used;

g) the mixing procedure used in 5.2.2;

h) the conditioning conditions used in 5.2.2.1 i), or 5.2.2.2.1 g) and 5.2.2.2.2 g);

i) for clause 6:

— the reference standard,

— the time for M_H,

— 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
(normative)

Internal mixer

NOTE 3 This annex will be deleted after publication of the second edition of ISO 2393, in which the use of internal mixers will be specified.

A.1 The internal mixer[1] shall have a nominal capacity of approximately 1 000 cm^3.

A.2 The rotor speed(s), ram pressure and coolant of the internal mixer shall be such that the time/temperature programme set out in 5.2.2.2.1 will be accomplished.

A.3 The batch size, in grams, shall be 1,05 to 1,10 times the nominal capacity, in cubic centimetres, of the internal mixer multiplied by the density, in grams per cubic centimetre, of the cold mix.

NOTE 4 If an old or worn internal mixer is used, the batch mass should be increased accordingly.

1) A type B Banbury internal mixer has been found to be satisfactory for this purpose. Other internal mixers may be used, if the mass, temperatures and time of mixing are adjusted to give comparable results.