International Standard 4097

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Rubber, ethylene-propylene-diene (EPDM) — Non-oil extended raw general purpose types — Evaluation procedures

Caoutchouc ethylène-propylène-diène (EPDM) — Types à usage général non étendus à l'huile — Méthodes d'évaluation

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO member bodies). The work of developing International Standards is carried out through ISO technical committees. Every member body interested in a subject for which a technical committee has been set up 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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 4097 was developed by Technical Committee ISO/TC 45, *Rubber and rubber products*, and was circulated to the member bodies in June 1978.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Spain
Austria	Hungary	Sri Lanka
Belgium	Ireland	Sweden
Brazil	Korea, Rep. of	Thailand
Bulgaria	Mexico	Turkey
Canada	Netherlands	United Kingdom
Czechoslovakia	Poland	USA
Egypt, Arab Rep. of	Romania	USSR
France	South Africa, Rep. of	Yugoslavia

No member body expressed disapproval of the document.

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Rubber, ethylene-propylene-diene (EPDM) — Non-oil extended raw general purpose types — Evaluation procedures

1 Scope and field of application

This International Standard specifies :

a) physical and chemical tests on raw rubber;

b) standard test recipe (materials, equipment and processing methods) for evaluating vulcanization properties of non-oil extended raw general purpose ethylene-propylene-diene rubbers (EPDM). This recipe may not be applicable to certain high ethylene types for which modifications may have to be made.

2 References

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

ISO 247, *Rubber — Determination of ash.*

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

ISO/R 289, *Determination of viscosity of natural and synthetic rubbers by the shearing disk viscometer.*

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

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

ISO 1796, *Raw rubber — Sample preparation.* [1)]

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

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

3 Sampling and sample preparation

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

3.2 Sample preparation shall be in accordance with ISO 1796.

4 Physical and chemical tests on raw polymer

4.1 Mooney viscosity

Determine the Mooney viscosity according to ISO/R 289 on a portion of the sample prepared as in 3.2. Record the result as ML 1 + 4 at 125 °C.

4.2 Volatile matter

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

4.3 Ash

Determine the ash in accordance with ISO 247.

1) At present at the stage of draft. (Revision of ISO 1796-1972.)

5 Standard test recipe

5.1 Standard test formula

The standard test formula is given in the table.

The materials used shall be NBS[1)] Standard reference materials as indicated in the table, or shall be in accordance with equivalent national standards.

Material	NBS Standard reference material number	Parts by mass
EPDM	–	100,0
Stearic acid	372	1,0
Oil furnace black HAF*	378	80,0
ASTM 103 oil**	–	50,0
Zinc oxide	370	5,0
Sulphur	371	1,5
Tetramethylthiuram disulphide (TMTD)	374	1,0
Mercaptobenzothiazole (MBT)	383	0,5
Total		239,0

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

** This oil may be obtained from Sun Oil Company, Industrial Products Department, 1608 Walnut Street, Philadelphia, Pennsylvania, 19103, USA. Alternative oils, such as Circosol 4240 or Shellflex 724, are suitable, but may give slightly different results.

ASTM 103 type oil has the following characteristics :

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

— viscosity gravity constant : 0,889 ± 0,002, calculated from the viscosity in seconds at 37,8 °C and the density at 15,6 °C, using the formula

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

where

d is the density at 15,6 °C;

V is the Saybolt universal viscosity at 37,8 °C.

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

Two alternative mixing procedures are specified :

Method A — Mill mixing.

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

NOTES

1 Details of a suitable internal mixer are given in the annex.

2 Mixing of ethylene-propylene-diene rubbers 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 rubbers, it is recommended that method B be used whenever such apparatus is available.

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 ± 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 — All mill openings should be adjusted to maintain a good rolling bank at the nip of the rolls during mixing.

	Duration (min)
b) Band the rubber on the fast roll with the mill set at 35 °C and 0,7 mm opening	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.	

1) National Bureau of Standards of the USA.

Then add the remainder of the mixure, opening the mill to 1,8 mm. When all the mixture as been incorporated (including that which has fallen into the tray), make two 3/4 cuts from each side. 13,0

NOTE — Do not cut the band until all visible free black has been incorporated.

d) Add accelerators and sulphur, evenly across the rolls still at 1,8 mm opening 3,0

e) Make three 3/4 cuts from each side, allowing 15 s between each cut 2,0

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

Total time 21,0

g) Sheet the batch to an approximate thickness of 6 mm and check weigh. Remove sufficient sample for oscillating disc curemeter testing.

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

i) Condition the batch for 2 to 24 h after mixing and prior to vulcanizing at a standard laboratory temperature (see ISO 471).

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

NOTE — All mill openings shall be adjusted to maintain a good rolling bank at the nip of the rolls during 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 min^{-1}) start the rotor and raise the ram.....	—	
b) Charge the polymer, the zinc oxide, the carbon black, the oil, 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 ± 5 °C. Check weigh the batch.

g) Rest the batch for at least 30 min or until it reaches room temperature.

5.2.2.2.2 Stage 2 — Final mill mixing procedure.

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 ± 5 °C and the mill opening to 1,5 mm. Band the masterbatch on the slow roll and add the sulphur and accelerators. Do not cut the band until the sulphur 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 to 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 an approximate thickness of 6 mm and check weigh. Remove sufficient sample for viscometer or oscillating disc curemeter testing.

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

g) Condition the batch for 2 to 24 h after mixing and prior to vulcanizing, at a standard laboratory temperature (see ISO 471).

6 Evaluation of vulcanization characteristics

6.1 Stress-strain properties

Vulcanize sheets at 160 °C for three periods chosen from a cure series of 10, 20, 30 , 40, 50 min.

NOTE — The three periods of cure selected should cover the undercure, optimum cure and overcure of the material under test.

Condition the vulcanized test slab for 16 to 72 h at a standard laboratory temperature (see ISO 471).

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

6.2 Evaluation according to oscillating disc curemeter test

Measure the following standard test parameters :

M_L, M_H, 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 selected to give at least 75 % full scale deflection at M_H
die temperature :	160 °C
pre-heat time :	None

NOTE — If macro dies are used, a pre-heat time of 1 min is necessary.

Annex

Internal mixer

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

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

A.3 The batch size should be the nominal capacity of the internal mixer in cubic centimetres, multiplied by the density (approximately 1,08 g/m^3) plus 5 to 10 %.

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

1) An internal mixer type B Banbury 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.