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**Reclaimed isobutene-isoprene (IIR)
rubber — Evaluation procedure**

*Caoutchouc isobutène-isoprène (IIR) régénéré — Méthode
d'évaluation*



Reference number
ISO/TS 16096:2014(E)

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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 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*.

NOTE 1 ISO/TC 45/SC 3 decided to publish a Technical Specification, because the result of the inter-laboratory test procedure (ITP) to validate the evaluation procedure on reclaimed isobutene-isoprene rubber may be dubious.

NOTE 2 If the result of this ITP shows that the procedure is reproducible and reproducible, then this Technical Specification will be converted into International Standard.

Reclaimed isobutene-isoprene (IIR) rubber — Evaluation procedure

1 Scope

This Technical Specification specifies

- physical and chemical tests on raw reclaimed isobutene-isoprene (IIR) rubber, and
- standard materials, standard test formulations, equipment, and processing methods for evaluating the vulcanization characteristics and the mechanical properties of reclaimed isobutene-isoprene rubber.

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 247:2006, *Rubber — Determination of ash*

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

ISO 1407:2011, *Rubber — Determination of solvent extract*

ISO 1408:1995, *Rubber — Determination of carbon black content — Pyrolytic and chemical degradation methods*

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 7619-1, *Rubber, vulcanized or thermoplastic — Determination of indentation hardness — Part 1: Durometer method (Shore hardness)*

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

ASTM D297-13, *Standard Test Methods for Rubber Products — Chemical Analysis*

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 sample 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 piece prepared as indicated in [3.2](#).

Record the result as ML(1 + 4) at 100 °C.

4.2 Acetone extract

Determine the acetone extract in accordance with method A or method B of ISO 1407:2011.

4.3 Ash

Determine the ash content in accordance with either method A or method B of ISO 247:2006.

4.4 Carbon black

Determine the carbon black content in accordance with ISO 1408:1995, Method A.

4.5 Rubber content

Determine the rubber content, R (in %), in accordance with ASTM D297-13, Part A, Clauses 11, 12, and 13.

$$R = 100 - (a + b + c)$$

where:

a is the carbon black content, in percent (%);

b is the ash content, in percent (%);

c is the acetone extract, in percent (%).

5 Preparation of test mix for evaluation

5.1 Standard test formulation

The standard test formulation for evaluation of reclaimed isobutene-isoprene rubber is given in [Table 1](#).

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

Table 1 — Standard test formulation

Material	Parts by mass
Reclaimed isobutene-isoprene rubber (IIR)	100,00 + x + y + z
Zinc oxide ^a	5,00
Sulfur ^a	2,00
ZBEC (zinc dibenzylthiocarbamate) ^a	1,50
Mercaptobenzothiazole (MBT)	1,00
NOTE 1 x is the number of parts of carbon black to 100 parts of rubber in reclaimed isobutene-isoprene rubber.	
NOTE 2 y is the number of parts of acetone extract to 100 parts of rubber in reclaimed isobutene-isoprene rubber.	
NOTE 3 z is the number of parts of ash to 100 parts of rubber in reclaimed isobutene-isoprene rubber.	
^a Powder materials are used (standard curing ingredients used in the industry).	

5.2 Mixing procedure — Mixing with a laboratory mill

The standard laboratory mill-batch mass, in grams, shall be enough to form proper rolling band. The surface temperature of the rolls shall be maintained at $50\text{ °C} \pm 5\text{ °C}$ throughout the mixing.

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 can be necessary.

A mill batch mass based on two times the formulation mass can also be used. However in this case, more adjustment to the mill openings is necessary.

	Duration min	Cumulative time min
a) Band the reclaimed rubber with the mill opening set at 1,2 mm.	1,0	1,0
b) Add the zinc oxide across the mill rolls at a uniform rate. When all the zinc oxide has been incorporated, make a 3/4 cut from each side. Do not cut the batch while powder is evident in the bank or on the milling surface. Be certain to return to the batch any materials that drop through the mill.	1,0	2,0
c) Add the MBT across the mill rolls at a uniform rate. When all the powder has been incorporated, make a 3/4 cut from each side.	1,0	3,0
d) Add the ZBEC across the mill rolls at a uniform rate. When all the powder has been incorporated, make a 3/4 cut from each side.	1,0	4,0
e) Add the sulfur across the mill rolls at a uniform rate. When all the sulfur has been incorporated, make a 3/4 cut from each side.	1,0	5,0
f) Cut the batch from the mill and take off the mill rolls.	2,0	7,0

g) Set the mill opening to 1,2 mm and pass the rolled batch 3,0 10,0
endwise through the mill 12 times.

Total time (max.) $\frac{\quad}{10,0}$

- h) Sheet the batch in grain direction to an approximate thickness of 6 mm and check-weigh the batch (see ISO 2393). If the mass of the batch differs from the theoretical value by more than + 0,5 % or - 1,5 %, discard the batch and re-mix.
- i) Remove sufficient material for curemeter testing.
- j) Sheet the batch to approximately 2,2 mm for preparing test slabs or to the appropriate thickness for preparing ISO ring or dumbbell test pieces in accordance with ISO 37.
- k) After mixing, condition the batch for at least 2 h, but not more than 24 h, if possible at standard laboratory temperature and humidity as defined in ISO 23529.

NOTE For very low Mooney viscosity reclaim rubber, tendency of reclaim sticking to the roll can be observed. Hence, cutting as mentioned in the procedure cannot be possible. In such case, scraper blade as shown in [Figure 1](#) can be employed. After scraping the material each time, it should be folded two times and passed through mill again to ensure proper blending of chemical.



Figure 1 — Example of scraper blade

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} , $t'_c(50)$, $t'_c(90)$, $t'_c(95)$, and $t'_c(99)$

in accordance with ISO 3417, using the following test conditions:

- oscillation frequency: 1,7 Hz (100 cycles per minute);
- amplitude of oscillation: 1° of arc (An amplitude of oscillation of 3° of arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1} .);
- torque range: to be chosen to give at least 75 % of full-scale deflection at M_H ;

- die temperature: $160\text{ °C} \pm 0,3\text{ °C}$;
- 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} , $t'_c(50)$, $t'_c(90)$, $t'_c(95)$, and $t'_c(99)$

in accordance with ISO 6502, using the following test conditions:

- oscillation frequency: 1,7 Hz (100 cycles per minute);
- amplitude of oscillation: $0,5^\circ$ of arc (An amplitude of oscillation of 1° of arc is permitted as an alternative. If such an amplitude is chosen, measure t_{s2} instead of t_{s1} .);
- torque range: to be chosen to give at least 75 % of full-scale deflection at F_{max} ;
- die temperature: $160\text{ °C} \pm 0,3\text{ °C}$;
- pre-heat time: none.

The two types of curemeter cannot give identical results.

7 Evaluation of Mooney viscosity of test mixes

Determine the Mooney viscosity in accordance with ISO 289-1.

Record the result as ML(1 + 4) at 100 °C .

8 Evaluation of tensile stress-strain properties of vulcanized test mixes

WARNING — Formation of nitrosamines is possible during the cure.

Vulcanize sheets at 160 °C for 30 min.

Condition the vulcanized sheets for 16 h to 96 h at a standard laboratory temperature and, if possible, at a standard laboratory humidity as defined in ISO 23529.

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

9 Evaluation of Shore hardness of vulcanized test mixes

Measure the Shore hardness using durometers with the A-scale for rubbers in the normal hardness range in accordance with ISO 7619-1.

10 Test report

The test report shall include the following:

- a) a reference to this Technical Specification (i.e. ISO/TS 16096);
- b) all details necessary for the identification of the sample;
- c) the method used for the ash determination (method A or method B of ISO 247:2006);
- d) the method used for the acetone extract determination (method A or method B of ISO 1407:2011);
- e) the method used for the carbon black determination (method A of ISO 1408:1995);

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- f) the reference materials used to prepare the test mix;
- g) the ambient conditions in the laboratory during preparation of the test mix;
- h) the type of curemeter used, the defined time at which M_H was measured and the amplitude of oscillation used for the curemeter test, as defined in [Clause 6](#);
- i) any unusual features noted during the determination;
- j) details of any operation not included in this Technical Specification or in the International Standards to which reference is made, as well as of any operation regarded as optional;
- k) the results and the units in which they have been expressed;
- l) the date of the test.

