

PD ISO/TS 16095:2014



BSI Standards Publication

# Reclaimed rubber derived from products containing mainly natural rubber — Evaluation procedure

**bsi.**

...making excellence a habit.™

**National foreword**

This Published Document is the UK implementation of ISO/TS 16095:2014.

The UK participation in its preparation was entrusted to Technical Committee PRI/50, Rubber - Raw, natural and synthetic, including latex and carbon black.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2014. Published by BSI Standards Limited 2014

ISBN 978 0 580 80801 2

ICS 83.040.10

**Compliance with a British Standard cannot confer immunity from legal obligations.**

This Published Document was published under the authority of the Standards Policy and Strategy Committee on 30 April 2014.

**Amendments issued since publication**

Date	Text affected
------	---------------

---

---

---

**Reclaimed rubber derived from  
products containing mainly natural  
rubber — Evaluation procedure**

*Caoutchouc régénéré dérivé principalement de produits contenant du  
caoutchouc naturel — Méthode d'évaluation*





**COPYRIGHT PROTECTED DOCUMENT**

© ISO 2014

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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](mailto:copyright@iso.org)  
Web [www.iso.org](http://www.iso.org)

Published in Switzerland

# Contents

Page

<b>Foreword</b> .....	<b>iv</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Sampling and sample preparation</b> .....	<b>1</b>
<b>4 Physical and chemical tests on raw rubber</b> .....	<b>2</b>
4.1 Mooney viscosity.....	2
4.2 Acetone extract.....	2
4.3 Ash.....	2
4.4 Carbon black.....	2
4.5 Rubber content.....	2
<b>5 Preparation of test mix for evaluation</b> .....	<b>2</b>
5.1 Standard test formulation.....	2
5.2 Mixing procedure — Mixing with a laboratory mill.....	3
<b>6 Evaluation of vulcanization characteristics by a curemeter test</b> .....	<b>4</b>
6.1 Using an oscillating disc curemeter.....	4
6.2 Using a rotorless curemeter.....	5
<b>7 Evaluation of Mooney viscosity of test mixes</b> .....	<b>5</b>
<b>8 Evaluation of tensile stress-strain properties of vulcanized test mixes</b> .....	<b>5</b>
<b>9 Evaluation of Shore hardness of vulcanized test mixes</b> .....	<b>5</b>
<b>10 Test report</b> .....	<b>6</b>

## 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](http://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](http://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 interlaboratory test procedure (ITP) to validate the evaluation procedure on reclaimed natural 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 rubber derived from products containing mainly natural rubber — Evaluation procedure

## 1 Scope

This Technical Specification defines

- the physical and chemical tests on raw reclaimed natural rubber, and
- the standard materials, standard test formulations, equipment, and processing methods for evaluating the vulcanization characteristics, and the mechanical properties of reclaimed natural 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 method A 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.

$$R = 100 - (a + b + c) \tag{1}$$

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 natural 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 natural rubber	100,00 + x + y + z
Stearic acid <sup>a</sup>	2,00
Zinc oxide <sup>a</sup>	5,00
Sulfur <sup>a</sup>	3,00
Mercaptobenzothiazole (MBT)	0,50
Diphenyl guanidine (DPG)	0,20
<b>Total</b>	<b>110,70 + x + y + z</b>
NOTE 1 x is the number of parts of carbon black to 100 parts of rubber in reclaimed natural rubber.	
NOTE 2 y is the number of parts of acetone extract to 100 parts of rubber in reclaimed natural rubber.	
NOTE 3 z is the number of parts of ash to 100 parts of rubber in reclaimed natural rubber.	
<sup>a</sup> 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 g, shall be enough to form proper rolling band. The surface temperature of the rolls shall be maintained at 40 °C ± 5 °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 might be necessary.

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

- a) Weigh accurately natural reclaimed rubber to the nearest 1 mg.
- b) Weigh accurately activators and curatives, to the nearest 0,02 mg, according to the test recipe given above in proportion to the amount of reclaim used.

	Duration min	Cumulative time min
c) Band the reclaimed rubber with the mill opening set at 1,2 mm.	1,0	1,0
d) Add the zinc oxide and stearic acid across the mill rolls at a uniform rate. When all the zinc oxide and stearic acid have 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 mill-ing surface. Be certain to return to the batch any materials that drop through the mill.	1,0	2,0
e) 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
f) Add the DPG 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
g) 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

h) Cut the batch from the mill and take off the mill rolls.	2,0	7,0
i) Set the mill opening to 1,2 mm and pass the rolled batch endwise through the mill six times.	3,0	10,0
Total time	10,0	

- j) 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.
- k) Remove sufficient material for curemeter testing.
- l) Sheet the batch in grain direction 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.
- m) 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. Mould the slab at 140 °C for 20 min. For buttons over 6 mm, 2 min extra time can be added.

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 might not be possible. In such case, scraper blade as shown in [Figure 1](#) can be used. After scraping the material each time, it should be folded two times and passed through mill again to ensure proper blending of chemical.



**Key**

a scraper blade

**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,  $t_{s1}$ ,  $t'_c(50)$ ,  $t'_c(90)$ , and  $t'_c(95)$

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: trials to be done at 140 °C ± 0,3 °C;
- pre-heat time: none.

## 6.2 Using a rotorless curemeter

Measure the following standard test parameters:

$F_L$ ,  $F_{max}$  at defined time,  $t_{s1}$ ,  $t'_c(50)$ ,  $t'_c(90)$ , and  $t'_c(95)$

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

- oscillation frequency: 1,7 Hz (100 cycles per minute);
- amplitude of oscillation: 0,5° 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: 140 °C ± 0,3 °C;
- pre-heat time: none.

The two types of curemeter might not 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 140 °C for 20 min.

Condition the vulcanized sheets/buttons 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 16095:2014);
- b) all details necessary for the identification of the sample;
- c) the method used for the ash determination (method A 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);
- 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.







# British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

## About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

## Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at [bsigroup.com/standards](http://bsigroup.com/standards) or contacting our Customer Services team or Knowledge Centre.

## Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at [bsigroup.com/shop](http://bsigroup.com/shop), where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

## Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to [bsigroup.com/subscriptions](http://bsigroup.com/subscriptions).

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

**PLUS** is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit [bsigroup.com/shop](http://bsigroup.com/shop).

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email [bsmusales@bsigroup.com](mailto:bsmusales@bsigroup.com).

## BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

## Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

## Copyright

All the data, software and documentation set out in all British Standards and other BSI publications are the property of and copyrighted by BSI, or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. Details and advice can be obtained from the Copyright & Licensing Department.

## Useful Contacts:

### Customer Services

**Tel:** +44 845 086 9001

**Email (orders):** [orders@bsigroup.com](mailto:orders@bsigroup.com)

**Email (enquiries):** [cservices@bsigroup.com](mailto:cservices@bsigroup.com)

### Subscriptions

**Tel:** +44 845 086 9001

**Email:** [subscriptions@bsigroup.com](mailto:subscriptions@bsigroup.com)

### Knowledge Centre

**Tel:** +44 20 8996 7004

**Email:** [knowledgecentre@bsigroup.com](mailto:knowledgecentre@bsigroup.com)

### Copyright & Licensing

**Tel:** +44 20 8996 7070

**Email:** [copyright@bsigroup.com](mailto:copyright@bsigroup.com)



...making excellence a habit.™