

BS ISO 18766:2014



BSI Standards Publication

# Rubber, vulcanized or thermoplastic — Low temperature testing — General introduction and guide

**bsi.**

...making excellence a habit.™

**National foreword**

This British Standard is the UK implementation of ISO 18766:2014.

The UK participation in its preparation was entrusted to Technical Committee PRI/22, Physical testing of rubber.

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 85096 7

ICS 83.060

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

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 July 2014.

**Amendments issued since publication**

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

---

INTERNATIONAL  
STANDARD

**ISO**  
**18766**

First edition  
2014-07-15

---

---

**Rubber, vulcanized or  
thermoplastic — Low temperature  
testing — General introduction and  
guide**

*Caoutchouc vulcanisé ou thermoplastique — Essais à basse  
température — Introduction générale et lignes directrices*



Reference number  
ISO 18766:2014(E)

© ISO 2014



**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 Terms and definitions</b> .....	<b>1</b>
<b>3 Types of low temperature test</b> .....	<b>1</b>
3.1 General.....	1
3.2 Change in stiffness.....	1
3.3 Brittleness point.....	2
3.4 Recovery tests.....	2
3.5 Crystallization.....	2
<b>4 Significance of low temperature tests</b> .....	<b>3</b>
<b>5 Comparison of tests</b> .....	<b>3</b>
<b>Bibliography</b> .....	<b>4</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 2, *Testing and analysis*.

# Rubber, vulcanized or thermoplastic — Low temperature testing — General introduction and guide

## 1 Scope

This International Standard provides a general introduction to, and guidance on, the methods of test for low temperature properties of vulcanized and thermoplastic rubbers.

It is intended to provide an understanding of the significance of the various low temperature properties and to assist in the selection of an appropriate test method.

## 2 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 2.1

#### low temperature test

test to measure any property at a temperature below standard laboratory temperature

## 3 Types of low temperature test

### 3.1 General

With reduction of temperature, rubbers become stiffer, until finally becoming hard and brittle, and also recovery from an applied deformation becomes more sluggish. The point of becoming hard and brittle is the glass transition. Any physical test can be made at sub-normal temperatures and, for particular purposes, it might be desirable to follow changes in, for example, tensile strength, dynamic modulus, resilience, or electrical resistivity as the temperature is lowered, depending on what is relevant to service. The glass transition temperature ( $T_g$ ) is most commonly determined from differential scanning calorimetry (DSC) or dynamic thermal mechanical analysis (DTMA) testing. In this respect, it should be noted that the measured  $T_g$  will depend on the frequency of the test used, a “fast” test yielding a higher  $T_g$  than a “slow” one. Further information can be found in Reference [6].

Largely for practical convenience, a number of specific low temperature test procedures have evolved for measuring these general trends in behaviour and have been widely standardized.

These low temperature tests can be grouped as follows:

- change in stiffness;
- brittleness point;
- rate of recovery (set and retraction).

In addition, some rubbers, for example natural rubber and polychloroprene, stiffen at low temperatures by partial crystallization. This is a gradual process continuing over many days or weeks and is most rapid at a particular temperature characteristic of each polymer, for example  $-25\text{ °C}$  for natural rubber. Hence, tests intended to measure the effect of crystallization have to detect changes in stiffness or recovery after periods of “ageing” at a low temperature.

### 3.2 Change in stiffness

Historically, before the introduction of thermal analysers, it was relatively difficult and expensive to measure tensile or compressive modulus at sub-ambient temperatures. Torsion tests, although rarely

used to measure stiffness at ambient temperature, were found to be very convenient for measuring change of stiffness as temperature is lowered. The system standardized for rubber as the Gehman test in ISO 1432 uses a torsion wire to provide the torque to twist a strip test piece. The stiffness is measured as a function of rising temperature. The measurements may be made in a liquid or gaseous environment, either at intervals or with continuous rise of temperature. In principle, these alternatives yield equivalent results.

The normal expression of results is to give the temperatures at which the relative modulus (calculated with respect to that at 23 °C) is 2, 5, 10, and 100. If required, the apparent torsional modulus at any temperature may be calculated but, because it depends on the test conditions, should not be taken as an absolute value.

With modern universal test machines with environmental cabinets and, in particular, dynamic thermal mechanical analysers, it can be argued that essentially the same data could be better obtained using the more commonly used modes of deformation, and which would match data obtained at ambient and elevated temperatures. However, the traditional ISO 1432 method continues to be widely used.

### 3.3 Brittleness point

A somewhat arbitrary method for determining the point at which the material becomes brittle is described in ISO 812. It operates on the basis of striking a test piece in the form of a simple cantilever with the striker moving at 2 m/s. The result is either pass or fail, failure being any crack or complete separation of the test piece. Various forms of apparatus are possible and the test may be conducted in a liquid or gaseous medium but, in principle, the results are equivalent.

Two procedures are specified: determination of brittleness temperature and determination of 50 % brittleness temperature. The former gives the lowest temperature at which there are no failures, while the latter finds the temperature at which 50 % of test pieces fail. It has been suggested that the 50 % method is more reproducible but might use more test pieces. For specification purposes, the test may be used as a go/no go procedure at one given temperature.

### 3.4 Recovery tests

The most straightforward way to measure recovery from a deformation at a low temperature would be to use an adaptation of the normal compression set or tension set tests. A test in compression is given in ISO 815-2. Essentially, it is the usual compression set procedure but with the deformed test piece being released and the recovery taking place at the specified low test temperature.

Although simple in principle, the test is practically rather difficult because of the need to unload the test piece and measure its recovery within a low temperature enclosure. Variability can be large and the test is best made with specially designed apparatus.

Traditionally, an alternative approach based on recovery in tension and known as the low temperature retraction test (TR test) has been widely used and is described in ISO 2921. The stretched test pieces are put into the heat transfer medium at a low temperature below the glass transition temperature. The test pieces are then allowed to recover as the temperature is raised. From a graph of percentage retraction against temperature, the temperatures corresponding to 10 %, 30 %, 50 %, and 70 % retraction are noted. This way of expressing the results is similar in principle to the results of the Gehman test for stiffness.

### 3.5 Crystallization

In principle, any of the low temperature tests can be used to study crystallization effects by holding the test piece at the low temperature for much longer than in the standard tests. A clause suggesting that this can be done is included in, for example, the TR test standard. However, a procedure based on hardness measurement has been standardized as ISO 3387. The hardness of a test piece is measured soon after being placed in the low temperature cabinet and after it has been held at the low temperature for a period of time, usually 24 h and 168 h. This procedure has a similar practical difficulty, of making a measurement at low temperature inside an enclosure, as for determining low temperature compression



set. One reason for introducing the hardness procedure was because it can be used with unvulcanized compound as well as vulcanized rubber. This would not be feasible with the other standard low temperature tests.

#### **4 Significance of low temperature tests**

None of the standard low temperature tests yields absolute values, as the results depend on the particular test conditions. Different conditions can give different results. Consequently, the results can be used as guidance as to performance at low temperature but might not precisely equate to the performance of a particular product. However, in the Gehman and TR tests, the results are essentially a measure of the change in a property, which is less dependent on the conditions.

It is obvious that the different tests measure different aspects of low temperature behaviour and are, hence, complementary. Which test is preferable in a given situation will depend on which aspect or aspects are relevant to service. For example, if remaining ductile is the only consideration then the brittleness test is appropriate. If recovery from deformation, as in a seal, is important then set or TR tests are more relevant. As mentioned earlier, a property other than those in the standardized low temperature tests might be the most appropriate for a particular circumstance.

#### **5 Comparison of tests**

Notwithstanding the fact that the various tests measure different aspects of behaviour, the question is often asked as to how the results compare. Published reports on comparisons of methods are discussed in Reference [6]. The indications are that the ranking of compounds is not always the same for all the tests but reasonable correlations can be expected between similar measures (hardness and Gehman modulus) or between measures at the same degree of stiffening [for example brittleness and TR10 or T10 (Gehman)].

## Bibliography

- [1] ISO 812, *Rubber, vulcanized or thermoplastic — Determination of low-temperature brittleness*
- [2] ISO 815-2, *Rubber, vulcanized or thermoplastic — Determination of compression set — Part 2: At low temperatures*
- [3] ISO 1432, *Rubber, vulcanized or thermoplastic — Determination of low-temperature stiffening (Gehman test)*
- [4] ISO 2921, *Rubber, vulcanized — Determination of low-temperature retraction (TR test)*
- [5] ISO 3387, *Rubber — Determination of crystallization effects by hardness measurements*
- [6] BROWN R.P. *Physical Testing of Rubber*. Springer, 2006







# 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.™