

BS ISO 11346:2014



BSI Standards Publication

# Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use

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**National foreword**

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**Rubber, vulcanized or thermoplastic —  
Estimation of life-time and maximum  
temperature of use**

*Caoutchouc vulcanisé ou thermoplastique — Estimation de la durée  
de vie et de la température maximale d'utilisation*





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<b>Contents</b>		Page
<b>Foreword</b> .....		<b>iv</b>
<b>Introduction</b> .....		<b>v</b>
<b>1 Scope</b> .....		<b>1</b>
<b>2 Normative references</b> .....		<b>1</b>
<b>3 Terms and definitions</b> .....		<b>1</b>
<b>4 Principle</b> .....		<b>1</b>
<b>5 Selection of tests and ageing oven</b> .....		<b>2</b>
<b>6 Selection of threshold value</b> .....		<b>2</b>
<b>7 Test pieces</b> .....		<b>2</b>
<b>8 Exposure temperatures</b> .....		<b>3</b>
<b>9 Exposure times</b> .....		<b>3</b>
<b>10 Procedure</b> .....		<b>3</b>
<b>11 Expression of results</b> .....		<b>4</b>
11.1 Arrhenius procedure.....		<b>4</b>
11.2 WLF procedure.....		<b>6</b>
11.3 Limitations.....		<b>8</b>
<b>12 Test report</b> .....		<b>8</b>
<b>Bibliography</b> .....		<b>10</b>

## Foreword

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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)).

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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*.

This third edition cancels and replaces the second edition (ISO 11346:2004), of which it constitutes a minor revision.

## Introduction

The rate of a chemical reaction normally increases with increase in temperature. By exposing test pieces to a series of elevated temperatures, the relation between the reaction rate of degradative mechanisms and temperature can be deduced. Estimates can then be made by extrapolation of the degree of degradation after a given time at a given temperature or the time at a given temperature to reach a given degree of degradation.

The reaction rate/temperature relationship can often be represented by the Arrhenius equation. The reaction rate at any given temperature is obtained from the change in the value of a selected property with exposure time at that temperature. The reaction rate can be represented by the time to a particular degree of degradation (threshold value) and this might be the only measure to use if the property/temperature relation is complex.

The Arrhenius approach is only suitable for chemical degradation reactions and might give wrong results for tests where physical (viscoelastic) changes cannot easily be separated from chemical changes.

An alternative approach for rubbers is to use the Williams Landel Ferry (WLF) equation. This equation performs a time/temperature transformation and no assumptions are made as to the form of the property/time relation at any temperature. Hence, in principle, it can be applied to any physical property, including set and relaxation, or where the property/time relation is complex. Further explanation of the use of the WLF equation can be found in the literature.<sup>[1]</sup>

During the preparation of this International Standard, account was taken of the contents of ISO 2578<sup>[2]</sup> and IEC 60216.<sup>[3]</sup>





# Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use

## 1 Scope

This International Standard specifies the principles and procedures for estimating the thermal endurance of rubbers from the results of exposure to elevated temperatures for long periods.

Two approaches are specified (see Introduction):

- one using the Arrhenius relation;
- the other using the WLF equation.

In this International Standard, the estimation of thermal endurance is based solely on the change in selected properties resulting from periods of exposure to elevated temperatures. The various properties of rubbers change at different rates on thermal ageing, hence comparisons between different rubbers can only be made using the same properties.

## 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 188, *Rubber, vulcanized or thermoplastic — Accelerated ageing and heat resistance tests*

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

## 3 Terms and definitions

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

### 3.1

#### **life-time**

time at which the material under test has reached the specified threshold value for the property tested at the temperature of use

### 3.2

#### **maximum temperature of use**

temperature at which the material under test has reached the specified threshold value for the property tested after the specified time

### 3.3

#### **threshold value**

particular degree of degradation which is taken as the maximum acceptable for the property being tested

Note 1 to entry: The time to reach the threshold value can be used to represent the reaction rate

## 4 Principle

At a chosen test temperature, the variations in the numerical value of a chosen property, for example a mechanical or viscoelastic property, are determined as a function of time.

The testing is continued until the relevant threshold value of that property has been exceeded.

Further tests are carried out at least two other temperatures.

For the Arrhenius procedure, the measures of the reaction rates obtained are plotted logarithmically as a function of the reciprocal of temperature and the straight line obtained is extrapolated back or interpolated to the temperature of use.

For the WLF procedure, the shift constants are calculated and used to transpose the property/time relation to the temperature of use.

## 5 Selection of tests and ageing oven

The tests chosen should preferably relate to properties which are likely to be of significance in practice. Wherever possible, use shall be made of methods of test specified in International Standards.

For general evaluations, hardness, and tensile stress-strain properties are commonly used, but stress relaxation or set in compression, for example, is desirable for sealing applications.

For the ageing of test pieces, a circulating-air oven meeting the requirements of ISO 188 shall be used.

## 6 Selection of threshold value

The threshold value shall be chosen as the degree of degradation that is the maximum acceptable for the property being tested and the end use. Commonly, 50 % of the initial value of the property is chosen.

The test should normally be continued for a long enough period for the threshold value to be reached. Although it is possible to extrapolate to greater degrees of degradation, this is not recommended.

## 7 Test pieces

**7.1** The dimensions and method of preparation of the test pieces shall be in accordance with the relevant test method standard.

**7.2** The minimum total number,  $n$ , of test pieces required for a destructive test method depends on:

- the number of test pieces,  $a$ , required for a single test in accordance with the test method standard;
- the number of different ageing periods,  $b$ , necessary to obtain the property/time relationship at any one exposure temperature;
- the number of exposure temperatures,  $c$ .

The minimum number of test pieces required is given in Formula (1):

$$n = abc + a \quad (1)$$

It is recommended that additional test pieces are aged at each temperature in case problems occur after several weeks, months or years of ageing, or an extra exposure temperature is required to improve precision.

The minimum number of test pieces required for a non-destructive test method is normally given in Formula (2):

$$n = ac \quad (2)$$

When measuring compression set, tension set, and relaxation, the tests are preferably done on the same test pieces, at the different times, to reduce the number of test pieces needed. This also reduces variations in the test results.

It might be necessary, in addition, to carry out trial runs to determine the exposure temperatures and the number of test points required at each temperature.

NOTE While it is possible to reduce the number of test pieces for each test from that given in the test method standard to economize on testing, extrapolation of the results requires the best possible precision and in some circumstances it might be advisable to increase the number of test pieces.

## 8 Exposure temperatures

Selection of the exposure temperatures involves knowing beforehand the approximate ageing characteristics of the material under test. With no previous knowledge of the material, exploratory tests will have to be carried out. This information will assist in selecting the exposure temperatures best suited for the evaluation of the material.

Test pieces shall be aged at not fewer than three temperatures covering a range adequate to establish the life-time estimation by extrapolation with the required degree of accuracy. The lowest exposure temperature shall be chosen so that the time taken to reach the threshold value is at least 1 000 h. Likewise, the highest temperature shall be chosen so that the time taken to reach the threshold value is not shorter than 100 h. The temperatures used should preferably be standard test temperatures taken from ISO 23529.

NOTE To obtain an estimate of the life-time with adequate precision will often require more than three test temperatures.

## 9 Exposure times

The properties chosen to measure the reaction rate shall be tested after each of at least five different exposure times at each temperature, but more exposure times will normally be needed if the shape of the property/time curve is to be established.

The exposure times shall be such as to enable the property chosen to measure the reaction rate to be characterized adequately. For thermo-oxidative ageing, a linear progression will be satisfactory in many cases. For physical relaxation, a logarithmic progression would be more appropriate. The requirements concerning minimum exposure times in [Clause 8](#) shall be respected.

## 10 Procedure

Measure the selected properties using unaged sets of test pieces conditioned as required by the relevant test method standards.

Place the required number of test pieces in each of the ovens maintained at the selected temperatures.

At the end of each exposure time, condition the test pieces to be examined as required by the relevant test method standard and measure the selected properties.

Continue this procedure until the required number of sets of test pieces have been tested.

## 11 Expression of results

### 11.1 Arrhenius procedure

The Arrhenius relation can be written in Formula (3):

$$\ln K(T) = B - \frac{E}{RT} \quad (3)$$

where

$K(T)$  is the reaction rate ( $\text{min}^{-1}$ );

$B$  is a constant;

$E$  is the activation energy ( $\text{J/mol}$ );

$R$  is the gas constant ( $8,314 \text{ J/mol K}$ );

$T$  is the absolute temperature ( $\text{K}$ ).

The stage the reaction has reached is given by the relation in Formula (4):

$$F_x(t) = K(T) \times t \quad (4)$$

where

$F_x(t)$  is a function describing the stage,  $x$ , the reaction has reached;

$t$  is the reaction time ( $\text{min}$ ).

There will be different reaction rates,  $K(T)$ , corresponding to different temperatures,  $T$ .

For each exposure temperature, plot the results for each property against time. For each plot, determine the reaction rate.

Often, a convenient measure of the reaction rate is the time for the property to reach the threshold value, determined by interpolation as illustrated in [Figure 1](#).

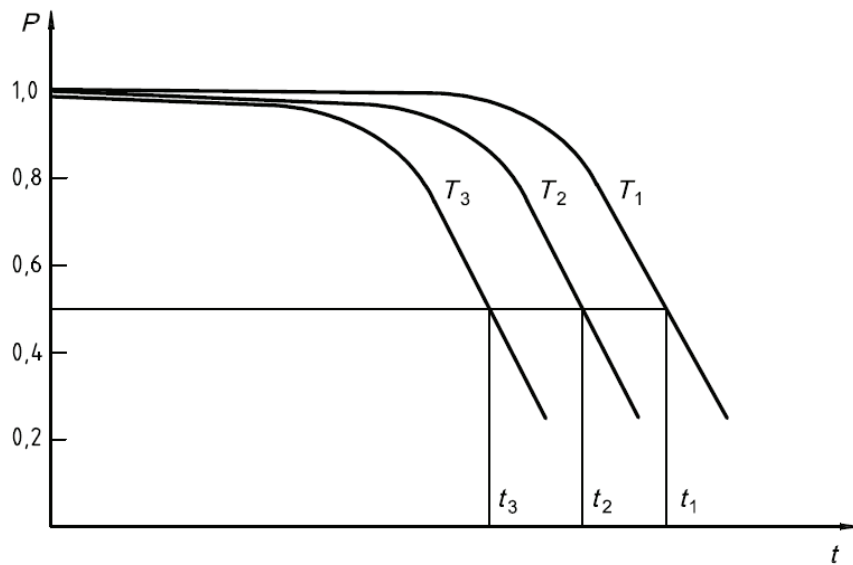
**NOTE** The ideal measure of the reaction rate would be the slope of the property-time plot, but this is rarely linear [i.e.  $F_x(t)$  is rarely linear]. It might be possible to fit a convenient relationship to the data or to obtain a convenient plot by plotting on a logarithmic scale.

Plot the logarithm of the reaction rate (e.g. time to threshold value) against the reciprocal of the absolute temperature and construct a best-fit straight line through the points by established statistical methods. An example is given in [Figure 2](#). If the data deviates from a straight line, this indicates that different reactions are taking place at the different temperatures and extrapolation of the data are invalid.

**The life-time** at a given temperature or service temperature ( $25 \text{ }^\circ\text{C}$  in [Figure 2](#)) is estimated by extrapolation of the line to that temperature. The reaction rate at that temperature will give the time to reach the threshold value.

**The maximum temperature of use** is estimated by extrapolation of the line to a specified reaction rate or time to reach the threshold value. A time of 20 000 h is commonly used when establishing a general maximum temperature of use.

**The activation energy** is obtained by dividing the slope of the line by  $R$ , the gas constant.



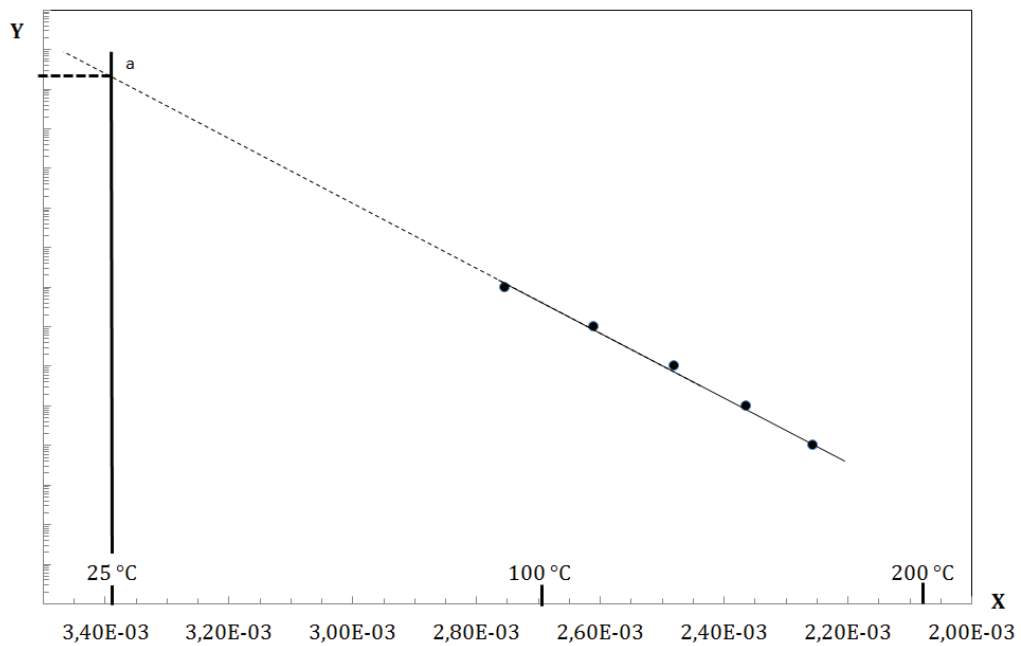
**Key**

$P$  value of property (as a fraction of initial value)

$t$  time

$T$  temperature

**Figure 1 — Change in property against time**



**Key**

$X$   $\frac{1}{T} \times 10^4$  ( $T$  in kelvins)

$Y$  log of reaction rate (time),  $t$

$a$  life time at 25 °C

**Figure 2 — Arrhenius plot (time against temperature)**

## 11.2 WLF procedure

The WLF equation that is generally used in Formula (5):

$$\log a_T = \frac{-a(T - T_0)}{b + (T - T_0)} \quad (5)$$

where

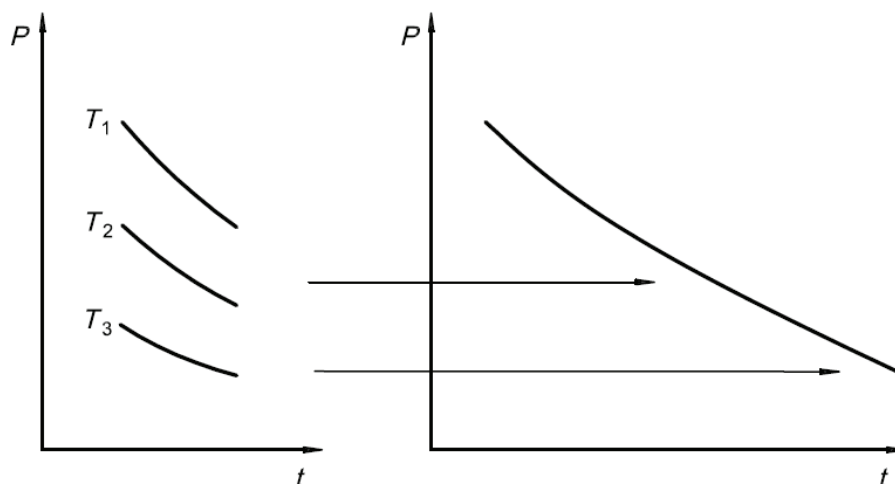
- $a_T$  is a so-called shift factor (see below);
- $a$  and  $b$  are constants which depend only on the material;
- $T_0$  is the reference temperature used to create the shift values.

For each exposure temperature, plot the results for each property as a function of time. Typically, a  $\log(\text{time})$   $x$ -axis is used.

Taking the reference temperature as fixed, slide the lines corresponding to each of the other temperatures in turn horizontally in the  $x$ -direction until the best possible overlap with the line at the reference temperature is obtained (see [Figure 3](#)). In this way, a “master curve” is constructed, at the reference temperature, which simulates how the material would behave over a much wider time scale than can be investigated by direct experimentation. The amount by which each line at a non-reference temperature is moved (movement in the positive direction is movement towards longer times and movement in the negative direction is movement towards shorter times) is the shift factor,  $a_T$  (or, if the  $x$ -axis is a logarithmic scale, the log of the shift factor). By definition, when  $T = T_0$ ,  $\log a_T = 0$ , and there is no shift.

Plot the value of  $\log a_T$  for each temperature against the corresponding temperature as shown in [Figure 4](#) (sometimes absolute temperature is used, although mathematically this is unnecessary since the temperatures are in fact temperature differences).

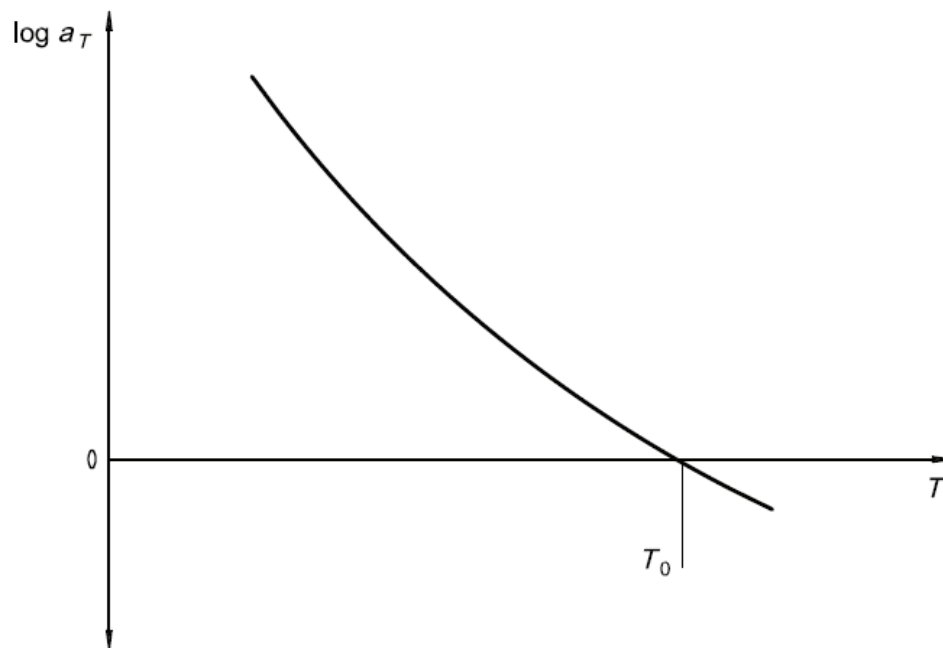
Use standard curve-fitting techniques to determine the best fit for the WLF equation to give values of the constants  $a$  and  $b$ .



### Key

- $P$  value of property
- $t$  time
- $T$  temperature

**Figure 3 — Construction of a “master curve”**



**Key**

$a_T$  shift factor

$T$  temperature

$T_0$  reference temperature

**Figure 4 — Shift factors plotted against temperature**

Alternatively, in the absence of curve-fitting software, the equation can be rewritten in the form of a straight line and then the same linear regression method as for the Arrhenius procedure used to find the coefficients. The straight line is given in Formula (6):

$$u = -rv + t \tag{6}$$

where

$$u = \frac{1}{\log a_T} \tag{7}$$

$$v = \frac{1}{T - T_0} \quad (8)$$

Having found the coefficients  $r$  and  $t$ , the constants in the WLF equation can be found in Formulae (9) and (10):

$$a = -\frac{1}{t} \quad (9)$$

and

$$b = -\frac{r}{t} \quad (10)$$

To obtain an estimate of the lifetime, use the WLF equation to determine the shift factor from the reference temperature to the temperature of interest. Apply that shift factor to each of the points on the master curve to obtain the required property-time curve and read the time to reach the threshold value.

To obtain an estimate of the maximum temperature of use, extrapolate the line to a specified reaction rate or time to reach the threshold value. A time of 20 000 h is commonly used when establishing a general maximum temperature of use.

### 11.3 Limitations

Although, in principle, the extrapolation can be made over a large temperature range and hence to extremely long times, consideration will have to be given to the increase in uncertainty inherent in extrapolation to long times and the possibility that the chemical reaction which takes place at high temperatures is gradually replaced by a different reaction at lower temperatures, especially where both scission and crosslinking reactions take place. Because of these considerations, extrapolations are generally limited to 30 °C to 40 °C beyond the last data point.

It is recommended that an estimate of the uncertainty of the results is made.

Caution should also be used when the results are analysed, because thermo-oxidative ageing is diffusion controlled and thus different results can be obtained when comparing thin and thick test pieces. The test conditions in the laboratory might also differ from service conditions, under which other causes of deterioration, such as light ageing and ozone attack, can be involved.

## 12 Test report

The test report shall include the following information:

a) sample details:

- 1) complete identification of the material tested,
- 2) the dimensions and method of preparation of the test pieces, with reference to the relevant International Standard,
- 3) the property selected, with reference to the relevant International Standard,
- 4) the threshold value of the property selected,
- 5) the test piece conditioning temperature and time;

b) test method:

- 1) a full reference to the test method used, that is reference to this International Standard (i.e. ISO 11346),



- 2) the type(s) of oven used, including details of the air-exchange rate and air speed,
  - 3) the exposure times and temperatures in the ovens,
  - 4) the numbers of test pieces used,
  - 5) the details of any procedures not specified in this standard;
- c) test results:
- 1) the graphs plotted as specified in [11.1](#) or [11.2](#),
  - 2) the predicted life-time at a given temperature, together with the temperature of use, or the maximum temperature of use at a given time;
- d) dates of the test.

## Bibliography

- [1] BROWN R.P. Practical Guide to the Assessment of the Useful Life of Elastomers, Rapra Technology Ltd., 2001
- [2] ISO 2578:1993, *Plastics — Determination of time-temperature limits after prolonged exposure to heat*
- [3] IEC 60216 (all parts), *Electrical insulating materials — Thermal endurance properties*







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