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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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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 document 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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 11346:2014), which has been technically revised.

The main changes are as follows:

— the accuracy via the use of a calculation method has been improved;

— the coefficient of determination and definition of a minimum value, which leads to significant improvement of regression curve accuracy and allows extrapolation to longer time periods has been introduced;

— the accuracy of test parameters has been increased;

— the formula to calculate the activation energy has been corrected;

— the threshold value (compression set) for seals has been introduced;

— different time-temperature collectives closer to real-world conditions have been introduced.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The rate of a chemical reaction normally increases with increasing temperature. By exposing test pieces to a series of elevated temperatures, the relation between the rate of degradative mechanisms and temperature can be deduced. Estimates can then be made by extrapolation, for a given temperature, of the degree of degradation after a given time or the time required 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 can be the only practical measure if the property-temperature relation is complex.

The Arrhenius approach is only suitable for chemical degradation reactions and can give incorrect 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 Reference [1].

NOTE The term equation is used for the relationships referred to here as formula.

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

1 Scope

This document 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 equation;

— the other using the WLF equation.

In this document, 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 comparison between different rubbers can only be made using the same properties.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. 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*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
life-time
time at which the material under test has reached the specified *threshold value* (3.4) for the property tested at the temperature of use or a time-temperature collective (respective to climate for outside application) closest to reality

3.1.1
life-time at a given temperature
life-time at a given service temperature (e.g. 25 °C)
time obtained by extrapolation of the line to that temperature

3.1.2
life-time at a given time-temperature collective
life-time at a given temperature (3.1.1) respectively at reference temperature divided by the *ageing factor* (3.2)

3.2
ageing factor
factor calculated using a time-temperature collective over one year that has been converted to a reference temperature

3.3
maximum temperature of use
temperature at which the material under test has reached the specified *threshold value* (3.4) for the property tested after the specified time

3.4
threshold value
specific 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 (the inverse of the reaction rate is proportional to the time to reach the threshold value).

4 Principle

The basic procedure to estimate the life-time and maximum temperature of use of rubbers consists of two parts.

a) Testing (see Clauses 5 to 10). Briefly, this involves:

— at a chosen test temperature, the variation in the numerical value of a chosen property, for example, a mechanical or viscoelastic property, is determined as a function of time;

— testing is continued until the relevant threshold value of the property is exceeded, and further measurements are carried out for at least two other temperatures.

b) Plotting of property-time curve and calculation using either the Arrhenius procedure (see 11.1 to 11.1.4) or WLF procedure (see 11.2).

— For the Arrhenius procedure, the measured reaction times are plotted logarithmically as a function of the reciprocal temperature, and the straight line obtained is extrapolated back or interpolated to a constant 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.

To improve comparability of the results every effort should be made to optimize the accuracy criteria. For this purpose, a curve fitting method and the coefficient of determination should be used when possible.

5 Selection of tests and ageing oven

The tests chosen should relate to properties which are likely to be of practical significance.

Test methods that are specified in International Standards shall be used when available.

For general evaluation, the hardness and tensile stress-strain properties are commonly used, while the stress relaxation or compression set are recommended for sealing applications.

For aging of test pieces, one of the described oven types and the corresponding method, which comply with the requirements of the ISO 188, shall be used. Once selected, the oven type and method shall not be changed within the test series.

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 during end use.

If threshold values are mentioned in the relevant product standard, it is recommended that they are used.

NOTE If there is no threshold value specified, 50 % of the initial value of the property is commonly chosen. For static sealing applications, a compression set of maximum 70 % is often chosen.

The test shall be carried out for a period that is sufficiently long to reach the threshold value.

7 Test pieces

7.1 General

The dimensions and method of preparation of the test pieces shall be in accordance with the relevant test method standard. To obtain comparable results, the use of identical test pieces across measurements is recommended.

7.2 Number of test pieces

It can be necessary to carry out trial runs to determine the exposure temperatures and the number of test points required at each temperature. Furthermore, increasing the number of test pieces can be necessary to improve accuracy.

The minimum number of test pieces depends on whether the test method is destructive or non-destructive and can be determined according to the following formulae.

a) For the destructive test method, the minimum number of test pieces, n, required is given by Formula (1):

$$n = abc + a \tag{1}$$

where

a is the number of test pieces required for a single test in accordance with the test method standard;

b is the number of different ageing periods necessary to obtain the property-time relationship at any one exposure temperature;

c is the number of exposure temperatures.

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

b) For the non-destructive test method, the minimum number of test pieces required is given by Formula (2):

$$n = ac \tag{2}$$

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

8 Exposure temperatures

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

Test pieces shall be aged at not fewer than three temperatures. Choose suitable temperatures for the material tested with intervals between 10 °C to 30 °C (depending on the elastomer and temperature range of use).

9 Exposure times

Increasing the exposure time significantly improves the accuracy of the result. Therefore, it is very useful to adjust the exposure time adequately depending on the expected life-time.

For the lowest exposure temperature, it is suggested to use the exposure times as indicated in Table 1.

Table 1 — Exposure times versus expected life-time for the lowest temperature

Expected life-time	Exposure time
> 2 years	> 1 month
> 10 years	> 3 months
> 25 years	> 6 months
> 50 years	> 9 months

The following paragraphs are not valid if stress relaxation with continuous recording is used. For discontinuous tests the properties chosen to measure the reaction rate shall be tested after each of at least six different exposure times for each temperature, however, more exposure times will often be needed when the shape of the property/time curve is to be established. For optimum accuracy, a coefficient of determination of $R^2 \geq 0{,}98$ is a good indicator and should be targeted.

The exposure times shall be such as to enable adequate characterization of the property chosen to measure the reaction rate. For thermo-oxidative ageing, a linear progression will be satisfactory in many cases. For physical relaxation, a logarithmic (e.g. $p = a \cdot \ln(t) + b$) or potential (e.g. $p = a \cdot t^b$) progression would be more appropriate. In this case, the function with the higher coefficient of determination (R^2) shall be used.

10 Test procedure

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

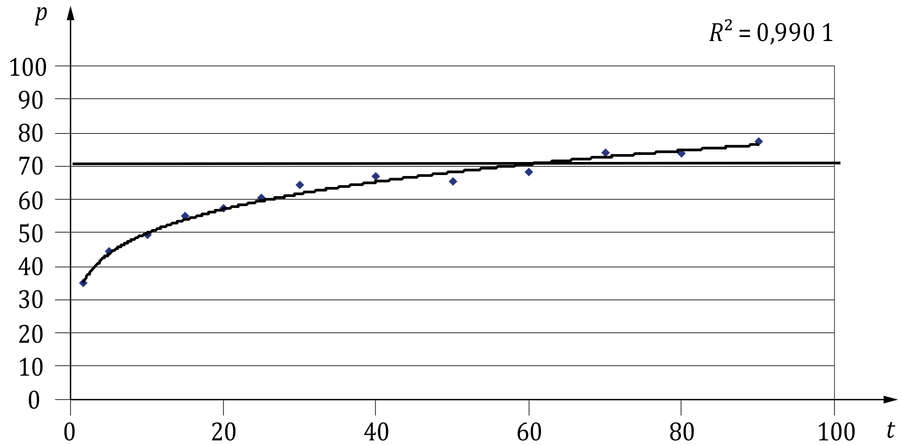
Place the required number of test pieces in each of the ovens maintained at the selected temperatures.

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 threshold values are reached for each temperature. For reasons of time, it is advisable to start with the lowest test temperature. After each testing point, it should be checked whether the prescribed minimum test time according to Table 1 is reached.

For each exposure temperature, plot the results for each property against time. Check if there are any outliers. Outliers are easily identified by a property-time curve displaying both the tested values and the smoothed curve. The use of the coefficient of determination (R^2) is a good tool to find the outliers.

The following paragraph is not valid if stress relaxation with continuous recording is used. An example of the property-time curve (for one temperature) to reach the threshold value with curve fitting is illustrated in Figure 1.



Key

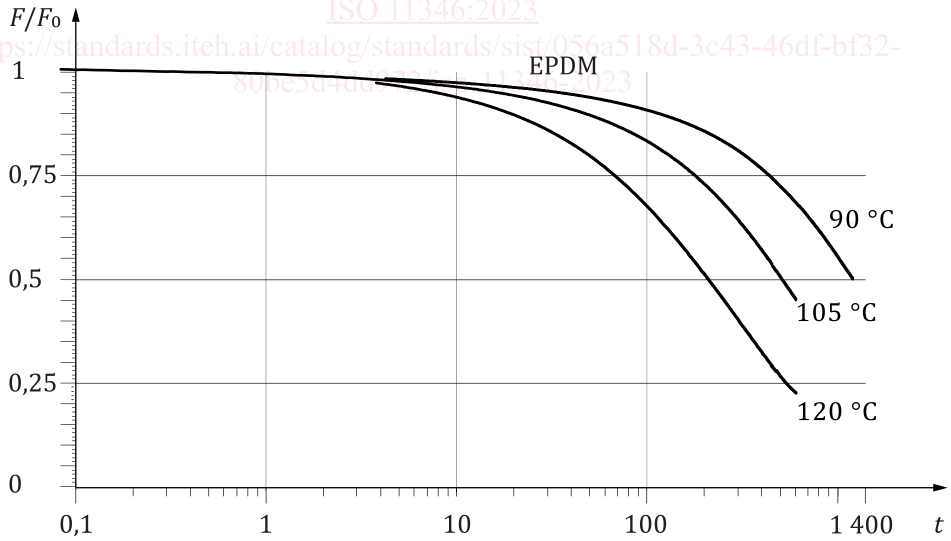
p value of property (deterioration in %)

t time (×100 h)

Figure 1 — Example of property-time curve with threshold value 70 %

NOTE For better understanding, an example is shown in Annex B.

When conducting tests with continuous recording of results, for example, stress relaxation testing, curve fitting is not needed. An example of such a graph is provided in Figure 2.



Key

F/F_0 fraction of initial value

t time (h)

Figure 2 — Example of property-time curve with stress relaxation

