INTERNATIONAL STANDARD

ISO

11346

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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Contents

Foreword

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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 $R^2 ≥ 0,98$ 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.

$R^2 = 0{,}990\ 1$

Key
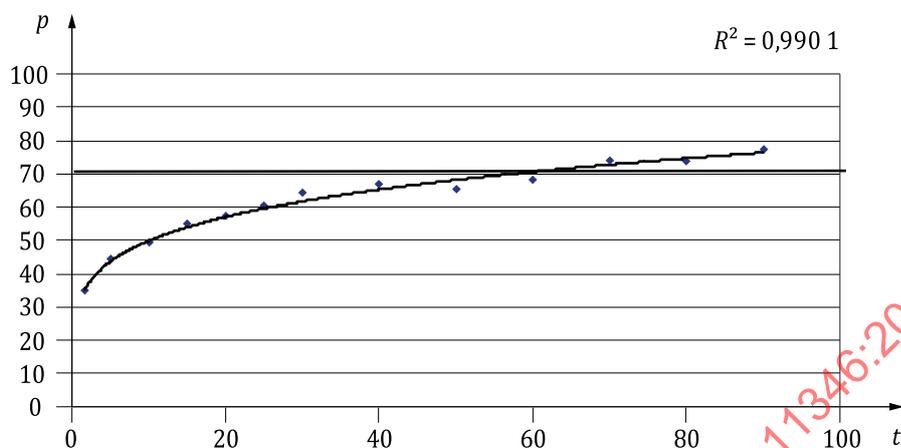
p value of property (deterioration in %)

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.
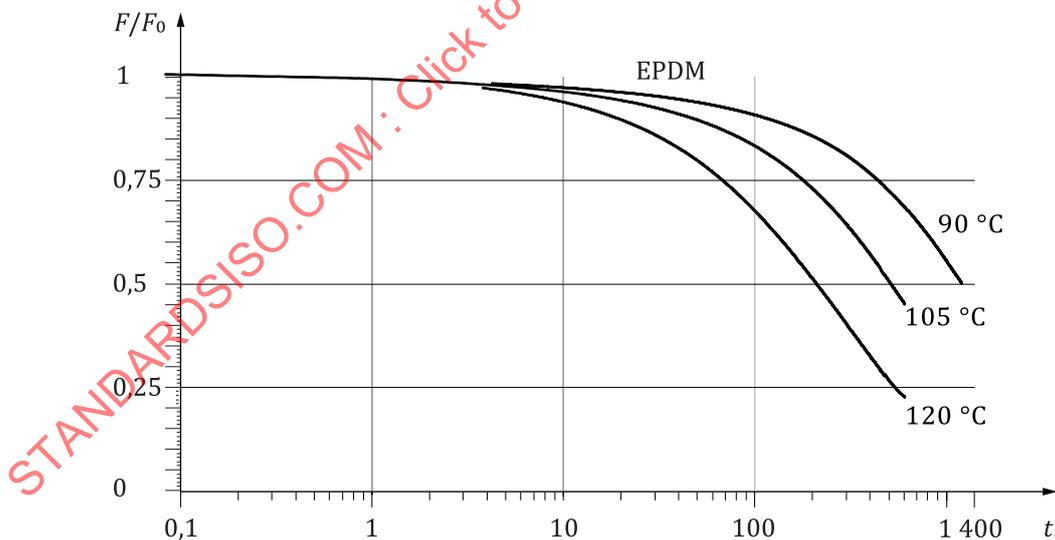
Key

F/F_0 fraction of initial value

t time (h)

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

11 Analysis of results

11.1 Arrhenius procedure

11.1.1 Relevant formulae and relations

The Arrhenius equation can be written as given in Formula (3):

$$\ln K(T) = \ln\left(\frac{1}{t}\right) = -\frac{E_a}{R}\cdot\frac{1}{T} + A \tag{3}$$

where

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

t is the reaction time, in min;

A is a constant;

E_a is the activation energy, in J/mol;

R is the universal gas constant, in 8,314 J/mol K;

T is the reaction temperature, in K.

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

$$F_x(t) = K(T) \times t \tag{4}$$

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

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

A convenient measure of the reaction rate is the time for the property to reach the threshold value, as determined by interpolation (see 11.1.2).

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 multiplying the slope of the line with R, the gas constant.

11.1.2 Preparation of test results and determination of reaction rates

After carrying out the procedure described in Clause 10, the test results shall be processed as follows.

Plot the measured values of the property p (e.g. elongation at break, discontinuous stress relaxation or compression set) over time for each exposure temperature and carry out the best fit.

When doing tests with continuous recording of results, for example stress relaxation testing, curve fitting is not needed.

Applicable curve-fitting functions are: logarithmic [e.g. $p = a \cdot \ln(t) + b$] or potential [e.g. $p = a \cdot t^b$]

where

p is the property selected;

a is the first regression parameter;

b is the second regression parameter.

For each plot, only the function with the higher coefficient of determination (R^2) shall be used. See Figure 3 for an example for the property to reach the threshold value.
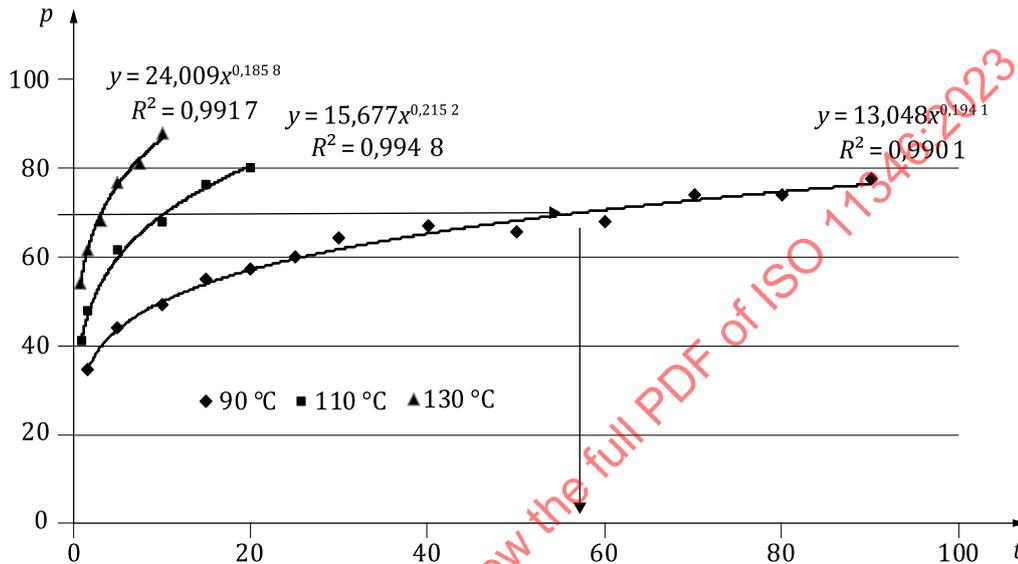

Key

p value of property (deterioration in %)

t time (× 100 h)

Figure 3 — Change of properties against time at three temperatures

The evaluation may be used if the property-time curve at any given temperature achieves the recommended coefficient of determination of $R^2 \geq 0{,}98$. In case of outliers, a decision shall be taken on how to continue the procedure (e.g. repeat testing at this point or increasing the number of testing points).

It is recommended to use a calculation software that can calculate a smoothed curve and display the curve function and coefficient of determination R^2.

For these graphs, extrapolation of the data are not allowed.

The reaction rate (time to threshold value) for the property (in Figure 3, the horizontal arrow line at 70 %) can be determined via the reverse function of the displayed calculated function (in Figure 3, the vertical arrow line) for each testing temperature.

The corresponding reverse function for:

— the logarithmic type ($p = a \cdot ln(t) + b$) is $t = e^{((p-b)/a)}$;

— the potential type ($p = a \cdot t^b$) is $t = \sqrt[b]{(p/a)}$.

Although it is possible to determine the reaction time graphically, for reasons of much higher accuracy, it is strongly recommended to use the calculation method as described above.

7

When using continuous recording of test values, for example, continuous stress relaxation, the time-to-threshold value is taken directly from the curve.

11.1.3 Calculation of life-time at a given temperature

Plot the logarithm of the reaction rate against the reciprocal of the absolute temperature and construct a best-fit straight line through the points. An example is given in Figure 4.

Key
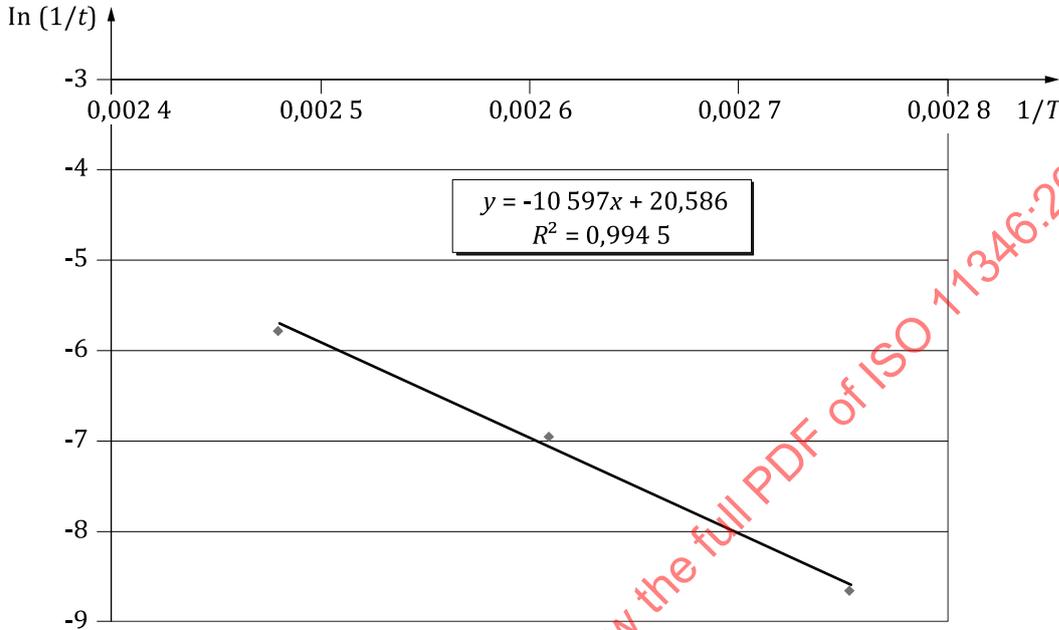
$\ln(1/t)$ ln of reciprocal time (h^{-1})

$1/T$ reciprocal absolute temperature (K^{-1})

Figure 4 — Example of an Arrhenius plot: Time against temperature

For the Arrhenius approach to be applicable, the required $R^2 \geq 0,98$ shall be maintained; if this is not the case, it indicates that different reactions take place at the different temperatures and the data extrapolation is invalid. In this case, the procedure described in 11.1.2 shall be rechecked.

NOTE 1   At this stage a higher level of accuracy is necessary, which is the reason for $R^2 > 0,98$.

The slope of the plot gives E_a/R (−10 597 in Figure 4) and the constant (20,586 in Figure 4) from the fitting curve gives the constant, A, in the Arrhenius equation in Formula (3). With this information, the life-time at a given temperature (or reference temperature) can be calculated.

11.1.4 Calculation of life-time at a given time-temperature collective (optional)

If the expected life-time at a given time-temperature collective is required, the method given in Annex A may be used.

11.2 WLF procedure

The generally used WLF equation is given by Formula (5):

$$\lg a_T = \frac{-a(T - T_0)}{b + (T - T_0)}$$

(5)

where

a_T is a shift factor;

a   is a constant which depend only on the material;

b   is a constant 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 lg(time) x-axis is used.

Taking the reference temperature as fixed, slide the lines corresponding to each of the other temperatures horizontally, in turn, in the x-direction until the best possible overlap with the line at the reference temperature is obtained (see Figure 5). 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 lg of the shift factor). By definition, when $T = T_0$, $\lg a_T = 0$, and there is no shift.

Plot the value of $\lg a_T$ for each temperature against the corresponding temperature, as shown in Figure 6 (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.
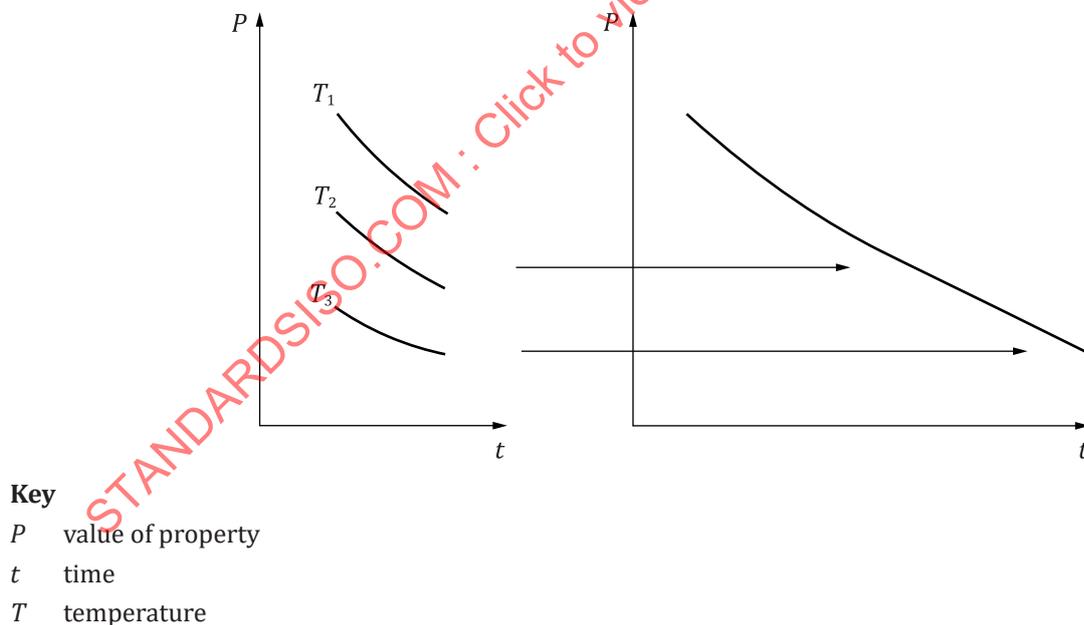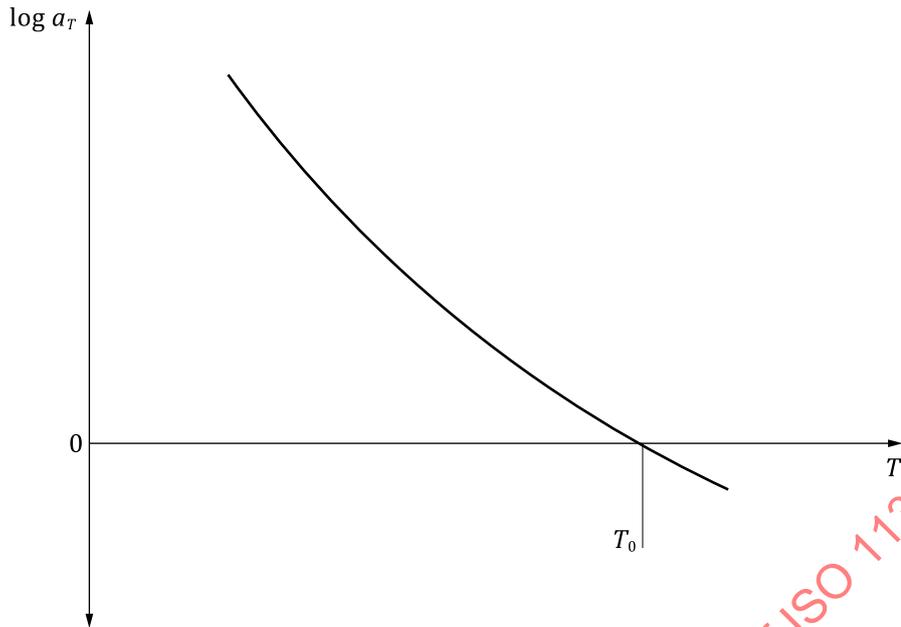

Key
P   value of property
t   time
T   temperature

Figure 5 — Construction of a master curve

Key

a_T shift factor

T temperature

T_0 reference temperature

Figure 6 — Shift factor plotted against temperature

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

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

where

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

$$v = \frac{1}{T - T_0} \tag{8}$$

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

$$a = -\frac{1}{t} \tag{9}$$

and

$$b = -\frac{r}{t} \tag{10}$$

To obtain an estimate of the life-time, use the WLF equation to determine the shift factor from the reference temperature to the temperature of interest. Apply this 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.

However, if the calculation method (as described above) can significantly improve the accuracy, for example, by applying the coefficient of determination R^2, extrapolations up to maximum 70 °C are permissible (but only in this case). This requires, above all, proof of the linearity of the Arrhenius plot with an R^2 of > 0,98. If this is not possible, it is recommended that an estimate of the uncertainty of the results be 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 can 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 document (i.e. ISO 11346:2023);

   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 document.

c) Test results:

   1) the graphs plotted, as specified in 11.1 or 11.2;

   2) the predicted life-time:

      i) for the Arrhenius procedure: the predicted life-time in years at a given time-temperature collective or a given temperature together with the threshold value used (e.g. 50 years/hot

time-temperature collective/compression set 70 %), or the maximum temperature of use at a given time;

    ii)   for the WLF procedure: 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)   Records of all single values of any measurement tested.

e)   Dates of the tests.

Annex A
(informative)

Calculation of life-time at a given time-temperature collective

This annex describes an additional option when evaluation of life-time in relation to any realistic climatic conditions is required. It is an estimation of life-time at a given time-temperature collective to reach the threshold value.

The following Arrhenius equation [i.e. Formula (A.1)] applies:

$$t_{\text{ref}} = \sum_{i=1}^{n} t_i \cdot e^{\left(\frac{E_a}{R} \cdot \left(\frac{1}{T_{\text{ref}}} - \frac{1}{T_i} \right) \right)} \tag{A.1}$$

where

T_i is the i-th temperature, in K;

T_{ref} is the reference temperature, in K;

t_i is the i-th time, in h;

t_{ref} is the time at reference temperature, in h;

E_a is the activation energy, in J/mol;

R is the universal gas constant, i.e. 8,314 J/mol K.

NOTE 1   The calculated times are accumulated according to Miner's Rule (see ISO 13760), as shown in Figure A.1.

Examples for realistic outdoor time-temperature collectives (climates) over one year are listed in Table A.1.

**Table A.1 — Time-temperature collectives over one year**

Collective Temperature °C	Hot	Moderate	Cold
		Time h	
−15		85	117
−5	8	1 279	3 093
5	1 129	3 337	3 376
15	3 457	2 584	1 701
25	2 194	882	397
35	1 073	358	77
45	553	191	
55	305	45	
65	42		
total	8 761	8 761	8 761

NOTE 2   The data in Table A.1 were obtained from field tests trials with copper pipes exposed to outdoor ageing for one year. It is the result of a systematic investigation at three different sites in Europe. Locations were Munich/Germany for "moderate", Sevilla/Spain for "hot" and Tromsoe/Norway for "cold" climate profiles.

Other time-temperature collectives may be used depending on the application. However, these time-temperature collectives shall be proven on the basis of a well-founded practical measurement over one year. They should be as close as possible to reality.

With the slope E_a/R, obtained from 11.1.3, the time-temperature collective shall now be calculated for a reference temperature, T_{ref}, using Formula (A.1) where the Miners rule (see ISO 13760) will be applied, leading to an ageing factor. The principle of this procedure is shown in Figure A.1. The step-shaped curve shows the time-temperature collective. By using Formula (A.1), the time at the reference temperature (horizontal line) is calculated.
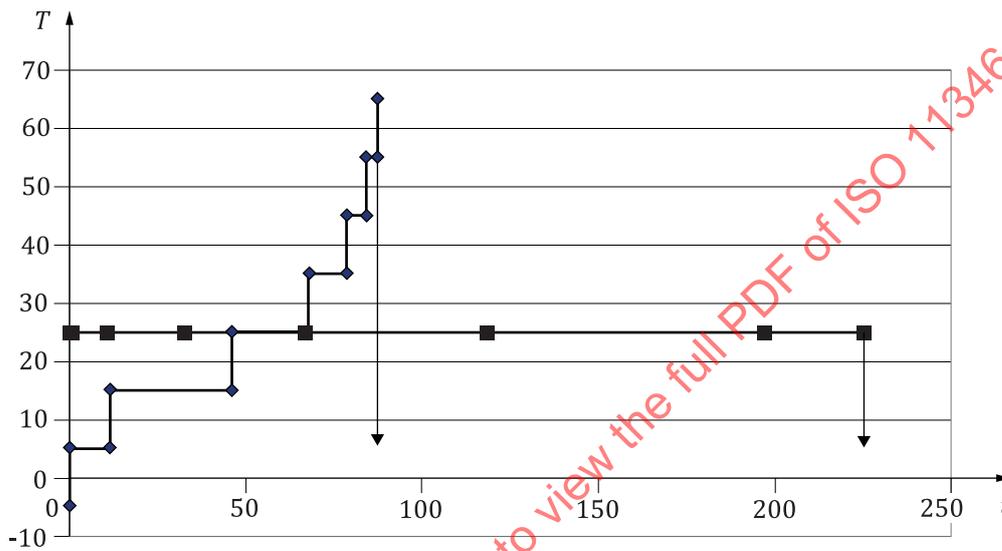
Key

T temperature (°C)

t time (×100 h)

Figure A.1 — Ageing factor

The ageing factor for a climate profile is the time at T_{ref}, calculated according to Formula (A.1) in relation to a year. In the example shown in Figure A.1, the ageing factor is equal to: 23 773/8 761 = 2,71. This means the ageing in the climate is 2,71 times higher than at a constant temperature (T_{ref}).

Provided that the T_{ref} is the same as that used for the life-time at a given temperature (as calculated in paragraph 11.1.3), the reaction time from the calculation of 11.1.3 divided by the ageing factor yields to the life-time at a given climate profile.

NOTE 3   For better understanding, an example is shown in Annex B.

It is recommended to use a specific software that is suitable to support the user in performing the described calculations.

Annex B
(informative)

Application example for the Arrhenius procedure using a calculation software for discontinuous measurements

B.1 General

For this example, the test parameter is the elongation at break (ε_B), threshold value for the decrease of ε_B ($\Delta\varepsilon_B$) is 50 % and the expected life-time is greater than 25 years.

## B.2 Measurement of test parameter, $\Delta\varepsilon_B$, to threshold value at test temperatures

B.2.1 Selection of the exposure time and exploratory test to determine lowest exposure temperature

According to Table 1, for the expected life-time of 25 years, a testing time of at least 6 months must be planned.

The ε_B versus time measurements are first taken at the lowest temperature (selected is 80 °C) followed by the calculation of $\Delta\varepsilon_B$ as shown in Table B.1.

Table B.1 — Exploratory test results: Time versus $\Delta\varepsilon_B$

Time h	$\Delta\varepsilon_B$ %	ε_B %
168	23,5	195
500	32,2	173
1 000	34,1	168
1 500	37,3	160
2 000		
3 000		
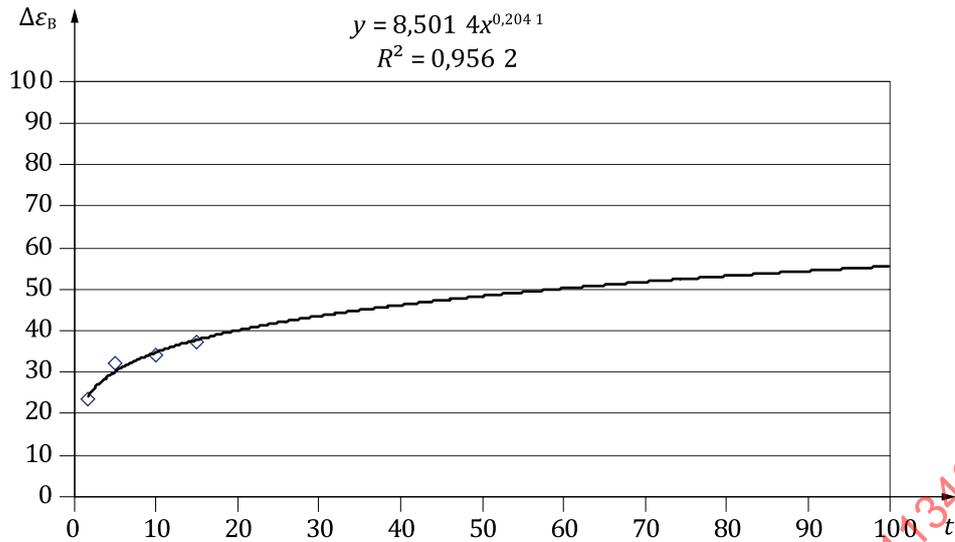
Key

$\Delta\varepsilon_B$ delta elongation at break (deterioration in %) t time (× 100 h)

Figure B.1 — Extrapolation of the test results to threshold value 50 %

Extrapolation of the data tested to threshold value as shown in Figure B.1 results in a testing time of approximately 5 890 h (greater than 6 months). Thus, testing can continue at the selected temperature of 80 °C.

If the result of the extrapolated time is less than 6 months, the lowest exposure temperature shall be reduced by 5 °C or 10 °C and the exploratory test repeated.

Further tests can now be started with a ΔT of +20 °C, i.e. 100 °C and 120 °C.

B.2.2 Execution of the test procedure and processing of the test results at three temperatures

The data and results of the testing series at three different temperatures are presented in Tables B.2 to B.4 and Figures B.2 to B.3.

Table B.2 — Testing series at 80°C

Time h	$\Delta\varepsilon_B$ %	ε_B %
168	23,5	195
500	32,2	173
1 000	34,1	168
1 500	37,3	160
2 000	41,2	150
3 000	47,1	135
4 000	47,1	135
5 000	49,0	130
6 000	53,7	118
7 000	55,7	113
8 000	56,5	111
9 000	60,8	100
10 000	60,8	100