INTERNATIONAL STANDARD

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Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use from an Arrhenius plot

Caoutchouc vulcanisé ou thermoplastique — Estimation de la durée de vie et de la température maximale d'utilisation au moyen d'un diagramme d'Arrhenius

Reference number
ISO 11346:1997(E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 11346 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 2, *Physical and degradation tests*.

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Introduction

With rising temperature, the reaction rate of a chemical reaction is normally increased. For many organic-chemical reactions a temperature rise of 10 °C means about 2 to 3 times higher reaction rate. The temperature dependence of chemical reactions can be described by the Arrhenius equation:

$$K(T)=A \bullet e^{-E/RT} \quad (1)$$

where

$K(T)$ =	reaction rate constant	(min^{-1})
A =	pre-exponential factor	(min^{-1})
E =	activation energy	(J/mol)
R =	gas constant	(8,314 J/mol K)
T =	absolute temperature	(K)

The state of a chemical reaction is given by the relation:

$$F_x(t)=K(t) \bullet t \quad (2)$$

where

$F_x(t)$ = function of the state of the reaction x

t = reaction time (min)

Due to different reaction rates K_i at different temperatures T_i the same threshold value F_a of a reaction will be reached by different reaction times t_i (equal-value times), e.g. t_1 to t_3 in figure 1:

$$F_a(t_i)=K_i(T_i) \bullet t_i \quad (3)$$

The Arrhenius equation (1) can be substituted in equation (3) as follows:

$$F_a(t_i)=A \bullet e^{-E/RT_i} \bullet t_i \quad (4)$$

or in logarithmic form with the constant terms combined in B

$$\ln t_i = E/RT_i + B \quad (5)$$

A plot of ln t versus 1/T gives a straight line with the slope E/R, and is known as an Arrhenius plot (see figure 2). The Arrhenius plot is however normally presented with $\log_{10}$ time against 1/T. The activation energy is constant over a temperature range in which the main ageing reaction is the same. For extrapolation of short-time data to predict long-term performance, an appropriate curve must be drawn through the short-time values.

Caution must be used when the results are analysed. Thermal-oxidative ageing is diffusion-controlled and thus different results can be achieved when comparing thin and thick test pieces. The ageing reactions can also differ at different temperatures and can affect the property being measured, especially where it is influenced by the balance between scission and cross-linking reactions. The test conditions in the laboratory may also differ from service conditions where other causes of deterioration such as light ageing and ozone attack may be involved.

During the preparation of this International Standard account was taken of the contents of ISO 2578 and IEC 216.

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Rubber, vulcanized or thermoplastic — Estimation of life-time and maximum temperature of use from an Arrhenius plot

1 Scope

This International Standard describes the principles and procedures for estimating the life-time and maximum temperature of use of vulcanized or thermoplastic rubbers, using an Arrhenius plot.

This method is suitable for different tests on rubber, but for tests under stress or deformation (creep, relaxation, etc.) physical (viscoelastic) changes of material cannot easily be separated from any chemical change. Then the Arrhenius equation is no longer the only possible model, and the Willams, Landel, Ferry (WLF) equation may be more suitable to represent the change in the material as a function of time.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 188:–[1], *Rubber, vulcanized - Accelerated ageing and heat-resistance tests.*

ISO 471:1995, *Rubber - Temperatures, humidities and times for conditioning and testing.*

ISO 2578:1993, *Plastics - Determination of time-temperature limits after prolonged exposure to heat.*

IEC 216 (all parts), *Guide for the determination of thermal endurance properties of electrical insulating materials.*

3 Definitions

For the purposes of this International Standard, the following definitions apply:

3.1 life-time: The 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: The 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: The value specified for the property or change in property being tested .

[1] To be published. (Revision of ISO 188:1982)

4 Principle

4.1 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, so that the time limit for that particular temperature of ageing can be determined.

Further tests are done at at least two other temperatures.

4.2 The time limits obtained are plotted on an Arrhenius plot as a function of the temperature, and the straight line obtained is extrapolated back to the temperature of use.

4.3 Although the extrapolation can be made to extremely long times, consideration must be given to the possibility that the chemical reaction at high temperatures is gradually replaced by a different reaction at lower temperatures. In such a case, the degradation curve will normally deviate from linearity. Because of these considerations, extrapolations are often limited to 30 °C to 40 °C beyond the last data point. If a longer extrapolation is needed, the greater uncertainty in the results must be appreciated.

5 Selection of test

The test chosen should preferably relate to a property which is likely to be of significance in practice. Wherever possible, use should be made of methods of test specified in International Standards.

The following properties are often used: tensile strength, elongation at break, stress relaxation in compression or tension, compression set, tension set and tension creep.

6 Selection of threshold value

The threshold value should be chosen to suit the conditions of use.

A change to 50% of the initial value of the property under investigation is often used. For properties like compression set and stress relaxation, a value not exceeding 50% is often used.

7 Test pieces

7.1 The dimensions and method of preparation of the test pieces shall be in accordance with the specifications given for the relevant test method.

7.2 The total number of test pieces needed depends on:

a) the number of test pieces required for the relevant test method;

b) the number of time-tests to be made to find the threshold value at a chosen temperature;

c) the number of test temperatures;

d) the number of control tests with reference test pieces to be made at every heating period.

It is advisable to age more than the minimum number of test pieces required, in case problems occur after several weeks, months or years of ageing.

7.3 The tests can often be done with logarithmic time intervals, for example 1 day, 2 days, 4 days, 1 week, 2 weeks, 4 weeks, 8 weeks, 16 weeks, 32 weeks, etc. Logarithmic time intervals can however be inappropriate for thermal-oxidative ageing, for chemical relaxation and for autocatalytic reactions, where closer test intervals may be needed.

7.4 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.

8 Temperature of test

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

8.2 Test pieces shall be tested 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 test temperature shall be chosen so that the time taken to reach the threshold value is at least 1000 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 471.

9 Ageing ovens

9.1 An ageing oven meeting the requirements of ISO 188 shall be used.

When tests are made in media other than air, the method of temperature control shall be adapted to the particular medium used.

9.2 When the tests are made in air, ovens with a known air exchange rate and a known air speed shall be used.

Air speed and air exchange rate shall be adequate to ensure that the rate of thermal deterioration is not influenced by accumulated products or evaporated components, or by oxygen depletion. Too high an air speed has been shown to increase deterioration caused by increased oxidation and volatilization of antioxidants and softeners.

9.3 Due to the risk of cross-contamination between test pieces of different rubber materials, use separate ovens or cells for each material.

10 Procedure

10.1 At the beginning of the test procedure, make the reference test run with the required number of test pieces, conditioned and tested in accordance with the appropriate standard test method.

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

10.3 At the end of each heating period, condition, if necessary, the test pieces to be examined under the appropriate controlled conditions and then carry out the tests by the previously chosen methods.

10.4 Continue this procedure until the numerical value of the property under investigation exceeds the relevant threshold value.

11 Expression of results

11.1 To facilitate the determination of the time at which the threshold value is reached, plot the results to give a curve of value of the selected property against time. Determine the values t_1, t_2, t_3, ... by interpolation. An example of such a graph is shown in figure 1.

11.2 Plot the log time at which the threshold value is reached for each of the test temperatures against the reciprocal 1/T of the corresponding temperature (T = absolute temperature in K). Construct a best-fit straight line through the plotted points. This may be done using established statistical methods.

If a straight line is not appropriate, carry out further tests at other, intermediate, temperatures. If a straight line is still not appropriate, the approach shall be abandoned.

Life-time: Extrapolate the line obtained to the temperature of use giving the life-time estimation. An example of such a plot is given in figure 2.

Maximum temperature of use: Extrapolate the line obtained to the specified time giving the maximum temperature of use estimation. 20 000 h is often used as the time for establishing a general maximum temperature of use.

12 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) sample details:

1) complete identification of the material tested,

2) dimensions and method of preparation of the test pieces, with reference to the relevant ISO standard,

3) selected property, with reference to the relevant ISO standard,

4) threshold value of the selected property,

5) times and temperatures for conditioning of test pieces;

c) ageing details:

1) oven type(s) used, with details of air exchange rate and air speed,

2) times and temperatures of exposure in the ovens,

3) precise details of the ageing conditions;

d) test results:

1) graphs plotted as specified in 11.1 and 11.2,

2) life-time prediction at a chosen test temperature, together with the temperature of use, or the maximum temperature of use at a chosen time;

e) the date of the test.

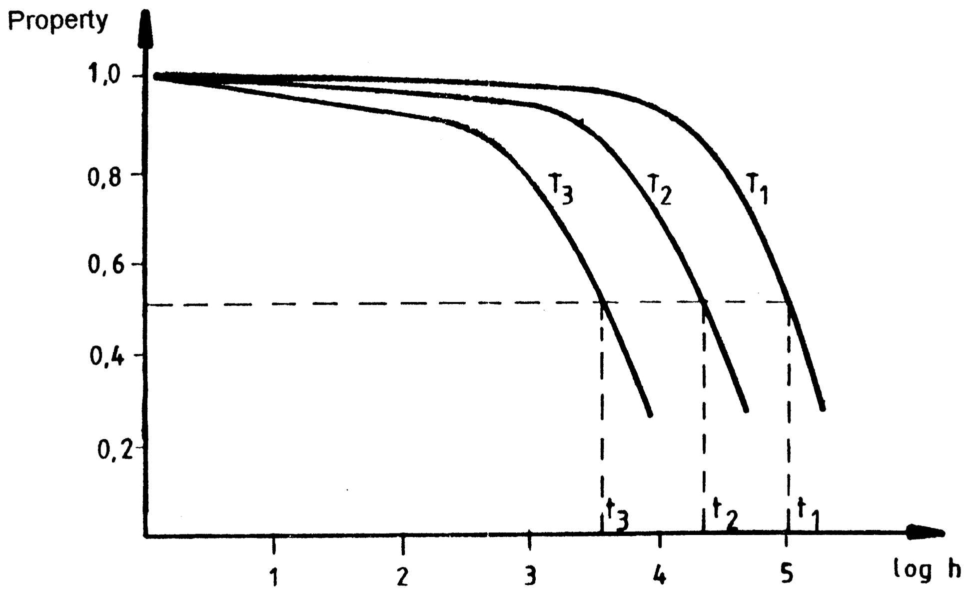

Figure 1 – Material property against time

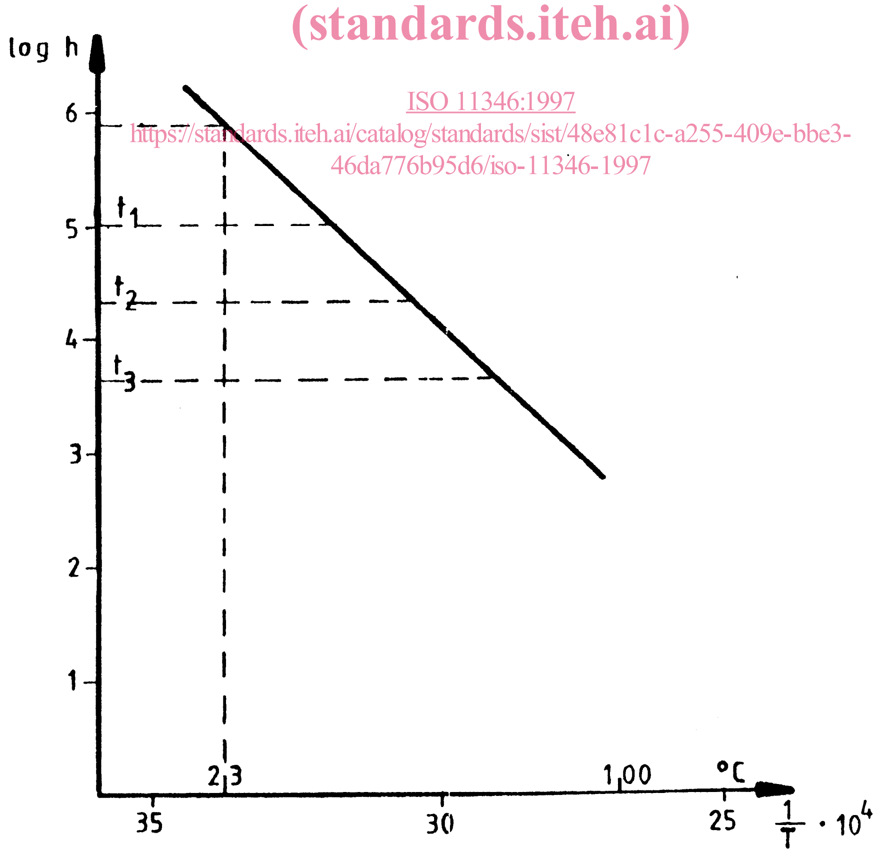

Figure 2 – Arrhenius plot (time against temperature)