INTERNATIONAL STANDARD

ISO 2578

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Plastics — Determination of time-temperature limits after prolonged exposure to heat

Plastiques — Détermination des limites temps-températures après exposition à l'action prolongée de la chaleur

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Contents

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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 2578 was prepared by Technical Committee ISO/TC 61, *Plastics*, Sub-Committee SC 6, *Ageing, chemical and environmental resistance*.

This second edition cancels and replaces the first edition (ISO 2578:1974), of which it constitutes a technical revision.

Annexes A and B form an integral part of this International Standard. Annex C is for information only.

Introduction

During the preparation of this International Standard, account was taken of the contents of IEC 216. Accordingly, the terms and definitions in this International Standard, as well as the procedures described, are in line or identical with those specified in IEC 216.

Plastics — Determination of time-temperature limits after prolonged exposure to heat

1 Scope

1.1 This International Standard specifies the principles and procedures for evaluating the thermal endurance properties of plastics exposed to elevated temperature for long periods.

1.2 The term thermal endurance is used here to refer to tests made in air, excluding any other influence or stress applied to the test specimens. Thermal endurance properties evaluated in different environments and/or with different stresses applied to the test specimens require different test procedures.

1.3 In this International Standard, the study of the thermal ageing of plastics is based solely on the change in certain properties resulting from a period of exposure to elevated temperature. The properties studied are always measured after the temperature has returned to ambient.

The various properties of plastics change at various rates on thermal ageing. To enable comparisons to be made of the thermal ageing of different plastics, the criteria for judgement depend on the type of property to be studied and its acceptable limiting value.

1.4 In the application of this standard it is assumed that a practically linear relationship exists between the logarithm of the time required to cause the predetermined property change and the reciprocal of the corresponding absolute temperature (Arrhenius Law).

For the plastics tested, no transition, in particular a first-order transition, should occur in the temperature range under study.

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 291:1977, *Plastics — Standard atmospheres for conditioning and testing.*

IEC 216-1:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 1: General guidelines for ageing procedures and evaluation of test results.*

IEC 216-2:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 2: Choice of the test criteria.*

IEC 216-3-1:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 3: Instructions for calculating thermal endurance characteristics — Section 1: Calculations using mean values of normally distributed complete data.*

IEC 216-3-3:—,[1] *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 3: Instructions for calculating thermal endurance characteristics — Section 3: Calculations for incomplete data.*

IEC 216-4-1:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 4: Ageing ovens — Section 1: Single-chamber ovens.*

IEC 216-5:1990, *Guide for the determination of thermal endurance properties of electrical insulating materials — Part 5: Guidelines for the application of thermal endurance characteristics.*

1) To be published. [15B (B.C.) 82]

3 Definitions

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

3.1 temperature index (TI): Number corresponding to the temperature, in degrees Celsius, derived from the thermal endurance relationship at a given time (normally 20 000 h).

3.2 relative temperature index (RTI): Temperature index of a test material, obtained at the time which corresponds to the known temperature index of a reference material when both materials are subjected to the same ageing and diagnostic procedures in a comparative test.

3.3 halving interval (HIC): Number corresponding to the temperature interval, in degrees Celsius, which expresses the halving of the time to end-point taken at the temperature of the TI or the RTI.

HIC is a measure of the slope of the thermal endurance graph. It is not constant, but varies with temperature even when the thermal endurance relationship is linear. In many practical cases, the error arising from the use of HIC in the temperature range of interest remains within acceptable limits.

3.4 thermal endurance graph (Arrhenius graph)**:** Graph in which the logarithm of time to reach a specified end-point in a thermal endurance test is plotted versus the reciprocal thermodynamic (absolute) test temperature.

3.5 threshold value: Value, corresponding to a percentage of the initial value of the property under investigation, at which the ageing test is stopped and the time to failure is calculated.

NOTES

1 The level of 50 % is often used to determine the threshold value.

2 If a minimum value is required after ageing, it may be agreed between the interested parties to use this minimum value as the threshold value, instead of a percentage of the initial value.

3.6 test-specimen failure time: Time required at the exposure temperature for a specimen to either fail the proof test or reach the threshold value of the characteristic under investigation, whichever is shorter.

4 Principle

4.1 Determination of time to failure

At a chosen temperature, the variations in the numerical value of a chosen characteristic (for example, a mechanical, optical or electrical property) are determined as a function of time.

The procedure is continued until the relevant threshold value of that characteristic has been reached, resulting in the time to failure at that particular temperature.

Further specimens are exposed at at least two other temperatures and the variations in the relevant characteristic determined. It is recommended to heat-age test specimens at three to four temperatures, and determine the time to failure for each of the temperatures.

4.2 Determination of TI

The times to failure are plotted versus the reciprocal values of the exposure temperatures. The intersection of this curve with the chosen time limit (in general 20 000 h) gives the temperature index sought.

4.3 Use of correlation coefficient

The reliability of the extrapolation of the graph depends on obtaining an acceptable Arrhenius plot, which may not be possible with materials showing behaviour related to a transition phenomenon in the chosen temperature range.

For this purpose the correlation coefficient r is calculated in accordance with annex B. If this calculation results in a value smaller than 0,95 (for three test temperatures; see also IEC 216), an additional test at a different test temperature may improve the linearity of data.

4.4 Determination of RTI

For determination of RTI, the chosen reference plastic, its thermal endurance and the method of determination are of central importance.

The reference plastic shall be of the same type as the tested plastic, and have a history of satisfactory service. It shall have a known temperature index for the property and a threshold value which are the same, or at least reasonably similar to, those to be employed in the RTI test. The TI and HIC of the reference material should also be approximately the same as the values expected for the tested plastic.

Since processing conditions may significantly affect the ageing characteristics of some materials, the sampling, cutting of sheet from the supply roll, cutting of anisotropic material in the same direction, moulding, curing, preconditioning, etc. shall be performed in the same manner for both materials, and the specimens shall be tested in the same thickness.

5 Choice of test

The test chosen shall relate to a characteristic which is likely to be of significance in practice and, wherever possible, use shall be made of test methods specified in International Standards. If the dimensions and/or form of the test specimens are altered by the heat treatment, then only test methods which are independent of these effects may be used.

6 Choice of end-point

For the selection of the end-point, two factors shall be agreed upon:

a) the period of time for which a time-temperature limit shall be estimated; for general purposes, a period of 20 000 h is recommended;

NOTE 3 Other times (shorter or longer than 20 000 h) may be chosen if necessary.

b) the acceptable threshold value of the chosen characteristic; this threshold value depends on the conditions of use foreseen.

7 Test specimens

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

7.2 For a criterion requiring a destructive test, the minimum total number (n) of test specimens needed depends on

— the number a of test specimens required for one test, according to the test specifications given for the relevant test method;

— the number b of tests necessary to determine the end-point at one exposure temperature;

— the number c of exposure temperatures;

— the number d of specimens needed for the initial test before heat ageing.

Thus the total number of specimens is:

$$n = abc + d$$

For a criterion requiring non-destructive testing, and for each exposure temperature, in most cases a group of five test specimens is adequate.

NOTES

4 When there is a large number of specimens to be tested, it may be possible in certain cases to deviate from the relevant test specifications and to reduce this number. However, it must be recognized that the precision of the test result depends to a large extent on the number of specimens tested.

5 In contrast, when the individual results are too scattered, an increase in the number of specimens may be necessary in order to obtain satisfactory precision.

6 It is advisable to make an approximate assessment, by means of preliminary tests, of the number and duration of the ageing tests required.

8 Exposure temperatures

8.1 Test specimens shall be exposed at not less than three temperatures, covering a range adequate to establish the time-temperature limit 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 5 000 h. Similarly, the highest temperature shall be chosen so that the time taken to reach the threshold value is not shorter than 100 h. The lowest exposure temperature shall not be more than 25 °C above the anticipated TI.

8.2 If the temperature limit sought is intended for a time other than 20 000 h (see clause 6, note 3), the lowest exposure temperature shall be chosen so that the time taken to reach the threshold value is at least one-fourth of the time limit chosen for extrapolation.

8.3 Selection of adequate exposure temperatures requires previously determined information on the material under test. If such information is not available, exploratory tests may help in selecting exposure temperatures which are suitable for evaluating the thermal endurance characteristics.

9 Ageing ovens

For heat ageing, ovens shall be used that meet the requirements specified in IEC 216-4, in particular with respect to the temperature tolerances and ventilation rates of air exchange.

10 Procedure

10.1 In addition to the specimens to be exposed to heat-ageing temperatures, an adequate number of test specimens shall be kept separately as a reserve

— for cases in which the accuracy requires heat ageing at an additional temperature;

— as reference specimens.

They shall be stored in an appropriately controlled atmosphere (see ISO 291).

10.2 Before the heat-ageing procedure is started, an initial test shall be made at room temperature with the required number of specimens conditioned and tested in accordance with the chosen test method.

Thermosetting materials shall be conditioned for 48 h at the lowest exposure temperature of the range selected.

NOTE 7 If necessary, thermoplastic materials should be annealed for 48 h at the lowest exposure temperature of the range selected.

10.3 Place the required number of specimens in each of the ovens maintained at the selected temperatures.

If there is a risk of cross-contamination between test specimens originating from different plastics, use separate ovens for each material.

10.4 At the end of each heat-ageing period (see note 8), the required number of test specimens is removed from the oven and conditioned, if necessary, under the appropriately controlled atmosphere (see ISO 291). The test, in accordance with the selected test criterion, shall be carried out at room temperature.

NOTE 8 After the first week, during which the number of determinations of the characteristic under investigation can vary depending on the material under test, the test durations are generally selected in accordance with annex C.

10.5 Continue this procedure until the numerical value of the characteristic under investigation reaches the relevant threshold value.

11 Evaluation of results

11.1 For both destructive and non-destructive tests, for each exposure temperature and for each heat-ageing period, the value of the chosen property is plotted as a function of the logarithm of the time (see figure 1). The point at which this graph intersects the horizontal line representing the end-point criterion is taken as the time to failure.

When applying a proof test, the times to failure shall be calculated as the mean values.[2)]

11.2 The calculation of the thermal endurance curve is based on these times to failures and the respective exposure temperatures. If mean values are used, the logarithmic mean represents the time to failure.

11.3 Calculate a first-order regression line in accordance with annex A.

11.4 Plot the times to failure versus exposure temperatures on graph paper having a logarithmic time scale as the ordinate and an abscissa based on the reciprocal of the absolute temperature but showing the correlated values in degrees Celsius. The first-order regression line is drawn through the points plotted on the graph, which thus represents the thermal endurance of the material under test. An example is given in figure 2.

2) See IEC 216-1:1990, subclause 11.1.

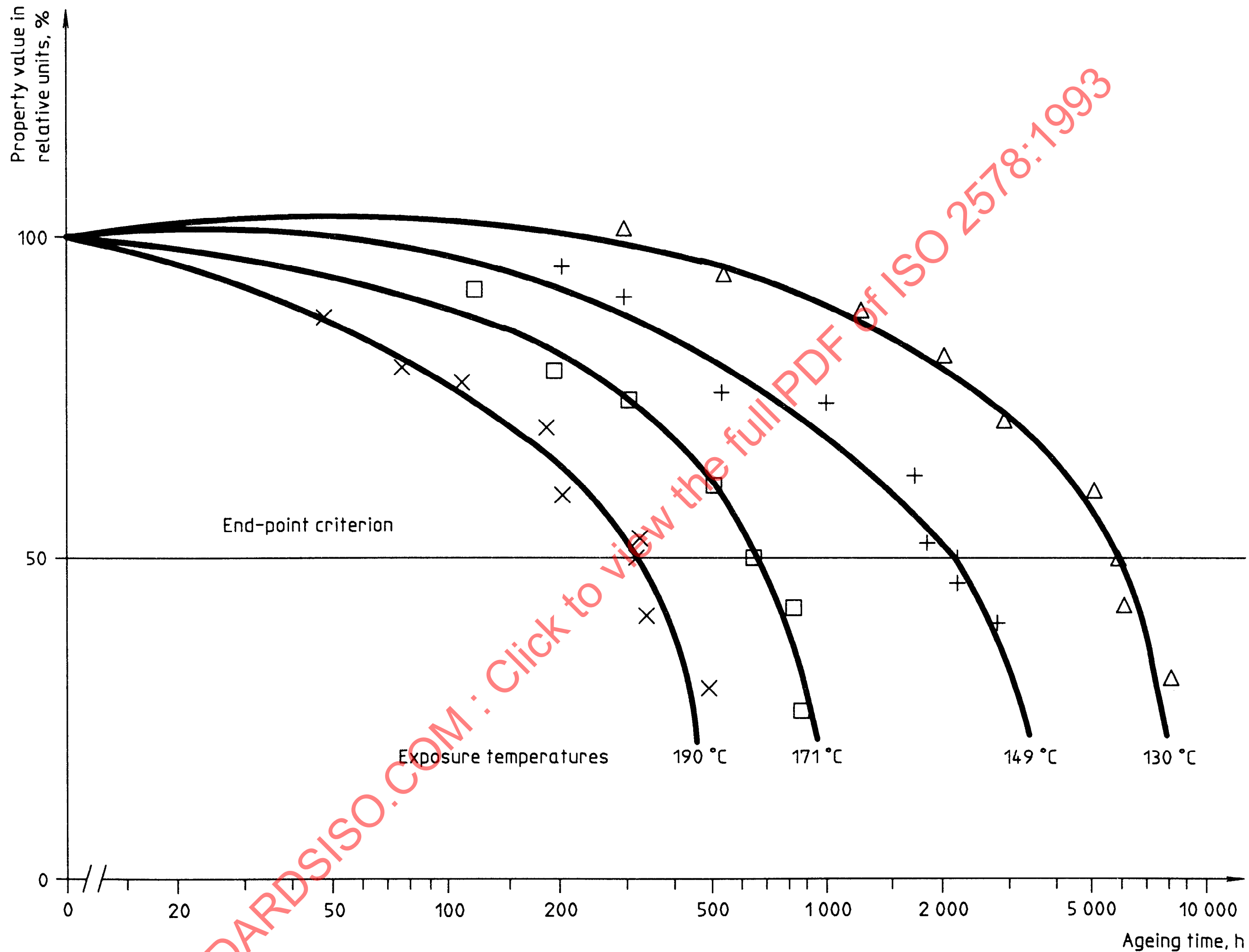

Figure 1 — Determination of the time to reach the end-point at each temperature — Property variation
(According to IEC 216-1)

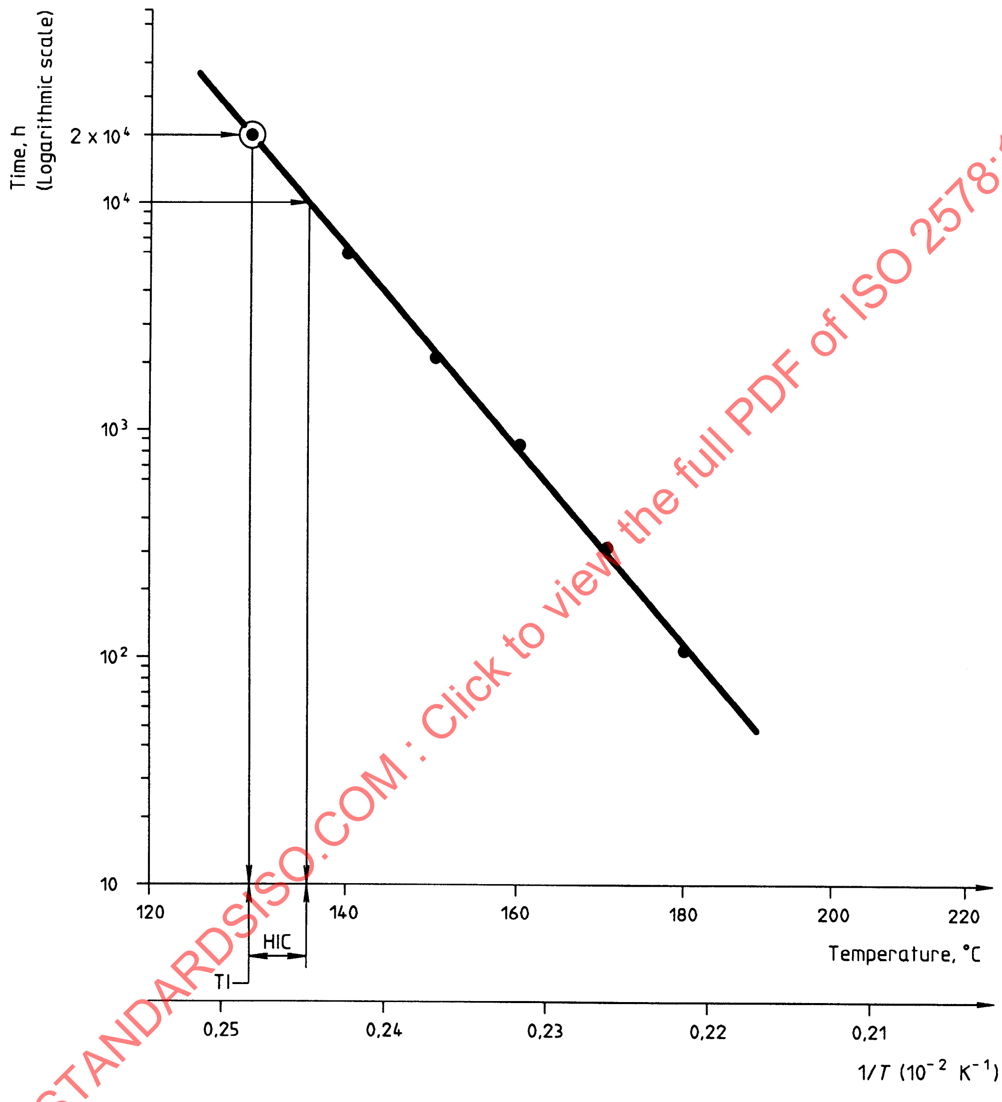

Figure 2 — Thermal endurance graph — Temperature index — Halving interval (According to IEC 216-1)

11.5 Calculate the temperature index (TI) corresponding to the time limit chosen (generally 20 000 h) and the halving interval (HIC) in accordance with annex A; calculate the correlation coefficient in accordance with annex B.

NOTES

9 When the temperature scale is chosen in such a way that equal intervals correspond to equal intervals of reciprocal kelvins, then the various points obtained will be found to lie on a straight line if a linear dependence exists.

10 When the temperature range used is comparatively small, a curve can be prepared using an abscissa scale proportional to the temperature; in this case the curve can be fit to a straight line only with great circumspection.

12 Determination of the relative temperature index

The relative temperature index (RTI) is a thermal endurance characteristic which is derived from the two thermal endurance relationships or curves resulting from the comparative testing of the test material and the reference material. The RTI is specifically related to the time corresponding to the TI originally determined for the reference material.

The RTI consists of two numbers, one representing the temperature derived using equation (1) and the other the associated HIC. The derivation may be numerical or graphical (details are given in IEC 216-3).

In the derivation and in figure 3, the following notation is used:

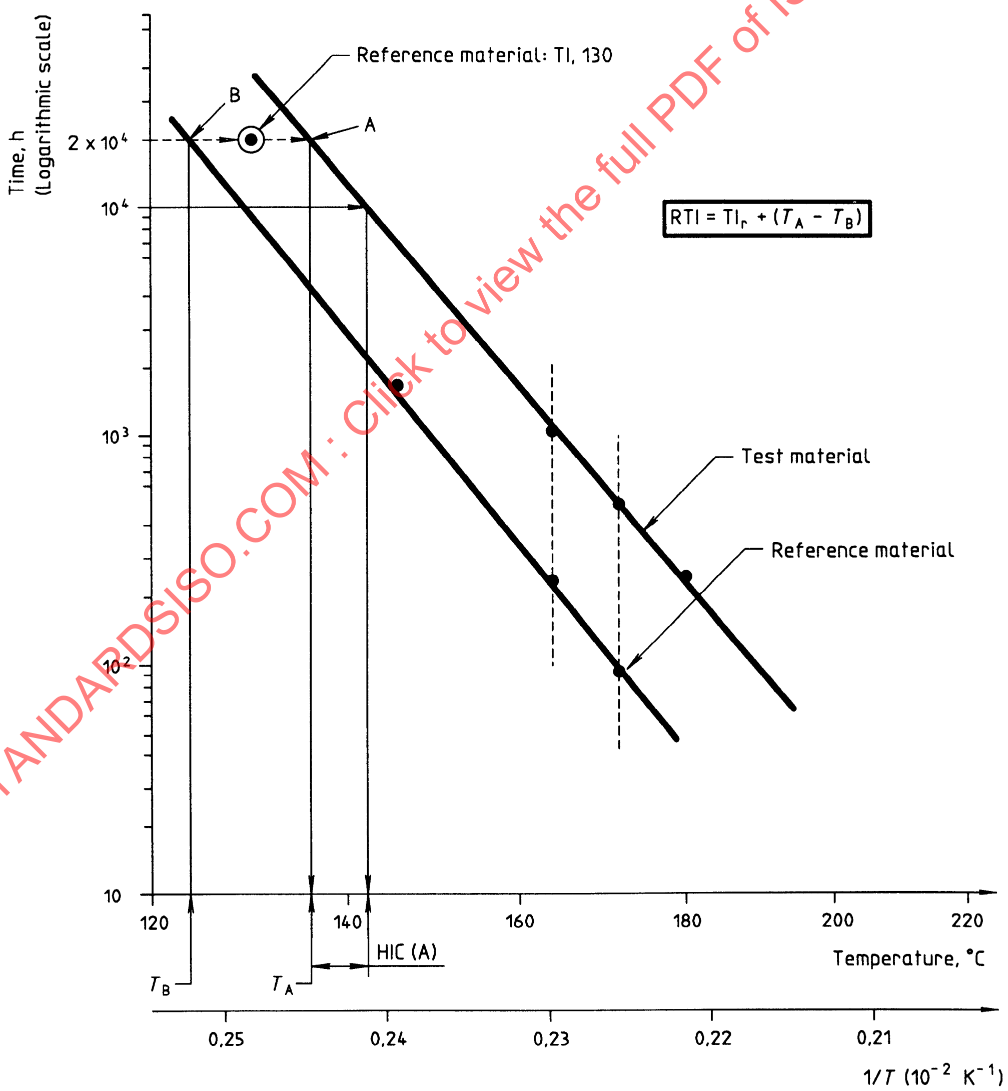

Figure 3 — Thermal endurance graph — Relative temperature index — Halving interval
(According to IEC 216-1)

TI_r original TI of the reference material;

t_o time corresponding to TI_r;

A point on the test material's thermal endurance relationship or graph from the comparative test, having coordinates θ_A, t_o;

B point on the reference material's thermal endurance relationship or graph, having coordinates θ_B, t_o;

HIC_r HIC for the reference material at point TI_r of its original thermal endurance relationship or graph;

HIC(A) HIC for the test material at point A;

HIC(B) HIC for the reference material at point B.

The points A and B may be determined either graphically or numerically, and the RTI then determined using equation (1):

$$RTI = TI_r + \theta_A - \theta_B \qquad \dots (1)$$

When reporting the RTI, the usual information regarding the property, end-point and test specimen data should be supplemented with corresponding information regarding the reference material.

NOTE 11 The criteria necessary to carry out the instructions given in 4.4 are under consideration.

13 Test report

The test report shall include the following particulars:

a) all information necessary for complete identification of the material tested;

b) reference to this International Standard;

c) precise details of the ageing conditions, if these are other than the exposure of unstressed specimens to hot air;

d) chosen characteristic, with reference to the standard concerning the corresponding test;

e) threshold value of the chosen characteristic;

f) shape, dimensions and method of preparation of the test specimens, with reference to the relevant standard;

g) conditioning;

h) type of oven, with details of rate and direction of airflow;

i) times and temperatures of exposure in ovens;

j) if necessary, the curves giving, for each temperature, the values of the characteristic plotted against time;

k) if applicable, a graph of the logarithm of lifetime plotted against temperature, with reference to the statistical method used;

l) the TI, HIC and correlation coefficient.

Annex A
(normative)

Calculation of the regression line

This annex presents a method for rapid plotting of the regression line for endurance data. This method may be used for any number of measurements at various exposure temperatures. If information about confidence limits is required, a more detailed analysis may be made in accordance with IEC 216.

It has been established that the deterioration of many plastics materials follows the equation:

$$L = A\mathrm{e}^{B/T} \qquad \text{. . . (A.1)}$$

where

L	is the thermal endurance; in hours;
T	is the absolute temperature, in kelvins;
A, B	are constants for each plastics material;
e	is the base of natural logarithms.

Equation (A.1) may be expressed as a linear function using logarithms:

$$\log_{10}L = \log_{10}A + (\log_{10}\mathrm{e})B/T \qquad \text{. . . (A.2)}$$

Let

$$Y = \log_{10}L$$

$$a = \log_{10}A$$

$$X = \frac{1}{T}$$

$$b = (\log_{10}\mathrm{e})B$$

Then

$$Y = a + bX \qquad \text{. . . (A.3)}$$

Thus, data from testing at higher temperatures may be plotted as $\log_{10}L$ versus $1/T$ on semilogarithmic graph paper and a straight line extrapolated to lower temperatures (see figure A.1). However, since the nature of logarithmic plots does not allow accurate extrapolation by drawing the best apparent straight line through the data points, a more rigorous method must be used for greater accuracy and uniformity. By the use of the method of least squares, the constant a and slope b may be derived in terms of the experimental data obtained. These equations are as follows:

$$a = \frac{\left(\sum Y - b\sum X\right)}{N} \qquad \text{. . . (A.4)}$$

$$b = \frac{N\sum XY - \sum X\sum Y}{N\sum X^2 - \left(\sum X\right)^2} \qquad \text{. . . (A.5)}$$

where

$X\ (= 1/T)$	is the reciprocal of the test temperature, in kelvins (°C + 273);
N	is the number of times to failure;
$Y\ (= \log_{10}L)$	is the logarithm of the time to failure.

Having determined the constant a and the slope b of the regression line, the temperature at any required lifetime may be calculated as follows:

$$Y = a + bX \qquad \text{. . . (A.6)}$$

$$T = \frac{1}{X} = \frac{b}{Y - a} \qquad \text{. . . (A.7)}$$

Temperature at 20 000 h, in degrees Celsius (TI)

$$\mathrm{TI} = \frac{b}{4{,}301\,0 - a} - 273 \qquad \text{. . . (A.8)}$$

Temperature at 2 000 h, in degrees Celsius

$$\mathrm{TI} = \frac{b}{3{,}301\,0 - a} - 273 \qquad \text{. . . (A.9)}$$

Temperature at 10 000 h, in degrees Celsius

$$\mathrm{TI} = \frac{b}{4{,}000\,0 - a} - 273 \qquad \text{. . . (A.10)}$$

To simplify the handling of the test data used in equations (A.4) to (A.10), it is suggested that the steps be followed as outlined below for a sample calculation (see tables A.1 and A.2):

a) In the column "°C" as illustrated in table A.2, list each temperature at which a set of specimens was tested.

b) In the second and third columns, list the reciprocals ($X = 1/T$) and the squares of the reciprocals

($X = 1/T^2$) of the above test temperatures, converted to kelvins (see also table A.1).

c) In the fourth column list the time to failure L, in hours, of each set of specimens, and in the fifth column list the $\log_{10}$ of the value L in the fourth column ($Y = \log_{10}L$).

d) In the sixth column, list the products of $X \cdot Y$.

e) Carry out summations for columns 2, 3, 5 and 6 and enter the summation (indicated by $\sum$) at the bottom of the respective column.

f) Indicate the number N of times to failure on the worksheet.

g) Using the values obtained in steps e) and f), compute b [equation (A.5)] and a [equation (A.4)] in that order. The constant a will always be negative.

h) Using constants a and b, solve for temperature, in degrees Celsius, at 20 000 h [equation (A.8)] and at 2 000 h [equation (A.9)].

i) Plot the above two temperature points from step h) on $\log_{10}L$ versus $1/T$ graph paper and draw the regression line through them.

j) Plot the times to failure L at their respective temperatures on the same graph.

k) Using constants a and b, solve for temperature, in degrees Celsius, at 10 000 h [equation (A.10)].

l) Calculate the HIC, in degrees Celsius, as the difference between the temperature for 20 000 h and the temperature for 10 000 h.

Table A.1 — Commonly used test temperatures, in degrees Celsius, corresponding values in kelvins with their reciprocal and reciprocal squared values (see table A.2)

θ °C	T K	$X = 1/T$ K^{-1}	$X^2 = 1/T^2$ K^{-2}
70	343	$2{,}915\ 4 \times 10^{-3}$	$8{,}499\ 56 \times 10^{-6}$
85	358	$2{,}793\ 2 \times 10^{-3}$	$7{,}801\ 97 \times 10^{-6}$
100	373	$2{,}680\ 9 \times 10^{-3}$	$7{,}187\ 22 \times 10^{-6}$
105	378	$2{,}645\ 50 \times 10^{-3}$	$6{,}998\ 68 \times 10^{-6}$
125	398	$2{,}512\ 56 \times 10^{-3}$	$6{,}312\ 97 \times 10^{-6}$
130	403	$2{,}481\ 39 \times 10^{-3}$	$6{,}157\ 29 \times 10^{-6}$
140	413	$2{,}421\ 31 \times 10^{-3}$	$5{,}862\ 73 \times 10^{-6}$
150	423	$2{,}364\ 07 \times 10^{-3}$	$5{,}588\ 81 \times 10^{-6}$
165	438	$2{,}283\ 11 \times 10^{-3}$	$5{,}212\ 57 \times 10^{-6}$
175	448	$2{,}232\ 14 \times 10^{-3}$	$4{,}982\ 46 \times 10^{-6}$
180	453	$2{,}207\ 51 \times 10^{-3}$	$4{,}873\ 08 \times 10^{-6}$
185	458	$2{,}183\ 41 \times 10^{-3}$	$4{,}767\ 26 \times 10^{-6}$
190	463	$2{,}159\ 83 \times 10^{-3}$	$4{,}664\ 85 \times 10^{-6}$
200	473	$2{,}114\ 16 \times 10^{-3}$	$4{,}469\ 69 \times 10^{-6}$
220	493	$2{,}028\ 40 \times 10^{-3}$	$4{,}114\ 40 \times 10^{-6}$
225	498	$2{,}008\ 03 \times 10^{-3}$	$4{,}032\ 19 \times 10^{-6}$
240	513	$1{,}949\ 32 \times 10^{-3}$	$3{,}799\ 84 \times 10^{-6}$
250	523	$1{,}912\ 05 \times 10^{-3}$	$3{,}633\ 92 \times 10^{-6}$
260	533	$1{,}876\ 17 \times 10^{-3}$	$3{,}520\ 02 \times 10^{-6}$
280	553	$1{,}808\ 32 \times 10^{-3}$	$3{,}270\ 01 \times 10^{-6}$
300	573	$1{,}745\ 20 \times 10^{-3}$	$3{,}045\ 73 \times 10^{-6}$
320	593	$1{,}686\ 34 \times 10^{-3}$	$2{,}843\ 74 \times 10^{-6}$

Table A.2 — Sample calculation

1	2	3	4	5	6
Temperature °C	$X = 1/T$ K^{-1}	$X^2 = 1/T^2$ K^{-2}	L h	$Y = \log_{10}L$	$XY = (\log_{10}L)/T$
170	$2{,}257\ 33 \times 10^{-3}$	$5{,}095\ 57 \times 10^{-6}$	5 600	3,748 19	$8{,}460\ 90 \times 10^{-3}$
185	$2{,}183\ 41 \times 10^{-3}$	$4{,}767\ 26 \times 10^{-6}$	2 600	3,414 97	$7{,}456\ 27 \times 10^{-3}$
200	$2{,}114\ 16 \times 10^{-3}$	$4{,}469\ 69 \times 10^{-6}$	1 500	3,176 09	$6{,}714\ 77 \times 10^{-3}$
215	$2{,}049\ 18 \times 10^{-3}$	$4{,}199\ 14 \times 10^{-6}$	640	2,806 18	$5{,}750\ 37 \times 10^{-3}$
Σ	$8{,}604\ 08 \times 10^{-3}$	$18{,}531\ 66 \times 10^{-6}$		13,145 43	$28{,}382\ 31 \times 10^{-3}$

$N = 4$

$$b = \frac{N\sum XY - \sum X \sum Y}{N\sum X^2 - (\sum X)^2} = \frac{(4 \times 28{,}382\ 31 \times 10^{-3}) - (8{,}604\ 08 \times 10^{-3} \times 13{,}145\ 43)}{(4 \times 18{,}531\ 66 \times 10^{-6}) - (8{,}604\ 08 \times 10^{-3} \times 8{,}604\ 08 \times 10^{-3})} = 4{,}406 \times 10^3$$

$$a = \frac{\sum Y - b\sum X}{N} = \frac{13{,}145\ 43 - (4{,}406 \times 10^3 \times 8{,}604\ 08 \times 10^{-3})}{4} = -6{,}190$$

$$\text{Temperature at 20 000 h, in degrees Celsius} = \frac{b}{Y - a} - 273 = \frac{4{,}406 \times 10^3}{4{,}301\ 0 + 6{,}190} - 273 = 147\ °\text{C}$$

$$\text{Temperature at 2 000 h, in degrees Celsius} = \frac{b}{Y - a} - 273 = \frac{4{,}406 \times 10^3}{3{,}301\ 0 + 6{,}190} - 273 = 191\ °\text{C}$$

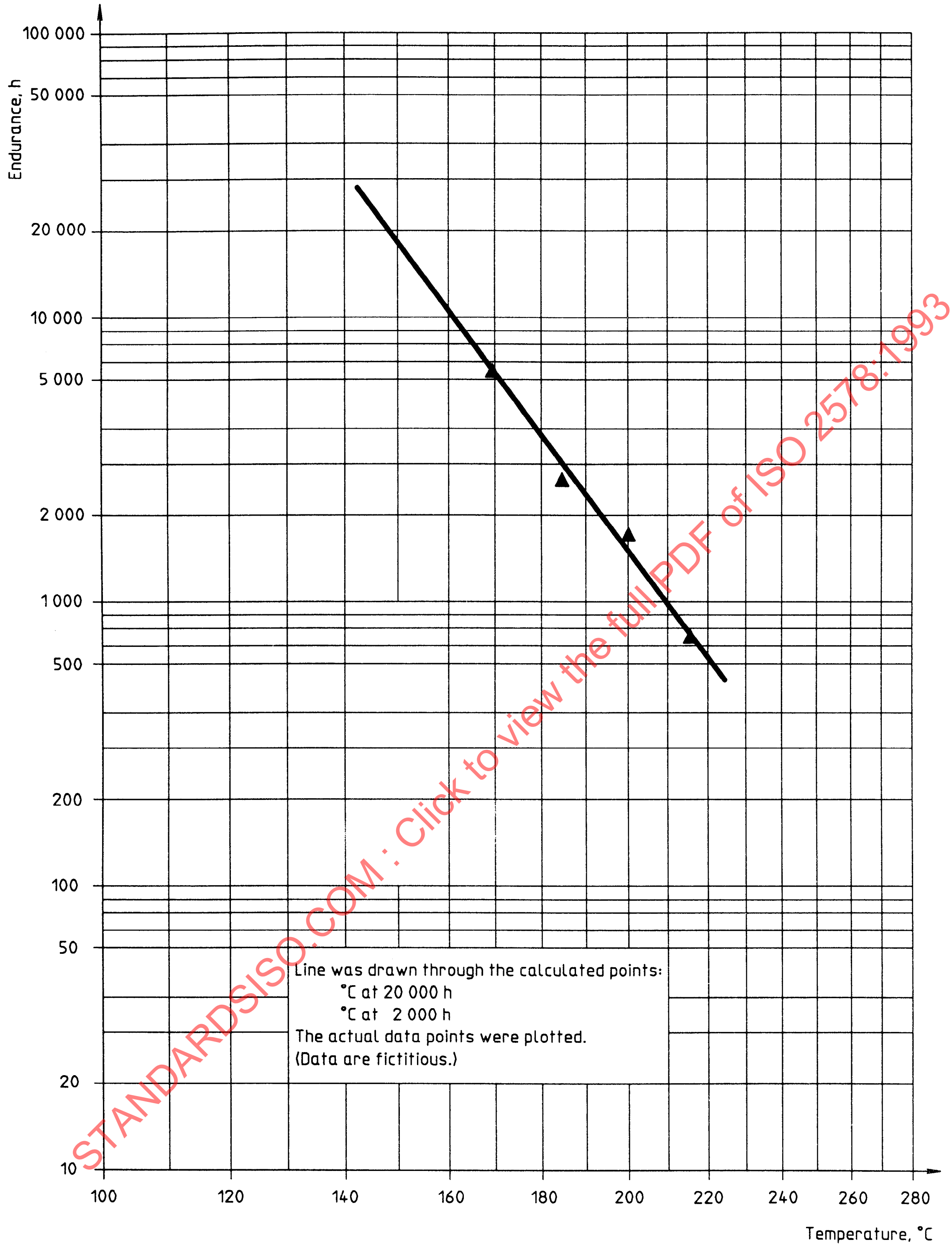

Figure A.1 — Plot of regression line based on sample calculation (see table A.2)