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

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

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FOREWORD

ISO (the International Organization for Standardization) is a worldwide federation of national standards institutes (ISO Member Bodies). The work of developing International Standards is carried out through ISO Technical Committees. Every Member Body interested in a subject for which a Technical Committee has been set up 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.

Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2578 was drawn up by Technical Committee ISO/TC 61, *Plastics*, and circulated to the Member Bodies in January 1974.

It has been approved by the Member Bodies of the following countries:

Austria	Hungary	South Africa, Rep. of
Belgium	India	Spain
Brazil	Israel	Sweden
Canada	Italy	Switzerland
Czechoslovakia	Japan	Turkey
Egypt, Arab Rep. of	Netherlands	United Kingdom
Finland	New Zealand	U.S.A.
France	Portugal	U.S.S.R.
Germany	Romania	

No Member Body expressed disapproval of the document.

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

0 INTRODUCTION

During the preparation of this International Standard, account was taken of the contents of IEC Publication 216.

1 SCOPE AND FIELD OF APPLICATION

1.1 This International Standard specifies the principles and procedures for evaluating the time-temperature limits of plastics exposed to the action of heat for long periods.

1.2 When the term "time-temperature limit" is not qualified, it refers to tests made in air, avoiding any other influence such as applied stress. Similar time-temperature limits may be determined in other environments or under applied stress, or both, the time-temperature limit being qualified accordingly.

1.3 Evaluation of these limits provides information on the possibilities of using a material under similar conditions for a specific purpose.

2 REFERENCES

IEC Publication 216, *Guide for the preparation of test procedures for evaluating the thermal endurance of electrical insulating materials.*

ISO/R 291, *Plastics – Standard atmospheres for conditioning and testing.*

3 DEFINITION

For the purpose of this International Standard, the following definition applies:

time-temperature limit: The highest temperature to which the particular plastic can be subjected for a determined time before the numerical value of a chosen characteristic reaches a predetermined critical (threshold) value.

4 PRINCIPLE

4.1 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, giving the time limit for that particular temperature.

Further specimens are exposed at at least two other temperatures and the variations in the relevant characteristic determined.

4.2 The time limits obtained are plotted as a function of the temperature. The intersection of this curve with the chosen time limit (in general 20 000 h) gives the time-temperature limit sought.

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

5 SELECTION OF TEST

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

6 SELECTION OF END POINT

For the selection of the end point, two factors have to be agreed:

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 – Other times (shorter or longer than 20 000 h) can 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 The total number of test specimens needed depends on :

- a) the required number of test specimens according to the specifications given 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 to be made.

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. Likewise the highest temperature shall be chosen so that the time taken to reach the threshold value is not shorter than 100 h.

8.2 If the temperature limit sought is intended for a time other than 20 000 h (see note, clause 6), 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 the temperature for exposure involves predicting or knowing beforehand the approximate temperature range in which the time-temperature limit of the material to be tested is located. With no previous knowledge of the material, exploratory tests must be made. This information will assist in selecting the exposure temperatures best suited for the evaluation of the material.

8.4 When evaluating the test results, a straight line may often be obtained when these data are plotted on graph paper constructed so that equal increments on the axes of abscissae and of ordinates respectively represent equal increments of $1/K$ (K = absolute temperature in kelvins) and $\log L$ (L = median or average life of specimens). This straight line indicates that the material deteriorates according to the Arrhenius equation. This means that, when raising the temperature in steps corresponding to equal increments of $1/K$ comparable ageing effects can be expected.

8.5 It is therefore convenient to choose testing temperatures corresponding to equal increments of $1/K$.

8.6 This can be done with the help of the plot of $1/K$ against temperature θ in Celsius degrees in figure 1, by measuring equal intervals on the $1/K$ -axis and deriving from them the corresponding temperatures on the θ -axis.

9 AGEING OVENS

It is essential that specimens be aged in ovens that will maintain the average temperature of the specimens within the following limits :

Temperature °C		Tolerance °C
over	up to	
—	100	± 2
100	200	± 3
200	400	± 4
400	—	± 5

When the tests are made in air, it is usually necessary to use a forced air circulation oven to achieve the required control of temperature. It is preferable that the introduction of fresh air in the oven be controlled, as in some cases the thermal degradation may be influenced by an abnormal supply of fresh air.

The temperature should preferably be recorded, either continuously or at frequent intervals.

When the tests are made in other atmospheres the method of temperature control has to be adapted to that particular atmosphere.

10 PROCEDURE

10.1 In addition to the specimens to be exposed, an adequate number of test specimens shall be kept as reference. Store these in the appropriate controlled atmosphere (see ISO/R 291).

10.2 At the beginning of the test procedure make the first reference test run with the required number of test specimens, conditioned and tested according to the appropriate standard method.

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 heating period, condition, if necessary, the test specimens to be examined, together with the reference specimens under the appropriate controlled conditions (see ISO/R 291) and then carry out the tests by the previously chosen methods.

The examination of reference specimens at each heating period provides an evaluation of the effect of storage time at ambient laboratory temperature on the selected characteristics.

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 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 characteristic against time on a logarithmic scale. Determine the values t_1 , t_2 , t_3 . . . by means of interpolation. An example of such a graph is given in figure 2.

11.2 Plot the log of the time at which the threshold value is reached for each of the testing temperatures against the corresponding temperature.

Construct a line of best fit through the plotted points. This may be done using established statistical methods. Extrapolate the line obtained to the time limit chosen (generally 20 000 h) giving the temperature limit sought for the time-limit chosen. An example of such a plot is given in figure 3.

NOTES

1 When the temperature scale is chosen in such a way that equal intervals correspond to equal intervals of $1/K$, then the various points obtained will be found to lie on a straight line in case a linear dependence is evident.

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

12 TEST REPORT

The test report shall include the following particulars :

- a) complete identification of the material tested;
- b) precise details of the ageing conditions, if these are other than the exposure of unstressed specimens to hot air;
- c) selected characteristic, with reference to the corresponding document on testing;
- d) threshold value of the selected characteristic;
- e) shape, dimensions and method of preparation of the test specimens, with reference to the relevant document;
- f) conditioning;
- g) type of ovens, with details of rate and direction of airflow;
- h) times and temperatures of exposure in ovens;
- i) if necessary, the curves giving for each temperature the values of the characteristic against time (curve 11.1);
- j) if applicable, a graph of log life plotted against temperature, with reference to the statistical method used;
- k) time-temperature limit for the selected characteristic.

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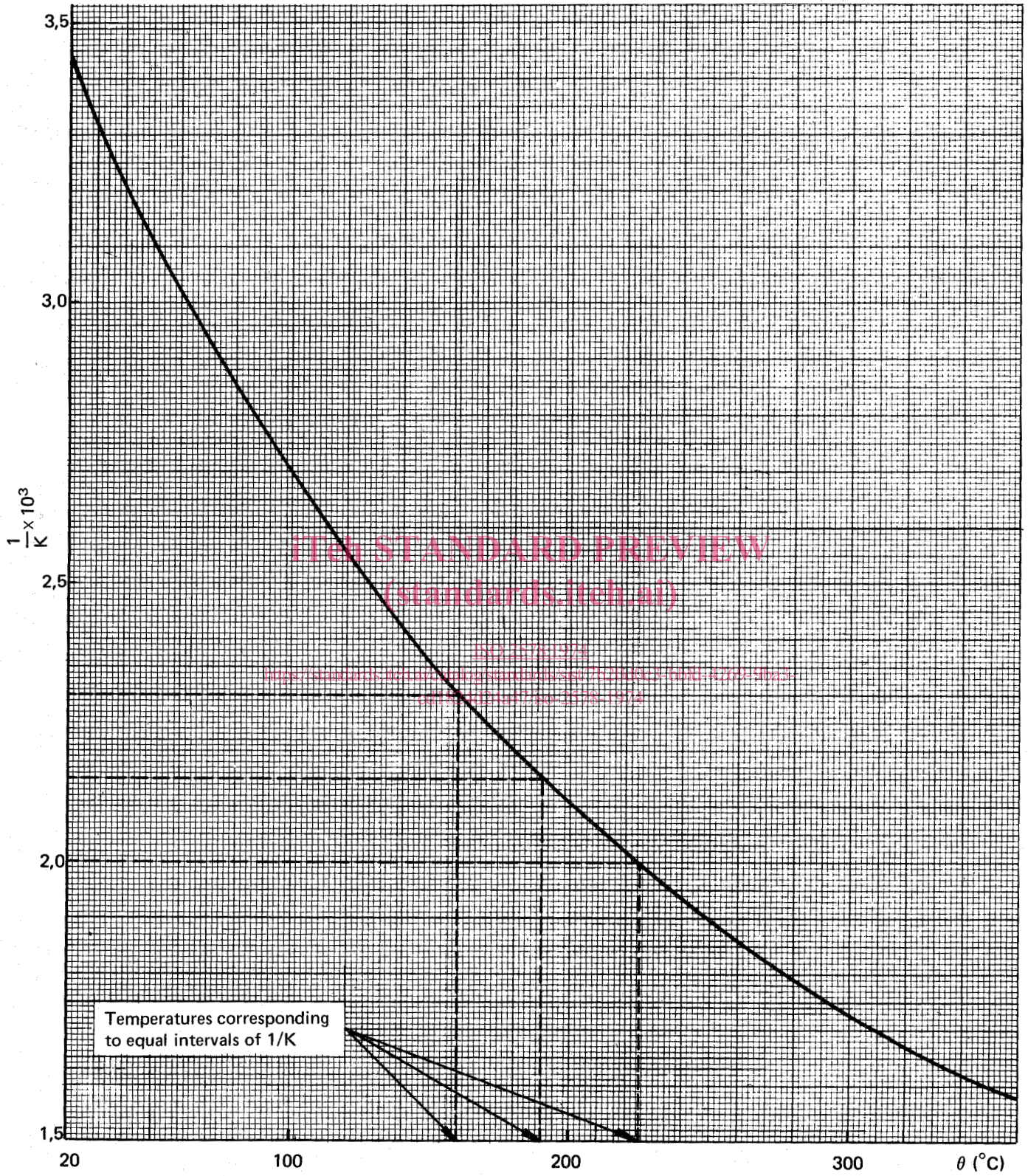
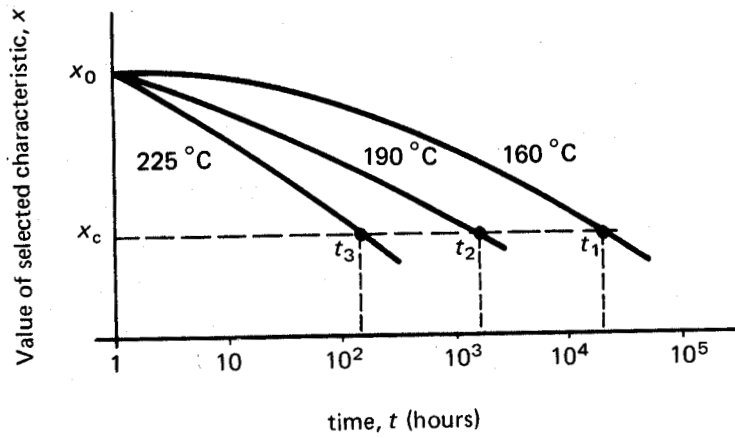


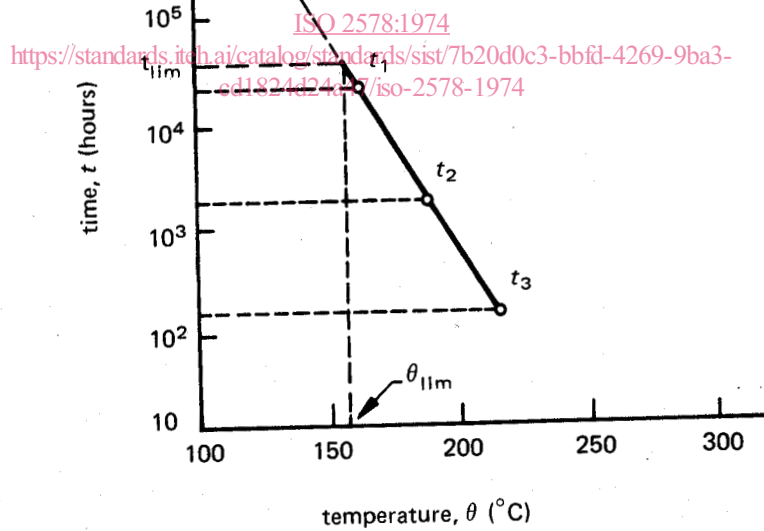
FIGURE 1 — Curve of the value of $1/K$ as a function of the temperature in Celsius degrees, K being the expression of the same temperature in kelvins



x_0 is the numerical value of the chosen characteristic at the beginning of the test;
 x_c is the critical (threshold) value of the same characteristic.

FIGURE 2

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t_{lim} is the time limit for extrapolation (here 20 000 h);
 θ_{lim} is the temperature limit sought.

FIGURE 3

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