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

ICS 83.080.01

Descriptors : plastics, environmental testing, thermal testing, temperature, thermal endurance tests

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Foreword

This translation has been made based on the original Japanese Industrial Standard established by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law:

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

Introduction This Japanese Industrial Standard has been prepared based on ISO 2578 : 1993, *Plastics—Determination of time-temperature limits after prolonged exposure to heat* without modifying its technical content and structure.

1 Scope

1.1 This Japanese Industrial 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 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 Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

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

IEC 60216-1 : 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 60216-2 : 1990 *Guide for the determination of thermal endurance properties of electrical insulating materials—Part 2 : Choice of the test criteria*

- IEC 60216-3-1 : 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 60216-3-3 : —* *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 60216-4-1 : 1990 *Guide for the determination of thermal endurance properties of electrical insulating materials—Part 4 : Ageing ovens—Section 1 : Single-chamber ovens*
- IEC 60216-5 : 1990 *Guide for the determination of thermal endurance properties of electrical insulating materials—Part 5 : Guidelines for the application of thermal endurance characteristics*

Note * To be published.

3 Definitions For the purposes of this 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 plot) 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 (K).

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.

Remarks 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 thermal endurance 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 and 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 least two other temperatures and the variations in the relevant characteristic determined. It is recommended to heat-age test specimens at three temperatures 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 behavior 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 60216), 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, and 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 between the parties concerned:

- 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;

Remarks 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 number (n) of test specimens needed depends on

- a : the number of test specimens required for one test, according to the test specifications given for the relevant test method;
- b : the number of tests necessary to determine the end-point at one exposure temperature;
- c : the number of exposure temperatures;
- d : the number of specimens needed for the initial test before heat ageing.

Thus the total number of specimens is:

$$n = a \cdot b \cdot c + d$$

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

Remarks 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 be not more than 25 °C above the anticipated TI.