BS EN 60216-1:2013

BSI Standards Publication

Electrical insulating materials — Thermal endurance properties

Part 1: Ageing procedures and evaluation of test results

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National foreword

This British Standard is the UK implementation of EN 60216-1:2013. It is identical to IEC 60216-1:2013. Together with BS EN 60216-8:2013, it supersedes BS EN 60216-1:2002 which is withdrawn.

The "simplified method" has been removed from IEC 60216-1:2002 in the new edition (IEC 60216-1:2013) and now forms IEC 60216-8:2013, "Instructions for calculating thermal endurance characteristics using simplified procedures".

The UK participation in its preparation was entrusted to Technical Committee GEL/112, Evaluation and qualification of electrical insulating materials and systems.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

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Foreword

The text of document 112/235/FDIS, future edition 6 of IEC 60216-1, prepared by IEC/TC 112 "Evaluation and qualification of electrical insulating materials and systems" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 60216-1:2013.

The following dates are fixed:

• latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2014-01-19
• latest date by which the national standards conflicting with the document have to be withdrawn	(dow)	2016-04-19

This document supersedes EN 60216-1:2001 (PART).

EN 60216-1:2013 includes the following significant changes with respect to EN 60216-1:2001:

This edition constitutes an editorial revision where the simplified method has been removed and now forms Part 8 of the EN 60216 Series: Instructions for calculating thermal endurance characteristics using simplified procedures.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CENELEC [and/or CEN] shall not be held responsible for identifying any or all such patent rights.

Endorsement notice

The text of the International Standard IEC 60216-1:2013 was approved by CENELEC as a European Standard without any modification.

In the official version, for Bibliography, the following notes have to be added for the standards indicated:

ISO 291	NOTE	Harmonised as EN ISO 291.
ISO 2578:1993	NOTE	Harmonised as EN ISO 2578:1998 (not modified).

Annex ZA
(normative)

Normative references to international publications with their corresponding European publications

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

Publication	Year	Title	EN/HD	Year
IEC 60212	-	Standard conditions for use prior to and during the testing of solid electrical insulating materials	EN 60212	-
IEC 60216-2	-	Electrical insulating materials - Thermal endurance properties - Part 2: Determination of thermal endurance properties of electrical insulating materials - Choice of test criteria	EN 60216-2	-
IEC 60216-3 + corr. December	2006 2009	Electrical insulating materials - Thermal endurance properties - Part 3: Instructions for calculating thermal endurance characteristics	EN 60216-3	2006
IEC 60216-4	Series	Electrical insulating materials - Thermal endurance properties	EN 60216-4	Series
IEC 60216-4-1	-	Electrical insulating materials - Thermal endurance properties - Part 4-1: Ageing ovens - Single-chamber ovens	EN 60216-4-1	-
IEC 60216-8	2013	Electrical insulating materials - Thermal endurance properties - Part 8: Instructions for calculating thermal endurance characteristics using simplified procedures	EN 60216-8	2013
IEC 60493-1	2011	Guide for the statistical analysis of ageing test data - Part 1: Methods based on mean values of normally distributed test results	-	-

CONTENTS

INTRODUCTION

The listing of the thermal capabilities of electrical insulating materials, based on service experience, was found to be impractical, owing to the rapid development of polymer and insulation technologies and the long time necessary to acquire appropriate service experience. Accelerated ageing and test procedures were therefore required to obtain the necessary information. The IEC 60216 series has been developed to formalize these procedures and the interpretation of their results.

Physico-chemical models postulated for the ageing processes led to the almost universal assumption of the Arrhenius equations to describe the rate of ageing. Out of this arose the concept of the temperature index (TI) as a single-point characteristic based upon accelerated ageing data. This is the numerical value of the temperature in °C at which the time taken for deterioration of a selected property to reach an accepted end-point is that specified (usually 20 000 h).

NOTE The term Arrhenius is widely used (and understood) to indicate a linear relationship between the logarithm of a time and the reciprocal of the thermodynamic (absolute or Kelvin) temperature. The correct usage is restricted to such a relationship between a reaction rate constant and the thermodynamic temperature. The common usage is employed throughout this standard.

The large statistical scatter of test data which was found, together with the frequent occurrence of substantial deviations from the ideal behavior, demonstrated the need for tests to assess the validity of the basic physico-chemical model. The application of conventional statistical tests, as set out in IEC 60493-1, fulfilled this requirement, resulting in the "confidence limit", (TC) of TI, but the simple, single-point TI was found inadequate to describe the capabilities of materials. This led to the concept of the "Thermal Endurance Profile" (TEP), incorporating the temperature index, its variation with specified ageing time, and a confidence limit.

A complicating factor is that the properties of a material subjected to thermal ageing may not all deteriorate at the same rate, and different end-points may be relevant for different applications. Consequently, a material may be assigned more than one temperature index, derived, for example, from the measurement of different properties and the use of different end-point times.

It was subsequently found that the statistical confidence index included in the TEP was not widely understood or used. However, the statistical tests were considered essential, particularly after minor modifications to make them relate better to practical circumstances: the concept of the halving interval (HIC) was introduced to indicate the rate of change of ageing time with temperature. TEP was then abandoned, with the TI and HIC being reported in a way which indicated whether or not the statistical tests had been fully satisfied. At the same time, the calculation procedures were made more comprehensive, enabling full statistical testing of data obtained using a diagnostic property of any type, including the particular case of partially incomplete data. Simultaneously with the development of the IEC 60216 series, other standards were being developed in ISO, intended to satisfy a similar requirement for plastics and rubber materials. These are ISO 2578 and ISO 11346 respectively, which use less rigorous statistical procedures and more restricted experimental techniques. A simplified calculation procedure is described in IEC 60216-8.

ELECTRICAL INSULATING MATERIALS – THERMAL ENDURANCE PROPERTIES –

Part 1: Ageing procedures and evaluation of test results

1 Scope

This part of IEC 60216 specifies the general ageing conditions and procedures to be used for deriving thermal endurance characteristics and gives guidance in using the detailed instructions and guidelines in the other parts of the standard.

Although originally developed for use with electrical insulating materials and simple combinations of such materials, the procedures are considered to be of more general applicability and are widely used in the assessment of materials not intended for use as electrical insulation.

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 relationship).

For the valid application of the standard, no transition, in particular no first-order transition should occur in the temperature range under study.

Throughout the rest of this standard the term "insulating materials" is always taken to mean "insulating materials and simple combinations of such materials".

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60212, *Standard conditions for use prior to and during the testing of solid electrical insulating materials*

IEC 60216-2, *Electrical insulating materials – Thermal endurance properties – Part 2: Determination of thermal endurance properties of electrical insulating materials – Choice of test criteria*

IEC 60216-3:2006, *Electrical insulating materials – Thermal endurance properties – Part 3: Instructions for calculating thermal endurance characteristics*

IEC 60216-4 (all Parts 4), *Electrical insulating materials – Thermal endurance properties – Part 4: Ageing ovens*

IEC 60216-4-1, *Electrical insulating materials – Thermal endurance properties – Part 4-1: Ageing ovens – Single-chamber ovens*

IEC 60216-8, *Electrical insulating materials – Thermal endurance properties – Part 8: Instructions for calculating thermal endurance characteristics using simplified procedures*[1]

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[1] To be published.

IEC 60493-1:2011, *Guide for the statistical analysis of ageing test data – Part 1: Methods based on mean values of normally distributed test results*

3 Terms, definitions, symbols and abbreviations

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply:

3.1.1
temperature index
TI
numerical value of the temperature in degrees Celsius derived from the thermal endurance relationship at a time of 20 000 h (or other specified time)

3.1.2
halving interval
HIC
numerical value of the temperature interval in Kelvin which expresses the halving of the time to end-point taken at the temperature equal to TI

[SOURCE: IEC 60050-212:2010, definition 212-12-13, modified – "equal to TI" replaces "corresponding to the temperature index or the relative temperature index"]

3.1.3
thermal endurance graph
graph in which the logarithm of the time to reach a specified end-point in a thermal endurance test is plotted against the reciprocal thermodynamic (absolute) test temperature

[SOURCE: IEC 60050-212:2010, definition 212-12-10, modified – "insertion of word "(absolute)"]

3.1.4
thermal endurance graph paper
graph paper having a logarithmic time scale as the ordinate, graduated in powers of ten (from 10 h to 100 000 h is often a convenient range)

Note 1 to entry: Values of the abscissa are proportional to the reciprocal of the thermodynamic (absolute) temperature. The abscissa is usually graduated in a non-linear (Celsius) temperature scale oriented with temperature increasing from left to right.

3.1.5
ordered data
set of data arranged in sequence so that, in the appropriate direction through the sequence, each member is greater than, or equal to, its predecessor

Note 1 to entry: Ascending order in this standard implies that the data is ordered in this way, the first order-statistic being the smallest.

3.1.6
order-statistics
each individual value in a set of ordered data is referred to as an order-statistics identified by its numerical position in the sequence

3.1.7
incomplete data
ordered data, where the values above and/or below defined points are not known

3.1.8
censored data
incomplete data, where the number of unknown values is known

Note 1 to entry: If the censoring is begun above/below a specified numerical value, the censoring is of type 1. If above/below a specified order-statistic, it is of type 2. This standard is concerned only with type 2.

3.1.9
degrees of freedom
number of data values minus the number of parameter values

3.1.10
variance of a data set
sum of the squares of the deviations of the data from a reference level defined by one or more parameters, divided by the number of degrees of freedom

Note 1 to entry: The reference level may for example, be a mean value (one parameter) or a line (two parameters, slope and intercept).

3.1.11
covariance of data sets
for two sets of data with equal numbers of elements where each element in one set corresponds to one in the other, the sum of the products of the deviations of the corresponding members from their set means, divided by the number of degrees of freedom

3.1.12
regression analysis
process of deducing the best-fit line expressing the relation of corresponding members of two data groups by minimizing the sum of squares of deviations of members of one of the groups from the line

Note 1 to entry: The parameters are referred to as the regression coefficients.

3.1.13
correlation coefficient
number expressing the completeness of the relation between members of two data sets, equal to the covariance divided by the square root of the product of the variances of the sets

Note 1 to entry: The value of its square is between 0 (no correlation) and 1 (complete correlation).

3.1.14
confidence limit
TC
statistical parameter, calculated from the test data, which with 95 % confidence constitutes a lower limit for the true value of the temperature index estimated by TI

Note 1 to entry: 95 % confidence implies that there is only 5 % probability that the true value of the temperature index is actually smaller than TC.

Note 2 to entry: In other connections, confidence values other than 95 % may sometimes be used; for example, in the linearity test for destructive test data.

3.1.15
destructive test
diagnostic property test, where the test specimen is irreversibly changed by the property measurement, in a way which precludes a repeated measurement on the same specimen

3.1.16
non-destructive test
diagnostic property test, where the properties of the test specimen are not permanently changed by the measurement, so that a further measurement on the same specimen may be made after appropriate treatment

3.1.17
proof test
diagnostic property test, where each test specimen is, at the end of each ageing cycle, subjected to a specified stress, further ageing cycles being conducted until the specimen fails on testing

3.1.18
temperature group
test group of specimens
number of specimens being exposed together to the same temperature ageing in the same oven

Note 1 to entry: Where there is no risk of ambiguity, either temperature groups or test groups may be referred to simply as groups.

3.1.19
test group
test group of specimens
number of specimens removed together from a temperature group (as above) for destructive testing

3.1.20
end point
property level that is effected by practical application to the equipment in the thermal endurance test

3.2 Symbols and abbreviations

Symbol	Meaning
a,b	Regression coefficients
$n_{a,b,c,d}$	Numbers of specimens for destructive tests
n	Number of y-values
N	Total number of test specimens
m_i	Number of specimens in temperature group i (censored data)
r	Correlation coefficient
F	Fisher distributed stochastic variable
x	Reciprocal thermodynamic temperature ($1/\Theta$)
y	Logarithm of time to end-point
ϑ	Temperature, °C
Θ	Temperature, thermodynamic (Kelvin)
Θ_0	Value in Kelvin (0 °C = 273,15 K)
τ	Time (to end-point)
χ^2	χ^2-distributed stochastic variable
TI	Temperature index
TC	Lower 95 % confidence limit of TI
HIC	Halving interval at temperature equal to TI
RTI	Relative temperature index

4 Synopsis of procedures – Full procedures

The standardized procedure for the evaluation of thermal properties of a material consists of a sequence of steps, as follows.

It is strongly recommended that the full evaluation procedure, as described below and in 5.1 to 5.8, be used.

a) Prepare suitable specimens appropriate for the intended property measurements (see 5.3).
b) Subject groups of specimens to ageing at several fixed levels of elevated temperature, either continuously, or cyclically for a number of periods between which the specimens are normally returned to room temperature or another standard temperature (see 5.5).
c) Subject specimens to a diagnostic procedure in order to reveal the degree of ageing. Diagnostic procedures may be non-destructive or destructive determinations of a property or potentially destructive proof tests (see 5.1 and 5.2).
d) Extend the continuous heat exposure or the thermal cycling until the specified end-point, i.e. failure of specimens or a specified degree of change in the measured property, is reached (see 5.1, 5.2 and 5.5).
e) Report the test results, showing the kind of ageing procedure (continuous or cyclic) and diagnostic procedure (see under item c) above); the ageing curves, or time or number of cycles to reach the end-point, for each specimen.
f) Evaluate these data numerically and present them graphically, as explained in 6.1 and 6.8.
g) Express the complete information in abbreviated numerical form, as described in 6.2 by means of the temperature index and halving interval.

The full experimental and evaluation procedures are given in Clause 5 and as far as 6.8.

A simplified procedure is given in IEC 60216-8.

5 Detailed experimental procedures

5.1 Selection of test procedures

5.1.1 General considerations

Each test procedure should specify the shape, dimensions and number of the test specimens, the temperatures and times of exposure, the property to which TI is related, the methods of its determination, the end-point, and the derivation of the thermal endurance characteristics from the experimental data.

The chosen property should reflect, in a significant fashion if possible, a function of the material in practical use. A choice of properties is given in IEC 60216-2.

To provide uniform conditions, the conditioning of specimens after removal from the oven and before measurement may need to be specified.

5.1.2 Selection of test properties for TI

If IEC material specifications are available, property requirements in terms of acceptable lower limits of TI values are usually given. If such material specifications are not available, a selection of properties and methods for the evaluation of thermal endurance is given in IEC 60216-2. (If such a method cannot be found, an international, national, or institution standard, or a specially devised method should be used, and in that order of preference.)

5.1.3 Determination of TI for times other than 20 000 h

In the majority of cases, the required thermal endurance characteristics are for a projected duration of 20 000 h. However, there is often a need for such information related to other longer or shorter times. In cases of longer times, for example, the times given as requirements or recommendations in the text of this standard (for example, 5 000 h for the minimum value of the longest time to end-point) shall be increased in the ratio of the actual specification time to 20 000 h. In the same way, the ageing cycle durations should be changed in approximately the same ratio. The temperature extrapolation again shall not exceed 25 K. In cases of shorter specification times, the related times may be decreased in the same ratio if necessary.

Particular care will be needed for very short specification times, since the higher ageing temperatures may lead into temperature regions which include transition points, for example, glass transition temperature or partial melting, with consequent non-linearity. Very long specification times may also lead to non-linearity (see also Annex A).

5.2 Selection of end-points

The thermal endurance of materials may need to be characterized by different endurance data (derived using different properties and/or end-points), in order to facilitate the adequate selection of the material in respect of its particular application in an insulation system. See IEC 60216-2.

There are two alternative ways in which the end-point may be defined:

a) As a percentage increase or decrease in the measured value of the property from the original level. This approach will provide comparisons among materials but bears a poorer relationship than item b) to the property values required in normal service. For the determination of the initial value, see 5.4.

b) As a fixed value of the property. This value might be selected with respect to usual service requirements. End-points of proof tests are predominantly given in the form of fixed values of the property.

The end-point should be selected to indicate a degree of deterioration of the insulating material which has reduced its ability to withstand a stress encountered in actual service in an insulation system. The degree of degradation indicated as the end-point of the test should be related to the allowable safe value for the material property which is desired in practice.

5.3 Preparation and number of test specimens

5.3.1 Preparation

The specimens used for the ageing test should constitute a random sample from the population investigated and are to be treated uniformly.

The material specifications or the test standards will contain all necessary instructions for the preparation of specimens.

The thickness of specimens is in some cases specified in the list of property measurements for the determination of thermal endurance. See IEC 60216-2. If not, the thickness shall be reported. Some physical properties are sensitive even to minor variations of specimen thickness. In such cases, the thickness after each ageing period may need to be determined and reported if required in the relevant specification.

The thickness is also important because the rate of ageing may vary with thickness. Ageing data of materials with different thicknesses are not always comparable. Consequently, a material may be assigned more than one thermal endurance characteristic derived from the measurement of properties at different thicknesses.

The tolerances of specimen dimensions should be the same as those normally used for general testing; where specimen dimensions need smaller tolerances than those normally used, these special tolerances should be given. Screening measurements ensure that specimens are of uniform quality and typical of the material to be tested.

Since processing conditions may significantly affect the ageing characteristics of some materials, it shall be ensured that, for example, sampling, cutting sheet from the supply roll, cutting of anisotropic material in a given direction, molding, curing, pre-conditioning, are performed in the same manner for all specimens.

5.3.2 Number of specimens

5.3.2.1 General

The accuracy of endurance test results depends largely on the number of specimens aged at each temperature. Instructions for an adequate number of specimens are given in IEC 60216-3. Generally, the following instructions (5.3.2.1 to 5.3.2.3), which influence the testing procedure given in 5.8, shall apply.

It is good practice to prepare additional specimens, or at least to provide a reserve of the original material batch from which such specimens may subsequently be prepared. In this way, any required ageing of additional specimens in case of unforeseen complications will introduce a minimum risk of producing systematic differences between groups of specimens. Such complications may arise, for example, if the thermal endurance relationship turns out to be non-linear, or if specimens are lost due to thermal runaway of an oven.

Where the test criterion for non-destructive or proof tests is based upon the initial value of the property, this should be determined from a group of specimens of at least twice the number of specimens in each temperature group. For destructive tests, see 5.3.2.4.

5.3.2.2 Number of specimens for non-destructive tests

For each exposure temperature, in most cases a group of five specimens will be adequate. However, further guidance will be found in IEC 60216-3.

5.3.2.3 Number of specimens for proof tests

In most cases a group of at least 11 specimens for each exposure temperature will be required. For graphical derivation and in some other cases the treatment of data may be simpler if the number of specimens in each group is odd. Further guidance will be found in IEC 60216-3.

5.3.2.4 Number of specimens for destructive tests

This number (N) is derived as follows: $N = n_a \times n_b \times n_c + n_d$

where

n_a is the number of specimens in a test group undergoing identical treatment at one temperature and discarded after determination of the property (usually five);

n_b is the number of treatments, i.e. total number of exposure times, at one temperature;

n_c is the number of ageing temperature levels;

n_d is the number of specimens in the group used to establish the initial value of the property. Normal practice is to select $n_d = 2n_a$ when the diagnostic criterion is a percentage change of the property from its initial level. When the criterion is an absolute property level, n_d is usually given the value of zero, unless reporting of the initial value is required.

5.4 Establishment of initial property value

Select the specimens for the determination of the initial value of the property to constitute a random subset of those prepared for ageing. Before determining the property value, these specimens shall be conditioned by exposure to the lowest level of ageing temperature of the test (see 5.5) for two days (48 h ± 6 h).

In some cases (for example, very thick specimens), times greater than two days may be necessary to establish a stable value.

Unless otherwise stated in the method for determining the diagnostic property (for example, parts of material specifications dealing with methods of test, or a method listed in IEC 60216-2), the initial value is the arithmetic mean of the test results.

5.5 Exposure temperatures and times

For TI determinations, test specimens should be exposed to not less than three, preferably at least four, temperatures covering a sufficient range to demonstrate a linear relationship between time to end-point and reciprocal thermodynamic (absolute) temperature.

To reduce the uncertainties in calculating the appropriate thermal endurance characteristic, the overall temperature range of thermal exposure needs to be carefully selected, observing the following requirements (if the required thermal endurance characteristics are for a projected duration of 20 000: see also 5.1.3):

a) the lowest exposure temperature shall be one which will result in a mean or median time to end-point of more than 5 000 h when determining TI (see also 5.1.3);

b) the extrapolation necessary to establish TI shall not be more than 25 K;

c) the highest exposure temperature shall be one which will result in a mean or median time to end-point of more than 100 h (if possible, less than 500 h).

For some materials, it is not possible to achieve a time to end-point of less than 500 h while retaining satisfactory linearity. However, it is important that a smaller range of mean times to end-point will lead to a larger confidence interval of the result for the same data dispersion.

Relevant and detailed instructions on how to proceed using non-destructive, proof or destructive test criteria are provided in 5.8.

Table 1 gives guidance in making initial selections.

A number of recommendations and suggestions, useful in establishing times and temperatures, will be found in Annex B.

5.6 Ageing ovens

Throughout the heat ageing period, ageing ovens shall maintain, in that part of the working space where specimens are placed, a temperature with tolerances as given in the IEC 60216-4 series. Unless otherwise specified, IEC 60216-4-1 shall apply.

The circulation of the air within the oven and the exchange of the air content should be adequate to ensure that the rate of thermal degradation is not influenced by accumulation of decomposition products or oxygen depletion (see 5.7).

5.7 Environmental conditions

5.7.1 General

The effects of special environmental conditions such as extreme humidity, chemical contamination or vibration in many cases may be more appropriately evaluated by insulation systems tests. However, environmental conditioning, the influence of atmospheres other than air and immersion in liquids such as oil may be important, but these are not the concern of this standard.

5.7.2 Atmospheric conditions during ageing

Unless otherwise specified, ageing shall be carried out in ovens operating in the normal laboratory atmosphere. However, for some materials very sensitive to the humidity in the ovens, more reliable results are obtained when the absolute humidity in the ageing oven room is controlled and equal to the absolute humidity corresponding to standard atmosphere B according to IEC 60212. This, or other specified conditions, shall then be reported.

5.7.3 Conditions for property measurement

Unless otherwise specified, the specimens shall be conditioned before measurement and measured under conditions as specified in the material standard specification.

5.8 Procedure for ageing

5.8.1 General

This subclause relates to the basic procedures for using

a) a non-destructive test,

b) a proof test,

c) a destructive test.

Prepare a number of specimens following the instructions in 5.3. If necessary, determine the initial value of the property as specified in 5.4. Divide the specimens by random selection into as many groups as there are exposure temperatures.

Establish the exposure temperatures and times in accordance with the instructions of 5.5 (see also Annex B).

Place one group for exposure in each of the ovens complying with 5.6, maintained as closely as possible to the temperatures selected from Table 1.

NOTE 1 It is suggested that individual specimens be identified to simplify their return to the correct oven after each test.

NOTE 2 Attention should be given to the recommendation in 5.3 to prepare an extra group of reserve specimens for the purposes stated in Annex B, in particular, to be able to initiate early the ageing of new specimens at an additional level of temperature.

5.8.2 Procedure using a non-destructive test

At the end of each cycle, remove the group of specimens from the respective oven and allow them to cool to room temperature unless otherwise specified (see 5.7). Some test properties may require measurement at the oven temperature, in which case the ageing is continuous.

Apply the appropriate test to each specimen and then return the group to the oven from which they came, at the same temperature as before, and expose for a further cycle. Continue the cycles of temperature exposure, cooling and application of the test until the average measured value for the specimens in the group has reached the end-point specified and provided at least one point beyond the end-point.

Evaluate the results as listed in 6.1 and detailed in IEC 60216-3 and report them as specified in 6.8.

5.8.3 Procedure using a proof test

Specimens for testing by a proof-test procedure shall be drawn at random from specimens which have successfully withstood screening by the proof test.

At the end of each cycle, remove all specimens from the oven. After each removal, allow the specimens to cool to room temperature and then subject each one to the specified proof test. Return specimens which have withstood the proof test to the oven from which they came, at the same temperature as before, and expose for a further cycle.

Continue the cycles of temperature exposure, cooling and application of the proof test until the failure of the median specimen number $(m + 1)/2$ if the number of specimens (m) is odd, or $(m/2 + 1)$ if the number of specimens is even. If the results show that this time to end-point is likely to be reached in about 10 periods of exposure, there is no need to alter the period of exposure originally selected. If the results do not show this, the period may be changed so that the median result may be expected in at least seven cycles (preferably about 10) provided this change in cycle time is made before the fourth cycle.

The cycles of temperature exposure may be continued until all specimens have failed, so that a more complete statistical analysis may be made (see IEC 60216-3).

Evaluate the results, as listed in 6.1 and detailed in IEC 60216-3, and report them as specified in 6.8.

5.8.4 Procedure using a destructive test

For each oven, select at random a test group of the assigned number (= n_a, see 5.3.2.4) of specimens and remove them from the oven after lengths of time chosen in such a way that the exposure times form a suitable sequence. See 5.5, Annex B and Table 1.

After each removal, allow the group of specimens to cool to room temperature unless otherwise specified. For materials in which a significant variation of properties with temperature or humidity is expected, unless otherwise specified, condition the specimens overnight in standard atmosphere B of IEC 60212. Test the specimens and plot the results and the arithmetic mean of the results (or a suitable transform thereof) against the logarithm of exposure time as given in IEC 60216-3.

Evaluate the results, as listed in 6.1 and detailed in Clause 6 of IEC 60216-3:2006, and report as specified in 6.8.

6 Evaluation

6.1 Numerical analysis of test data

The numerical calculation procedures for the full analysis of data are specified in 6.3 to 6.7. The analysis of TI data is based on the assumption that there is a linear relation between the logarithm of the time to end-point and the reciprocal of the thermodynamic ageing temperature.

The method of evaluation of TI results is by the numerical procedure detailed in IEC 60216-3 together with a graphical presentation as shown in Figure 1.

A simplified procedure is available in IEC 60216-8.

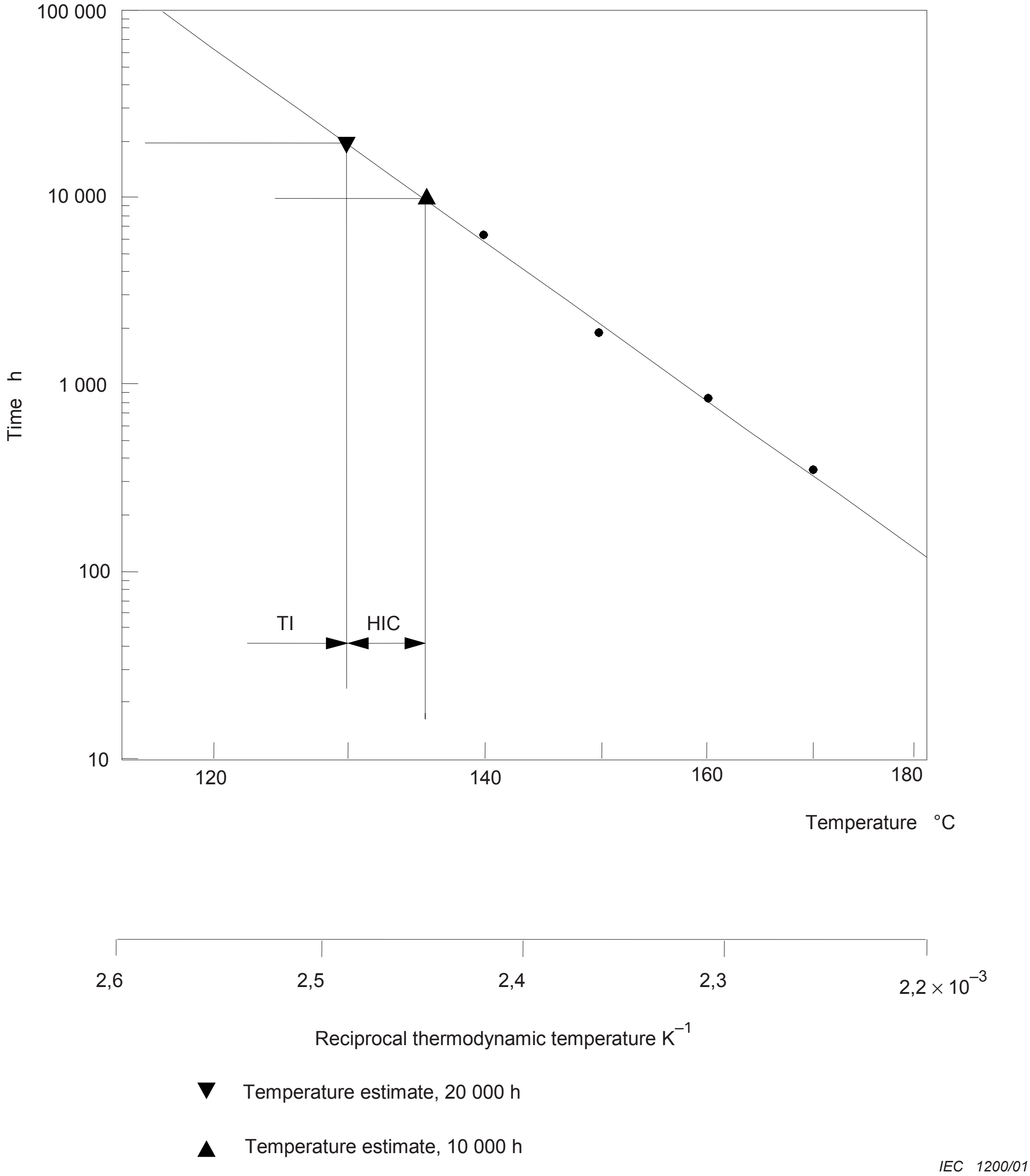


Figure 1 – Thermal endurance graph

6.2 Thermal endurance characteristics and formats

The thermal endurance characteristics are

- the temperature index, TI,
- the halving interval, HIC.

The thermal endurance of an electrical insulating material is always given for a specific property and end-point. If this is disregarded, any reference to thermal endurance properties ceases to be meaningful since the properties of a material subjected to thermal ageing may not all deteriorate at the same rate. Consequently, a material may be assigned more than one temperature index or halving interval derived, for example, from the measurement of different properties.

Where the derivation is by the numerical method and the statistical conditions concerning linearity and dispersion are satisfied, the format is as follows:

– TI (HIC): TI value (HIC value);

for example, TI (HIC): 152 (9,0).

The value of TI shall be expressed as the nearest integral value, and of HIC to one decimal place.

Where the derivation is graphical or the statistical conditions are not satisfied the format is as follows:

– TI_g = TI value, HIC_g = HIC value;

for example, TI_g = 152, HIC_g = 9,0.

If a time different from 20 000 h has been used for deriving the TI, the relevant time expressed in kh shall be stated, followed by kh. The format of the TI is then:

– TI time in kh (HIC): TI value (HIC value);

for example, TI 40 kh (HIC): 131(10,0),

and correspondingly for TI_g

for example, TI_g 40 kh = 131, HIC_g = 10,0.

Where the derivation is by the simplified procedure (see IEC 60216-8) the format is:

– TI_s = TI value, HIC_s = HIC value;

for example, TI_s = 152, HIC_s = 9,0.

6.3 Times to end-point, *x*- and *y*-values

6.3.1 General

For each temperature group, the *x*-value shall be calculated using the following equation:

$$x = 1/(\vartheta + \Theta_0) \qquad (1)$$

where ϑ is the ageing temperature in degrees Celsius, and Θ_0 = 273,15 K.

6.3.2 Non-destructive tests

Within each temperature group of specimens, a property value after each ageing period is obtained for each specimen. From these values, if necessary by interpolation (see Figure 2), obtain the time to end-point and calculate its logarithm as the *y*-value to be used in 6.4.

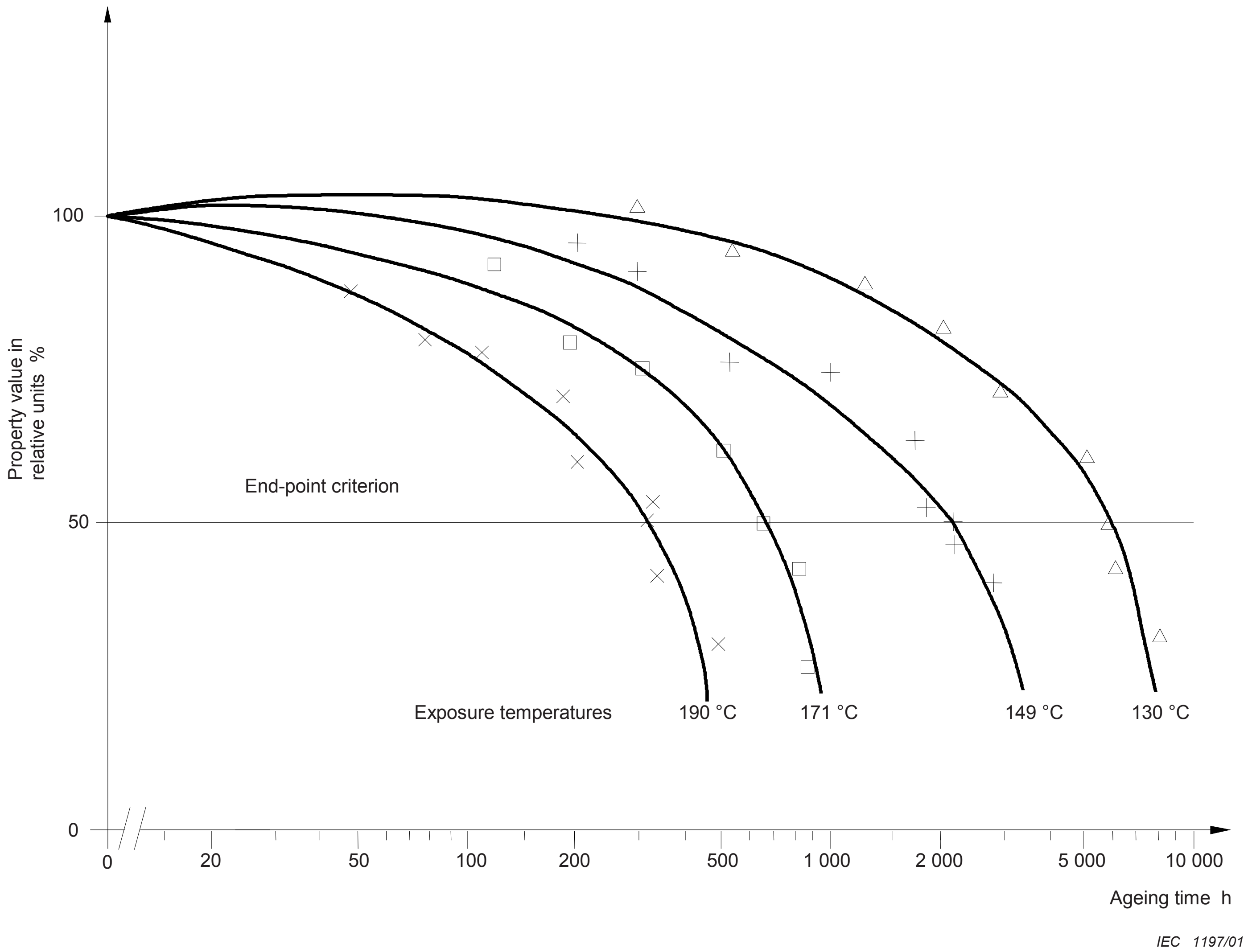


Figure 2 – Property variation – Determination of time to end-point at each temperature (destructive and non-destructive tests)

6.3.3 Proof tests

For each specimen in each temperature group, calculate the time to the mid-point of the ageing period immediately prior to reaching the end-point and take the logarithm of this time as the value of y.

A time to end-point within the first ageing period shall be treated as invalid. In this case, either

a) start again with a new group of specimens, or

b) ignore the specimen and reduce the value ascribed to the number (m_i) of specimens in group i by one.

If the end-point is reached for more than one specimen during the first period, discard the group and test a further group, paying particular attention to any critical points of experimental procedure.

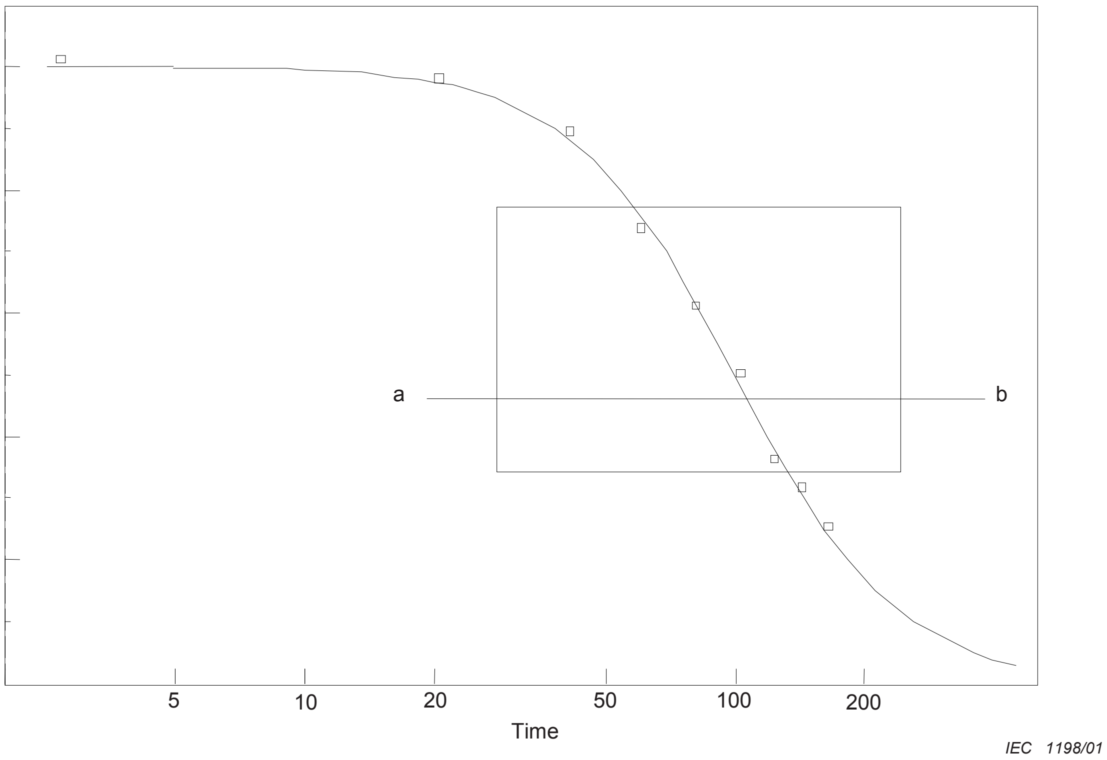
6.3.4 Destructive tests

Since each specimen is destroyed in making the relevant property measurement, it is not possible to measure directly the time to end-point for any specimen. Hypothetical times to end-point are calculated using a mathematical procedure described in detail in 6.1.4 of IEC 60216-3:2006.

This procedure is based on the assumption that the ageing rate of all specimens aged at one temperature is the same and can therefore be determined from the ageing rate of the property means of the successive groups tested. An approximately linear region of the ageing graph is selected (Figure 3) and a line parallel to the mean ageing graph drawn through each (time, property) point. The intercept of this line with the end-point line gives the logarithm of the required time to end-point (see Figure 4).

NOTE The ageing graph is formed by plotting the value of property or a suitable transform of its value against the logarithm of the exposure time. It is necessary to ensure that the intercept of the regression line with the time axis gives the same value as the mean of the intercepts of the individual lines.

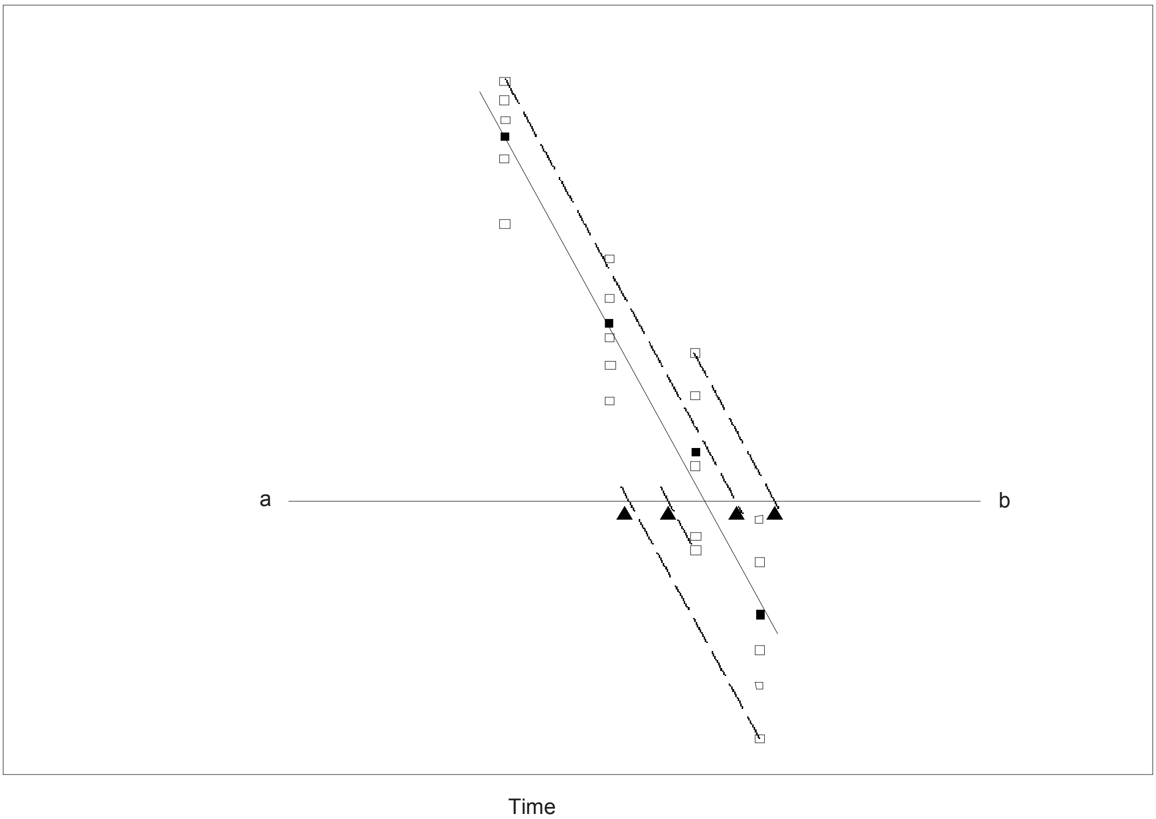
The procedure is carried out numerically, and appropriate statistical tests are introduced. The y-values derived are used in the calculations of 6.4 (see 6.1.4 of IEC 60216-3:2006).



Detail in inset rectangle shown in Figure 4.

a ________________ b = Value of property at end-point

Figure 3 – Estimation of times to end-point – Property value (ordinate, arbitrary units) versus time (abscissa, log scale, arbitrary units)



IEC 1199/01

Key

□ Data point

■ Group mean property value

▲ Pointer to time-to-end-point estimate

a ______ b Value of property at end-point

———— Regression line

------------ Estimating line parallel to regression

For the sake of clarity, estimating lines are not shown for all data points.

Figure 4 – Destructive tests – Estimation of time to end-point

6.4 Means and variances

6.4.1 Complete data

For non-destructive tests and proof tests where the time to end-point is known for all specimens in a temperature group, the mean $\overline{y}$ and variance s^2 of the y-values shall be calculated from the equations:

$$\overline{y} = \sum y / n \tag{2}$$

$$s^2 = \frac{\left[\sum y^2 - \left(\sum y\right)^2 / n\right]}{(n-1)} \tag{3}$$

where n is the number of y-values in the group.

For destructive tests, the same procedure shall be used, applied to the hypothetical values of y obtained as in 6.3.

6.4.2 Incomplete (censored) data

For non-destructive tests and proof tests where ageing has been terminated before all specimens in a group have reached end-point, the mean and variance estimates shall be calculated as in 6.2.1.2 of IEC 60216-3:2006.

6.5 General means and variances and regression analysis

The weighted mean and variance of the y-values and the weighted mean and central second moment of the x-values shall be calculated as in 6.2.2 of IEC 60216-3:2006.

The regression analysis for slope and intercept of the thermal endurance graph and the tests for deviations from linearity shall be as in 6.2.3 of IEC 60216-3:2006.

6.6 Statistical tests and data requirements

6.6.1 General

The following statistical tests are fully specified in 6.3 of IEC 60216-3:2006 and are summarized in Annexes A and B of IEC 60216-3. These tests have been designed to test all important aspects of the data which might invalidate derivation of thermal endurance characteristics, as well as to decide whether a failure to satisfy the statistical requirements is of practical significance.

A simplified procedure, requiring a limited subset of the following tests is reported in IEC 601216-8.

6.6.2 Data of all types

Before application of the statistical tests, it is necessary that the data satisfy the following requirements:

a) the mean value of time to end-point at the lowest test temperature shall be not less than 5 000 h (or $\tau/4$ when a time τ different from 20 000 is specified for the temperature index);
b) the difference between the temperature index and the lowest test temperature shall not be greater than 25 K.

If either of these conditions is not met, the value of TI cannot be reported. In order to carry out valid calculations, one or more further group(s) of specimens shall be aged at such a lower temperature as will enable the conditions to be met.

When a set of data is satisfactory with respect to the above requirements, the statistical requirement is that the difference (TI – TC) between the temperature index (TI) and its lower 95 % confidence limit (TC), is not more than 0,6 HIC. This difference is dependent on the scatter of the data points, the deviations from linearity in the regression analysis, the number of data points and the extent of extrapolation.

The general calculation procedures outlined here and detailed in IEC 60216-3 are based on the principles set out in IEC 60493-1:2011. These may be briefly expressed as follows (see 3.7.1 of IEC 60493-1:2011).

1) The relation between the mean of the logarithms of the times taken to reach the specified end-point (times to end-point) and the reciprocal of the thermodynamic (absolute) temperature is linear.

2) The values of the deviations of the logarithms of the times to end-point from the linear relation are normally distributed, with a variance which is independent of the ageing temperature.

The first assumption is tested by the so-called Fisher test (F-test). In this test, a test parameter F is calculated from the experimental data and compared with a tabulated value F_0. If $F < F_0$, the assumption of linearity is accepted and the calculation continued. If not, the assumption is *a priori* rejected, but, since in special cases it is possible to detect a statistically significant non-linearity which is of little practical importance, the calculations may, under specified conditions, be continued in a modified way (for details, see IEC 60216-3).

F_0 is chosen so that the test is carried out on significance level 0,05, which means that there is a probability of 5 % of rejecting the assumption, even if correct (and 95 % probability of accepting it when correct).

The second assumption is tested by the Bartlett's χ^2-test. The test parameter χ^2 is calculated from the data and compared with tabulated values of χ^2. If the test parameter χ^2 is greater than the value tabulated for a significance level of 0,05, the values of χ^2 and the corresponding probability P from the table shall be reported.

In the case of destructive tests (6.3.3), the linearity of the property values as a function of time in the vicinity of the end-point is also tested by the F-test (see 6.1.4.4 of IEC 60216-3:2006).

When the scatter of the data is such that the value of TI – TC lies between 0,6 HIC and 1,6 HIC, it is still possible to report an adjusted value TI_a instead of the calculated value TI such that the difference between the reported value TI_a and the lower confidence limit of TI calculated by the usual procedure is less than or equal to 0,6 HIC (the value TI is replaced by TI_a = TC + 0,6 HIC; see 4.4(3) and 7.3 of IEC 60216-3:2006).

Calculation procedures and suitable restrictions have been developed to meet these circumstances and are given in detail in IEC 60216-3. A flow chart and decision table setting out the procedures and conditions are given in Annexes A and B of IEC 60216-3:2006.

6.6.3 Proof tests

For proof-test data, the time to end-point is considered to be the mid-point of the ageing cycle leading up to failure. Failures at the end of the first ageing cycle cannot be accepted. Either a new group, possibly with a shorter cycle time, should be started, or the first cycle failure ignored and the nominal size of the group reduced by one (for example, a temperature group of 21 would be treated in the mathematical process as 20; see 6.1.3 of IEC 60216-3:2006). In either case, the specimen preparation technique should be carefully examined.

In all cases, ageing shall be continued until more than one-half of the test specimens in each group have failed to pass the proof test. It is not necessary for all groups to be equal in size or for equal numbers to have failed.

6.6.4 Destructive tests

It is normally required that at least three (and preferably more) ageing groups at each temperature be selected for linearity test, which should be satisfied at significance level 0,05 (see Figures 2 and 3 and 6.1.4.4 of IEC 60216-3:2006), and at least one of the means of these groups should lie above and at least one below the end-point. It is permitted for these conditions not to be satisfied in specified circumstances (either a small extrapolation or linearity test at significance level 0,005 may be permitted; see 6.1.4.4 of IEC 60216-3:2006).

6.7 Thermal endurance graph and thermal endurance characteristics

Calculate the temperature ϑ_1 corresponding to a time to end-point of 20 000 h (or such other time τ_1 chosen for the temperature index using Equation (47) of 6.3.3 of IEC 60216-3:2006).

In the same way, calculate the temperature ϑ_2 corresponding to a time to end-point of 10 000 h, or else $\tau_1/2$. The difference $\vartheta_2 - \vartheta_1$ is the value of the halving interval HIC.

Calculate the temperature ϑ_3 corresponding to a time to end-point of 1 000 h, or else $\tau_1/20$. Using the points ϑ_1 and ϑ_3 and their corresponding times, plot the thermal endurance regression line on thermal endurance graph paper.

Using Equations (46) to (50) of 6.3.3 of IEC 60216-3:2006, calculate the lower confidence limits of temperature estimate for times of 20 000 h, 1 000 h (or alternatives as in the previous paragraphs) and at least five intermediate times. Plot these time-temperature pairs on the thermal endurance graph and draw a smooth curve passing through these points.

On the same graph, plot the ageing temperatures, the times to end-point (measured or hypothetical), and the mean times.

The thermal endurance characteristics are as derived in the calculations of 6.5 (see also 7.2 and 7.3 of IEC 60216-3:2006).

6.8 Test report

The test report shall include the following:

a) a description of the tested material including dimensions and any conditioning of the specimens;
b) the property investigated, the chosen end-point, and, if this is a percentage value, the initial value of the property;
c) the test method used for determination of the property (for example, by reference to an IEC publication);
d) any relevant information on the test procedure, for example, ageing environment;
e) the individual test temperatures, with the appropriate data
   1) for non-destructive tests, the individual times to end-point, with the graphs of variation of property with ageing time,
   2) for proof tests, the numbers and durations of the ageing cycles, with the numbers of specimens reaching end-point during the cycles,
   3) for destructive tests, the ageing times and individual property values, with the graphs of variation of property with ageing time;
f) the thermal endurance graph;
g) the temperature index and halving interval reported in the format defined in 6.2;
h) the values of χ^2 and $k-1$ if required by 6.3.1 of IEC 60216-3:2006;
i) any first cycle failures in accordance with 5.1.2 of IEC 60216-3:2006.

Table 1 – Suggested exposure temperatures and times

Estimated value of TI in range °C	Exposure temperature °C Boxes: duration of exposure cycle in days																							
	120	**130**	**140**	**150**	**160**	**170**	**180**	**190**	**200**	**210**	**220**	**230**	**240**	**250**	**260**	**270**	**280**	**290**	**300**	**310**	**320**	**330**	**340**	**360**
95-104	28		14		7		3		1															
105-114		28		14		7		3		1														
115-124			28		14		7		3		1													
125-134				28		14		7		3		1												
135-144					28		14		7		3		1											
145-154						28		14		7		3		1										
155-164							28		14		7		3		1									
165-174								28		14		7		3		1								
175-184									28		14		7		3		1							
185-194										28		14		7		3		1						
195-204											28		14		7		3		1					
205-214												28		14		7		3		1				
215-224													28		14		7		3		1			
225-234														28		14		7		3		1		
235-244															28		14		7		3		1	
245-254																28		14		7		3		1

Further discussion and recommendations will be found in Annex B.

NOTE 1 This table is intended primarily for cyclic proof testing and non-destructive tests, but may also be used as a guide for selection of suitable time intervals for destructive tests. In this case, cycle times of 56 days, or even more, may be required.

NOTE 2 When extending the test program by submitting additional specimens to ageing at temperatures below the lower of the originally planned ageing temperatures, a temperature interval of 10 K and a cycle duration of 42 days for TI determination should be considered.

Annex A
(informative)

Dispersion and non-linearity

A.1 Data dispersion

Tests for the acceptability of data dispersion are detailed in IEC 60216-3.

If the data dispersion is not high through inadequate experimental technique, the effect of the high dispersion can be overcome by the use of a larger number of data values, i.e. more test specimens. This does not necessarily imply a complete repeat of the experimental work, since it is possible (if material is available) to test further specimens and add the results to the original data. These further tests may be at lower or intermediate temperatures but should not generally be at higher temperatures than originally selected.

In the case of proof tests with incomplete data (usually censored at the median), it may be possible to obtain a sufficient increase in data group size by continuing the exposure until further test specimens have failed the proof test.

The size of the confidence interval is roughly proportional to the square root of the reciprocal of the total number of data values.

A.2 Non-linearity

A.2.1 Mechanisms of thermal degradation

The model upon which the thermal endurance testing of electrical insulating materials according to this standard is based is the applicability of the theory of thermally activated chemical rate processes. This model is valid when the selected end-point of the diagnostic property is correlated with a particular degree of molecular change in the material which is subject to ageing. The validity of the model is, therefore, not dependent on the more stringent condition of a linear relationship between the level of the diagnostic property and the degree of molecular change.

In addition to the above-mentioned basic assumption, some general assumptions regarding the chemical mechanisms of the thermal ageing need to be satisfied:

a) the material or combination of materials should be uniform in the macro-physical sense;
b) the thermal degradation should proceed in a homogeneous phase;
c) the ageing reaction should be essentially irreversible.

A.2.2 Non-linearity of data groups

Non-linearity of data is indicated by failure of the F-test in data evaluation, when at the same time the data dispersion is large enough for the confidence interval of the result to be higher than acceptable (see 6.3 of IEC 60216-3: 2006). It may arise from inadequate experimental technique (for example, oven temperature errors); such non-linearity may be corrected by further testing. However, in many cases, the deviations arise from the ageing behavior of the material; this happens with many thermoplastic materials or other materials where the ageing temperature range includes, or is close to, a transition temperature of some kind, or where there is more than one ageing mechanism at work.

In such cases, it may be possible to obtain an acceptable result by further testing at a lower temperature. This will have the effect of decreasing the extrapolation, which is one of the influences in determining the size of the confidence interval, and also make the errors associated with the non-linearity less serious.

It is also possible that acceptable results will be obtained where further testing at a lower temperature has been carried out by removing the results at the highest temperature(s), since the deviations may only become significant at the higher temperatures.

If these expedients are not successful, it will be necessary to test at a temperature low enough for extrapolation not to be required.

Annex B
(informative)

Exposure temperatures and times

B.1 General

Table 1 serves for the selection of ageing temperatures and cycle durations when planning a thermal endurance test. The row in table 1 corresponding to the estimated TI shows suggested ageing times in days at oven temperatures which appear at the head of the respective columns. Early results of the ageing test may motivate an adjustment of ageing cycles or additional ageing temperatures.

It is advisable to distinguish between:

– cyclic and continuous ageing;

– destructive, non-destructive and proof tests for determination of the degree of deterioration.

The following recommendations and suggestions may be found helpful in establishing the ageing temperatures and times.

B.2 Temperatures

a) The highest exposure temperature should be one which will result in a median time to end-point between 100 h and 500 h (see 5.5).

b) The chosen exposure temperatures should differ by equal intervals, normally by 20 K, if the entire temperature range of the test is expected to produce the same ageing mechanisms (see Table 1). If this rule results in changes of mechanism (for example, when a transformation point like melting or softening is exceeded), then the maximum exposure temperature will need to be limited. In such cases, or if the value of HIC is known or expected to be less than 10 K, the difference between the levels of ageing temperature may need to be reduced, but to not less than 10 K (so that oven temperature tolerance effects will be acceptable).

c) Selection of the exposure temperatures involves estimating or knowing beforehand the approximate value of the temperature index of the material to be tested. If such information is not available, preliminary screening tests may be performed to produce a forecast of the value of TI.

B.3 Times

B.3.1 Cyclic ageing

For proof tests and non-destructive tests, it is necessary to minimize errors caused by differences of handling, testing and thermal cycling between the groups exposed at the selected temperatures. To achieve this, select the cycle length so that the mean or median time to end-point is reached in about 10 cycles but not less than seven.

For non-destructive tests, although Table 1 suggests constant cycle lengths, test times following a geometric series may be used.

B.3.2 Continuous ageing

For destructive tests, the ageing of each tested group is continuous, and it is therefore not necessary that the mean times to end-point at the different ageing temperatures be reached in approximately equal multiples of the cycle lengths given in table 1. However, the planned number of groups of specimens at each temperature (see 5.3) should be at least five, 10 being preferred, if possible. The time interval between tests of groups should be planned so that the results of at least two groups of specimens are available before the mean time to end-point, and at least one after: the rate of change of property with time in this interval should be reasonably linear. See 6.3.3 and IEC 60216-3:2006.

B.4 Delayed groups of specimens

A sequential procedure may be justified when an unknown material is tested. In such cases it is often convenient to start by loading the ageing oven with one-half of the prepared specimens and performing measurements after the second or third exposure cycle of the recommended series. After a few cycles, the remaining specimens can be placed in the oven and the points on the ageing curve (property variation curve) (see Figures 2, 3 and 4), which are deemed necessary, determined.

A sequential procedure may also be justified where the envisaged accuracy of the evaluation requires additional specimens to be aged, for example, in the case where the thermal endurance relationship turns out to be non-linear. If the decision to extend the original test programme is taken after its completion, the duration of the complete procedure may become prohibitive. Instead, the trend of the thermal endurance relationship may be roughly estimated after the first or second failure at the lowest ageing temperature of the original programme. Ageing at lower temperature(s) of one or two additional group(s) of specimens in case of suspected non-linearity can then be initiated immediately to produce the complete test data within a time limit which is still acceptable.

A procedure which has frequently been found very useful involves the delayed introduction of test groups following the sequence in the Table B.1 below.

This example is based on nine test groups identified as A, B, C, D, E, F, G, H, I being exposed at one temperature.

Five of the test groups are placed in the oven at the beginning of the sequence. After successive delays (see footnote in Table B.1 below), three further groups are added.

Groups are tested as indicated in the table.

Table B.1 – Groups

Start of cycle	Groups to be loaded into ageing oven	Remove from oven and test groups
1	B C D E F	A (unaged)
2 [a]	G	
3 [a]	H	
4 [a]	I	
5		B
9		C
13		D
17		E
21		F

[a] Delayed after start of cycle by a time equal to the sum of the conditioning time and the time taken for testing a group.

If the end-point has not been reached after the testing of Group F, Groups G – I may be tested after appropriate further ageing.

If end-point is reached in one of the Groups B – F, Groups G – I are immediately removed from the oven and tested after conditioning. If, for example, Group C has reached end-point (nine cycles), Groups G, H and I would have received respectively six, seven and eight cycles at testing. In this way, the total amount of testing is reduced, without loss of discrimination.

These values are intended solely as illustration and may be changed as the work requires.

Annex C
(informative)

Concepts in earlier editions

C.1 Relative temperature index (RTI)

The relative temperature index was defined in the fourth edition of IEC 60216-1:1990 as follows:

"The temperature index of a test material obtained from 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 (see Figure 4)".

In obtaining the RTI, the systematic errors observed in the determination of TI are to a large extent compensated. The characteristic has now been made the subject of a new, independent standard (in preparation).

NOTE 1 Figure 3 of IEC 602316-1:1990 (fourth edition) is redrawn here as Figure C.1.

NOTE 2 The above definition is not strictly compatible with the figure and also differs slightly from the definition in the second edition of IEC 60216-1:1974.

C.2 Thermal endurance profile (TEP)

The thermal endurance profile was introduced in the second edition of IEC 60216-1:1974 and defined as follows:

"The thermal endurance profile consists of the two numbers corresponding to the temperatures in degrees Celsius derived from the thermal endurance graph at 20 000 h and 5 000 h, followed by a number corresponding to the lower 95 % unilateral confidence limit on the temperature at 5 000 h."

It was deleted from IEC 60216-1 in the fourth edition, so that the halving interval could be stated explicitly without confusion, and because it was felt that the actual value of a lower confidence limit was not a very useful characteristic to be quoted for a material. It was felt to be much more important that assurance could be given that the difference between the calculated TI and its lower confidence limit was less than a specified value.

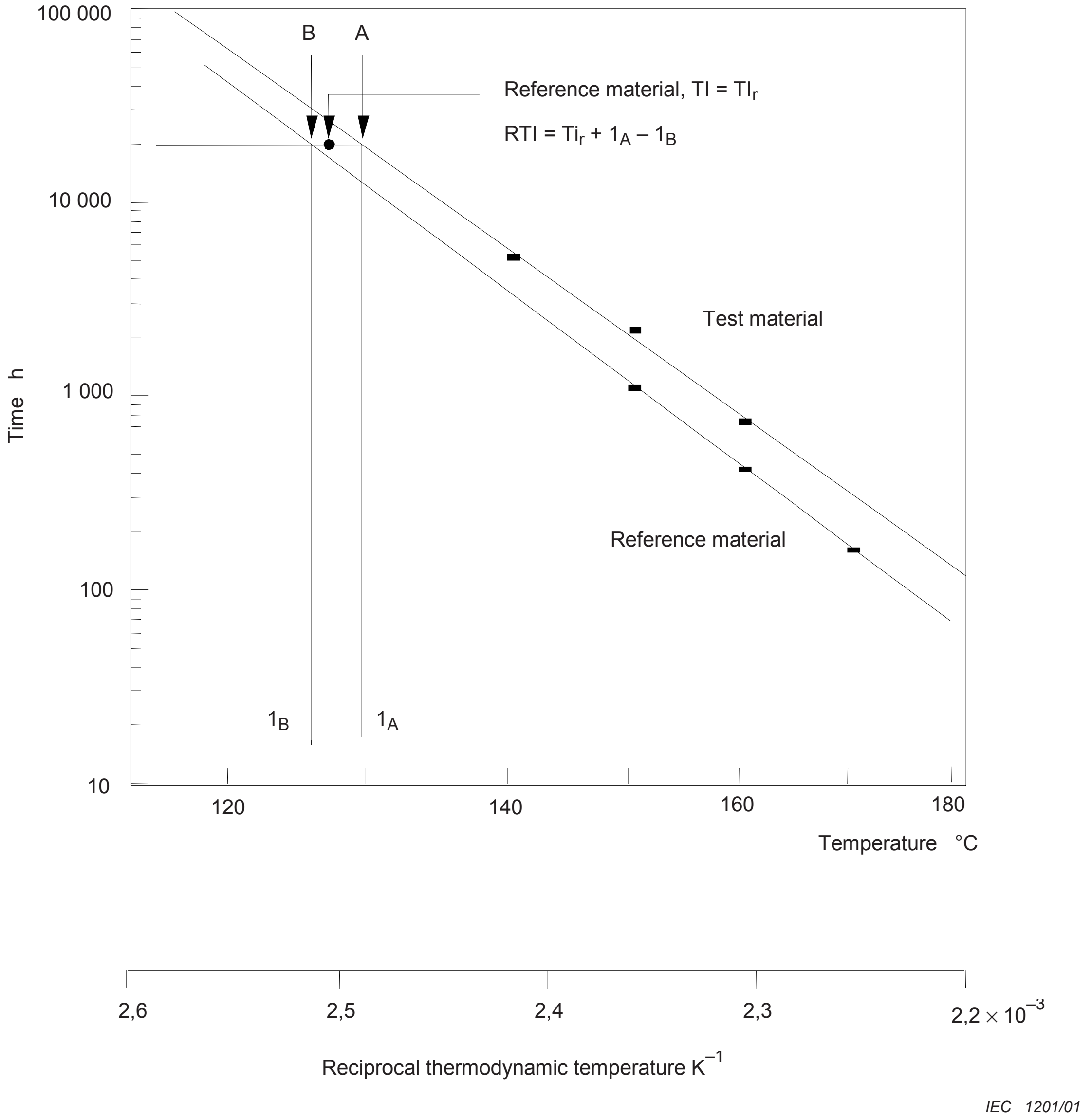


Figure C.1 – Relative temperature index
(Adapted from Figure 3, IEC 60216-1:1990, 4th edition)

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