

Electrical insulating materials — Thermal endurance properties —

Part 6: Determination of thermal endurance indices (TI and RTE) of an insulating material using the fixed time frame method

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National foreword

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**Electrical insulating materials -
Thermal endurance properties
Part 6: Determination of thermal endurance indices (TI and RTE)
of an insulating material using the fixed time frame method
(IEC 60216-6:2006)**

Matériaux isolants électriques -
Propriétés d'endurance thermique
Partie 6: Détermination des indices
d'endurance thermique (TI et RTE)
d'un matériau isolant en utilisant
la méthode de "trame de durées fixes
(fixed time frame)"
(CEI 60216-6:2006)

Elektroisolierstoffe -
Eigenschaften hinsichtlich des
thermischen Langzeitverhaltens
Teil 6: Bestimmung der thermischen
Langzeitkennwerte (TI und RTE)
eines Isolierstoffes unter Anwendung
des Festzeitrahmenverfahrens
(IEC 60216-6:2006)

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Foreword

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This European Standard supersedes EN 60216-6:2004.

The significant technical change with respect to EN 60216-6:2004 is as follows:

EN 60216-6:2006 has been supplemented by Annex G and the corresponding software.

The following dates were fixed:

- latest date by which the EN has to be implemented
at national level by publication of an identical
national standard or by endorsement (dop) 2007-06-01
- latest date by which the national standards conflicting
with the EN have to be withdrawn (dow) 2009-09-01

Annex ZA has been added by CENELEC.

Endorsement notice

The text of the International Standard IEC 60216-6:2006 was approved by CENELEC as a European Standard without any modification.

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ELECTRICAL INSULATING MATERIALS – THERMAL ENDURANCE PROPERTIES –

Part 6: Determination of thermal endurance indices (TI and RTE) of an insulating material using the fixed time frame method

1 Scope

This part of IEC 60216 specifies the experimental and calculation procedures for deriving the thermal endurance characteristics, temperature index (TI) and relative thermal endurance index (RTE) of a material using the “fixed time frame method (FTFM)”.

In this protocol, the ageing takes place for a small number of fixed times, using the appropriate number of ageing temperatures throughout each time, the properties of the specimens being measured at the end of the relevant time interval. This differs from the procedure of IEC 60216-1, where ageing is conducted at a small number of fixed temperatures, property measurement taking place after ageing times dependent on the progress of ageing.

The diagnostic tests employed in the fixed time frame method are restricted to destructive tests. The method has not as yet been applied to non-destructive or proof test procedures.

Both the TI and the RTE determined according to the FTFM protocol are derived from experimental data obtained in accordance with the instructions of IEC 60216-1 and IEC 60216-2 as modified in this standard. The calculation procedures and statistical tests are modified from those of IEC 60216-3 and IEC 60216-5.

2 Normative references

The following referenced documents are indispensable for the application of this document. 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 60216-1:2001, *Electrical insulating materials – Properties of thermal endurance – Part 1: Ageing procedures and evaluation of test results*

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 60216-3:2002, *Electrical insulating materials – Thermal endurance properties – Part 3: Instructions for calculating thermal endurance characteristics*

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

IEC 60216-4-2, *Electrical insulating materials – Thermal endurance properties – Part 4-2: Ageing ovens – Precision ovens for use up to 300 °C*

IEC 60216-4-3, *Electrical insulating materials – Thermal endurance properties – Part 4-3: Ageing ovens – Multi-chamber ovens*

IEC 60216-5, *Electrical insulating materials – Thermal endurance properties – Part 5: Determination of relative thermal endurance index (RTE) of an insulating material*

IEC 60493-1:1974, *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 abbreviated terms

For the purposes of this document, the following terms, definitions, symbols and abbreviations apply.

3.1 Terms, abbreviations and definitions

3.1.1

assessed thermal endurance index

ATE

numerical value of the temperature in degrees Celsius, up to which the control material possesses known, satisfactory service performance in the specified application

NOTE 1 The ATE of a specific material may vary between different applications of the material.

NOTE 2 ATE is sometimes referred to as “absolute” thermal endurance index.

3.1.2

ageing temperature

temperature in degrees Celsius at which a group of specimens is thermally aged

3.1.3

end-point temperature

temperature in degrees Celsius at which a specimen is considered to have reached end-point after ageing for a specified time

3.1.4

candidate material

material for which an estimate of the thermal endurance is required to be determined

NOTE The determination is made by simultaneous thermal ageing of the material and a control material.

3.1.5

central second moment of a data group

sum of the squares of the differences between the data values and the value of the group mean divided by the number of data in the group

3.1.6

95 % confidence limit

statistical parameter, calculated from test data, which with 95 % confidence constitutes an upper or lower limit for the true value of a quantity estimated by statistical analysis

NOTE 1 This implies that there is only 5 % probability that the true value of the quantity estimated is actually larger (or smaller) than the upper (or lower) confidence limit.

NOTE 2 In other connections, confidence values other than 95 % may sometimes be used, e.g. in the linearity test for destructive test data.

3.1.7

control material

material with known assessed thermal endurance index (ATE), preferably derived from service experience, used as a reference for comparative tests with the candidate material

3.1.8

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

NOTE 2 In this standard, the two data sets are the values of the independent variable and the means of the corresponding dependent variable groups.

3.1.9

correlation time (RTE)

estimated time to end-point of the control material at a temperature equal to its ATE in degrees Celsius

3.1.10

correlation time (TI)

hypothetical time to end-point used to calculate TI

NOTE Its usual value is 20 000 h.

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, 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

degrees of freedom

number of data values minus the number of parameter values

3.1.13

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

NOTE An example of a destructive test is measurement of electric strength. An example of a non-destructive test is measurement of $\tan \delta$.

3.1.14

end-point line

line parallel to the temperature axis intercepting the property axis at the end-point value

3.1.15**halving interval****HIC**

numerical value of the temperature interval in kelvins which expresses the halving of the time to end-point taken at a time equal to TI

3.1.16**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

3.1.17**regression coefficients**

coefficients of the equation of the best fit line derived by regression analysis

3.1.18**relative thermal endurance index****RTE**

estimate of the thermal endurance of a candidate material, made by thermal ageing simultaneously with the control material, as described in this standard

NOTE The value of RTE is the value of the temperature in degrees Celsius at which the estimated time to end-point of the candidate material is the same as the estimated time to end-point of the control material at a temperature equal to its ATE.

3.1.19**significance**

probability of a value of a statistical function greater than a specified value

NOTE The value is equal to $(1-p)$ where p is the cumulative distribution function value. Significance is conventionally printed in upper case (P).

3.1.20**standard deviation**

square root of the variance of a data group or sub-group

3.1.21**standard error of an estimate of the true value of a data group property**

value of the standard deviation of the hypothetical sampling population of which the group property may be considered to be a member

NOTE For an estimate of the group mean, the standard error is equal to the group standard deviation divided by the square root of the number of data in the group, and indicates the uncertainty in the estimate of the true value of the mean. This standard is concerned only with means and the difference between two means.

3.1.22**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)

3.1.23**temperature group (of specimens)**

number of specimens being exposed together to thermal ageing at the same temperature in the same oven

NOTE Where there is no risk of ambiguity, either temperature groups or test groups may be referred to simply as "groups".

3.1.24

test group (of specimens)

number of specimens removed together from a temperature group for destructive testing

NOTE Where there is no risk of ambiguity, either temperature groups or test groups may be referred to simply as "groups".

3.1.25

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

3.1.26

thermal endurance graph paper

graph paper having a logarithmic time scale as the ordinate and values proportional to the reciprocal of the thermodynamic (absolute) temperature as the abscissa

NOTE The ordinate is usually graduated in powers of ten (from 10 h to 100 000 h is often a convenient range). The abscissa is usually graduated in a non-linear (Celsius) temperature scale oriented with temperature increasing from left to right.

3.1.27

time group (of specimens)

all test groups removed for testing at the same time

3.1.28

variance of a data group

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 The reference level may, for example be a mean value (1 parameter) or a line (2 parameters, here intercept on the axis of the independent variable and slope).

3.2 Symbols and abbreviated terms

The following symbols are used in the calculations of Clauses 6, 7 and 12.

Symbol	Description	Clause
a	Regression coefficient: intercept of regression line with x -axis	6.4.3
b	Regression coefficient: slope of regression line relative to y -axis	6.4.3
b_r	Parameter derived from b for calculation of $\hat{Y}_c$	6.5.3
b_p	Regression coefficient for destructive test calculations	6.3.4
c	Parameter in calculation of χ^2	6.5.1
F	F -distributed variance ratio for linearity test	6.3.3, 6.5.2
g, h, i, j	Indexing parameters for regression calculations	6.3, 6.4
HIC	Halving interval	7.1
k	Number of ageing times	6.1.1
N	Total number of x_{ij} values	6.4.2
n_i	Number of x_{ij} values in time group i	6.1.1
P	Significance of the value of a statistical test function	Annexes A, B, and C
p_e	End-point property value	6.3

p_{gh}	Property value h in temperature group g (time group i implied)	6.3
$\bar{p}_g$	Mean property value in temperature group g (time group i implied)	6.3
q	Base of logarithms in calculation of χ^2	6.5.1
r	Number of temperature groups selected in time group i	6.3.2
r^2	Square of correlation coefficient	6.4.3
s^2	Total (non-regression) variance of x -values	6.5.2
s_{1g}^2	Variance of property values in temperature group g (time group i implied)	6.3.2
s_a^2	Value of s^2 adjusted to allow for acceptable non-linearity	6.5.2
s_r^2	Parameter derived from s^2 for calculation of $\hat{Y}_c$	6.5.3
t	Student's t -distributed stochastic variable	6.5.3
TC, TC _a	Lower confidence limit of TI or TI _a (see s_a^2 above)	7.1
$t_{p,N}$	Value of t with probability p and N degrees of freedom	6.5.3
x_{ij}	Value of x , index number j , in time group i	6.3.4
$\bar{x}$	General mean of x -values	6.4.2
$\hat{X}$, $\hat{X}_c$	Estimate of x , and its confidence limit	6.5.3
y_i	Value of y for time group i	6.1.1
$\bar{y}$	General mean of y -values	6.4.2
$\hat{Y}$, $\hat{Y}_c$	Estimate of y , and its confidence limit	6.5.3
z_{ij}	Reciprocal kelvin temperature for ϑ_{ij}	6.1.1
$\mu_2(y)$	Central 2 nd moment of y values	6.4.2
ν	Total number of property values in time group (i implied)	6.3.2
χ^2	χ^2 distributed variable for variance equality (Bartlett's) test	6.5.1
ϑ_{ij}	Ageing temperature for specimen group j in time group i	6.1.1
θ_0	273,15 K (corresponding to 0 °C)	6.1.1
τ_i	Ageing time for time group i	6.1.1

4 FTFM protocol

4.1 Principles and objectives

4.1.1 Principles of FTFM protocol

The FTFM (fixed time frame method) protocol is based upon the principle that thermal ageing for determination of thermal endurance characteristics is carried out over a small number of fixed times, with a sufficient range of ageing temperatures at each time to ensure that the property values determined reach the end-point in a satisfactory manner.

In this it differs from the fixed temperature frame procedure of IEC 60216-1, where a small number of ageing temperatures is employed, with ageing being carried out with testing at intervals, until the end-point has been reached.

4.1.2 Objective of FTFM protocol

The objective of the protocol is to achieve the following advantages:

The determination of thermal endurance characteristics is completed in a fixed, pre-determined time.

This enables much more efficient planning of the determination, and will often have substantial commercial advantage. A simple TI determination will be completed in 5 kh, whereas by the fixed temperature frame procedure, it may be necessary for ageing to be considerably prolonged past this time to achieve the end-point at the lowest chosen ageing temperature.

Each temperature to end-point (i.e. time-group mean) in the thermal endurance regression is based on the temperatures selected in a time group. The number of temperatures selected may be any number between three (3) and the number of temperature groups in a time group.

Since the largest source of systematic error in the fixed temperature frame procedure is temperature error (actual indication error or temperature distribution error), systematic errors can be considerably reduced. Errors from this source can lead to results which are either inaccurate or invalid through incorrect assessment of linearity.

5 TI determination

5.1 Ageing procedures

Each test procedure shall specify the shape, dimensions and number of the test specimens, the 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, if possible, reflect in a significant fashion 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.

5.2 Ageing times and temperatures

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, the times given as requirements or recommendations in the text of this standard (e.g. 5 kh for the minimum value of the longest ageing time) shall be increased in the ratio of the actual specification time to 20 kh.

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, e.g. glass transition temperature or partial melting, with consequent non-linearity. Very long specification times may also lead to non-linearity.

Recommendations for ageing times and temperatures are given in Annex D.

5.3 Test specimens

5.3.1 Preparation

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

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, moulding, curing, preconditioning, are performed in the same manner for all specimens.

The material specifications or the standards for the diagnostic test methods 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 shall be the same as those normally used for general testing. Where specimen dimensions need smaller tolerances than those normally used, these special tolerances shall be given.

Screening measurements ensure that specimens are of uniform quality and typical of the material to be tested.

5.3.2 Number of specimens

The accuracy of endurance test results depends largely on the number of specimens aged at each temperature.

The total number of specimens (N) is derived as follows:

$$N = a \times b \times c + d$$

where

- 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);
- b* is the number of treatments, i.e. total number of exposure temperatures, at one time;
- c* is the number of ageing time levels;
- d* is the number of specimens in the group used to establish the initial value of the property. Normal practice is to select $d = 2a$ when the diagnostic criterion is a percentage change of the property from its initial level. When the criterion is an absolute property level, *d* is usually given the value of zero, unless reporting of the initial value is required.

It is good practice to prepare additional specimens, or at least to provide a reserve from 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.

5.4 Diagnostic tests

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 in that order of preference. In this case, the diagnostic test shall be stated in the report, including the property, measurement procedure and end-point.

5.5 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. 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 5.6;
- 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. 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.

5.6 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 5.2), for two days (48 ± 6) h.

NOTE In some cases (e.g. 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 materials 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.

5.7 Ageing conditions

5.7.1 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 IEC 60216-4. Unless otherwise specified, IEC 60216-4-1 shall apply. IEC 60216-4-2 and 60216-4-3 may be specified in special cases.

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 5.7.2).

5.7.2 Environmental conditions

Unless otherwise specified, the 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 maintained at the value equal to the absolute humidity of standard atmosphere B according to IEC 60212. This, or other specified conditions, shall then be reported.

NOTE 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. Although environmental conditioning, the influence of atmospheres other than air and immersion in liquids, such as oil, may be important, these are not the concern of this standard.

5.7.3 Conditions for property measurement

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

5.8 Procedure for ageing

Establish a testing scheme, as for example outlined in Annex D.

Prepare a number of specimens following the instructions of 5.3.2. If necessary, determine the initial value of the property as specified in 5.6. Divide the specimens by random selection into test groups appropriate for the testing scheme. Place the appropriate numbers of groups in each of the ovens at the required temperature.

NOTE Attention should be given to the recommendation in Annex D (NOTE 2) to prepare extra groups of specimens should the thermal endurance characteristics of the material be unsuited to the basic recommendation of Annex D.

After each ageing time, select at random one group from each of the appropriate ageing ovens and remove it from the oven. Allow to cool to room temperature unless otherwise specified. If specified, condition for the specified time in the specified atmosphere, and test the specimens by the specified test procedure.

It is recommended to carry out calculations as data become available, particularly for the shortest exposure time.

Evaluate the results as specified in Clause 6.

6 Calculation procedures

6.1 General principles

6.1.1 Thermal endurance calculation

The general calculation procedures and instructions given in 6.4 are based on the principles set out in IEC 60493-1, modified as follows (see 3.7.1 of IEC 60493-1:1974).

- a) The relation between the mean of the reciprocals (x) of the thermodynamic (absolute) temperatures at which the specified end-point is reached and the logarithm (y) of the ageing time is linear.
- b) The values of the deviations of the values of x from the linear relation are normally distributed with a variance which is independent of the ageing time.

The data used in the general calculation procedures are obtained from the experimental data by a preliminary calculation. Calculation data comprise values of z , y , n and k , where

$z_{ij} = 1 / (\vartheta_{ij} + \theta_0)$ = reciprocal of thermodynamic value in K of ageing temperature ϑ_{ij} in °C;

$y_i = \log \tau_i$ = logarithm of value of ageing time in h (τ_i);

n_i = number of z values in group number i aged for time τ_i ;

k = number of ageing times or groups of x values.

NOTE Any number may be used as the base for logarithms, provided consistency is observed throughout calculations. The use of natural logarithms (base e) is recommended, since most computer programming languages and scientific calculators have this facility.

6.1.2 Property value – equivalent temperature transform

(Calculation of hypothetical ageing temperature derived from the value of a property)

When destructive test criteria are employed, each test specimen is destroyed in obtaining a property value: for this reason, time and/or temperature values necessary to reach end-point cannot be directly measured. To enable estimates of the times to end-point to be obtained, the following assumptions are made that in the vicinity of the end-point (for one ageing time):

- a) the relation between the mean property values and the reciprocals of the thermodynamic temperatures is approximately linear;
- b) the values of the deviations of the individual property values from this linear relation are normally distributed with a variance which is independent of the temperature;