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TECHNICAL SPECIFICATION

Electrical insulation materials – Thermal endurance properties – Part 7-1: Accelerated determination of relative thermal endurance using analytical test methods (RTE_A) – Instructions for calculations based on activation energy

IEC TS 60216-7-1:2015-03(en)

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Technical specifications are subject to review within three years of publication to decide whether they can be transformed into International Standards.

IEC TS 60216-7-1, which is a technical specification, has been prepared by IEC technical committee 112: Evaluation and qualification of electrical insulation materials and systems.

The text of this technical specification is based on the following documents:

Enquiry draft	Report on voting
112/298/DTS	112/314/RVC

Full information on the voting for the approval of this technical specification can be found in the report on voting indicated in the above table.

This publication has been drafted in accordance with the ISO/IEC Directives, Part 2.

A list of all parts in the IEC 60216 series, published under the general title *Electrical insulating materials – Thermal endurance properties*, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC web site under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- transformed into an International standard,
- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

A bilingual version of this publication may be issued at a later date.

INTRODUCTION

The existing procedures of the IEC 60216 series for the evaluation of thermal endurance of an electrical insulation material can be time consuming. These methods are therefore of limited use during development of new materials or screening of existing products for use as a material in an electrical insulation. There is an important demand from industry for a rapid test method of relative thermal endurance (RTE) / temperature index (TI) and halving interval (HIC) to reduce project times and cost. A short-term test procedure for conventional thermal endurance characterization is proposed in IEC 60216-5 and a simplified approach to data processing is described in IEC 60216-8. Non-conventional methodology for thermal endurance characterization which can reduce further test times is considered in this technical specification.

The basic procedure is based on thermal analysis methods (DSC and TGA in particular, but not restricted to them) to evaluate the activation energy of the thermal degradation of the material. The activation energy is directly correlated with the HIC of the thermal endurance.

With this information, a single-point thermal endurance test, according to IEC 60216-1 and IEC 60216-5, at the highest temperature of those selected for the conventional thermal ageing procedure, is sufficient to calculate the temperature corresponding to a selected life, typically 20 000 h, i.e. an estimate of TI. However, due to the inherent uncertainty associated with this analytical approach, only RTE can be provided for material characterization. This is obtained performing the single-point thermal endurance test in the same conditions of temperature and environment as a reference material of known thermal endurance characteristics, i.e. TI and HIC.

The analytical test methods described in this technical specification may satisfy the demand of shortening the insulating material characterization procedure, if used with care and considering the restrictions these methods imply. At present, the universal applicability and the accuracy of these methods is not validated, thus a round robin test is required to provide an IEC standard based on these procedures. This part of IEC 60216 is therefore published as a technical specification.

A general assessment process of the procedures will be developed in other sub-parts of IEC 60216-7.

ELECTRICAL INSULATION MATERIALS – THERMAL ENDURANCE PROPERTIES –

Part 7-1: Accelerated determination of relative thermal endurance using analytical test methods (RTE_A) – Instructions for calculations based on activation energy

1 Scope

This technical specification describes the procedure for the evaluation of the thermal endurance of electrical insulating materials, based on thermal analysis methods for the evaluation of the activation energy of the thermal degradation reaction and a conventional life test providing a life point in the thermal endurance graph. The purpose of the test procedure is to estimate the relative temperature index (RTE).

Predictions of thermal endurance based on this procedure are limited to ageing reactions where one single reaction is predominant and directly correlated to the end-point criteria for a specific application.

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 60085, *Electrical insulation – Thermal evaluation and designation*

IEC 60216-1, *Electrical insulating materials – Thermal endurance properties – 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 60216-5, *Electrical insulating materials – Thermal endurance properties – Part 5: Determination of relative thermal endurance index (RTE) of an insulating material.*

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

ISO 11357-6, *Plastics – Differential scanning calorimetry (DSC) – Part 6: Determination of oxidation induction time (isothermal OIT) and oxidation induction temperature (dynamic OIT)*

ISO 11358-2, *Plastics – Thermogravimetry (TG) of polymers – Part 2: Determination of activation energy*

ISO 11358-3, *Plastics – Thermogravimetry (TG) of polymers – Part 3: Determination of the activation energy using the Ozawa-Friedman plot and analysis of the reaction kinetics*

3 Terms, definitions and abbreviations

3.1 Terms and definitions

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

3.1.1
reaction rate
r
change of the concentration of a chemical entity as a function of time

[SOURCE: IUPAC "Goldbook"]

3.1.2
extent of reaction
ξ
progress of a chemical reaction equal to the number of chemical transformations

[SOURCE: IUPAC "Goldbook"]

3.1.3
rate of conversion
$\dot{\xi}$
time derivative of the extent of reaction

[SOURCE: IUPAC "Goldbook"]

3.1.4
order of reaction
n
indication of the number of entities affecting the macroscopic rate of reaction

[SOURCE: IUPAC "Goldbook"]

3.1.5
diagnostic property
$\boldsymbol{p}$
property to which TI is related

Note 1 to entry: See definition in IEC 60216-1.

3.1.6
rate law
empirical differential rate equation, an expression for the rate of a particular reaction in terms of concentrations of chemical species

[SOURCE: IUPAC "Goldbook"]

3.1.7
reaction rate constant
k
proportionality factor k of a rate law

[SOURCE: IUPAC "Goldbook"]

3.1.8
Arrhenius equation
empirical exponential law relating reaction rate constant to reciprocal of absolute temperature

[SOURCE: IUPAC "Goldbook"]

3.1.9
activation energy (Arrhenius activation energy)
E_a
empirical parameter characterizing the exponential temperature dependence of the reaction rate constant

[SOURCE: IUPAC "Goldbook"]

3.1.10
pre-exponential factor
A
coefficient of the Arrhenius equation

[SOURCE: IUPAC "Goldbook"]

3.1.11
end-point
limit for a diagnostic property value based on which the thermal endurance is evaluated

3.1.12
time to end-point
failure time
time to reach the end-point or conventional failure

3.1.13
relative temperature endurance index
RTE
numerical 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 reference material at a temperature equal to its assessed temperature index

Note 1 to entry: RTE_A is the relative temperature endurance index calculated through the analytical procedure.

3.1.14
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)

Note 1 to entry: TI_A is the temperature index calculated through the analytical procedure.

[SOURCE: IEC 60050-212:2010, 212-12-11, modified according to IEC 60216-1].

3.1.15
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

Note 1 to entry: HIC_A is the halving interval calculated through the analytical procedure.

[SOURCE: IEC 60050-212:2010, 212-12-13, modified according to IEC 60216-1]

3.1.16
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 test temperature

[SOURCE: IEC 60050-212:2010, 212-12-10]

3.1.17
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) and values of the abscissa are proportional to the reciprocal of the thermodynamic (absolute) temperature

Note 1 to entry: The abscissa is usually graduated in a non-linear (Celsius) temperature scale oriented with temperature increasing from left to right.

3.2 Abbreviations

DSC	scanning calorimetry
FTIR	Fourier transform infrared
GC-MS	gas chromatography–mass spectrometry
HIC	halving interval
OIT	oxygen induction time
RTE	relative temperature endurance index
TGA	thermogravimetric analysis
TI	temperature index

4 General considerations

4.1 Thermal degradation kinetics

The general principles of IEC 60216 to determine the temperature index and halving interval are based on the implicit assumption of a first-order kinetic of the thermal degradation process of the insulation material. Only under these conditions the thermal endurance graph is linear and halving interval is independent of the concentration of the reactants.

It is a plausible assumption that the condition of an insulating material at the time of reaching the defined end-point criteria according to IEC 60216 is correlated to a certain conversion of the thermal degradation process. Therefore, by knowing the reaction mechanism and kinetics of the thermal degradation process of an insulation material, it should be possible to estimate the thermal endurance of an insulating material.

The most important mechanisms for the degradation of insulating materials are thermal oxidation, pyrolysis, and hydrolysis of the basic polymer. Unfortunately, from a theoretical point of view, none of these reactions can be considered a priori as reactions with first-order kinetics. Pyrolysis reactions follow in general zero-order kinetics, and oxidation and hydrolysis reactions are reactions of higher order because concentration of oxygen, respectively water, will determine the total reaction rate. The degradation reaction can be considered a heterogeneous reaction having the insulating material as the solid phase and the environmental atmosphere as the gas phase. Various processes will influence the total reaction rate, such as adsorption of reactants (oxygen, water) and desorption (reaction products), as well as diffusion of reactants and products in the solid and fluid phase. Only if one of these reactions is predominant, can the overall observable reaction rate follow first-order kinetics (“pseudo first-order”).

The most common method for the evaluation of the activation energy of a chemical reaction is the determination of the concentration of reactants and/or products as a function of time and