IEC TR 60216-7-2

Edition 2.0 2024-09

TECHNICAL REPORT

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Electrical insulating materials – Thermal endurance properties – Part 7-2: Accelerated determination of relative thermal endurance using analytical test methods (RTEA) – Results of the round robin tests to validate procedures of IEC TS 60216-7-1 by non-isothermal kinetic analysis of thermogravimetric data

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ELECTRICAL INSULATING MATERIALS – THERMAL ENDURANCE PROPERTIES –

Part 7-2: Accelerated determination of relative thermal endurance using analytical test methods (RTEA) – Results of the round robin tests to validate procedures of IEC TS 60216-7-1 by non-isothermal kinetic analysis of thermogravimetric data

FOREWORD

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IEC TR 60216-7-2 has been prepared by IEC technical committee 112: Evaluation and qualification of electrical insulating materials and systems. It is a Technical Report.

This second edition cancels and replaces the first edition published in 2016. This edition constitutes a technical revision.

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This edition includes the following significant technical changes with respect to the previous edition:

a) Annex A (informative) has been added to provide a round robin test with a different polymer type – polybuthylene terephthalate (PBY) – as an additional use case of the method in accordance with IEC TS 60216-7-1;

b) Tables 3 to 11 have been corrected by adding units, and texts have been refined for more technical clarifications of the procedures and observations.

The text of this Technical Report is based on the following documents:

Draft	Report on voting
112/651/DTR	112/658/RVDTR

Full information on the voting for its approval can be found in the report on voting indicated in the above table.

The language used for the development of this Technical Report is English.

This document was drafted in accordance with ISO/IEC Directives, Part 2, and developed in accordance with ISO/IEC Directives, Part 1 and ISO/IEC Directives, IEC Supplement, available at www.iec.ch/members_experts/refdocs. The main document types developed by IEC are described in greater detail at www.iec.ch/publications.

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 document will remain unchanged until the stability date indicated on the IEC website under webstore.iec.ch in the data related to the specific document. At this date, the document will be

- reconfirmed,
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- revised.

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INTRODUCTION

IEC technical committee 112, (IEC TC 112) has been working on the development of IEC TS 60216-7-1 [1][1] that considers the use of activation energy determined through thermal analytical tools plus abbreviated conventional heat ageing to determine a thermal index on a polymeric compound. At the same time, the Underwriters Laboratories Long-Term Thermal Aging Forum (UL LTTA Forum) has been discussing alternative methods that can speed up the determination of a thermal index. Members of the IEC TC 112 and of the UL LTTA Forum have made joint efforts to determine whether the Technical Specification developed by IEC TC 112 can be used to offer an alternative method of evaluating polymeric compounds for a thermal index.

Members of IEC TC 112 and the UL LTTA Forum decided to conduct a round robin test (RRT) using thermogravimetric analysis (TGA) according to ISO 11358-2 [3] on a known compound, with a known activation energy determined through conventional ageing with a view to validate the acceptability of IEC TS 60216-7-1, and to determine whether a similar thermal index can be calculated. The round robin testing was conducted with conventional TGA by multiple heating rates. However, running isothermal tests can be a follow-up of this RRT.

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[1] Numbers in square brackets refer to the Bibliography.

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Part 7-2: Accelerated determination of relative thermal endurance using analytical test methods (RTEA) – Results of the round robin tests to validate procedures of IEC TS 60216-7-1 by non-isothermal kinetic analysis of thermogravimetric data

1 Scope

This part of IEC 60216 is intended to validate the procedures of IEC TS 60216-7-1 in providing a similar temperature index to conventional methods used in other parts of the IEC 60216 series.

The round robin test results do not provide statistical analysis for precision. The round robin test focuses on preliminary studies to understand the evaluation and calculation procedures, influence on apparatus, and data variance among laboratories before determination of precision.

2 Normative references

There are no normative references in this document.

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3 Terms and definitions

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

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- IEC Electropedia: available at https://www.electropedia.org/
- ISO Online browsing platform: available at https://www.iso.org/obp

3.1
activation energy
Arrhenius activation energy
E_a
empirical parameter characterizing the exponential temperature dependence of the reaction rate constant

3.2
end-point
limit for a diagnostic property value based on which the thermal endurance is evaluated

3.3
time to end-point
failure time
time to reach the end-point or conventional failure

3.4
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.

3.5
temperature endurance 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 [2], modified – "characterizing the thermal capability of an insulating material or an insulation system" has been replaced with "derived from the thermal endurance relationship at a time of 20 000 h (or other specified time)" and the two notes to entry have been replaced by a new note to entry.]

3.6
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

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

3.7
degree of conversion
α
quantity of products present at a particular time and temperature during a reaction compared with the final quantity of the products

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[SOURCE: ISO 11358-2:2021, 3.3 [3], modified – The symbol "*C*" has been replaced with "α" and the notes to entry have been deleted.]

4 Test specimens

For the round robin test, one generic type of polymer, liquid crystal polyester (LCP), was pre-selected. Although it is known that materials can undergo more than one transition, the round robin test verified the assumption that one single thermal degradation reaction is predominant and directly correlated to the end-point of dielectric strength as a diagnostic property.

NOTE Since different materials can undergo more than one transition, the validity of results obtained from the evaluation of thermal endurance properties using TGA are assessed for the different materials.

LCP originally has very little entwining of molecules exhibiting crystalline properties as a liquid. Hence, there is less thermal transformation between solid and liquid, or between oven ageing conditions of conventional thermal endurance test and TGA conditions at higher temperature ranges. In addition, LCP molecular chains align themselves when moulded, and this generates a self-reinforcing effect, thereby resulting in high mechanical and electrical stress resistance.

In this round robin, two LCP materials (LCP sample A, LCP sample B) were chosen as test samples which already have the conventional heat oven ageing data of dielectric strength, tensile strength, and impact strength to validate the acceptability of whether or not RTE_A can be similar to RTE. Both sample A and sample B consist of 30 % glass fibres reinforced materials. Configurations of monomers are the only differences between the samples which influence the difference in thermal resistance, as shown in Table 1.

The samples were homogenized by freeze-pulverization from test plaques. 100 mg each of freeze-pulverized powders from the same batch were prepared and provided to eleven testing laboratories for evaluation, after pre-drying at 140 °C for 4 h.

Table 1 – Heat ageing properties of the test specimens by the conventional procedure described in IEC 60216-5 [4]

Temperature in ovens	Time to end-point at 50 % retention of initial dielectric strength h		Time to end-point at 50 % retention of initial tensile strength h		Time to end-point at 50 % retention of initial impact strength h	
°C	LCP Sample A	LCP Sample B	LCP Sample A	LCP Sample B	LCP Sample A	LCP Sample B
290		1 141		1 215		1 860
285	2 896		1 789		2 870	
280		1 917		3 229		2 655
275	5 591		3 083		4 164	
270		4 300		4 597		3 920
265	8 255		6 706		8 412	
260		5 848		7 625		6 640
250						9 600
TI (°C)	250,0	241,5	249,1	246,2	249,1	234,7
E_a (kJ/mol)	130,6	142,3	165,2	145,9	134,5	102,9

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5 Test apparatus

5.1 Thermogravimetric analyser (TGA)

A thermogravimetric analyser (TGA) in accordance with ISO 11358-1 [5] was used for the determination of RTE_A concerning the test samples. In fact, a number of commercial instruments suitable for the measurement are available and various models of TGAs were used for evaluation of the test samples by the participating laboratories. Before the RRT, weight and temperature calibrations were implemented based on ISO 11358-1 and TGA apparatus manufacturer's guidance.

5.2 Purge gas supplied into the TGA furnace

For purge gas into the TGA furnace, air was chosen to assume oxidative thermal degradation, as well as degradation of electrical and mechanical strengths with test specimens in oven ageing. Most of the laboratory participants selected dry air (water content less than 1 ppm[2]), but air supplied from the facility (compressed air with or without an air dryer) was used in a few laboratories.

[2] ppm = parts per million.

6 Test procedures

6.1 General

Thermal analysis with TGA of the test samples was evaluated with reference to ISO 11358-2 [3] and IEC TS 60216-7-1 in principle. A few modifications of test conditions and more detailed procedures were added as follows.

6.2 Preconditioning of test samples

5 mg ± 0,5 mg of the test sample were initially measured in each laboratory and mounted on the empty pan in the furnace opened. Then the furnace was closed and pre-conditioned in equilibrium at 100 °C for 1 h before heating tests were started. The weight value just before the heating test (time at 0 s in the heating run, or 60 min at the end of the equilibrium) was used for calculation on the degree of conversion.

NOTE ISO 11358-2 [3] requires using test samples of identical mass ±1 % of the initial weight in multiple heating conditions which is much narrower than the above. Influence on the initial mass deviation is taken into consideration in 7.2.

6.3 TGA tests with multiple heating rates

Multiple heating rates testing at 1 K/min, 2 K/min, 4 K/min, 6 K/min and 8 K/min were selected for evaluation which resulted in the lowest and highest heating rates differing by a factor of 8, in accordance with ISO 11358-2 [3]. Evaluation temperature range was set between 100 °C and 700 °C. Each heating rate test was run one time each for sample A and sample B, but 8 K/min was evaluated twice as an approximate check and to consider repeatability.

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6.4 Calculation of the activation energy (E_a)

After TGA data with multiple heating rates were obtained, the activation energies were calculated for given degrees of conversion in accordance with Equation (2) in ISO 11358-2:2021 [3]. Then, both values of degree of conversion and the activation energies were plotted between 1 % and 19 % with 2 % interval of degree of conversion and a cubic curve fitting approximately was performed as shown in Figure 1. Equation (2) in ISO 11358-2:2021 [3] was used for the selection of appropriate activation energy and degree of conversion to determine RTE_A.

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For example, if the activation energy of a reference material was already determined as 150 kJ/mol by the conventional heat ageing (e.g. dielectric strength), the corresponding degree of conversion of the reference material can be read and obtained with the equation of the fitting curve graph (see Figure 1). Then the corresponding degree of conversion for this reference material can be used for reading the activation energy of a candidate material from another graph which was also evaluated with ISO 11358-2 [3] and had a similar degree of conversion versus the activation energy fitting curve for the candidate material.

All TGA raw data were submitted by eleven participating laboratories and analysis with ISO 11358-2 [3] was carried out by one of the laboratories with the analytical tool, to avoid any discrepancy among various software calculations.