IEC TR 60216-7-2

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TECHNICAL REPORT

colour inside

Electrical insulating materials – Thermal endurance properties – Part 7-2: 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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FOREWORD

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

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

Enquiry draft	Report on voting
112/354/DTR	112/370/RVC

Full information on the voting for the approval of this Technical Report 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 website under "http://webstore.iec.ch" in the data related to the specific publication. At this date, the publication will be

- reconfirmed,
- withdrawn,
- replaced by a revised edition, or
- amended.

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

IMPORTANT – The 'colour inside' logo on the cover page of this publication indicates that it contains colours which are considered to be useful for the correct understanding of its contents. Users should therefore print this document using a colour printer.

INTRODUCTION

IEC technical committee 112, (IEC/TC112) has been working on the development of IEC TS 60216-7-1 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 UL LTTA Forum has been discussing alternative methods that could speed up the determination of a thermal index. Members of the IEC/TC112 and of the UL LTTA Forum have joined efforts to determine whether the Technical Specification developed by IEC/TC112 can be used to offer an alternative method of evaluating polymeric compounds for a thermal index.

Members of IEC/TC112 and the UL LTTA Forum decided to conduct a round robin test (RRT) using thermogravimetric analysis (TGA) according to ISO 11358-2 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 could 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.

ELECTRICAL INSULATING MATERIALS – THERMAL ENDURANCE PROPERTIES –

Part 7-2: 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

The purpose of this part of IEC 60216, which is a Technical Report, is 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.

These round robin test results do not provide statistical analysis for precisions. 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 precisions.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

[SOURCE: IUPAC “Goldbook”]

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, modified – the two notes have been deleted and replaced by a new note.]

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

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

[SOURCE: ISO 11358-2:2014, 3.3, modified — the notes have been deleted]

4 Test specimens

For the round robin study, one generic type of polymer, liquid crystal polyester (LCP), was pre-selected as the round robin study which assumes one single thermal degradation reaction is predominant and directly correlated to the end-point of dielectric strength as a diagnostic property.

LCP originally has very little entwining of molecules which exhibits crystalline properties as a liquid and therefore, 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 range. 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 for 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. Configurations of monomers are only different between the samples which influence the difference in thermal resistance, as shown in Table 1.

The samples were homogenized by freeze-pulverization from the test plaques respectively. 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

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
Ea	130,6	142,3	165,2	145,9	134,5	102,9
TI	250,0	241,5	249,1	246,2	249,1	234,7

5 Test apparatus

5.1 Thermogravimetric analyser (TGA)

A thermogravimetric analyser (TGA) in accordance with ISO 11358-1 was used for determination of RTE_A concerning the test samples. In fact, a number of commercial instruments suitable for the document are available and various models of TGAs that the laboratory participants have were used for evaluation of the test samples. 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[1]), but air supplied from the facility (compressed air with or without air dryer) was used in a few laboratories.

6 Test procedures

6.1 General

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

1 ppm = part per million.

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 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 gave the lowest and highest heating rates that differ by a factor of 8, in accordance with ISO 11358-2. 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.

6.4 Calculation of the activation energy (E_a)

After TGA data with multiple heating rates were obtained, the activation energies were calculated per certain given degrees of conversion in accordance with Equation (2) in ISO 11358-2:2014. Then, both values of degree of conversion and the activation energies were plotted between 1 % and 19 % with 2 % interval of degree of conversion to analyse the cubic approximation for drawing the fitting curve of the plots as shown in Figure 1. Equation (2) in ISO 11358-2:2014 was used for selection of appropriate activation energy and degree of conversion to determine RTE_A.

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 and had another fitting curve of activation energy and degree of conversion for the candidate material.

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