IEC TR 60216-7-2

Edition 2.0 2024-09
REDLINE VERSION

TECHNICAL REPORT

colour inside

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

INTERNATIONAL
ELECTROTECHNICAL
COMMISSION

ICS 19.020; 29.020; 29.035.01

ISBN 978-2-8322-9761-2

Warning! Make sure that you obtained this publication from an authorized distributor.

® Registered trademark of the International Electrotechnical Commission

CONTENTS

FOREWORD 4
INTRODUCTION 2
1 Scope 7
2 Normative references 7
3 Terms and definitions 7
4 Test specimens 8
5 Test apparatus 9
5.1 Thermogravimetric analyser (TGA) 9
5.2 Purge gas supplied into the TGA furnace 9
6 Test procedures 10
6.1 General 10
6.2 Preconditioning of test samples 10
6.3 TGA tests with multiple heating rates 10
6.4 Calculation of the activation energy (E_a) 10
6.5 Determination of thermal endurance using TGA 11
6.5.1 General 11
6.5.2 Determination of RTE_A by given degree of conversion from reference material (Method A) 11
6.5.3 Determination of TI_A by fixed degree of conversion at 0,05 (Method B) 12
7 Round robin test results 12
7.1 TGA test results 12
7.2 Degree of conversion correlated to the activation energy from conventional heat ageing data 12
7.3 HIC_A determined by Method A and Method B 13
7.4 RTE_A determined by Method A and TI_A by Method B 14
7.5 Difference between RTE_A and TI determined by the conventional heat ageing tests 16
8 Observations from the round robin test results 18
8.1 General 18
8.2 Sample weight variation 18
8.3 Humidity and hydrolysis of the sample 20
8.4 Considerations on repeatability of TGA curves 20
8.5 Baseline drift and responsiveness to heating rates of TGA 21
9 Conclusion ~~and recommendation~~ 25
Annex A (informative) Additional round robin studies with polybuthylene terephthalate 26
A.1 Objectives 26
A.2 Test specimens 26
A.3 Test apparatus 26
A.4 Test procedures 27
A.5 Test results 27
A.6 Observations 32
Bibliography 35

Figure 1 – Fitting curve of plots between degree of conversion and activation energy determined by ISO 11358-2 [3] (example) 11

Figure 2 – Correlation between the initial sample mass of sample A and the difference of RTE_A (TI_A) from TI 19

Figure 3 – Correlation between the initial sample mass of sample B and the difference of RTE_A (TI_A) from TI 19

Figure 4 – Overlay charts of TGA curves in multiple heating rates in multiple laboratories (enlarged) 22

Figure 5 – Logarithm plots for activation energy calculation 23

Figure 6 – Fitting curves of degree of conversion versus activation energy by TGA 24

Figure A.1 – Effect of sample amount on E_a (data provided by laboratory E) 33

Figure A.2 – Summary of factors affecting the TGA kinetic study for determination of RTE_A and TI_A 34

Table 1 – Heat ageing properties of the test specimens by the conventional procedure described in IEC 60216-5 [4] 9

Table 2 – Degree of conversion identical to the activation energy of the conventional heat ageing 13

Table 3 – HIC_A determined by Method A and Method B for dielectric strength 13

Table 4 – HIC_A determined by Method A and Method B for tensile strength 14

Table 5 – HIC_A determined by Method A and Method B for impact strength 14

Table 6 – RTE_A determined by Method A and TI_A by Method B for dielectric strength 15

Table 7 – RTE_A determined by Method A and TI_A by Method B for tensile strength 15

Table 8 – RTE_A determined by Method A and TI_A by Method B for impact strength 16

Table 9 – Difference between RTE_A or TI_A, and TI for dielectric strength 16

Table 10 – Difference between RTE_A or TI_A, and TI for tensile strength 17

Table 11 – Difference between RTE_A or TI_A, and TI for impact strength 17

Table 12 – Comparison of degree of conversion with original or rerun data at 8 K/min 21

Table A.1 – Heat ageing properties of the PBT test specimens by the conventional procedure in accordance with IEC 60216-5 [4] 26

Table A.2 – Degrees of conversion at the activation energy identical to that from conventional heat ageing 27

Table A.3 – HIC_A determined by Method A and Method B for dielectric strength 28

Table A.4 – HIC_A determined by Method A and Method B for tensile strength 28

Table A.5 – HIC_A determined by Method A and Method B for impact strength 29

Table A.6 – RTE_A determined by Method A and TI_A by Method B for dielectric strength 29

Table A.7 – RTE_A determined by Method A and TI_A by Method B for tensile strength 30

Table A.8 – RTE_A determined by Method A and TI_A by Method B for impact strength 30

Table A.9 – Difference between RTE_A or TI_A, and TI for dielectric strength 31

Table A.10 – Difference between RTE_A or TI_A, and TI for tensile strength 31

Table A.11 – Difference between RTE_A or TI_A, and TI for impact strength 32

INTERNATIONAL ELECTROTECHNICAL COMMISSION

_____

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

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
6) All users should ensure that they have the latest edition of this publication.
7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
9) IEC draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). IEC takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, IEC had not received notice of (a) patent(s), which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at https://patents.iec.ch. IEC shall not be held responsible for identifying any or all such patent rights.

This redline version of the official IEC Standard allows the user to identify the changes made to the previous edition IEC TR 60216-7-2:2016. A vertical bar appears in the margin wherever a change has been made. Additions are in green text, deletions are in strikethrough red text.

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.

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,
- withdrawn, or
- revised.

IMPORTANT – The "colour inside" logo on the cover page of this document 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 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 ~~could~~ 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 ~~could~~ 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.

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

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

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.

IEC TR 60216-7-2

Edition 2.0 2024-09

TECHNICAL REPORT

colour inside

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



IEC TR 60216-7-2:2024-09(en)

CONTENTS

FOREWORD 4
INTRODUCTION 6
1 Scope 7
2 Normative references 7
3 Terms and definitions 7
4 Test specimens 8
5 Test apparatus 9
5.1 Thermogravimetric analyser (TGA) 9
5.2 Purge gas supplied into the TGA furnace 9
6 Test procedures 10
6.1 General 10
6.2 Preconditioning of test samples 10
6.3 TGA tests with multiple heating rates 10
6.4 Calculation of the activation energy (E_a) 10
6.5 Determination of thermal endurance using TGA 11
6.5.1 General 11
6.5.2 Determination of RTE_A by given degree of conversion from reference material (Method A) 11
6.5.3 Determination of TI_A by fixed degree of conversion at 0,05 (Method B) 12
7 Round robin test results 12
7.1 TGA test results 12
7.2 Degree of conversion correlated to the activation energy from conventional heat ageing data 12
7.3 HIC_A determined by Method A and Method B 13
7.4 RTE_A determined by Method A and TI_A by Method B 14
7.5 Difference between RTE_A and TI determined by the conventional heat ageing tests 16
8 Observations from the round robin test results 18
8.1 General 18
8.2 Sample weight variation 18
8.3 Humidity and hydrolysis of the sample 20
8.4 Considerations on repeatability of TGA curves 20
8.5 Baseline drift and responsiveness to heating rates of TGA 21
9 Conclusion 25
Annex A (informative) Additional round robin studies with polybuthylene terephthalate 26
A.1 Objectives 26
A.2 Test specimens 26
A.3 Test apparatus 26
A.4 Test procedures 27
A.5 Test results 27
A.6 Observations 32
Bibliography 35

Figure 1 – Fitting curve of plots between degree of conversion and activation energy determined by ISO 11358-2 [3] (example) 11

Figure 2 – Correlation between the initial sample mass of sample A and the difference of RTE_A (TI_A) from TI 19

Figure 3 – Correlation between the initial sample mass of sample B and the difference of RTE_A (TI_A) from TI 19

Figure 4 – Overlay charts of TGA curves in multiple heating rates in multiple laboratories (enlarged) 22

Figure 5 – Logarithm plots for activation energy calculation 23

Figure 6 – Fitting curves of degree of conversion versus activation energy by TGA 24

Figure A.1 – Effect of sample amount on E_a (data provided by laboratory E) 33

Figure A.2 – Summary of factors affecting the TGA kinetic study for determination of RTE_A and TI_A 34

Table 1 – Heat ageing properties of the test specimens by the conventional procedure described in IEC 60216-5 [4] 9

Table 2 – Degree of conversion identical to the activation energy of the conventional heat ageing 13

Table 3 – HIC_A determined by Method A and Method B for dielectric strength 13

Table 4 – HIC_A determined by Method A and Method B for tensile strength 14

Table 5 – HIC_A determined by Method A and Method B for impact strength 14

Table 6 – RTE_A determined by Method A and TI_A by Method B for dielectric strength 15

Table 7 – RTE_A determined by Method A and TI_A by Method B for tensile strength 15

Table 8 – RTE_A determined by Method A and TI_A by Method B for impact strength 16

Table 9 – Difference between RTE_A or TI_A, and TI for dielectric strength 16

Table 10 – Difference between RTE_A or TI_A, and TI for tensile strength 17

Table 11 – Difference between RTE_A or TI_A, and TI for impact strength 17

Table 12 – Comparison of degree of conversion with original or rerun data at 8 K/min 21

Table A.1 – Heat ageing properties of the PBT test specimens by the conventional procedure in accordance with IEC 60216-5 [4] 26

Table A.2 – Degrees of conversion at the activation energy identical to that from conventional heat ageing 27

Table A.3 – HIC_A determined by Method A and Method B for dielectric strength 28

Table A.4 – HIC_A determined by Method A and Method B for tensile strength 28

Table A.5 – HIC_A determined by Method A and Method B for impact strength 29

Table A.6 – RTE_A determined by Method A and TI_A by Method B for dielectric strength 29

Table A.7 – RTE_A determined by Method A and TI_A by Method B for tensile strength 30

Table A.8 – RTE_A determined by Method A and TI_A by Method B for impact strength 30

Table A.9 – Difference between RTE_A or TI_A, and TI for dielectric strength 31

Table A.10 – Difference between RTE_A or TI_A, and TI for tensile strength 31

Table A.11 – Difference between RTE_A or TI_A, and TI for impact strength 32

INTERNATIONAL ELECTROTECHNICAL COMMISSION

_____

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

1) The International Electrotechnical Commission (IEC) is a worldwide organization for standardization comprising all national electrotechnical committees (IEC National Committees). The object of IEC is to promote international co-operation on all questions concerning standardization in the electrical and electronic fields. To this end and in addition to other activities, IEC publishes International Standards, Technical Specifications, Technical Reports, Publicly Available Specifications (PAS) and Guides (hereafter referred to as "IEC Publication(s)"). Their preparation is entrusted to technical committees; any IEC National Committee interested in the subject dealt with may participate in this preparatory work. International, governmental and non-governmental organizations liaising with the IEC also participate in this preparation. IEC collaborates closely with the International Organization for Standardization (ISO) in accordance with conditions determined by agreement between the two organizations.
2) The formal decisions or agreements of IEC on technical matters express, as nearly as possible, an international consensus of opinion on the relevant subjects since each technical committee has representation from all interested IEC National Committees.
3) IEC Publications have the form of recommendations for international use and are accepted by IEC National Committees in that sense. While all reasonable efforts are made to ensure that the technical content of IEC Publications is accurate, IEC cannot be held responsible for the way in which they are used or for any misinterpretation by any end user.
4) In order to promote international uniformity, IEC National Committees undertake to apply IEC Publications transparently to the maximum extent possible in their national and regional publications. Any divergence between any IEC Publication and the corresponding national or regional publication shall be clearly indicated in the latter.
5) IEC itself does not provide any attestation of conformity. Independent certification bodies provide conformity assessment services and, in some areas, access to IEC marks of conformity. IEC is not responsible for any services carried out by independent certification bodies.
6) All users should ensure that they have the latest edition of this publication.
7) No liability shall attach to IEC or its directors, employees, servants or agents including individual experts and members of its technical committees and IEC National Committees for any personal injury, property damage or other damage of any nature whatsoever, whether direct or indirect, or for costs (including legal fees) and expenses arising out of the publication, use of, or reliance upon, this IEC Publication or any other IEC Publications.
8) Attention is drawn to the Normative references cited in this publication. Use of the referenced publications is indispensable for the correct application of this publication.
9) IEC draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). IEC takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, IEC had not received notice of (a) patent(s), which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at https://patents.iec.ch. IEC shall not be held responsible for identifying any or all such patent rights.

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.

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,
- withdrawn, or
- revised.

IMPORTANT – The "colour inside" logo on the cover page of this document 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 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.

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

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

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.