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INTERNATIONAL STANDARD

colour inside

Nuclear power plants – Instrumentation and control important to safety – Electrical equipment condition monitoring methods – Part 4: Oxidation induction techniques

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FOREWORD

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This redline version of the official IEC Standard allows the user to identify the changes made to the previous edition IEC/IEEE 62582-4:2011. 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/IEEE 62582-4 was prepared by subcommittee 45A: Instrumentation, control and electrical power systems of nuclear facilities, of IEC technical committee 45: Nuclear instrumentation, in cooperation with Nuclear Power Engineering Committee of the IEEE, under the IEC/IEEE Dual Logo Agreement between IEC and IEEE. It is an International Standard.

This document is published as an IEC/IEEE Dual Logo standard.

This second edition cancels and replaces the first edition, published in 2011. This edition constitutes a technical revision.

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

a) Consideration of publication of IEC/IEEE 60780-323;
b) An example added in Annex B and update;
c) Annex C added.

The text of this International Standard is based on the following IEC documents:

Draft	Report on voting
45A/1435/FDIS	45A/1445/RVD

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 International Standard is English.

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

A list of all parts of IEC/IEEE 62582 series, under the general title *Nuclear power plants – Instrumentation and control important to safety – Electrical equipment condition monitoring methods*, can be found on the IEC website.

The IEC Technical Committee and IEEE Technical Committee have 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,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

a) Technical background, main issues and organisation of this standard

This part of IEC/IEEE 62582 specifically focuses on oxidation induction methods for condition monitoring for the management of ageing of electrical equipment installed in nuclear power plants. The methods are primarily suited to samples taken from materials that are polyolefin-based, but they can also be used for some materials based on ethylene-propylene polymers and for some ethylene vinyl acetate materials.

This part 4 of IEC/IEEE 62582 is the fourth part of the IEC/IEEE 62582 series. ~~It contains detailed descriptions of condition monitoring based on oxidation induction measurements.~~

IEC/IEEE 62582 series is issued with a joint logo which makes it applicable to the management of ageing of electrical equipment qualified to IEEE as well as IEC Standards.

~~Historically, IEEE Std 323-2003 introduced~~ IEC/IEEE 60780-323 includes the concept and role that condition based qualification could be used in equipment qualification as an adjunct to qualified life. In equipment qualification, the condition of the equipment for which acceptable performance was demonstrated is the qualified condition. The qualified condition is the condition of equipment, prior to the start of a design basis event, for which the equipment was demonstrated to meet the design requirements for the specified service conditions.

Significant research has been performed on condition monitoring techniques and the use of these techniques in equipment qualification as noted in NUREG/CR-6704, Vol. 2 (BNL-NUREG-52610) JNES-SS-0903, 2009 and IAEA-TECDOC-1825:2017.

It is intended that this document be used by test laboratories, operators of nuclear power plants, systems evaluators, and licensors.

b) Situation of the current standard in the structure of the IEC SC 45A standard series

IEC/IEEE 62582-4 is the third level IEC SC 45A document tackling the specific issue of application and performance of oxidation induction measurements in the management of ageing of electrical instrument and control equipment in nuclear power plants.

IEC/IEEE 62582-4 is to be read in association with IEC/IEEE 62582-1, which provides background and guidelines for the application of methods for condition monitoring of electrical equipment important to safety of nuclear power plants.

For more details on the structure of the IEC SC 45A standard series, see item d) of this introduction.

c) Recommendations and limitations regarding the application of this standard

It is important to note that this document establishes no additional functional requirements for safety systems.

d) Description of the structure of the IEC SC45A standard series and relationships with other IEC documents and other bodies documents (IAEA, ISO)

~~The top-level document of the IEC SC 45A standard series is IEC 61513. It provides general requirements for I&C systems and equipment that are used to perform functions important to safety in NPPs. IEC 61513 structures the IEC SC 45A standard series.~~

~~IEC 61513 refers directly to other IEC SC 45A standards for general topics related to categorisation of functions and classification of systems, qualification, separation of systems,~~

~~defence against common cause failure, software aspects of computer-based systems, hardware aspects of computer-based systems, and control room design. The standards referenced directly at this second level should be considered together with IEC 61513 as a consistent document set.~~

~~At a third level, IEC SC 45A standards not directly referenced by IEC 61513 are standards related to specific equipment, technical methods, or specific activities. Usually these documents, which make reference to second-level documents for general topics, can be used on their own.~~

~~A fourth level extending the IEC SC 45A standard series, corresponds to the Technical Reports which are not normative.~~

~~IEC 61513 has adopted a presentation format similar to the basic safety publication IEC 61508 with an overall safety life-cycle framework and a system life-cycle framework and provides an interpretation of the general requirements of IEC 61508-1, IEC 61508-2 and IEC 61508-4, for the nuclear application sector. Compliance with IEC 61513 will facilitate consistency with the requirements of IEC 61508 as they have been interpreted for the nuclear industry. In this framework IEC 60880 and IEC 62138 correspond to IEC 61508-3 for the nuclear application sector.~~

~~IEC 61513 refers to ISO as well as to IAEA 50-C-QA (now replaced by IAEA GS-R-3) for topics related to quality assurance (QA).~~

~~The IEC SC 45A standards series consistently implements and details the principles and basic safety aspects provided in the IAEA code on the safety of NPPs and in the IAEA safety series, in particular the Requirements NS-R-1, establishing safety requirements related to the design of Nuclear Power Plants, and the Safety Guide NS-G-1.3 dealing with instrumentation and control systems important to safety in Nuclear Power Plants. The terminology and definitions used by SC 45A standards are consistent with those used by the IAEA.~~

The IEC SC 45A standard series comprises a hierarchy of four levels. The top-level documents of the IEC SC 45A standard series are IEC 61513 and IEC 63046.

IEC 61513 provides general requirements for instrumentation and control (I&C) systems and equipment that are used to perform functions important to safety in nuclear power plants (NPPs). IEC 63046 provides general requirements for electrical power systems of NPPs; it covers power supply systems including the supply systems of the I&C systems.

IEC 61513 and IEC 63046 are to be considered in conjunction and at the same level. IEC 61513 and IEC 63046 structure the IEC SC 45A standard series and shape a complete framework establishing general requirements for instrumentation, control and electrical power systems for nuclear power plants.

IEC 61513 and IEC 63046 refer directly to other IEC SC 45A standards for general requirements for specific topics, such as categorization of functions and classification of systems, qualification, separation, defence against common cause failure, control room design, electromagnetic compatibility, human factors engineering, cybersecurity, software and hardware aspects for programmable digital systems, coordination of safety and security requirements and management of ageing. The standards referenced directly at this second level should be considered together with IEC 61513 and IEC 63046 as a consistent document set.

At a third level, IEC SC 45A standards not directly referenced by IEC 61513 or by IEC 63046 are standards related to specific requirements for specific equipment, technical methods, or activities. Usually these documents, which make reference to second-level documents for general requirements, can be used on their own.

A fourth level extending the IEC SC 45 standard series, corresponds to the Technical Reports which are not normative.

The IEC SC 45A standards series consistently implements and details the safety and security principles and basic aspects provided in the relevant IAEA safety standards and in the relevant documents of the IAEA nuclear security series (NSS). In particular this includes the IAEA requirements SSR-2/1 , establishing safety requirements related to the design of nuclear power plants (NPPs), the IAEA safety guide SSG-30 dealing with the safety classification of structures, systems and components in NPPs, the IAEA safety guide SSG-39 dealing with the design of instrumentation and control systems for NPPs, the IAEA safety guide SSG-34 dealing with the design of electrical power systems for NPPs, the IAEA safety guide SSG-51 dealing with human factors engineering in the design of NPPs and the implementing guide NSS17 for computer security at nuclear facilities. The safety and security terminology and definitions used by the SC 45A standards are consistent with those used by the IAEA.

IEC 61513 and IEC 63046 have adopted a presentation format similar to the basic safety publication IEC 61508 with an overall life-cycle framework and a system life-cycle framework. Regarding nuclear safety, IEC 61513 and IEC 63046 provide the interpretation of the general requirements of IEC 61508-1, IEC 61508-2 and IEC 61508-4, for the nuclear application sector. In this framework, IEC 60880, IEC 62138 and IEC 62566 correspond to IEC 61508-3 for the nuclear application sector.

IEC 61513 and IEC 63046 refer to ISO 9001 as well as to IAEA GSR part 2 and IAEA GS-G-3.1 and IAEA GS-G-3.5 for topics related to quality assurance (QA).

At level 2, regarding nuclear security, IEC 62645 is the entry document for the IEC/SC 45A security standards. It builds upon the valid high level principles and main concepts of the generic security standards, in particular ISO/IEC 27001 and ISO/IEC 27002; it adapts them and completes them to fit the nuclear context and coordinates with the IEC 62443 series. At level 2, IEC 60964 is the entry document for the IEC/SC 45A control rooms standards, IEC 63351 is the entry document for the human factors engineering standards and IEC 62342 is the entry document for the ageing management standards.

NOTE 1 It is assumed that for the design of I&C systems in NPPs that implement conventional safety functions (e.g. to address worker safety, asset protection, chemical hazards, process energy hazards) international or national standards would be applied.

NOTE 2 IEC TR 64000 provides a more comprehensive description of the overall structure of the IEC SC 45A standards series and of its relationship with other standards bodies and standards.

NUCLEAR POWER PLANTS – INSTRUMENTATION AND CONTROL IMPORTANT TO SAFETY – ELECTRICAL EQUIPMENT CONDITION MONITORING METHODS –

Part 4: Oxidation induction techniques

1 Scope ~~and object~~

This part of IEC/IEEE 62582 specifies methods for condition monitoring of organic and polymeric materials in instrumentation and control systems using oxidation induction techniques in the detail necessary to produce accurate and reproducible measurements. It includes the requirements for sample preparation, the measurement system and conditions, and the reporting of the measurement results.

The different parts of IEC/IEEE 62582 are measurement standards, primarily for use in the management of ageing in initial qualification and after installation. IEC/IEEE 62582-1 includes requirements for the application of the other parts of the IEC/IEEE 62582 series and some elements which are common to all methods. Information on the role of condition monitoring in the qualification of equipment important to safety is found in ~~IEEE Std 323~~ IEC/IEEE 60780-323.

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.

ISO, IEC and IEEE maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp
- IEEE Standards Dictionary Online: available at http://dictionary.ieee.org

3.1
Oxidation induction time
OIT
relative measure of a stabilised material's resistance to oxidative decomposition, determined by the calorimetric measurement of the time interval to the onset of exothermic oxidation of the material at a specified temperature in an oxygen atmosphere, under atmospheric pressure

Note 1 to entry: OIT is expressed in minutes (min).

3.2
Oxidation induction temperature
OITP
calorimetric measurement of the temperature of the onset of exothermic oxidation of the material when subjected to a specified heating rate in an oxygen atmosphere, under atmospheric pressure

Note 1 to entry: OITP is expressed in degrees Celsius (°C).

4 Abbreviated terms and acronyms

CSPE	chlorosulphonated polyethylene
DSC	differential scanning calorimeter
EPDM	ethylene propylene diene monomer
EPR	ethylene propylene rubber
EVA	ethylene vinyl acetate
OIT	oxidation induction time
OITP	oxidation induction temperature
PE	polyethylene
PEEK	poly ether ether ketone
PVC	poly vinyl chloride
STA	simultaneous thermal analyzer
XLPE	cross-linked polyethylene

5 General description

Oxidation induction methods are based on the detection of the oxidation exotherm that occurs when a sample is heated in the presence of oxygen. This exotherm is sensitive to the level of degradation in some organic and polymeric materials and can be used as an indicator of ageing. There are two oxidation induction methods available, based on the time required to reach the onset of oxidation at a constant temperature (oxidation induction time – OIT) or based on the temperature at the onset of oxidation during a constant temperature ramp rate (oxidation induction temperature – OITP). The two methods are complementary, in that OITP is often effective in those materials where OIT is difficult to determine. OIT and OITP decrease with increasing degradation. The methods are generally related to the amount of antioxidants present in the material. As degradation proceeds, these antioxidants are depleted. For material without antioxidants OIT is not suitable, but OITP may give a useful result. In general, OIT and OITP are used to measure the thermal stability of polymers under isothermal (OIT) and dynamic (OITP) heating conditions in oxygen.

6 Applicability and reproducibility

The oxidation induction method is primarily suited to samples taken from materials (such as cable jackets or insulation) that are polyolefin-based (e.g. PE, XLPE). It can also be used for some materials based on ethylene-propylene polymers (e.g. EPR, EPDM) and for some ethylene vinyl acetate (EVA) materials. Oxidation Induction techniques may not be applicable to high temperature polymers in some cases. It is not applicable to high temperature polymers, such as PEEK. The method can be applied to other materials but problems with interpretation of the results may reduce the effectiveness of the test.

The method is generally not suitable for chlorinated polymers (e.g. PVC, CSPE) because of the corrosive degradation products evolved during the measurements, which can damage the instrument. For these materials, smaller sample masses (1 mg to 2 mg) may enable the method to be used with care. In addition, some modern systems are designed with features that protect the equipment from volatile off gases. To determine if the equipment is protected or if additional protective components are needed, consult the equipment manufacturer.

The method is not suitable for field use in the nuclear power plant but uses samples taken from the plant, which are then measured in the laboratory. Each OIT measurement in the laboratory can take up to 90 min to complete for unaged samples, decreasing for heavily aged samples, whereas OITP measurements typically take 30 min to 40 min.

Measurements of OIT typically have a standard deviation of 5 % to 10 % of the mean value whereas measurements of OITP typically have a standard deviation of 1 % to 3 % of the mean value, both within the same laboratory and between different laboratories. Some of this variation arises from inhomogeneity of the sample materials, which becomes significant when making condition measurements on samples whose mass is very low. OITP measurements are usually more reproducible than OIT measurements but require baseline data for interpretation of the changes.

7 Measurement procedure

7.1 Stabilisation of the polymeric materials

An appropriate time period shall be allowed for the polymeric materials in recently manufactured equipment to stabilise before any condition monitoring or accelerated ageing programmes are carried out. The time period over which the polymeric materials stabilise is normally dependent on the processing additives and polymer composition. If manufacturers' stabilisation time data are not available, a period of 6 months shall be allowed.

7.2 Sampling

7.2.1 General

Measurements of OIT or OITP provide information on the status of the equipment only at the specific location which has been sampled. The selection of the sample locations for condition monitoring shall be made based on the environmental conditions in representative areas during plant operation. It is important that these locations represent as wide a range of ageing conditions as possible with special consideration given to locations where ageing conditions could be severe, e.g. hotspots. The location of the sampling and available information about the environmental time history at the sample location selected shall be documented.

7.2.2 Sample requirements

To enable up to 5 measurements to be made on one specific sample, a minimum of 50 mg of material is needed. The material to be sampled shall be cleaned of surface debris. No solvents shall be used to clean the surface. Samples typically may take the form of slivers or scrapings of material taken from the surface of a cable jacket or a thin slice through insulation at a termination. The location of the sampling position shall be noted, including its radial distribution (i.e. whether it is a surface sample or a through thickness slice).

When sampling dual layer materials, e.g. cables, it shall be clear in the report how the samples have been obtained. The measured value will be dependent on the proportion of each layer included in the sample tested. For this reason, it is recommended that the layers are not mixed when technically possible.

Sampling and measurement procedures shall comply with local instructions, taking into account the safety of personnel and equipment

Care shall be taken to avoid unsuitable conditions in storage during the time period between sampling and measurements. It is recommended that samples be stored in the dark at temperatures not exceeding 25 °C and at humidity conditions within 45 % ~~and~~ to 75 %.

7.2.3 Precautions

When taking samples for OIT/OITP in the field, the equipment shall be visually inspected before and after the sampling in order to document that the equipment is not damaged.

If samples are to be taken from operational equipment in plant, the impact of such sampling on future operational use and qualification of such equipment shall be evaluated prior to sampling.

~~NOTE~~ Where removal of material from operational equipment is considered detrimental to qualification or future use, the equipment should be removed from service or repaired according to the utility's local procedures to ensure that qualification is maintained.

7.3 Sample preparation

Samples for each OIT or OITP measurement shall be ~~in the range 10 mg ± 2 mg in weight~~ 5 mg ± 1 mg in mass. Some older, less sensitive equipment may require the use of larger samples. In this case, 10 mg ± 1 mg of material should be used for the measurements. Regardless of which value is used, the mass of the sample in each measurement should be consistent. Each sample shall be chopped or ground into pieces with max dimensions of 1 mm. It is recommended that the chopped sample should be screened with a mesh to provide a particle size not greater than 0,85 mm as consistent sample preparation is important to enable reproducible oxidation of the sample during measurement. The chopped or granular sample shall be placed into a sample pan appropriate to the instrument being used and packed to achieve a good contact to the sample pan.

The sample pans shall be of aluminium, ceramic, or platinum and be open or have lids with holes or mesh to allow free access for oxygen during the measurement. A minimum of three samples shall be measured. If the results of measurements on three samples have a standard deviation > 10 % of the mean value for OIT or > 3 % of the mean value for OITP, an additional two samples should be measured if sufficient material is available. Out of these five measurements the highest and lowest will be removed and use the mean value of the remaining three samples.

~~NOTE 1~~ If smaller sample ~~weights~~ mass needs to be used, e.g. for chlorinated materials, this should be noted in the measurement report.

~~NOTE 2 If the results of measurements on three samples have a standard deviation >10 % of the mean value for OIT or >3 % of the mean value for OITP, an additional two samples should be measured.~~

7.4 Instrumentation

The instrument used for oxidation induction measurements shall be capable of determining exotherms in the sub-milliwatt range, e.g. a differential scanning calorimeter (DSC), simultaneous thermal analyzer (STA), or differential thermal analyzer (DTA). It shall be capable of maintaining an isothermal stability of ± 0,3 °C over the duration of the measurement, typically up to 90 min. The temperature ramp rate shall be programmable.

The instrument shall allow purging of the sample chamber with specific gases at a controllable rate. The distance between the gas-switching point and the instrument cell needs to be kept as short as possible, with a dead time of less than 1 min, to minimise the switching volume. Accordingly, for a flow rate of 75 ml·min^{-1}, the dead volume shall be less than 75 ml.

For analysis purposes, the difference in heat flow between a reference pan and the sample pan as a function of time (for OIT measurements) or temperature (for OITP measurements) shall be measured.

7.5 Calibration

The instrument shall be calibrated according to the manufacturer's recommendations and the relevant QA (quality assurance) procedure, using a suitable calibration standard for the temperature ranges being used (e.g. lead/indium/tin). Measurement of a reference sample shall be carried out prior to each batch of OIT or OITP measurements to verify this calibration.

7.6 OIT measurement method

7.6.1 Measurement procedure

The measurement procedure is illustrated in Figure 1. It includes the following steps.

- The sample is heated in nitrogen at a rate of temperature rise of 50 °C ·min^{-1} until 10 °C below the set temperature T_{set}. The ramp rate is then reduced to 5 °C·min^{-1} to reach the set temperature.
- The sample is then held for 2 min at the set temperature in nitrogen after which the atmosphere in the instrument is switched to oxygen.
- The oxidation exotherm is detected by a rapid increase in heat flow.
- The time from switching the atmosphere to oxygen until the sample starts oxidising is determined. This time is the oxidation induction time.

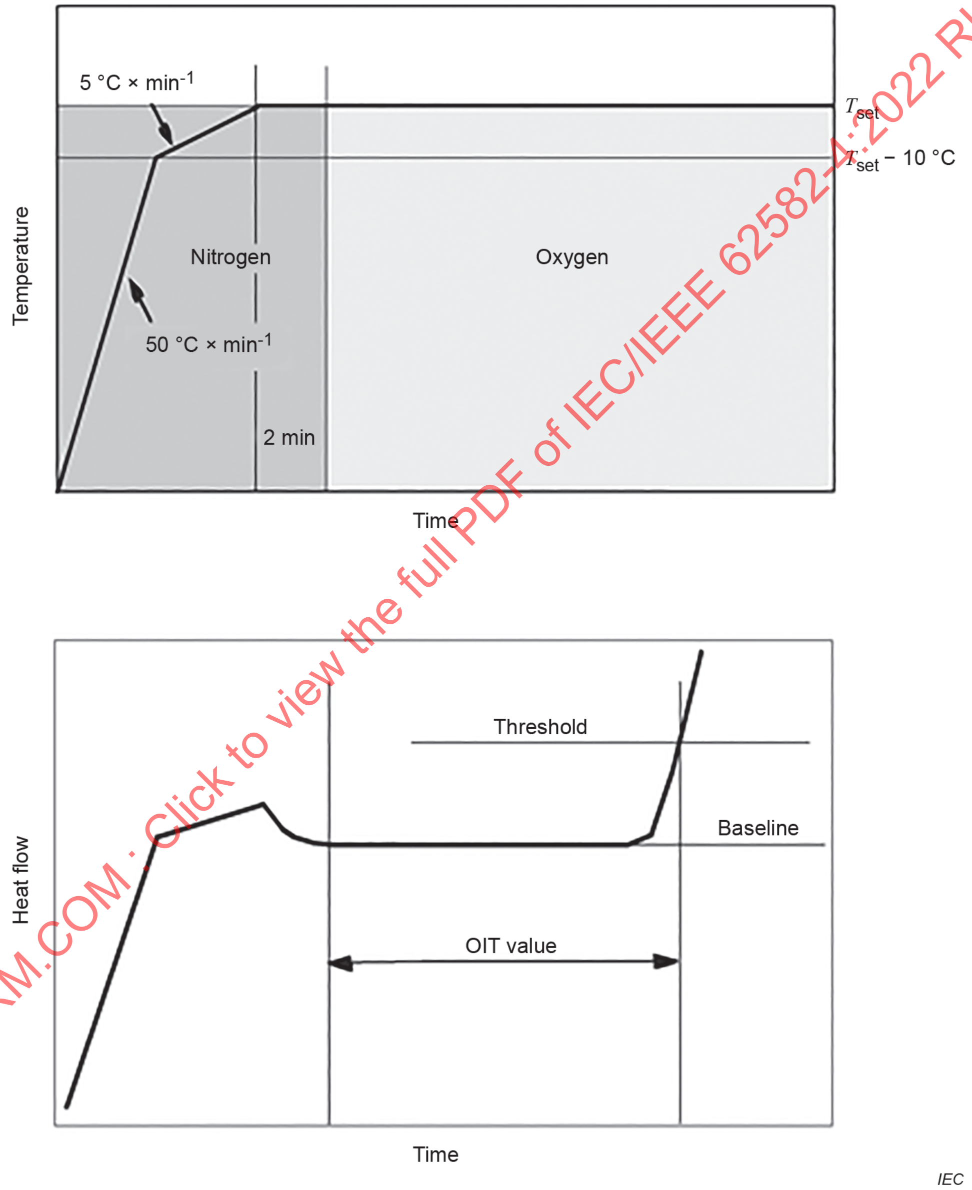


Figure 1 – OIT measurement – Schematic of temperature and gas profile and corresponding heat flow

7.6.2 Temperature profile

The reproducibility of OIT measurements is dependent on using a standardised thermal history. T_{set} for OIT measurements shall be 210 °C, provided that the oxidation induction time for unaged material is at least 30 min. The OIT value is highly dependent on T_{set} selected, see example in Annex C. If the OIT is less than 30 min for unaged material, then T_{set} shall be reduced in 10 °C increments until the OIT is > 30 min. If the OIT is > 90 min for unaged material, then T_{set} shall be increased in 10 °C increments until the OIT is < 90 min. Once the value of T_{set} has been selected for a specific material, the same value shall be used for all subsequent measurements on that material.

NOTE OIT > 90 min for unaged material is acceptable provided that the heat flow observed during the oxidation exotherm is sufficient to exceed the required threshold value (see 7.6.4.2).

7.6.3 Gas flow

The flow rate for oxygen during OIT tests shall be 75 ml·min^{-1} ± 25 ml·min^{-1}. The flow rate for nitrogen during the initial phase of OIT tests is not critical but it is recommended that 75 ml·min^{-1} ± 25 ml·min^{-1} be used.

NOTE Oxidation induction measurements can be affected by the oxygen flow rate used during the tests. For low flow rates (< 50 ml·min^{-1}), this can result in increased induction times in OIT tests. For the range of flow rates from 50 ml·min^{-1} to 100 ml·min^{-1}, oxidation induction times are not strongly dependent on the oxygen flow rate.

7.6.4 Determining the value of oxidation onset

7.6.4.1 Definition of the baseline

The threshold for oxidation induction is measured relative to a baseline, as shown in Figure 2. There will usually be a period of constant heat flow prior to the onset of oxidation; this is used as the baseline. In some materials, there is a linear change in heat flow before the onset of oxidation. This can also be used as a baseline and is referred to as a sloping baseline.

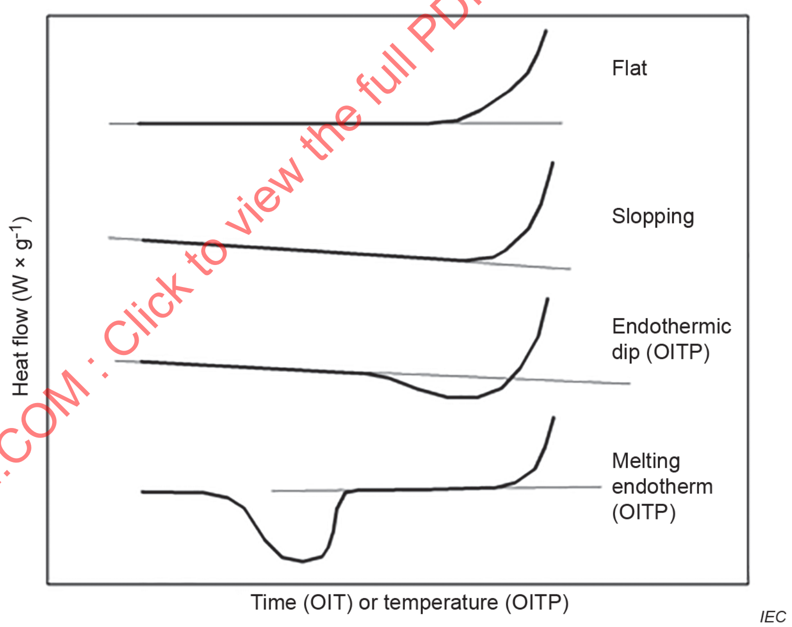


Figure 2 – Schematic showing the types of baselines (flat, sloping, endothermic dip, melting endotherm) observed for OIT and OITP measurements

7.6.4.2 Definition of the threshold and onset time

The threshold shall be defined at 0,1 W·g^{-1} relative to the baseline. The onset ~~time~~ is defined ~~by~~ as the ~~intersection of the test curve with~~ time at which the test curve crosses the threshold relative to the baseline, as shown in Figure 3.

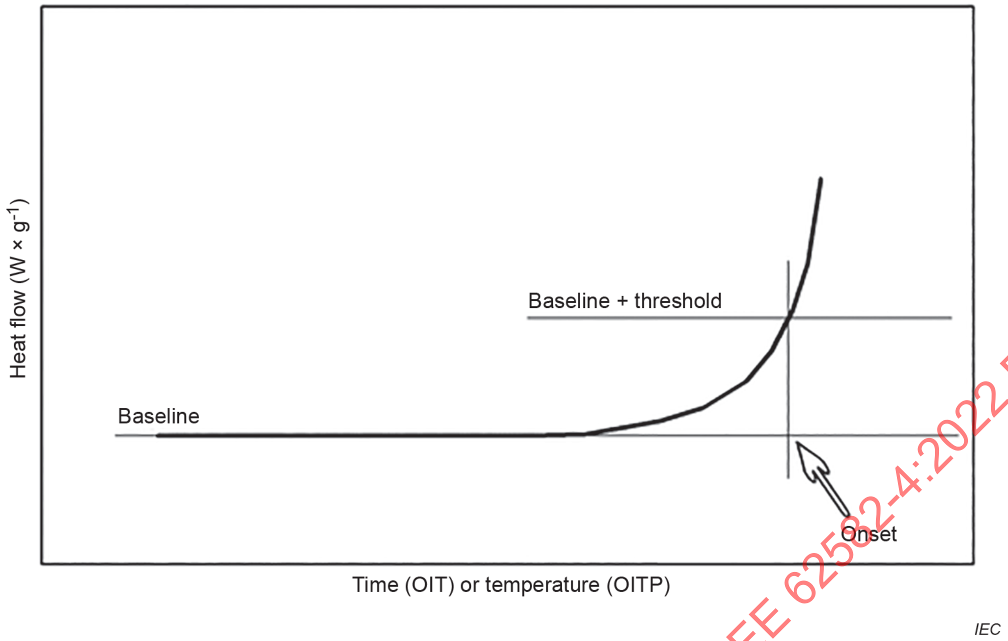


Figure 3 – Schematic showing definition of onset value for OIT and OITP measurements

Examples of the types of OIT thermogram that are observed in practice are given in Annex A.

7.6.5 Reporting

The measurement report shall include the following items.

a) Identification of the equipment sampled. This shall include:
   - details of the material being sampled, e.g. the generic polymer type, specific formulation numbers;
   - where the sample was taken from, e.g. surface scraping, through thickness slice;
   - for samples taken in plant, location within the plant.

b) Prehistory of the equipment sampled. This shall include:
   - time in service, or ageing time for laboratory aged samples;
   - the environmental conditions to which it has been exposed, e.g. temperature, radiation.

c) Sampling method, including sample preparation, have dual layer been separated (7.2).

d) Place and date of the measurements.

e) Instrument used and software version used for analysis (7.4).

f) Calibration procedure (7.5).

g) Type of sample pan used (7.3).

h) Oxygen flow rate during test (7.6.3).

i) Temperature profile, including ramp rates and hold times (7.6.2).

   ~~NOTE~~ If the instrument is capable of generating the information, the actual temperature profile should be included.

j) Baseline type and the rationale for using that specific baseline (7.6.4.1).

k) Onset type and the rationale for the selection of the onset in multiple onsets.

l) Threshold value used and the rationale if a non-standard value is used (7.6.4.2).

m) Number of samples measured (7.3).

n) Mean value of OIT, and standard deviation, in minutes.

o) Examples of the heat flow vs. time plot, particularly if the material does not show a flat baseline with single well-defined onset.

7.7 OITP measurement method

7.7.1 Measurement procedure

The measurement procedure is illustrated in Figure 4. It includes the following steps:

- The sample is heated in the instrument in oxygen at 10 °C·min^{-1}.
- The oxidation exotherm is detected by a rapid increase in heat flow.
- The temperature at which the sample starts oxidising is determined. This is the oxidation induction temperature.

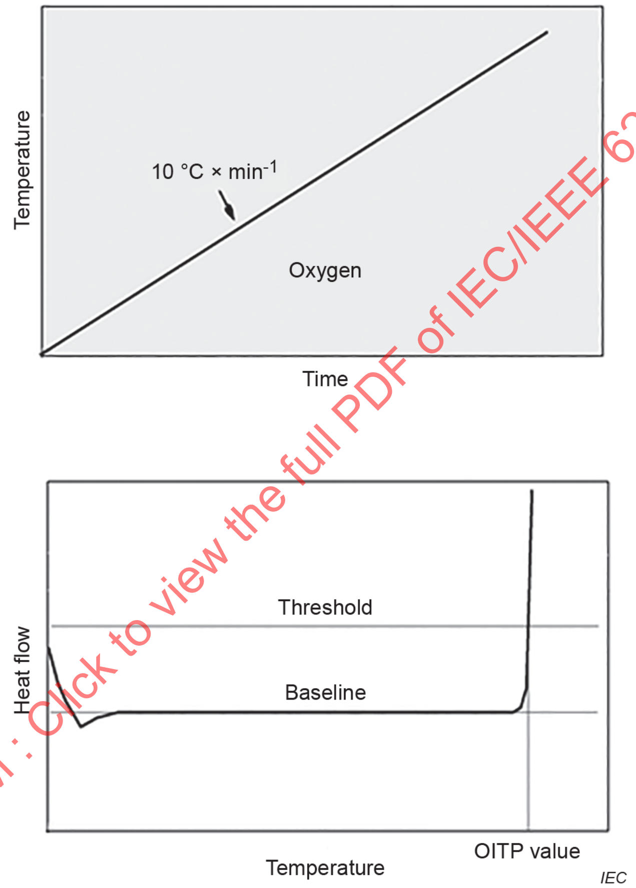


Figure 4 – Schematic of the temperature for OITP measurements and the corresponding heat flow

7.7.2 Temperature profile

The temperature ramp rate shall be 10 °C·min^{-1} in oxygen from the start temperature until the oxidation onset is observed. When carrying out consecutive measurements, the starting temperature shall be < 50 °C.

7.7.3 Gas flow

The flow rate for oxygen during OITP measurements shall be 75 ml·min^{-1} ± 25 ml·min^{-1}.

NOTE Oxidation induction measurements can be affected by the oxygen flow rate used during the tests. For low flow rates (< 50 ml·min^{-1}), this can result in increased induction temperatures in OITP tests. For the range of flow rates from 50 ml·min^{-1} to 100 ml·min^{-1}, oxidation induction temperatures are not strongly dependent on the oxygen flow rate.

7.7.4 Determining the value of oxidation onset

7.7.4.1 Definition of the baseline

The threshold for oxidation induction is measured relative to a baseline, as shown in Figure 2. There will usually be a period of constant heat flow prior to the onset of oxidation; this is used as the baseline. In some materials, there is a linear change in heat flow before the onset of oxidation. This can also be used as a baseline and is referred to as a sloping baseline.

7.7.4.2 Definition of the threshold and onset temperature

The threshold shall be defined at 0,5 W·g^{-1} relative to the baseline. The onset ~~temperature~~ is defined by the ~~intersection of the test curve with~~ temperature at which test curve crosses the threshold relative to the baseline, as shown in Figure 3.

NOTE Heat flows during OITP measurements are considerably higher than in OIT measurements. The selection of a higher threshold value than that used for OIT measurements enables a more consistent value to be obtained for the onset.

Examples of the types of OITP thermogram that are observed in practice are given in Annex A.

7.7.5 Reporting

The measurement report shall include the following items.

a) Identification of the equipment sampled. This shall include:
   - details of the material being sampled e.g. the generic polymer type, specific formulation numbers;
   - where the sample was taken from, e.g. surface scraping, through thickness slice;
   - for samples taken in plant, location within the plant.

b) Prehistory of the equipment sampled. This shall include:
   - time in service, or ageing time for laboratory aged samples;
   - the environmental conditions to which it has been exposed, e.g. temperature, radiation.

c) Sampling method, including sample preparation, have dual layers been separated (7.2).

d) Instrument used and software version used for analysis (7.4).

e) Place and date of the measurement.

f) Calibration procedure (7.5).

g) Type of sample pan used (7.3).

h) Oxygen flow rate (7.7.3).

i) Temperature ramp rate.

   ~~NOTE~~ If the instrument is capable of generating the information, the actual temperature profile should be included.

j) Baseline type and the rationale for using that specific baseline (7.7.4.1).

k) Threshold value used and the rationale if a non-standard value is used (7.7.4.2).

l) Number of samples measured.

m) Mean value of OITP, and standard deviation, in °C.

n) Examples of the heat flow versus temperature plot, particularly if the material does not show a flat baseline with a well-defined onset.

An example of a measurement report is given in Annex B.

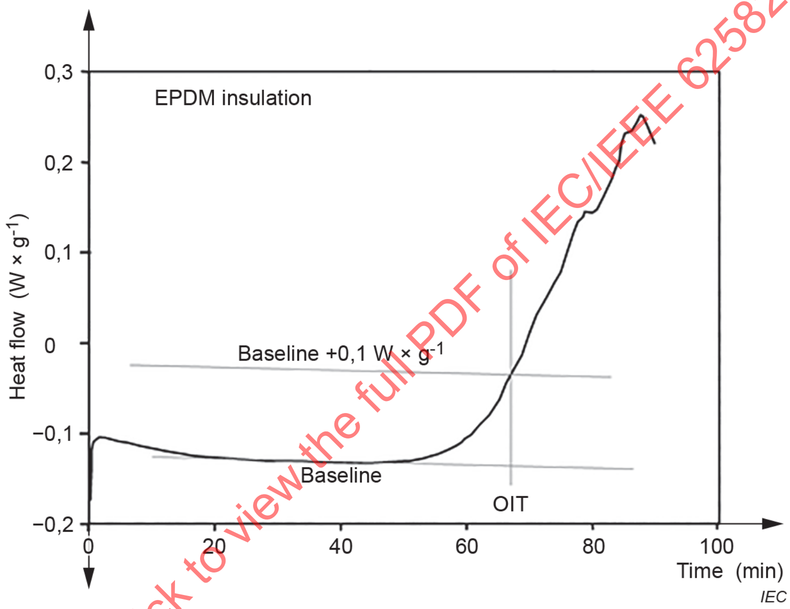
Annex A
(informative)

Interpretation of thermogram

A.1 Interpretation of OIT thermograms

The ideal OIT plot of heat flow versus time (as shown in Figure 3) is rarely seen in practice. More usually, the baseline is sloping or is difficult to define. Some of the types of plot that are observed are shown below. The approach that could be used for each type is briefly discussed. All of the examples are taken from actual OIT plots.

Where there is a clear baseline and a well-defined onset, it is straight forward to define the baseline and the onset value at the threshold of 0,1 W·g^{-1}, as shown in Figure A.1.

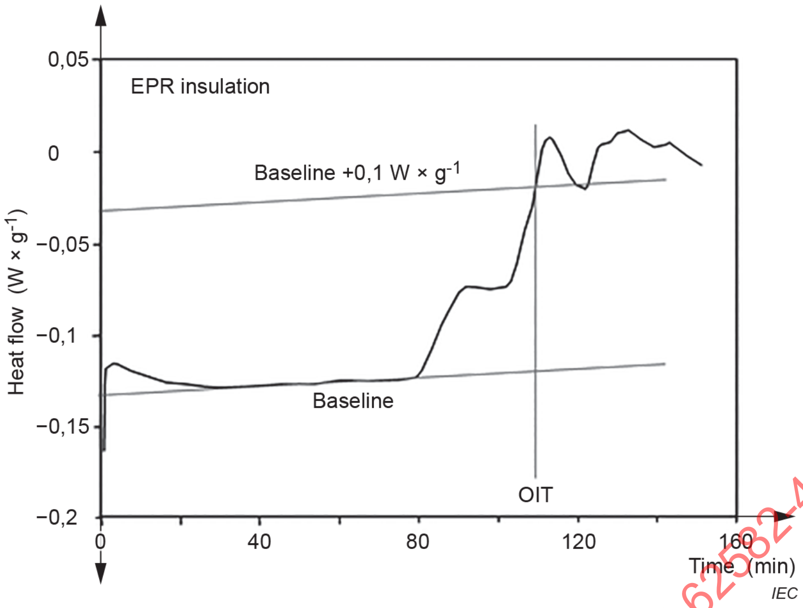


Set temperature T_{set} 210 °C

Figure A.1 – Example of an OIT plot with clear baseline and onset

Multiple onsets are often seen in EPR and EVA based materials. In this case, the standard threshold value of 0,1 W·g^{-1} may ignore one of the onsets, as shown in Figure A.2. However, the first onset may be important in defining degradation so it may be appropriate for an additional value of the threshold to be used. Alternatively, several onset values may be determined at different threshold levels. Where this is the case, the rationale for selecting the threshold and onset should be given in the measurement report.

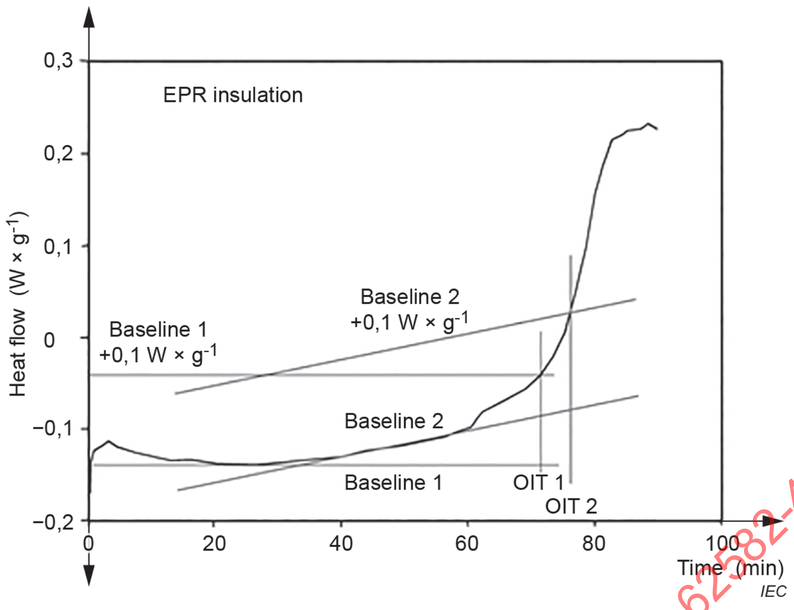
In the two examples shown in Figure A.1 and Figure A.2, the baseline was relatively simple to define, with a good section of either constant heat flow or linearly increasing heat flow. However, in many cases it is not obvious how to define the baseline. The example in Figure A.3 shows one of this type of plot. One could use the short section of linearly increasing baseline (baseline 2) or use the minimum value of the heat flow (baseline 1). Note that the choice of baseline makes a large difference to the value of the onset time, particularly where the onset of oxidation is gradual, as in this example. It is therefore very important to use a consistent definition of the baseline between measurements. This variation in onset value depending on the baseline used is one of the reasons why measurements of OIT from different laboratories can be variable.



Set temperature T_{set} 210 °C

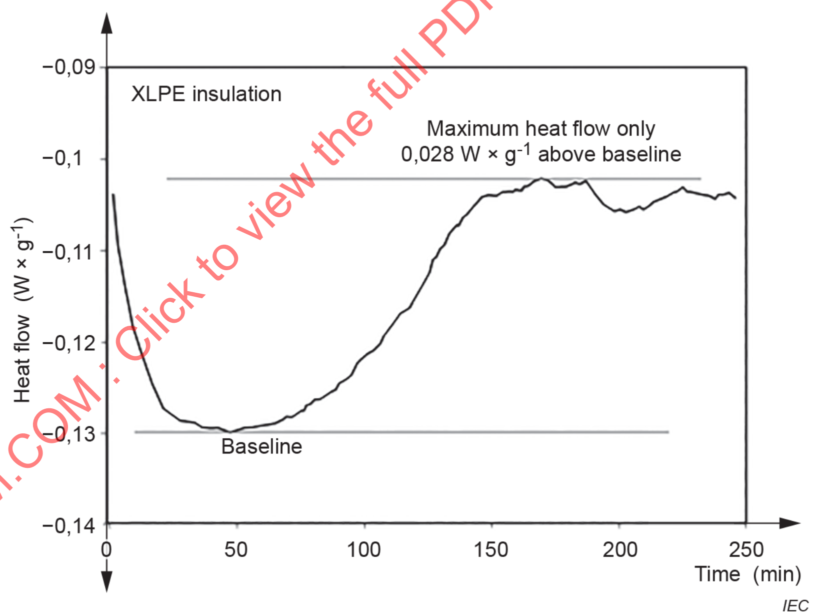
Figure A.2 – Example of OIT plot with multiple onsets

Another area that can cause problems of interpretation is where the heat flows during oxidation are too low for the standard threshold value of 0,1 W·g^{-1} to be appropriate. In the example shown in Figure A.4, the maximum heat flow observed is only 0,028 W·g^{-1} above the minimum and there is no clearly defined baseline (note that the long timescale of the test has compressed the time axis relative to the other examples). For this material, one could use the minimum heat flow as the baseline and use a lower threshold value to define the onset. However, the test plot indicates that, for this particular material, the OIT measurement procedure was not appropriate. Note that the long time scale and low heat flow indicate that the temperature selected for the test was too low.



Set temperature T_{set} 210 °C

Figure A.3 – Example of OIT plot where the baseline is difficult to define



Set temperature T_{set} 190 °C

Figure A.4 – Example of OIT plot where heat flow is too low to use standard 0,1 W·g^{-1} threshold

A.2 Interpretation of OITP thermograms

Interpretation of OITP plots is usually more straightforward than for OIT plots. This is because baselines are usually consistent and single onsets with a high heat flow are normally observed. This makes definition of the onset value less dependent on the baseline used. Figure A.5 shows the type of plot seen in an OITP measurement on a non-crystalline polymer sample.

In materials which are semi-crystalline e.g. XLPE, a melting endotherm will be observed. This is usually at a much lower temperature than the oxidation onset, so that a suitable baseline can still be defined. Figure A.6 shows an example of the OITP plot for a semi-crystalline material.

EPR and EVA based materials often show a small endothermic dip immediately prior to the onset of oxidation. In this case, the baseline should be defined prior to the endothermic dip, as shown in Figure A.7. If the lowest point of the endotherm is used instead (baseline 2 in Figure A.7), this should be stated clearly in the measurement report. It can be seen that the value of the onset temperature is not strongly dependent on the baseline selected.

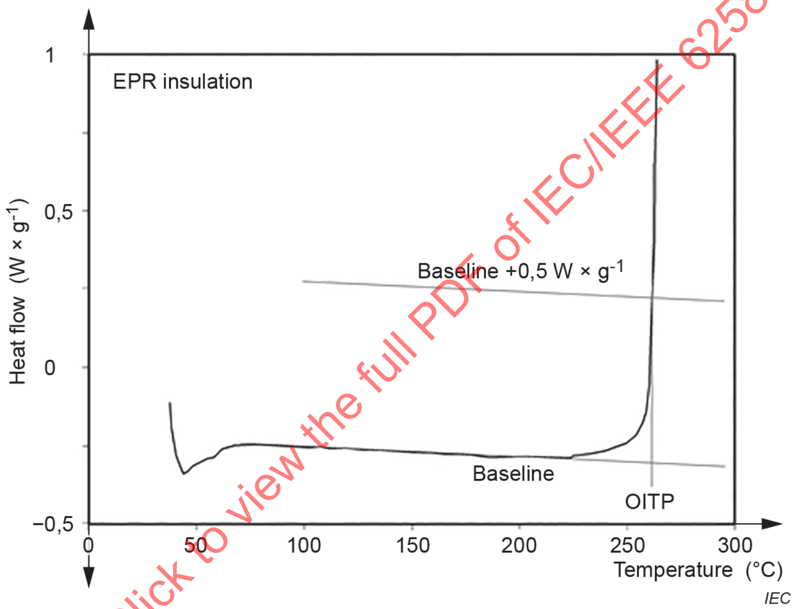


Figure A.5 – Example of an OITP plot with a well-defined baseline and onset

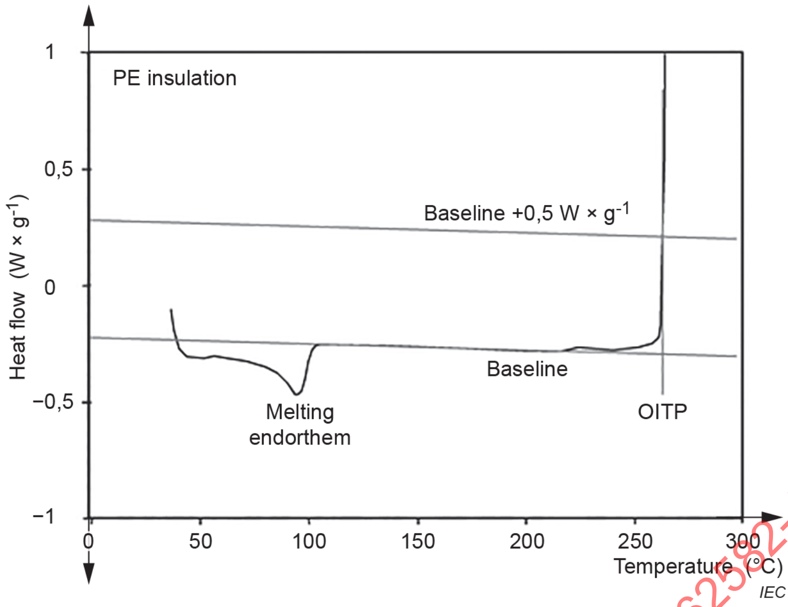


Figure A.6 – Example of an OITP plot for a semi-crystalline material showing a melting endotherm prior to the oxidation onset

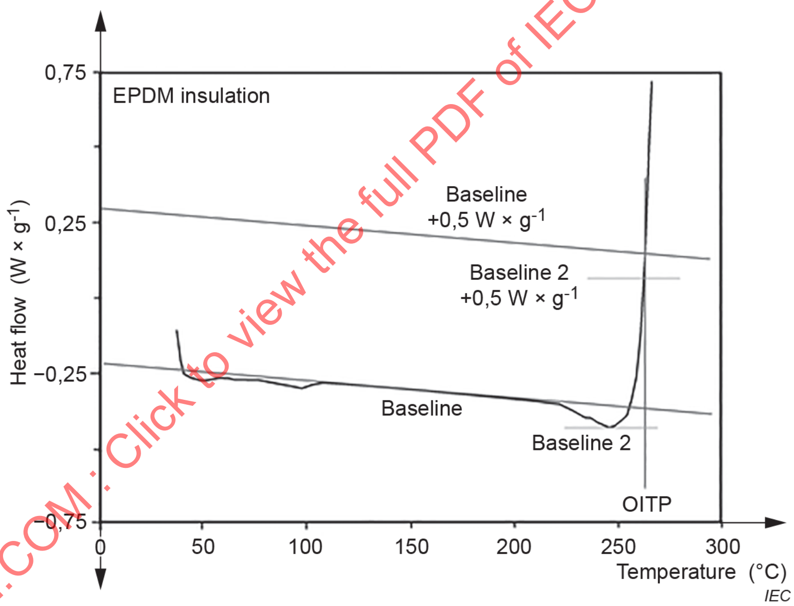


Figure A.7 – Example of an OITP plot showing an endothermic dip immediately prior to the oxidation onset

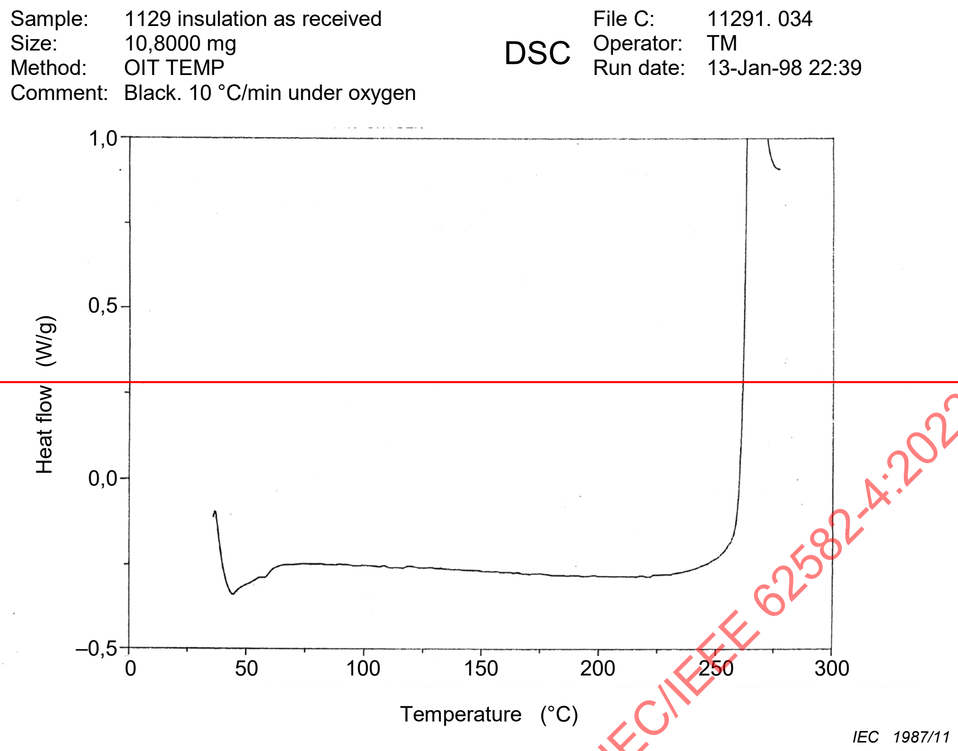
Annex B
(informative)

Example of a measurement report from OITP ~~measurements~~ and OIT

~~This example is from the round-robin test programme carried out as part of an IAEA coordinated research programme on cable ageing.~~

~~**Specimen ID**~~	~~1129 wire insulation, manufactured by AIW, U.S.A. Taken from cable with cable code D14~~
~~**Material**~~	~~4 core EPR + CSPE bonded insulation on stranded Cu – black. Insulation thickness 1 mm~~
~~**Sample type**~~	~~Through thickness slice~~
~~**Pre-history**~~	~~Artificially aged in dry heat test chamber. Method IEC 60068 Test Ba. Ageing conditions: 120 °C for 42 days~~
~~**Place and date of measurement**~~	~~13 January 1998~~ ~~Ontario Hydro~~
~~**Sampling method and weight**~~	~~Chopped with sharp blade to <1 mm, screened with no. 20 mesh to 0,85 mm sample weight 10 mg~~
~~**Instrument**~~	~~DuPont 2100 DSC~~
~~**Calibration method**~~	~~Indium/lead/tin standard sample. See calibration report No xxxx~~
~~**Sample pan**~~	~~Al open pan~~
~~**Oxygen flow rate**~~	~~100 ml·min^{-1}~~
~~**Temperature ramp rate**~~	~~10 °C·min^{-1}~~
~~**Baseline type**~~	~~sloping~~
~~**Threshold value**~~	~~0,1 W·g^{-1}~~
~~**No. of samples measured**~~	~~3~~
~~**OITP value**~~	~~Mean value 255,8 °C; Standard deviation 0,6 °C~~

~~Example of test plot (see Figure B.1):~~



~~**Figure B.1 – Example of test plot**~~

~~Signature:~~

~~Date:~~

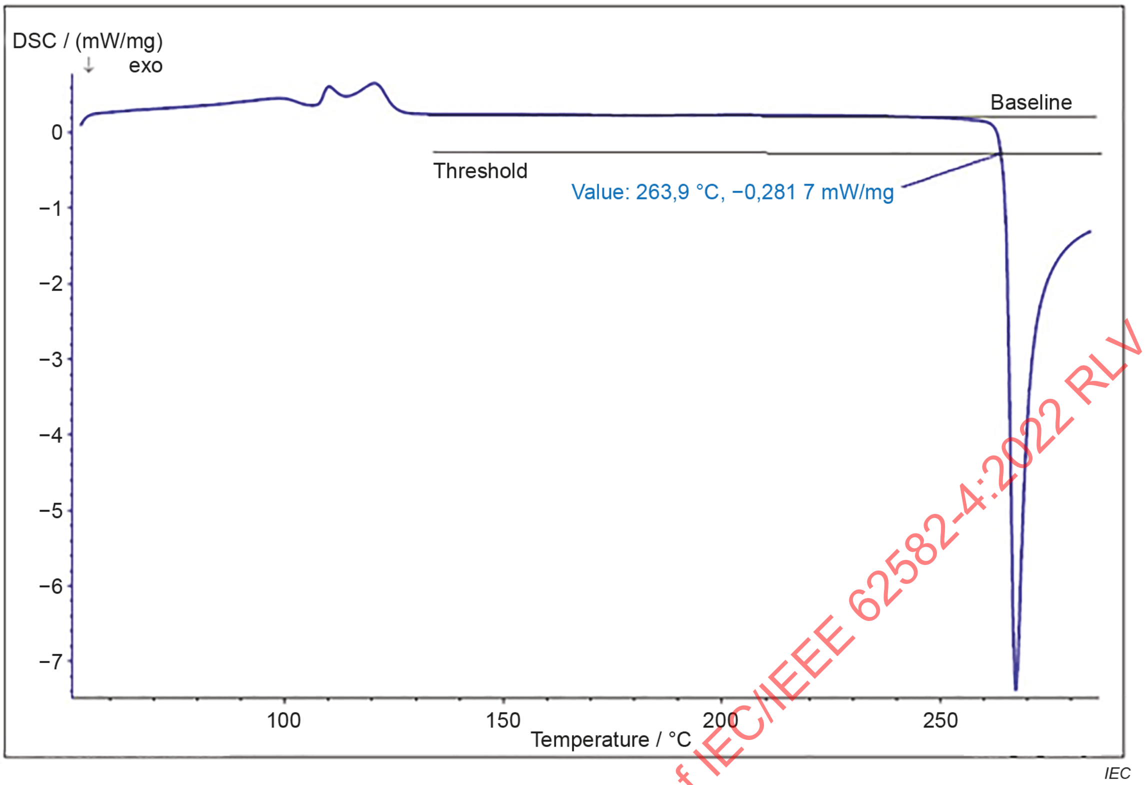
B.1 OITP measurements

These examples in Table B.1 and Figure B.1 are from the round-robin test programme carried out as part of an IAEA coordinated research programme on cable ageing (IAEA-TECDOC-1825:2017).

Table B.1 – Example of a measurement report from OITP

Specimen ID	Rockbestos Firewall III
Material	XLPE flame retardent insulation
Sample type	Through thickness slice
Prehistory	As-received, unaged
Place and date of measurement	3-Dec-2012 VUJE, Slovakia
Sampling method and mass	Chopped with sharp blade to < 1 mm, screened with no. 20 mesh to 0,85 mm sample mass 10 mg
Instrument	Netzsch DSC 200F3
Calibration method	KNO3, In, Sn, Bi, Zn, CsCl standard sample.
Sample pan	Al open pan
Oxygen flow rate	100 ml·min^{-1}
Temperature ramp rate	10 °C ·min^{-1}
Baseline type	flat
Threshold value	0,5 W·g^{-1}
No. of samples measured	3
OITP value	Mean value 262,4 °C; Standard deviation 1,4 °C

Example of test plot (see Figure B.1):



NOTE Exotherm is down in this plot.

Figure B.1 – Example of OITP test plot

Signature:

Date:

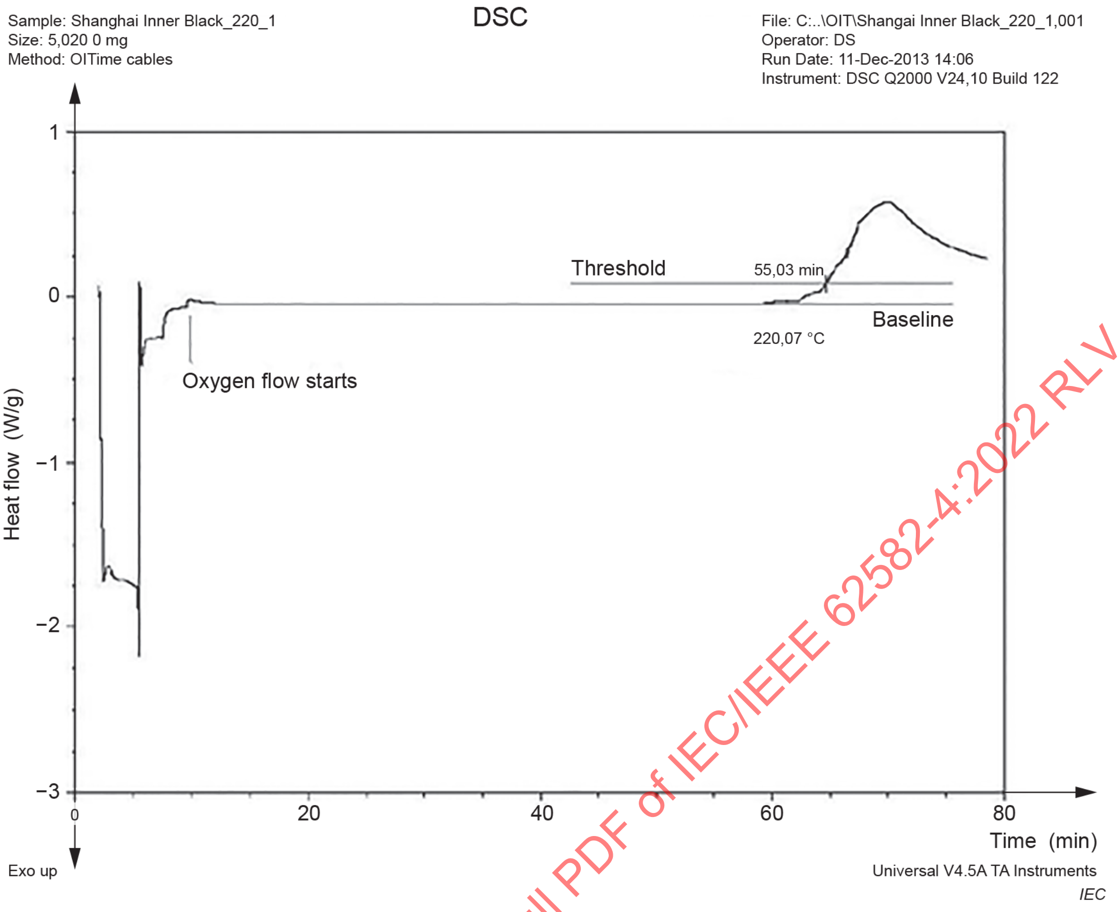
B.2 OIT measurements

These examples in Table B.2 and in Figure B.2 are from the round-robin test programme carried out as part of an IAEA coordinated research programme on cable ageing (IAEA-TECDOC-1825:2017).

Table B.2 – Example of a measurement report from OIT

Specimen ID	**WDZA-HK1-KEGJ**
Material	XLPO halogen-free flame retardent insulation (black), Shanghai Special Cable, China
Sample type	Through thickness slice
Prehistory	As-received, unaged
Place and date of measurement	11-Dec-2013 NIST, USA
Sampling method and mass	Chopped with sharp blade to <1 mm sample mass 5 mg
Instrument	TA Instruments Universal V4.5A
Calibration method	Indium/lead/tin standard sample
Sample pan	Al open pan
Oxygen flow rate	100 ml·min^{-1}
Set temperature	220 °C
Temperature ramp rate	50 °C ·min–1 to 210 °C, then 10 °C ·min^{-1} to set temperature
Baseline type	flat
Threshold value	0,1 W·g^{-1}
No. of samples measured	3
OIT value	Mean value 53,0 min; Standard deviation 2,3 min

Example of test plot (see Figure B.2):



NOTE Exotherm is up in this plot.

Figure B.2 – Example of OIT test plot

Signature:

Date:

Annex C
(informative)

Influence of set temperature on the OIT value

The OIT value measured is highly dependent on the set temperature selected. The loss of antioxidants can normally be described as a thermally activated process. Figure C.1 shows an example of the ratio between the OIT value at different temperatures T and at 200 °C.

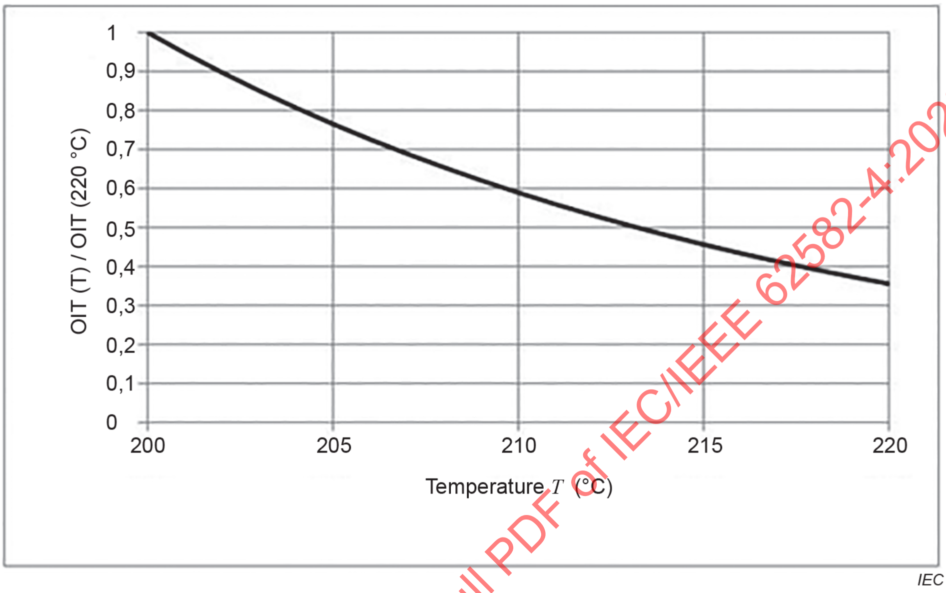


Figure C.1 – Example of the influence of set temperature on the OIT value

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~~IEC 60780, *Nuclear power plants – Electrical equipment of the safety system – Qualification*~~

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INTERNATIONAL STANDARD

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Nuclear power plants – Instrumentation and control important to safety – Electrical equipment condition monitoring methods – Part 4: Oxidation induction techniques

Centrales nucléaires de puissance – Instrumentation et contrôle-commande importants pour la sûreté – Méthodes de surveillance de l'état des matériels électriques – Partie 4: Techniques d'induction à l'oxydation

IEC/IEEE 62582-4:2022-11(en-fr)

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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NUCLEAR POWER PLANTS – INSTRUMENTATION AND CONTROL IMPORTANT TO SAFETY – ELECTRICAL EQUIPMENT CONDITION MONITORING METHODS –

Part 4: Oxidation induction techniques

FOREWORD

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IEC/IEEE 62582-4 was prepared by subcommittee 45A: Instrumentation, control and electrical power systems of nuclear facilities, of IEC technical committee 45: Nuclear instrumentation, in cooperation with Nuclear Power Engineering Committee of the IEEE, under the IEC/IEEE Dual Logo Agreement between IEC and IEEE. It is an International Standard.

This document is published as an IEC/IEEE Dual Logo standard.

This second edition cancels and replaces the first edition, published in 2011. This edition constitutes a technical revision.

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

a) Consideration of publication of IEC/IEEE 60780-323;
b) An example added in Annex B and update;
c) Annex C added.

The text of this International Standard is based on the following IEC documents:

Draft	Report on voting
45A/1435/FDIS	45A/1445/RVD

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 International Standard is English.

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

A list of all parts of IEC/IEEE 62582 series, under the general title *Nuclear power plants – Instrumentation and control important to safety – Electrical equipment condition monitoring methods*, can be found on the IEC website.

The IEC Technical Committee and IEEE Technical Committee have 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,
- replaced by a revised edition, or
- amended.

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INTRODUCTION

a) Technical background, main issues and organisation of this standard

This part of IEC/IEEE 62582 specifically focuses on oxidation induction methods for condition monitoring for the management of ageing of electrical equipment installed in nuclear power plants. The methods are primarily suited to samples taken from materials that are polyolefin-based, but they can also be used for some materials based on ethylene-propylene polymers and for some ethylene vinyl acetate materials.

This part 4 of IEC/IEEE 62582 is the fourth part of the IEC/IEEE 62582 series.

IEC/IEEE 62582 series is issued with a joint logo which makes it applicable to the management of ageing of electrical equipment qualified to IEEE as well as IEC Standards.

IEC/IEEE 60780-323 includes the concept and role that condition based qualification could be used in equipment qualification as an adjunct to qualified life. In equipment qualification, the condition of the equipment for which acceptable performance was demonstrated is the qualified condition. The qualified condition is the condition of equipment, prior to the start of a design basis event, for which the equipment was demonstrated to meet the design requirements for the specified service conditions.

Significant research has been performed on condition monitoring techniques and the use of these techniques in equipment qualification as noted in NUREG/CR-6704, Vol. 2 (BNL-NUREG-52610) JNES-SS-0903, 2009 and IAEA-TECDOC-1825:2017.

It is intended that this document be used by test laboratories, operators of nuclear power plants, systems evaluators, and licensors.

b) Situation of the current standard in the structure of the IEC SC 45A standard series

IEC/IEEE 62582-4 is the third level IEC SC 45A document tackling the specific issue of application and performance of oxidation induction measurements in the management of ageing of electrical instrument and control equipment in nuclear power plants.

IEC/IEEE 62582-4 is to be read in association with IEC/IEEE 62582-1, which provides background and guidelines for the application of methods for condition monitoring of electrical equipment important to safety of nuclear power plants.

For more details on the structure of the IEC SC 45A standard series, see item d) of this introduction.

c) Recommendations and limitations regarding the application of this standard

It is important to note that this document establishes no additional functional requirements for safety systems.

d) Description of the structure of the IEC SC45A standard series and relationships with other IEC documents and other bodies documents (IAEA, ISO)

The IEC SC 45A standard series comprises a hierarchy of four levels. The top-level documents of the IEC SC 45A standard series are IEC 61513 and IEC 63046.

IEC 61513 provides general requirements for instrumentation and control (I&C) systems and equipment that are used to perform functions important to safety in nuclear power plants (NPPs). IEC 63046 provides general requirements for electrical power systems of NPPs; it covers power supply systems including the supply systems of the I&C systems.

IEC 61513 and IEC 63046 are to be considered in conjunction and at the same level. IEC 61513 and IEC 63046 structure the IEC SC 45A standard series and shape a complete framework establishing general requirements for instrumentation, control and electrical power systems for nuclear power plants.

IEC 61513 and IEC 63046 refer directly to other IEC SC 45A standards for general requirements for specific topics, such as categorization of functions and classification of systems, qualification, separation, defence against common cause failure, control room design, electromagnetic compatibility, human factors engineering, cybersecurity, software and hardware aspects for programmable digital systems, coordination of safety and security requirements and management of ageing. The standards referenced directly at this second level should be considered together with IEC 61513 and IEC 63046 as a consistent document set.

At a third level, IEC SC 45A standards not directly referenced by IEC 61513 or by IEC 63046 are standards related to specific requirements for specific equipment, technical methods, or activities. Usually these documents, which make reference to second-level documents for general requirements, can be used on their own.

A fourth level extending the IEC SC 45 standard series, corresponds to the Technical Reports which are not normative.

The IEC SC 45A standards series consistently implements and details the safety and security principles and basic aspects provided in the relevant IAEA safety standards and in the relevant documents of the IAEA nuclear security series (NSS). In particular this includes the IAEA requirements SSR-2/1 , establishing safety requirements related to the design of nuclear power plants (NPPs), the IAEA safety guide SSG-30 dealing with the safety classification of structures, systems and components in NPPs, the IAEA safety guide SSG-39 dealing with the design of instrumentation and control systems for NPPs, the IAEA safety guide SSG-34 dealing with the design of electrical power systems for NPPs, the IAEA safety guide SSG-51 dealing with human factors engineering in the design of NPPs and the implementing guide NSS17 for computer security at nuclear facilities. The safety and security terminology and definitions used by the SC 45A standards are consistent with those used by the IAEA.

IEC 61513 and IEC 63046 have adopted a presentation format similar to the basic safety publication IEC 61508 with an overall life-cycle framework and a system life-cycle framework. Regarding nuclear safety, IEC 61513 and IEC 63046 provide the interpretation of the general requirements of IEC 61508-1, IEC 61508-2 and IEC 61508-4, for the nuclear application sector. In this framework, IEC 60880, IEC 62138 and IEC 62566 correspond to IEC 61508-3 for the nuclear application sector.

IEC 61513 and IEC 63046 refer to ISO 9001 as well as to IAEA GSR part 2 and IAEA GS-G-3.1 and IAEA GS-G-3.5 for topics related to quality assurance (QA).

At level 2, regarding nuclear security, IEC 62645 is the entry document for the IEC/SC 45A security standards. It builds upon the valid high level principles and main concepts of the generic security standards, in particular ISO/IEC 27001 and ISO/IEC 27002; it adapts them and completes them to fit the nuclear context and coordinates with the IEC 62443 series. At level 2, IEC 60964 is the entry document for the IEC/SC 45A control rooms standards, IEC 63351 is the entry document for the human factors engineering standards and IEC 62342 is the entry document for the ageing management standards.

NOTE 1 It is assumed that for the design of I&C systems in NPPs that implement conventional safety functions (e.g. to address worker safety, asset protection, chemical hazards, process energy hazards) international or national standards would be applied.

NOTE 2 IEC TR 64000 provides a more comprehensive description of the overall structure of the IEC SC 45A standards series and of its relationship with other standards bodies and standards.

NUCLEAR POWER PLANTS – INSTRUMENTATION AND CONTROL IMPORTANT TO SAFETY – ELECTRICAL EQUIPMENT CONDITION MONITORING METHODS –

Part 4: Oxidation induction techniques

1 Scope

This part of IEC/IEEE 62582 specifies methods for condition monitoring of organic and polymeric materials in instrumentation and control systems using oxidation induction techniques in the detail necessary to produce accurate and reproducible measurements. It includes the requirements for sample preparation, the measurement system and conditions, and the reporting of the measurement results.

The different parts of IEC/IEEE 62582 are measurement standards, primarily for use in the management of ageing in initial qualification and after installation. IEC/IEEE 62582-1 includes requirements for the application of the other parts of the IEC/IEEE 62582 series and some elements which are common to all methods. Information on the role of condition monitoring in the qualification of equipment important to safety is found in IEC/IEEE 60780-323.

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.

ISO, IEC and IEEE maintain terminological databases for use in standardization at the following addresses:

- IEC Electropedia: available at http://www.electropedia.org/
- ISO Online browsing platform: available at http://www.iso.org/obp
- IEEE Standards Dictionary Online: available at http://dictionary.ieee.org

3.1
Oxidation induction time
OIT
relative measure of a stabilised material's resistance to oxidative decomposition, determined by the calorimetric measurement of the time interval to the onset of exothermic oxidation of the material at a specified temperature in an oxygen atmosphere, under atmospheric pressure

Note 1 to entry: OIT is expressed in minutes (min).

3.2
Oxidation induction temperature
OITP
calorimetric measurement of the temperature of the onset of exothermic oxidation of the material when subjected to a specified heating rate in an oxygen atmosphere, under atmospheric pressure

Note 1 to entry: OITP is expressed in degrees Celsius (°C).

4 Abbreviated terms and acronyms

CSPE	chlorosulphonated polyethylene
DSC	differential scanning calorimeter
EPDM	ethylene propylene diene monomer
EPR	ethylene propylene rubber
EVA	ethylene vinyl acetate
OIT	oxidation induction time
OITP	oxidation induction temperature
PE	polyethylene
PEEK	poly ether ether ketone
PVC	poly vinyl chloride
STA	simultaneous thermal analyzer
XLPE	cross-linked polyethylene

5 General description

Oxidation induction methods are based on the detection of the oxidation exotherm that occurs when a sample is heated in the presence of oxygen. This exotherm is sensitive to the level of degradation in some organic and polymeric materials and can be used as an indicator of ageing. There are two oxidation induction methods available, based on the time required to reach the onset of oxidation at a constant temperature (oxidation induction time – OIT) or based on the temperature at the onset of oxidation during a constant temperature ramp rate (oxidation induction temperature – OITP). The two methods are complementary, in that OITP is often effective in those materials where OIT is difficult to determine. OIT and OITP decrease with increasing degradation. The methods are generally related to the amount of antioxidants present in the material. As degradation proceeds, these antioxidants are depleted. For material without antioxidants OIT is not suitable, but OITP may give a useful result. In general, OIT and OITP are used to measure the thermal stability of polymers under isothermal (OIT) and dynamic (OITP) heating conditions in oxygen.

6 Applicability and reproducibility

The oxidation induction method is primarily suited to samples taken from materials (such as cable jackets or insulation) that are polyolefin-based (e.g. PE, XLPE). It can also be used for some materials based on ethylene-propylene polymers (e.g. EPR, EPDM) and for some ethylene vinyl acetate (EVA) materials. Oxidation Induction techniques may not be applicable to high temperature polymers in some cases. It is not applicable to high temperature polymers, such as PEEK. The method can be applied to other materials but problems with interpretation of the results may reduce the effectiveness of the test.

The method is generally not suitable for chlorinated polymers (e.g. PVC, CSPE) because of the corrosive degradation products evolved during the measurements, which can damage the instrument. For these materials, smaller sample masses (1 mg to 2 mg) may enable the method to be used with care. In addition, some modern systems are designed with features that protect the equipment from volatile off gases. To determine if the equipment is protected or if additional protective components are needed, consult the equipment manufacturer.

The method is not suitable for field use in the nuclear power plant but uses samples taken from the plant, which are then measured in the laboratory. Each OIT measurement in the laboratory can take up to 90 min to complete for unaged samples, decreasing for heavily aged samples, whereas OITP measurements typically take 30 min to 40 min.

Measurements of OIT typically have a standard deviation of 5 % to 10 % of the mean value whereas measurements of OITP typically have a standard deviation of 1 % to 3 % of the mean value, both within the same laboratory and between different laboratories. Some of this variation arises from inhomogeneity of the sample materials, which becomes significant when making condition measurements on samples whose mass is very low. OITP measurements are usually more reproducible than OIT measurements but require baseline data for interpretation of the changes.

7 Measurement procedure

7.1 Stabilisation of the polymeric materials

An appropriate time period shall be allowed for the polymeric materials in recently manufactured equipment to stabilise before any condition monitoring or accelerated ageing programmes are carried out. The time period over which the polymeric materials stabilise is normally dependent on the processing additives and polymer composition. If manufacturers' stabilisation time data are not available, a period of 6 months shall be allowed.

7.2 Sampling

7.2.1 General

Measurements of OIT or OITP provide information on the status of the equipment only at the specific location which has been sampled. The selection of the sample locations for condition monitoring shall be made based on the environmental conditions in representative areas during plant operation. It is important that these locations represent as wide a range of ageing conditions as possible with special consideration given to locations where ageing conditions could be severe, e.g. hotspots. The location of the sampling and available information about the environmental time history at the sample location selected shall be documented.

7.2.2 Sample requirements

To enable up to 5 measurements to be made on one specific sample, a minimum of 50 mg of material is needed. The material to be sampled shall be cleaned of surface debris. No solvents shall be used to clean the surface. Samples typically may take the form of slivers or scrapings of material taken from the surface of a cable jacket or a thin slice through insulation at a termination. The location of the sampling position shall be noted, including its radial distribution (i.e. whether it is a surface sample or a through thickness slice).

When sampling dual layer materials, e.g. cables, it shall be clear in the report how the samples have been obtained. The measured value will be dependent on the proportion of each layer included in the sample tested. For this reason, it is recommended that the layers are not mixed when technically possible.

Sampling and measurement procedures shall comply with local instructions, taking into account the safety of personnel and equipment

Care shall be taken to avoid unsuitable conditions in storage during the time period between sampling and measurements. It is recommended that samples be stored in the dark at temperatures not exceeding 25 °C and at humidity conditions within 45 % to 75 %.

7.2.3 Precautions

When taking samples for OIT/OITP in the field, the equipment shall be visually inspected before and after the sampling in order to document that the equipment is not damaged.

If samples are to be taken from operational equipment in plant, the impact of such sampling on future operational use and qualification of such equipment shall be evaluated prior to sampling.

Where removal of material from operational equipment is considered detrimental to qualification or future use, the equipment should be removed from service or repaired according to the utility's local procedures to ensure that qualification is maintained.

7.3 Sample preparation

Samples for each OIT or OITP measurement shall be 5 mg ± 1 mg in mass. Some older, less sensitive equipment may require the use of larger samples. In this case, 10 mg ± 1 mg of material should be used for the measurements. Regardless of which value is used, the mass of the sample in each measurement should be consistent. Each sample shall be chopped or ground into pieces with max dimensions of 1 mm. It is recommended that the chopped sample should be screened with a mesh to provide a particle size not greater than 0,85 mm as consistent sample preparation is important to enable reproducible oxidation of the sample during measurement. The chopped or granular sample shall be placed into a sample pan appropriate to the instrument being used and packed to achieve a good contact to the sample pan.

The sample pans shall be of aluminium, ceramic, or platinum and be open or have lids with holes or mesh to allow free access for oxygen during the measurement. A minimum of three samples shall be measured. If the results of measurements on three samples have a standard deviation > 10 % of the mean value for OIT or > 3 % of the mean value for OITP, an additional two samples should be measured if sufficient material is available. Out of these five measurements the highest and lowest will be removed and use the mean value of the remaining three samples.

If smaller sample mass needs to be used, e.g. for chlorinated materials, this should be noted in the measurement report.

7.4 Instrumentation

The instrument used for oxidation induction measurements shall be capable of determining exotherms in the sub-milliwatt range, e.g. a differential scanning calorimeter (DSC), simultaneous thermal analyzer (STA), or differential thermal analyzer (DTA). It shall be capable of maintaining an isothermal stability of ± 0,3 °C over the duration of the measurement, typically up to 90 min. The temperature ramp rate shall be programmable.

The instrument shall allow purging of the sample chamber with specific gases at a controllable rate. The distance between the gas-switching point and the instrument cell needs to be kept as short as possible, with a dead time of less than 1 min, to minimise the switching volume. Accordingly, for a flow rate of 75 ml·min^{-1}, the dead volume shall be less than 75 ml.

For analysis purposes, the difference in heat flow between a reference pan and the sample pan as a function of time (for OIT measurements) or temperature (for OITP measurements) shall be measured.

7.5 Calibration

The instrument shall be calibrated according to the manufacturer's recommendations and the relevant QA (quality assurance) procedure, using a suitable calibration standard for the temperature ranges being used (e.g. lead/indium/tin). Measurement of a reference sample shall be carried out prior to each batch of OIT or OITP measurements to verify this calibration.

7.6 OIT measurement method

7.6.1 Measurement procedure

The measurement procedure is illustrated in Figure 1. It includes the following steps.

- The sample is heated in nitrogen at a rate of temperature rise of 50 °C ·min^{-1} until 10 °C below the set temperature T_{set}. The ramp rate is then reduced to 5 °C·min^{-1} to reach the set temperature.
- The sample is then held for 2 min at the set temperature in nitrogen after which the atmosphere in the instrument is switched to oxygen.
- The oxidation exotherm is detected by a rapid increase in heat flow.
- The time from switching the atmosphere to oxygen until the sample starts oxidising is determined. This time is the oxidation induction time.

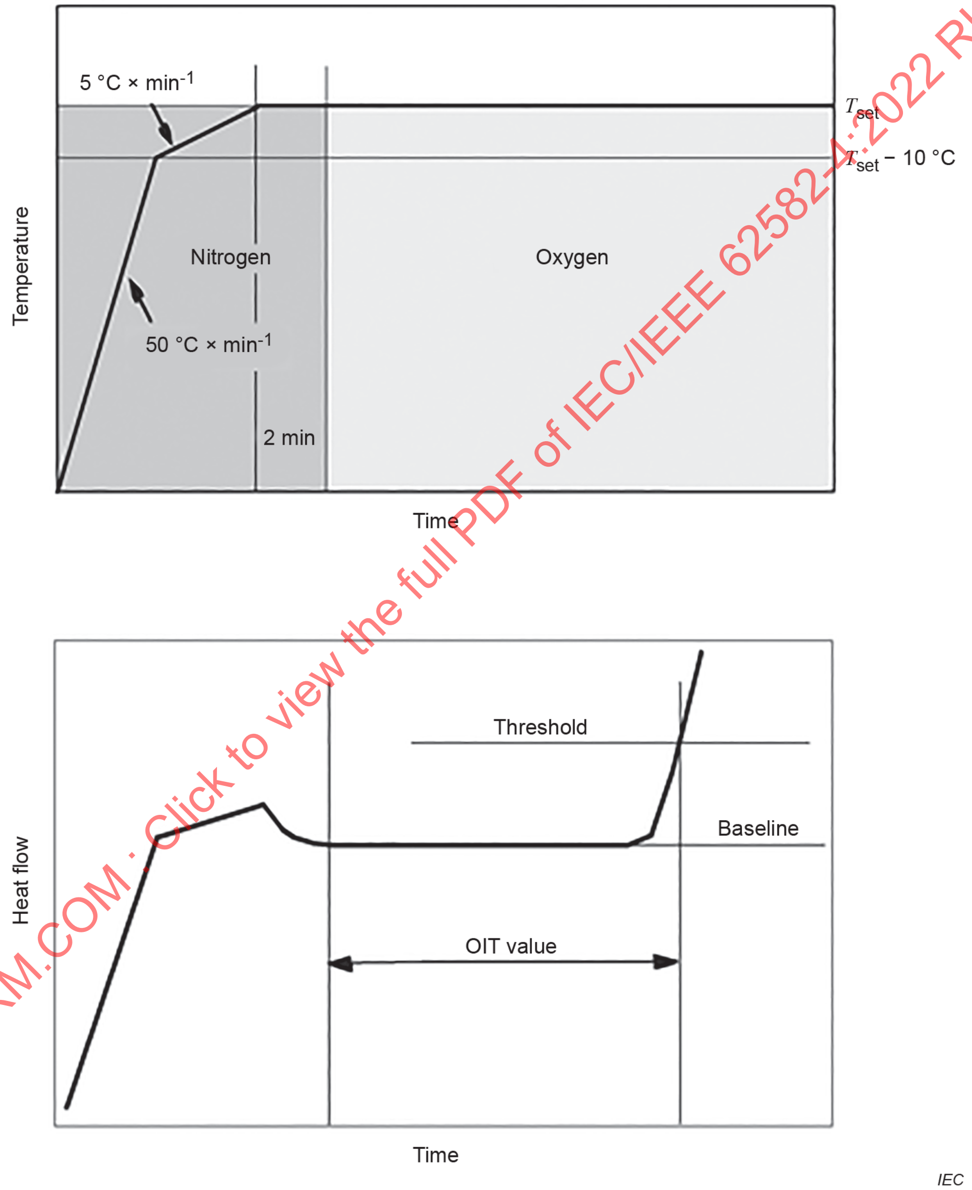


Figure 1 – OIT measurement – Schematic of temperature and gas profile and corresponding heat flow

7.6.2 Temperature profile

The reproducibility of OIT measurements is dependent on using a standardised thermal history. T_{set} for OIT measurements shall be 210 °C, provided that the oxidation induction time for unaged material is at least 30 min. The OIT value is highly dependent on T_{set} selected, see example in Annex C. If the OIT is less than 30 min for unaged material, then T_{set} shall be reduced in 10 °C increments until the OIT is > 30 min. If the OIT is > 90 min for unaged material, then T_{set} shall be increased in 10 °C increments until the OIT is < 90 min. Once the value of T_{set} has been selected for a specific material, the same value shall be used for all subsequent measurements on that material.

NOTE OIT > 90 min for unaged material is acceptable provided that the heat flow observed during the oxidation exotherm is sufficient to exceed the required threshold value (see 7.6.4.2).

7.6.3 Gas flow

The flow rate for oxygen during OIT tests shall be 75 ml·min^{-1} ± 25 ml·min^{-1}. The flow rate for nitrogen during the initial phase of OIT tests is not critical but it is recommended that 75 ml·min^{-1} ± 25 ml·min^{-1} be used.

NOTE Oxidation induction measurements can be affected by the oxygen flow rate used during the tests. For low flow rates (< 50 ml·min^{-1}), this can result in increased induction times in OIT tests. For the range of flow rates from 50 ml·min^{-1} to 100 ml·min^{-1}, oxidation induction times are not strongly dependent on the oxygen flow rate.

7.6.4 Determining the value of oxidation onset

7.6.4.1 Definition of the baseline

The threshold for oxidation induction is measured relative to a baseline, as shown in Figure 2. There will usually be a period of constant heat flow prior to the onset of oxidation; this is used as the baseline. In some materials, there is a linear change in heat flow before the onset of oxidation. This can also be used as a baseline and is referred to as a sloping baseline.

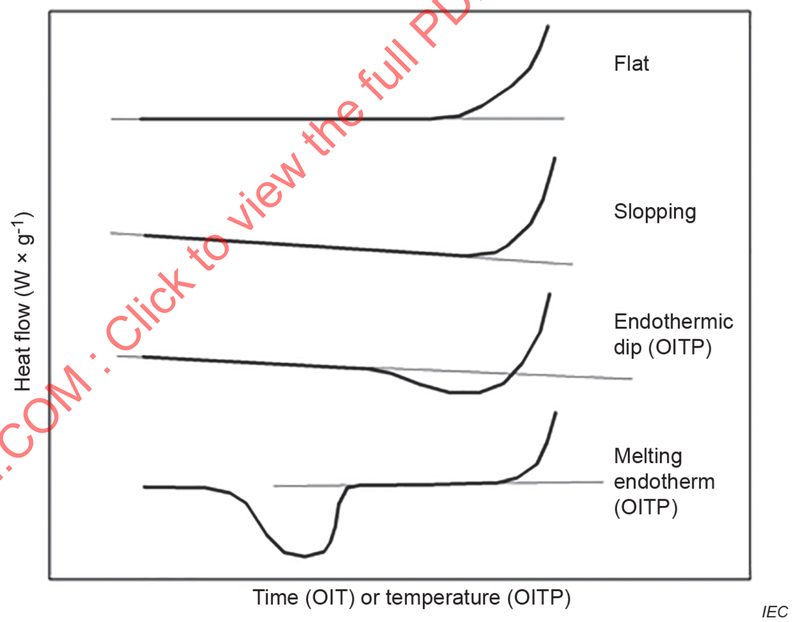


Figure 2 – Schematic showing the types of baselines (flat, sloping, endothermic dip, melting endotherm) observed for OIT and OITP measurements

7.6.4.2 Definition of the threshold and onset time

The threshold shall be defined at 0,1 W·g^{-1} relative to the baseline. The onset is defined as the time at which the test curve crosses the threshold relative to the baseline, as shown in Figure 3.

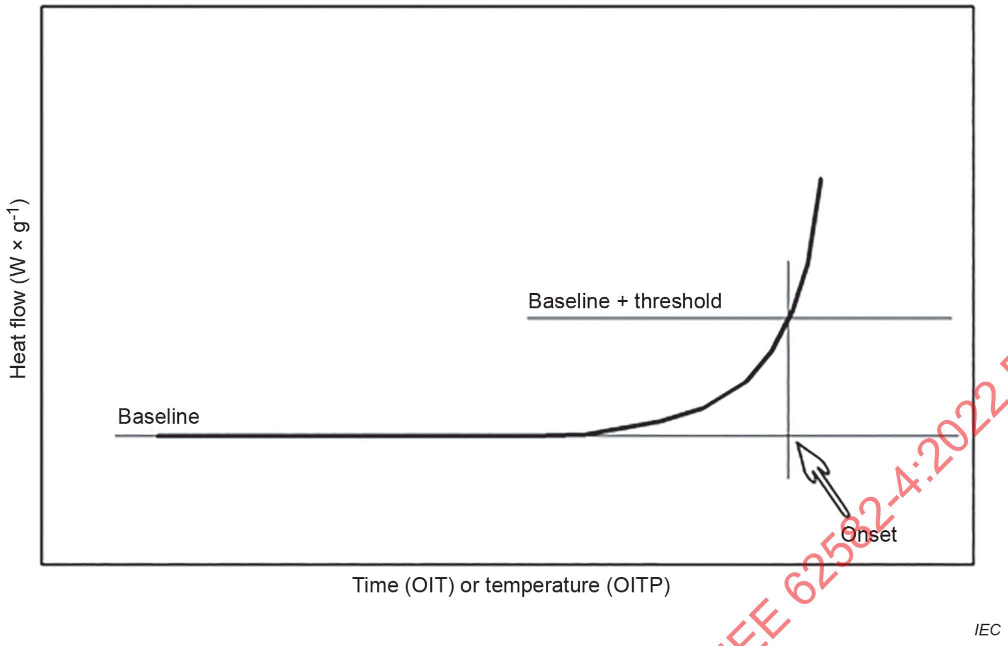


Figure 3 – Schematic showing definition of onset value for OIT and OITP measurements

Examples of the types of OIT thermogram that are observed in practice are given in Annex A.

7.6.5 Reporting

The measurement report shall include the following items.

a) Identification of the equipment sampled. This shall include:
   - details of the material being sampled, e.g. the generic polymer type, specific formulation numbers;
   - where the sample was taken from, e.g. surface scraping, through thickness slice;
   - for samples taken in plant, location within the plant.

b) Prehistory of the equipment sampled. This shall include:
   - time in service, or ageing time for laboratory aged samples;
   - the environmental conditions to which it has been exposed, e.g. temperature, radiation.

c) Sampling method, including sample preparation, have dual layer been separated (7.2).

d) Place and date of the measurements.

e) Instrument used and software version used for analysis (7.4).

f) Calibration procedure (7.5).

g) Type of sample pan used (7.3).

h) Oxygen flow rate during test (7.6.3).

i) Temperature profile, including ramp rates and hold times (7.6.2).

   If the instrument is capable of generating the information, the actual temperature profile should be included.

j) Baseline type and the rationale for using that specific baseline (7.6.4.1).

k) Onset type and the rationale for the selection of the onset in multiple onsets.

l) Threshold value used and the rationale if a non-standard value is used (7.6.4.2).

m) Number of samples measured (7.3).

n) Mean value of OIT, and standard deviation, in minutes.

o) Examples of the heat flow vs. time plot, particularly if the material does not show a flat baseline with single well-defined onset.

7.7 OITP measurement method

7.7.1 Measurement procedure

The measurement procedure is illustrated in Figure 4. It includes the following steps:

- The sample is heated in the instrument in oxygen at 10 °C·min^{-1}.
- The oxidation exotherm is detected by a rapid increase in heat flow.
- The temperature at which the sample starts oxidising is determined. This is the oxidation induction temperature.

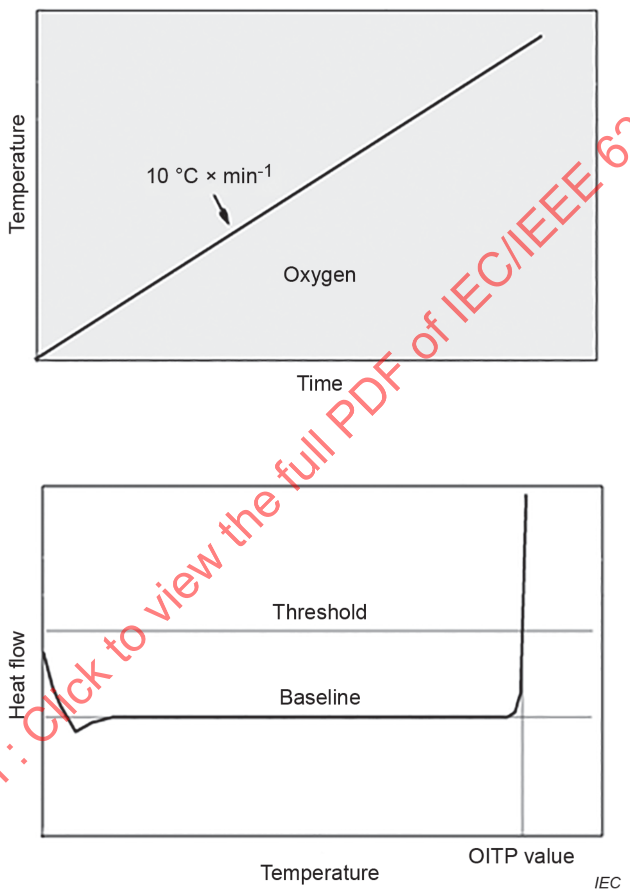


Figure 4 – Schematic of the temperature for OITP measurements and the corresponding heat flow

7.7.2 Temperature profile

The temperature ramp rate shall be 10 °C·min^{-1} in oxygen from the start temperature until the oxidation onset is observed. When carrying out consecutive measurements, the starting temperature shall be < 50 °C.

7.7.3 Gas flow

The flow rate for oxygen during OITP measurements shall be 75 ml·min^{-1} ± 25 ml·min^{-1}.

NOTE Oxidation induction measurements can be affected by the oxygen flow rate used during the tests. For low flow rates (< 50 $ml{\cdot}min^{-1}$), this can result in increased induction temperatures in OITP tests. For the range of flow rates from 50 $ml{\cdot}min^{-1}$ to 100 $ml{\cdot}min^{-1}$, oxidation induction temperatures are not strongly dependent on the oxygen flow rate.

7.7.4 Determining the value of oxidation onset

7.7.4.1 Definition of the baseline

The threshold for oxidation induction is measured relative to a baseline, as shown in Figure 2. There will usually be a period of constant heat flow prior to the onset of oxidation; this is used as the baseline. In some materials, there is a linear change in heat flow before the onset of oxidation. This can also be used as a baseline and is referred to as a sloping baseline.

7.7.4.2 Definition of the threshold and onset temperature

The threshold shall be defined at 0,5 $W{\cdot}g^{-1}$ relative to the baseline. The onset is defined by the temperature at which test curve crosses the threshold relative to the baseline, as shown in Figure 3.

NOTE Heat flows during OITP measurements are considerably higher than in OIT measurements. The selection of a higher threshold value than that used for OIT measurements enables a more consistent value to be obtained for the onset.

Examples of the types of OITP thermogram that are observed in practice are given in Annex A.

7.7.5 Reporting

The measurement report shall include the following items.

a) Identification of the equipment sampled. This shall include:
   - details of the material being sampled e.g. the generic polymer type, specific formulation numbers;
   - where the sample was taken from, e.g. surface scraping, through thickness slice;
   - for samples taken in plant, location within the plant.
b) Prehistory of the equipment sampled. This shall include:
   - time in service, or ageing time for laboratory aged samples;
   - the environmental conditions to which it has been exposed, e.g. temperature, radiation.
c) Sampling method, including sample preparation, have dual layers been separated (7.2).
d) Instrument used and software version used for analysis (7.4).
e) Place and date of the measurement.
f) Calibration procedure (7.5).
g) Type of sample pan used (7.3).
h) Oxygen flow rate (7.7.3).
i) Temperature ramp rate.

   If the instrument is capable of generating the information, the actual temperature profile should be included.
j) Baseline type and the rationale for using that specific baseline (7.7.4.1).
k) Threshold value used and the rationale if a non-standard value is used (7.7.4.2).
l) Number of samples measured.
m) Mean value of OITP, and standard deviation, in °C.
n) Examples of the heat flow versus temperature plot, particularly if the material does not show a flat baseline with a well-defined onset.

An example of a measurement report is given in Annex B.

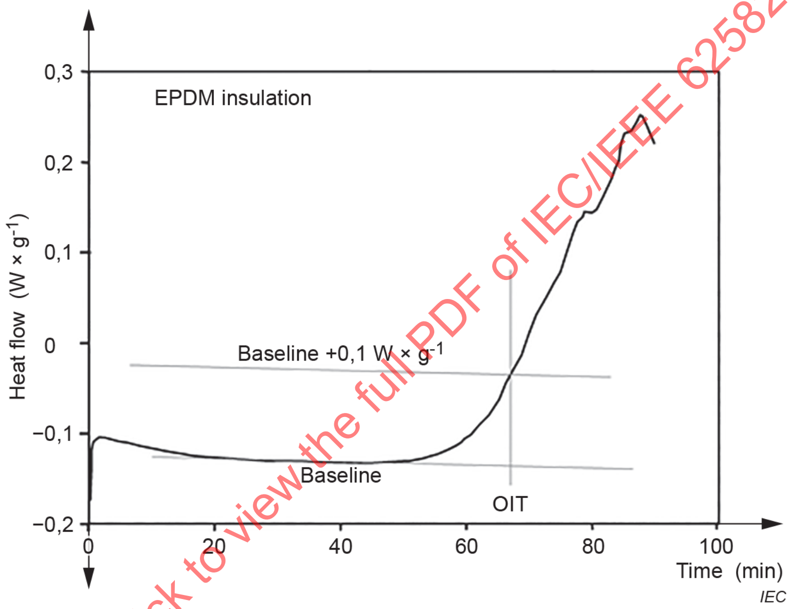
Annex A
(informative)

Interpretation of thermogram

A.1 Interpretation of OIT thermograms

The ideal OIT plot of heat flow versus time (as shown in Figure 3) is rarely seen in practice. More usually, the baseline is sloping or is difficult to define. Some of the types of plot that are observed are shown below. The approach that could be used for each type is briefly discussed. All of the examples are taken from actual OIT plots.

Where there is a clear baseline and a well-defined onset, it is straight forward to define the baseline and the onset value at the threshold of 0,1 W·g^{-1}, as shown in Figure A.1.

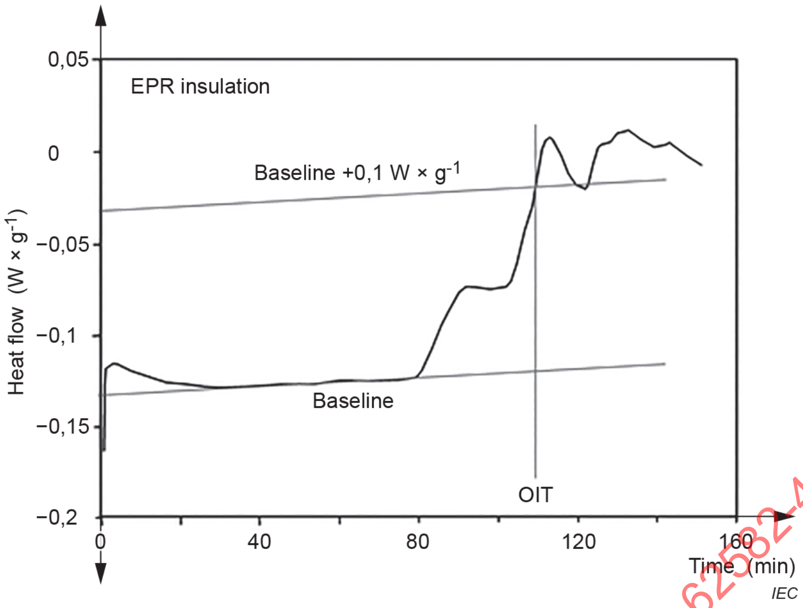


Set temperature T_{set} 210 °C

Figure A.1 – Example of an OIT plot with clear baseline and onset

Multiple onsets are often seen in EPR and EVA based materials. In this case, the standard threshold value of 0,1 W·g^{-1} may ignore one of the onsets, as shown in Figure A.2. However, the first onset may be important in defining degradation so it may be appropriate for an additional value of the threshold to be used. Alternatively, several onset values may be determined at different threshold levels. Where this is the case, the rationale for selecting the threshold and onset should be given in the measurement report.

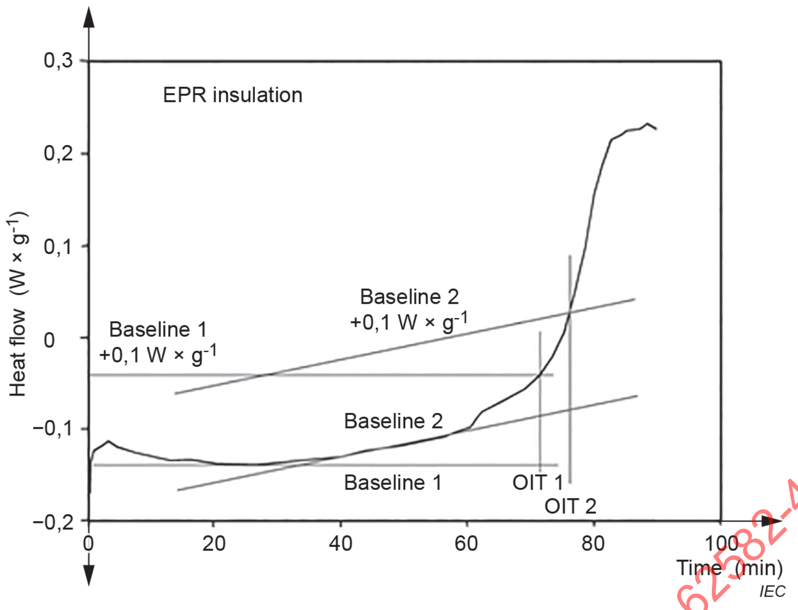
In the two examples shown in Figure A.1 and Figure A.2, the baseline was relatively simple to define, with a good section of either constant heat flow or linearly increasing heat flow. However, in many cases it is not obvious how to define the baseline. The example in Figure A.3 shows one of this type of plot. One could use the short section of linearly increasing baseline (baseline 2) or use the minimum value of the heat flow (baseline 1). Note that the choice of baseline makes a large difference to the value of the onset time, particularly where the onset of oxidation is gradual, as in this example. It is therefore very important to use a consistent definition of the baseline between measurements. This variation in onset value depending on the baseline used is one of the reasons why measurements of OIT from different laboratories can be variable.



Set temperature T_{set} 210 °C

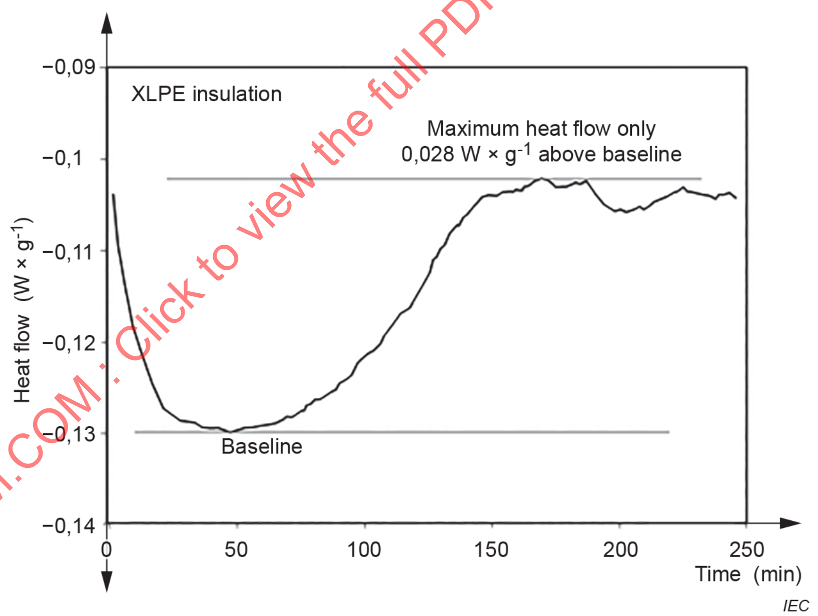
Figure A.2 – Example of OIT plot with multiple onsets

Another area that can cause problems of interpretation is where the heat flows during oxidation are too low for the standard threshold value of 0,1 W·g^{-1} to be appropriate. In the example shown in Figure A.4, the maximum heat flow observed is only 0,028 W·g^{-1} above the minimum and there is no clearly defined baseline (note that the long timescale of the test has compressed the time axis relative to the other examples). For this material, one could use the minimum heat flow as the baseline and use a lower threshold value to define the onset. However, the test plot indicates that, for this particular material, the OIT measurement procedure was not appropriate. Note that the long time scale and low heat flow indicate that the temperature selected for the test was too low.



Set temperature T_{set} 210 °C

Figure A.3 – Example of OIT plot where the baseline is difficult to define



Set temperature T_{set} 190 °C

Figure A.4 – Example of OIT plot where heat flow is too low to use standard 0,1 W·g^{-1} threshold

A.2 Interpretation of OITP thermograms

Interpretation of OITP plots is usually more straightforward than for OIT plots. This is because baselines are usually consistent and single onsets with a high heat flow are normally observed. This makes definition of the onset value less dependent on the baseline used. Figure A.5 shows the type of plot seen in an OITP measurement on a non-crystalline polymer sample.

In materials which are semi-crystalline e.g. XLPE, a melting endotherm will be observed. This is usually at a much lower temperature than the oxidation onset, so that a suitable baseline can still be defined. Figure A.6 shows an example of the OITP plot for a semi-crystalline material.

EPR and EVA based materials often show a small endothermic dip immediately prior to the onset of oxidation. In this case, the baseline should be defined prior to the endothermic dip, as shown in Figure A.7. If the lowest point of the endotherm is used instead (baseline 2 in Figure A.7), this should be stated clearly in the measurement report. It can be seen that the value of the onset temperature is not strongly dependent on the baseline selected.

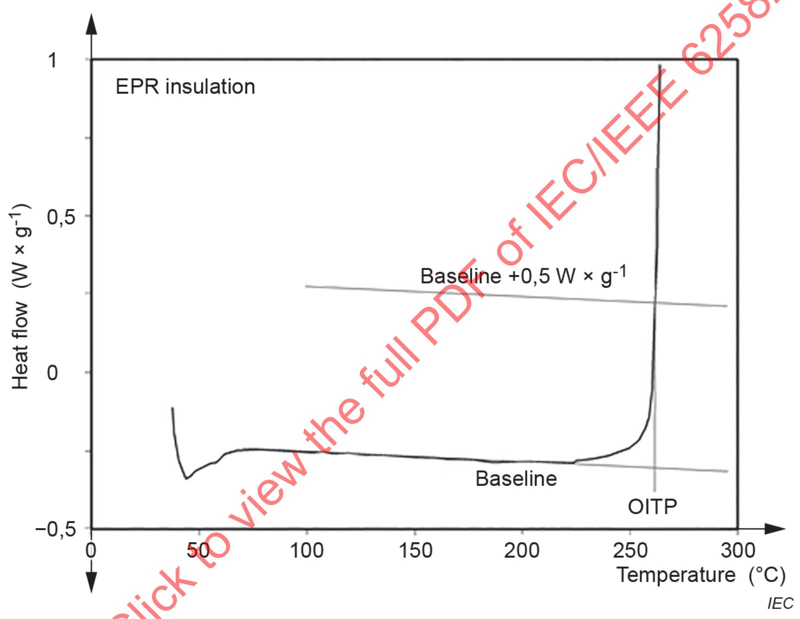


Figure A.5 – Example of an OITP plot with a well-defined baseline and onset

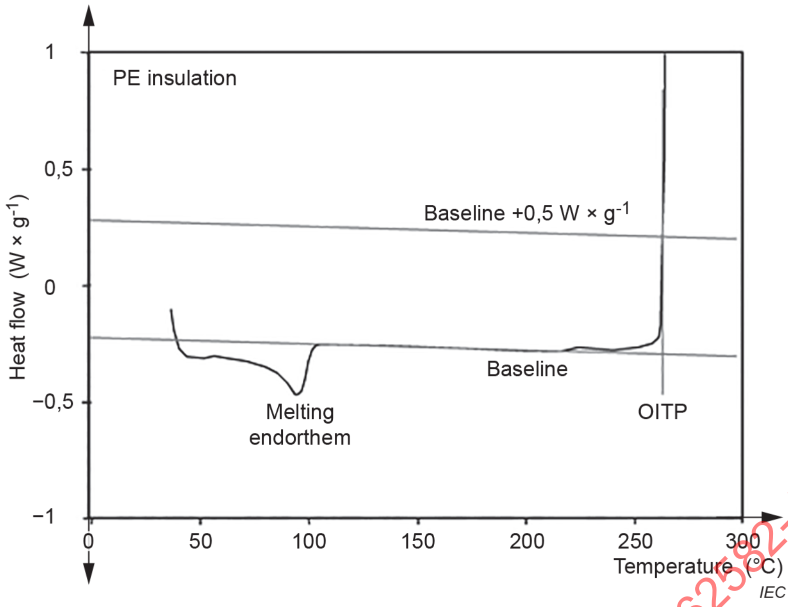


Figure A.6 – Example of an OITP plot for a semi-crystalline material showing a melting endotherm prior to the oxidation onset

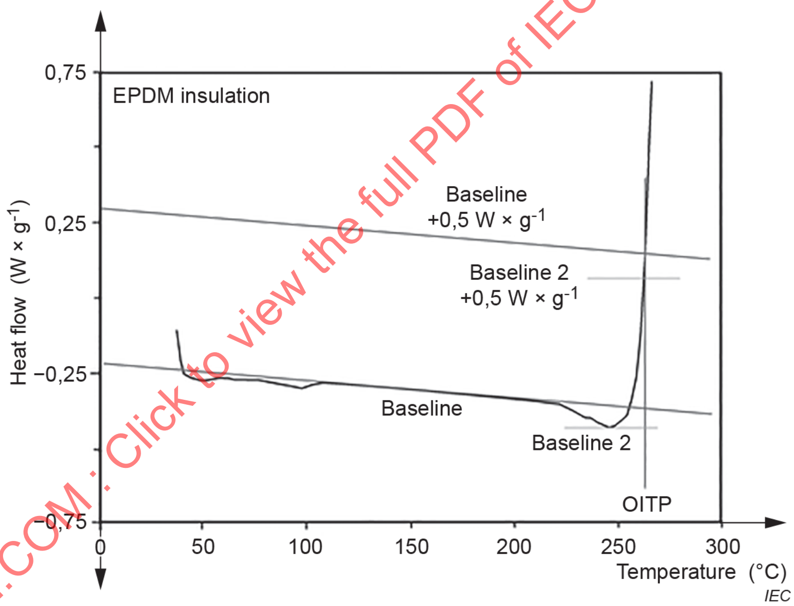


Figure A.7 – Example of an OITP plot showing an endothermic dip immediately prior to the oxidation onset

Annex B
(informative)

Example of a measurement report from OITP and OIT

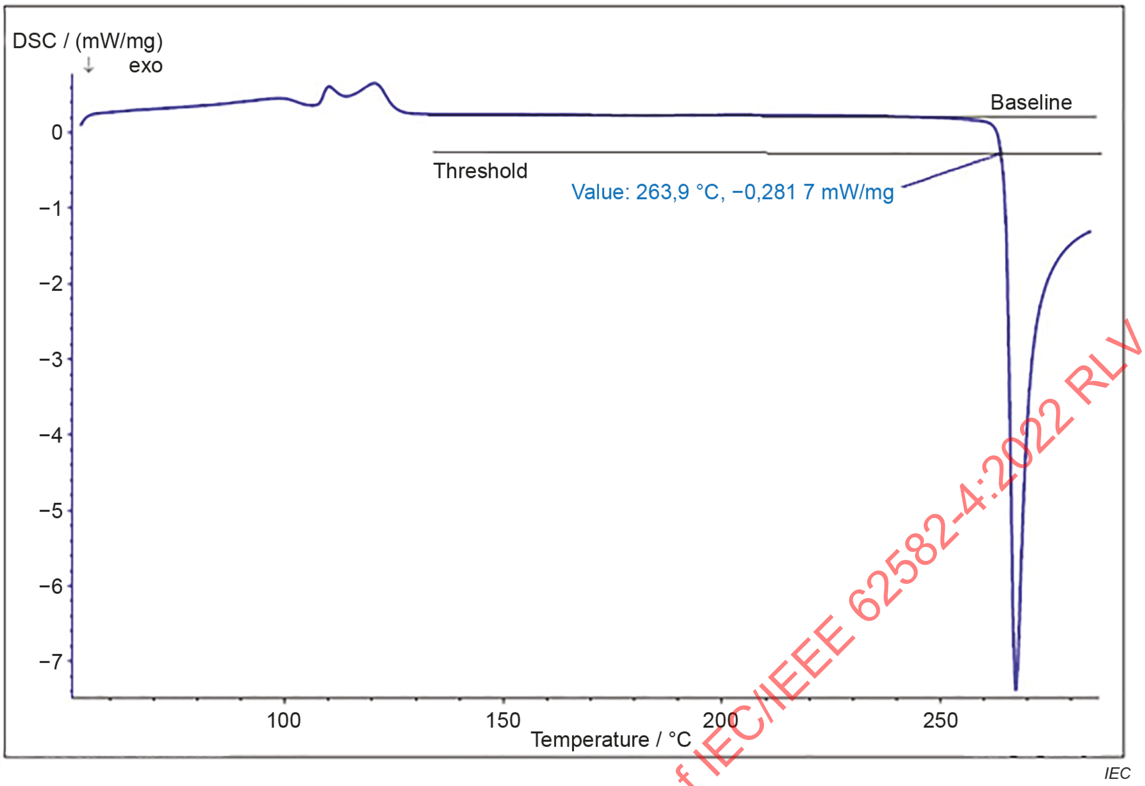
B.1 OITP measurements

These examples in Table B.1 and Figure B.1 are from the round-robin test programme carried out as part of an IAEA coordinated research programme on cable ageing (IAEA-TECDOC-1825:2017).

Table B.1 – Example of a measurement report from OITP

Specimen ID	Rockbestos Firewall III
Material	XLPE flame retardent insulation
Sample type	Through thickness slice
Prehistory	As-received, unaged
Place and date of measurement	3-Dec-2012 VUJE, Slovakia
Sampling method and mass	Chopped with sharp blade to < 1 mm, screened with no. 20 mesh to 0,85 mm sample mass 10 mg
Instrument	Netzsch DSC 200F3
Calibration method	KNO3, In, Sn, Bi, Zn, CsCl standard sample.
Sample pan	Al open pan
Oxygen flow rate	100 ml·min^{-1}
Temperature ramp rate	10 °C ·min^{-1}
Baseline type	flat
Threshold value	0,5 W·g^{-1}
No. of samples measured	3
OITP value	Mean value 262,4 °C; Standard deviation 1,4 °C

Example of test plot (see Figure B.1):



NOTE Exotherm is down in this plot.

Figure B.1 – Example of OITP test plot

Signature:

Date:

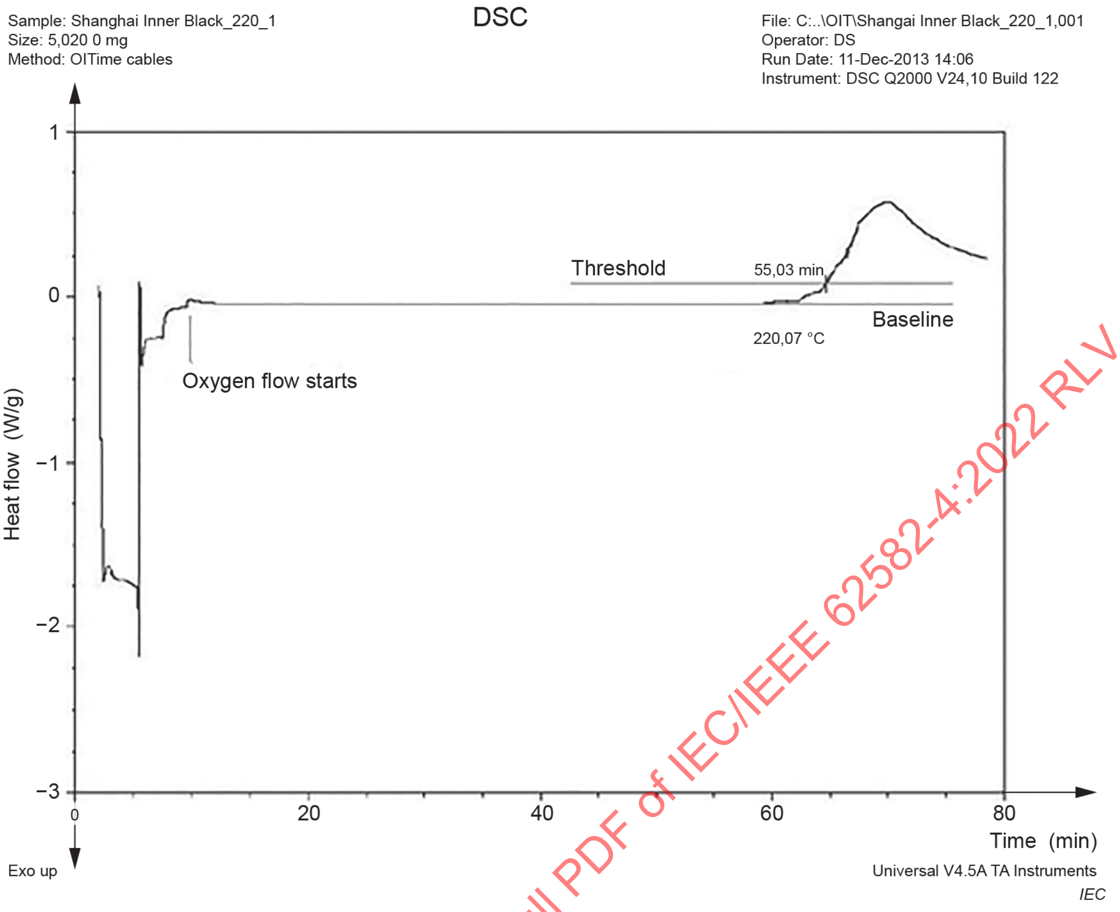
B.2 OIT measurements

These examples in Table B.2 and in Figure B.2 are from the round-robin test programme carried out as part of an IAEA coordinated research programme on cable ageing (IAEA-TECDOC-1825:2017).

Table B.2 – Example of a measurement report from OIT

Specimen ID	**WDZA-HK1-KEGJ**
Material	XLPO halogen-free flame retardent insulation (black), Shanghai Special Cable, China
Sample type	Through thickness slice
Prehistory	As-received, unaged
Place and date of measurement	11-Dec-2013 NIST, USA
Sampling method and mass	Chopped with sharp blade to <1 mm sample mass 5 mg
Instrument	TA Instruments Universal V4.5A
Calibration method	Indium/lead/tin standard sample
Sample pan	Al open pan
Oxygen flow rate	100 ml·min^{-1}
Set temperature	220 °C
Temperature ramp rate	50 °C ·min–1 to 210 °C, then 10 °C ·min^{-1} to set temperature
Baseline type	flat
Threshold value	0,1 W·g^{-1}
No. of samples measured	3
OIT value	Mean value 53,0 min; Standard deviation 2,3 min

Example of test plot (see Figure B.2):



NOTE Exotherm is up in this plot.

Figure B.2 – Example of OIT test plot

Signature:

Date:

Annex C
(informative)

Influence of set temperature on the OIT value

The OIT value measured is highly dependent on the set temperature selected. The loss of antioxidants can normally be described as a thermally activated process. Figure C.1 shows an example of the ratio between the OIT value at different temperatures T and at 200 °C.

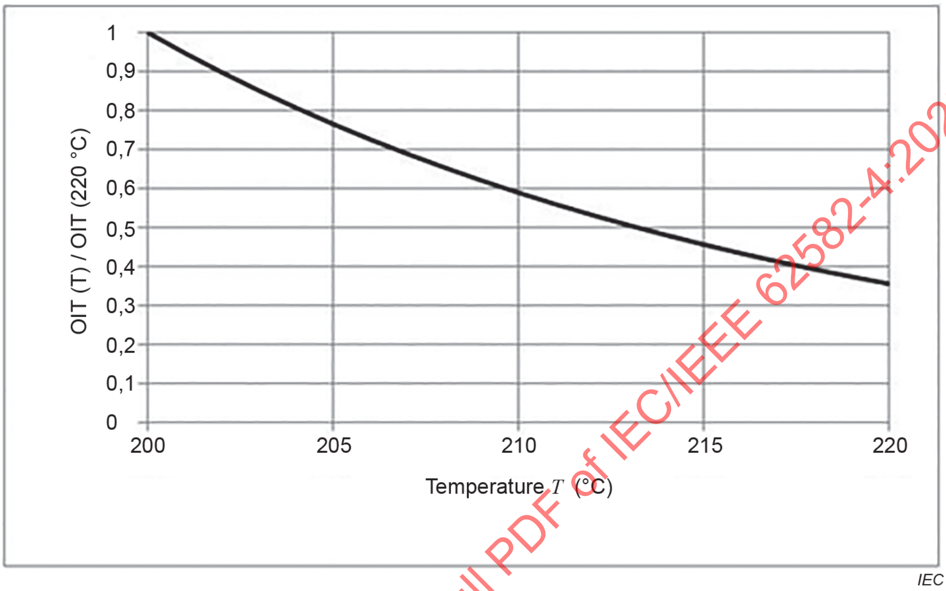


Figure C.1 – Example of the influence of set temperature on the OIT value

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SOMMAIRE

COMMISSION ÉLECTROTECHNIQUE INTERNATIONALE

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CENTRALES NUCLÉAIRES DE PUISSANCE – INSTRUMENTATION ET CONTRÔLE-COMMANDE IMPORTANTS POUR LA SÛRETÉ – MÉTHODES DE SURVEILLANCE DE L'ÉTAT DES MATÉRIELS ÉLECTRIQUES –

Partie 4: Techniques d'induction à l'oxydation

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L'IEC/IEEE 62582-4 a été établie par le sous-comité 45A: Systèmes d'instrumentation, de contrôle-commande et d'alimentation électrique des installations nucléaires, du comité d'études 45 de l'IEC: Instrumentation nucléaire, en coopération avec le ''Nuclear Power Engineering Committee'' de l'IEEE, selon l'accord double logo IEC/IEEE. Il s'agit d'une Norme internationale.

Le présent document est publié en tant que norme IEC/IEEE double logo.

Cette deuxième édition annule et remplace la première édition, parue en 2011. Cette édition constitue une révision technique.

Cette édition inclut les modifications techniques majeures suivantes par rapport à l'édition précédente:

a) Prise en considération de la publication de l'IEC/IEEE 60780-323;
b) Ajout d'un exemple dans l'Annexe B et mise à jour;
c) Ajout de l'Annexe C.

Le texte de cette Norme internationale est issu des documents suivants de l'IEC:

Projet	Rapport de vote
45A/1435/FDIS	45A/1445/RVD

Le rapport de vote indiqué dans le tableau ci-dessus donne toute information sur le vote ayant abouti à son approbation.

La version française de cette norme n'a pas été soumise au vote.

La langue employée pour l'élaboration de cette Norme internationale est l'anglais.

Ce document a été rédigé conformément aux règles données dans les Directives ISO/IEC, Partie 2, disponibles à l'adresse www.iec.ch/members_experts/refdocs. Les principaux types de documents développés par l'IEC sont décrits plus en détail sous www.iec.ch/standardsdev/publications.

Une liste de toutes les parties de la série IEC/IEEE 62582, publiées sous le titre général *Centrales nucléaires de puissance – Instrumentation et contrôle-commande importants pour la sûreté – Méthodes de surveillance de l'état des matériels électriques*, se trouve sur le site web de l'IEC.

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INTRODUCTION

a) Contexte technique, questions importantes et structure de la présente norme

La présente partie de l'IEC/IEEE 62582 s'intéresse plus particulièrement aux méthodes d'induction à l'oxydation utilisées dans le cadre de la surveillance de l'état pour la gestion du vieillissement des matériels électriques installés dans les centrales nucléaires de puissance. Ces méthodes sont principalement adaptées pour les échantillons prélevés sur des matériaux de la famille des polyoléfines, mais elles peuvent aussi être utilisées pour certains matériaux de la famille des polymères éthylène-propylène et pour certains matériaux acétate de vinyle éthylène.

La présente partie (4) de l'IEC/IEEE 62582 est la quatrième partie de la série IEC/IEEE 62582.

La série IEC/IEEE 62582 est publiée en double logo, ce qui la rend applicable pour la gestion du vieillissement des matériels électriques qualifiés tant dans le cadre des normes IEEE que dans celui des normes IEC.

La norme IEC/IEEE 60780-323 couvre le concept et le rôle complémentaire que peut jouer la qualification reposant sur l'état du matériel dans le cadre de la qualification des matériels au niveau de la durée de vie qualifiée. Dans le cadre de la qualification du matériel, l'état du matériel pour lequel des performances acceptables ont été prouvées correspond à l'état qualifié. L'état qualifié est l'état du matériel qui prévaut au début d'un événement de dimensionnement, pour lequel il a été démontré que le matériel satisfaisait aux exigences de conception pour les conditions de service spécifiées.

Des recherches importantes ont été réalisées sur les techniques de surveillance de l'état des matériels et l'utilisation de ces techniques dans le cadre de la qualification desdits matériels, comme cela est indiqué dans les documents NUREG/CR-6704, Vol. 2 (BNL-NUREG-52610), JNES-SS-0903, 2009 et AIEA TECDOC-1825:2017.

L'objectif du présent document est d'être utilisé par les laboratoires d'essai, les exploitants de centrales nucléaires de puissance, les évaluateurs de systèmes et les régulateurs.

b) Position de la présente norme dans la structure de la série de normes du SC 45A de l'IEC

L'IEC/IEEE 62582-4 est le document du SC 45A de l'IEC de troisième niveau qui traite du problème particulier de l'application et des performances des mesurages réalisés par induction à l'oxydation dans le cadre de la gestion du vieillissement des matériels électriques et des matériels de commande utilisés dans les centrales nucléaires de puissance.

La norme IEC/IEEE 62582-4 doit être lue avec l'IEC/IEEE 62582-1, qui fournit les éléments de contexte et des lignes directrices pour l'application des méthodes de surveillance de l'état des matériels électriques importants pour la sûreté des centrales nucléaires de puissance.

Pour de plus amples informations sur la collection de normes du SC 45A de l'IEC, voir l'article d) de la présente introduction.

c) Recommandations et limites relatives à l'application de la présente norme

Il est important de noter que le présent document n'établit pas d'exigence fonctionnelle supplémentaire pour les systèmes de sûreté.

d) Description de la structure de la série de normes du SC 45A de l'IEC et relations avec d'autres documents de l'IEC, et d'autres organisations (AIEA, ISO)

La série de normes du SC 45A de l'IEC comprend une hiérarchie de quatre niveaux. Les documents de niveau supérieur de la série de normes du SC 45A de l'IEC sont les normes IEC 61513 et IEC 63046.

L'IEC 61513 fournit des exigences générales relatives aux systèmes et matériels d'instrumentation et de contrôle-commande (I&C) utilisés pour accomplir les fonctions importantes pour la sûreté des centrales nucléaires de puissance (NPPs – *Nuclear power plants*). L'IEC 63046 fournit des exigences générales relatives aux systèmes d'alimentation électrique des NPP; elle couvre les systèmes d'alimentation électrique jusqu'à et y compris les alimentations des systèmes d'instrumentation et de contrôle-commande.

L'IEC 61513 et l'IEC 63046 doivent être étudiées ensemble et au même niveau. L'IEC 61513 et l'IEC 63046 structurent la série de normes du SC 45A de l'IEC et forment un cadre complet qui établit les exigences générales relatives aux systèmes et matériels d'instrumentation et de contrôle-commande, ainsi qu'aux systèmes d'alimentation électriques des centrales nucléaires de puissance.

L'IEC 61513 et l'IEC 63046 font directement référence aux autres normes du SC 45A de l'IEC pour les exigences générales relatives à des sujets particuliers, tels que la catégorisation des fonctions et le classement des systèmes, la qualification, la séparation des systèmes, la protection contre la défaillance de cause commune, la conception des salles de commande, la compatibilité électromagnétique, l'ergonomie, la cybersécurité, les aspects logiciels et matériels relatifs aux systèmes numériques programmables, la coordination des exigences de sûreté et de sécurité et la gestion du vieillissement. Il convient de considérer les normes référencées directement à ce deuxième niveau, ainsi que l'IEC 61513 et l'IEC 63046, comme un ensemble cohérent de documents.

Au troisième niveau, les normes du SC 45A de l'IEC, qui ne sont pas référencées directement par l'IEC 61513 ou l'IEC 63046, sont liées à des exigences spécifiques relatives à des matériels particuliers, ainsi qu'à des méthodes techniques ou à des activités spécifiques. Généralement, ces documents, qui font référence aux documents de deuxième niveau pour les exigences générales, peuvent être utilisés seuls.

Un quatrième niveau qui est une extension de la série de normes du SC 45A de l'IEC correspond aux rapports techniques qui ne sont pas des documents normatifs.

La série de normes du SC 45A de l'IEC met en œuvre et détaille de manière cohérente les principes de sûreté et de sécurité, ainsi que les aspects fondamentaux fournis dans les normes de sûreté pertinentes de l'AIEA et dans les documents pertinents de la série sur la sécurité nucléaire (NSS – *nuclear security series*) de l'AIEA. Cette disposition inclut plus particulièrement les exigences SSR-2/1 de l'AIEA qui établissent les exigences de sûreté relatives à la conception des centrales nucléaires de puissance, le guide de sûreté SSG-30 de l'AIEA qui traite du classement de sûreté des structures, systèmes et composants des centrales nucléaires de puissance, le guide de sûreté SSG-39 de l'AIEA qui traite de la conception de l'instrumentation et du contrôle-commande des centrales nucléaires de puissance, le guide de sûreté SSG-34 de l'AIEA qui traite de la conception des systèmes d'alimentation électrique des centrales nucléaires de puissance, le guide de sûreté SSG-51 de l'AIEA qui traite de l'ergonomie dans la conception des centrales nucléaires de puissance et le guide de mise en œuvre NSS17 relatif à la sécurité informatique des installations nucléaires. La terminologie et les définitions utilisées pour la sûreté et la sécurité dans les normes produites par le SC 45A sont conformes à celles utilisées par l'AIEA.

L'IEC 61513 et l'IEC 63046 ont adopté une présentation similaire à celle de la publication fondamentale de sécurité IEC 61508, avec un cadre de cycle de vie global et un cadre de cycle de vie du système. En ce qui concerne la sûreté nucléaire, l'IEC 61513 et l'IEC 63046 interprètent les exigences générales des normes IEC 61508-1, IEC 61508-2 et IEC 61508-4, pour le secteur des applications nucléaires. Dans ce cadre, l'IEC 60880, l'IEC 62138 et l'IEC 62566 correspondent à l'IEC 61508-3 pour le secteur des applications nucléaires.

L'IEC 61513 et l'IEC 63046 font référence à l'ISO 9001, ainsi qu'aux documents AIEA GSR partie 2, AIEA GS-G-3.1 et AIEA GS-G-3.5 pour les sujets liés à l'assurance qualité (AQ).

Au niveau 2, concernant la sécurité nucléaire, l'IEC 62645 constitue le document chapeau pour les normes de sécurité produites par le SC 45A de l'IEC. Cette norme s'appuie sur les principes de haut niveau et les principaux concepts valides des normes de sécurité génériques, notamment l'ISO/IEC 27001 et l'ISO/IEC 27002, qu'elle adapte et complète pour répondre au contexte nucléaire et les coordonne avec la série IEC 62443. Au niveau 2, l'IEC 60964 constitue le document chapeau pour les normes du SC 45A de l'IEC portant sur les salles de commande, l'IEC 63351 constitue le document chapeau pour les normes portant sur l'ergonomie et l'IEC 62342 constitue le document chapeau pour les normes portant sur la gestion du vieillissement.

NOTE 1 Par hypothèse, des normes nationales ou internationales sont appliquées pour la conception des systèmes d'instrumentation et de contrôle-commande dans les centrales nucléaires de puissance qui mettent en œuvre des fonctions de sûreté conventionnelles (par exemple, pour assurer la sécurité des travailleurs et la protection des biens, répondre aux dangers chimiques et aux dangers liés à l'énergie procédé).

NOTE 2 L'IEC TR 64000 fournit une description plus complète de la structure globale de la série de normes produites par le SC 45A de l'IEC et de sa relation avec d'autres organismes de normalisation et d'autres normes.

CENTRALES NUCLÉAIRES DE PUISSANCE – INSTRUMENTATION ET CONTRÔLE-COMMANDE IMPORTANTS POUR LA SÛRETÉ – MÉTHODES DE SURVEILLANCE DE L’ÉTAT DES MATÉRIELS ÉLECTRIQUES –

Partie 4: Techniques d’induction à l’oxydation

1 Domaine d'application

La présente partie de l'IEC/IEEE 62582 établit des méthodes de surveillance de l'état des matériaux organiques et polymères présents dans les systèmes d’instrumentation et de contrôle-commande, par l’utilisation des techniques d’induction à l’oxydation selon un processus détaillé nécessaire pour obtenir des mesurages reproductibles et exacts. La présente partie comprend les exigences relatives à la préparation d’échantillons, au système et aux conditions de mesure, ainsi qu’au compte-rendu des résultats de mesure.

Les différentes parties de l’IEC/IEEE 62582 sont des normes de mesure, principalement destinées à être utilisées pour la gestion du vieillissement dans le cadre de la qualification initiale et après installation. L’IEC/IEEE 62582-1 comprend des exigences pour l'application des autres parties de la série IEC/IEEE 62582 et certains éléments communs à toutes les méthodes. L’IEC/IEEE 60780-323 fournit des informations concernant le rôle de la surveillance de l’état dans la qualification des matériels importants pour la sûreté.

2 Références normatives

Le présent document ne contient aucune référence normative.

3 Termes et définitions

Pour les besoins du présent document, les termes et définitions suivants s'appliquent.

L'ISO, l'IEC et l’IEEE tiennent à jour des bases de données terminologiques destinées à être utilisées en normalisation, consultables aux adresses suivantes:

- IEC Electropedia: disponible à l'adresse http://www.electropedia.org/
- ISO Online browsing platform: disponible à l'adresse http://www.iso.org/obp
- IEEE Standards Dictionary Online: disponible à l'adresse http://dictionary.ieee.org

3.1
temps d’induction à l’oxydation
OIT
mesure relative de la résistance stabilisée d’un matériau à la décomposition par oxydation, déterminée par le mesurage calorimétrique de l’intervalle de temps qui précède le début de l’oxydation exothermique du matériau à une température spécifiée, sous atmosphère d’oxygène et sous pression atmosphérique

Note 1 à l’article: L’OIT est exprimé en minutes (min).

Note 2 à l'article: L’abréviation "OIT" est dérivée du terme anglais développé correspondant "Oxidation Induction Time".

3.2
température d’induction à l’oxydation
OITP
mesurage calorimétrique de la température au début de l’oxydation exothermique du matériau, lorsque celui-ci est soumis à un échauffement spécifié sous atmosphère d’oxygène et sous pression atmosphérique

Note 1 à l’article: L’OITP est exprimée en degrés Celsius (°C).

Note 2 à l'article: L’abréviation "OITP" est dérivée du terme anglais développé correspondant "Oxidation Induction Temperature".

4 Abréviations et acronymes

CSPE	(chlorosulphonated polyethylene) polyéthylène chlorosulphoné
DSC	(differential scanning calorimeter) calorimètre différentiel à balayage
EPDM	(ethylene propylene diene monomer) caoutchouc d'éthylène-propylène-diène
EPR	(ethylene propylene rubber) caoutchouc d’éthylène-propylène
EVA	(ethylene vinyl acetate) éthylène-acétate de vinyle
OIT	(oxidation induction time) temps d’induction à l’oxydation
OITP	(oxidation induction temperature) température d’induction à l’oxydation
PE	polyéthylène
PEEK	(poly ether ketone) polyéther éther cétone
PVC	(poly vinyl chloride) chlorure de polyvinyle
STA	(simultaneous thermal analyzer) analyseur thermique simultané
XLPE	(cross-linked polyethylene) polyéthylène réticulé

5 Description générale

Les méthodes d’induction à l’oxydation reposent sur la détection de l’oxydation exothermique qui survient lorsqu’un échantillon est chauffé en présence d’oxygène. Cette réaction exothermique est sensible au niveau de dégradation de certains matériaux organiques et polymères et peut être utilisée comme un indicateur du vieillissement. Il existe deux méthodes d’induction à l’oxydation, qui reposent sur la durée exigée pour que débute l’oxydation à température constante (temps d’induction à l’oxydation – OIT) ou sur la température observée au début de l’oxydation lors d’une vitesse constante d’augmentation de la température (température d’induction à l’oxydation – OITP). Les deux méthodes sont complémentaires, en ce sens que l’OITP est souvent efficace pour les matériaux pour lesquels l’OIT est difficile à déterminer. L’OIT et l’OIPT diminuent avec l'augmentation de la dégradation. Les méthodes sont généralement liées à la quantité d’antioxydants présents dans le matériau. Au fur et à mesure de la dégradation, ces antioxydants s’épuisent. Pour les matériaux qui ne comportent pas d’antioxydants, l’OIT n’est pas adapté, mais l’OIPT peut donner des résultats utiles. En général, l'OIT et l'OITP permettent de mesurer la stabilité thermique des polymères dans des conditions de chauffage isotherme (OIT) et adiabatique (OITP) sous oxygène.

6 Applicabilité et reproductibilité

La méthode d'induction à l'oxydation est principalement adaptée aux échantillons prélevés sur les matériaux (tels que les gaines externes ou l'isolant des câbles) qui sont de la famille des polyoléfines (par exemple, le PE, le XLPE). Elle peut aussi être utilisée pour certains matériaux de la famille des polymères éthylène-propylène (par exemple, l'EPR, l'EPDM) et pour certains matériaux éthylène-acétate de vinyle EVA. Les techniques d'induction à l'oxydation peuvent ne pas être applicables aux polymères à haute température dans certains cas. La méthode d'induction à l'oxydation n'est pas applicable aux polymères à haute température, tels que le PEEK. La méthode peut être appliquée à d'autres matériaux, mais les problèmes d'interprétation des résultats peuvent réduire l'efficacité de l'essai.

La méthode n'est généralement pas adaptée pour les polymères chlorés (par exemple, le PVC, le CSPE) en raison des produits de dégradation corrosifs dégagés pendant les mesurages, qui peuvent endommager l'appareil de mesure. Pour ces matériaux, des masses d'échantillons plus faibles (de 1 mg à 2 mg) peuvent permettre d'utiliser la méthode avec précaution. En outre, certains systèmes modernes sont conçus avec des caractéristiques qui protègent le matériel contre les gaz d'échappement volatils. Pour déterminer si le matériel est protégé ou si des composants de protection supplémentaires sont nécessaires, consulter le fabricant dudit matériel.

La méthode n'est pas adaptée pour une utilisation sur le terrain dans une centrale nucléaire de puissance, mais elle utilise des échantillons prélevés sur la centrale, qui sont ensuite mesurés en laboratoire. Chaque mesurage OIT en laboratoire peut durer jusqu'à 90 min pour les échantillons non vieillis, et diminuer pour les échantillons fortement vieillis, alors que les mesurages OITP durent généralement de 30 min à 40 min.

Les mesurages OIT présentent généralement un écart type compris entre 5 % et 10 % de la valeur moyenne, tandis que les mesurages OITP présentent généralement un écart type compris entre 1 % et 3 % de la valeur moyenne. Dans les deux cas, les mesurages sont effectués dans le même laboratoire et dans des laboratoires différents. Une partie de cette variation est due à l'inhomogénéité des matériaux des échantillons, qui devient significative lors de mesurages de l'état sur des échantillons dont la masse est très faible. Les mesurages OITP sont généralement plus reproductibles que les mesurages OIT, mais ils exigent des données de référence pour pouvoir interpréter les variations.

7 Procédure de mesure

7.1 Stabilisation des matériaux polymères

Une durée appropriée doit être prévue pour que les matériaux polymères des matériels fabriqués récemment se stabilisent avant que des programmes de surveillance de l'état ou de vieillissement accéléré ne soient réalisés. La durée pendant laquelle les matériaux polymères se stabilisent dépend normalement des additifs de traitement et de la composition des polymères. En l'absence de données du fabricant sur le temps de stabilisation, un délai de 6 mois doit être prévu.

7.2 Échantillonnage

7.2.1 Généralités

Les mesurages OIT ou OITP donnent des informations sur l'état du matériel uniquement à l'emplacement spécifique soumis à échantillonnage. Le choix des emplacements d'échantillonnage à des fins de surveillance de l'état doit s'effectuer sur la base des conditions environnementales dans des zones représentatives pendant le fonctionnement de la centrale. Il est important que ces emplacements représentent une plage de conditions de vieillissement aussi étendue que possible, avec une attention particulière pour les emplacements auxquels les conditions de vieillissement peuvent être sévères, par exemple, les points chauds. L'emplacement d'échantillonnage et les informations disponibles concernant l'historique environnemental de l'emplacement choisi doivent être documentés.

7.2.2 Exigences applicables à l'échantillon

Pour que cinq mesurages puissent être réalisés sur un échantillon particulier, une quantité minimale de 50 mg de matériau est nécessaire. Le matériau à échantillonner doit être nettoyé des débris de surface. Aucun solvant ne doit être utilisé pour nettoyer la surface. Les échantillons peuvent généralement prendre la forme d'éclats ou de raclures de matériau prélevés à la surface d'une gaine externe de câble ou d'une fine tranche à travers l'isolant d'une extrémité. L'emplacement de la position d'échantillonnage doit être noté, y compris sa distribution radiale (c'est-à-dire s'il s'agit d'un échantillon de surface ou d'une tranche d'épaisseur traversante).

Lors de l'échantillonnage de matériaux à double couche, par exemple des câbles, le rapport doit indiquer clairement le mode d'obtention des échantillons. La valeur mesurée dépend de la proportion de chaque couche incluse dans l'échantillon soumis à l'essai. Pour cette raison, il est recommandé que les couches ne soient pas mélangées lorsque cela est techniquement possible.

Les procédures d'échantillonnage et de mesure doivent être conformes aux consignes locales, compte tenu de la sécurité du personnel et des matériels.

Il faut veiller à ce que les conditions de stockage ne soient pas inadéquates au cours de la durée comprise entre l'échantillonnage et les mesurages. Il est recommandé que les échantillons soient stockés dans l'obscurité à des températures qui ne dépassent pas 25 °C et dans des conditions d'humidité comprises entre 45 % et 75 %.

7.2.3 Précautions

Lors du prélèvement sur le terrain des échantillons destinés aux mesurages OIT ou OITP, le matériel doit faire l'objet d'une inspection visuelle avant et après l'échantillonnage afin de documenter qu'il n'est pas endommagé.

Si des échantillons doivent être prélevés sur un matériel opérationnel de la centrale, la conséquence de cet échantillonnage sur l'utilisation opérationnelle future et la qualification de ce matériel doit être évaluée avant l'échantillonnage.

Lorsque le retrait d'un matériau constitutif du matériel opérationnel est considéré comme préjudiciable à la qualification ou à une utilisation future, il convient de retirer ce matériel du service ou de le réparer selon les procédures locales du réseau pour assurer le maintien de la qualification.

7.3 Préparation des échantillons

Les échantillons pour chaque mesurage OIT ou OITP doivent avoir une masse de 5 mg ± 1 mg. Certains matériels plus anciens et moins sensibles peuvent exiger l'utilisation d'échantillons plus grands. Dans ce cas, il convient d'utiliser 10 mg ±1 mg de matériau pour les mesurages. Quelle que soit la valeur utilisée, il convient que la masse de l'échantillon utilisé dans chaque mesurage soit cohérente. Chaque échantillon doit être haché ou broyé en morceaux d'une dimension maximale de 1 mm. Il convient de tamiser l'échantillon haché à l'aide d'une maille pour obtenir une taille de particule qui ne dépasse pas 0,85 mm, dans la mesure où une préparation cohérente de l'échantillon est importante pour permettre une oxydation reproductible de ce dernier pendant le mesurage. L'échantillon haché ou granulaire doit être placé dans une coupelle appropriée à l'appareil de mesure utilisé et emballé de manière à obtenir un bon contact avec la coupelle.

Les coupelles d'échantillonnage doivent être en aluminium, en céramique ou en platine et être ouvertes ou équipées de couvercles avec des trous ou des mailles pour laisser librement circuler l'oxygène pendant le mesurage. Trois échantillons au moins doivent être mesurés. Lorsque les résultats des mesurages sur trois échantillons présentent un écart type > 10 % de la valeur moyenne pour la méthode OIT ou un écart type > 3 % pour la méthode OITP, il convient de mesurer deux échantillons supplémentaires si la quantité de matériaux présents est suffisante. Le mesurage le plus élevé et le mesurage le plus faible sont éliminés de ces cinq mesurages et la valeur moyenne des trois échantillons restants est appliquée.

S'il est nécessaire d'utiliser une masse d'échantillons plus petite, par exemple, pour les matériaux chlorés, il convient de noter ce point dans le rapport de mesure.

7.4 Instrumentation

L'appareil de mesure utilisé pour les mesurages d'induction à l'oxydation doit être capable de déterminer les réactions exothermiques dans la plage des sous-milliwatts, par exemple, un calorimètre différentiel à balayage (DSC), un analyseur thermique simultané (STA) ou un analyseur thermique différentiel (DTA – *differential thermal analyzer*). Il doit être également capable de maintenir une stabilité isotherme de ± 0,3 °C pendant la durée du mesurage, généralement jusqu'à 90 min. La vitesse d'augmentation de la température doit être programmable.

L'appareil de mesure doit permettre de purger la chambre à échantillons avec des gaz spécifiques à un débit contrôlable. Il est nécessaire de maintenir une distance aussi courte que possible entre le point de commutation du gaz et la cellule de l'appareil, avec un temps mort inférieur à 1 min, afin de réduire le plus possible le volume de commutation. En conséquence, pour un débit de 75 ml min^{-1}, le volume mort doit être inférieur à 75 ml.

À des fins d'analyse, la différence de flux thermique entre une coupelle de référence et la coupelle d'échantillonnage en fonction du temps (pour les mesurages OIT) ou en fonction de la température (pour les mesurages OITP) doit être mesurée.

7.5 Étalonnage

L'appareil de mesure doit être étalonné selon les recommandations du fabricant et selon la procédure d'assurance qualité (AQ) appropriée, au moyen d'un étalon de référence adapté pour les plages de températures utilisées (par exemple, plomb/indium/étain). Le mesurage d'un échantillon de référence doit être effectué avant chaque lot de mesurages OIT ou OITP pour vérifier cet étalonnage.