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INTERNATIONAL STANDARD

NORME INTERNATIONALE

Electrical insulating materials – Determination of the effects of ionizing radiation on insulating materials –
Part 2: Procedures for irradiation and test

Matériaux isolants électriques – détermination des effets des rayonnements Ionisants sur les matériaux isolants –
Partie 2: Méthodes d'irradiation et d'essai

IEC 60544-2:2012

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

ELECTRICAL INSULATING MATERIALS – DETERMINATION OF THE EFFECTS OF IONIZING RADIATION ON INSULATING MATERIALS –

Part 2: Procedures for irradiation and test

FOREWORD

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International Standard IEC 60544-2 has been prepared by IEC technical committee 112: Evaluation and qualification of electrical insulating materials and systems.

This third edition cancels and replaces the second edition, published in 1991, and constitutes a technical revision.

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

– alignment with standards recently developed by SC 45A as well as with other parts in the IEC 60544 series.

The text of this standard is based on the following documents:

FDIS	Report on voting
112/208/FDIS	112/216/RVD

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

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

A list of all parts of the IEC 60544 series can be found, under the general title *Electrical insulating materials – Determination of the effects of ionizing radiation on insulating materials*, on the IEC website.

Future standards in this series will carry the new general title as cited above. Titles of existing standards in this series will be updated at the time of the next edition.

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

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- withdrawn,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

INTRODUCTION

When selecting insulating materials for applications in radiation environments, the component designers should have available reliable test data to compare candidate materials. To be meaningful, the performance data should be obtained on each material by standardized procedures, and the procedures should be designed to demonstrate the influence that variations of the service conditions have on the significant properties. This point is of particular concern where in normal service conditions low dose rates exist and where the insulation materials have been selected from radiation endurance data obtained from tests conducted at high dose rates.

Environmental conditions shall be well controlled and documented during the measurement of radiation effects. Important environmental parameters include temperature, reactive medium and mechanical and electrical stresses present during the irradiation. If air is present, radiation-induced species can enter into reactions with oxygen that would not occur in its absence. This is responsible for an observed influence of the absorbed dose rate for certain types of polymers if irradiated in air. As a result, the resistance may be several orders of magnitude lower than when the sample is irradiated under vacuum or in the presence of inert gas. This is generally called the "dose-rate effect", which is described and reviewed in references [1] to [14][1].

NOTE For the user of this Part of IEC 60544 who wants to go into more detail, the cited references are listed in the Bibliography. Where these are not publications in internationally available journals, addresses where the cited scientific reports can be obtained are given at the end of the references.

The irradiation time can become relevant because of time-dependent complications caused by:

a) physical effects such as diffusion-limited oxidation [8], [10]; and

b) chemical phenomena such as rate-determining hydroperoxide breakdown reactions [10], [14].

Typical diffusion-limited effects are commonly observed in radiation studies of polymers in air. Their importance depends upon the interrelationship of the geometry of the polymer with the oxygen permeation and consumption rates, both of which depend upon temperature [10]. This means that the irradiation of thick samples in air may result in oxidation only near the air-exposed surfaces of the sample, resulting in material property changes similar to those obtained by irradiation in an oxygen-free environment. Therefore, when the material is to be used in air for a long period of time at a low dose rate, depositing the same total dose at a high dose rate in a short exposure period may not determine its durability. Previous experiments or considerations of sample thickness combined with estimates of oxygen permeation and consumption rates [8], [10] may eliminate such concerns. A technique that may be useful for eliminating oxygen diffusion effects by increasing the surrounding oxygen pressure is under investigation [8].

Radiation-induced reactions will be influenced by temperature. An increase in reaction rate with temperature can result in a synergistic effect of radiation and heat. In the case of the more commonly used thermal ageing prediction, the Arrhenius method is employed; this makes use of an equation based on fundamental chemical kinetics. Despite considerable ongoing investigations of radiation ageing methodologies, this field is much less developed [9]. General equations involving dose, time, Arrhenius activation energy, dose rate and temperature are being tested for modelling of ageing experiments [10-12]. It should be noted that sequential application of radiation and heat, as it is frequently practised, can give very different results depending on the order in which they are performed, and that synergistic effects may not be properly simulated [13], [14].

The electrical and mechanical properties required of insulating materials and the acceptable amount of radiation-induced changes are so varied that it is not possible to establish

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[1] References in square brackets refer to the bibliography.

acceptable properties within the framework of a recommendation. The same holds for the irradiation conditions. Therefore, this standard recommends only a few properties and irradiation conditions which previous experience has shown to be appropriate. The properties recommended are those that are especially sensitive to radiation. For a specific application, other properties may have to be selected.

Part 1 of IEC 60544 constitutes an introduction dealing very broadly with the problems involved in evaluating radiation effects. It also provides a guide to dosimetry terminology, several methods of determining the exposure and absorbed dose, and methods of calculating the absorbed dose in any specific material from the dosimetry method applied. The present part describes procedures for irradiation and test. Part 4 of IEC 60544 defines a classification system to categorize the radiation endurance of insulating materials. It provides a set of parameters characterizing the suitability for radiation service. It is a guide for the selection, indexing and specification of insulating materials. The earlier Part 3 of IEC 60544 has been incorporated into the present Part 2.

ELECTRICAL INSULATING MATERIALS – DETERMINATION OF THE EFFECTS OF IONIZING RADIATION ON INSULATING MATERIALS –

Part 2: Procedures for irradiation and test

1 Scope

This Part of IEC 60544 specifies the controls maintained over the exposure conditions during and after the irradiation of insulating materials with ionizing radiation prior to the determination of radiation-induced changes in physical or chemical properties.

This standard specifies a number of potentially significant irradiation conditions as well as various parameters which can influence the radiation-induced reactions under these conditions.

The objective of this standard is to emphasize the importance of selecting suitable specimens, exposure conditions and test methods for determining the effect of radiation on appropriately chosen properties. Since many materials are used either in air or in inert environments, standard exposure conditions are recommended for both of these situations.

It should be noted that this standard does not consider measurements which are performed during the irradiation.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

IEC 60093, *Methods of test for volume resistivity and surface resistivity of solid electrical insulating materials*

IEC 60167, *Methods of test for the determination of the insulation resistance of solid insulating materials*

IEC 60212, *Standard conditions for use prior to and during the testing of solid electrical insulating materials*

IEC 60243-1, *Electrical strength of insulating materials –Test methods – Part 1: Tests at power frequencies*

IEC 60544-1, *Electrical insulating materials – Determination of the effects of ionizing radiation – Part 1: Radiation interaction and dosimetry*

IEC 60544-4, *Electrical insulating materials – Determination of the effects of ionizing radiation – Part 4: Classification system for service in radiation environments*

ISO 37, *Rubber, vulcanized or thermoplastic – Determination of tensile stress-strain properties*

ISO 48, *Rubber, vulcanized or thermoplastic – Determination of hardness (hardness between 10 IRHD and 100 IRHD)*

ISO 178, *Plastics – Determination of flexural properties*

ISO 179 (all parts), *Plastics – Determination of Charpy impact properties*

ISO 527 (all parts), *Plastics – Determination of tensile properties*

ISO 815 (all parts), *Rubber, vulcanized or thermoplastic – Determination of compression set*

ISO 868, *Plastics and ebonite – Determination of indentation hardness by means of a durometer (Shore hardness)*

3 Irradiation

3.1 Type of radiation and dosimetry

The following types of radiation are covered by the standard:

- X- and γ-rays;
- electrons;
- protons;
- neutrons;
- combined γ-rays and neutrons ("reactor" radiation).

In general, the radiation effects may be different for different types of radiation. However, in many practical applications, it has been found that with analogous experimental conditions, equal absorbed dose and equal linear energy transfer, the changes in properties will be only slightly dependent on the type of radiation [15-17]. Thus, the preferred type of radiation should be one for which the absorbed dose measurement is simple and precise, for example ^{60}Co γ-rays or fast electrons. For a comparison of the effect of reactor radiation with γ-rays or fast electrons, specimens with the same chemical composition can be irradiated with these various types of radiation and the radiation-induced changes can be compared.

Radiation-induced changes are related to the absorbed radiation energy, expressed by the absorbed dose. Recommended methods of dosimetry are listed in IEC 60544-1. The definitions of absorbed dose, absorbed dose rate and the units are also given in IEC 60544-1 and repeated here for convenience.

The absorbed dose, D, is the quotient of $\mathrm{d}\bar{\varepsilon}$ by $\mathrm{d}m$, where $\mathrm{d}\bar{\varepsilon}$ is the mean energy imparted by ionizing radiation to the matter in a volume element and $\mathrm{d}m$ is the mass of the matter in that volume element.

$$D = \frac{\mathrm{d}\bar{\varepsilon}}{\mathrm{d}m}$$

The absorbed dose rate, D, is the increment of the absorbed dose $\mathrm{d}D$ in the time interval $\mathrm{d}t$.

$$D = \frac{\mathrm{d}D}{\mathrm{d}t}$$

Units

The SI unit of absorbed dose is the gray (Gy);

1 Gy = 1 J/kg (= 10^2 rad).

Usual multiples for higher doses are the kilogray (kGy) or megagray (MGy).

The SI unit of absorbed dose rate is the gray per second;

1 Gy/s = 1 W/kg (=10^2 rad/s = 0,36 Mrad/h).

3.2 Irradiation conditions

The irradiation conditions which must be established are as follows:

- type and energy of the radiation;
- absorbed dose;
- absorbed dose rate;
- surrounding medium;
- temperature;
- mechanical, electrical and other stresses;
- sample thickness.

It is preferable to use γ-rays, X-rays or electrons for the irradiation (see 3.1). Their energy should be so chosen that the homogeneity of the absorbed dose in the sample is within ±15 %.

3.3 Sample preparation

The test specimens shall be carefully prepared in accordance with the appropriate IEC and ISO standards, because a variation in test results may be due to differences in the quality of test specimens.

Because the effect of radiation can depend on the dimensions of the specimens, these shall be uniform for all comparison studies. It is preferable to irradiate the test specimens in the geometry needed for subsequent tests. If, however, the test specimens have to be cut from a larger irradiated test piece, the position of the specimen in the test piece shall be reported.

Non-irradiated control specimens shall be produced in the same manner and subjected to the same conditioning and post-irradiation treatment as the irradiated specimens.

3.4 Irradiation procedures

3.4.1 Irradiation dose-rate control

The exposure rate is usually non-uniform in the radiation field. In addition, it is reduced by the energy absorption in the specimen itself. Therefore, the absorbed dose cannot be homogeneous. Improvements in homogeneity may be achieved by filtering methods, by irradiation of the specimens from several directions, by traversing the radiation field at a constant rate or by scanning the specimen with the radiation beam. The homogeneity of the absorbed dose rate should be improved rotating or moving the sample during the irradiation, for example, by means of suitable equipments. It is expected that variations in dose rate within ±15 % will not appreciably affect the results (see 3.2); variations outside this recommended value shall be reported.

3.4.2 Irradiation temperature control

The specimens shall be conditioned at the irradiation temperature for 48 h, or until an approximate equilibrium with the irradiation temperature is ensured.

The temperatures shall be chosen from the standardized series given in IEC 60212.

The temperature of the specimens during irradiation shall be determined by the use of a supplementary specimen containing a temperature-measuring device, irradiated under the same conditions as the other specimens. The measuring device and its position in the specimen have to be carefully chosen so to avoid that the irradiation influences the temperature measurements.

The temperature variations are a function of the actual temperature of the experiment. Larger tolerances (e.g. ±5 K) are allowed at ambient temperatures up to approximately 40 °C, smaller tolerances (e.g. ±2 K) are reasonable at higher temperatures where temperature control is used. Deviations of more than ±2 K shall be reported.

Irradiation at high dose rates may cause the temperature to rise. The temperature may be controlled in any way that does not affect the material properties or radiation conditions.

Irradiations in the region of a transition (e.g. melting, glass or secondary transition) shall be noted, since degradation behaviour can change significantly as a material passes through such a transition.

3.4.3 Irradiation in air

Specimens to be irradiated in air shall be arranged so that free access to air is ensured on all sides. The build-up of radiation-induced reaction products is to be prevented (e.g. by a flow of fresh air over the specimen), except in cases where it is desirable to determine whether the products (e.g. O_3 or HCl) affect the material properties.

If the nature of the radiation source requires that the specimens be enclosed in a container, package the specimens in the standard atmosphere. In general, the conditions in the container (e.g. pressure and chemical composition of atmosphere) will be changed by irradiation. This could seriously affect the results. Therefore, the air within the container should be changed frequently. It shall be stated in the report that irradiation was made in a closed container, the material of which the container was made, the ratio between the volumes of specimens and air, and how often the air was renewed. The possibility of a pressure rise by heating or by reaction products is to be considered in the design of the container so that this effect is minimized.

3.4.4 Irradiation in a medium other than air

Specimens to be irradiated in a gas other than air shall be conditioned in a container at a pressure of ≤1 Pa (10^{-5} bar) for at least 8 h, followed by three flushes with the gas. After flushing, the specimens shall remain in the container filled with gas at the temperature of the irradiation until an approximate equilibrium of the specimens with the gas is ensured. During irradiation it is best to maintain a continuous flow of gas through the specimen container. When necessary, a sealed container may be used if the gas is changed periodically. Sealing the container for the entire exposure is permitted only if it is unavoidable due to the nature of the source. The details of the method shall be reported.

Specimens to be irradiated in a liquid medium shall be immersed for a sufficient period of time to reach approximate equilibrium with the liquid before the irradiation. The radiation resistance may be influenced by swelling induced during the conditioning time. During the entire period of irradiation the specimens shall be completely immersed in the liquid. Stirring of the liquid, streaming or other methods used to supply new liquid to the specimen shall be reported.

3.4.5 Irradiation in a vacuum

Specimens to be irradiated in a vacuum shall be conditioned in a container at a pressure of ≤1 Pa (10^{-5} bar) for at least 24 h and that pressure shall not be exceeded throughout the irradiation.

3.4.6 Irradiation at high pressure

Specimens to be irradiated at high pressure shall be conditioned in a container at that pressure for sufficient lengths of time to reach approximate equilibrium, and the selected pressure shall be maintained throughout the irradiation. A possible technique for irradiation under oxygen pressure is described in [8]. Details of the exposure conditions shall be reported.

3.4.7 Irradiation during mechanical stressing

The specimens shall be arranged on a suitable fixture so that they will be subject to a mechanical stress during irradiation. A description of the method shall be reported.

3.4.8 Irradiation during electrical stressing

The specimens shall be arranged on a suitable fixture so that they will be subject to an electrical stress during irradiation. A description of the method shall be reported.

3.4.9 Combined irradiation procedures

When any combination of two or more of the variables listed in the above procedures is used, the combined procedure shall incorporate all the appropriate features of the separate procedures involved.

3.5 Post-irradiation effects

The irradiation of polymers results in the formation of free radicals or other reactive species. The rate at which some of these are formed may be much greater than their reaction rate; this leads to the accumulation of reactive species within the irradiated material and to the possibility of continuing reactions after the specimen has been removed from the radiation field. Because of this effect, specimens shall be tested as soon as possible (preferably within one week) after the end of irradiation.

3.6 Specified irradiation conditions

Problems related to assessing the effects at long-term service conditions by short-term laboratory tests are discussed in the Introduction. Two irradiation conditions are given below which are intended to provide a measure of the time-related oxygen effects:

- Short time exposure in non-oxidizing conditions, e.g. either in the absence of oxygen or for thick samples at high absorbed dose rates usually in excess of 1 Gy/s.

  Since radiation heating can occur at high dose rates, the upper limit is governed by the specified test temperature.
- Long time exposure conditions in the presence of oxygen (ambient air) at low dose rates up to 3×10^{-2} Gy/s.

NOTE The recommended long time exposure employs a dose rate that was chosen as a compromise between long-term field service conditions and practical test durations. It can still be several orders of magnitude higher than the dose rate that occurs in many long-term applications of interest. Further significant dose rate effects may apply due to these differences, and the size will depend on the polymer type and sample thickness. At present, test procedures predicting life times at much lower dose rates than 3×10^{-2} Gy/s are subject to research [9 – 12].

For application in nuclear reactor service, it is preferable to irradiate the specimens at two temperatures: room temperature (23 ±5) °C and 80 °C. Consideration should be given to 3.4.2.

4 Test

4.1 General

The radiation resistance can be characterized by:

- the absorbed dose required to produce a predetermined change in a property (see 4.3.1), or
- the amount of change in a property produced by a fixed value of absorbed dose (see 4.3.2).

To establish radiation resistance the following points shall be defined:

- irradiation conditions (see Clause 3);
- properties whose changes may be evaluated (see 4.2);
- end-point criteria of properties and/or values of absorbed dose (see 4.3).

The tests are intended to determine permanent changes in the properties of the material. Transient changes occurring during the irradiation are not dealt with in this standard.

4.2 Test procedures

Some properties which may be considered for monitoring radiation effects are listed in Table 1 together with the appropriate test procedures. Although electrical properties can change drastically when a material fails, they are much less sensitive than mechanical properties for monitoring damage built up before failure [18], [19]. Mechanical properties may be improved initially in plastics which crosslink, but with higher absorbed doses most plastics become brittle and technically unusable. This process of becoming brittle should be considered when the properties to be tested are chosen.

For normal application, experience has shown that the most appropriate mechanical properties are

- the flexural stress at maximum load for rigid plastics, and
- the percentage elongation at break for flexible plastics and elastomers.

Should the application warrant it, the user may specify an alternative property taken from Table 1 or any alternative procedure. Also, since the radiation source and container have a limited volume over which the radiation field is sufficiently uniform, this may imply restrictions in sample size.

4.3 Evaluation criteria

4.3.1 End-point criteria

The end-point criterion may be expressed as an absolute property value or a percentage of the initial value. Either method may be used to classify materials for radiation resistance. Table 1 provides examples of ranking materials using a percentage of the initial value. The assessment of a radiation index is given in IEC 60544-4.

For a specific application or service condition, a more appropriate end-point value may be selected that will reflect end-use requirements.