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INTERNATIONAL STANDARD

NORME INTERNATIONALE

Electrical insulating materials – Determination of the effects of ionizing radiation – Part 1: Radiation interaction and dosimetry

Matériaux isolants électriques – Détermination des effets des rayonnements ionisants – Partie 1: Interaction des rayonnements et dosimétrie

IEC 60544-1:2013

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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

Part 1: Radiation interaction and dosimetry

FOREWORD

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International Standard IEC 60544-1 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 1994 and constitutes a technical revision.

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

a) recent advances in simulation methods of radiation interaction with different matter enables the prediction of the energy-deposition profile in matter and design the irradiation procedure;
b) many new dosimetry systems have become available.

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

FDIS	Report on voting
112/254/FDIS	112/262/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 in the IEC 60544 series, published under the general title *Electrical insulating materials – Determination of the effects of ionizing radiation*, can be found on the IEC website.

The committee has decided that the contents of this publication will remain unchanged until the stability date indicated on the IEC 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- withdrawn,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

The establishment of suitable criteria for the evaluation of the radiation resistance of insulating materials is very complex, since such criteria depend upon the conditions under which the materials are used. For instance, if an insulated cable is flexed during a refuelling operation in a reactor, the service life will be that time during which the cable receives a radiation dose sufficient to reduce to a specified value one or more of the relevant mechanical properties. Temperature of operation, composition of the surrounding atmosphere and the time interval during which the total dose is received (dose rate or flux) are important factors which also determine the rate and mechanisms of chemical changes. In some applications, temporary changes may be the limiting factor.

Given this, it becomes necessary to define the radiation fields in which materials are exposed and the radiation dose subsequently absorbed by the material. It is also necessary to establish procedures for testing the mechanical and electrical properties of materials which will define the radiation degradation and link those properties with application requirements in order to provide an appropriate classification system.

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

Part 1: Radiation interaction and dosimetry

1 Scope

This part of IEC 60544 deals broadly with the aspects to be considered in evaluating the effects of ionizing radiation on all types of organic insulating materials. It also provides, for X-rays, γ-rays, and electrons, a guide to

– dosimetry terminology,
– methods for dose measurements,
– testing carried out at irradiation facilities,
– evaluation and testing of material characteristics and properties,
– documenting the irradiation process.

Dosimetry that might be carried out at locations of use of the material is not described in this standard.

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 60544-2, *Electrical insulating materials – Determination of the effects of ionizing radiation on insulating materials – Part 2: Procedures for irradiation and test*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions in ICRU Report 33 [1][1]. as well as the following definitions apply.

3.1
exposure
X
measure of an electromagnetic radiation field (X- or γ-radiation) to which a material is exposed

Note 1 to entry: The exposure is the quotient obtained by dividing d*Q* by d*m*, where d*Q* is the absolute value of the total charge of the ions of one sign produced in the air when all of the electrons (and positrons) liberated by photons in air of mass d*m* are completely stopped in air:

1 References in square brackets refer to the Bibliography.

$$X = \frac{\mathrm{d}Q}{\mathrm{d}m} \tag{1}$$

The SI unit of exposure is the coulomb (C) per kilogram: C/kg. The old unit is the roentgen R: 1 R = 2,58 × 10^{-4} C/kg.

The exposure thus describes the effect of an electromagnetic field on matter in terms of the ionization that the radiation produces in a standard reference material, air.

3.2
electron charge fluence
Q'
quotient obtained by dividing dQ by dA, where dQ is the electron charge impinging during the time t on the area dA:

$$Q' = \frac{\mathrm{d}Q}{\mathrm{d}A} \tag{2}$$

3.3
electron current density
j
quotient obtained by dividing dQ' by dt, where dQ' is the electron charge fluence during the time interval dt:

$$j = \frac{\mathrm{d}Q'}{\mathrm{d}t} = \frac{\mathrm{d}^2 Q}{\mathrm{d}A\,\mathrm{d}t} \tag{3}$$

3.4
absorbed dose
D
measure of the energy imparted to the irradiated material, regardless of the nature of the radiation field

Note 1 to entry: The absorbed dose D is the quotient obtained by dividing d$\bar{\varepsilon}$ by dm where d$\bar{\varepsilon}$ is the mean energy imparted by ionizing radiation to matter of mass dm:

$$D = \frac{\mathrm{d}\bar{\varepsilon}}{\mathrm{d}m} \tag{4}$$

The SI unit is the gray (Gy). The old unit is the rad:

1 Gy = 1 J × kg^{-1} (= 10^2 rad).

Since this definition does not specify the absorbing material, the gray can be used only with reference to a specific material. The absorbed dose is determined in part by the composition of the irradiated material. When exposed to the same radiation field, therefore, different materials usually receive different absorbed doses.

Note 2 to entry: For purposes of dosimetry, it has been found convenient to specify dose in terms of dose to water. The dose to other materials can be found by applying cavity theory.

3.5
absorbed dose rate
$\dot{D}$
quotient obtained by dividing dD by dt, where dD is the increment of absorbed dose in the time interval dt:

$$\dot{D} = \frac{\mathrm{d}D}{\mathrm{d}t} \tag{5}$$

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

$$1\ \text{Gy} \times \text{s}^{-1} = 1\ \text{W} \times \text{kg}^{-1}\ (= 10^2\ \text{rad} \times \text{s}^{-1} = 0{,}36\ \text{Mrad} \times \text{h}^{-1})$$

4 Radiation-induced changes and their evaluation

4.1 General

Although the various types of radiation interact with matter in different ways, the primary process is the production of ions and electrically excited states of molecules which, in turn, may lead to the formation of free radicals. The technique to detect ions, excited states and radicals (short-lived intermediate species) are briefly described in Clause A.4. Radiation-generated mobile electrons, which become trapped at sites of low potential energy, are also produced. The first phenomenon leads to permanent chemical, mechanical, and electrical changes of the material; the second results in temporary electrical changes in performance [2].

4.2 Permanent changes

In polymeric materials, the formation of free radicals during irradiation leads to scission and cross-linking processes that modify the chemical structure of the insulation, generally leading to deterioration of the mechanical properties. This mechanical deterioration frequently gives rise to significant electrical property changes. However, important electrical property changes sometimes occur before mechanical degradation becomes serious. For example, a change in dissipation factor or in permittivity might become serious for the reliable functioning of a resonant circuit. The extent of scission and cross-linking processes depends on the absorbed dose, the absorbed dose rate, the material geometry and the environmental conditions present during the irradiation. Because the free radicals sometimes decay slowly, there may also be post-irradiation effects.

4.3 Environmental conditions and material geometry

Environmental conditions and test specimen geometry 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, the irradiation time (flux and dose rate) has also been demonstrated to be a very important experimental parameter because of oxygen diffusion effects and hydroperoxide breakdown rate constants. Both factors are time dependent. The conditions that influence oxygen diffusion and equilibrium concentrations in the polymer shall be controlled. These include: temperature, oxygen pressure, material geometry and the time during which the dose is applied.

If the effect of simultaneous stresses, e.g. radiation at high temperature, is simulated by sequential stressing, other results are to be expected. Further, there can be differences in results if the sample is first irradiated and then heat aged or vice versa.

4.4 Post-irradiation effects

In organic polymers, there may be post-irradiation effects due to the gradual decay of various reactants, such as residual free radicals. Due allowance shall be made for this type of behaviour in any evaluation procedure. The tests shall be made at recorded intervals after irradiation, maintaining specimen storage in a standard laboratory atmosphere. The reaction of oxygen with residual free radicals can cause further degradation.

4.5 Temporary effects

4.5.1 Performing measurements during irradiation is not within the scope of this part of IEC 60544. Despite this, some basic aspects will be discussed briefly. The temporary effects appear primarily as changes in electrical properties such as induced conductivity, both during and for some time after irradiation. Hence, measurement of the induced conductivity

could be used as an evaluation property to determine the temporary radiation effects. These effects are primarily dose-rate dependent.

4.5.2 Experience has shown that the induced conductivity is usually not quite proportional to the absorbed dose rate $\dot{D}$, but varies as $\dot{D}^{\alpha}$, where α is smaller than unity. Hence, the radiation sensitivity is described by the relation:

$$\sigma_i = k\,\dot{D}^{\alpha} \tag{6}$$

To determine k and α, at least two measurements are needed. A further complication comes from the fact that k and α also depend on the integrated dose absorbed by the sample.

The measurement of the induced conductivity is actually quite delicate, since photoelectrons and Compton electrons in the electrode materials will tend to perturb the intrinsic induced current of the specimen. Ionic currents through the ionized atmosphere will also introduce errors in the measurement if they are not eliminated. Experimental procedures eliminating most of the disturbing effects, while remaining relatively simple, shall be defined.

NOTE It is convenient to use a simple figure such as the induced conductivity σ_i or σ_i/σ_o, its ratio to the dark conductivity σ_o measured in the same experimental conditions, per unit dose rate to characterize the sensitivity of the materials to temporary effects.

5 Facilities for irradiation of material samples for evaluation of properties

5.1 General

Irradiation of material samples for evaluation of properties shall be performed at irradiation facilities that have undergone installation qualification, operational qualification and performance qualification, see e.g. ISO 11137 [3].

Three principal types of radiation sources are used:

- gamma radiation from radionuclides such as ^{60}Co (1,25 MeV) and ^{137}Cs (0,66 MeV);
- electrons from accelerators;
- X-rays generated from accelerated electrons.

The design and properties of an irradiation facility have implications for absorbed dose distribution in the samples and attainable absorbed dose range. Major considerations in the design of an irradiation facility are the uniformity of the distribution of absorbed dose in the given product, efficient utilization of radiation energy.

5.2 Gamma-ray irradiators

Large capacity gamma radiation facilities usually use ^{60}Co as the radiation source. The sources are often in the form of individual source capsules arranged in an array to maximize the volume available for irradiations. The dose rates that are available will be dependent on the distance from the sources at which the samples are placed. Typically, dose rates in the range 10 kGy/h (2,78 Gy/s) down to 1 Gy/h (0,278 mGy/s) are possible. This covers the range of dose rates that are of particular interest for materials degradation testing.

5.3 Electron-beam irradiators

Electron beam irradiators use accelerators that generate electron beam in the energy-range of 300 KeV – 10 MeV. At present, various types of accelerating procedures are available; examples include electro-static type and high-frequency (radio-frequency) type. With respect to radiation resistance testing, electro-static type of (0,5 – 3)MeV is widely used. In an electro-static accelerating system, thermo-electrons are emitted from a cathode and the

emitted electrons are accelerated with high voltage applied between electrodes. Electron beams are electro-magnetically scanned in a scanning horn and taken out from the window (typically made of a thin foil of Titanium). The operation of electron accelerators is simple and safe, i.e. there is no radiation if the power is switched off, compared to a ^{60}Co gamma irradiation facility. Depending on voltage (energy), beam current, scan width, distance between the window and samples, static or conveyor irradiation, the dose rate may change, but typically it is in the order of kGy/s, which is much higher compared with gamma irradiator. Penetration of the electron beam in samples shall be taken into account (see Clause A.3).

5.4 X-ray (Bremsstrahlung) irradiators

X-rays (or Bremsstrahlung) are created when accelerated electrons are slowed down in an absorbing material. The fraction of kinetic energy of the electrons that is converted into X-rays (conversion efficiency) is higher for absorbers with a higher atomic number, and therefore materials such as tungsten are used as X-ray converters. The conversion efficiency also increases with increasing electron energy. At 5 MeV it is about 5 % in tungsten, increasing to about 12,5 % at 10 MeV, and the low conversion efficiency at this energy has limited the use of this type of irradiators. The advent of high-power electron accelerators in the range from 5 MeV to 10 MeV has renewed interest in the use of X-rays for irradiation of products.

In contrast to radionuclide sources, which emit nearly mono-energetic photons, X-ray sources emit a broad spectrum of photons from the maximum energy of the electrons to zero energy. For example, an X-ray beam generated by 5 MeV electrons has approximately the same penetration characteristics as ^{60}Co radiation. Other characteristics of the X-ray beam, such as scanning and pulsing of the beam, are derived from the characteristics of the electron beam that generated the X-rays.

6 Dosimetry methods

6.1 General

It is necessary to ensure that the correct absorbed dose is applied during irradiation. The dose shall be measured, and measurement systems have been developed for this purpose. Much of the development of these systems rests on the early development of dosimetry systems for personnel radiation protection and for medical treatment. However, the doses used in material testing are generally higher, ranging from a few kGy to 100 kGy or more and new dosimetry systems have been developed for measurements of these doses. Dose shall be measured with traceability to national standards, and the uncertainty known, including the effect of influencing parameters.

Absolute methods of dosimetry are maintained as national standards by a number of national laboratories. These dosimeters provide dose measurement by means of physical measurements that do not depend on calibration of the dosimeter in a known radiation field. Other dosimeters are calibrated against these national standard dosimeters, thereby providing measurement traceability to the national standard dosimeters.

A number of dosimeter systems are in use at irradiation facilities and laboratories for measurement of dose distribution for facility characterization and in products and samples to be irradiated. These dosimeters are also used for monitoring the irradiation process. Selection of a dosimeter system depends on the measurement task to be carried and of the properties of the dosimeter. Dosimetry methods and dosimeter systems are described in several ISO/ASTM standards and guides [4 – 18]. More details of several of these dosimetry systems are found in ICRU 80 [19].

6.2 Absolute dosimetry methods

6.2.1 Gamma-rays

Free-air ionization chambers are used to measure exposure X up to 3 MeV, i.e. they are designed to measure the quantity of charge dQ produced in air and the mass dm of air where the ionizing electrons are liberated. Ionization chambers can be used if the dose rate is not too high [20].

Calorimeters operate by absorbing energy from the radiation field in which they are placed; they retain this energy until it is converted to thermal energy and this heat quantity is evaluated by measuring the rise in temperature of the calorimetric absorber [21].

6.2.2 Electron beams

In addition to calorimetric methods, measurement of electron current density has been used to measure electron charge or current per unit area of radiation fields of electron accelerators. This method is not a dosimetric method, but enables the calibration of absorbed dose if the mean electron energy impinging on the charge absorber of the densitometer and the relative depth-dose distribution in the same absorber material are known.

6.3 Dosimetry systems

6.3.1 Reference standard dosimetry systems

Reference standard dosimetry systems are used as standards to calibrate the dosimetry systems that are used for routine measurements. The uncertainty of the reference standard dosimetry system will affect the uncertainty of the system being calibrated and it is therefore important that the reference standard dosimetry system is of high metrological quality. In this context, the concept of high metrological quality implies a system with low uncertainty and with traceability to appropriate national or international standards. It also implies that the response of the reference standard dosimeter is not significantly influenced by environment.

The expanded uncertainty achievable with measurements made using a reference standard dosimetry system is typically of the order of ± 3 % (k = 2, which corresponds approximately to a 95 % level of confidence for normally distributed data). In certain specific applications, for example the use of electrons of energy below 1 MeV, practical limitations of the techniques may mean that the reference standard dosimetry systems have a larger uncertainty.

Examples of reference standard dosimetry systems are given in Table 1.

NOTE ASTM E 2628-09 “Standard practice for dosimetry for radiation processing” [22] is a valuable guideline concerning Table 1 and Table 2.

Table 1 – Examples of reference standard dosimeters

Dosimeter	Description	Reference	Dose range Gy	Dose rate range Gy/s	Influencing parameters
Fricke solution	Liquid solution of ferrous and ferric ions in 0,4 M sulphuric acid. Measured by spectrophotometry	ASTM E1026 [23]	20 to 4×10^2	$< 10^6$	Temperature
Alanine/EPR (electron paramagnetic resonance)	Pellet or film containing alanine. Measured by EPR spectroscopy of radiation induced radical	ISO/ASTM 51607 [8]	1 to 10^5	$< 10^8$	Temperature Humidity
Dichromate	Liquid solution of chromium ions in 0,1 M perchloric acid. Measured by spectrophotometry.	ISO/ASTM 51401 [9]	2×10^3 to 5×10^4	Pulsed: < 600 Gy/pulse (12,5 pps) Continuous: $< 7{,}5 \times 10^{-3}$	Temperature
Ceric-cerous sulphate	Liquid solution of ceric and cerous ions in 0,4 M sulphuric acid. Measured by spectrophotometry or potentiometry	ISO/ASTM 51205 [10]	5×10^2 to 10^5	$< 10^6$	Temperature
Ethanol chlorobenzene (Classification dependent on solution composition and method of measurement)	Liquid solutions of various compositions containing chlorobenzene in ethanol. Measured by titration	ISO/ASTM 51538 [11]	10 to 2×10^6	$< 10^6$	Temperature

6.3.2 Routine dosimetry systems

The classification of a dosimetry system as a routine dosimetry system is based on its application i.e. routine absorbed dose measurements, including dose mapping and process monitoring. A routine dosimetry system comprises dosimeters and the associated measurement equipment and quality system documentation necessary to ensure traceability to appropriate national or international standards. The response of routine dosimeters is often influenced by the environment in a complex way.

The expanded uncertainty achievable with measurements made using a routine dosimetry system is typically of the order of ± 6 % ($k = 2$).

Examples of routine dosimetry systems are given in Table 2. Dosimeters in Table 1 can also be used as routine systems.