BS EN 60544-1:2013



BSI Standards Publication

Electrical insulating materials — Determination of the effects of ionizing radiation

Part 1: Radiation interaction and dosimetry



BS EN 60544-1:2013 BRITISH STANDARD

National foreword

This British Standard is the UK implementation of EN 60544-1:2013. It is identical to IEC 60544-1:2013. It supersedes BS EN 60544-1:1995 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee GEL/112, Evaluation and qualification of electrical insulating materials and systems.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 78240 4 ICS 29.035.01

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 September 2013.

Amendments/corrigenda issued since publication

Date Text affected

EUROPEAN STANDARD

EN 60544-1

NORME EUROPÉENNE EUROPÄISCHE NORM

September 2013

ICS 17.240; 29.035.01

Supersedes EN 60544-1:1994

English version

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

(IEC 60544-1:2013)

Matériaux isolants électriques -Détermination des effets des rayonnements ionisants -Partie 1: Interaction des rayonnements et dosimétrie (CEI 60544-1:2013) Elektroisolierstoffe - Bestimmung der Wirkung ionisierender Strahlung -Teil 1: Einfluss der Strahlenwirkung und Dosimetrie (IEC 60544-1:2013)

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Foreword

The text of document 112/254/FDIS, future edition 3 of IEC 60544-1, prepared by IEC TC 112 "Evaluation and qualification of electrical insulating materials and systems" was submitted to the IEC-CENELEC parallel vote and approved by CENELEC as EN 60544-1:2013.

The following dates are fixed:

•	latest date by which the document has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2014-05-01
•	latest date by which the national standards conflicting with the document have to be withdrawn	(dow)	2016-08-01

This document supersedes EN 60544-1:1994.

EN 60544-1:2013 includes the following significant technical changes with respect to EN 60544-1:1994:

- 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.

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The text of the International Standard IEC 60544-1:2013 was approved by CENELEC as a European Standard without any modification.

In the official version, for Bibliography, the following note has to be added for the standard indicated:

ISO 11137 series NOTE Harmonised in EN ISO 11137 series.

Annex ZA (normative)

Normative references to international publications with their corresponding European publications

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.

NOTE When an international publication has been modified by common modifications, indicated by (mod), the relevant EN/HD applies.

<u>Publication</u>	<u>Year</u>	<u>Title</u>	EN/HD	<u>Year</u>
IEC 60544-2	-	Electrical insulating materials - Determination of the effects of ionizing radiation on insulatin 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	EN 60544-4	-

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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 dQ by dm, where dQ 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 dm are completely stopped in air:

¹ References in square brackets refer to the Bibliography.

$$X = \frac{dQ}{dm} \tag{1}$$

The SI unit of exposure is the coulomb(C) per kilogram: C/kg. The old unit is the roentgen R: $1~R=2.58\times10^{-4}~C/kg$.

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

 Ω'

quotient obtained by dividing dQ by dA, where dQ is the electron charge impinging during the time t on the area dA:

$$Q' = \frac{dQ}{dA} \tag{2}$$

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:

$$j = \frac{dQ'}{dt} = \frac{d^2 Q}{dA dt}$$
 (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 $d\overline{\varepsilon}$ by dm where $d\overline{\varepsilon}$ is the mean energy imparted by ionizing radiation to matter of mass dm:

$$D = \frac{d\overline{\varepsilon}}{dm} \tag{4}$$

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

1 Gy = 1 J
$$\times$$
 kg⁻¹ (= 10² 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

D

quotient obtained by dividing dD by dt, where dD is the increment of absorbed dose in the time interval dt:

$$\dot{D} = \frac{dD}{dt} \tag{5}$$

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}^{α} , where α is smaller than unity. Hence, the radiation sensitivity is described by the relation:

$$\sigma_{i} = k \stackrel{\cdot}{D}^{\alpha} \tag{6}$$

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 $\sigma_{\rm i}$ or $\sigma_{\rm i}/\sigma_{\rm o}$, its ratio to the dark conductivity $\sigma_{\rm o}$ 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 ⁶⁰Co (1,25 MeV) and ¹³⁷Cs (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 ⁶⁰Co 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 ⁶⁰Co 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 ⁶⁰Co 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 \pm 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	< 108	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 × 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 \pm 6 % (k = 2).

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

Table 2 – Examples of routine dosimeter systems

Dosimeter	Description	Reference	Dose range Gy	Dose rate range Gy/s	Influencing parameters
Calorimeter	Assembly consisting of calorimetric body (absorber), thermal insulation, and temperature sensor with wiring	ISO/ASTM 51631 [12]	10 ² to 10 ⁵	> ~10	Temperature
Cellulose triacetate	Untinted cellulose triacetate (CTA) film. Measured by spectrophotometry	ISO/ASTM 51650 [13]	5 × 10 ³ to 10 ⁶	$3 \times 10^{-2} \text{ to}$ 3×10^{-7}	Temperature Humidity
Ethanol chlorobenzene (classification dependent on solution composition and method of measurement)	Liquid solutions of various compositions containing chlorobenzene in ethanol. Measured by spectrophotometry or oscillometry	ISO/ASTM 51538 [11]	10 to 2 × 10 ⁶	< 10 ⁶	Temperature
LiF photo- fluorescent	Lithium fluoride based photo-fluorescent film. Measured by photo-stimulated luminescence	ASTM E2304 [24]	5 × 10 ⁻² to 3 × 10 ²	0,3 to 2 × 10 ⁴	Temperature Humidity Ambient light
PMMA (polymethylmetha crylate)	Specially developed PMMA materials. Measured by spectrophotometry	ISO/ASTM 51276 [14]	10 ² to 10 ⁵	10 ⁻² to 10 ⁷	Temperature Humidity Ambient light
Radiochromic film	Specially prepared film containing dye precursors. Measured by spectrophotometry	ISO/ASTM 51275 [15]	10 ⁰ to 10 ⁵	< 10 ¹³	Temperature Humidity Ambient light
Radiochromic liquid	Specially prepared solution containing dye precursors. Measured by spectrophotometry	ISO/ASTM 51540 [16]	$5 \times 10^{-1} \text{ to}$ 4×10^{4}	<10 ⁻² to 10 ¹¹	Temperature Ambient light
Radiochromic optical waveguide	Specially prepared optical waveguide containing dye precursors. Measured by spectrophotometry	ISO/ASTM 51310 [17]	10 ⁰ to 10 ⁴	10 ⁻³ to 10 ³	Temperature Ambient light
TLD (thermoluminesen ce detector)	A phosphor, alone, or incorporated in a material. Measured by thermoluminescence	ISO/ASTM 51956 [18]	10 ⁻⁴ to 10 ³	10 ⁻² to 10 ¹⁰	Temperature Humidity Ambient light

6.3.3 Measurement uncertainty

To be meaningful, a measurement of absorbed dose shall be accompanied by an estimate of uncertainty. Components of uncertainty should be identified as belonging to one of two categories:

Type A — those evaluated by statistical methods, or

Type B — those evaluated by other means.

Estimates of the expanded uncertainty of an absorbed dose measurement should be made with a coverage factor k = 2.

NOTE The identification of Type A and Type B uncertainties is based on the methodology published in 1995 by the International Organization for Standardization (ISO) in the Guide to the Expression of Uncertainty in Measurement [25]. The purpose of using this kind of characterization is to promote an understanding of how uncertainty statements are arrived at and to provide a basis for the international comparison of measurement results.

6.3.4 Dosimeter calibration

The radiation induced response of dosimeters depends to a larger or smaller extent on the environment at which they are irradiated. For example, temperature, humidity and dose rate may affect the response, and it is therefore needed to calibrate dosimeters using conditions that are as close as possible to the conditions of use of the dosimeters.

Calibration of dosimeters should therefore be carried out using one of the following two methods:

- a) Irradiation of dosimeters at the irradiation facility where the dosimeters will be used ("inplant") together with transfer standard dosimeters issued and analysed by a national standards laboratory or an accredited dosimetry calibration laboratory.
- b) Irradiations of dosimeters at a national standards laboratory or an accredited dosimetry calibration laboratory. Calibration irradiations carried out in this way will results in a calibration curve generated under a single set of influencing parameters. The user of the dosimeter shall therefore evaluate the effect of influencing parameter values. This is most readily done by a verification irradiation "in-plant" at selected doses.

Measurement traceability is defined in the International vocabulary of metrology [26] as follows:

"the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons."

Calibration is an important step in obtaining measurement traceability.

6.3.5 Dosimeter selection

The selection and use of a specific dosimetry system in a given application shall be justified taking into account at least the following:

- · dose range;
- radiation type:
- · effect of influencing parameters;
- required level of uncertainty;
- · required spatial resolution.

The dosimetry system shall be calibrated in accordance with 6.3.4.

The uncertainty associated with measurements made with the dosimetry system shall be established and documented. All dose measurements shall be accompanied by an estimate of uncertainty.

Documentation shall be established and maintained to ensure compliance with the minimum requirements specified in standards for the dosimetry system. The user's quality system might be more detailed than these minimum requirements.

Tables 1 and 2 gives a non-exhaustive list of reference and routine dosimetry methods with some of their characteristics, such as:

- range of absorbed doses and absorbed dose rates;
- influence of the radiation energy;

- influence of temperature or humidity;
- material and thickness of dosimeter material;
- type of measurement;
- observations of practical interest;
- bibliographical references.

Clause A.2 gives an example for calculation of absorbed dose.

7 Characterization of irradiation facilities

Irradiation facilities that are to be used for evaluating the behaviour of electrical insulating materials shall be characterized before use. The parameters that need to be determined will be dependent on the type of facility being used.

For a gamma irradiation facility, the following parameters shall be determined:

- The dose rate distribution shall be measured within the volume of the facility that will be used for sample exposure to radiation. This mapping shall be in sufficient detail to enable the locations available for a specific dose rate to be determined.
- The ambient temperature within the radiation facility shall be measured while the sources are in their normal operating position.
- The time for which samples are exposed shall be measured. The measurement method shall have the ability to take into account any interruptions to the irradiation, e.g. retraction of the sources to allow access for removal of samples.

For electron beam and X-ray facilities, the following parameters shall be determined:

- The dose as a function of the beam current, beam spot size and beam width shall be measured.
- If a conveyor system is used to transport samples through the radiation field, the dose as a function of conveyor speed shall also be measured.

NOTE There are a number of standards available for dosimetry and dose mapping specifically for use in radiation processing facilities [4-7, 27].

8 Dose mapping of samples for test

8.1 Charged particle equilibrium

For irradiation in gamma facilities it is recommend to provide secondary electron equilibrium in the irradiated sample leading to more uniform dose distribution throughout the sample, see Clause A.1.

8.2 Depth-dose distribution (limitations)

Since the absorbed dose distribution through the specimen being irradiated will vary and is a function of the thickness of the specimen, its density and the energy of the incident radiation, it is necessary to decide how much variation in dose one is willing to tolerate as the radiation penetrates the specimen. The most commonly used irradiation facilities have radiation sources in the energy range of 0,5 MeV to 1,5 MeV. If, for a point source, one arbitrarily sets a limit of 25 % for the difference between the absorbed dose at the front and rear of the specimen (25 % attenuation through the specimen), then the specimen thickness is limited to 2,8 cm for 0,5 MeV and 5,0 cm for 1,5 MeV radiation, assuming no build-up, a specimen of unit density equal to 1 g/cm³ and unidirectional radiation (see Clause A.1). For other source geometries (e.g. slab sources) these thicknesses will be significantly different.

9 Monitoring of the irradiation

Irradiation testing of insulating materials can require long term testing under low dose rate conditions that are best carried out in a gamma irradiation facility. The decay of the radionuclide used in the sources shall be taken into account when performing such tests. It is recommended that dose rate mapping of gamma facilities (as described in Clause 7) be carried out at intervals not exceeding the half-life of the radionuclide in use.

In electron beam and X-ray facilities, the electron beam characteristics shall be monitored during irradiation and any interruptions or changes in characteristics shall be recorded, together with any actions taken.

Annex A (informative)

Radiation chemical aspects in interaction and dosimetry

A.1 Charged-particle equilibrium thickness

Whenever a material is irradiated from one side only with X- or γ -rays uncontaminated with secondary electrons, there is initially (in the first absorber) a build-up of energy deposition (absorbed dose) as the radiation penetrates the material. After some finite thickness, the radiation-energy deposition decreases. The thickness necessary to reach the maximum energy deposition is commonly called the charged-particle equilibrium thickness, and is a function of the radiation energy and the electron density of the material being irradiated. For larger thicknesses there is charged-particle equilibrium in the material.

Figure A.1 is a typical plot of energy deposition as a function of thickness. Whenever one is irradiating a specimen from all sides, it is necessary to surround the specimen with an absorber in order to ensure charged-particle equilibrium throughout the specimen. In practical cases with highly scattered radiation, build-up is not observed. It is recommended however to use built-up layers in order to provide well-defined irradiation conditions.

Figure A.2 is a plot of absorber thickness as a function of energy for material of electron density 3.3×10^{23} cm⁻³. Figure A.3 is a plot of thickness of water (or a material of the same electron density) as a function of energy for a given attenuation of unidirectional irradiation.

NOTE The electron density n of any material can be evaluated from:

$$n = \rho \frac{N_A}{M} \sum_i Z_i \text{ [cm}^{-3}] = \frac{N_A}{M} \sum_i Z_i \text{ [g}^{-1}]$$
 (A.1)

where

 ρ is the density of material (g/cm³);

 N_{Δ} is the 6,023 × 10²³ mol⁻¹, Avogadro's constant;

M is the molar mass (g/mol);

 Z_i is the atomic number of element i;

 $\Sigma_i Z_i$ is the total number of electrons per molecule.

Since 1/M ($\Sigma_i Z_i$) is about 1/2 for elements up to Z=17 (excluding H), for organic materials, this equation can be simplified to read:

$$n = \rho \frac{N_A}{2} 3 \times 10^{23} \ \rho \ [\text{cm}^{-3}] = 3 \times 10^{23} \ [\text{g}^{-1}]$$
 (A.2)

Figures A.1 to A.3 are calculated with this approximation.

The curve in Figure A.2 will shift to the left as the electron density of the absorber increases above this calculated value and to the right for materials of lesser electron density. Therefore, an equivalent thickness to use is that obtained from Figure A.2 divided by the ratio of the electron density of the absorber to 3.3×10^{23} cm⁻³ (electron density of water).

For example, it is required to irradiate polytetrafluoroethylene (PTFE) film with 1,1 MeV photons. Referring to Figure A.2, it is noted that 0,5 cm of material of electron density of

 3.3×10^{23} cm⁻³ is needed to ensure charged-particle equilibrium. Therefore, this thickness of water shall surround the film.

For PTFE with a density of ρ = 2,2 g/cm³ one calculates:

$$\frac{n_{\text{PTFE}}}{n_{\text{H}_2\text{O}}} = \frac{3 \times 10^{23} \times 2.2}{3.3 \times 10^{23}} = 2 \tag{A.3}$$

and

$$d_{\text{PTFE}} = \frac{d_{\text{H}_2\text{O}}}{n_{\text{PTFE}}/n_{\text{H}_2\text{O}}} = 0.25 \text{ cm}$$
 (A.4)

This means that 0,25 cm of PTFE has to surround the film.

Figure A.3 is a plot of thickness as a function of energy of a sample for 10 % and 25 % attenuation through a specimen of unit density equal to 1 g/cm³ for unidirectional radiation. The curves will shift to the left for higher-density material and to the right for lower-density material. The accurate thickness for 10 % or 25 % attenuation in the specimen will be the value obtained from Figure A.3 divided by the ratio of the electron density of the specimen to $3.3 \times 10^{23} \ g^{-1}$. Since the curves are calculated on the basis of attenuation only, and build-up in the thicker specimens is neglected, the curves represent a maximum attenuation for a given energy and thickness, and for unidirectional radiation. Non-unidirectional radiation results in larger attenuation.

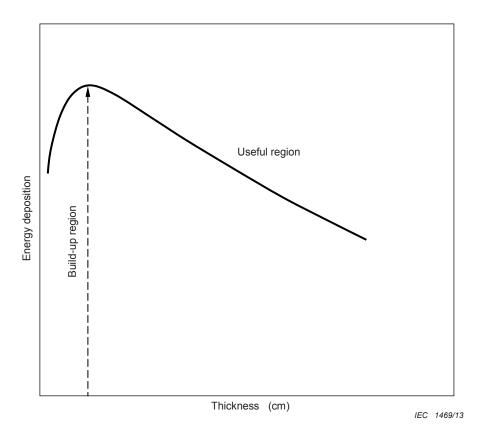


Figure A.1 - Absorbed dose as a function of thickness

The part of the curve to the left of the maximum is not well known; therefore, it is necessary to have a sample of sufficient thickness to fall either at or to the right of the maximum.

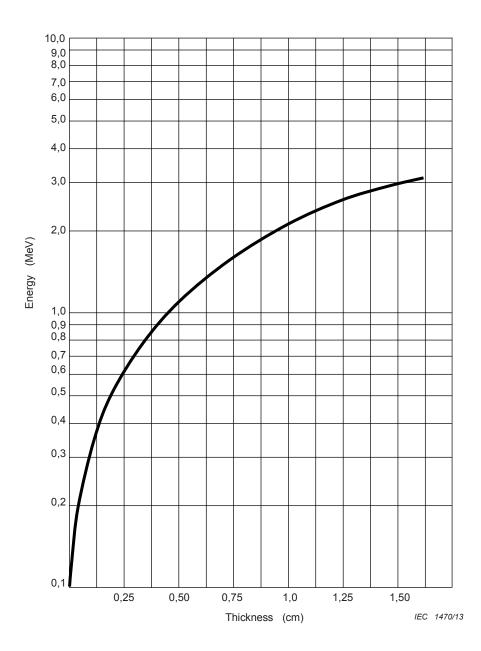


Figure A.2 – Absorber thickness for charged-particle equilibrium as a function of energy for a material with an electron density of $3.3 \times 10^{23}~\text{cm}^{-3}$ (water)

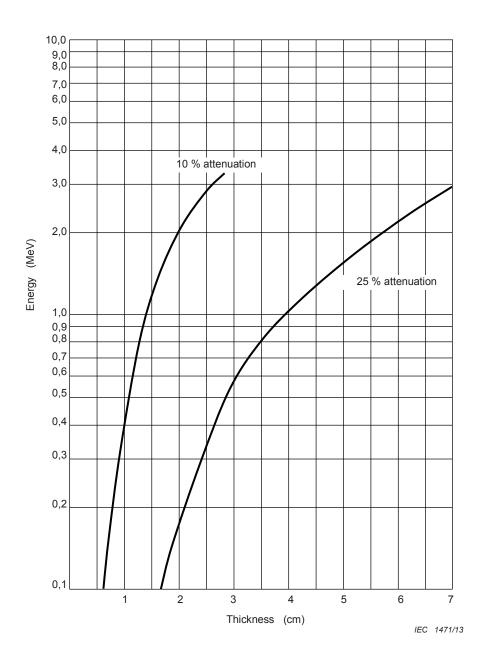


Figure A.3 – Thickness of water (1 g/cm³) as a function of photon energy for a given attenuation of unidirectional X-ray or γ -ray radiation

A.2 Calculation of absorbed dose

For irradiations using a photon source, the dosimeter may be considered as a cavity in the material interest and the interpretation of absorbed dose in materials is as follows:

If the sensitive region of the dosimeter is very thin compared to the range of the highest energy secondary electrons, then most of the energy deposited in the dosimeter and in the material surrounding it results from secondary electrons produced outside the dosimeter (that is, in the equilibrium layer of material). Thus, the absorbed dose in the material, $D_{\rm m}$ is given by:

$$D_{\rm m} = (S/\rho)_{\rm m} / (S/\rho)_{\rm d} \times D_{\rm d}$$
 (A.5)

 $(S/\rho)_m$ and $(S/\rho)_d$ is the mass collision stopping power for the surrounding material and dosimeter, respectively;

 D_{d} is the absorbed dose in the dosimeter.

Values of mass collision stopping powers are given in Table A.1.

If the sensitive region of the dosimeter has a thickness much greater than the range of the highest energy secondary electrons, then most of the energy deposited in it results from the secondary electrons produced within the dosimeter itself. Thus, the absorbed dose in the material is given by:

$$D_{\rm m} = (\mu_{\rm en}/\rho)_{\rm m} / (\mu_{\rm en}/\rho)_{\rm d} \times D_{\rm d}$$
 (A.6)

where

 $(\mu_{\rm en}/\rho)_{\rm m}$ and $(\mu_{\rm en}/\rho)_{\rm d}$ are the mass energy-absorption coefficients for the material and dosimeter, respectively.

Values of mass energy absorption coefficient are given in Table A.2.

If the sensitive region of the dosimeter has a thickness between the two limits discussed above then the equations may be combined with appropriate weighing factors to reflect the relative contribution of each term.

The collision stopping powers and energy absorption coefficients are energy dependent. Ratios of mass energy absorption coefficients can be plotted. Similarly, ratios of mass collision stopping powers can be plotted.

NOTE Such tables and figures are available, for example, at the following web sites: Stopping power (electrons): http://physics.nist.gov/PhysRefData/Star/Text/ESTAR.html Energy absorption (x-ray): http://srdata.nist.gov/gateway/gateway/keyword=mass+energy+absorption+coefficient.

For a photon energy spectrum including energies down to 50 keV, the ratios of mass energy absorption coefficient are essentially equal to unity for most polymer materials compared to water.

A correction for radiation energy dependence may be necessary in the case of materials having photon absorption properties greatly different from water (such as bone or metal). If the photon energy spectrum at the point of interest has a significant component below 0,2 MeV and if the spectrum is known, then a more accurate absorbed dose value can be obtained by integration of Equation (A.6) over the entire photon energy spectrum.

For irradiations using an electron source, the absorbed dose in the material can be interpreted as follows:

At any given depth in a material of interest, the dose may be determined with a dosimeter that is thin compared to the range of the incident electrons. The absorbed dose may be calculated using Equation (A.5). However, all of the following conditions are necessary:

- a) the depth in the material shall be less than the incident electron range;
- b) the ratio of mass collision stopping powers (material/dosimeter) shall be essentially constant:
- c) the degraded electrons at a given depth shall have sufficient energy to traverse the dosimeter thickness.

Therefore, for materials considered in this standard, Equation (A.5) is valid for electron energies from about 0,05 MeV to 0,1 MeV.

For most cases, this method of absorbed dose interpretation requires an incident beam energy of at least 0,5 MeV. However, energies down to 0,1 MeV may be used for surface irradiation products. Measurements of absorbed dose in a material at such low incident energies may be difficult to interpret.

Table A.1 – Electron mass collision stopping powers, S/ρ (MeV cm²/g)

Electron energy MeV	Air	Water	Polystyrene	LiF
0,01	19,75	22,56	22,23	17,96
0,02	11,57	13,17	12,96	10,55
0,04	6,848	7,777	7,637	6,252
0,06	5,111	5,797	5,688	4,670
0,08	4,198	4,757	4,666	3,838
0,1	3,633	4,115	4,034	3,323
0,2	2,470	2,793	2,735	2,261
0,4	1,902	2,145	2,101	1,737
0,6	1,743	1,956	1,911	1,583
0,8	1,683	1,879	1,832	1,521
1	1,661	1,844	1,794	1,491
2	1,684	1,825	1,768	1,474
4	1,790	1,877	1,816	1,513
6	1,870	1,919	1,859	1,547
8	1,931	1,951	1,891	1,572
10	1,979	1,976	1,916	1,592
20	2,134	2,051	1,989	1,654
40	2,282	2,120	2,053	1,711
60	2,347	2,157	2,089	1,742

Table A.2 – Photon mass energy absorption coefficients, $\mu_{\rm en}$ $I\rho$ (cm²/g)

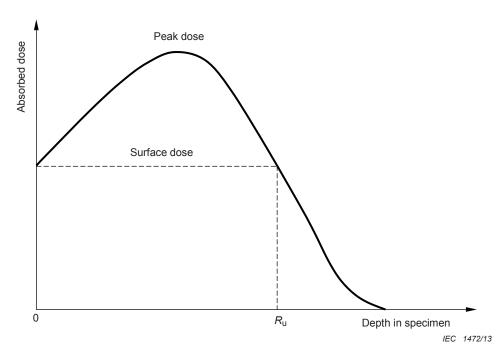
Photon energy MeV	Air	Water	Polystyrene	LiF
0,01	4,742	4,944	1,918	5,733
0,02	0,538 9	0,550 3	0,207 5	0,649 4
0,04	0,068 33	0,069 47	0,032 64	0,078 90
0,06	0,030 41	0,031 90	0,021 72	0,032 23
0,08	0,024 07	0,025 97	0,021 60	0,023 85
0,1	0,023 25	0,025 46	0,022 96	0,022 29
0,2	0,026 72	0,031 92	0,028 57	0,024 84
0,4	0,029 49	0,032 79	0,031 75	0,027 34
0,6	0,029 53	0,032 84	0,031 82	0,027 36
0,8	0,028 82	0,032 06	0,031 06	0,026 71
1	0,027 89	0,031 03	0,030 06	0,025 85
2	0,023 45	0,026 08	0,025 24	0,021 73
4	0,018 70	0,020 66	0,019 79	0,017 33
6	0,016 47	0,018 06	0,017 08	0,015 28
8	0,015 25	0,016 58	0,015 50	0,014 14
10	0,014 50	0,015 66	0,014 48	0,013 45
20	0,013 11	0,013 82	0,012 32	0,012 11
40	0,012 62	0,012 98	0,011 28	0,011 54
60	0,012 42	0,012 61	0,010 86	0,011 23

A.3 Depth-dose distribution for electron beam irradiation

Recently, many calculation codes have become available to estimate energy deposition of electrons in materials as a function of depth. The examples include free or commercial software such as ED MULTI, EGS 5 (Electron gamma shower, version 5), PENELOPE (penetration and energy loss of positron and electron) etc. By using these codes, depth-dose distribution curves such as those shown in Figures A.4 to Figure A.6 would be obtained. Comparisons between simulation and experimental results (with stack of thin film, as indicated Figures A.7 and Figure A.8) show reasonably sufficient agreement. These codes would be useful to design irradiation conditions.

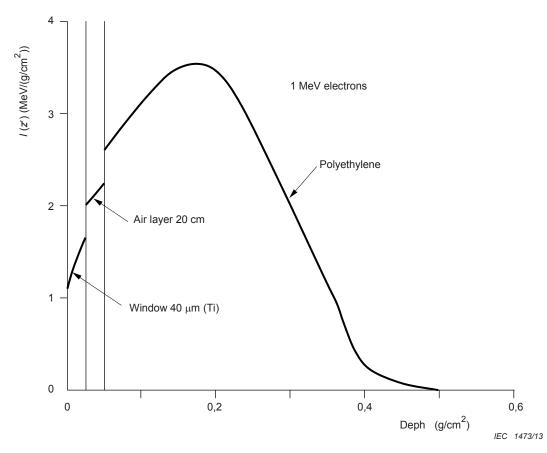
NOTE 1 ED MULTI and PENELOPE are OECD (Organisation for Economic Cooperation and Development)/NEA (Nuclear Energy Agency) codes, available through: http://www.oecd-nea.org/dbprog/ .

NOTE 2 EGS 5 is a free software, see URL at http://rcwww.kek.jp/research/egs/egs5.html



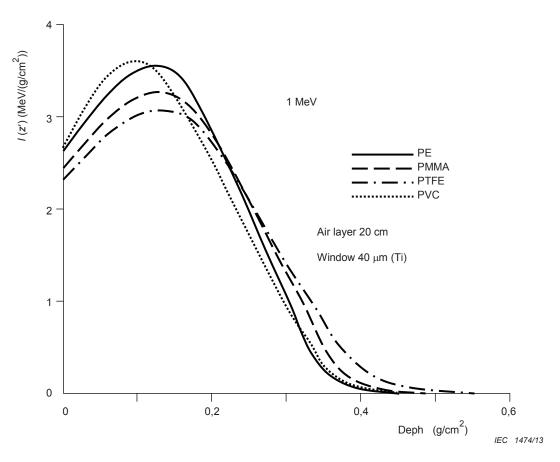
NOTE Acceleration voltage > 0.5 MV, R_u = useful range.

Figure A.4 – Typical depth-dose distribution in a homogeneous material obtained with electron accelerators for radiation processing



NOTE z': total depth in three-layer absorber consisting of beam window, air gap, and polyethylene layer.

Figure A.5 – Example of calculated results of energy deposition function, I(z'), for a slab layer of polyethylene exposed to 1 MeV electron



NOTE z': total depth in three-layer absorber consisting of beam window, air gap and insulating material layer.

Figure A.6 – Example of calculated results of energy deposition function, I(z'), for typical organic insulators exposed to 1 MeV electron

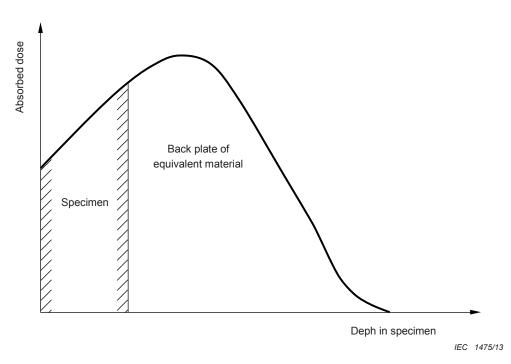


Figure A.7a) - Specimen and a backplate of the equivalent material

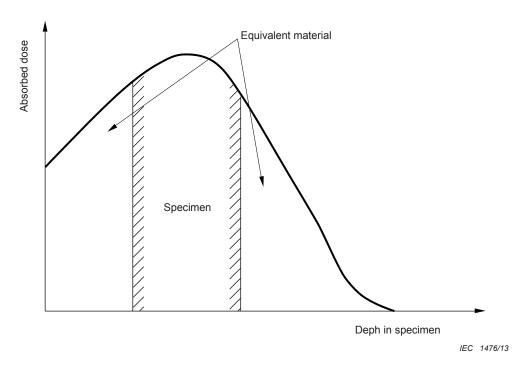
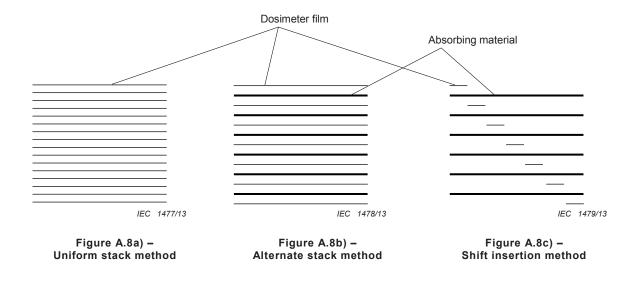


Figure A.7b) - Specimen sandwiched between two plates of the equivalent material

Figure A.7 – Two methods of arranging the irradiation samples in order to take into account the typical depth-dose distributions



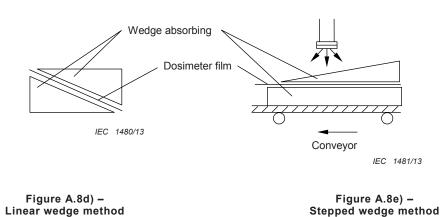


Figure A.8 – Methods of arranging the irradiation samples for measuring electron depth-dose distributions with a stack of slab insulating materials and wedge-shape insulating materials

A.4 Fundamental aspect of radiation chemistry

A.4.1 Radiation and materials

The definition of radiation is, typically, electro-magnetic waves or corpuscular beams that can ionise material (in a typical case, air), that is, eject an electron.

Examples of radiation include

- a) alpha-ray (nucleus of helium He from radioisotope),
- b) beta-ray (electron from radio-isotope),
- c) gamma-ray (electro-magnetic wave resulting from transition between energy levels of a nucleus),
- d) X-ray (electro-magnetic wave resulting from transition between energy levels of an atom),
- e) neutron beam,
- f) electron beam (accelerated with a particle-accelerator),

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g) ion beam (such as proton, helium and other heavier nuclei accelerated with a particle-accelerator), etc.

Among them, from the viewpoint of current radiation resistance test, the most widely used radiations are gamma-rays and electron beams.

A.4.2 Interactions of radiation with materials

Interaction of radiation with materials depends on the energy of radiation; the interaction scheme varies from implantation (when the energy of incident electro-magnetic wave/particle is low), sputtering, excitation, ionization and nuclear reaction (when the energy of incident radiation is sufficiently high). In radiation chemical degradation, ionization and excitation are most fundamental events. In the case of Co-60 gamma ray irradiation, secondary electrons are ejected through the Compton effect, and these secondary electrons can interact with the material. In the case of electron beam irradiation, the incident electrons can interact with the material directly. High energy (incident and secondary) electrons interact directly or indirectly with electrons (of the irradiated material) through Coulomb interaction, and results in ionization and excitation. Ionized or excited species can form radicals (chemically reactive atoms or molecules having an unpaired electron) and the radicals induce subsequent reactions. The analysing technique is described in A.4.3. In the case of polymer, the final events are cross-linking (new bond formation between two polymer chains), scission (fragmentation into two or more smaller pieces), un-saturation (formation of double bond. other functional bonds, etc.) and graft-polymerization. With respect to radiation resistance test, cross-linking and scission are of primary interest. This scheme is illustrated in Figure A.9.

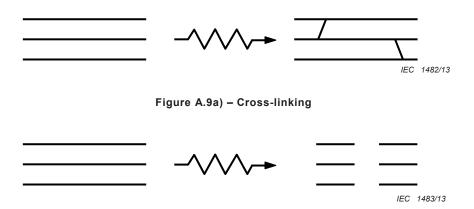


Figure A.9b) - Degradation

Figure A.9 - Scheme of radiation effects of polymers

NOTE Some concerns may exist on the effect of beta-rays relative to gamma rays, especially in the case of accidents. However, since the beta rays are electrons having energies in the range of keV - MeV, the effect can be sufficiently represented by electron beams.

A.4.3 Techniques to analyse intermediate short-lived species

A.4.3.1 ESR (electron spin resonance)

Polymers undergo radical formation upon irradiation. If radicals, having an un-paired electron, are placed in an electro-magnetic field, their energy levels split in two stages of higher and lower energy, known as the Zeeman effect. This splitting width is proportional to the magnetic field applied. If the sample is subjected to microwaves and the magnetic field is scanned, the resonant condition is satisfied as follows:

$$h \times v = g \times \beta \times H$$

where

h is Planck's constant $(6,6 \times 10^{-34} \text{ Js})$;

- v is the frequency of microwave (in s⁻¹);
- g is the g-factor of the radical;
- β is Bohr's magneton (9,3 × 10⁻²⁴ J/T; Tesla);
- H is the magnetic field (in T), respectively.

At this resonant condition, the microwave is absorbed by radicals to transit from lower energy level towards a higher one. Depending on the structure of the radical, especially the presence and distance of H and/or C atoms having nuclear spin, fine and/or super-fine structure are often observed and can be indicative of specific chemical structure. Interpretation of the absorption spectra and assignments of radicals are sometimes not an easy task. Stability of radicals is evaluated through annealing at a fixed temperature as a function of storage time or at a fixed storage time with raising the temperature.

A.4.3.2 Laser flash photolysis and pulse radiolysis

Laser flash photolysis uses eximer laser (excited dimer laser, such as from Ar-F or Kr-F) that provides pulsed laser light and pulse radiolysis uses a linear electron accelerator (linac, typically in MeV) that generates a pulsed electron beam. Analysing light from Xe flash lamp, it generates pulsed light in the wide wavelength of $200-900\,\mathrm{nm}$, but pulse width is much longer than that of subsequent pulsed laser light or electron; Xe light can be presumed as stable. When pulsed laser or electron beam are applied to the samples as a solution in a quartz glass cell, it causes ionization and excitation within a very short time, and subsequent diffusion and chemical reaction takes place. This technique focuses on a rather shorter time region than the ESR.

The intermediates absorb analysing light according to Lambert-Beer's law: $\log_{10}(I_0/I)$ (defined as absorbance or optical density) = $\varepsilon C(t)\ell$

where

- I_0 is the intensity of the incident analysing light;
- *I* is the intensity of out-coming light through the cell;
- is the molar absorption coefficient (in ℓ mol⁻¹cm⁻¹) unique to the chemicals and function of wavelength;
- ℓ is the length of the glass cell (in cm).

By scanning the wavelength, the absorption band specific to intermediate species of interest can be found. Through observing the change of absorbance at the wavelength as a function of time, the change in concentration, and therefore life-time or reactivity, as well as the rate constant with other chemicals or additives, if any, can be evaluated. Time resolution, according to the state-of-the-art is in the order of femto (10⁻¹⁵) s or faster, from the moment of energy deposition from radiation to material.

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