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Guide for dosimetry in radiation research on food and agricultural products

*Guide de la dosimétrie pour la recherche dans le domaine de
l'irradiation des produits alimentaires et agricoles*



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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75% of the member bodies casting a vote.

ASTM International is one of the world's largest voluntary standards development organizations with global participation from affected stakeholders. ASTM technical committees follow rigorous due process balloting procedures.

A project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this project, ASTM Subcommittee E10.01, Radiation Processing: Dosimetry and Applications, is responsible for the development and maintenance of these dosimetry standards with unrestricted participation and input from appropriate ISO member bodies.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. Neither ISO nor ASTM International shall be held responsible for identifying any or all such patent rights.

International Standard ISO/ASTM 51900 was developed by ASTM Committee E10, Nuclear Technology and Applications, through Subcommittee E10.01, and by Technical Committee ISO/TC 85, Nuclear energy.

This second edition cancels and replaces the first edition (ISO/ASTM 51900:2002), which has been technically revised.



Standard Guide for Dosimetry in Radiation Research on Food and Agricultural Products¹

This standard is issued under the fixed designation ISO/ASTM 51900; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision.

1. Scope

1.1 This guide covers the minimum requirements for dosimetry needed to conduct research on the effect of radiation on food and agricultural products. Such research includes establishment of the quantitative relationship between absorbed dose and the relevant effects in these products. This guide also describes the overall need for dosimetry in such research, and in reporting of the results. Dosimetry must be considered as an integral part of the experiment.

NOTE 1—The Codex Alimentarius Commission has developed an international General Standard and a Code of Practice that address the application of ionizing radiation to the treatment of foods and that strongly emphasize the role of dosimetry for ensuring that irradiation will be properly performed (1).²

NOTE 2—This guide includes tutorial information in the form of Notes. Researchers should also refer to the references provided at the end of the standard, and other applicable scientific literature, to assist in the experimental methodology as applied to dosimetry (2-10).

1.2 This guide covers research conducted using the following types of ionizing radiation: gamma radiation, X-ray (bremsstrahlung), and electron beams.

1.3 This guide describes dosimetry requirements for establishing the experimental method and for routine experiments. It does not include dosimetry requirements for installation qualification or operational qualification of the irradiation facility. These subjects are treated in ISO/ASTM Practices 51204, 51431, 51608, 51649, and 51702.

1.4 This guide is not intended to limit the flexibility of the experimenter in the determination of the experimental methodology. The purpose of the guide is to ensure that the radiation source and experimental methodology are chosen such that the results of the experiment will be useful and understandable to other scientists and regulatory agencies.

1.5 The overall uncertainty in the absorbed-dose measurement and the inherent absorbed-dose variation within the irradiated sample should be taken into account (see ISO/ASTM Guide 51707).

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced documents

2.1 ASTM Standards:³

E 170 Terminology Relating to Radiation Measurements and Dosimetry

E 925 Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Slit Width does not Exceed 2 nm

E 1026 Practice for Using the Fricke Reference-Standard Dosimetry System

E 2232 Guide for Selection and Use of Mathematical Methods for Calculating Absorbed Dose in Radiation Processing Applications

E 2303 Guide for Absorbed-Dose Mapping in Radiation Processing Facilities

E 2304 Practice for Use of a LiF Photo-Fluorescent Film Dosimetry System

E 2381 Guide for Dosimetry In Radiation Processing of Fluidized Beds and Fluid Streams

F 1355 Guide for Irradiation of Fresh Agricultural Produce as a Phytosanitary Treatment

F 1356 Practice for Irradiation of Fresh and Frozen Red Meat and Poultry to Control Pathogens and Other Microorganisms

F 1640 Guide for Selection and Use of Packaging Materials for Foods to Be Irradiated

F 1736 Guide for Irradiation of Finfish and Aquatic Invertebrates Used as Food to Control Pathogens and Spoilage Microorganisms

F 1885 Guide for Irradiation of Dried Spices, Herbs, and Vegetable Seasonings to Control Pathogens and Other Microorganisms

2.2 ISO/ASTM Standards:³

51204 Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing

51205 Practice for Use of a Ceric-Cerous Sulfate Dosimetry System

¹ This guide is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.01 on Radiation Processing: Dosimetry and Applications, and is also under the jurisdiction of ISO/TC 85/WG 3.

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² The boldface numbers in parentheses refer to the bibliography at the end of this guide.

³ For referenced ASTM and ISO/ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.



- 51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing
- 51275 Practice for Use of a Radiochromic Film Dosimetry System
- 51276 Practice for Use of a Polymethylmethacrylate Dosimetry System
- 51310 Practice for Use of a Radiochromic Optical Waveguide Dosimetry System
- 51431 Practice for Dosimetry in Electron Beam and X-ray (Bremsstrahlung) Irradiation Facilities for Food Processing
- 51538 Practice for Use of the Ethanol-Chlorobenzene Dosimetry System
- 51540 Practice for Use of a Radiochromic Liquid Dosimetry System
- 51607 Practice for Use of the Alanine-EPR Dosimetry System
- 51608 Practice for Dosimetry in an X-ray (Bremsstrahlung) Facility for Radiation Processing
- 51649 Practice for Dosimetry in Electron Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV
- 51650 Practice for Use of Cellulose Triacetate Dosimetry Systems
- 51702 Practice for Dosimetry in a Gamma Irradiation Facility for Radiation Processing
- 51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing
- 51818 Guide for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies Between 80 and 300 keV
- 51956 Practice for Use of Thermoluminescence Dosimetry (TLD) Systems for Radiation Processing
- 52116 Practice for Dosimetry for a Self-Contained Dry Storage Gamma Irradiator

2.3 International Commission on Radiation Units and Measurements (ICRU) Reports:⁴

ICRU 60 Fundamental Quantities and Units for Ionizing Radiation

2.4 NPL Report:

CIRM 29 : Guidelines for Calibration of Dosimeters for Use in Radiation Processing, Sharpe, P., and Miller, A., August, 1999

3. Terminology

3.1 Definitions:

3.1.1 *absorbed dose (D)*—quantity of ionizing radiation energy imparted per unit mass of a specified material. The SI unit of absorbed dose is the gray (Gy), where 1 gray is equivalent to the absorption of 1 joule per kilogram of the specified material (1 Gy = 1 J/kg). The mathematical relationship is the quotient of $d\bar{\epsilon}$ by dm , where $d\bar{\epsilon}$ is the mean incremental energy imparted by ionizing radiation to matter of incremental mass dm .

$$D = d\bar{\epsilon}/dm \quad (1)$$

3.1.1.1 *Discussion*—The discontinued unit for absorbed dose is the rad (1 rad = 100 erg/g = 0.01 Gy). Absorbed dose is sometimes referred to simply as dose.

3.1.2 *absorbed-dose mapping*—measurement of absorbed dose within an irradiated product to produce a one-, two- or three-dimensional distribution of absorbed dose, thus rendering a map of absorbed-dose values.

3.1.3 *absorbed-dose rate $\dot{D}$* —absorbed dose in a material per incremental time interval, that is, the quotient of dD by dt (see ICRU 60).

$$\dot{D} = dD/dt \quad (2)$$

Unit: $\text{Gy} \cdot \text{s}^{-1}$

3.1.4 *accredited dosimetry calibration laboratory*—dosimetry laboratory with formal recognition by an accrediting organization that the dosimetry laboratory is competent to carry out specific activities which lead to the calibration or calibration verification of dosimetry systems in accordance with documented requirements of the accrediting organization.

3.1.5 *bremsstrahlung*—broad-spectrum electromagnetic radiation emitted when an energetic charge particle is influenced by a strong electric or magnetic field, such as that in the vicinity of an atomic nucleus.

3.1.6 *charged-particle equilibrium*—condition in which the kinetic energy of charged particles, excluding rest mass, entering an infinitesimal volume of the irradiated material equals the kinetic energy of charged particles emerging from it.

3.1.7 *dose uniformity ratio*—ratio of the maximum to the minimum absorbed dose within the irradiated product.

3.1.8 *dosimeter*—device that, when irradiated, exhibits a quantifiable change that can be related to absorbed dose in a given material using appropriate measurement instruments and procedures.

3.1.9 *dosimeter response*—reproducible, quantifiable radiation effect produced in the dosimeter by a given absorbed dose.

3.1.10 *dosimetry system*—system used for determining absorbed dose, consisting of dosimeters, measurement instruments and their associated reference standards, and procedures for the system's use.

3.1.11 *electron equilibrium*—charged-particle equilibrium when the charged particles are electrons set in motion by photons irradiating the material. See charged-particle equilibrium.

3.1.12 *reference-standard dosimeter*—dosimeter of high metrological quality, used as a standard to provide measurements traceable to measurements made using primary-standard dosimeters.

3.1.13 *repeatability (of results of measurements)*—closeness of the agreement between the results of successive measurements of the same measurand carried out subject to all of the following conditions; the same measurement procedure, the same observer, the same measuring instrument, used under the same conditions, the same location, and repetition over a short period of time.

⁴ Available from the International Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814 USA.



3.1.13.1 *Discussion*—These conditions are called “repeatability conditions.” Repeatability may be expressed quantitatively in terms of the dispersion characteristics of the results.

3.1.14 *reproducibility (of results of measurements)*—closeness of agreement between the results of measurements of the same measurand, where the measurements are carried out under changed conditions such as differing: principle or method of measurement, observer, measuring instrument, location, conditions of use, and time.

3.1.14.1 *Discussion*—A valid statement of reproducibility requires specification of the conditions that were changed for the measurements. Reproducibility may be expressed quantitatively in terms of the dispersion characteristics of the results. In this context, results of measurement are understood to be corrected results.

3.1.15 *routine dosimeter*—dosimeter calibrated against a primary-, reference-, or transfer-standard dosimeter and used for routine absorbed-dose measurement.

3.1.16 *simulated product*—material with radiation attenuation and scattering properties similar to those of the product, material or substance to be irradiated.

3.1.17 *traceability*—property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

3.1.18 *transfer-standard dosimeter*—dosimeter, often a reference-standard dosimeter, suitable for transport between different locations, used to compare absorbed-dose measurements.

3.1.19 *transit dose*—absorbed dose delivered to a product (or a dosimeter) while it travels between the non-irradiation position and the irradiation position, or in the case of a movable source while the source moves into and out of its irradiation position.

3.1.20 *uncertainty (of measurement)*—parameter associated with the result of a measurement, that characterizes the dispersion of the values that reasonably could be attributed to the measurand or derived quantity (see ISO/ASTM Guide 51707).

3.1.21 *X-radiation*—ionizing electromagnetic radiation, which includes both bremsstrahlung and the characteristic radiation emitted when atomic electrons make transitions to more tightly bound states.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *residual*—difference between the observed value and the value calculated by the regression model.

3.2.2 *target dose*—absorbed dose intended for the volume of interest within the irradiated sample.

NOTE 3—Definitions of other terms used in this standard that pertain to radiation measurement and dosimetry may be found in ASTM Terminology E 170. Definitions in Terminology E 170 are compatible with ICRU 60; that document, therefore, may be used as an alternative reference.

4. Significance and use

4.1 This guide is intended to provide direction on dosimetry for experiments in food and agricultural research, and on the reporting of dosimetry results. Research concerning the effec-

tiveness of irradiation of food and agricultural products to achieve a defined benefit involves very different absorbed-dose specifications from one study and one product to another. For example, the absorbed dose required to sterilize fruit flies is much lower than the doses required to inactivate some bacterial pathogens in meat, or to decontaminate spices.

NOTE 4—Examples of the relevant effects of irradiation include reduction of viable food-borne bacteria, viruses and parasites and phytosanitary treatment (such as disinfestation of fruits and vegetables), prevention of sprouting, delay of ripening, and changes in product chemistry and quality. Further discussion of these effects is outside the scope of this guide. Refer to ASTM Guides F 1355, F 1356, F 1736 and F 1885.

4.2 Proper reporting of the irradiation aspect is important since the degree of biological effect may be a function of various factors such as the radiation source, the absorbed-dose rate, energy of the incident radiation, environmental effects during irradiation, and the type of incident radiation. This guide attempts to highlight the information, including the methodology and results of the absorbed-dose measurements, necessary for an experiment to be repeatable by other researchers.

NOTE 5—Factors that may influence the response of agricultural products to ionizing radiation include genus, species, variety, vigor, life stage, initial quality, state of ripeness, temperature, moisture content, pH, packaging, shipping, and storage conditions. Although these factors are not discussed in this guide, they should be considered when planning experiments (see ASTM Guides F 1355, F 1356, F 1640, F 1736 and F 1885).

4.3 Ideally, an experiment should be designed to irradiate the sample as uniformly as possible. In practice, a certain variation in absorbed dose will exist throughout the sample. Absorbed-dose mapping is used to determine the magnitude, location, and reproducibility of the maximum ($D_{\max}$) and minimum absorbed dose ($D_{\min}$) for a given set of experimental parameters. Dosimeters used for dose mapping must be capable of responding to doses and dose gradients likely to occur within irradiated samples.

4.4 Theoretical calculations may provide useful information about absorbed-dose distribution in the irradiated sample, especially near material interfaces (see ASTM Guide E 2232).

5. Irradiation facilities and modes of operation

5.1 *Types of Facilities*—This guide covers the use of gamma radiation, X-ray (bremsstrahlung), and accelerated electrons used for studying the effects of ionizing radiation on food and agricultural products.

NOTE 6—Sections 5 and 6 give a brief overview of types of irradiation facilities and radiation source characteristics. Radiation source characteristics, the type of radiation produced, the energy of the photons or electrons, and the sizes and densities of the samples to be irradiated, will all be factors in determining how the incident radiation is absorbed in the experimental samples. Researchers unfamiliar with radiation source characteristics are strongly recommended to review appropriate reference materials before beginning experimentation (2-10).

5.2 *Self-Contained Dry Storage Gamma and X-ray (bremsstrahlung) Irradiators*—Much of the research currently being conducted on food and agricultural products is accomplished by using gamma radiation from either ^{137}Cs or ^{60}Co



self-contained irradiators or X-ray (bremsstrahlung) self-contained irradiators. These devices are self-shielded using lead (or other appropriate high atomic number material), and usually have a mechanism to move the sample container from the load to the irradiation position.

NOTE 7—Typically, self-contained dry storage gamma irradiators have a limited irradiation volume. This type of irradiator is classified as ANSI Category I, Self-contained Dry Storage Gamma Irradiators (11).

5.2.1 In self-contained gamma irradiators, a common approach is to distribute the source in an annular array, such that the absorbed dose is relatively uniform around the center where the sample is irradiated.

5.2.2 For gamma and X-ray units, another method is to rotate the sample container on an irradiator turntable within the radiation field to achieve a more uniform dose within the sample.

5.3 *Self-Contained Wet Storage Gamma Irradiators*—Irradiation of samples may also be carried out in a wet-storage gamma irradiator. In these facilities, the source is contained in a storage pool (usually containing water), which is shielded at all times. The samples to be irradiated are enclosed in a water-tight chamber and lowered into the water next to the radiation source.

NOTE 8—This type of irradiator is classified as ANSI Category III, Self-Contained Wet Source Storage Gamma Irradiators (12).

5.4 *Large-Scale Gamma Irradiation Facilities*—Gamma irradiation of research samples is also carried out in large-scale irradiators, either pool-type or dry-storage. In these facilities the source typically consists of a series of rods that contain ^{60}Co and can be raised or lowered into a large irradiation room. When retracted from the irradiation room, the source is shielded by water (pool-type), or an appropriate material of high atomic number (dry-storage), or both.

NOTE 9—These types of irradiators are classified as ANSI Category IV, Wet Source Storage Gamma Irradiators or ANSI Category II, Dry Source Storage Gamma Irradiators (13).

5.4.1 *Continuous Operation*—A common method of use is for the irradiation of sample containers to be carried on a conveyor in one or more revolutions around a central source in order to obtain a more uniform absorbed dose. The source is retracted from the irradiation room when the irradiator is not in use.

5.4.2 *Batch Operation*—An alternative approach is to place the sample containers in the irradiation room while the source is shielded, and move the source into the irradiation position for the time required to achieve the desired absorbed dose.

5.5 *Electron and X-ray (Bremsstrahlung) Facilities:*

5.5.1 *Electron Facility*—Radiation sources for electrons (with energies greater than 300 keV) are either direct action (potential-drop) or indirect-action (microwave-powered) accelerators. The radiation fields depend on the characteristics and the design of the accelerators. Included among these characteristics are the electron beam parameters, that is, the electron energy spectrum, average electron beam current and beam current distribution on the product surface.

5.5.1.1 Typically, accelerators produce a narrow beam of electrons that is diffused to cover the width of the conveyor, which is the location at which samples will be irradiated. Diffusion of the electron beam may be accomplished using a magnetic scanner (to sweep the beam back and forth rapidly), a magnetic defocusing lens, or scattering foils.

5.5.2 *X-ray (Bremsstrahlung) Facility*—An X-ray (bremsstrahlung) generator emits short-wavelength electromagnetic radiation, which is analogous to gamma radiation from radioactive isotopic sources. Although their effects on irradiated materials are generally similar, these kind of radiation differ in their energy spectra, angular distribution, and dose rates.

5.5.2.1 Electrons are accelerated towards a metal target or “converter” of high atomic number (typically tungsten or tantalum). The collision of the electrons with the target generates X-ray (bremsstrahlung) with a broad continuous energy spectrum).

5.5.3 *Sample Transport*—Samples are typically carried on a conveyor through the radiation field. Because of the narrow angular distribution of the radiation, use of conveyors to transport samples through the irradiation field, in contrast to use of static irradiation systems or shuffle-dwell systems will enhance the dose uniformity.

5.5.4 Refer to ISO/ASTM Practices 51431, 51608, and 51649 for more detailed information on electron and X-ray (bremsstrahlung) facilities and modes of operation.

6. Radiation source characteristics

6.1 *Gamma Irradiators:*

6.1.1 The radiation source used in the gamma facilities considered in this guide consists of sealed elements of ^{60}Co or ^{137}Cs that are typically linear rods arranged in one or more planar or cylindrical arrays.

6.1.2 Cobalt-60 emits photons with discrete energies of approximately 1.17 and 1.33 MeV in nearly equal proportions. Cesium-137 emits photons with energy of approximately 0.662 MeV (14).

6.1.3 The radioactive decay half-lives for ^{60}Co and ^{137}Cs are regularly reviewed and updated. A recent publication gave values of 1925.20 ± 0.25 days for ^{60}Co and 11018.3 ± 9.5 days for ^{137}Cs (15).

6.1.4 For gamma-ray sources, the only variation in the source output is the known reduction in the activity caused by radioactive decay. This reduction in the source activity, which necessitates an increase in the irradiation time to deliver the same dose, may be calculated or obtained from tables provided by the irradiator manufacturer (refer to ISO/ASTM Practice 51204).

NOTE 10—Errors in the decay calculation may be introduced by the existence of radioimpurities in the radiation source (for example, a small amount of ^{134}Cs present as an impurity in ^{137}Cs).

6.2 *Electron Accelerator (Electron and X-ray (Bremsstrahlung) Modes):*

6.2.1 For an electron accelerator, the principal beam characteristics are the electron energy spectrum, the beam current and where applicable the instantaneous (per pulse) current



together with pulse length and pulse repetition frequency (see ISO/ASTM Practices 51431, 51649 and 51818).

6.2.1.1 Direct-action electron accelerators employ direct current (dc) or pulsed high-voltage generators and typically produce electron energies up to 5 MeV.

6.2.1.2 Indirect-action electron accelerators use microwave or very high frequency (VHF) alternating current (ac) to produce electron energies typically from 3 MeV to 15 MeV.

6.2.2 For an X-ray (bremsstrahlung) facility, besides beam characteristics noted in 6.2.1, X-ray target design is a critical parameter. Although the X-ray (bremsstrahlung) is similar to gamma radiation from the radionuclides ^{60}Co or ^{137}Cs and thus their effects on materials are generally similar, these kinds of radiation differ in their energy spectra, angular distributions, and absorbed-dose rates. The continuous energy spectrum of the X-ray (bremsstrahlung) extends up to the maximum energy of the electrons incident on the X-ray target (see ISO/ASTM Practice 51608). In some cases, spectrum filtration is used to reduce the low energy component of the radiation, so as to improve dose uniformity.

NOTE 11—In some countries, regulations may limit the maximum energy for electrons or X-ray (bremsstrahlung) used to irradiate food for human consumption.

7. Dosimetry systems

7.1 Dosimetry systems are used to determine absorbed dose, usually in terms of absorbed dose to water. They consist of dosimeters, measurement instruments and their associated reference standards, and procedures for the system's use.

7.2 Dosimeters may be divided into four basic classes according to their relative quality and areas of application: primary-standard, reference-standard, transfer-standard, and routine dosimeters. ISO/ASTM Guide 51261 provides information about the selection of dosimetry systems for different applications. Most research will be conducted using routine dosimeters and transfer-standard dosimeters.

7.2.1 *Primary-Standard Dosimeters*—Primary-standard dosimeters are established and maintained by national standards laboratories for calibration of radiation environments (fields) and other classes of dosimeters. The two most commonly used primary-standard dosimeters are ionization chambers and calorimeters.

7.2.2 *Reference-Standard Dosimeters*—Reference-standard dosimeters are used to calibrate radiation environments and

routine dosimeters. Examples of reference-standard dosimeters, along with their useful dose ranges, are listed in ISO/ASTM Guide 51261.

7.2.3 *Transfer-Standard Dosimeters*—Transfer-standard dosimeters are specifically selected dosimeters used for transferring absorbed-dose information from an accredited or national standards laboratory to an irradiation facility in order to establish traceability for that facility. These dosimeters should be carefully used under conditions that are specified by the issuing laboratory. Transfer-standard dosimeters may be selected from either reference-standard dosimeters or routine dosimeters taking into consideration the criteria listed in ISO/ASTM Guide 51261.

7.2.4 *Routine Dosimeters*—Routine dosimeters may be used for process control and dose mapping. Proper dosimetric techniques, including calibration, should be employed to ensure that measurements are reliable and accurate. Examples of routine dosimeters, along with their useful dose ranges, are listed in Table 1 and ISO/ASTM Guide 51261.

7.3 Select a routine dosimetry system suitable for the research taking into consideration those parameters associated with the radiation source and experimental set-up that may influence dosimeter response (for example, absorbed-dose range, radiation source, dose rate, energy, and environmental conditions, such as temperature, humidity and light) and the uncertainty associated with it. More detailed selection criteria are listed in ISO/ASTM Guide 51261. For electron accelerator applications, it is also essential to consider the influences of absorbed-dose rate (average and peak dose rate for pulsed accelerators), pulse rate and pulse width (if applicable) when selecting a dosimeter.

7.3.1 For use, handling, storage, special precautions, calibration, etc., for a specific routine dosimetry system, an ASTM or ISO/ASTM standard and manufacturers recommendations shall be used.

7.4 Prior to use, a dosimetry system shall be calibrated. The calibration curve supplied by the dosimeter supplier should be considered as general information and should not be used without further verification of its applicability. The calibration of the existing lot of dosimeters should be checked at regular intervals as appropriate. This check could take the form of a calibration verification exercise.

7.4.1 Calibration verification can be performed by irradiating sets of dosimeters at each of three dose points (spanning the

TABLE 1 Examples of routine dosimeters (see ISO/ASTM Guide 51261)

Dosimeter	Measurement Instrument	Useful Absorbed Dose Range (Gy)	References
Alanine	EPR spectrometer	1 to 10^5	ISO/ASTM 51607
Polymethylmethacrylate	UV/Visible spectrophotometer	10^2 to 10^5	ISO/ASTM 51276
Cellulose triacetate	Spectrophotometer	5×10^3 to 3×10^5	ISO/ASTM 51650
Thermoluminescence (TLD)	Thermoluminescence reader	1 to 10^5	ISO/ASTM 51956
Lithium Fluoride Film	Fluorimeter	50 to 3×10^5	ASTM E 2304
Radiochromic dye films, solutions, optical wave guide	Visible spectrophotometer	1 to 10^5	ISO/ASTM 51275 ISO/ASTM 51540 ISO/ASTM 51310
Ceric cerous sulfate solution	Potentiometer or UV spectrophotometer	5×10^2 to 5×10^4	ISO/ASTM 51205
Fricke solution	UV spectrophotometer	20 to 4×10^2	ASTM E 1026
Ethanol chlorobenzene solution	Spectrophotometer, color titration, high frequency conductivity	10^1 to 2×10^6	ISO/ASTM 51538
MOSFET	Electronic Reader	1 to 2×10^2	(16)



calibration range) using the same dosimeter holder as used for calibration irradiation and at the reference location where the dose rate is known. These dose values are then compared with the values calculated by using the response values of the irradiated dosimeters and the current response function. Agreement at the three points within the uncertainty of the system confirms the validity of the response function.

7.4.2 Measurement Instruments—The measurement instrument is an integral part of the dosimetry system, thus, the calibration of a dosimetry system should be regarded as being specific to a particular instrument. Calibration that is established with one instrument is not valid for another one. The effect of any changes, or repairs, to the measurement instrument should be assessed. A major repair may require either a calibration check (for example, a calibration verification exercise), or a complete re-calibration of the dosimetry system. Before use, the instrument shall be calibrated such that the measurements have traceability to a national or international standard. Guidance on spectrophotometer calibration, a common measurement instrument for dosimeters, is given in ASTM Practice E 925.

7.4.3 Calibration Procedure—The calibration procedure for a dosimetry system mainly consists of the irradiation of dosimeters to a number of known absorbed doses spanning the required dose range, the reading of the irradiated dosimeters using a calibrated measurement instrument, and the generation of a response function (calibration curve). Refer to ISO/ASTM Practice 51261 for more details.

7.4.3.1 Calibration Irradiation of Dosimeters—Dosimeters should be irradiated over a dose range broader than that of the intended use. The non-linear nature of most response functions means that extrapolations are not acceptable. For dose ranges of less than one decade, use at least four dose points distributed in an arithmetic progression (for example, 1, 3, 5, and 7 kGy). For dose ranges of greater than one decade use at least five dose points per decade and distribute the dose points in a geometric progression (for example, 0.1, 0.16, 0.25, 0.4, 0.63, 1.0, 1.6, 2.5, 4.0, 6.3, 10 kGy for two decades). Use a set of dosimeters consisting of 3 to 5 dosimeters at each dose point.

7.4.3.2 Environmental Conditions—For many dosimeters, the radiation-induced response is influenced by the environmental conditions, such as temperature during and after irradiation, humidity and dose rate. Because the dosimetry system calibration relationship is valid for the conditions present during the calibration procedure, the calibration conditions must be as similar as possible to those present during routine dose measurements in order to limit errors due to these effects. Thus, calibration irradiations may be performed in one of two ways, by irradiating the dosimeters at an irradiator that provides an absorbed dose (or an absorbed-dose rate) having measurement traceability to nationally or internationally recognized standards, or at the irradiator where the research experiments are being conducted.

NOTE 12—Although not treated in detail in this guide, quantitative data relating to environmental factors, such as temperature and moisture content of the irradiated food or agricultural products, should be reported because they may affect the response of dosimeters (see ISO/ASTM Guide 51261).

7.4.3.3 Irradiation at an Accredited Dosimetry Calibration Laboratory—This method involves irradiation of the routine dosimeters in the reference radiation field of a calibration laboratory. Although exactly matching the actual research conditions in a calibration laboratory is not generally possible, an attempt should be made to irradiate dosimeters using dose rate and temperature conditions as closely as possible to those that will be experienced during research experiments.

7.4.3.4 Irradiation in a Self-contained Irradiator—This method involves irradiating routine dosimeters at a static reference location in the radiation field where the dose rate is accurately known (this location should be preferably where the research samples would be irradiated). The dose rate shall be known at this reference location using transfer-standard dosimeters from an accredited dosimetry calibration laboratory. This dose rate value is valid for an irradiation geometry and for the specific dosimeter holder and its location in the radiation field. If the routine and transfer-standard dosimeters are of significantly different shape and size, the holder design needs special considerations; for example, the attenuation characteristics should be similar for both. Various irradiation times can be selected to give the specified dose to the routine dosimeters. Refer to ISO/ASTM Guide 52116.

NOTE 13—The certified dose rate should be measured by a national or accredited laboratory.

7.4.3.5 Irradiation at the Production Facility—Some production facilities may be trying to expand their portfolio of irradiation applications, and have irradiation time available for radiation research. In this case, calibration irradiation of routine dosimeters in a production irradiator using reference or transfer-standard dosimeters provides a calibration curve or response function valid for an actual production irradiation condition existing during the calibration. This method takes combined environmental factors into account to the extent that the reference or transfer dosimeter response can be corrected for differences in environmental factors between the calibration laboratory and production irradiator. Refer to ISO/ASTM Guide 51261.

7.4.3.6 Generation of Response Function—From the calibration (experimental) data generate a function that will enable dose to be determined from a measured dosimeter response (see ISO/ASTM Guides 51261 and 51707). Although this could be accomplished using a hand-drawn graph of response versus dose, in practice a mathematical fitting procedure (regression analysis) is generally used to obtain an analytical relationship between the dosimeter response and the dose. Strictly speaking, dose should be used as the independent variable (x variable). However, this may result in an expression that is difficult to solve for dose, which is the quantity required. In practice, letting dose be the dependent variable (y variable) is more convenient. Provided that the dose range is not greater than one decade, this procedure will not result in an appreciable error (see ISO/ASTM Guide 51707 and NPL Report CIRM 29).

NOTE 14—Commercially available software provides solutions for the inversion of polynomials, making the application of non-linear regression straightforward to manage. Mathematical functions that resemble the



expected behavior of the dosimeter can also be inverted analytically. Refer to Annex A1.

NOTE 15—In general, there is no recommended type of mathematical expression to represent the non-linear relationship between response and dose. In many cases, a polynomial function (for example, $response = a + b \times dose + c \times dose^2 + \dots$) will adequately describe the relationship. Individual dosimeter response values should be used, that is, readings from replicate dosimeters irradiated to the same dose should not be averaged. This enables an estimation of the dosimeter-to-dosimeter variability and allows outlying results to be identified. In selecting a polynomial function, the main consideration is to use the lowest order of polynomial that will adequately represent the data. One of the best methods of determining the required order is by examining the distribution of residuals with dose for increasing orders of the polynomial (see CIRM 29). The polynomial order of choice is the lowest order that does not exhibit a systematic trend.

NOTE 16—The residual (or error) represents unexplained (or residual) variation after fitting a regression model. It is the difference (or left over) between the observed value of the variable and the value predicted by the regression model. The 'relative residual' is the residual divided by either the observed or the predicted value. Fitting software use residuals, not relative residuals. The sum of residuals (including squared sum of residuals) is the quantity being minimized when fitting the calibration curve. A plot of residuals may reveal a pattern, gradually moving from positive to negative, and vice versa. Increase the order of polynomial if such a pattern exists. The lowest order that does not exhibit a systematic trend is the polynomial of choice.

NOTE 17—With commercially available software, the utilization of non-polynomial functions should be considered before the polynomial equation. The expected physical/chemical relationship is likely to be a non-polynomial. If the response of the dosimeter obeys a known mathematical relationship or physical law, for example, logarithmic, that function should be used.

7.4.3.7 The researcher is encouraged to contact a national laboratory or an accredited dosimetry calibration laboratory for guidance.

8. Performance qualification

8.1 Demonstrate and document that the experimental procedure will produce a reproducible dose distribution in a given sample. Documentation shall include descriptions of instrumentation and equipment necessary for the precise repetition of the experiment.

8.2 Equipment Testing and Calibration:

8.2.1 *Processing Equipment*—The absorbed dose in the sample depends on the experimental parameters.

8.2.1.1 Test the required equipment and instrumentation.

8.2.1.2 Implement a documented calibration program to ensure that the required equipment and instrumentation that may influence dose delivery are calibrated periodically. Examples are the calibration of the timing mechanism, and verification of the turntable rotation.

8.2.2 *Measurement Instruments*—The accuracy of the absorbed-dose determination depends on the correct operation and calibration of the analytical equipment used in the reading of the dosimeters.

8.2.2.1 Check the performance of the analytical equipment periodically to ensure that the equipment is functioning according to performance specifications. Repeat this check following any equipment modification or servicing and prior to the use of the equipment for a dosimetry system calibration. This check

can be accomplished by using reference standards such as calibrated optical density filters, wavelength standards, or calibrated thickness gauges supplied by the manufacturer or national or accredited standards laboratories.

8.2.2.2 Periodically, the performance of the equipment should be checked and the equipment recalibrated.

9. Experimental methodology and dose mapping

9.1 *Objective*—The purpose of dosimetry is to help establish the most suitable irradiation geometry for samples and products including selection of all key process parameters, and to provide evidence of reproducibility of dose and dose distribution.

9.1.1 The absorbed dose received by any portion of a sample or product depends on the irradiation and experimental parameters, such as the source type and geometry, irradiation geometry, product composition and density, and product distribution.

9.2 *Dose Mapping*—This objective is accomplished through mapping the absorbed-dose distribution throughout the sample, whether this comprises a single item, such as an onion or potato, or group of items such as a box of onions or potatoes. See ASTM Guide E 2303.

9.2.1 Ideally, the radiation process should be designed to irradiate the sample uniformly. In practice, a certain variation in absorbed dose through the sample will exist. Absorbed-dose mapping is used to determine the magnitude and locations of D_{max} and D_{min} for a given set of experimental parameters, including a specific reference geometry that should then be used throughout the life of the experiment. Changing the dimension of the container (box, crate) or the product configuration may require a remeasurement of the dose distribution.

9.2.2 Dose mapping involves placing dosimeters throughout the sample, both on the surface and within the sample. Placement patterns that can identify the locations of D_{max} and D_{min} should be selected (see ASTM Guide E 2303). Some dosimeters exist in the form of strips or sheets, enabling the researcher to obtain one or two dimensional dose distribution and reducing the number of dosimeters needed to produce a dose map.

NOTE 18—Prior to performing dose mapping the researcher should review the appropriate ISO/ASTM standards and scientific literature (5, 6, 9). Dosimetry data from previously performed experiments or theoretical calculations may provide useful information for determining the number and location of dosimeters needed for this exercise.

9.2.3 An example of a dosimeter placement array in a three-dimensional grid pattern that might represent a box or crate of food or agricultural product is given in Fig. 1. Dosimeters (illustrated as small rectangles) are placed throughout the box in order to obtain a map of the absorbed dose (dose map) within the box or crate. When replications of the dose mapping procedure are performed using a specific reference geometry, the degree of reproducibility in dose distribution can be obtained.

NOTE 19—When conducting research pertaining to the reduction of parasites, pathogenic bacteria, viruses, etc., small samples are often used in order to minimize the amount of food or agricultural material inoculated

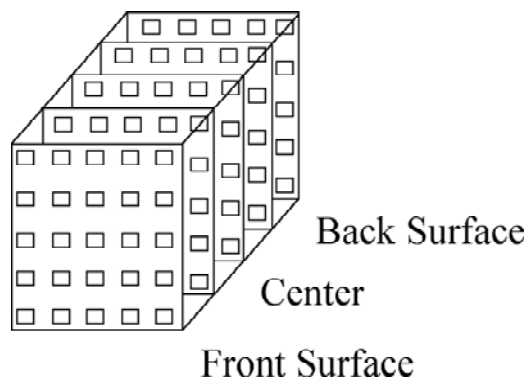


FIG. 1 Dosimeter placement for dose mapping a product container for photon irradiation

with the biological entity. This practice facilitates containment of the infectious biological agent and makes it easier for laboratory personnel to safely handle the material in a controlled setting. These samples may be of very small volume and weight. In this case the researcher may use small dosimeters to map the absorbed dose in un-inoculated material. In cases where the material to be irradiated is only a few milliliters in volume or a few grams in mass, dosimeters may be attached to the sides of the sample containers and the absorbed dose measured in that manner.

NOTE 20—In the case of free-flowing fluid or powder samples, absorbed-dose mapping may not be practicable. In those cases, theoretical calculations of the absorbed-dose distribution may be appropriate (see ASTM Guide E 2232). For liquids, suspension of dosimeters in semi-solid agars may be a practical solution.

9.2.4 Locations and values of $D_{\max}$ and $D_{\min}$ in the sample should be determined from the dose mapping data and reported. In cases where there is a significant difference between $D_{\max}$ and $D_{\min}$ in the sample, an attempt should be made to minimize the difference (see 9.3). If the difference is still significantly larger than the uncertainty in the dose measurements, correlate radiation effects at a specific location in the sample with the absorbed dose at that location.

NOTE 21—For example, a researcher is conducting experiments to determine the effect of ionizing radiation on inhibition of sprouting in potatoes. Many potatoes are required to obtain statistically valid data. When a crate (box) of potatoes is irradiated on a rotating platform, from two or four sides, there may be a range of absorbed doses, such that the potatoes in the center of the box will receive an absorbed dose that is less than those on the sides or corners. It is important, therefore, that the potatoes are not mixed after irradiation, and that the dose distribution within the crate (box) of potatoes is known. The potatoes should be removed carefully so that the research data on individual potatoes can be correlated with the absorbed dose that was received and the specific biological effect (in this case inhibition of sprouting) that was induced by that absorbed dose.

9.2.5 Changes in the product handling system or radiation source characteristics may affect the absorbed-dose distribution and thus require repeating the dose mapping procedure.

9.3 Dose Non-Uniformity:

9.3.1 If the incident radiation is attenuated substantially in the irradiated sample, large absorbed-dose gradients may exist. The dose uniformity in the sample may be improved by irradiating the sample on a rotating turntable, or by irradiating it from two or four sides. Re-designing of the packaging

configuration should also be considered. Large gradients in absorbed dose can also be minimized by reducing the sample volume.

9.3.2 To reduce the effect of low-energy radiation scattered from materials outside the sample, a container of composition similar to that of the sample should be selected (such as polyethylene, polystyrene or polymethylmethacrylate for biological samples).

9.3.3 In some irradiation geometries, especially when the gamma radiation or X-ray (bremsstrahlung) is collimated, the absorbed dose at the surface of the sample may be significantly lower than the equilibrium dose, which occurs at a depth in the material equivalent to the maximum range of the secondary electrons (about 3 mm polymeric material for cobalt-60 gamma radiation and 3 to 5 mm for 5 MeV X-ray (bremsstrahlung)). To achieve better dose uniformity within the sample, the container wall thickness should be approximately equal to this range. See Ref (17) for an explanation of secondary electrons and electron equilibrium.

9.3.4 Absorbed-dose variation may be significant in food or agricultural products where density and composition vary within the product. Examples include interfaces between soft tissue and bone or seed pit and fruit tissue. An attempt to quantify this absorbed-dose variation should be made by using a sufficient number of dosimeters placed at or near such interfaces.

9.3.5 In some applications the irradiated sample may be relatively close to the radiation sources, resulting in a significant absorbed-dose gradient near the periphery of the sample. Therefore, choosing a dosimetry system that is able to detect these gradients is important. Small dosimeters or dosimeter film in strips or sheets may be employed to obtain useful information about such dose gradients.

9.3.6 Once the reference geometry is established and a defined set of experimental parameters is finalized for a given experimental project, dosimetry is used to characterize absorbed-dose variations when conditions fluctuate statistically through normal experimental conditions. The extent of such variations may be established through dose mapping several identical samples using identical irradiation conditions. The resulting statistical variations in the absorbed-dose distributions in the irradiated sample should be evaluated and reported as part of the experimental results.

9.4 *Routine Monitoring Positions*—If the location of the $D_{\min}$ is not readily accessible during experimentations, alternative routine monitoring position(s) may be used for routine dose monitoring (see 10.2). The relationships between the dose at these locations and $D_{\min}$ must be established (during dose mapping), shown to be reproducible, and documented.

9.5 *Chilled or Frozen Foods*—Absorbed dose is not affected by the temperature of the food or agricultural product. The dosimeter response, however, can be affected by temperature. In fact, the response of nearly all dosimeters is temperature-dependent, and this dependence often varies with the absorbed-dose level and/or dose rate. Thus, for chilled and frozen food applications, dose mapping may be performed following one of two methods:

9.5.1 Absorbed-dose mapping may be performed with the sample or simulated sample at ambient temperature. This requires that there be no change in any parameter that may affect the absorbed dose during irradiation of the chilled or frozen food. Dose mapping at ambient temperature includes placement of one or more dosimeters at a routine monitoring position (see 9.4) that would be isolated from temperature gradients in the sample during routine experiments. Routine dosimeters should be placed at this routine monitoring position during experiments with chilled or frozen samples (see 10.2).

9.5.2 Absorbed-dose mapping may be performed at the temperature to which the sample will be chilled or frozen during actual experiments using a dosimetry system that can be characterized at this temperature or whose response is not significantly affected by temperature. The temperature of the sample and the dosimeters during irradiation must be maintained relatively constant (for example, by using an insulated container).

9.6 *Bulk-Flow Irradiators*—Absorbed-dose mapping as described in 9.2 may not be feasible for products flowing through the irradiation zone. In this case, $D_{\max}$ and $D_{\min}$ should be estimated by using an appropriate number of dosimeters mixed randomly with, and carried by the product, through the irradiation zone (4). Enough dosimeters should be used to obtain statistically significant results. Calculation of the $D_{\max}$ and $D_{\min}$ may be an appropriate alternative (see ASTM E 2381).

10. Dosimetry during experimentation

10.1 *Objective*—Once the reproducibility of the absorbed-dose distribution has been established for a given set of experimental conditions (see 9.3.6), the measurement of the absorbed dose periodically during the experiments, with proper statistics, is a critical requisite of Good Laboratory Practices (GLPs).

10.2 In a reproducible experiment, absorbed-dose measurement at the location of $D_{\max}$ and $D_{\min}$ or at the alternative routine monitoring positions identified during the dose mapping procedure (see 9.4) should be sufficient to indicate whether the experimental conditions are within specification (see Fig. 2).

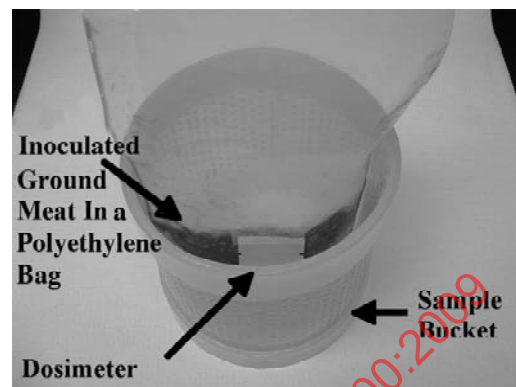
10.3 *Sample Loading Configuration*—Experiments should use the same sample loading configuration and irradiation geometry that was defined earlier (see Section 9).

10.4 *Selection of a Target Dose*—The experimental protocol usually includes choosing several target doses in order to help determine the optimal absorbed dose for the process/effect under consideration.

10.4.1 It is essential that the selection of target doses take into account the actual dose variation throughout the sample. Thus, the step width (interval/difference) between successive target doses should be larger than the difference between the $D_{\max}$ and $D_{\min}$ for the respective target doses (see Fig. 3).

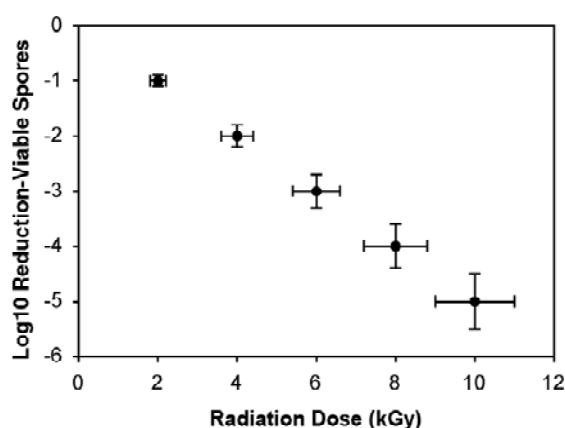
10.4.2 When applicable, transit dose shall be considered and quantified (see ISO/ASTM Guide 52116).

10.5 *Environmental Effects*—Because dosimeter response is affected by the environmental conditions, care should be taken



NOTE—This experimental set-up for irradiation of ground meat inoculated with food-borne pathogens was designed to determine the effect of ionizing radiation on pathogen viability. One hundred grams of ground meat, inoculated with a food-borne pathogen, were placed in a plastic bag used for microbial samples which was then placed vertically in the center of a plastic container (sample bucket). A dose map for the sample configuration (weight, dimensions, density) was first generated using non-inoculated ground meat. The ground meat sample has been configured to improve the dose uniformity. The sample and bucket are then positioned in the center of the radiation field of a self-contained, dry-storage gamma irradiator when in the irradiation position. This same geometry (the reference geometry) is used for all replicate experiments. The sample bucket has the same approximate density as the meat sample. Because placement of a dosimeter within the sample would be a safety hazard, it is attached to the side of the sample bucket at a predetermined position (routine monitoring position) to monitor absorbed dose during routine experimentation. All replicate measurements are subject to statistical analysis.

FIG. 2 Experimental set-up for the irradiation of ground meat



NOTE—An example of selecting the step width of the target doses. This graphic represents the decrease in viability of bacterial spores inoculated into a food product in response to ionizing radiation. Note the experiment has been designed such that the error bars for the absorbed doses, which include the $D_{\max}$ and $D_{\min}$, do not overlap. Dose effect curves may be either linear or non-linear depending on the effect measured.

FIG. 3 Step width selection of target dose

to ensure that these conditions during experiments are similar to those during calibration irradiation of the dosimeters. The