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Guide for selection and calibration of dosimetry systems for radiation processing

Guide de choix et d'étalonnage des appareils de mesure dosimétrique pour le traitement par irradiation

Reference number
ISO 15556:1998(E)

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.

International Standard ISO 15556 was prepared by the American Society for Testing and Materials (ASTM) Subcommittee E10.01 (as E 1261-94) and was adopted, under a special "fast-track procedure", by Technical Committee ISO/TC 85, *Nuclear energy*, in parallel with its approval by the ISO member bodies.

A new ISO/TC 85 Working Group WG 3, *High-level dosimetry for radiation processing*, was formed to review the voting comments from the ISO "Fast-track procedure" and to maintain these standards. The USA holds the convenership of this working group.

International Standard ISO 15556 is one of 20 standards developed and published by ASTM. The 20 fast-tracked standards and their associated ASTM designations are listed below:

ISO Designation	ASTM Designation	Title
15554	E 1204-93	*Practice for dosimetry in gamma irradiation facilities for food processing*
15555	E 1205-93	*Practice for use of a ceric-cerous sulfate dosimetry system*
15556	E 1261-94	*Guide for selection and calibration of dosimetry systems for radiation processing*
15557	E 1275-93	*Practice for use of a radiochromic film dosimetry system*
15558	E 1276-96	*Practice for use of a polymethylmethacrylate dosimetry system*
15559	E 1310-94	*Practice for use of a radiochromic optical waveguide dosimetry system*
15560	E 1400-95a	*Practice for characterization and performance of a high-dose radiation dosimetry calibration laboratory*
15561	E 1401-96	*Practice for use of a dichromate dosimetry system*

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15562	E 1431-91	*Practice for dosimetry in electron and bremsstrahlung irradiation facilities for food processing*
15563	E 1538-93	*Practice for use of the ethanol-chlorobenzene dosimetry system*
15564	E 1539-93	*Guide for use of radiation-sensitive indicators*
15565	E 1540-93	*Practice for use of a radiochromic liquid dosimetry system*
15566	E 1607-94	*Practice for use of the alanine-EPR dosimetry system*
15567	E 1608-94	*Practice for dosimetry in an X-ray (bremsstrahlung) facility for radiation processing*
15568	E 1631-96	*Practice for use of calorimetric dosimetry systems for electron beam dose measurements and dosimeter calibrations*
15569	E 1649-94	*Practice for dosimetry in an electron-beam facility for radiation processing at energies between 300 keV and 25 MeV*
15570	E 1650-94	*Practice for use of cellulose acetate dosimetry system*
15571	E 1702-95	*Practice for dosimetry in a gamma irradiation facility for radiation processing*
15572	E 1707-95	*Guide for estimating uncertainties in dosimetry for radiation processing*
15573	E 1818-96	*Practice for dosimetry in an electron-beam facility for radiation processing at energies between 80 keV and 300 keV*

Designation: E 1261 – 94

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Standard Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing[1]

This standard is issued under the fixed designation E 1261; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This guide provides the basis for selecting and calibrating dosimetry systems used to measure absorbed dose in gamma-ray or X-ray fields and in electron beams used for radiation processing. It discusses the types of dosimetry systems that may be employed during calibration or on a routine basis as part of quality assurance in commercial radiation processing of products. This guide also discusses interpretation of absorbed dose and briefly outlines the uncertainties associated with the dosimetry measurements. The details of the calibration of the analytical instrumentation are addressed in individual dosimetry system standard practices.

1.2 The absorbed-dose range covered is from about 1 Gy (100 rad) to 1 MGy (100 Mrad). Source energies covered are from 0.1 to 50 MeV photons and electrons.

1.3 Standard practices and guides for specific dosimetry systems and applications are covered in other standards. Dosimetry for radiation processing with neutrons or heavy charged particles is not covered in this guide.

1.4 *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:*

E 170 Terminology Relating to Radiation Measurements and Dosimetry[2]

E 178 Practice for Dealing with Outlying Observations[3]

E 666 Practice for Calculating Absorbed Dose from Gamma or X Radiation[2]

E 668 Practice for the Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices[2]

E 1026 Practice for Using the Fricke Reference Standard Dosimetry System[2]

E 1204 Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing[2]

E 1205 Practice for Use of a Ceric-Cerous Sulfate Dosimetry System[2]

E 1275 Practice for Use of a Radiochromic Film Dosimetry System[2]

E 1276 Practice for Use of a Polymethylmethacrylate Dosimetry System[2]

E 1310 Practice for Use of a Radiochromic Optical Waveguide Dosimetry System[2]

E 1400 Practice for Characterization and Performance of a High-Dose Radiation Dosimetry Calibration Laboratory[2]

E 1401 Practice for Use of a Dichromate Dosimetry System[2]

E 1431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing[2]

E 1538 Practice for Use of the Ethanol-Chlorobenzene Dosimetry System[2]

E 1540 Practice for Use of a Radiochromic Liquid Dosimetry System[2]

E 1607 Practice for Use of the Alanine–EPR Dosimetry System[2]

E 1631 Practice for Use of Calorimetric Dosimetry Systems for Electron Beam Dose Measurements and Dosimeter Calibrations[2]

2.2 *International Commission on Radiation Units and Measurements Reports:*

ICRU Report 14 Radiation Dosimetry: X-rays and Gamma rays with Maximum Photon Energies Between 0.6 and 50 MeV[4]

ICRU Report 17 Radiation Dosimetry: X-rays Generated at Potentials of 5 to 150 kV[4]

ICRU Report 33 Radiation Quantities and Units[4]

ICRU Report 34 The Dosimetry of Pulsed Radiation[4]

ICRU Report 35 Radiation Dosimetry: Electron Beams with Energies between 1 and 50 MeV[4]

ICRU Report 37 Stopping Powers for Electrons and Positrons[4]

3. Terminology

3.1 *Descriptions of Terms Specific to This Standard:*

3.1.1 *accredited dosimetry calibration laboratory*—a laboratory that meets specific performance criteria and has been tested and approved by a recognized accrediting organization.

3.1.2 *calibration curve*—graphical or mathematical rela-

[1] This guide is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.01 on Dosimetry for Radiation Processing.

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[2] *Annual Book of ASTM Standards*, Vol 12.02.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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

tionship between dosimeter response and absorbed dose for a given dosimetry system.

DISCUSSION—This term is also referred to as the response function.

3.1.3 *calibration facility*—combination of an ionizing radiation source and its associated instrumentation that provides uniform and reproducible absorbed-dose rates at specific locations in a specific material traceable to national standards, and therefore, may be used to calibrate the absorbed-dose response of routine or other types of dosimeters.

3.1.4 *dosimeter batch*—quantity of dosimeters made from a specific mass of material with uniform composition, fabricated in a single production run under controlled, consistent conditions, and having a unique identification code.

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

3.1.6 *electron equilibrium*—a condition that exists in material under irradiation when the energies, number, and direction of electrons induced by the radiation are constant throughout the volume of interest; thus, within such a volume, the sum of the energies of the electrons entering it is equal to the sum of the energies of all the electrons leaving it.

3.1.7 *measurement quality assurance plan*—a documented program for a measurement process that quantifies the total uncertainty of the measurement (both random and non-random components); this plan shall demonstrate traceability to national standards, and shall show that the total uncertainty meets the requirements of the specific application.

3.1.8 *primary standard dosimeter*—dosimeter, of the highest metrological quality, established and maintained as an absorbed dose standard by a national or international standards organization.

3.1.9 *quality assurance*—all systematic actions necessary to provide adequate confidence that a measurement is performed to a predefined level of quality.

3.1.10 *reference standard dosimeter*—dosimeter, of high metrological quality, used as a standard to provide measurements traceable to, and consistent with, measurements made using primary standard dosimeters.

3.1.11 *routine dosimeter*—dosimeter calibrated against a primary, reference, or transfer standard dosimeter and used for routine dosimetry measurements.

3.1.12 *simulated product*—mass of material with attenuation and scattering properties similar to those of a particular material or combination of materials.

DISCUSSION—This term is sometimes referred to as dummy product.

3.1.13 *stock*—part of a batch held by the user.

3.1.14 *traceability*—the ability to show that a measurement is consistent with appropriate national or international standards through an unbroken chain of comparisons.

3.1.15 *transfer standard dosimeter*—dosimeter, often a reference standard dosimeter, intended for transport between different locations for use as an intermediary to compare absorbed dose measurements.

3.2 Other terms used in this guide may be found in Terminology E 170, ICRU Report 33, and Ref (**1**).[5]

4. Significance and Use

4.1 Ionizing radiation is used to produce various desired effects in products. Examples include the sterilization of medical products, processing of food, modification of polymers, irradiation of electronic devices, and curing of inks, coatings, and adhesives (**2, 3**). The absorbed doses employed vary according to the application. The doses cover a range from about 10 Gy to more than 100 kGy.

4.2 Regulations for sterilization of medical products and radiation processing of food exist in many countries. These regulations may require that the response of the dosimetry system be calibrated and traceable to national standards (**4, 5, 6**). Adequate dosimetry, with proper statistical controls and documentation, is necessary to ensure that the products are properly processed.

4.3 Proper dosimetric measurements must be employed to ensure that the product receives the desired absorbed dose. The dosimeters must be calibrated. Calibration of a routine dosimetry system can be carried out directly in a national or secondary standards laboratory by standardized irradiation of routine dosimeters. It may be carried out through the use of a local (in-house) calibration facility (7) or in a production irradiator. All possible factors that may affect the response of dosimeters, including environmental conditions and variations of such conditions within a processing facility, should be known and taken into account. The associated analytical instrumentation must also be calibrated.

5. Dosimeter Classes and Applications

5.1 Dosimeters may be divided into four basic classes in accordance with their relative quality and areas of applications (see Section 3).

5.1.1 *Primary Standard Dosimeter*—Primary standard dosimeters are established and maintained by national standards laboratories for calibration of radiation fields. The two most commonly used primary standard dosimeters are ionization chambers and calorimeters (for details, see ICRU Reports 14, 17, 34, and 35).

5.1.2 *Reference Standard Dosimeter*—Reference standard dosimeters are used to calibrate radiation fields and routine dosimeters. A widely used reference dosimeter is the ferrous sulfate (Fricke) aqueous solution (see Practice E 1026). Examples of reference dosimeters are listed in Table 1; more details of the characteristics of several systems may be found in Appendix X3.

5.1.3 *Routine Dosimeters*—Examples of routine dosimeters are listed in Table 2; more details of the characteristics of several of these systems may be found in Appendix X3.

5.1.4 *Transfer Standard Dosimeters*—Transfer standard dosimeters are specially selected dosimeters used for transferring dose information from an accredited or national standards laboratory to a local irradiation facility in order to establish traceability for the local calibration facility. Normally, these dosimeters are used under conditions that are

[5] The boldface numbers in parentheses refer to the list of references at the end of this guide.

TABLE 1 Examples of Reference Standard Dosimeters

Dosimeter	Readout System	Useful Absorbed Dose, Gy	References[A]
Calorimeter	Thermometer	10^2 to 10^5	8
Alanine	EPR spectrometer	1 to 10^5	9
Ceric-cerous sulfate solution	UV spectrophotometer or electrochemical potentiometer	10^3 to 10^5	10, 11
Ethanol-chlorobenzene solution	Spectrophotometer, color titration, high frequency conductivity	10 to 2×10^6	12, 13
Ferrous sulfate solution	UV spectrophotometer	20 to 4×10^2	14
Potassium/silver dichromate	UV/visible spectrophotometer	10^3 to 10^5	15

[A] These references are not exhaustive; others may be found in the literature.

TABLE 2 Examples of Routine Dosimeters

Dosimeter	Readout System	Useful Absorbed Dose, Gy	References[A]
Alanine	EPR spectrometer	1 to 10^5	9
Dyed polymethylmethacrylate	Visible spectrophotometer	10^3 to 5×10^4	16, 17, 18
Clear polymethylmethacrylate	UV spectrophotometer	10^3 to 10^5	16, 19
Cellulose triacetate	Spectrophotometer	10^4 to 4×10^5	20
Lithium borate, lithium fluoride	Thermoluminescence reader	10^{-4} to 10^3	21
Lithium fluoride (optical grade)	UV/Visible spectrophotometer	10^2 to 10^6	22
Radiochromic dye films, solutions, papers, optical wave guide	Visible spectrophotometer	1 to 10^5	23, 24, 25
Ceric-cerous sulfate solution	Potentiometer or UV spectrophotometer	10^3 to 10^5	10
Ferrous-cupric sulfate solution	UV spectrophotometer	10^3 to 5×10^3	26
Ethanol-chlorobenzene solution	Spectrophotometer, color titration, high-frequency conductivity	10^2 to 10^5	13
Amino acids	Lyoluminescence reader	10^{-5} to 10^4	27

[A] These references are not exhaustive; others may be found in the literature.

carefully controlled by the issuing laboratory. They are selected from the list of available reference standard dosimeters (Table 1) or routine dosimeters (Table 2) that have characteristics listed in Table 3 that meet the required application requirements. In addition to the references given in Tables 1 and 2, relevant information on some other types of dosimeters may be found in Practices E 668, E 1275, and E 1276.

NOTE 1—None of the reference standard dosimeters or routine dosimeter listed have all of the desirable characteristics given in Table 3 for an "ideal" transfer standard dosimeter. However, such dosimeters may be used as transfer standard dosimeters if the absence of one or more desirable characteristics has negligible effect on the response of the dosimeter, or if correction factors can be applied to bring the dosimeter's response into conformity within the necessary limits of uncertainty for the application.

TABLE 3 Characteristics of Transfer Standard Dosimeters

Long shelf life
Easily calibrated
Stable
Portable
Mailable
Broad absorbed-dose range
Radiation absorption properties similar to those of irradiated product
Relatively insensitive to extremes of environmental conditions
Correctable systematic errors (for example, temperature, humidity, etc.)
Produced in reproducible lots
Small dimensions compared to distances over which absorbed-dose gradients become significant

6. Criteria for Selection of Routine Dosimetry Systems

6.1 The selection of an appropriate dosimetry system requires matching its performance with the specific application criteria. The following operational criteria should be considered in selecting a suitable dosimetry system.

NOTE 2—Availability of adequate information on the performance characteristics of the dosimetry systems should be considered in selecting a dosimetry system.

6.1.1 Suitability of the dosimeter for the absorbed-dose range of interest and for use with a specific product,

6.1.2 Stability and reproducibility of the system,

6.1.3 Ease of system calibration,

6.1.4 Traceability of system calibration to national standards,

6.1.5 Ability to control or correct system response for systematic errors, such as those caused by temperature and humidity,

6.1.6 Ease and simplicity of use,

6.1.7 Availability of dosimeters in reasonably large quantities,

6.1.8 Overall initial and operational cost of the system, including dosimeters, readout equipment, and labor,

6.1.9 Time required for dosimeter response development, and labor required for dosimeter readout and interpretation,

6.1.10 Ruggedness of the system—resistance to damage during routine handling and use in a processing environment,

6.1.11 Variance of the dosimetry system response data within established limits about a fitted calibration curve over the absorbed-dose range of interest. Suitable regression analysis methods should be used to fit the curve, and could include linear, polynomial, or exponential functions,

6.1.12 Dependence of dosimeter response on environmental conditions (such as temperature, humidity, and light) before, during, and after calibration and production irradiation. Effects of environmental conditions on the dosimeter readout equipment shall also be considered,

6.1.13 Dependence of dosimeter response on absorbed-dose rate or incremental delivery of absorbed dose, or both,

6.1.14 Stability of dosimeter response both before and after irradiation,

6.1.15 Variation of dosimeter response within a batch or between batches,

6.1.16 Effects of size, location, and composition of the dosimeter on the radiation field or the interpretation of the absorbed-dose measurement. In cases where it is desirable to measure absorbed dose at the interface of different materials (for example, at a bone-tissue interface or the surface of a product), dosimeters should be used that are thin compared to distances over which the absorbed-dose gradient is significant, and

6.1.17 Effects of differences in radiation energy spectra between calibration and product irradiation fields.

7. Analytical Instrument Performance

7.1 Check the performance of the analytical instrumenta-

tion prior to the reading of dosimeters irradiated for a dosimetry system calibration.

7.1.1 Check the analytical instrumentation in accordance with documented procedures (for example, the operating manual) to ensure that the instrument is functioning in accordance with appropriate performance specifications.

7.1.1.1 For optical absorbance measurements using a spectrophotometer, check and document that the wavelength and absorbance scales are within the documented specifications at or near the analysis wavelength using optical density filters and wavelength standards traceable to national standards.

7.1.1.2 For thickness measurements using a thickness gage, check and document that the instrument is within documented specifications using gage blocks traceable to national standards.

7.2 For some analytical instrumentation, correct performance can be demonstrated by showing that the readings of dosimeters given known absorbed doses are in agreement with the expected readings within the limits of the dosimetry system uncertainty.

8. Dosimetry System Calibration

8.1 *General:*

8.1.1 The calibration of a dosimetry system consists of the irradiation of dosimeters to a number of known absorbed doses over the range of use, analysis of the dosimeters using calibrated analytical equipment, and the generation of a calibration curve. Calibration verification is performed periodically to confirm the continued validity of the calibration curve.

8.1.2 The calibration of reference or transfer standard dosimeters shall be performed using a calibration facility that has an absorbed-dose rate traceable to national standards. Gamma calibration facilities shall meet the requirements specified in Practice E 1400. Electron beam and X-ray (bremsstrahlung) calibration facilities should meet similar requirements.

NOTE 3—In several countries, the national standard is realized indirectly through a calibrated radiation field. For example, the absorbed-dose rate in the center of a ^{60}Co source array at the U.S. National Institute of Standards and Technology (NIST) has been well characterized by calorimetry and is one of the national standards used by NIST to irradiate reference standard, transfer standard, and routine dosimeters to known absorbed-dose levels.

NOTE 4—The response of reference and transfer standard dosimeters to environmental effects such as temperature and relative humidity, absorbed-dose rate, and energy spectrum, should be documented. Differences in the dosimeter response between calibration and use conditions should be taken into account using known correction factors.

8.1.3 The calibration of routine dosimeters can be performed in three different ways. One way (described in 8.3) calls for routine dosimeters to be irradiated in a gamma, electron beam, or X-ray (bremsstrahlung) calibration facility meeting the requirements of Practice E 1400. The second way (described in 8.4) calls for routine dosimeters to be irradiated in an in-house calibration facility that has an absorbed-dose rate measured by reference or transfer standard dosimeters. The third way (described in 8.5) calls for routine dosimeters to be irradiated together with reference or transfer standard dosimeters in the production irradiator.

NOTE 5—The response of some routine dosimeters may be affected by the combined effect of several environmental factors such as temperature, absorbed-dose rate, energy spectrum, and relative humidity, including possible seasonal variations of some of these factors. For these cases, it may not be possible to take these combined effects into account by applying a correction factor. To ensure that the calibration curve is valid for the conditions of use, the calibration of routine dosimeters should be performed using irradiation conditions similar to those in the actual production irradiator and verified prior to use.

8.1.3.1 The calibration of routine dosimeters using a gamma, electron beam, or X-ray calibration facility meeting the requirements of Practice E 1400 has the advantage that the dosimeters are irradiated to accurately known absorbed doses under well-controlled and documented conditions. However, use of these routine dosimeters under different environmental conditions in a production irradiator may introduce uncertainties that are difficult to quantify. Transporting of the dosimeters to and from the calibration facility may also introduce uncertainties from pre- and post-irradiation storage effects.

8.1.3.2 The calibration of routine dosimeters using an in-house calibration facility has the advantage that the pre- and post-irradiation storage conditions of the dosimeters can be controlled so that they are similar to those encountered during routine production. However, it may not be possible for the in-house calibration facility to meet all the irradiation requirements of Practice E 1400. In addition, use of transfer standard dosimeters is required to provide traceability to national standards.

8.1.3.3 The calibration of routine dosimeters by irradiation of the dosimeters together with reference or transfer standard dosimeters in the production irradiator has the advantage that the environmetal conditions are similar to those encountered during routine production, reducing the requirement to make corrections for the routine dosimeter for environmental effects. However, great care must be taken to ensure that the routine and reference or transfer standard dosimeters irradiated together receive the same absorbed dose (23).

8.2 *Analytical Instrument Calibration:*

8.2.1 Analytical instrumentation shall be calibrated annually in accordance with written procedures by qualified individuals.

8.2.2 Calibration should provide traceability and consistency to national standards if available.

8.2.3 Verification of the calibration curve shall be performed if any maintenance or modification of the analytical instrumentation occurs that may affect its performance. In this case, it shall be demonstrated that the measurements of dosimeter response are within specified limits over the full dose range.

8.3 *Irradiation of Dosimeters Using a Calibration Facility:*

8.3.1 Irradiate reference standard, transfer standard, or routine dosimeters to known absorbed doses in a national calibration facility or a calibration facility that has an absorbed-dose rate traceable to national standards. Gamma, electron beam, or X-ray calibration facilities shall meet the requirements specified in Practice E 1400.

8.3.2 Calibrate routine dosimeters using irradiation conditions similar to those in the actual production irradiator.

Employ an energy spectrum, absorbed-dose rate, and irradiation temperature as close as practical to those encountered during routine use.

8.3.3 Adverse environmental conditions (such as high or low temperature and humidity) during transport of the dosimeters to and from the calibration facility may affect the dosimeter response.

8.3.4 Package dosimeters to minimize the effects of environmental conditions during transport.

8.3.5 Environmental monitors such as maximum temperature indicators may be included in the dosimeter package during transport to document environmental extremes.

8.3.6 Confirm that the environmental conditions during transfer have not changed the response of the dosimeter. This may be achieved by sending dosimeters irradiated to known absorbed doses along with the dosimeters sent for calibration. The readings of the two sets of dosimeters should be compared when they are returned with readings from additional dosimeters given the same absorbed dose and stored under controlled conditions.

8.3.7 Position the dosimeters in the calibration radiation field in a defined, reproducible location. The variation in absorbed-dose rate within the volume occupied by the dosimeters should be within ±1 %.

8.3.8 When using a gamma-ray source or X-ray beam for calibration, surround the dosimeter with a sufficient amount of material to achieve approximate electron equilibrium conditions (28).

NOTE 6—The appropriate thickness of such material depends on the energy of the radiation (see Practices E 666 and E 668). For measurement of absorbed dose in water, use materials that have radiation-absorption properties essentially equivalent to water. For example, for a ^{60}Co source, 3 to 5 mm of polystyrene (or equivalent polymeric material) should surround the dosimeter in all directions.

8.3.9 Control or monitor the temperature during gamma irradiation of the dosimeters.

NOTE 7—It may be difficult to control or monitor the temperature of the dosimeter during electron or X-ray irradiation.

8.3.10 If the response of the dosimeters is affected by humidity and they are not sealed, control or monitor the relative humidity during irradiation.

8.3.11 For each absorbed dose point, use the number of dosimeters required to achieve the desired confidence level (see Section 9 of Practice E 668).

8.3.12 The number of sets of dosimeters required to determine the calibration curve of the dosimetry system depends on the absorbed-dose range of utilization. Use at least five sets for each factor of ten span of absorbed dose, or at least four sets if the range of utilization is less than a factor of ten.

NOTE 8—To determine mathematically the minimum number of sets to be used, divide the maximum dose in the range of utilization (D_{max}) by the minimum dose (D_{min}), then, calculate log(base 10) of this ratio: $Q = \log(D_{max}/D_{min})$. If Q is less than 1, use a minimum of four sets. If Q is equal to or greater than 1, calculate the multiple $5 \times Q$, and round this to the nearest integer value. This value represents the minimum number of sets to be used.

8.3.13 Specify the calibration dose in terms of the material of interest. The calibration dose is usually specified in terms of absorbed dose in water. See Appendix X1 for conversion factors for calculating the absorbed dose in different materials.

8.3.14 Consider the possibility of combined environmental effects (see Note 5).

8.4 *Irradiation of Dosimeters Using an In-House Calibration Facility:*

8.4.1 Routine dosimetry systems may be calibrated by irradiating dosimeters in an in-house calibration facility.

8.4.2 The absorbed-dose rate in the in-house calibration facility shall be demonstrated to be traceable to appropriate national standards by direct measurement intercomparisons or calibrations using transfer standard dosimeters supplied by a nationally recognized radiation dosimetry calibration laboratory.

8.4.3 Measurement intercomparisons or calibrations of absorbed-dose rates of the in-house calibration facility shall be performed at least once every three years and after any change in source activity or geometry.

8.4.4 Procedures, protocols, and training of personnel shall be provided to ensure that the correct absorbed dose is given to dosimeters.

8.4.5 All criteria given in 8.3.2 to 8.3.14 shall be met.

8.4.6 Calibration of routine dosimeters in an in-house calibration facility reduces the possibility of changes in the response due to adverse storage conditions during transport of dosimeters. After irradiation, dosimeters should be stored under similar conditions to those encountered in the production irradiator and read at approximately the same time after irradiation as the dosimeters used in routine dosimetry.

8.5 *Irradiation of Dosimeters Using a Production Irradiator:*

8.5.1 Calibration of routine dosimeters in the production irradiator using reference or transfer standard dosimeters provides a calibration curve valid for the actual production irradiation conditions 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 facility and production irradiator.

8.5.2 Use reference or transfer standard dosimeters supplied and analyzed by a nationally recognized radiation dosimetry calibration laboratory to demonstrate traceability to national standards.

8.5.3 Reference or transfer standard dosimeters obtained commercially or prepared in accordance with published standards and analyzed on site may be used provided that the reference or transfer standard dosimetry systems have been calibrated in accordance with 8.1.

8.5.4 Calibrate routine dosimeters by irradiating them together with reference or transfer standard dosimeters under actual production irradiation conditions over the entire range of normal use. Ensure that the routine and reference or transfer standard dosimeters receive the same absorbed dose.

8.5.5 Design a calibration package to ensure that the dosimeters do not shield each other significantly during irradiation. The calibration package shall contain the number of routine dosimeters required to achieve the desired confidence level and one or more reference or transfer standard dosimeter (see Section 9 of Practice E 668).

NOTE 9—This absorbed dose variation can be confirmed by irradiating calibration packages containing one type of dosimeter in all dosimeter positions within the calibration package.

8.5.5.1 For gamma or X-ray sources, the calibration package should provide a sufficient thickness of water-equivalent material to achieve approximate electron equilibrium conditions (see Note 6).

8.5.5.2 When thick and thin dosimeters are irradiated together, surround the thin dosimeters by sufficient polymeric material to ensure that the attenuation characteristics are similar and to ensure that the dosimeters receive the same dose.

8.5.6 To calibrate the dosimeters under conditions similar to those used for processing, place the calibration packages with product or simulated product in volumes where the absorbed-dose variation over the area containing dosimeters is within specified limits.

8.5.7 Use a sufficient number of routine and reference or transfer standard dosimeters to give statistically significant results. Irradiate at least five calibration packages to different absorbed doses covering the absorbed-dose range of utilization for each factor of ten span of absorbed dose (see Note 8).

NOTE 10—The absorbed-dose rate, temperature, and energy spectrum may vary depending on the location of the dosimeter on or in the irradiation unit. These factors may have to be considered when evaluating overall dosimeter uncertainties during routine use.

8.6 *Dosimeter Analysis:*

8.6.1 Analyze dosimeters using analytical instrumentation with calibration traceable to national standards.

8.6.2 Check the performance of the analytical instrumentation (see 7.1).

8.6.3 If the dosimeter response changes with time after irradiation, analyze dosimeters at approximately the time after irradiation when dosimeters will be analyzed during routine production.

8.6.4 Document and retain all analysis data.

8.7 *Calibration Curve:*

8.7.1 Calculate and document the mean response, $\bar{k}$, and the sample standard deviation (S_{n-1}) for each set of dosimeters at each absorbed-dose value. The sample standard deviation, S_{n-1}, is calculated from the sample data set of n values as follows:

$$S_{n-1} = \sqrt{\frac{\Sigma(k_i - \bar{k})^2}{n-1}} \quad (1)$$

where k_i = ith value of k.

8.7.2 Calculate the coefficients of variation, CV, for each absorbed dose value as follows:

$$CV = \frac{S_{n-1}}{\bar{k}} \times 100(\%) \quad (2)$$

NOTE 11—In general, if any CV values are greater than 2 %, then a redetermination of the data should be considered, or the stock of dosimeters should be rejected.

8.7.3 Graphically plot dosimeter response versus absorbed dose, or use a suitable computer code, or both, to derive this relationship in mathematical form. Choose an analytical form (for example, linear, polynomial, or exponential) that provides an appropriate fit to the measured data.

8.7.4 Examine the resulting calibration curve for goodness of fit within specified limits.

8.7.5 Repeat this calibration procedure to the extent necessary if any response value exceeds accepted statistical limits of the determined curve, and if discarding this value would result in there being insufficient data to adequately define the curve (see Practice E 178 for guidance on dealing with outliers).

8.8 *Calibration Verification:*

8.8.1 Verify that calibration curves for routine dosimetry systems obtained by irradiating dosimeters in a dosimetry calibration facility or in an in-house calibration facility are valid for the actual conditions of use in the production facility.

8.8.2 Calibration verification may be performed by irradiating the routine dosimeters together with reference standard or transfer standard dosimeters to three different absorbed doses in the production irradiator. The reference standard or transfer standard dosimeters should be of a different type than the routine dosimeters, to reduce the probability that both types of dosimeters are influenced by the same combined environmental effects. Ensure that the routine and reference standard or transfer standard dosimeters receive the same absorbed dose (see 8.5.5 for guidance).

8.8.3 Compare absorbed-dose values obtained from the calibration curve with the absorbed doses measured by the reference standard or transfer standard dosimeters.

8.8.3.1 If the difference between the transfer standard and routine dosimeter measurement of absorbed dose exceeds the estimated combined uncertainty in the two systems exclusive of environmental effects, the calibration curve may be adjusted by a constant factor to give agreement with the reference or transfer standard dosimeters.

8.8.3.2 If the difference varies significantly at different absorbed doses, recalibration using the method described in 8.5 may be necessary.

8.8.4 Calibration curves supplied by a manufacturer shall not be used.

8.8.5 Calibration curves generated by one analytical instrument shall not be used for another instrument unless it has been demonstrated that the measurements of the dosimeters' response is within specified limits over the full absorbed dose range.

8.9 *Absorbed-Dose Rate Effects:*

8.9.1 For some routine dosimetry systems, the dosimeter response at different absorbed-dose rates for the same given absorbed dose may differ over portions of the system's working range. In these portions, the higher absorbed-dose rate response may diverge from the lower absorbed-dose rate response. This divergence may be dependent on several factors, such as the absorbed dose and type of radiation (gamma, electron beam, or X ray). In these divergence tests, other factors that could influence dosimeter response, for example irradiation temperature, should be either fixed or kept within a narrow range. The divergence may be checked by several methods.

8.9.2 As appropriate for the intended application, irradiate dosimeters using gamma, electron beam, or X-ray facilities with absorbed-dose rates that span the range expected in the production facility. Irradiate the dosimeters to the same absorbed dose at two or more dose rates. Repeat this process for several absorbed doses to the highest absorbed dose of interest. Compare the resultant responses of

the dosimeters. Divergence of the response curves provides an indication of the magnitude of the absorbed-dose rate effect.

NOTE 12—This effect may vary for each batch of routine dosimeters.

8.9.3 For dosimetry systems known to have negligible absorbed-dose rate dependence in the low absorbed-dose portion, irradiate the dosimeters in a gamma, electron beam, or X-ray production facility to an absorbed dose in the low absorbed-dose portion of the working range of the dosimetry system. Interpret the absorbed dose using the calibration curve obtained from dosimeters irradiated at a dosimetry calibration facility. Change the production facility irradiation parameters (for example, conveyor speed, electron beam current, or dwell time) to increase the absorbed dose. Compare the resultant dosimeter response curve to the original calibration curve. The resultant response curve may show divergence from the calibration curve as the absorbed dose increases. The resultant response curve can be related to the original calibration curve at low absorbed doses to provide corrected response at higher absorbed doses.

8.10 *Frequency of Calibration:*

8.10.1 Calibrate the dosimetry system for each new batch of reference standard, transfer standard, or routine dosimeters prior to use.

8.10.1.1 At an interval not exceeding one year, re-calibrate the dosimetry system for each batch of reference standard, transfer standard, or routine dosimeters. This re-calibration shall include the analytical instrumentation. Depending on seasonal variations in ambient conditions (for example, temperature and relative humidity) the interval of re-calibration may have to be decreased (see Note 5).

8.10.2 Check the calibration of the dosimetry system for each new stock of reference standard or transfer standard dosimeters and routine dosimeters using at least three absorbed doses over the range of application in order to verify that their responses are the same as for the current stock.

8.10.3 Periodically verify the calibration of the dosimetry system for each new batch of routine dosimeters.

NOTE 13—One method of verification is to irradiate together reference standard or transfer standard dosimeters, dosimeters from the new batch, and dosimeters from the current batch to ensure consistency of absorbed-dose measurements when a new batch of routine dosimeters is introduced.

8.11 *Calibration Uncertainty*—The overall uncertainty in the calibration of the routine dosimetry system used in radiation processing should be within ±6 % at a 95 % confidence level. This level of uncertainty should be achievable if these calibration procedures are followed.

9. Interpretation of Absorbed Dose in a Product

9.1 Generally, the absorbed dose in an irradiated food or polymer product is specified in terms of absorbed dose in water because most food or polymer products are nearly water-equivalent in terms of radiation absorption properties. If the radiation absorption properties of the product differ from those of water, then interpretation of the absorbed dose in the product may be accomplished by means of cavity theory (see Appendix X1).

9.2 Electron equilibrium conditions may not exist within dosimeters placed throughout the product under actual processing conditions. This particularly is the case near interfaces of different materials, for example, at bone-tissue interfaces or on the surface of a product package. Absorbed-dose measured under non-equilibrium conditions is sometimes used to monitor the absorbed dose within the product using the procedures described in Practices E 1204 and E 1431.

10. Minimum Documentation Requirements

10.1 Record the routine dosimetry system used with each product irradiated. Identify the dosimeter manufacturer, type and batch number, and instruments used for analysis.

10.2 Record the dosimeter calibration data, including date, reference standard or transfer standard, and description of the facility used.

10.3 Record or reference a description of the radiation source used in processing, including the type, nominal activity or beam parameters, and any available information on the energy spectrum.

10.4 Record the irradiation environmental conditions for the routine dosimeter, including temperature, pressure (if other than atmospheric), relative humidity, and surrounding atmosphere (if other than air).

10.5 Record or reference the method used to convert dosimetry measurements to absorbed-dose values in water or the product (see Section 9).

10.6 Record the value and the assigned uncertainty of the absorbed dose to the product for each irradiation.

10.7 Record or reference the measurement quality assurance plan used for the routine dosimetry.

11. Precision and Bias

11.1 To be meaningful, a measurement of absorbed dose shall be accompanied by an estimate of the uncertainty in the measured value. Factors contributing to this uncertainty may be separated into two types, precision (random) and bias (systematic). The uncertainty should be stated as an estimate of the probable limits of error, combining both precision and bias **(29, 30)**. See Appendix X2 for a discussion on sources of uncertainties.

11.2 *Precision*—For a measurement of absorbed dose, the precision (random uncertainty) of the measurement process is due to at least two effects. These are variations in intrinsic response of the individual dosimeters and of associated instrumentation. The precision due to these two effects may be determined by replicate measurements of a specific absorbed dose. The number of replicates is determined by the variability in dosimetry system response and specified confidence level (see Practice E 668 for general guidance on determining this number). There may be other sources of random uncertainty contributing to the total estimate of the precision of the measurements. Assuming normal distribution, all values of such components of uncertainty may be summed in quadrature (that is, the square root of the sum of the squares) and the results reported at the specified confidence level. Determine the precision for the dosimetric system at the time of calibration and repeat the procedure if a change takes place that might affect the precision.

11.3 *Bias:*

11.3.1 All nonrandom factors contributing to the uncer-