<span id="page-0-0"></span>

# **Standard Practice for Use of a LiF Photo-Fluorescent Film Dosimetry System<sup>1</sup>**

This standard is issued under the fixed designation E2304; 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  $(\varepsilon)$  indicates an editorial change since the last revision or reapproval.

# **1. Scope**

1.1 This practice covers the handling, testing, and procedure for using a lithium fluoride (LiF)-based photo-fluorescent film dosimetry system to measure absorbed dose (relative to water) in materials irradiated by photons or electrons. Other alkali halides that may also exhibit photofluorescence (for example, NaCl, NaF, and KCl) are not covered in this practice. Although various alkali halides have been used for dosimetry for years utilizing thermoluminescence, the use of photoluminescence is relatively new.

1.2 This practice applies to photo-fluorescent film dosimeters (referred hereafter as photo-fluorescent dosimeters) that can be used within part or all of the following ranges:

- 1.2.1 Absorbed dose range of  $5 \times 10^{-2}$  to  $3 \times 10^{2}$  kGy  $(1-3)^{2}$  $(1-3)^{2}$ .
- 1.2.2 Absorbed dose rate range of  $0.3$  to  $2 \times 10^4$  Gy/s  $(2-5)$  $(2-5)$ ).

1.2.3 Radiation energy range for photons of 0.05 to 10 MeV **(2)**.

1.2.4 Radiation energy range for electrons of 0.1 to 10 MeV **[\(2\)](#page-3-0)**.

1.2.5 Radiation temperature range of -20 to +60°C **[\(6,7\)](#page-3-0)**.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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:*<sup>3</sup>

- [E170](#page-2-0) [Terminology Relating to Radiation Measurements and](http://dx.doi.org/10.1520/E0170) **[Dosimetry](http://dx.doi.org/10.1520/E0170)**
- E275 [Practice for Describing and Measuring Performance of](http://dx.doi.org/10.1520/E0275) Ultraviolet [and Visible Spectrophotometers](http://dx.doi.org/10.1520/E0275)
- E925 [Practice for Monitoring the Calibration of Ultraviolet-](http://dx.doi.org/10.1520/E0925)[Visible Spectrophotometers whose Spectral Bandwidth](http://dx.doi.org/10.1520/E0925) [does not Exceed 2 nm](http://dx.doi.org/10.1520/E0925)
- 2.2 *ISO/ASTM Standards:*
- [51204](#page-4-0) Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing
- [51261](#page-3-0) Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing
- [51431](#page-1-0) Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing
- [51608](#page-4-0) Practice for Dosimetry in an X-ray (Bremsstrahlung) Facility for Radiation Processing
- [51649](#page-4-0) Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV
- [51702](#page--1-0) Practice for Dosimetry in a Gamma Irradiation Facility for Radiation Processing
- [51707](#page-4-0) Guide for Estimating Uncertainties in Dosimetry for Radiation Processing
- 51818 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 80 keV and 300 keV
- [51956](#page-1-0) Practice for Thermoluminescence-Dosimetry (TLD) Systems for Radiation Processing

2.3 *International Commission on Radiation Units and Measurements (ICRU) Reports:*<sup>4</sup>

- [ICRU Report 14](#page-1-0) Radiation Dosimetry: X-rays and Gamma rays with Maximum Photon Energies Between 0.6 and 50 MeV
- [ICRU Report 17](#page-4-0) Radiation Dosimetry: X-rays Generated at Potentials of 5 to 150 kV
- [ICRU Report 34](#page-5-0) The Dosimetry of Pulsed Radiation
- [ICRU Report 35](#page-5-0) Radiation Dosimetry: Electron Beams with Energies Between 1 and 50 MeV
- [ICRU Report 60](#page-1-0) Fundamental Quantities and Units for Ionizing Radiation

 $1$  This practice is under the jurisdiction of ASTM Committee [E61](http://www.astm.org/COMMIT/COMMITTEE/E61.htm) on Radiation Processingand is the direct responsibility of Subcommittee [E61.02](http://www.astm.org/COMMIT/SUBCOMMIT/E6102.htm) on Dosimetry **Systems**.

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<sup>&</sup>lt;sup>2</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

<sup>&</sup>lt;sup>3</sup> For referenced 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.

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

# <span id="page-1-0"></span>**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<sup>-1</sup>). The mathematical relationship is the quotient of  $d\bar{\varepsilon}$  by  $dm$ , where  $d\bar{\varepsilon}$  is the mean incremental energy imparted by ionizing radiation to matter of incremental mass *dm* (see ICRU 60).

$$
D = \frac{d\bar{\varepsilon}}{dm}
$$

3.1.1.1 *Discussion—*Absorbed dose is sometimes referred to simply as dose. For a photon source under conditions of charged particle-equilibrium, the absorbed dose, *D*, may be expressed as:

$$
D = \varphi E \frac{\mu_{en}}{\rho}
$$

where:

 $\varphi$  = particle fluence (m<sup>-2</sup>),<br>  $E$  = energy of the ionizing

 $=$  energy of the ionizing radiation  $(J)$ , and  $\mu_{en}$  / $\rho$  = mass energy absorption coefficient (m<sup>2</sup> kg<sup>-1</sup>).

If bremsstrahlung production within the specified material is negligible, the mass energy absorption coefficient  $(\mu_{en}/\rho)$  is equal to the mass energy transfer coefficient  $(\mu_t/\rho)$ , and absorbed dose is equal to kerma if, in addition, charged-particle equilibrium exists.

3.1.2 *alkali halide—*a binary compound consisting of a halogen (any of the five elements fluorine, chlorine, bromine, iodine, and astatine) and an alkali metal (for example, lithium, sodium, and potassium).

3.1.3 *analysis wavelength—*wavelength used in a spectrophotometric instrument to help determine a desired dosimetric quantity, for example, absorbed dose, by means of the measurement of optical absorbance, optical density, reflectance or luminescence.

3.1.4 *calibration facility—*combination of an ionizing radiation source and its associated instrumentation that provides a uniform and reproducible absorbed dose, or absorbed-dose rate traceable to national or international standards at a specified location and within a specific material, and that may be used to derive the dosimetry system's response function or calibration curve.

3.1.5 *charged-particle equilibrium—*the condition that exists in an incremental volume within a material under irradiation if the kinetic energies and number of charged particles (of each type) entering the volume are equal to those leaving the volume.

3.1.6 *color center—*imperfections (for example, negativeor positive-ion vacancies) within the ionic lattice of compounds that have trapped electrons or electron holes. These centers, upon excitation by energy in the form of light or heat, can produce luminescence.

3.1.7 *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.

3.1.8 *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.

3.1.9 *electron equilibrium—*charged particle equilibrium for electrons.

3.1.10 *fluorescence—*one of the four main luminescence mechanisms. In many materials, it involves the liberated electrons falling back to the valence band—directly or via a relaxation state—to fill an electron hole, resulting in the release of a photon. In the case of alkali-halides the liberated electrons do not fall back to the valance band, but are excited to a higher state within the color center, and subsequently fall back to the center's ground state, resulting in the release of a photon.

3.1.11 *fluorescence signal, Ef —*the photometric reading by a spectrofluorimeter in terms of light intensity incident on the photodetector. Typically, the value measured is some quantity proportional to the standardized quantity, irradiance,  $E_i$  (for example, volts or amperes per unit area of detector surface, V  $cm^{-2}$  or A  $cm^{-2}$ ).

3.1.12 *fluorescence standard—*a solid or liquid material that produces a fluorescence upon excitation, with an emitted radiance that is calibrated and made traceable to a recognized standard.

3.1.13 *fluorimeter—*instrument used to measure the amount of fluorescence signal,  $E_f$ , emitted from a sample upon excitation by an energy source (usually in the form of light).

3.1.14 *irradiance*,  $E_i$ —a radiometric term for the radiant flux that is incident upon a surface, having units of  $W m<sup>-2</sup>$ . Also see *radiance*.

NOTE 1—The standard symbol for irradiance is *E*; however, for this document the subscript, *i*, was added to distinguish irradiance from energy of ionizing radiation (see 3.1.1) and fluorescence signal.

3.1.15 *luminescence—*photon emission from a solid or liquid phosphor material during, or after, exposure to a form of energy. The main luminescence mechanisms are fluorescence, phosphorescence, thermoluminescence, and photoluminescence.

3.1.16 *measurement quality assurance plan—*a documented program for the measurement process that ensures on a continuing basis that the overall uncertainty meets the requirements of the specific application. This plan requires traceability to, and consistency with, nationally or internationally recognized standards.

3.1.17 *measurement traceability—*the ability to demonstrate by means of an unbroken chain of comparisons that a measurement is in agreement within acceptable limits of uncertainty with comparable nationally or internationally recognized standards.

3.1.18 *net fluorescence,* ∆*Ef —*measured fluorescence signal,  $E_f$  from an irradiated sample, subtracted by the pre-irradiation fluorescence,  $E_o$ , as follows:

$$
\Delta E_{f}=E_{f}-E_{o}
$$

<span id="page-2-0"></span>3.1.19 *photo-fluorescent film dosimeter—*a film-type dosimeter, which upon excitation by visible or UV light, emits fluorescent light.

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

3.1.21 *quality assurance—*all systematic actions necessary to provide adequate confidence that a calibration, measurement, or process is performed to a predefined level of quality.

3.1.22 *radiance, L—*radiant flux (watts) in a light beam, emanating from a surface, or falling on a surface, in a given direction, per unit of projected area of the surface  $(m<sup>2</sup>)$  as viewed from that direction, per unit of solid angle (steradians). Has units of W m-2 sr-1. See also, *irradiance*.

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

3.1.24 *stock—*part of a dosimeter batch, held by the user.

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

3.1.26 *verification—*confirmation by examination of objective evidence that specified requirements have been met.

3.1.26.1 *Discussion—*In the case of measuring equipment, the result of verification leads to a decision to restore to service or to perform adjustments, repair, downgrade, or declare obsolete. In all cases it is required that a written trace of the verification performed be kept on the instrument's individual record.

3.2 Definitions of other terms used in this standard that pertain to radiation measurement and dosimetry may be found in Terminology E170. Definitions in Terminology [E170](#page-0-0) are compatible with ICRU 60; that document, therefore, may be used as an alternative reference.

# **4. Significance and Use**

4.1 A lithium fluoride (LiF)-based photo-fluorescent film dosimetry system provides a means of determining absorbed dose to materials by the photo-stimulated emission of wavelengths longer than that of the stimulation wavelength. The absorbed dose is obtained from the amount of the light emission. Imperfections within the ionic lattice of alkali-halide compounds such as LiF act as traps for electrons and electron holes (positively charged negative-ion vacancies). These imperfections are known as color centers because of the part they play in the compound's ability to absorb and then release energy in the form of visible-light photons. Like an atom, these color centers have discrete, allowed energy levels, and electrons can be removed from these sites when energy of the appropriate wavelength and intensity is transferred to the material. The resulting fluorescence spectra contain discrete peaks that can cover a range of wavelengths, depending upon the type of alkali-halide **[\(8\)](#page-3-0)**. An example of fluorescence spectra from a LiF-based dosimeter is provided in Fig. 1. A system of optical filters within a light-detecting instrument (that is, fluorimeter) can be used to block all but a narrow range of wavelengths that are desired for use. Theories on how color



NOTE 1-Also shown are transmission curves for green and red emission filters.

**FIG. 1 Excitation Spectrum and Resulting Fluorescence Spectrum from the Sunna LiF-based Film Dosimeter**

<span id="page-3-0"></span>centers are formed, how luminescence mechanisms work, and their application in dosimetry are found in Refs **[\(8-13\)](#page-7-0)**. For characterization studies on specific photo-fluorescent dosimeters see Refs **[\(1-7](#page-6-0))** and **[\(14-1](#page-6-0)9)**.

4.2 In the application of a specific dosimetry system, absorbed dose is determined by use of an experimentallyderived calibration curve. The calibration curve for the photofluorescent dosimeter is the functional relationship between  $\Delta E_f$  and *D*, and is determined by measuring the net fluorescence of sets of dosimeters irradiated to known absorbed doses. These absorbed doses span the range of utilization of the system.

4.3 Photo-fluorescent dosimetry systems require calibration traceable to national standards. See ISO/ASTM Guide 51261.

4.4 The absorbed dose is usually specified relative to water. Absorbed dose in other materials may be determined by applying the conversion factors discussed in ISO/ASTM Guide 51261.

4.5 During calibration and use, possible effects of influence quantities such as temperature, light exposure, post-irradiation stabilization of signal, and absorbed-dose rate need to be taken into account.

4.6 Photo-fluorescent dosimeters are sensitive to light, especially during irradiation and post-irradiation stabilization **[\(7\)](#page-4-0)**. Some color centers are sensitive to the UV and blue regions of the spectrum, while other centers are only sensitive to the UV. Therefore, they need to be packaged in appropriate light-tight packaging shortly after manufacture, and during use they need to be packaged or the appropriate filters placed over room lighting. Filtering the light fixtures involved during irradiation may be required for irradiations using low-energy X-rays or electrons where unpackaged dosimeters are used.

4.7 The signal from photo-fluorescent dosimeters either increases or decreases with time following irradiation, depending on the color center utilized **[\(19\)](#page-6-0)**. This stabilization process, which can last from hours to days depending on storage temperature (and dose for some color centers) can be accelerated and stabilized by heat treating the dosimeters after irradiation and before readout (see [9.2\)](#page-5-0).

### **5. Apparatus**

5.1 *Components of the Dosimetry System—*The following shall be used to determine absorbed dose with photofluorescent dosimetry systems:

5.1.1 *Photo-fluorescent Film Dosimeters,* being of sufficient transparency to both excitation and emission wavelengths used. Prior to calibration, data must exist that verifies that the fluorescence spectra of the batch used matches the fluorescence spectra of the batch for which performance data is published.

5.1.2 *Fluorimeter,* having lamp or light-emitting diode (LED)-based light source, excitation and emission filters, and photo-multiplier tube (PMT) or photo-diode based light detector. The fluorimeter can be of the type where the light source and light detector are on the same side of the film (that is, reflection type), or on opposite sides of the film (that is, transmission type). Documentation is needed to ensure that proper excitation and emission filters, and light detector of proper wavelength sensitivity, are utilized.

5.1.3 *Holder,* to position the dosimeter reproducibly in the path between the excitation source and detector.

5.1.4 *Fluorescence Standard,* of the appropriate wavelength, if available (see [7.9.6\)](#page-4-0), to be used during system calibration and for periodic checks on the stability of the fluorimeter response (that is, stability of excitation light and light detector).

NOTE 2—Published literature should provide the period of usefulness of the fluorescence standard under typical conditions of use, and any certificate of calibration should include an expiration date.

5.1.5 *Calibrated Laboratory Oven,* as appropriate, with a temperature stability and uniformity that results in a maximum standard deviation of  $\pm 2$  % in the response of treated dosimeters.

#### **6. Performance Check of Instrumentation**

6.1 At periodic intervals between calibrations, the individual component instruments of the dosimetry system (that is, fluorimeter and oven, as appropriate) shall have their performance verified. These performance verifications should be performed at least monthly during periods of use, and after any maintenance or modification of the instrument that may affect its performance. These periodic checks should verify the stability of the instruments, and should demonstrate that they perform as they did when the dosimetry was calibrated. Detailed instrument calibration and performance verification procedures are provided in ISO/ASTM Guide 51261.

6.2 To perform a performance check on the fluorimeter, the same fluorescence standard used during the initial system calibration (see [7.9.6\)](#page-4-0) shall be used.

## **7. Calibration of the Dosimetry System**

7.1 Prior to use, the dosimetry system (consisting of a specific batch of dosimeters and specific measurement instruments) shall be calibrated in accordance with the user's documented procedure that specifies details of the calibration process and quality assurance requirements. This calibration process shall be repeated at regular intervals to ensure that the accuracy of the absorbed dose measurement is maintained within required limits. Calibration methods are described in ISO/ASTM Guide [51261.](#page-4-0)

7.2 *Calibration Irradiation of Dosimeters—*Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations shall be performed in one of three ways by irradiating the dosimeters at:

7.2.1 An accredited calibration laboratory that provides an absorbed dose (or an absorbed-dose rate) having measurement traceability to nationally or internationally recognized standards, or

7.2.2 An in-house calibration facility that provides an absorbed dose (or an absorbed-dose rate) having measurement traceability to nationally or internationally recognized standards, or

<span id="page-4-0"></span>7.2.3 A production or research irradiation facility together with reference- or transfer-standard dosimeters that have measurement traceability to nationally or internationally recognized standards.

7.3 Determine the absorbed dose rate of the calibration field by use of a reference or transfer standard dosimetry system (see ISO/ASTM Guide 51261, Practices E1026 and E1205).

7.4 After irradiation, heat treat dosimeters, if desired, to stabilize post-irradiation growth (see [9.2.3](#page-5-0) and [9.2.4\)](#page-5-0).

7.5 If necessary to avoid light effects, keep dosimeters in their packaging until time of readout.

7.6 After irradiation, read out dosimeters (see [8.3\)](#page-5-0) to determine the fluorescence,  $E_f$ . Calculate the sample standard deviation (see ISO/ASTM Guide 51707).

7.7 If applicable, determine the net fluorescence, ∆*Ef* .

NOTE 3—Some dosimetry systems may exhibit background signals that are low and consistent enough that users can ignore the background (that is, can use  $E_f$  instead of  $\Delta E_f$ ).

7.8 For graphical analysis, plot the mean  $\Delta E_f$  (or mean  $E_f$ , as applicable) of each dosimeter set versus absorbed dose.The individual values of  $\Delta E_f$  or  $E_f$  not a mean should be used if regression analysis is used. Choose an analytical form (for example, linear, polynomial, or exponential) that provides an adequate fit to the measured data (see ISO/ASTM Guide [51707\)](#page-5-0).

### 7.9 *Measurement Instruments:*

7.9.1 For the calibration of the instruments, and for the verification of instrument performance between calibrations, see ISO/ASTM Guide [51261](#page-5-0) and/or instrument-specific operating manuals.

7.9.2 Calibrations of the individual instruments used in the analysis of the dosimeters, including an oven as appropriate, shall be performed prior to the calibration of the dosimetry system.

7.9.3 Prior to the initial calibration of the dosimetry system, the excitation and emission filters within the fluorimeter shall each be verified (documentation or certificates) to produce a peak wavelength and bandwidth (FWHM) that are within 2 % and 50 %, respectively, of values for the filters used during the performance testing of thedosimetry system used.

7.9.4 Documentation on the wavelength and bandwidth of the fluorimeter's excitation and emission filters shall provide identification numbers (serial numbers) specific to the filters used. If these filters are not easily accessible for serial number verification, then the serial number of the fluorimeter may be used.

7.9.5 The photo-detector within the fluorimeter shall be verified to have a wavelength sensitivity range appropriate for the dosimeter used.

7.9.6 A fluorescence standard of the appropriate wavelength, and having published data indicating adequate signal stability over time, shall be used to measure the fluorimeter response during the calibration of the system. This baseline fluorimeter response shall then be checked on a periodic basis to ensure the stability of the instrument over the period of its use (see Section [6\)](#page-3-0). If an appropriate nationally or internationally recognized fluorescent standard exists, the user's fluorescent standard shall have a calibration traceable to the nationally or internationally recognized standard.

### **8. Procedure**

8.1 *Examination and Pre-irradiation Storage Procedure:*

8.1.1 Prolonged exposure of unirradiated dosimeters to light can change the background signal. This can be of significance especially for low-dose applications. If needed, keep dosimeters stored in light-tight packaging or cover room lighting with appropriate filters (for example, filters that block UV or UV/blue wavelengths).

NOTE 4—Dosimeters are especially sensitive to light during the irradiation and stabilization periods (see [9.3.3\)](#page-6-0).

8.1.2 Dosimeters typically can be handled with fingers without affecting the signal.

8.1.3 Visually inspect the dosimeters, and discard any that show imperfections, such as discoloration or foreign material, that could give rise to erroneous readings.

8.1.4 Because LiF does not easily absorb water, especially if contained within a polymeric matrix that also does not readily absorb water **[\(3,](#page-5-0) [5,](#page-5-0) [7\)](#page-5-0)**, to avoid humidity effects on the dosimeter signal it is recommended that dosimeters be stored in areas that will not reach a relative humidity above 70 %, or use dosimeters packaged in hermetically-sealed pouches.

8.1.5 Although the signal from control dosimeters is typically not affected by the extremes in temperature, it is recommended practice to store dosimeters in moderatetemperature controlled areas.

8.1.6 Follow manufacturer's recommendations regarding these or other storage issues.

8.2 *Pre-Irradiation Procedure:*

8.2.1 For relatively low fluorescence signals, it may be necessary to measure the pre-irradiation fluorescence,  $E<sub>o</sub>$ , for each individual dosimeter film at the selected fluorescence wavelength or wavelength band. For relatively high fluorescence signals, it may be sufficient to use a pre-determined average  $E<sub>o</sub>$ , obtained by reading a sufficient number of randomly-selected unirradiated dosimeters within the batch.

8.2.2 Package the dosimeters or use appropriate filters over room lighting to provide controlled light exposure during irradiation (see [9.3.3\)](#page-6-0).

8.2.3 Label the package or dosimeters appropriately for identification.

8.2.4 For calibration, use an irradiation facility that meets the requirements of Section [7](#page-3-0) and follow the procedures in that section.

8.2.5 For general application of the dosimetry system in industrial process monitoring, see ISO/ASTM Practices [51204,](#page-5-0) [51608,](#page-0-0) and [51649.](#page-0-0)

NOTE 5—The dosimeters may be irradiated either in the product undergoing processing, or in a medium of similar composition. In each case, the medium should have appropriate dimensions so as to approximate electron equilibrium conditions. Such equilibrium conditions may not exist, however, within dosimeters placed throughout the product under actual processing conditions. This is particularly true near interfaces of different materials. Irradiation under non-equilibrium conditions, such as on the surface of a product package, may nevertheless be sufficient to monitor the absorbed dose delivered to the product and may under certain **E2304 − 03 (2011)**

<span id="page-5-0"></span>conditions be related to absorbed dose within the product by correction factors. For a detailed discussion of this subject, see ISO/ASTM Guide [51261](#page-6-0) and ISO/ASTM Practice [51204.](#page-0-0)

8.3 *Post-irradiation Analysis Procedure:*

8.3.1 Control light exposure to dosimeters until time of readout (see [9.3.3\)](#page-6-0).

8.3.2 At a pre-selected time after irradiation (the same time used during calibration):

8.3.2.1 Position the dosimeter in the fluorimeter as per the instrument manufacturer's instructions.

8.3.2.2 Read out the fluorescence signal,  $E_f$  of the dosimeter and record the value.

8.3.2.3 If heat treatment is used to accelerate the stabilization of the dosimeter signal, and the magnitude of the dosimeter reading has a significant dependence on when the heat treatment is performed, this treatment shall be performed at a consistent, time after irradiation (see 9.2.3 and 9.2.4).

NOTE 6—The signal from some color centers can vary during a typical readout process **(7)**. In most cases this alteration is only significant if heat treatment is not used and the same dosimeter is going to be read multiple times. Follow manufacturer's recommendations or conduct a simple test to determine if the signal is varying.

8.3.3 If applicable (see [8.2.1\)](#page-4-0), for each dosimeter calculate the net fluorescence,  $\Delta E_f = E_f - E_o$ , and record it.

8.3.4 For each set of dosimeters, calculate the associated sample mean and standard deviation of  $E_f$  (or  $\Delta E_f$ , as applicable). See ISO/ASTM Guide 51707 for information on calculating the sample mean and sample standard deviation.

#### **9. Characterization of Each Stock of Dosimeters**

#### 9.1 *Reproducibility of Fluorescence Signal:*

9.1.1 For each stock of dosimeters used, the reproducibility of the fluorescence signal shall be determined by analyzing all sets of dosimeters irradiated during the calibration procedure.

9.1.2 Use the sample standard deviations determined during calibration (see 8.3.4) to calculate coefficients of variation (CV) for each set of dosimeters. See ISO/ASTM Guide 51707 for information on calculating the CV.

9.1.3 Calculate the pooled value of CV (see ISO/ASTM Guide [51707\)](#page-6-0).

9.1.4 Document these CVs and note any that are unusually large.

NOTE 7-In general, if any CV values are greater than 2 %, then a re-determination of the data should be considered using a larger sample of dosimeters, or the stock of dosimeters rejected.

#### 9.2 *Post-Irradiation Characterization:*

9.2.1 LiF-based photo-fluorescent dosimeters will exhibit either a post-irradiation growth or fade of signal, depending on the color center observed (2, 3, 5, and [15\)](#page-6-0). Depending on the particualr color center utilized, the growth or fade can be as much as 15 to 50 %, and the time to stabilize can be from hours to many days. The time it takes for stabilization to occur is dependent on the storage temperature and, for some color centers, the absorbed dose. In order to determine whether this change in signal is significant in a given application, measure the fluorescence signal at the selected fluorescence wavelength over the anticipated period of analysis and over the range of expected storage conditions. Data from the scientific literature or the manufacturer can also be used to obtain this information.

9.2.2 In cases where the post-irradiation change of signal is slow enough, the user can select a specific time after irradiation to read out the dosimeters. This readout time shall be the same one used during calibration and must be used consistently. To determine if this change of signal is indeed slow enough for this readout protocol to be used, the user can use the slope of the curve to determine the maximum amount that this readout time can vary while still maintaining acceptable uncertainty in the measured dose.

9.2.3 If the user determines that the post-irradiation signal obtained in 9.2.1 is too fast to use the readout protocol in 9.2.2, then they have the option of (*1*) waiting for the signal to stabilize, (*2*) applying correction factors for such timedependent variations, taking into account the calibration curve for that batch of dosimeters or, (*3*) heat treating the dosimeters in a laboratory oven to quickly stabilize the signal.

NOTE 8—The post-irradiation signal from LiF-based photo-fluorescent dosimeters can be stabilized by heat treating for a relatively short duration after irradiation at temperatures around 70°C **(2, [3,](#page-6-0) [5\)](#page-6-0)**. The minimum treatment duration required depends on the color center utilized, and is typically in the range of 15 to 25 min.

9.2.4 If the user decides to heat treat the dosimeters, determine the period of time after irradiation that the heat treatment can be started without introducing appreciable error into the measured dose. This needs to be determined because the magnitude of the final stabilized value after heat treatment is dependent on when during the post-irradiation process the treatment is performed. In order to determine this heat treatment start "window," and the associated uncertainty in the measured dose from using this window, measure the fluorescence at the selected fluorescence wavelength over the range of anticipated heat treatment start times, and over the range of expected storage conditions **[\(2\)](#page-6-0)**. Data from scientific literature or the manufacturer can also be used to obtain this information.

9.2.5 For a given set of irradiation conditions, the procedures in 9.2.1, 9.2.2, and 9.2.4, which help identify the dosimeter singal's post-irradiation rate of change and heat treatment windows, need to be performed only once for a given fluorescence wavelength and model of dosimeter.

#### 9.3 *Environmental Influences:*

9.3.1 The irradiation and storage temperatures influence the signal from LiF photo-fluorescent dosimeters, and the degree of influence is dependent on the color center utilized. For these dosimeters, a typical irradiation temperature coefficient below room temperature is on the order of  $+0.5\%$  /°C to  $+1\%$  /°C and is typically not dose dependent. The irradiation temperature coefficient above room temperature is dose dependent, and can vary from -0.1 %/ $\degree$ C to +0.5 %/ $\degree$ C for both meat and sterilization dose levels.

9.3.2 Influences of ambient humidity on dosimeters before, during, and after irradiation can become significant if humidity approaches 90 % r.h. for an extended time. Typically for LiF, a maximum effect of 4 % on the dosimeter signal is observed for dosimeters stored at high humidity for 1 day or more **[\(7\)](#page-6-0)**. Although LiF does not easily absorb water, especially if contained within a polymeric matrix that also does not readily

<span id="page-6-0"></span>absorb water, it is recommended practice that dosimeters be stored in areas that do not reach a relative humidity above 70 %.

9.3.3 Light exposure to dosimeters during irradiation and post-irradiation storage influences the dosimeter signal. Package the dosimeters or use appropriate filters over room lighting to provide controlled light exposure.

9.3.4 During the characterization, calibration, and use of the dosimetry system, the effects, if any, of irradiation and storage temperature, light exposure, absorbed dose rate, and incident energy spectrum on the dosimeter response shall be determined and taken into account **[\(1-7,14,15\)](#page-7-0)**.

NOTE 9—Information regarding the magnitude of such effects on the dosemetric measurements may be obtained from sources such as scientific literature, dosimeter manufacturers, distributors, and qualified testing organizations.

### **10. Application of Dosimetry System**

10.1 The number of dosimeters required for the measurement of absorbed dose on or within a material is determined by the precision required for the application. Appendix X3 of Practice E668 describes a statistical method for determining this number.

10.2 Follow the procedures in accordance with [8.2](#page-4-0) and [8.3.](#page-5-0)

10.3 Determine the absorbed dose from the mean fluorescence signals (or net fluorescence signal as applicable) and the system calibration curve that results from following the procedures of Section [7.](#page-3-0)

10.4 Record the measured absorbed dose and all other relevant data outlined in Section 11.

# **11. Minimum Documentation Requirements**

11.1 Record the dosimeter manufacturer, type, and batch identification number.

11.2 Record or reference the date of calibration of the dosimetry system, irradiation source, and associated instruments used.

11.3 Record the environmental conditions during irradiation, including temperature, humidity, and composition of atmosphere surrounding dosimeters (if other than air).

11.4 Record the date of irradiation, the fluorescence readings, the date(s) on which the irradiated dosimeters were analyzed, and the resulting determined absorbed dose.

11.5 Record or reference the uncertainty in the determined absorbed dose.

11.6 Record or reference the measurement quality assurance plan used for the application of the dosimetry system.

# **12. Measurement Uncertainty**

12.1 To be meaningful, a measurement of absorbed dose shall be accompanied by an estimate of uncertainty.

12.2 Components of uncertainty shall be identified as belonging to one of two categories:

12.2.1 *Type A—*Those evaluated by statistical methods, or

12.2.2 *Type B—*Those evaluated by other means.

12.3 Other ways of categorizing uncertainty have been widely used and may be useful in reporting uncertainty. For example, the terms precision and bias or random and systematic (non-random) are used to describe different categories of uncertainty.

12.3.1 If this practice is followed, the estimate of the expanded uncertainty of an absorbed dose determined by this dosimetry system should be well within the 6 % value recommended in ISO/ASTM Guide [51261](#page-0-0) for routing dosimetry systems for a coverage factor  $k = 2$  (which corresponds approximately to a 95 % level of confidence for normally distributed data).

NOTE 10—The identification of Type A and Type B uncertainties are based on the methodology for estimating uncertainties published in 1993 by the International Organization for Standardization (ISO) in the Guide to the expression of Uncertainty in Measurement **[\(19\)](#page-7-0)**. The purpose of using this type of characterization is to promote an understanding of how uncertainty statements are arrived at and to provide a basis for the international comparison of measurement results.

NOTE 11-ISO/ASTM Guide [51707](#page-0-0) defines possible sources of uncertainty in dosimetry performed in radiation processing facilities, and offers procedures for estimating the magnitude of the resulting uncertainties in the measurement of absorbed dose using a dosimetry system. The document defines and discusses basic concepts of measurement, including estimation of the measured value of a quantity, "true" value, error and uncertainty. Components of uncertainty are discussed and methods are provided for estimating their values. Methods are also provided for calculating the combined standard uncertainty and estimating expanded (overall) uncertainty.

# **13. Keywords**

13.1 absorbed dose; calibration facility; dosimeter; dosimeter calibration; dosimetry system; electron beam; food irradiation; gamma ray; ionizing radiation; photo-fluorescent dosimeter; quality assurance; radiation processing; routine dosimeter

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