Designation: E2304 – 03

An American National Standard

Standard Practice for
Use of a LiF Photo-Fluorescent Film Dosimetry System[1]

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 (ε) 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 5×10^{-2} to 3×10^{2} kGy (**1-3**).[2]

1.2.2 Absorbed dose rate range of 0.3 to 2×10^{4} Gy/s (**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**).

1.2.5 Radiation temperature range of -20 to +60°C (**6**,**7**).

1.3 *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]

E170 Terminology Relating to Radiation Measurements and Dosimetry

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

E925 Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Bandwidth does not Exceed 2 nm

2.2 *ISO/ASTM Standards:*

51204 Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing

51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing

51431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing

51608 Practice for Dosimetry in an X-ray (Bremsstrahlung) Facility for Radiation Processing

51649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV

51702 Practice for Dosimetry in a Gamma Irradiation Facility for Radiation Processing

51707 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 Practice for Thermoluminescence-Dosimetry (TLD) Systems for Radiation Processing

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

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

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

ICRU Report 34 The Dosimetry of Pulsed Radiation

ICRU Report 35 Radiation Dosimetry: Electron Beams with Energies Between 1 and 50 MeV

ICRU Report 60 Fundamental Quantities and Units for Ionizing Radiation

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

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

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

[3] 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.

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

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^{-1}). 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:

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

where:

ϕ = particle fluence (m^{-2}),

E = energy of the ionizing radiation (J), and

μ_{en}/ρ = mass energy absorption coefficient (m^2 kg^{-1}).

If bremsstrahlung production within the specified material is negligible, the mass energy absorption coefficient (μ_{en}/ρ) is equal to the mass energy transfer coefficient (μ_{tr}/ρ), 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, negative- or 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, E_f*—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^{-2}. 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, ΔE_f*—measured fluorescence signal, E_f, from an irradiated sample, subtracted by the pre-irradiation fluorescence, E_o, as follows:

$$\Delta E_f = E_f - E_o$$

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^2) 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.

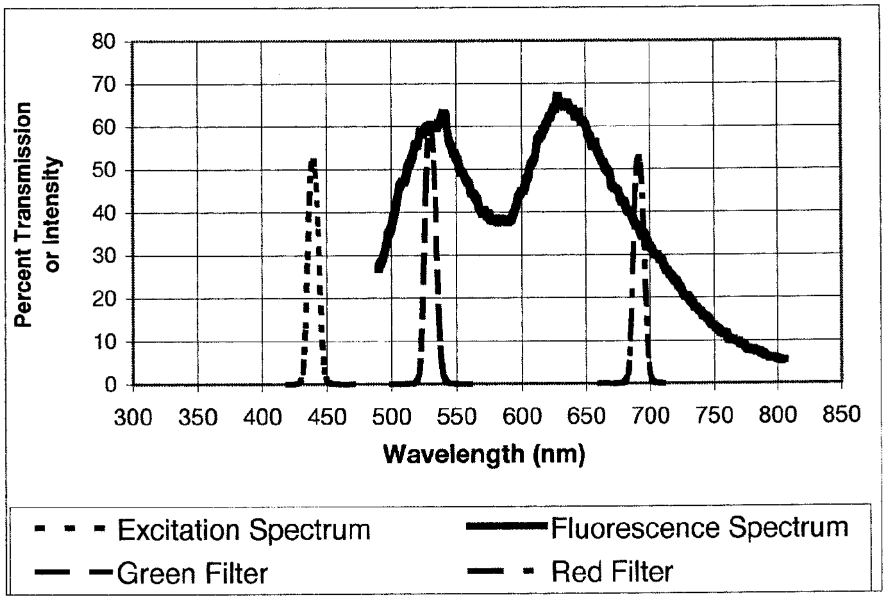
3.2 Definitions of other terms used in this standard that pertain to radiation measurement and dosimetry may be found in Terminology E170. Definitions in Terminology E170 are compatible with ICRU 60; that document, therefore, may be used as an alternative reference.

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**). 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 centers are formed, how luminescence mechanisms work, and their application in dosimetry are found in Refs (**8-13**). For characterization studies on specific photo-fluorescent dosimeters see Refs (**1-7**) and (**14-19**).

4.2 In the application of a specific dosimetry system, absorbed dose is determined by use of an experimentally-derived calibration curve. The calibration curve for the photo-fluorescent dosimeter is the functional relationship between ΔE_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 .



NOTE—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

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 .

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**). 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**). 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).

5. Apparatus

5.1 *Components of the Dosimetry System*—The following shall be used to determine absorbed dose with photo-fluorescent 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), 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 ±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 .

6.2 To perform a performance check on the fluorimeter, the same fluorescence standard used during the initial system calibration (see 7.9.6) 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 .

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

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 , Practices E1026 and E1205).

7.4 After irradiation, heat treat dosimeters, if desired, to stabilize post-irradiation growth (see 9.2.3 and 9.2.4).

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) to determine the fluorescence, E_f. Calculate the sample standard deviation (see ISO/ASTM Guide).

7.7 If applicable, determine the net fluorescence, ΔE_f.

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 ΔE_f).

7.8 For graphical analysis, plot the mean ΔE_f (or mean E_f, as applicable) of each dosimeter set versus absorbed dose.The