



Standard Practice for Use of a Radiochromic Liquid Dosimetry System¹

This standard is issued under the fixed designation ISO/ASTM 51540; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision.

1. Scope

1.1 This practice covers the procedures for preparation, handling, testing, and using radiochromic liquid dosimetry systems of radiochromic dye solutions held in sealed or capped containers (for example, ampoules, vials). It also covers the use of spectrophotometric or photometric readout equipment for measuring absorbed dose in materials irradiated by photons and electrons.

1.2 This practice applies to radiochromic liquid dosimeter solutions that can be used within part or all of the specified ranges as follows:

1.2.1 The absorbed dose range is from 0.5 to 40 000 Gy for photons and electrons.

1.2.2 The absorbed dose rate is from 10^{-3} to 10^{11} Gy/s.

1.2.3 The radiation energy range for photons is from 0.01 to 20 MeV.

1.2.4 The radiation energy range for electrons is from 0.01 to 20 MeV.

NOTE 1—Since electrons with energies less than 0.01 MeV may not penetrate the container of the solution, the solutions may be stirred in an open beaker with the electrons entering the solutions directly (1).²

1.2.5 The irradiation temperature range is from -40 to $+60^{\circ}\text{C}$.

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.*

¹ This practice is under the jurisdiction of ASTM Committee E61 on Radiation Processing and is the direct responsibility of Subcommittee E61.02. on , and is also under the jurisdiction of ISO/TC 85/WG 3.

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

2. Referenced documents

2.1 ASTM Standards:³

C912 Practice for Designing a Process for Cleaning Technical Glasses

E170 Terminology Relating to Radiation Measurements and Dosimetry

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

E666 Practice for Calculating Absorbed Dose From Gamma or X Radiation

E668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices

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

E958 Practice for Estimation of the Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers

E1026 Practice for Using the Fricke Dosimetry System

2.2 ISO/ASTM Standards:³

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

51400 Practice for Characterization and Performance of a High-Dose Gamma Radiation Dosimetry Calibration Laboratory

51707 Guide for Estimating Uncertainties in Dosimetry 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

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

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



ICRU Report 34 The Dosimetry of Pulsed Radiation
 ICRU Report 35 Radiation Dosimetry: Electron Beams with
 Energies between 1 and 50 MeV
 ICRU Report 37 Stopping Powers for Electrons and Photons
 ICRU Report 44 Tissue Substitutes in Radiation Dosimetry
 and Measurement
 ICRU Report 60 Fundamental Quantities and Units for
 Ionizing Radiation

3. Terminology

3.1 Definitions:

3.1.1 *calibration curve*—graphical representation of the dosimetry system's response function.

3.1.2 *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.

3.1.3 *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.

3.1.4 *measurement quality assurance plan*—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.

3.1.5 *molar linear absorption coefficient* (ϵ_m)—constant relating the spectrophotometric absorbance, A_λ , of an optically absorbing molecular species at a given wavelength (λ) per unit pathlength (d) to the molar concentration, c , of that species in solution (2-4): $\epsilon_m = A_\lambda/(d \times c)$. SI Unit: $\text{m}^2 \text{mol}^{-1}$.

3.1.6 *net absorbance*, ΔA —change in measured optical absorbance at a selected wavelength determined as the absolute difference between the pre-irradiation absorbance, A_0 , and the post-irradiation absorbance, A , as follows (2, 3): $\Delta A = |A - A_0|$.

3.1.7 *radiochromic liquid dosimeter*—specially prepared solution containing ingredients that undergo change in optical absorbance under ionizing radiation. This change in optical absorbance can be related to absorbed dose in water.

3.1.8 *response function*—mathematical representation of the relationship between dosimeter response and absorbed dose for a given dosimetry system.

3.1.9 *specific net absorbance* (Δk)—Net absorbance, ΔA , at a selected wavelength divided by the optical pathlength, d , through the dosimeter material as follows: $\Delta k = \Delta A/d$.

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

4. Significance and use

4.1 The radiochromic liquid dosimetry system provides a means of measuring absorbed dose in materials (5-7). Under the influence of ionizing radiation, chemical reactions take

place in the radiochromic solution modifying the amplitudes of optical absorption bands (8-10). Absorbance values are measured at the selected wavelength(s) within these affected absorption bands (see also ISO/ASTM Guide 51261).

4.2 In the use of a specific dosimetry system, a calibration curve or response function relates the dosimeter's response to an absorbed dose traceable to a nationally or internationally recognized standard (11, 12).

4.3 The absorbed dose that is measured is usually specified in water. Absorbed dose in other materials may be evaluated by applying the conversion factors discussed in ISO/ASTM Guide 51261.

NOTE 2—For a comprehensive discussion of various dosimetry methods applicable to the radiation types and energies discussed in this practice, see ICRU Reports 14, 17, 34, 35, and 37.

4.4 These dosimetry systems may be used in the industrial radiation processing of a variety of products, for example the sterilization of medical devices and radiation processing of foods (5, 7, 13).

4.5 The available dynamic range indicated in 1.2.1 is achieved by using a variety of radiochromic leuco dyes (Table 1) in a variety of solutions (Table 2).

4.6 The ingredients of the solutions, in particular the solvents, can be varied so as to simulate a number of materials in terms of the photon mass energy-absorption coefficients, (μ_{en}/ρ) , for X-rays and gamma-rays and electron mass collision stopping powers, $[(1/\rho) dE/dx]$, over a broad spectral energy range from 0.01 to 100 MeV (18). For special applications certain tissue-equivalent radiochromic solutions have been designed to simulate various materials and anatomical tissues, in terms of values of (μ_{en}/ρ) for photons and $[(1/\rho) dE/dx]$ for electrons (18) (see also ICRU Report 44). Tabulations of the values of (μ_{en}/ρ) for water (19), the anatomical tissues (17, 19), and three specially designed radiochromic solutions, for photons over the energy range from 0.01 to 20 MeV, and tabulations of the values of $[(1/\rho) dE/dx]$ (17) for water, the tissues and the radiochromic solutions for electrons over the energy range from 0.01 to 20 MeV are given in Refs (12, 13, 18). For additional information see ISO/ASTM Guide 51261, ASTM Practice E666, and ICRU Reports 14, 17, 35, 37, and 44.

5. Apparatus

5.1 The following shall be used to determine absorbed dose with radiochromic liquid dosimetry systems:

5.1.1 Batch or Portion of a Batch of Radiochromic Liquid.

5.1.2 *Spectrophotometer or Photometer*, having documentation covering analysis wavelengths, accuracy of wavelength selection, absorbance determination, analysis bandwidth, and stray light rejection. The spectrophotometer should be able to read visible spectrum absorbance values of up to 2 with an uncertainty of no more than $\pm 1\%$.

5.1.3 *Glass Cuvettes*, having optical windows and path lengths of 5 to 100 mm, depending on the dose range of interest and on the size of the dosimeter ampoule used for irradiation.

TABLE 1 Three Available Radiochromic Leuco Dyes, Their Molecular Structures, Molecular Weights, and Values of ϵ_m and Color Index Numbers of the Parent Dyes (14, 15)

Radiochromic Leuco Dye (code)	Molecular Structure	Molecular Weight	Molar Linear Absorption Coefficient ^A (L mol ⁻¹ cm ⁻¹)	Color Index No.
Pararosaniline cyanide (PRC)	(See diagram below left)	314.376	140 000 ($\lambda = 550$ nm)	42 500
Hexa(hydroxyethyl)pararosaniline cyanide (HHEVC)	(See diagram below center)	578.715	100 000 ($\lambda = 600$ nm)	(none given)
New fuchsin cyanide (NFC)	(See diagram below right)	356.455	130 000 ($\lambda = 560$ nm)	42 500

^AThese values of molar linear absorption coefficients are given in Ref (14, 16) for 2-methoxyethanol solutions containing 17 mM acetic acid. The values may vary somewhat in other solvents and with other additives.

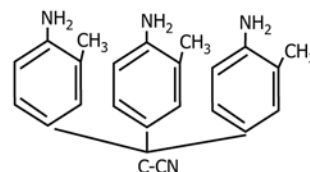
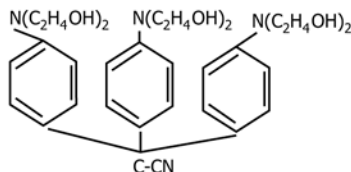
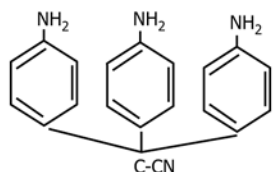


TABLE 2 Selected Radiochromic Solution Formulations and the Radiation Chemical Yields of Dye Cations in Solution

Radiochromic Leuco Dye (See Table 1)	Solution Formulation	Radiochromic Leuco Dye Concentration (mmol L ⁻¹)	Wavelength for Spectrophotometer, nm	Radiation Chemical Yield, $\mu\text{mol J}^{-1}$	Nominal Dose Range, Gy	References
HHEVC	Dissolve in 2-methoxy ethanol containing 17 mmol L ⁻¹ acetic acid	5	599	0.025	10–1000	(5)
PRC	Dissolve in 2-methoxy ethanol containing 51 mmol L ⁻¹ acetic acid	5	549	0.033	10–3000	(1)
NFC	Dissolve in dimethyl sulfoxide containing 17 mmol L ⁻¹ acetic acid	0.1	554	0.0031	100–30 000	(14)
PRC	Dissolve in dimethyl sulfoxide containing 17 mmol L ⁻¹ acetic acid and 30 mmol L ⁻¹ nitrobenzene	5	554	0.0040	3–40 000	(11)
HHEVC	Dissolve in mixture of 85 % n-propanol and 15 % triethylphosphate (by volume), containing 34 mmol L ⁻¹ acetic acid, 500 parts-per-million nitrobenzoic acid and 10 % polyvinyl butyral (by weight)	2	605	0.0051	50–5000	(15)
NFC	Dissolve in mixture of 85 % triethylphosphate and 15 % dimethyl sulfoxide (by volume), containing 68 mM acetic acid, 500 parts-per-million nitrobenzoic acid and 10 % polyvinyl butyral (by weight)	2	557	0.0055	100–10 000	(12)
HHEVC	Dissolve in mixture of 85 % triethylphosphate and 15 % dimethyl sulfoxide (by volume), containing 68 mM acetic acid, 500 parts-per-million nitrobenzoic acid and 10 % polyvinyl butyral (by weight)	100	608	0.28	0.5–10	(17)

Glass flow cells with parallel optical windows may be an alternative means of holding the solutions for spectrophotometry.

5.1.4 Clean *glass containers* may be used for storage or irradiation of the solutions.

5.1.4.1 Containers for storing the solutions should have glass, aluminum, or polyethylene liners for the lids. The lids should be compatible with the unirradiated and irradiated solution.

5.1.4.2 Use glass ampoules which are flame sealed for containing the solution during irradiation, or alternatively, glass vials with lids having aluminum or polyethylene liners, or disposable plastic vials, using only polymeric materials known to be resistant to any chemical effects by the solvents that are used. Another type of container for irradiation may be a cuvette

equipped with a tightly closed cap. The solution should be stored at <30°C in the dark.

NOTE 3—Any glass container should be cleaned with laboratory distilled water and detergent, rinsed with doubly distilled water and then with ethanol, dried at elevated temperature (>300°C) and cooled to ambient laboratory temperature before being used to hold the dosimetric solution. For more detail on cleaning glassware, see ASTM Practice C912.

NOTE 4—The glass ampoules or vials for irradiation commonly have capacities of 2 to 5 mL. The glass is commonly amber to protect the solution from stray ultraviolet light.



6. Performance check of instrumentation

6.1 Check and document the performance of the photometer or spectrophotometer. (For detailed information on these performance checks, see ASTM Practices E275, E925, and E958.) Use reference standards traceable to national or international standards.

6.1.1 When using a photometer, estimate and document the accuracy of the absorbance at time intervals not to exceed one month during periods of use, or whenever there are indications of poor performance.

6.1.2 When using a spectrophotometer, estimate and document the accuracy of the wavelength and absorbance at or near the selected analysis wavelength(s) at time intervals not to exceed one month during periods of use, or whenever there are indications of poor performance.

6.1.3 Document the comparison of information obtained in 6.1.1 or 6.1.2 with the original instrument specification to verify adequate performance and take appropriate corrective action if required.

7. Preparation of dosimeters

7.1 *Solvents*—The solvents for dissolving radiochromic dye precursors include a number of liquid polar organic solvents, reagent grade or better. Examples include: ethanol, isopropanol, *n*-propanol, 2-methoxyethanol, 2-ethoxy-ethanol, *N,N*-dimethylformamide, dimethylsulfoxide, triethylphosphate (1, 9-11, 14-16, 18). The choice of the solvent depends on which dosimeter formulation is needed for a given use (see 7.3). Use a solvent from a bottle that has not previously been opened if the solvent is likely to degrade after opening (for example, by forming peroxides).

7.2 *Dye Precursors*—The radiochromic dye precursor solutes for liquid dosimeters include the leuco dyes listed in Table 1.

7.3 *Formulations, Molar Linear Absorption Coefficients, and Radiation Chemical Yields*—A variety of combinations of solvents, radiochromic dyes, and selective additives are possible as indicated in the literature. Table 1 lists nominal values of molar linear absorption coefficients, also derived from Refs (14, 16).

7.4 *Preparation of Dosimeter Solutions*—Table 2 lists representative formulations of radiochromic liquid dosimeters.

7.4.1 These solutions are made at room temperature in a covered Erlenmeyer flask, using a magnetic stirring apparatus or other automatic stirring system. Stirring should be carried out long enough to ensure complete dissolution. It is preferable in each case to dissolve the additives (for example, acetic acid, nitrobenzoic acid, polyvinyl butyral) before adding the leuco dye.

7.4.2 Exercise care in filling ampoules to avoid depositing solution in the ampoule neck. Subsequent heating during sealing may cause an undesirable chemical change in the dosimetric solution remaining in the ampoule's neck. For the same reason, exercise care to avoid heating the body of the ampoule during sealing. (**Warning**—Some leuco dye solutes and some solvents may be flammable or toxic or may become

an irritant on extended exposure. Appropriate precautions as recommended by the suppliers of ingredients shall be exercised.

NOTE 5—Some of the solutions listed in Table 2 are supplied as standard reference dosimeters, with well-characterized linear responses over specified dose ranges, irradiation temperature-dependence values, radiation chemical yields, and linear molar absorption coefficients (12, 16). Such solutions do not always need calibration and may be used with appropriate radiation chemical yields and values of ϵ_m at an assigned spectrophotometric wavelength for the evaluation of absorbed dose in water (see (16) and ASTM Practice E1026). When preparing solutions from ingredients as described in Table 2, each new solution should be calibrated since batches of commercially supplied leuco dyes may vary in quality (5, 9, 11, 13).

8. Calibration of the dosimetry system

8.1 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 procedure 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).

8.2 *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:

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

8.2.2 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

8.2.3 A production or research irradiation facility together with reference or transfer standard dosimeters that have measurement traceability to nationally or internationally recognized standards.

8.3 When the radiochromic liquid dosimeter is used as a reference or transfer standard dosimeter, the calibration irradiation may be performed only as stated in 8.2.1, or in 8.2.2 at a facility that meets the requirements in Practice ISO/ASTM 51400.

8.4 *Measurement Instrument Calibration and Performance Verification*—For the calibration of the instruments, and for the verification of instrument performance between calibrations, see ISO/ASTM Guide 51261 and/or instrument-specific operating manuals.

8.5 Fig. 1 shows a calibration curve for a typical radiochromic liquid dosimeter.

9. Measurement and analysis

9.1 Depending on the radiochromic solution used (see Tables 1 and 2), set the spectrophotometer at the appropriate wavelength. Use the same bandwidth for calibrations and routine measurements.

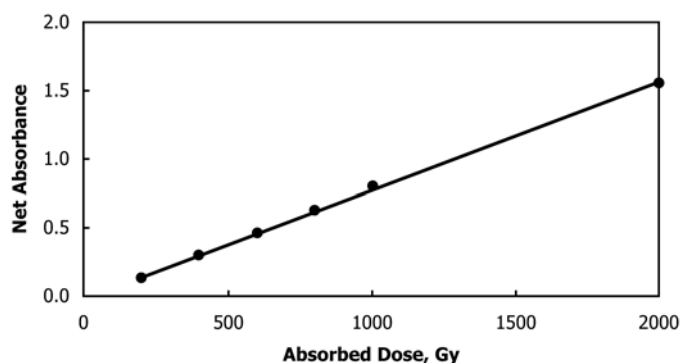


FIG. 1 Calibration Curve of a Typical Radiochromic Liquid Dosimeter [2 mM HHEVC in a Mixture of 85 % TEP and 15 % DMSO (by Volume), Containing 68 mmol·L⁻¹ Acetic Acid and 500 ppm Nitrobenzoic Acid and 10 % PVB (by Weight)], in Terms of $\Delta A_{608 \text{ nm}}$ Versus D (19)

9.2 Set the balance of the spectrophotometer to zero with only air (no cuvette or flow cell) in the light path(s).

NOTE 6—For the formulations in Table 2, control of temperature during spectrophotometry is not essential because the temperature coefficient during spectrophotometric measurements is between 0 and -0.01 % per degree Celsius. However, the temperature during measurement should be within the temperature range from 20 to 30°C.

9.3 Select a clean cuvette of a selected optical pathlength. Fill the cuvette with the solvent (or solvent mixture) used for the radiochromic solution being calibrated, and measure the absorbance (with air only in the reference beam of the spectrophotometer). Record this value ($A_{s,0}$).

NOTE 7—Choice of the cuvette pathlength depends on the maximum absorbance that can be measured accurately by the spectrophotometer and on the dose range and dosimetric solution's concentration chosen for a given calibration.

9.4 Empty the cuvette and rinse at least once with the dosimeter solution from an ampoule. Discard the rinse portion of the solution and fill the cuvette to the appropriate level with more solution from the same ampoule. Carefully wipe off any solution on the exterior surface of the cuvette and measure the absorbance. Repeat this procedure for all unirradiated and irradiated solutions, rinsing with blank solvent or solution between ampoules.

NOTE 8—Inadequate rinsing of the cuvette can lead to errors due to solution carryover (cross-contamination). Techniques for minimizing this effect are discussed in Refs (13) and (14).

9.5 Periodically during the measurement process, remeasure the absorbance of the solvent (or solvent mixture), A_s , first rinsing the cuvette with the solvent (or solvent mixture). Compare A_s with $A_{s,0}$ in order to detect and correct for any drift in the zero balance of the spectrophotometer or contamination of the cuvette.

9.6 Always check zero reading with only air in the light paths of the instrument.

9.7 Using a mean absorbance, A_0 , of the unirradiated dosimeters, calculate the net absorbance, ΔA_i , for each irradiated dosimeter.

10. Use of dosimetry systems

10.1 Use a minimum of two dosimeters for each dose measurement. The number of dosimeters required for the measurement of absorbed dose at a location on or within a material is determined by the uncertainty of the dosimetry system and the acceptable uncertainty associated with the use. Appendix X3 of ASTM Practice E668 describes a statistical method for determining this number.

10.2 Control or monitor the temperature of the dosimeters during irradiation. Take into account any temperature variations that affect dosimeter response (that is, specific net absorbance). For the formulations in Table 2 the temperature dependence of dosimeter response during irradiation between 20 and 50°C is -0.2 % per degree Celsius (13).

10.3 Determine the absorbed dose values from the net absorbance values and the calibration curve or response function.

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

11. Minimum documentation requirements

11.1 Calibration:

11.1.1 Record the dosimeter type and batch number (code).

11.1.2 Record or reference the irradiation date, irradiation temperature, temperature variation (if any), dose range, radiation source, and associated instrumentation used to calibrate and analyze the dosimeters.

11.2 Application:

11.2.1 Record the date and temperature of irradiation, temperature variation (if any), and the date and temperature of absorbance measurement, for each dosimeter.

11.2.2 Record or reference the radiation source type and characteristics.

11.2.3 Record the absorbance, net absorbance value, temperature correction (if applicable), and resulting absorbed dose for each dosimeter. Reference the calibration curve or response function used to obtain the absorbed dose values.

11.2.4 Record or reference the uncertainty in the value of the absorbed dose.

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

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 for reporting uncertainty. For example, the terms *precision* and *bias* or *random* and *systematic* (non-random) are used to describe different categories of uncertainty.

12.4 If this practice is followed, the estimate of the expanded uncertainty of an absorbed dose determined by this



dosimetry system should be less than 6 % for a coverage factor $k=2$ (which corresponds approximately to a 95 % level of confidence for normally distributed data).

NOTE 9—The identification of Type A and Type B uncertainties is based on the methodology for estimating uncertainties published in 1995 by the International Organization for Standardization (ISO) in the Guide to the Expression of Uncertainty in Measurement (20). The purpose of this type of characterization is to promote an understanding of how uncertainty statements are developed and to provide a basis for the international comparison of measurement results.

NOTE 10—ISO/ASTM Guide 51707 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; dosimetry; electron beams; gamma radiation; ionizing radiation; liquid dosimeter; quality control; radiation processing; radiochromic dosimetry; radiochromic liquid; radiochromic solution; ICS 17.240

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⁵ Available from the International Organization for Standardization, 1 rue de Varembe, Case Postale 56, CH-1211, Geneva 20, Switzerland.



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