



Standard Practice for Use of a Ceric-Cerous Sulfate Dosimetry System¹

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1. Scope

1.1 This practice covers the preparation, testing, and procedure for using the ceric-cerous sulfate dosimetry system to measure absorbed dose in water when exposed to ionizing radiation. The system consists of a dosimeter and appropriate analytical instrumentation. For simplicity, the system will be referred to as the ceric-cerous system. It is classified as a reference standard dosimetry system (see Guide E 1261). Ceric-cerous dosimeters are also used as transfer standard dosimeters or routine dosimeters.

1.2 This practice describes both the spectrophotometric and the potentiometric readout procedures for the ceric-cerous systems.

1.3 This practice applies only to γ rays, X rays, and high energy electrons.

1.4 This practice applies provided the following are satisfied:

1.4.1 The absorbed-dose range shall be between 5×10^2 and 5×10^4 Gy (1).²

1.4.2 The absorbed-dose rate shall be less than 10^6 Gy/s (1).

1.4.3 For radionuclide gamma-ray sources, the initial photon energy shall be greater than 0.6 MeV. For bremsstrahlung photons, the initial energy of the electrons used to produce the bremsstrahlung photons shall be equal to or greater than 2 MeV. For electron beams, the initial electron energy shall be greater than 8 MeV.

NOTE 1—The lower energy limits are appropriate for a cylindrical dosimeter ampoule of 12-mm diameter. Corrections for dose gradients across an ampoule of that diameter or less are not required for photons, but may be required for electron beams (2). The ceric-cerous system may be used at lower energies by employing thinner (in the beam direction) dosimeter containers (see ICRU Report 35).

1.4.4 The irradiation temperature of the dosimeter shall be between 0 and 62°C (3).

NOTE 2—The temperature coefficient of dosimeter response is known only in this range. For use outside this range, the dosimetry system should be calibrated for the required range of irradiation temperatures.

1.5 *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:

C 912 Practice for Designing a Process for Cleaning Technical Glasses³

D 941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer⁴

D 1193 Specification for Reagent Water⁵

E 170 Terminology Relating to Radiation Measurements and Dosimetry⁶

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods⁷

E 178 Practice for Dealing with Outlying Observations⁷

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers⁸

E 456 Terminology Related to Quality and Statistics⁷

E 666 Practice for Calculating Absorbed Dose from Gamma or X Radiation⁶

E 668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices⁶

E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers⁸

E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers⁸

E 1026 Practice for Using the Fricke Reference Standard Dosimetry System⁶

E 1261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing⁶

E 1400 Practice for Characterization and Performance of a High-Dose Gamma Radiation Dosimetry Calibration Laboratory⁶

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

³ *Annual Book of ASTM Standards*, Vol 15.02.

⁴ Discontinued; see 1993 *Annual Book of ASTM Standards*, Vol 05.01.

⁵ *Annual Book of ASTM Standards*, Vol 11.01.

⁶ *Annual Book of ASTM Standards*, Vol 12.02.

⁷ *Annual Book of ASTM Standards*, Vol 14.02.

⁸ *Annual Book of ASTM Standards*, Vol 03.06.

E 1401 Practice for Use of a Dichromate Dosimetry System⁶

E 1607 Practice for Use of the Alanine-EPR Dosimetry System⁶

E 1707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing⁶

2.2 *International Commission on Radiation Units and Measurements (ICRU) Reports:*⁹

ICRU Report 14 - Radiation Dosimetry: X-Rays and Gamma Rays with Maximum Photon Energies Between 0.6 and 60 MeV

ICRU Report 33 - Radiation Quantities and Units

ICRU Report 34 - The Dosimetry of Pulsed Radiation

ICRU Report 35 - Radiation Dosimetry: Electrons with Initial Energies Between 1 and 50 MeV

ICRU Report 37 - Stopping Powers for Electrons and Positrons

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 Gy is equivalent to the absorption of 1 J/kg of the specified material (1 Gy = 1 J/kg). The mathematical relationship is the quotient of $d\bar{\epsilon}$ by dm , where $d\bar{\epsilon}$ is the mean incremental energy imparted by ionizing radiation to matter of incremental mass dm (see ICRU 33).

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

3.1.1.1 *Discussion*—The discontinued unit for absorbed dose is the rad (1 rad = 100 erg/g = 0.01 Gy). Absorbed dose is sometimes referred to simply as dose. For a photon source under conditions of charged particle equilibrium, the absorbed dose, D , may be expressed as:

$$D = \phi \cdot E \cdot \frac{\mu_{en}}{\rho} \quad (2)$$

where:

ϕ = particle fluence (particles/m²),

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

μ_{en}/ρ = mass energy absorption coefficient (m²/kg). 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 *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.

3.1.3 *electropotential*—difference in potential, E , between irradiated and unirradiated solutions in an electrochemical cell, measured in millivolts.

3.1.4 *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.

3.1.5 *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.

3.1.6 *molar linear absorption coefficient, ϵ_m* —a 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:

$$\epsilon_m = \frac{A_\lambda}{d \cdot c} \quad (3)$$

SI unit: m²mol⁻¹

3.1.6.1 *Discussion*—The measurement is sometimes expressed in units of L mol⁻¹ cm⁻¹.

3.1.7 *net absorbance, ΔA* —change in measured optical absorbance at a selected wavelength determined as the absolute difference between the pre-irradiation absorbance, A_o , and the post-irradiation absorbance, A , as follows:

$$\Delta A = |A - A_o| \quad (4)$$

3.1.8 *radiation chemical yield, $G(\chi)$* —the quotient of $n(\chi)$ by $\bar{\epsilon}$, where $n(\chi)$ is the mean amount of a specified entity, χ , produced, or changed by the mean energy, $\bar{\epsilon}$, imparted to the matter.

$$G(\chi) = \frac{n(\chi)}{\bar{\epsilon}} \quad (5)$$

SI unit: mol J⁻¹

3.1.9 *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.

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

3.2 For definitions of other terms used in this practice that pertain to radiation measurement and dosimetry, refer to Terminology E 170. Definitions in Terminology E 170 are compatible with ICRU 33; that document, therefore, may be used as an alternative reference.

4. Significance and Use

4.1 The ceric-cerous system provides a reliable means for measuring absorbed dose in water. It is based on a process of reduction of ceric ions to cerous ions in acidic aqueous solution by ionizing radiation (**1, 4**).

4.2 The dosimeter is a solution of ceric sulfate and cerous sulfate in sulfuric acid in an appropriate container such as a

⁹ Available from International Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814, USA.

flame-sealed glass ampoule. The solution indicates a level of absorbed dose by a change (decrease) in optical absorbance at a specified wavelength in the ultraviolet region, or a change (increase) in electropotential. A calibrated spectrophotometer is used to determine the change in absorbance and a potentiometer, with a specially designed cell, is used to determine the change in potential in millivolts.

4.3 The dosimeter response has a temperature dependence during irradiation that is approximately equal to -0.2% per degree Celsius between 0 and 62°C (3, 5, 6).

4.4 For calibration with photons, the ceric-cerous dosimeter shall be irradiated under conditions that approximate electron equilibrium.

4.5 The absorbed dose in other materials irradiated under equivalent conditions may be calculated. Procedures for making such calculations are given in Practices E 666 and E 668 and Guide E 1261.

5. Interferences

5.1 The ceric-cerous dosimetric solution response is sensitive to impurities, particularly organic impurities. Even in trace quantities, impurities can cause a detectable change in the observed response (7). For high-accuracy results, organic materials shall not be used for any component in contact with the solution. The effect of trace impurities is minimized by the addition of cerous ions to the solution (8, 9)

5.2 Undesirable chemical changes in the dosimetric solution can occur if care is not taken during flame-sealing of the ampoules (see 8.7).

6. Apparatus

6.1 *Spectrophotometric Method*—For the analysis of the dosimetric solution, use a high-precision spectrophotometer capable of measuring absorbance values up to two with an uncertainty of no more than $\pm 1\%$ in the region from 254 to 320 nm. Use quartz cuvettes with 10-mm path length for spectrophotometric measurements of absorbance of the solution.

6.2 *Potentiometric Method*—Use an electrochemical cell, similar to that in Appendix X1 (see Fig. X1.1). Measure the electropotential across the cell with a high-precision digital potentiometer that is capable of measuring d-c potentials in the range from 1 to 100 mV within an uncertainty of $\pm 1\%$.

NOTE 3—As shown in Fig. X1.1, the electrochemical cell has two compartments separated by a porous junction, such as a glass frit, a ceramic or kaolin junction, or a fibreglass wick. The inner compartment is filled with unirradiated solution. The lower compartment is filled with solution transferred from an irradiated or unirradiated ampoule. The potential difference, E , generated between the platinum electrodes in the two compartments is measured by a digital potentiometer.

6.3 *Glassware*—Use borosilicate glass or equivalent chemically resistant glass to store the reagents and the prepared dosimetric solution. Clean all glassware, except ampoules,

using chromic acid solution or an equivalent cleaning agent (see Practice C 912). Rinse at least three times with double-distilled water. Dry thoroughly and store under conditions that will minimize exposure to dust.

6.4 *Glass Ampoules*—If required, clean glass ampoules in boiling double-distilled water. Rinse twice with double-distilled water and oven dry.

NOTE 4—The dosimetric ampoule normally used has a capacity of approximately 2 mL. Quick-break glass ampoules, or Type 1 glass colorbreak ampoules or equivalent containers, are commonly used. Commercially available ampoules have been found to give reproducible results without requiring additional cleaning.

7. Reagents

7.1 Analytical reagent grade (or better) chemicals shall be used for preparing all solutions.¹⁰

7.2 Use of double-distilled water from coupled all-glass and silica stills is recommended for high-range dosimeters. For low-range stock solutions, use triply-distilled water. Water purity is very important since it is the major component of the dosimetric solutions, and therefore may be the prime source of contamination. Use of deionized water is not recommended. Type III reagent water as specified in Specification D 1193 is considered to be of sufficient quality for use in preparing all solutions.

NOTE 5—Double-distilled water distilled from an alkaline potassium permanganate (KMnO_4) solution (2 g KMnO_4 plus 5 g sodium hydroxide (NaOH) pellets in 2 L of distilled water) has been found to be adequate for routine preparation of the dosimetric solution. High-purity water is commercially available from some suppliers. Such water labeled HPLC (high-pressure liquid chromatographic) grade is usually sufficiently free from organics to be used in this practice.

7.3 Do not store purified water used in this practice in plastic containers or in containers with plastic caps or plastic cap liners.

8. Preparation of the Dosimetric Solution

8.1 The recommended concentrations for the ceric-cerous dosimeter to measure absorbed doses from about 5 to 50 kGy (high-range dosimeter) are 0.015- M ceric sulfate [$\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$] and 0.015- M cerous sulfate [$\text{Ce}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$]. For measurement of absorbed doses from about 0.5 to 10 kGy (low-range dosimeter), the recommended concentrations are 0.003- M [$\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$] and 0.003- M [$\text{Ce}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$].

8.2 The dosimeters specified in 8.1 may be formulated from the following nominal stock solutions: (a) 0.4- M and 4- M sulfuric acid (H_2SO_4), (b) 0.1- M $\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}$, and (c) 0.1- M $\text{Ce}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}$. Procedures for preparing these solutions are given in Appendix X2.

¹⁰ Reagent specifications are available from American Chemical Society, 1115 16th St., Northwest, Washington, DC 20036, USA.