



# Standard Practice for Using the Fricke Dosimetry System<sup>1</sup>

This standard is issued under the fixed designation ISO/ASTM 51026; 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, testing and using the acidic aqueous ferrous ammonium sulfate solution dosimetry system to measure absorbed dose to water when exposed to ionizing radiation. The system consists of a dosimeter and appropriate analytical instrumentation. The system will be referred to as the Fricke dosimetry system. The Fricke dosimetry system may be used as either a reference standard dosimetry system or a routine dosimetry system.

1.2 This practice is one of a set of standards that provides recommendations for properly implementing dosimetry in radiation processing, and describes a means of achieving compliance with the requirements of ISO/ASTM Practice [52628](#) for the Fricke dosimetry system. It is intended to be read in conjunction with ISO/ASTM Practice [52628](#).

1.3 The practice describes the spectrophotometric analysis procedures for the Fricke dosimetry system.

1.4 This practice applies only to gamma radiation, X-radiation (bremsstrahlung), and high-energy electrons.

1.5 This practice applies provided the following are satisfied:

1.5.1 The absorbed dose range shall be from 20 to 400 Gy **(1)**.<sup>2</sup>

1.5.2 The absorbed-dose rate does not exceed  $10^6$  Gy·s<sup>-1</sup> **(2)**.

1.5.3 For radioisotope gamma sources, the initial photon energy is greater than 0.6 MeV. For X-radiation (bremsstrahlung), the initial energy of the electrons used to produce the photons is equal to or greater than 2 MeV. For electron beams, the initial electron energy is greater than 8 MeV.

NOTE 1—The lower energy limits given are appropriate for a cylindrical dosimeter ampoule of 12 mm diameter. Corrections for displacement effects and dose gradient across the ampoule may be required for electron beams **(3)**. The Fricke dosimetry system may be used at lower energies by

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee [E61](#) on Radiation Processing and is the direct responsibility of Subcommittee [E61.02](#) on Dosimetry Systems and is also under the jurisdiction of ISO/TC 85/WG 3.

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<sup>2</sup> The boldface numbers that appear in parentheses refer to a bibliography at the end of this practice.

employing thinner (in the beam direction) dosimeter containers (see ICRU Report 35).

1.5.4 The irradiation temperature of the dosimeter should be within the range of 10 to 60°C.

1.6 *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>

[C912 Practice for Designing a Process for Cleaning Technical Glasses](#)

[E170 Terminology Relating to Radiation Measurements and Dosimetry](#)

[E178 Practice for Dealing With Outlying Observations](#)

[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](#)

### 2.2 ISO/ASTM Standards:<sup>3</sup>

[51261 Practice for Calibration of Routine Dosimetry Systems for Radiation Processing](#)

[51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing](#)

[52628 Practice for Dosimetry in Radiation Processing](#)

### 2.3 ISO/IEC Standard:

[ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories<sup>4</sup>](#)

<sup>3</sup> For referenced ASTM and ISO/ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.



2.4 *International Commission on Radiation Units and Measurements (ICRU) Reports:*<sup>5</sup>

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

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

ICRU Report 64 Dosimetry of High-Energy Photon Beams based on Standards of Absorbed Dose to Water

ICRU Report 80 Dosimetry Systems for Use in Radiation Processing

ICRU Report 85a Fundamental Quantities and Units for Ionizing Radiation

2.5 *Joint Committee for Guides in Metrology (JCGM) Reports:*<sup>6</sup>

JCGM 100:2008 GUM 1995, with minor corrections, Evaluation of measurement data – Guide to the expression of uncertainty in measurement

2.6 *National Research Council Canada (NRCC):*  
PIRS-0815 The IRS Fricke Dosimetry System<sup>7</sup>

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *approved laboratory*—laboratory that is a recognized national metrology institute; or has been formally accredited to ISO/IEC 17025; or has a quality system consistent with the requirements of ISO/IEC 17025.

3.1.1.1 *Discussion*—A recognized national metrology institute or other calibration laboratory accredited to ISO/IEC 17025 should be used in order to ensure traceability to a national or international standard. A calibration certificate provided by a laboratory not having formal recognition or accreditation will not necessarily be proof of traceability to a national or international standard.

3.1.2 *molar linear absorption coefficient* ( $\epsilon_m$ )—a constant relating the spectrophotometric absorbance ( $A_\lambda$ ) of an optically absorbing molecular species at a given wavelength ( $\lambda$ ) per unit pathlength ( $d$ ) to the molar concentration ( $c$ ) of that species in solution:

$$\epsilon_m = \frac{A_\lambda}{(d \times c)} \quad (1)$$

Unit:  $\text{m}^2 \cdot \text{mol}^{-1}$

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

$$G(x) = \left( \frac{n(x)}{\bar{\epsilon}} \right) \quad (2)$$

Unit:  $\text{mol} \cdot \text{J}^{-1}$

<sup>5</sup> Available from International Commission on Radiation Units and Measurements (ICRU), 7910 Woodmont Ave., Suite 400, Bethesda, MD 20841-3095, <http://www.icru.org>.

<sup>6</sup> Document produced by Working Group 1 of the Joint Committee for Guides in Metrology (JCGM/WG1). Available free of charge at the BIPM website (<http://www.bipm.org>).

<sup>7</sup> Available from the National Research Council, Ionizing Radiation Standards, Ottawa, Ontario, K1A 0R6.

3.1.4 *reference standard dosimetry system*—dosimetry system, generally having the highest metrological quality available at a given location or in a given organization, from which measurements made there are derived.

3.1.5 *type I dosimeter*—dosimeter of high metrological quality, the response of which is affected by individual influence quantities in a well-defined way that can be expressed in terms of independent correction factors.

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 E170 are compatible with ICRU 85a; that document, therefore, may be used as an alternative reference.

### 4. Significance and use

4.1 The Fricke dosimetry system provides a reliable means for measurement of absorbed dose to water, based on a process of oxidation of ferrous ions to ferric ions in acidic aqueous solution by ionizing radiation (ICRU 80, PIRS-0815,(4)). In situations not requiring traceability to national standards, this system can be used for absolute determination of absorbed dose without calibration, as the radiation chemical yield of ferric ions is well characterized (see Appendix X3).

4.2 The dosimeter is an air-saturated solution of ferrous sulfate or ferrous ammonium sulfate that indicates absorbed dose by an increase in optical absorbance at a specified wavelength. A temperature-controlled calibrated spectrophotometer is used to measure the absorbance.

### 5. Effect of influence quantities

5.1 The Fricke dosimeter response (change in optical absorbance) to a given radiation dose is dependent on irradiation temperature and measurement temperature. Thus, corrections may have to be applied for changes to the radiation chemical yield ( $G$ ) for irradiation temperature and to the molar linear absorption coefficient ( $\epsilon$ ) for measurement temperatures. Both  $\epsilon(\text{Fe}^{3+})$  and  $G(\text{Fe}^{3+})$  increase with increase in temperature. The subscripts indicate the temperature of irradiation and measurement, as applicable. Both of the temperatures are in °C.

$$\epsilon_{T_{meas}} = \epsilon_{25} [1 + 0.0069 (T_{meas} - 25)] \quad (3)$$

$$G_{T_{irrad}} = G_{25} [1 + 0.0012 (T_{irrad} - 25)] \quad (4)$$

5.2 The radiation chemical yield depends on the type and energy of the radiation employed and, in particular, changes significantly at low photon energies (5).

### 6. Interferences

6.1 The Fricke dosimeter response is extremely sensitive to impurities in the solution, particularly organic impurities. Even in trace quantities, impurities can cause a detectable change in the observed response. For high accuracy, organic materials shall not be used for any component in contact with the solution, unless it has been demonstrated that the materials do not affect the dosimeter response.



6.2 Traces of metal ions in the irradiated and unirradiated dosimetric solutions can also affect dosimeter response. Therefore, do not use metal in any component in contact with the solutions.

6.3 If flame sealed ampoules are used as the dosimeters, exercise care in filling ampoules to avoid depositing solution in the ampoule neck. Subsequent heating during sealing of the ampoule may cause undesirable chemical change in the dosimetric solution remaining inside the ampoule neck. For the same reason, exercise care to avoid heating the body of the ampoule during sealing.

6.4 Thermal oxidation (as indicated by an increase in optical absorbance), in the absence of radiation, is a function of ambient temperature. At normal laboratory temperatures (about 20 to 25°C), this effect may be significant if there is a long period of time between solution preparation and photometric measurement. This interference is discussed further in 9.3.

6.5 The dosimetric solution is somewhat sensitive to ultraviolet light and should be kept in the dark for long-term storage. No special precautions are required during routine handling under normal laboratory lighting conditions, but strong UV sources such as sunlight should be avoided.

## 7. Apparatus

7.1 For the analysis of the dosimetric solution, use a high-precision spectrophotometer capable of measuring absorbance values up to 2 with an uncertainty of no more than  $\pm 1\%$  in the region of 300 nm. Use a quartz cuvette with 5- or 10-mm pathlength for spectrophotometric measurement of the solution. The cuvette capacity must be small enough to allow it to be thoroughly rinsed by the dosimeter solution and still leave an adequate amount of that solution to fill the cuvette to the appropriate level for the absorbance measurement. For dosimeter ampoules of less than 2 mL, this may require the use of semi-microcapacity cuvettes. Other solution handling techniques, such as the use of micro-capacity flow cells, may be employed provided precautions are taken to avoid cross-contamination. Either control the temperature of the dosimetric solution during measurement at  $25 \pm 0.5^\circ\text{C}$ , or determine the solution temperature during the spectrophotometric analysis and correct the measured absorbance to  $25^\circ\text{C}$  using Eq 3.

7.2 Use borosilicate glass or equivalent chemically-resistant glass to store the reagents and the prepared dosimetric solution. Clean all apparatus thoroughly before use (see Practice C912).

7.2.1 Store the cleaned glassware in a clean, dust-free environment. For extreme accuracy, bake the glassware in vacuum at  $550^\circ\text{C}$  for at least 1 h (6).

7.2.2 As an alternative method to baking the glassware, the dosimeter containers (for example, ampoules) may be filled with the dosimetric solution and irradiated to a dose of at least 500 Gy. When a container is needed, pour out the irradiated solution, rinse the container at least three times with unirradiated solution and then refill with the dosimetric solution to be irradiated. The time between filling, irradiation and measurement should be as short as practical, preferably no more than a few hours. Refer to Note 2.

7.3 Use a sealed glass ampoule or other appropriate glass container to hold the dosimetric solution during irradiation.

NOTE 2—To minimize errors due to differences in radiation absorption properties between the container material and the Fricke solution, it is possible to use plastic containers (for example, PMMA or polystyrene) to hold Fricke solution. However, the interferences discussed in Section 6 may result in a reduction in accuracy. To reduce these problems, the plastic containers may be conditioned by irradiating them filled with dosimetric solution to approximately 500 Gy. The containers should then be thoroughly rinsed with unirradiated solution before use.

## 8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used. Unless otherwise indicated, all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society (or equivalent) where such specifications are available.<sup>8</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the measurements. Methods of obtaining higher purity of chemicals exist (for example, crystallization or distillation), but are not discussed here.

8.2 *Purity of Water*—Water purity is very important since water is the major constituent of the dosimetric solution, and therefore, may be the prime source of contamination. The use of double-distilled water from coupled all-glass and silica stills or water from a high-quality commercial purification unit capable of achieving Total Oxidizable Carbon (T.O.C.) content below 5 ppb is recommended. Use of deionized water is not recommended.

NOTE 3—Double-distilled water distilled from an alkaline permanganate ( $\text{KMnO}_4$ ) solution (2 g  $\text{KMnO}_4$  plus 5 g sodium hydroxide ( $\text{NaOH}$ ) 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. Water labelled HPLC (high pressure liquid chromatography) grade is usually sufficiently free of organic impurities to be used in this practice.

### 8.3 Reagents:

8.3.1 *Ferrous Ammonium Sulfate*— $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ .

8.3.2 *Sodium Chloride* ( $\text{NaCl}$ ).

8.3.3 *Sulfuric Acid* ( $\text{H}_2\text{SO}_4$ ).

## 9. Preparation of dosimeters

### 9.1 Prepare dosimetric solution:

9.1.1 Dissolve 0.392 g of ferrous ammonium sulfate,  $(\text{NH}_4)_2\text{Fe}(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$ , and 0.058 g of sodium chloride,  $\text{NaCl}$ , in 12.5 mL of  $0.4 \text{ mol}\cdot\text{L}^{-1}$  sulfuric acid,  $\text{H}_2\text{SO}_4$ . Dilute to 1 L in a volumetric flask with air-saturated  $0.4 \text{ mol}\cdot\text{L}^{-1}$  sulfuric acid at  $25^\circ\text{C}$ . To make 0.4 M solution, use 41.0 g of 96.7 % sulfuric acid plus water to make 1 L of solution.

NOTE 4—Sodium chloride is used to reduce any adverse effects on the response of the dosimeter due to trace organic impurities.

<sup>8</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.



9.1.2 If the final solution is not yet air-saturated, it should be done. Shaking of the solution is normally sufficient to achieve this. Alternatively, bubble high-purity air through the solution, taking care to avoid any possible organic contamination of the air. The oxygen concentration in air-saturated solution is adequate to ensure the dosimeter's linear response up to 400 Gy. Store the dosimetric solution in clean borosilicate glass containers in the dark.

9.2 The dosimetric solution has the following concentrations:  $1 \times 10^{-3}$  mol·L<sup>-1</sup> ferrous ammonium sulfate;  $1 \times 10^{-3}$  mol·L<sup>-1</sup> sodium chloride; and 0.4 mol·L<sup>-1</sup> sulfuric acid.

9.3 The dosimetric solution will slowly oxidize at room temperature resulting in an increase in the optical absorbance of the unirradiated solution. If the solution has not been used for some time, measure the absorbance of the unirradiated solution, as described in 10.4. If the absorbance of a 10-mm pathlength sample is greater than 0.1, do not use that solution. Prepare a fresh batch of solution to replace it.

NOTE 5—Oxidation of the solution at room temperature can be significantly reduced by refrigerating the solution, but refrigeration may also change the oxygen concentration.

9.4 Rinse the dosimeter containers (ampoules or other types) at least three times with the dosimetric solution before filling them for irradiation. Even with careful rinsing, there will always be solution remaining; subsequent rinsing will help mitigate this effect.

9.5 Fill clean containers with the dosimetric solution. If flame sealing the dosimeters, observe the precautions in 6.3.

9.6 An alternative method of preparation using concentrated stock solution is described in Appendix X1. Each dilution made from the stock solution should be treated as a separate batch for the purposes of calibration.

## 10. Calibration of the dosimetry system

10.1 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 and quality assurance requirements. This calibration 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 Practice 51261.

NOTE 6—The quality of the Fricke dosimetry system is potentially high if prepared and used correctly and it is capable of dose determination using published  $\epsilon$  or  $G$  values, or the value of their product. However, doses determined in this way cannot be considered traceable to national or international standards without additional evidence, such as comparison with known traceable standards. For completeness, details of this method are given in Appendix X3, but it is not recommended in situations where traceability to national or international standards is a regulatory requirement.

10.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system.

10.2.1 When the Fricke dosimeter is used in a reference standard dosimetry system, calibration irradiations shall be

performed at an approved laboratory, as defined in 3.1.1, and have demonstrable traceability to nationally or internationally recognized standards.

10.2.2 When the Fricke dosimeter is used in a routine dosimetry system, the calibration irradiation may be performed in accordance with 10.2.1, or at a production or research irradiation facility together with reference- or transfer-standard dosimeters from a system that has measurement traceability to nationally or internationally recognized standards.

10.2.3 Specify the calibration dose in terms of absorbed dose to water.

10.2.4 For calibration with photons, the Fricke dosimeter shall be irradiated under conditions that approximate electron equilibrium.

10.2.5 When using an electron beam for irradiation, locate the dosimeters in a well-characterized position within the radiation field.

10.2.6 Ensure that the radiation field within the volume occupied by the dosimeters is as uniform as possible. The variation in dose rate within this volume should be known, and be within acceptable limits for the uncertainty of calibration required.

10.2.7 The dosimeter shall be calibrated in a radiation field of the same type and energy as that in which it is to be used, unless evidence is available to demonstrate equivalence of response.

10.2.8 Calibrate each batch of dosimeters prior to use.

10.2.9 Separate five dosimeters from the remainder of the batch and do not irradiate them. Use them in determining  $A_0$  (see 10.5.1).

10.2.10 Control (or monitor) the temperature of the dosimeters during irradiation. Calculate or measure the mean irradiation temperature of each dosimeter to an accuracy of  $\pm 2^\circ\text{C}$ , or better.

10.2.11 Use a set of at least three dosimeters for each absorbed dose value.

10.2.12 Irradiate these sets of dosimeters to at least five known dose values covering the range of utilization in order to determine the calibration curve for the dosimetry system.

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

10.3.1 Check the wavelength scale of the spectrophotometer and establish its accuracy. The emission spectrum from a low-pressure mercury arc lamp can be used for this purpose. Such a lamp may be obtained from the spectrophotometer manufacturer or other scientific laboratory instrument suppliers. Other appropriate wavelength standards are holmium oxide filters or solutions. For more details, see Practices E275, E925, and E958.

NOTE 7—For example, holmium oxide solutions in sealed cuvettes for use in the wavelength region of 240 to 650 nm are available as certified wavelength standards (SRM 2034).<sup>9</sup>

<sup>9</sup> Available from the National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899.



10.3.2 Check the accuracy of the photometric (absorbance) scale of the spectrophotometer, especially in the ultraviolet region before and after each set of measurements. Certified absorbance standard filters or solutions are available for this purpose.

NOTE 8—Solutions such as SRM 931f and SRM 935 (7) and metal-on-quartz filters such as SRM 2031<sup>9</sup> are examples of absorbance standards. Note that SRM 935 is a pure chemical that must be diluted by the user.

#### 10.4 Measurement:

10.4.1 Set the spectral bandwidth of the spectrophotometer at no more than 2 nm. Determine the optimum wavelength of the absorbance peak of the solution by making a spectral scan of an irradiated sample. The optimum wavelength is the wavelength that corresponds to the maximum absorbance value and should be between 302 nm and 305 nm. This peak is fairly broad, and the optimum wavelength will vary depending on the quality of the spectrophotometer. Set the wavelength at the peak and leave it there for the subsequent measurements.

10.4.2 Set the balance of the spectrophotometer to zero with only air (no cuvette) in the light path(s).

10.4.3 Fill a clean cuvette (or flow cell) of 5 to 10 mm pathlength with distilled water. Carefully wipe off any liquid on the exterior surfaces of the cuvette. Measure and record the absorbance. See [Note 9](#).

NOTE 9—Inadequate rinsing of the cuvette (or flow cell) between dosimeter solutions can lead to errors due to solution carryover (cross-contamination). Techniques for minimizing this effect are discussed in Ref (7). Micropipettes or Hamilton dispensers are recommended for the rinsing procedure. It is not meaningful to attempt to determine the quality of the water from absorbance measurements of water since pure water has an absorbance of approximately 0.0002 at 303 nm. The loss of light due to reflection from the cuvette surfaces will increase the absorbance.

10.4.4 Empty the water from the cuvette (or flow cell) and rinse it at least twice with the solution from an ampoule, or other container. Discard the rinse solution and fill to the appropriate level with more solution from the same container. Carefully wipe off any solution on the exterior surfaces of the cuvette. Place the cuvette in the sample holder of the spectrophotometer and measure the absorbance as soon as the instrument reading has stabilized. If necessary, correct the absorbance for temperature during measurement (see 7.1). The temperature correction should be applied to the measured absorbance after subtraction of the absorbance of the cell containing water (10.4.3). It is important to read the absorbance of each dosimeter at the same elapsed time after putting it in the spectrophotometer light beam. This is necessary because the absorbance increases slowly with time while the dosimeter is in the light beam (probably due to oxidation of the solution by the UV light). Repeat this procedure for all unirradiated and irradiated solutions.

10.4.5 Check the spectrophotometer zero after each measurement using only air in the light path(s). Measure the unirradiated solution before and after the irradiated solutions are read. Periodically during the measurement process, remeasure the absorbance of a distilled water to detect any contamination of the cuvette (or flow cell) and take appropriate corrective actions if required.

#### 10.5 Analysis:

10.5.1 Calculate the mean absorbance of the unirradiated dosimeters,  $A_0$  (see 10.2.9). Calculate the net absorbance,  $\Delta A$ , for each irradiated dosimeter by subtracting  $A_0$  from its absorbance ( $A_1$ ), as follows:

$$\Delta A = A_i - A_0 \quad (5)$$

10.5.2 Correct the measured net absorbance  $\Delta A$  to the net absorbance expected for an irradiation temperature of 25°C using the formula:

$$\Delta A_{25} = \Delta A / (1 + 0.0012 (T_{irrad} - 25)) \quad (6)$$

10.5.3 Prepare a calibration curve by plotting the corrected  $\Delta A$  values versus absorbed dose,  $D$ . Fit the data by means of a least-squares method with an appropriate analytical form that provides a best fit to the data. The data for these Fricke dosimeters should fit a first (or at the high end of the dose range a second) order polynomial of the form:

$$\Delta A_{25} = b_0 + b_1 D + b_2 D^2 \quad (7)$$

10.5.4 As a guide, the value of  $b_1$  should be approximately 0.00360 Gy<sup>-1</sup> for absorbance measurements made in a 10 mm pathlength optical cell.

NOTE 10—Computer software is available commercially for performing least-squares fits of data with polynomials or other analytical forms. Further information on mathematical methods for handling calibration data is given in ISO/ASTM Practice 51261.

10.5.5 The calibration curve should tend towards  $\Delta A = 0$  at zero dose. An appreciable  $\Delta A$  intercept value is indicative of contamination of the dosimetric solution with impurities.

NOTE 11—Impure solutions may exhibit a linear response in terms of  $\Delta A$  versus dose, but with an enhanced radiation chemical yield ( $G$ ). A useful method to check the purity of Fricke solution is to compare the dosimetric response ( $\Delta A$ ) of irradiated Fricke solution with and without NaCl (all other aspects of the preparation remaining the same). If the solutions are pure, the difference in response of solutions with and without NaCl should be less than 0.5 %.

10.5.6 Compare the net absorbance values of a given calibration with those expected based on the value of  $b_1$  given in 10.5.4 and the appropriate optical path length. For cobalt-60 radiation, agreement should be within  $\pm 2$  % if the dosimetric solutions were properly prepared and all associated analysis equipment was properly calibrated. Agreement of the dosimetric response values from batch to batch over the useful range of the system should be within  $\pm 1$  %.

10.5.7 Estimate the reproducibility (precision) of the individual dosimeter results either from the results of replicate measurements or from the statistics of the least-squares fit to the data. The reproducibility, expressed as one standard deviation, should not exceed 0.002 absorbance for an optical pathlength of 10 mm. Suspected data outliers should be tested using statistical procedures such as those found in Practice E178.

## 11. Application of dosimetry system

11.1 For most applications, use a minimum of two dosimeters for each dose measurement. The number of dosimeters required for the measurement of absorbed dose on or within a material is determined by the reproducibility associated with



the dosimetry system and the required measurement uncertainty associated with the application. Appendix X3 of *Prattice E668* describes a statistical method for determining this number.

11.2 Use the irradiation and measurement procedures in accordance with 10.2.3, 10.2.9, 10.2.10, 10.4.1 to 10.4.5, 10.5.1 and 10.5.2.

11.3 Determine the absorbed dose from the net absorbance values and the calibration curve.

NOTE 12—The absorbed dose in materials other than water irradiated under equivalent conditions may be calculated using procedures given in *Prattices E666* and *E668*.

11.4 Record the calculated absorbed dose values and all other relevant data as outlined in Section 12.

## 12. Minimum documentation requirements

### 12.1 Calibration:

12.1.1 Record the dosimeter batch number (code).

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

### 12.2 Application:

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

12.2.2 Record or reference the radiation source type and characteristics.

12.2.3 Record the absorbance, net absorbance value, temperature correction (if applicable), and resulting absorbed dose

for each dosimeter. Reference the calibration curve used to obtain the absorbed dose values.

12.2.4 Record or reference the measurement uncertainty in absorbed dose (refer to Section 13).

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

## 13. Measurement uncertainty

13.1 All dose measurements need to be accompanied by an estimate of uncertainty. Appropriate procedures are recommended in ISO/ASTM Guide 51707 (see also GUM).

13.2 All components of uncertainty should be included in the estimate, including those arising from calibration, dosimeter reproducibility, instrument reproducibility, and the effect of influence quantities. A full quantitative analysis of components of uncertainty may be referred to as an uncertainty budget, and is then often presented in the form of a table. Typically, the uncertainty budget will identify all significant components of uncertainty, together with their methods of estimation, statistical distributions and magnitudes.

13.3 If this practice is followed, the estimate of the expanded uncertainty of an absorbed dose determined by this dosimetry system should be less than 3 % for a coverage factor  $k = 2$  (which corresponds approximately to a 95 % level of confidence for normally distributed data).

## 14. Keywords

14.1 absorbed dose; dosimetry; ferrous ammonium sulfate dosimeter; ferrous sulfate dosimeter; Fricke dosimeter; reference-standard dosimetry system

## APPENDIXES

### (informative)

#### X1. Alternative Method for Preparation of Dosimetric Solution

X1.1 As an alternative to the preparation of the dosimetric solution described in 9.1, two separate concentrated stock solutions may be prepared and diluted as needed.

X1.2 Prepare concentrated stock solutions as follows:

X1.2.1 Dissolve 19.608 g of ferrous ammonium sulfate in 50 mL of 0.4 mol·L<sup>-1</sup> sulfuric acid, H<sub>2</sub>SO<sub>4</sub>, and add 0.4 mol·L<sup>-1</sup> sulfuric acid to make 100 mL of solution. The resulting concentration is 0.5 mol·L<sup>-1</sup> ferrous ammonium sulfate, (NH<sub>4</sub>)<sub>2</sub>Fe(SO<sub>4</sub>)<sub>2</sub> · 6H<sub>2</sub>O.

X1.2.2 Dissolve 2.923 g of sodium chloride in 50 mL of 0.4 mol·L<sup>-1</sup> sulfuric acid and add 0.4 mol·L<sup>-1</sup> sulfuric acid to make 100 mL of solution. The resulting concentration is 0.5 mol·L<sup>-1</sup> sodium chloride, NaCl.

X1.2.3 Store these stock solutions in clean borosilicate glass containers in the dark.

X1.3 Prepare the dosimetric solution from the concentrated solutions as follows:

X1.3.1 Pipette 1 mL of ferrous ammonium sulfate solution and 1 mL of sodium chloride solution from the stock solutions into a 500 mL volumetric flask.

X1.3.2 Add 0.4 mol·L<sup>-1</sup> sulfuric acid to make 500 mL of solution.

X1.4 The resulting dosimetric solution will have the same molar concentration as that given in 9.2.

X1.5 Prepare the dosimetric solution fresh each time it is needed and store in clean borosilicate glass containers in the dark.

## X2. Procedure for Determination of $\epsilon$ for Ferric Ions

X2.1 Even though it is recommended in [Appendix X3](#) that the product  $\epsilon \cdot G$  be used in calculating the dose, there is some merit in determining the value of  $\epsilon$  experimentally for the particular spectrophotometer utilized in measuring the dosimeter absorbance. In so doing, the performance of the spectrophotometer is independently verified. A series of ferric ion solutions of different concentrations is prepared and measured with the spectrophotometer. The molar linear absorption coefficient is determined from the slope of the line (plot of  $\Delta A/d$  versus concentration). The slope should have a value close to  $219 \text{ m}^2 \cdot \text{mol}^{-1}$ .

X2.2 Prepare a concentrated ferric ion solution as follows:

X2.2.1 Weigh about 100 mg of spectrographically pure (purity of at least 99.99 %) iron wire to the nearest 0.1 mg.

X2.2.2 Place the iron wire in a long-necked 1000-mL calibrated volumetric flask.

X2.2.3 Add 60 mL of distilled water and 22.5 mL of concentrated sulfuric acid (density of  $1.84 \text{ g} \cdot \text{cm}^{-3}$ ).

X2.2.4 Heat the unstoppered flask gently under a hood until the wire is completely dissolved. With a beaker inverted over the mouth of the flask, allow the solution to cool. During these procedures, fix the neck of the flask at an angle of  $45^\circ$  to the horizontal to prevent loss of solution.

X2.2.5 Add 3 to 5 mL of 35 % hydrogen peroxide,  $\text{H}_2\text{O}_2$ , solution to the flask and boil under reflux condensation for  $\frac{1}{2}$  to 1 h, or until the bubbles of excess peroxide are driven off. Allow the flask to cool and place it in a thermostat set at  $25 \pm 0.5^\circ\text{C}$ .  $\text{H}_2\text{O}_2$  will absorb slightly at 303 nm, so it is imperative that the bubbles have been driven off.

X2.2.6 Dilute the solution with distilled water to give 1000 mL of solution.

X2.3 The molarity ( $\text{mol} \cdot \text{L}^{-1}$ ) of ferric ions,  $C_{\text{ref}}$ , in this reference solution is calculated by the following:

$$C_{\text{ref}} = m_{\text{Fe}} \cdot k / V \quad (\text{X2.1})$$

where:

$m_{\text{Fe}}$  = mass of iron dissolved, kg,  
 $V$  = volume of the final solution, L, and  
 $k$  = conversion factor equal to  $17.91 \text{ mol} \cdot \text{kg}^{-1}$  of iron.

X2.4 The reference solution will have an absorbance of about 4 at 303 nm. Pipette samples of the reference solution of 1, 5, 10, 15, 20, and 25 mL into six 100-mL volumetric flasks. Dilute each sample by filling to the 100-mL mark with  $0.4 \text{ mol} \cdot \text{L}^{-1}$  sulfuric acid. These samples should have absorbances ranging from about 0.04 to 1.

X2.5 The molarity of each diluted sample solution,  $C_s$ , is calculated by the following:

$$C_s = C_{\text{ref}} / S \quad (\text{X2.2})$$

where:

$S$  = final volume (100 mL) divided by the initial sample volumes (1, 5, 10, 15, 20, and 25 mL).

X2.6 Measure the absorbance of the diluted sample solutions in a 10-mm pathlength cuvette at a temperature of  $25 \pm 0.5^\circ\text{C}$  using the procedures of [10.3](#).

X2.7 Plot the absorbance/pathlength values versus concentration of the samples. The result should be a straight line; the slope is the molar linear absorption coefficient. Compare this slope with the reference value of  $219 \text{ m}^2 \cdot \text{mol}^{-1}$ .

## X3. Procedure for Calculation of Dose Based on $\epsilon$ and $G$

X3.1 Calculate the mean absorbance of the unirradiated dosimeters,  $A_0$  (see [10.2.9](#)). Calculate the net absorbance,  $\Delta A$ , for each irradiated dosimeter by subtracting  $A_0$  from its absorbance ( $A_i$ ), as follows:

$$\Delta A = A_i - A_0 \quad (\text{X3.1})$$

X3.2 The basic equation for calculation of absorbed dose in the dosimeter solution,  $D_F$ , is as follows:

$$D_F = \Delta A / (\epsilon \cdot G \cdot \rho \cdot d) \quad (\text{X3.2})$$

where:

$D_F$  = absorbed dose to the Fricke solution (Gy),  
 $\Delta A$  = net absorbance at the optimum wavelength (302 to 305 nm),  
 $\rho$  = density of the dosimetric solution, equal to  $1.024 \times 10^3 \text{ kg} \cdot \text{m}^{-3}$  at  $25^\circ\text{C}$ ,  
 $\epsilon$  = molar linear absorption coefficient of the ferric ions ( $\text{Fe}^{3+}$ ),  $\text{m}^2 \cdot \text{mol}^{-1}$ ,

$G$  = radiation chemical yield of ferric ions ( $\text{Fe}^{3+}$ ),  $\text{mol} \cdot \text{J}^{-1}$ , and  
 $d$  = optical pathlength of the dosimetric solution in the cuvette, m.

X3.3 When using [Eq X3.2](#), the values of the parameters on the right side of the equation must be those for the appropriate temperatures, that is, the value of  $\epsilon$  must be its value for the temperature at which the absorbance of the Fricke solution was measured and the value of  $G$  must be its value for the temperature at which the Fricke solution was irradiated. The values of  $\epsilon$  and  $G$  are well known for  $25^\circ\text{C}$  and their temperature coefficients are known. The values of  $\epsilon$ ,  $G$ , or the product  $\epsilon \cdot G$  for a given temperature  $T^\circ\text{C}$  can be calculated using [Eq X3.3](#) and [Eq X3.4](#) (see NRCC Report PIRS-0815). Both  $\epsilon(\text{Fe}^{3+})$  and  $G(\text{Fe}^{3+})$  increase with increase in temperature.

$$\epsilon_{T_{\text{meas}}} = \epsilon_{25} [1 + 0.0069 (T_{\text{meas}} - 25)] \quad (\text{X3.3})$$



$$G_{T_{irrad}} = G_{25}[1 + 0.0012 (T_{irrad} - 25)] \quad (X3.4)$$

NOTE X3.1—Although ICRU 35 is an important reference, equation (5.7) within ICRU 35 gives the temperatures in the brackets in the incorrect order. ICRU 64 gives the equation in the correct form, but it references ICRU 35 with no mention of the error.

X3.4 The values of  $\rho$  and  $d$  should also correspond to their values at the temperature of the Fricke solution when the absorbance was measured. The use of “room temperature” values is usually considered adequate for precise dosimetry. Eq X3.3 and Eq X3.4 are valid for an irradiation temperature range of 10 to 60°C, and for an absorbance measurement temperature range of 15 to 35°C.

NOTE X3.2—Based on Eq X3.3 and Eq X3.4, it can be seen that  $\epsilon(\text{Fe}^{3+})$  and  $G(\text{Fe}^{3+})$  increase with increase in temperature. The recommended values for  $\epsilon$  and  $G$  at 25°C are:  $\epsilon = 219 \text{ m}^2 \cdot \text{mol}^{-1}$  and  $G = 1.61 \times 10^{-6} \text{ mol} \cdot \text{J}^{-1}$  (see ICRU Reports 14 and 35). A procedure for determining  $\epsilon$  is given in Appendix X2.

X3.5 The values of  $\epsilon$  and  $G$  may have to be corrected for temperature according to Eq X3.3 and Eq X3.4. The temperature at which the absorbance of the Fricke solution was measured and the temperature at which it was irradiated must be known.

$$D_F = \frac{\Delta A [1 + 0.0069 (25 - T_{meas})] [1 + 0.0012 (25 - T_{irrad})]}{(\epsilon_{25} \cdot G_{25} \cdot \rho \cdot d)} \quad (X3.5)$$

NOTE X3.3—In bringing the “correction” from the denominator to the numerator, the temperature subtraction in the round brackets has been reversed. This is not an exact way to achieve the results of Eq X3.3 and Eq X3.4, but the errors caused are usually considered negligible. For example, if the absorbance were measured at 30°C, Eq X3.5 would give a value 0.1 % smaller than Eq X3.3, and if the irradiation temperature were 60°C, Eq X3.5 would give a value 0.2 % smaller than Eq X3.4.

X3.6 To calculate the absorbed dose to water,  $D_w$ , use the following (ICRU Report 35):

$$D_w = 1.004 D_F \quad (X3.6)$$

X3.7 The absorbed dose to water,  $D_w$ , can also be determined from the mean absorbed dose in the Fricke solution,  $D_F$ , using the following equation (ICRU 64):

$$D_w = (\mu_{en}/\rho)_{w,F} \cdot P_{w,F} \cdot D_F \quad (X3.7)$$

where  $(\mu_{en}/\rho)_{w,F}$  is the ratio of the mass-energy absorption coefficients of water to Fricke solution;  $P_{w,F}$  is the correction factor of the perturbation introduced by the dosimeter vessel if it is not water-equivalent. If a single plastic irradiation cell is used,  $P_{w,F}$  is generally negligible.

X3.8 It has been recommended that the product  $\epsilon \cdot G$  be used in (Eq X3.2 and Eq X3.5) rather than individually determined  $\epsilon$  and  $G$  values (see ICRU Report 35). This is due to the large systematic errors in the measurement of  $\epsilon$  that have been observed with a number of investigators. For irradiation and absorbance measurement at 25°C, the recommended value for  $\epsilon \cdot G$  at 303 nm is  $3.52 \times 10^{-4} \text{ m}^2 \cdot \text{J}^{-1}$ . For irradiation or absorbance measurement at other than 25°C, correct the value of  $\epsilon \cdot G$  using Eq X3.3 and Eq X3.4.

X3.9 For an irradiation and absorbance measurement temperature of 25°C, with a 10-mm pathlength cuvette, and using the value of  $\epsilon \cdot G$  recommended in X3.8, Eq X3.2 reduces to:

$$D_w = 278 \Delta A \quad (X3.8)$$

NOTE X3.4—Impure solutions may exhibit a linear response in terms of  $\Delta A$  versus dose, but with an enhanced radiation chemical yield ( $G$ ). Alternatively, impurities may result in changes in response at low doses, resulting in an apparent intercept in the  $\Delta A$  versus dose function. A useful method to check the purity of Fricke solution is to compare the dosimetric response ( $\Delta A$ ) of irradiated Fricke solution with and without NaCl (all other aspects of the preparation remaining the same). If the solutions are pure, the difference in response of solutions with and without NaCl should be less than 0.5 %.

## Bibliography

- (1) Sehested, K., “The Fricke Dosimeter,” *Manual on Radiation Dosimetry*, edited by Holm, N. W., and Berry, R. J., Marcel Dekker, pp. 313–317, 1970.
- (2) Holm, N. W., and Zagorski, Z. P., “Aqueous Chemical Dosimetry,” *Manual on Radiation Dosimetry*, edited by Holm, N. W., and Berry, R. J., Marcel Dekker, pp. 87–104, 1970.
- (3) Sharpe, P. H. G., and Burns, D. T., “The Relative Response of Fricke, Dichromate and Aniline Dosimeters to  $^{60}\text{Co}$  and High Energy Electron Beam Radiation,” *Journal of Radiation Physics and Chemistry*, Vol 46, 1995, p. 1273.
- (4) Fricke, H., and Hart, E. J., “Chemical Dosimetry,” *Radiation Dosimetry*, 2nd Edition, Vol 2, Academic Press, pp. 167–239, 1966.
- (5) McLaughlin, W. L., Boyd, A. W., Chadwick, K. H., McDonald, J. C., and Miller, A., Chapter 8 and Appendix X2 in *Dosimetry for Radiation Processing*, Taylor and Francis, London, 1989.
- (6) Ellis, S. C., “The Dissemination of Absorbed Dose Standards by Chemical Dosimetry Mechanism and Use of the Fricke Dosimeter,” *Ionizing Radiation Metrology*, pp. 163–180, 1977.
- (7) Burke, R. W., and Mavrodineanu, R., “Standard Reference Materials: Certification and Use of Acidic Potassium Dichromate Solutions as an Ultraviolet Absorbance Standard-SRM 935,” NBS Special Publication 260-54, 1977.



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