Designation: E 1026 – 95

Standard Practice for
Using the Fricke Reference Standard Dosimetry System[1]

This standard is issued under the fixed designation E 1026; 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 preparation, testing and procedure for using the acidic aqueous ferrous ammonium sulfate solution 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 Fricke system. It is classified as a reference standard dosimeter (see Guide E 1261).

1.2 The practice describes the spectrophotometric analysis procedures for the Fricke dosimeter.

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 from 40 to 400 Gy **(1)**.[2]

1.4.2 The absorbed dose rate shall not exceed 10^6 Gy·s^{-1}**(2)**.

1.4.3 For radioisotope 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 (see ICRU Reports 34 and 35).

NOTE 1—The lower energy limits given 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. The Fricke 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 should be within the range of 10 to 60°C.

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 Glasses[3]
D 1193 Specification for Reagent Water[4]
E 170 Terminology Relating to Radiation Measurements and Dosimetry[5]
E 178 Practice for Dealing with Outlying Observations[6]
E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[7]
E 666 Practice for Calculating Absorbed Dose from Gamma or X-Radiation[5]
E 668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices[5]
E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers[8]
E 958 Practice for Measuring Practical Spectral Bandwidth of UltraViolet-Visible Spectrophotometers[9]
E 1205 Method for Using the Ceric-Cerous Sulfate Dosimetry System[5]
E 1261 Guide for Selection and Application of Dosimetry Systems for Radiation Processing of Food[5]

2.2 *International Commission on Radiation Units and Measurements (ICRU) Reports:*[10]

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

3. Terminology

3.1 *absorbed dose, D*—the quotient of *de* by *dm*, where *de*

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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 that appear in parentheses refer to a list of references at the end of this practice.

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

[4] *Annual Book of ASTM Standards*, Vol 11.01.

[5] *Annual Book of ASTM Standards*, Vol 12.02.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

[7] *Annual Book of ASTM Standards*, Vol 03.05.

[8] *Annual Book of ASTM Standards*, Vol 14.01.

[9] *Annual Book of ASTM Standards*, Vol 03.06.

[10] Available from the International Commission on Radiation Units and Measurements (ICRU), 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814.

is the mean energy imparted by ionizing radiation to matter of mass *dm* (see ICRU Report 33).

$$D = de/dm$$

The special name of the unit for absorbed dose is the gray (Gy).

$$1\ \text{Gy} = 1\ \text{J}\cdot\text{kg}^{-1}$$

3.1.1 *Discussion*—Formerly, the special unit for absorbed dose was the rad.

$$1\ \text{rad} = 10^{-2}\ J\cdot\text{kg}^{-1} = 10^{-2}\ Gy$$

3.2 *calibration facility*—combination of an ionizing radiation source and its associated instrumentation that provides traceable, uniform, and reproducible absorbed dose rates at specific locations and in a specific material. It may be used to calibrate the response of routine or other types of dosimeters as a function of absorbed dose.

3.3 *measurement quality assurance plan*—a documented program for the measurement process that quantifies the total uncertainty of the measurements (both random and systematic error components). This plan shall demonstrate traceability to national standards, and shall show that the total uncertainty meets the requirements of the specific application.

3.4 *molar linear absorption coefficient*, ϵ—quotient given by the relation from Beer's law as follows:

$$\epsilon = A/(M\cdot d)$$

where:

A = absorbance at a specified wavelength,
M = molar concentration of the ion of interest, and
d = optical pathlength within the solution measured by the spectrophotometer (see ICRU Report 35). Units: $\text{m}^2\text{mol}^{-1}$

3.4.1 *Discussion*—This quantity is often referred to in the literature as the *molar extinction coefficient*.

3.5 *net absorbance*, ΔA—the difference between the optical absorbance of an unirradiated dosimetric solution, A_o, and the optical absorbance of an irradiated dosimetric solution, A_i:

$$\Delta A = A_i - A_o$$

3.6 *radiation chemical yield, G(x)*—the quotient of *n(x)* by *e* where *n(x)* is the mean amount of substance of a specified entity, *x*, produced, destroyed or changed by the mean energy imparted, *e*, to the matter (see ICRU Report 33).

$$G(x) = n(x)/e$$

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

3.6.1 *Discussion*—This quantity is often referred to as *G value*. The former special unit was $(100\ \text{eV})^{-1}$.

3.7 *reference standard dosimetry system*—combination of a dosimeter and appropriate analytical instrumentation of high metrological quality that is traceable to national standards.

3.8 *traceability*—the ability to show that a measurement is consistent with appropriate national standards through an unbroken chain of comparisons.

3.9 For other terms, see Terminology E 170.

4. Significance and Use

4.1 The Fricke dosimetry system provides a reliable means for measurement of absorbed dose in water, based on a process of oxidation of ferrous ions to ferric ions in acidic aqueous solution by ionizing radiation **(3)**. In situations not requiring traceability to national standards, this system can be used for absolute determination of absorbed dose, as the radiation chemical yield of ferric ions is well known.

4.1.1 Fricke dosimetry cannot be used in situations where traceability to national standards of absorbed dose is required unless the dosimetry system is calibrated in a calibration facility. Irradiation of Fricke dosimeters in a calibration facility is required in order to provide a means of verifying the expected response of the dosimetry system.

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

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

4.4 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.

4.5 There are two factors associated with use of the Fricke system at energies below those specified in 1.4.3:

4.5.1 The radiation chemical yield changes at low photon energies **(4)**, and

4.5.2 Dose gradients across the dosimeter with a dimension in the beam direction exceeding 12 mm require corrections in dosimeter response at energies below 8 MeV for electrons (see ICRU Report 35).

5. Interferences

5.1 The Fricke dosimetric solution response is extremely sensitive to impurities, particularly organic impurities. Even in trace quantities, impurities can cause a detectable change in the observed response. For high accuracy results, organic materials shall not be used for any component in contact with the solution.

5.2 Traces of metal ions in the dosimetric or reference solutions can also cause problems. Therefore, do not use metal in any component in contact with the solutions.

5.3 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's neck. For the same reason, exercise care to avoid heating the body of the ampoule during sealing.

5.4 Thermal oxidation (as indicated by an increase in absorbance), in the absence of radiation, is a function of temperature. At normal laboratory temperatures (about 20 to 25°C), this effect may cause a problem if there is a long period of time between solution preparation and measurement. This interference is discussed further in 8.4.

5.5 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.

6. Apparatus

6.1 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 ±1 % in the region of 300 nm. Use a quartz cuvette with 5 or 10 mm path length 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 2 mL or less, this may require the use of micro-capacity 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. Control the temperature of the dosimetric solution during measurement at 25 ± 0.2°C. If this is not possible, determine the solution temperature during the spectrophotometric analysis and correct the results using (Eq 4) in 10.3.5.

6.2 Use borosilicate glass or equivalent chemically-resistant glass to store the reagents and the prepared dosimeter solution. Clean all apparatus thoroughly before use (see Practice C 912).

6.2.1 One method of cleaning that may be used consists of the following steps in sequence:

6.2.1.1 Ultrasonic cleaning in hot (60°C or higher) distilled water for at least ½ h,

6.2.1.2 Thorough rinsing in hot fuming sulfuric acid,

6.2.1.3 Rinsing at least twice with distilled water, and

6.2.1.4 Drying in an oven at a temperature of at least 100°C for a minimum of 1 h.

6.2.1.5 Store the cleaned glassware in a clean, dust-free environment. For extreme accuracy, bake the glassware in vacuum at 550°C for at least 1 h **(5)**.

6.2.2 As an alternative method, the dosimeter containers 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.

6.3 Use a sealed glass ampoule or other appropriate glass container to hold the dosimetric solution during irradiation. For photons, surround the container with material of thickness sufficient to produce approximate electron equilibrium conditions during calibration irradiations. For measurement of absorbed dose in water, use materials that have radiation absorption properties essentially equivalent to water, for example, polystyrene or polyethylene. The appropriate thickness of such material depends on the energy of the photon radiation (see Practices E 666 and E 668).

NOTE 2—The dosimetric ampoule commonly used has a capacity of about 2 mL. Certain types of plastic containers for the dosimetric solution may be used; however, the results will be less accurate because of interferences discussed in Section 5 **(5)**. To avoid these problems with plastic containers, condition them by irradiating them filled with dosimetric solution through two cycles as discussed in 6.2.2. Then fill the containers with unirradiated solution, irradiate them, and measure the absorbance immediately. The time between filling and measurement should be as short as practical, preferably no more than a few hours.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.[11] 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 determination.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type II of Specification D 1193.

7.2.1 Use of triply-distilled water from coupled all-glass and silica stills is recommended. Water purity is very important since it is the major constituent of the dosimetric solution, and, therefore, may be the prime source of contamination. Use of deionized water is not recommended. Type II reagent water as specified in Specification D 1193 is considered to be of sufficient quality for use in preparing all solutions.

NOTE 3—High purity water is commercially available. Such water, labeled HPLC (high pressure liquid chromatography) grade, is usually sufficiently free of organic impurities to be used in this practice.

7.3 *Reagents*:

7.3.1 *Ferrous Ammonium Sulfate*—($Fe(NH_4)_2(SO_4)_2$-($6H_2O$)).

7.3.2 *Sodium Chloride* (NaCl).

7.3.3 *Sulfuric Acid* (H_2SO_4).

8. Preparation of Dosimeters

8.1 Perform the following steps:

8.1.1 Dissolve 0.392 g of ferrous ammonium sulfate, $Fe(NH_4)_2(SO_4)_2(6H_2O)$, and 0.058 g of sodium chloride, NaCl, in 12.5 mL of 0.4 $mol \cdot L^{-1}$ sulfuric acid, H_2SO_4. Dilute to 1 L in a volumetric flask with 0.4 $mol \cdot L^{-1}$ sulfuric acid at 25°C.

8.1.2 Air saturate the resultant dosimetric solution. One method for doing this is to bubble high-purity air through the solution, taking care to avoid any possible organic contamination of the air. Store the dosimetric solution in clean borosilicate glass containers in the dark.

8.2 The dosimetric solution has the following concentrations: 1×10^{-3} $mol \cdot L^{-1}$ ferrous ammonium sulfate; 1×10^{-3} $mol \cdot L^{-1}$ sodium chloride; and 0.4 $mol \cdot L^{-1}$ sulfuric acid.

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

8.4 The dosimetric solution will slowly oxidize at room temperature resulting in an increase in the absorbance of the unirradiated solution. This effect can be significantly reduced by refrigerating the solution. If the solution has not been used for some time, measure the absorbance of the unirradiated solution, as described in 10.2. If the absorbance of a 1-cm pathlength sample is greater than 0.1, then do not use that solution. Prepare a fresh batch of solution to replace it.

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[11] *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.

8.5 Rinse the dosimeter containers (ampoules or other types) at least three times with the dosimetric solution before filling them for irradiation.

8.6 The quantity of dosimetric solution produced by the procedure in 8.1 is convenient when dosimeters are needed in small quantities. If more dosimeters are required on a regular basis, then it may be more convenient to prepare a concentrated stock solution from which a small quantity may be drawn and diluted as needed (see Appendix X1 for details).

9. Spectrophotometer Calibration

9.1 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 E 275, E 925, and E 958.

NOTE 4—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).[12]

9.2 Check the accuracy of the photometric (absorbance) scale of the spectrophotometer, especially in the ultraviolet region. Certified absorbance standard filters or solutions are available for this purpose.

NOTE 5—Examples of absorbance standards are solutions of various concentrations such as SRM 931d and SRM 935 **(6)** and metal-on-quartz filters such as SRM 2031.[11]

10. Irradiation and Measurement Procedures

10.1 *Irradiation*:

10.1.1 Fill clean containers with the dosimetric solution, observing the precautions in 5.3 on flame sealing. Separate five dosimeters from the remainder of the batch and do not irradiate them. Use them in determining A_o (see 10.3.1).

10.1.2 Specify the dose in terms of absorbed dose in water (for example, see Method E 1205).

10.1.3 Position the dosimeters in the radiation field in a defined, reproducible location.

10.1.4 When using a gamma-ray source for irradiation, surround the dosimeters with a sufficient amount of water-equivalent material to achieve approximate electron equilibrium conditions.

NOTE 6—For example, for a ^{60}Co source, 3 to 5 mm of polystyrene (or equivalent polymeric material) should surround the dosimeter in all directions.

10.1.5 When using an electron beam for irradiation, locate the dosimeters in a well-characterized position within the radiation field. In addition, it may be necessary to surround the dosimeters with water-equivalent material.

10.1.6 Make the irradiation field within the volume occupied by the dosimeters as uniform as possible. The variation in dose rate within this volume should be within ±1 %.

10.1.7 Control (or monitor) the temperature of the dosimeters during irradiation. Take into account any temperature variations that affect dosimeter response (see 10.3.5).

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

10.2 *Measurement*:

10.2.1 Set the spectral bandwidth of the spectrophotometer at no more than 2 nm, and maintain the sample compartment and dosimetric solution at 25°C during the measurement. Determine the exact wavelength of the absorbance peak of the solution by making a spectral scan of an irradiated sample. The nominal peak wavelength is about 303 nm. Set the wavelength at the peak and leave it there for all subsequent measurements.

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

10.2.3 Fill a clean cuvette (or flow cell) of 5 or 10 mm pathlength with double-distilled water. Carefully wipe the cuvette exterior windows through which the light beam passes with a clean, lint-free tissue or cloth. Measure and record the absorbance.

10.2.4 Empty the water from the cuvette (or flow cell) and rinse it at least once with the solution from an ampoule. Discard the rinse solution and fill to the appropriate level with more solution from the same ampoule. Carefully wipe off any solution on the exterior surfaces of the cuvette as instructed in 10.2.3. Place the cuvette in the sample holder and measure the absorbance as soon as the instrument reading has stabilized. 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 with time if the dosimeter is left in the instrument (probably due to oxidation of the solution by the UV light). Repeat this procedure for all unirradiated and irradiated solutions.

NOTE 7—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 **6**.

10.2.5 Check the zero balance after each solution is measured with only air in the light path(s). Periodically during the measurement process, remeasure the absorbance of distilled water to detect any contamination of the cuvette (or flow cell) and take appropriate corrective actions if required.

10.3 *Analysis*:

10.3.1 Calculate the mean absorbance of the unirradiated dosimeters, A_o (see 10.1.1). Calculate the net absorbance,ΔA, for each irradiated dosimeter by subtracting A_o from its absorbance, A_i, as follows:

$$\Delta A = A_i - A_o \tag{1}$$

10.3.2 The basic equation for calculation of absorbed dose in the dosimetric solution is as follows:

$$D_F = \Delta A/(\rho \epsilon G d) \tag{2}$$

where:

D_F = absorbed dose in Fricke solution, Gy,
ΔA = change in absorbance at about 303 nm,
ρ = density of the dosimetric solution, equal to 1.024×10^3 kg·m^{-3}

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[12] Available from the National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899.

ϵ = molar linear absorption coefficient, $m^2 \cdot mol^{-1}$,
G = radiation chemical yield of ferric ions, $mol \cdot J^{-1}$, and
d = optical pathlength of the dosimetric solution in the cuvette, m.

10.3.3 To calculate the absorbed dose in water (ICRU Report 35), use the following:

$$D_W = 1.004\, D_F \tag{3}$$

10.3.4 (Eq 3) is valid only for an irradiation temperature of 25°C and for a temperature of 25°C for the dosimetric solution during the spectrophotometric measurement.

10.3.5 It has been recommended that the product $\epsilon \cdot G$ be used in (Eq 2) rather than individually determined ϵ and G values (see ICRU Report 35). This is due to the large systematic errors in the measurement of ϵ that have been observed with a number of investigators. The recommended value for $\epsilon \cdot G$ at 303 nm is $352 \times 10^{-6}\ m^2 \cdot J^{-1}$. For irradiation or absorbance measurement at other than 25°C **(7)**, correct the value of $\epsilon \cdot G$ by use of the following:

$$\epsilon_t \cdot G_{t'} = 352 \times 10^{-6}[1 + 0.007(t - 25)] \times [1 + 0.0015(t' - 25)] \tag{4}$$

where:

t = temperature of dosimetric solution during spectrophotometric analysis, °C, that affects the value of ϵ, and
t' = irradiation temperature, °C, that affects the value of G.

(Eq 4) is valid for an irradiation temperature range of 10 to 60°C, and for an absorbance measurement temperature range of 15 to 35°C.

NOTE 8—The recommended values for ϵ and G at 25°C are: $\epsilon = 219\ m^2 \cdot mol^{-1}$ and $G = 1.61 \times 10^{-6}\ mol \cdot J^{-1}$(see ICRU Reports 14 and 35). A procedure for determining ϵ is given in Appendix X2.

10.3.6 For an irradiation and absorbance measurement temperature of 25°C, with a 1-cm pathlength cuvette, and using the value of $\epsilon \cdot G$ recommended in 10.3.5, (Eq 3) reduces to:

$$D_W = 278\, \Delta A \tag{5}$$

10.4 *Calibration*:

10.4.1 If Fricke dosimetry is to be used in situations where traceability to national standards is required, the system shall be calibrated. This can be done by irradiating samples in a known radiation field of a calibration facility. Use the procedures of 10.1 to perform the irradiations. Only a few dose levels are needed to check both the absolute response and the linearity of the response. Analyze the dosimeters and calculate the doses using the procedures of 10.3. Compare the results with the calibration doses. The results from the two methods should not differ by more than the overall uncertainty of the dosimeter response. A greater difference is an indication of possible contamination of the solution or some other problem that must be resolved.

11. Minimum Documentation Requirements

11.1 Record calibration data and results (if performed).

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

11.1.2 Record or reference the 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 (if appropriate) or the calculations used to obtain the absorbed dose values.

11.2.4 Record or reference the precision and bias 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. Precision and Bias

12.1 To be meaningful, a measurement of absorbed dose shall be accompanied by an estimate of the uncertainty in the measured value. Factors contributing to the total uncertainty may be separated into two types, precision (random) and bias (systematic). Guide E 1261 discusses sources of uncertainty and lists some of the possible factors that affect precision and bias. Additional information is given in Practice E 668 and Ref **(8)**.

12.2 The random and systematic uncertainties involved in measuring absorbed dose using this dosimetry system should be estimated or determined. The overall uncertainty in absorbed dose should be estimated from a combination of these uncertainties, and the procedure for combining these uncertainties should be specifically stated or referenced in all results.

12.3 If care is taken in carrying out this practice, the combined uncertainty of an absorbed dose determined by the dosimetry system should be within ±2 % at a 99 % confidence level (see ICRU Report 35).

13. Keywords

13.1 absorbed dose; dosimetry; Fricke; reference standard dosimetry

APPENDIXES

(Nonmandatory Information)

X1. ALTERNATIVE METHOD FOR PREPARATION OF DOSIMETRIC SOLUTION

X1.1 As an alternative to the preparation of the dosimetric solution described in 8.1, concentrated stock solutions of 0.5 mol·L^{-1} ferrous ammonium sulfate, $Fe(NH_4)_2(SO_4)_2(6H_2O)$, and 0.5 mol·L^{-1} sodium chloride, NaCl, 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^{-1} sulfuric acid, H_2SO_4, and add 0.4 mol·L^{-1} sulfuric acid to make 100 mL of solution.

X1.2.2 Dissolve 2.923 g of sodium chloride in 50 mL of 0.4 mol·L^{-1} sulfuric acid and add 0.4 mol·L^{-1} sulfuric acid to make 100 mL of solution.

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 0.5 mol·L^{-1} ferrous ammonium sulfate and 1 mL of 0.5 mol·L^{-1} sodium chloride from the stock solutions into a 500 mL volumetric flask.

X1.3.2 Add 0.4 mol·L^{-1} sulfuric acid to make 500 mL of solution.

X1.4 The resulting dosimetric solution will have the same molar concentration as that given in 8.2.

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 PREPARING THE REFERENCE SOLUTION OF FERRIC IONS

X2.1 Even though it is recommended in 10.3 that the product $\epsilon \cdot G$ be used in calculating the dose, there is some merit in determining the value of ϵ 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 of the plot concentration versus absorbance. The slope should have a value close to 219 m^2·mol^{-1} (see 10.3.4).

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

X2.2.1 Weigh 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 g·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° to the horizontal to prevent loss of solution.

X2.2.5 Add 3 to 5 mL of 35 % hydrogen peroxide, H_2O_2, solution to the flask and boil under reflux condensation for ½ to 1 h, or until the bubbles of excess peroxide are driven off. Allow the flask to cool and place in a thermostat set at 25°C.

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

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

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

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

X2.4 The reference solution will have an absorbance of about 4. 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 mol·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:

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

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 1-cm pathlength cuvette at a temperature of 25°C using the procedures of 10.2.

X2.7 Plot the absorbance 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 m^2·mol^{-1}.

NOTICE: This standard has either been superceded and replaced by a new version or discontinued.
Contact ASTM International (www.astm.org) for the latest information.

REFERENCES

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