



Designation: E 1026 – 95

Standard Practice for Using the Fricke Reference Standard Dosimetry System¹

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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 Glasses³

D 1193 Specification for Reagent Water⁴

E 170 Terminology Relating to Radiation Measurements and Dosimetry⁵

E 178 Practice for Dealing with Outlying Observations⁶

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers⁷

E 666 Practice for Calculating Absorbed Dose from Gamma or X-Radiation⁵

E 668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices⁵

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

E 958 Practice for Measuring Practical Spectral Bandwidth of UltraViolet-Visible Spectrophotometers⁹

E 1205 Method for Using the Ceric-Cerous Sulfate Dosimetry System⁵

E 1261 Guide for Selection and Application of Dosimetry Systems for Radiation Processing of Food⁵

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

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.

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.

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

$$D = d/dm$$

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.

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

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 $\pm 1\%$ 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 $25 \pm 0.2^\circ\text{C}$. 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 $\frac{1}{2}$ 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*— $(\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O})$.

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, $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, and 0.058 g of sodium chloride, NaCl, in 12.5 mL of $0.4 \text{ mol}\cdot\text{L}^{-1}$ sulfuric acid, H_2SO_4 . Dilute to 1 L in a volumetric flask with $0.4 \text{ mol}\cdot\text{L}^{-1}$ 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: $1 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ ferrous ammonium sulfate; $1 \times 10^{-3} \text{ mol}\cdot\text{L}^{-1}$ sodium chloride; and $0.4 \text{ mol}\cdot\text{L}^{-1}$ 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.

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.