Designation: E1026 – 13

AnAmerican National Standard

Standard Practice for
Using the Fricke Dosimetry System[1]

This standard is issued under the fixed designation E1026; 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 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 Practice E2628 for the Fricke dosimetry system. It is intended to be read in conjunction with Practice E2628.

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**).[2]

1.5.2 The absorbed-dose rate does not exceed 10^6 Gy·s^{-1} (**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 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:*[3]

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
E2628 Practice for Dosimetry in Radiation Processing

2.2 *ISO/ASTM Standards:*[3]

ISO/ASTM 51261 Practice for Calibration of Routine Dosimetry Systems for Radiation Processing
ISO/ASTM 51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

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[1] 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.

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

2.3 *ISO/IEC Standard:*

ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories[4]

2.4 *International Commission on Radiation Units and Measurements (ICRU) Reports:*[5]

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:*[6]

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[7]

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* (ε_m)—a constant relating the spectrophotometric absorbance (A_λ) of an optically absorbing molecular species at a given wavelength (λ) per unit pathlength (d) to the molar concentration (c) of that species in solution:

$$\varepsilon_m = \frac{A_\lambda}{(d \times c)} \tag{1}$$

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

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

$$G(x) = \left(\frac{n(x)}{\bar{\varepsilon}}\right) \tag{2}$$

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

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 (**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 (ICRU 80).

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 (ε) for measurement temperatures. both $\varepsilon(Fe^{3+})$ and $G(Fe^{3+})$ increase with increase in temperature. The subscripts indicate the temperature of irradiation and measurement, as applicable.

$$\varepsilon_{T_{meas}} = \varepsilon_{25}[1 + 0.0069\,(T_{meas} - 25)] \tag{3}$$

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

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

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[4] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

[6] 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).

[7] Available from the National Research Council, Ionizing Radiation Standards, Institute for National Measurement Standards, Ottawa, Ontario. K1A 0R6.

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 ±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 ± 0.5°C, or determine the solution temperature during the spectrophotometric analysis and correct the measured absorbance to 25°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°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 where such specifications are available.[8] 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 is recommended. Alternatively, water from a high-quality commercial purification unit capable of achieving Total Oxidizable Carbon (T.O.C.) content below 5 ppb may be used. Use of deionized water is not recommended.

NOTE 3—Double-distilled water distilled from an alkaline permanganate ($KMnO_4$) solution (2 g $KMnO_4$ plus 5 g sodium hydroxide (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*—$(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$.

8.3.2 *Sodium Chloride* (NaCl).

8.3.3 *Sulfuric Acid* (H_2SO_4).

9. Preparation of Dosimeters

9.1 Prepare dosimetric solution:

9.1.1 Dissolve 0.392 g of ferrous ammonium sulfate, $(NH_4)_2Fe(SO_4)_2 \cdot 6H_2O$, 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 air-saturated 0.4 $mol{\cdot}L^{-1}$ sulfuric acid at 25°C. To make 0.4 M solution, use 41.0 g of 96.7 % sulfuric acid plus water to make 1 L of solution.

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[8] *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.