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Practice for use of a ceric-cerous sulfate dosimetry system

Pratique de l'utilisation d'un système dosimétrique au sulfate cérique-céreux

Reference number
ISO/ASTM 51205:2009(E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75% of the member bodies casting a vote.

ASTM International is one of the world's largest voluntary standards development organizations with global participation from affected stakeholders. ASTM technical committees follow rigorous due process balloting procedures.

A pilot project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this pilot project, ASTM Subcommittee E10.01, Radiation Processing: Dosimetry and Applications, is responsible for the development and maintenance of these dosimetry standards with unrestricted participation and input from appropriate ISO member bodies.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. Neither ISO nor ASTM International shall be held responsible for identifying any or all such patent rights.

International Standard ISO/ASTM 51205 was developed by ASTM Committee E10, Nuclear Technology and Applications, through Subcommittee E10.01, and by Technical Committee ISO/TC 85, Nuclear energy.

This second edition cancels and replaces the first edition (ISO/ASTM 51205:2002), which has been technically revised.

Standard Practice for
Use of a Ceric-Cerous Sulfate Dosimetry System[1]

This standard is issued under the fixed designation ISO/ASTM 51205; 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 ceric-cerous sulfate dosimetry system to determine absorbed dose (in terms of absorbed dose to water) in materials irradiated by photons (gamma radiation or X-radiation/bremsstrahlung) or high-energy electrons. The system consists of a dosimeter and appropriate analytical instrumentation. For simplicity, the system will be referred to as the ceric-cerous system. It is classified as a reference–standard dosimetry system (see ISO/ASTM Guide 51261). Ceric-cerous dosimeters are also used as transfer–standard dosimeters or routine dosimeters.

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

1.3 This practice applies provided the following conditions are satisfied:.

1.3.1 The absorbed-dose range is between 0.5 and 50 kGy (**1**).[2]

1.3.2 The absorbed-dose rate is less than 10^6 Gy s^{-1}(**1**).

1.3.3 For radionuclide gamma-ray sources, the initial photon energy is greater than 0.6 MeV. For bremsstrahlung photons, the energy of the electrons used to produce the bremsstrahlung 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 are appropriate for a cylindrical dosimeter ampoule of 12-mm diameter. Corrections for dose gradients across an ampoule of that diameter or less are not required for photons, but may be required for electron beams (**2**). The ceric-cerous system may be used at lower energies by employing thinner (in the beam direction) dosimeters.

1.3.4 The irradiation temperature of the dosimeter is above 0°C and below 62°C (**3**).

NOTE 2—The temperature dependence of dosimeter response is known only in this range (see 4.3). Use outside this range requires determination of the temperature dependence.

1.4 *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]

C 912 Practice for Designing a Process for Cleaning Technical Glasses

E 170 Terminology Relating to Radiation Measurements and Dosimetry

E 178 Practice for Dealing With Outlying Observations

E 275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

E 666 Practice for Calculating Absorbed Dose From Gamma or X Radiation

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

E 925 Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Slit Width does not Exceed 2 nm

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

2.2 *ISO/ASTM Standards:*[3]

51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing

51400 Practice for Characterization and Performance of a High-Dose Radiation Dosimetry Calibration Laboratory

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

2.3 *International Commission on Radiation Units and Measurements (ICRU) Reports:*[4]

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

ICRU Report 34 The Dosimetry of Pulsed Radiation

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

ICRU Report 37 Stopping Powers for Electrons and Positrons

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[1] This guide is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.01 on Radiation Processing: Dosimetry and Applications, and is also under the jurisdiction of ISO/TC 85/WG 3.

Current edition approved June 18, 2008. Published June 2009. Originally published as ASTM E 1205–88. Last previous ASTM edition E 1205–99. ASTM E 1205–93 was adopted by ISO in 1998 with the intermediate designation ISO 15555:1998(E). The present International Standard ISO/ASTM 51205:2009(E) is a major revision of ISO/ASTM 51205-2002(E) which replaced ISO 15555.

[2] The boldface numbers in parentheses refer to the bibliography at the end of this standard.

[3] For referenced ASTM and ISO/ASTM standards, visit the ASTM website, 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.

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

ICRU Report 60 Radiation Quantities and Units for Ionizing Radiation

3. Terminology

3.1 *Definitions:*

3.1.1 *calibration*—set of operations under specified conditions, which establishes the relationship between values indicated by a measuring instrument or measuring system, and the corresponding values realized by standards traceable to a nationally or internationally recognized laboratory.

3.1.1.1 *Discussion*—Calibration conditions include environmental and irradiation conditions present during irradiation, storage and measurement of the dosimeters that are used for the generation of a calibration curve. To achieve stable environmental conditions, it may be necessary to condition the dosimeters before performing the calibration procedure.

3.1.2 *calibration curve*—graphical representation of the dosimetry system's response function.

3.1.3 *ceric-cerous dosimeter*—specially prepared solution of ceric sulfate and cerous sulfate in sulfuric acid, individually sealed in an appropriate container such as a glass ampoule, where the radiation-induced changes in electropotential or optical absorbance of the solution are related to absorbed dose to water.

3.1.4 *measurement quality assurance plan*—documented program for the measurement process that ensures that the expanded uncertainty consistently meets the requirements of the specific application. This plan requires traceability to nationally or internationally recognized standards.

3.1.5 *molar linear absorption coefficient,* ε_m—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:

$$\varepsilon_m = \frac{A_\lambda}{d \cdot c} \qquad (1)$$

SI unit: $m^2\ mol^{-1}$

3.1.5.1 *Discussion*—The measurement is sometimes expressed in units of $L\ mol^{-1}\ cm^{-1}$.

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

$$\Delta A = |A - A_o| \qquad (2)$$

3.1.7 *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.

$$G(x) = \frac{n(x)}{\bar{\varepsilon}} \qquad (3)$$

SI unit: $mol\ J^{-1}$

3.1.8 *reference–standard dosimeter*—dosimeter of high metrological quality used as a standard to provide measurements traceable to measurements made using primary–standard dosimeters.

3.1.9 *response function*—mathematical representation of the relationship between dosimeter response and absorbed dose, for a given dosimetry system.

3.1.10 *routine dosimeter*—dosimeter calibrated against a primary–, reference–, or transfer–standard dosimeter and used for routine absorbed-dose measurements.

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *electropotential, E*—difference in potential between the solutions in the two compartments of an electrochemical cell, measured in millivolts.

3.3 For definitions of other terms used in this practice that pertain to radiation measurement and dosimetry, refer to ASTM Terminology E 170. Definitions in ASTM Terminology E 170 are compatible with ICRU 60; that document, therefore, may be used as an alternative reference.

4. Significance and use

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

NOTE 3—The ceric-cerous system described in the practice has cerous sulfate added to the initial solution to reduce the effect of organic impurities and to allow the potentiometric method of measurement. Other systems used for dosimetry include solutions of ceric sulfate or ceric ammonium sulfate in sulfuric acid without the initial addition of cerous sulfate. These other systems are based on the same process of reduction of ceric ions to cerous ions but are not included in this practice.

4.2 The dosimeter is a solution of ceric sulfate and cerous sulfate in sulfuric acid in an appropriate container such as a flame-sealed glass ampoule. The solution indicates a level of absorbed dose by a change (decrease) in optical absorbance at a specified wavelength in the ultraviolet region, or a change (increase) in electropotential. A calibrated spectrophotometer is used to determine the absorbance and a potentiometer, with a specially designed cell, is used to determine the electropotential in millivolts.

4.3 The dosimeter response has an irradiation temperature dependence since the radiation chemical yield ($G(Ce^{3+})$) depends on temperature. The dependence of $G(Ce^{3+})$ is approximately equal to −0.2 % per degree Celsius between 0 and 62°C **(3, 5, 6)**. This irradiation temperature dependence has a slight dependence on the initial cerous ion concentration (see 10.6.3).

4.4 The absorbed dose to materials other than water when irradiated under equivalent conditions may be calculated. Procedures for making such calculations are given in ASTM Practices E 666 and E 668 and ISO/ASTM Guide 51261.

NOTE 4—For a comprehensive discussion of various dosimetry methods applicable to the radiation types and energies discussed in this practice, see ICRU Reports 14, 34, 35, and 37.

5. Interferences

5.1 The ceric-cerous dosimetric solution response is sensitive to impurities, particularly organic impurities. Even in trace quantities, impurities can cause a detectable change in the observed response **(7)**. Organic materials shall not be used for any component in contact with the solution unless they have been tested and shown to have no effect. The effect of trace impurities is minimized by the addition of cerous ions to the solution **(8, 9)**. Water purification methods found to be adequate for use in preparing ceric-cerous dosimeters are decribed in 7.2.

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

6. Apparatus

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

6.2 *Potentiometric Method*—Use an electrochemical cell, similar to that described in Annex A1 (see Fig. A1.1). Measure the electropotential across the cell with a high-precision digital potentiometer that is capable of measuring dc potentials in the range from 1 to 100 mV within an uncertainty of 1 %.

NOTE 5—As shown in Fig. A1.1, the electrochemical cell has two compartments separated by a porous junction, such as a glass frit, a ceramic or kaolin junction, or a fibreglass wick. The inner compartment is filled with unirradiated solution. The lower compartment is filled with solution transferred from an irradiated or unirradiated ampoule. The electropotential, E, generated between the platinum electrodes in the two compartments is measured by a digital potentiometer.

6.3 *Glassware*—Use borosilicate glass or equivalent chemically resistant glass to store the reagents and the prepared dosimetric solution. Clean all glassware, except ampoules, using chromic acid cleaning solution or an equivalent cleaning agent (see ASTM Practice C 912). Rinse at least three times with double-distilled water. Dry thoroughly and store under conditions that will minimize exposure to dust.

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

NOTE 6—The dosimetric ampoule normally used has a capacity of approximately 2 mL. Quick-break glass ampoules, or Type 1 glass colorbreak ampoules or equivalent containers, are commonly used. Commercially available ampoules have been found to give reproducible results without requiring additional cleaning.

7. Reagents

7.1 Analytical reagent grade chemicals shall be used for preparing all solutions.[5]

7.2 Water quality is very important since it is the major component of the dosimetric solutions, and therefore may be the prime source of contamination. The water quality is more important for ceric-cerous dosimeters used for measurements in the lower absorbed-dose range than for those used in the upper absorbed-dose range. For high-range dosimeters double-distilled water from coupled all-glass and silica stills can be used. For low-range dosimeters, use triply-distilled water. Alternatively, use water from a high-quality commercial purification unit capable of achieving Total Oxidizable Carbon (T.O.C.) content below 5 ppb. Use of deionized water is not recommended.

NOTE 7—Double-distilled water distilled from an alkaline potassium permanganate ($KMnO_4$) solution (2 g $KMnO_4$ plus 5 g sodium hydroxide (NaOH) pellets in 2 L of distilled water) has been found to be adequate for routine preparation of the dosimetric solution. High-purity water is commercially available from some suppliers. Such water labeled HPLC (high-pressure liquid chromatographic) grade is usually sufficiently free from organics to be used in this practice.

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

8. Preparation of the dosimeters

8.1 The recommended concentrations for the ceric-cerous dosimeter to measure absorbed doses from about 5 to 50 kGy (high-range dosimeter) are 15 mmol dm^{-3}ceric sulfate $[Ce(SO_4)_2 \cdot 4H_2O]$ and 15 mmol dm^{-3} cerous sulfate $[Ce_2(SO_4)_3 \cdot 8H_2O]$. For measurement of absorbed doses from about 0.5 to 10 kGy (low-range dosimeter), the recommended concentrations are 3 mmol $dm^{-3}[Ce(SO_4)_2 \cdot 4H_2O]$ and 3 mmol $dm^{-3}[Ce_2(SO_4)_3 \cdot 8H_2O]$.

8.2 The dosimetric solutions specified in 8.1 may be formulated from the following nominal stock solutions: *(a)* 0.4 mol dm^{-3} and 4 mol dm^{-3} sulfuric acid (H_2SO_4), *(b)* 0.1 mol dm^{-3} $Ce(SO_4)_2 \cdot 4H_2O$, and *(c)* 0.1 mol dm^{-3} $Ce_2(SO_4)_3 \cdot 8H_2O$. Procedures for preparing these solutions are given in Annex A2. (**Warning**—Concentrated sulfuric acid is corrosive and can cause serious burns. Ceric-cerous solutions are skin irritants. Appropriate precautions should be exercised in handling these materials.)

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

8.3 Use the following equations to determine the volume in millilitres of each stock solution necessary to prepare 1 L of dosimetric solution:

High Range **Low Range**

$$\frac{V_1}{1000} = \frac{0.015}{c_1} \qquad \frac{V_1}{1000} = \frac{0.003}{c_1} \tag{4}$$

$$\frac{V_2}{1000} = \frac{0.015}{c_2} \qquad \frac{V_2}{1000} = \frac{0.003}{c_2} \tag{5}$$

$$\frac{V_3}{1000 - V_1} = \frac{0.4}{c_3} \qquad \frac{V_3}{1000 - V_1} = \frac{0.4}{c_3} \tag{6}$$

$$V_4 = 1000 - V_1 - V_2 - V_3 \qquad V_4 = 1000 - V_1 - V_2 - V_3 \tag{7}$$

where:

V_1 = volume of nominal 0.1 mol dm^{-3} ceric-sulfate stock solution,
V_2 = volume of nominal 0.1 mol dm^{-3} cerous-sulfate stock solution,
V_3 = volume of nominal 4 mol dm^{-3} sulfuric-acid stock solution,
V_4 = volume of distilled water,
c_1 = actual concentration of the ceric-sulfate stock solution,
c_2 = actual concentration of the cerous-sulfate stock solution, and
c_3 = actual concentration of the nominal 4 mol dm^{-3} sulfuric-acid stock solution.

NOTE 8—If the nominal concentrations of $c_1 = c_2 = 0.1$ mol dm^{-3}, and $c_3 = 4$ mol dm^{-3} are assumed, then $V_1 = V_2 = 150$ mL for the high range and $V_1 = V_2 = 30$ mL for the low range; $V_3 = 85$ mL for the high range and $V_3 = 97$ mL for the low range. If the concentrations of the various stock solutions are significantly different from the nominal values, then use Eq 4-6 to determine the exact volumes. To prepare a volume of the dosimetric solution other than 1000 mL, the result of these equations should be multiplied by the ratio of the desired volume in millilitres to 1000 mL.

8.4 Determine all of the volumes given in 8.3 using a calibrated graduated cylinder that can be read to within ±0.5 mL.

8.5 Transfer the volume of each component of the dosimetric solution into a 1-L or larger glass storage container. Rinse the graduated cylinder used for measuring V_1, V_2, and V_3 by using some portion of the distilled water of V_4. Stopper the container and shake well. Before use, allow the dosimetric solution to stand for at least five days in the dark.

8.6 Quality control testing of the dosimetric solution prior to ampouling is performed by comparing the measurement of dosimetric solution parameters, such as ceric-ion concentration, cerous-ion concentration, ceric-ion molar linear absorption coefficient, radiation chemical yield for the cerous ion, and density with acceptable values. Procedures for performing these measurements are given in Annex A3. Quality control testing following ampouling is performed by comparing calibration data for the new dosimeter batch with data obtained from previous batches (see 10.5.3).

8.7 Prepare dosimeters by filling ampoules with approximately 2 mL of dosimetric solution. Take care not to contaminate the dosimetric solution with impurities. Exercise care in filling ampoules to avoid depositing solution in the ampoule neck. Subsequent heating during sealing may cause an undesirable chemical change in the dosimetric solution remaining inside the ampoule neck. Flame seal the ampoules, exercising care to avoid heating the body of the ampoule during sealing.

8.8 Store dosimeters in a dark place at room temperature (23 ± 5°C).

9. Analytical instrument performance

9.1 *Spectrophotometer Performance*:

9.1.1 Check the wavelength scale of the spectrophotometer. Appropriate wavelength standards are holmium-oxide filters and solutions. For more details see ASTM Practices E 275, E 925, and E 958.

NOTE 9—For example, holmium-oxide solutions in sealed cuvettes are available as certified wavelength standards (SRM 2034) for use in the wavelength region from 240 to 650 nm **(10).**

9.1.2 Check the accuracy of the photometric (absorbance) scale of the spectrophotometer. Certified absorbance standard filters or solutions are available for this purpose.

NOTE 10—Examples of absorbance standards are solutions of various concentrations, such as SRM 931d **(11)** and SRM 935 **(12)**, and metal-on-quartz filters, such as SRM 2031 **(13, 14)**.

9.1.3 Check the linearity of the absorbance scale of the spectrophotometer as a function of the ceric-ion concentration. This should be done at the peak of the absorbance spectrum for the ceric ion at 320 nm at a constant temperature, preferably 25°C. The standardized ceric-sulfate stock solution (0.1 mol dm^{-3} nominal in 0.4 mol dm^{-3} H_2SO_4), as described in A2.3, may be used for this measurement. The plot of measured absorbance, A, per unit path length versus concentration shall be linear. The slope of the line gives, ε_m, the molar linear absorption coefficient.

NOTE 11—A reference value for ε_m is 561 $m^2 \cdot mol^{-1} \pm 0.4$ % at 320 nm **(3)**.

9.2 *Potentiometer and Electrochemical Cell Performance*:

9.2.1 For the potentiometer method, correct performance can be demonstrated by showing that the readings of dosimeters given known absorbed doses are in agreement with the expected readings within the limits of the dosimetry system uncertainty (see Section 13).

NOTE 12—This method is only applicable for reference-standard dosimetry systems where the long-term stability of the response has been demonstrated and documented.

10. Calibration of the dosimetry system

10.1 The dosimetry system shall be calibrated prior to use and at intervals thereafter in accordance with the user's documented procedure that specifies details of the calibration process and quality assurance requirements. Calibration requirements are given in ISO/ASTM Guide 51261.

10.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations shall be performed at a national or accredited laboratory using criteria specified in ISO/ASTM Practice 51400.

10.2.1 Specify the dose in terms of absorbed dose to water.

10.2.2 When the ceric-cerous dosimeter is used as a routine dosimeter, the calibration irradiation may be performed by irradiating the dosimeters at *(a)* a national or accredited laboratory using criteria specified in ISO/ASTM Practice 51400, *(b)* an in-house calibration facility that provides an absorbed dose (or an absorbed-dose rate) having measurement traceability to nationally or internationally recognized standards, or *(c)* a production irradiator under actual production irradiation conditions, together with reference– or transfer––standard dosimeters that have measurement traceability to nationally or internationally recognized standards.

10.3 *Measurement Instrument Calibration and Performance Verification*—For the calibration of instruments (spectrophotometer or multimeter), and for the verification of instrument performance between calibrations, see ISO/ASTM Guide 51261 and/or instrument-specific manuals.

10.4 *Spectrophotometric Measurement*:

10.4.1 For the spectrophotometric measurement, separate at least five dosimeters from the remainder of the batch and do not irradiate them. Use them in determining $\bar{A}_o$.

10.4.2 For spectrophotometric measurement of both unirradiated and irradiated dosimeters, dilute high-range dosimetric solutions by a factor of 100 and low-range dosimetric solutions by a factor of 50.

10.4.2.1 Pipette 0.25 mL of high-range dosimetric solution or 0.5 mL of low-range dosimetric solution into a clean, dry 25-mL volumetric flask.

10.4.2.2 Rinse the pipette with 0.4 mol dm^{-3} H_2SO_4 into the flask and make up to volume with 0.4 mol dm^{-3} H_2SO_4.

10.4.2.3 Stopper the 25-mL flask, and mix well.

10.4.3 Transfer an appropriate amount into a quartz spectrophotometric cuvette (sample cell) from the 25-mL volumetric flask.

10.4.4 Read the absorbance, *A*, in the spectrophotometer at 320 nm.

10.4.5 Calculate the mean absorbance of the unirradiated dosimeters, $\bar{A}_o$. Calculate the net absorbance, Δ*A*, for each irradiated dosimeter:

$$\Delta A = \bar{A}_o - A \qquad (8)$$

10.5 *Potentiometric Measurement*:

10.5.1 Place contents of an unirradiated dosimeter (ampoule) into both compartments of the electrochemical cell. See Annex A1 for a description of the electrochemical cell.[6]

10.5.2 Allow the solution to remain in the electrochemical cell for about 30 min in order to establish equilibrium across the porous junction. For a new batch of dosimeters, or if a cell has not been used for one or more days, solution should be left in both compartments for at least 16 h to ensure equilibrium across the porous junction.

10.5.3 When the cell is being used for the first time, the filled cell should be left to stand for at least 24 h before making any measurements.

10.5.4 If the cell is not going to be used for more than three days, drain all solution from the cell. Rinse both the inner and outer compartments three times with distilled water, and allow the cell to air dry. Refer to 10.5.1 and 10.5.2 before reusing the cell.

10.5.5 Drain the inner compartment and refill it with the contents of another unirradiated dosimeter.

10.5.6 Connect the digital potentiometer across the cell. If the electropotential, *E*, is equal to zero (within ±0.2 mV), the cell is ready for use. Read at least three unirradiated dosimeters, and determine average value $\bar{E}_o$.

10.5.7 Expel the unirradiated solution from the outer compartment and draw in the solution from each irradiated dosimeter (ampoule) in turn, starting with the lowest and proceeding to the highest absorbed dose. In each case, before measuring the electropotential for any particular dosimeter, rinse the cell by drawing in a little less than half of that dosimeter's solution in order to reduce the effects of the previous dosimeter. Expel the rinse solution into a waste container, and then draw in sufficient solution from that remaining in the dosimeter ampoule to fully cover the porous junction.

NOTE 13—Inadequate rinsing of the cell between dosimetric solutions can lead to errors due to solution carryover. If the approximate absorbed doses are known, read the dosimeters that received similar absorbed doses together to minimize the errors from this effect.

10.5.8 Read the electropotential, *E*, in millivolts, across the cell for each dosimeter after temperature equilibrium is established within the cell. Subtract the average electropotential, $\bar{E}_o$, to determine Δ*E*, the net electropotential value. Measure the readout temperature near the electrochemical cell, and apply correction for this temperature.

NOTE 14—The electropotential, *E*, within the electrochemical cell, has a positive temperature coefficient of 0.33 % per °C between 25 and 30°C **(8)**.

10.6 *Analysis*:

10.6.1 Obtain a response function for Δ*A* or Δ*E* as a function of the 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 ceric-cerous dosimeters should fit a third or fourth order polynomial of the form:

$$v = b_0 + b_1 D + b_2 D^2 + b_2 D^3 + b_4 D^4 \qquad (9)$$

where:

v = Δ*A* or Δ*E*,
b_4 = 0 for third order polynomial, $b_4 \neq 0$ for fourth order polynomial.

NOTE 15—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 Guide 51707.

10.6.2 The inverse of the response function determined in 10.6.1 will provide the calibration–temperature dose for irradiations performed at the temperature, T_c, used for the calibration irradiations.

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[6] Electrochemical cells can be obtained from MDS Nordion, 447 March Road, Ottaw, Ontario, Canada K2K 1X8.

10.6.3 The irradiation temperature dependence of the radiation chemical yield $G(\mathrm{Ce}^{3+})$ varies with the initial cerous ion concentration **(3)**. The variation of $G(\mathrm{Ce}^{3+})$ with temperature, T (°C), for the high range and low range solutions is given by the following equations:

where:

$$G(\mathrm{Ce}^{3+})_T = (2.33544 - 0.0052 \times T) \times 1.036 \times 10^{-7} \quad \text{(high range)} \tag{10}$$

$$G(\mathrm{Ce}^{3+})_T = (2.42452 - 0.0052 \times T) \times 1.036 \times 10^{-7} \quad \text{(low range)} \tag{11}$$

10.6.4 The absorbed dose is approximately inversely proportional to $G(\mathrm{Ce}^{3+})$ over the absorbed-dose range for the solution. For irradiations at an effective irradiation temperature, T_{eff}, correct the absorbed dose by the ratio $G(\mathrm{Ce}^{3+})_{T_c}$ / $G(\mathrm{Ce}^{3+})_{T_{\mathrm{eff}}}$.

10.6.5 For quality control, compare the net absorbances or net electropotentials determined for a given calibration with the results obtained from previous batches. Agreement should be within 3 % if the dosimetric solutions were properly prepared and all associated analysis equipment was properly calibrated.

10.6.6 Estimate the component of uncertainty that can be evaluated by statistical methods (Type A) of the individual dosimeter results from the results of replicate measurements at a given absorbed-dose level. Type A uncertainty provides a measure of acceptable performance of the dosimetry system. For the high-range dosimeter, the Type A uncertainty, expressed as one standard deviation, should not exceed 0.005 absorbance units for an optical pathlength of 10 mm or 2 % of the electropotential value. For the low-range dosimeter, the Type A uncertainty should not exceed 0.010 absorbance units or 2 % of the electropotential value. Suspected data outliers should be tested using statistical procedures, such as those found in ASTM Practice E 178.

10.7 *Alternative Method for Determining Absorbed Dose*—If the procedures for quantifying dosimetric solution parameters given in Annex A3 are carried out, approximate values for the absorbed dose can be obtained from analytical functions.

10.7.1 For spectrophotometric readings, calculate an approximate value for the absorbed dose, D_s, in grays, using the following equation:

$$D_s = \frac{f \cdot \Delta A}{G\,(\mathrm{Ce}^{3+}) \cdot \varepsilon_m \cdot \rho \cdot d} \tag{12}$$

where:

f = dilution factor for the irradiated dosimeters,
ΔA = change in absorbance of irradiated dosimeter,
$G(\mathrm{Ce}^{3+})$ = average value for $G(\mathrm{Ce}^{3+})$ determined from Eq A3.9,
ε_m = molar-linear absorption coefficient ($\mathrm{m}^2\ \mathrm{mol}^{-1}$),
ρ = density of the dosimetric solution, kg m^{-3}, and
d = path length of spectrophotometer cell, m.

10.7.2 For potentiometric readings, calculate an approximate value for the absorbed dose, D_p, in grays using the following equation:

$$D_\rho = \frac{10^3}{\rho G(\mathrm{Ce}^{3+})}\left[c_4 - \left(\frac{c_4 + c_5}{1 + \frac{c_5}{c_4}\,\mathrm{antilog}\,\frac{\Delta E}{59.16}}\right)\right] \tag{13}$$

where:

ρ = density of the dosimetric solution, kg m^{-3},
$G(\mathrm{Ce}^{3+})$ = average value for $G(\mathrm{Ce}^{3+})$ determined from Eq A3.11,
ΔE = electropotential minus average electropotential for unirradiated dosimeters, and
c_4 and c_5 = concentrations of the ceric and cerous ions in the unirradiated dosimetric solutions, respectively, mol dm^{-3}.

10.7.3 Determine the relationship for the calculated absorbed doses as a function of the absorbed-dose values used for the calibration irradiations. The function describing this relationship may be used for the determination of the absorbed dose.

NOTE 16—The calculated absorbed dose values should be close to the absorbed dose values used for the calibration irradiations, providing a good fit to a low order (first to third order) polynomial. If the calculated absorbed dose values differ by more than the expanded uncertainty with a coverage factor k=2 from the absorbed-dose values used for the calibration irradiations, there is an indication of possible contamination of the solution, or some other problem that needs to be resolved.

11. Application of dosimetry system

11.1 For use as a transfer-standard dosimeter, use a minimum of two dosimeters for each absorbed-dose measurement. The number of dosimeters required for the measurement of absorbed dose on or within a material is determined by the estimated uncertainty of the dosimetry system and the required uncertainty associated with the application. Appendix X3 of ASTM Practice E 668 describes a statistical method for determining this number.

11.2 Use the irradiation and measurement procedures in accordance with Section 10.

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

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 of the Dosimetry System*:

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

12.1.2 Record or reference the date, irradiation temperature, temperature variation (if any), absorbed-dose range, radiation source, and associated instrumentation used to measure the dosimeter response.

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 or electropotential, net absorbance or net electropotential value, temperature correction (if applicable), and resulting absorbed dose for each dosimeter.

Reference the calibration curve or response function used to obtain the absorbed-dose values.

12.2.4 Record or reference the components of uncertainty in the value of the absorbed dose.

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

13. Measurement uncertainty

13.1 To be meaningful, a measurement of absorbed dose shall be accompanied by an estimate of uncertainty.

13.2 Components of uncertainty shall be identified as belonging to one of two categories:

13.2.1 Type *A*—those that are evaluated by statistical methods or

13.2.2 Type *B*—those evaluated by other means.

13.3 Other ways of categorizing uncertainty have been widely used and may be useful for reporting uncertainty. For example, the terms *precision* and *bias* or *random* and *systematic* (non-random) are used to describe different categories of uncertainty.

13.4 If this practice is followed, the estimate of the expanded uncertainty of an absorbed dose determined by this dosimetry system should be less than 4 % for a coverage factor $k = 2$ (which corresponds approximately to a 95 % level of confidence for normally distributed data).

NOTE 17—The identification of Type A and Type B uncertainties is based on methodology published in 1995 by the International Organization for Standardization (ISO) in the Guide to the Expression of Uncertainty in Measurement **(15)**. The purpose of this type of characterization is to promote an understanding of how uncertainty statements are developed and to provide a basis for the international comparison of measurement results.

NOTE 18—ISO/ASTM Guide 51707 defines possible sources of uncertainty in dosimetry performed in radiation processing facilities and offers procedures for estimating the magnitude of the resulting uncertainties in the measurement of absorbed dose using a dosimetry system. The document defines and discusses basic concepts of measurement, including estimation of the measured value of a quantity, "true" value, error, and uncertainty. Components of uncertainty are discussed, and methods are provided for evaluating and estimating their values. Methods are also provided for calculating the combined standard uncertainty and estimating expanded (overall) uncertainty.

14. Keywords

14.1 absorbed dose; ceric-cerous sulfate dosimeter; dose; dose measurement; dosimeter; dosimetry system; electron beam; gamma radiation; ionizing radiation; irradiation; photons; radiation; radiation processing; reference-standard dosimeter; X-radiation; ICS 17.240

ANNEXES

(informative)

A1. ELECTROCHEMICAL CELL

A1.1 The electrochemical cell shown in Fig. A1.1 has both an inner and outer compartment, *A* and *B*, respectively, separated by a porous junction, *C*, such as a glass frit, a ceramic or kaolin junction, or a fibreglass wick.

A1.2 Compartment *A* contains the unirradiated dosimetric solution.

A1.3 Compartment *B* contains either unirradiated or irradiated dosimetric solution.

A1.4 The porous junction, *C*, provides contact between the two solutions and shall have a porosity of less than 2 µm.

A1.5 The small open tip of compartment *B* is inserted into the neck of a dosimeter ampoule, *D*. The glass syringe, *E*, is used alternately to draw into compartment *B* the dosimetric solution and then expel the solution after measurement.

A1.6 Leads *F* and *G* provide a means for measuring the potential difference across the electrochemical cell when they are connected to a potentiometer or multimeter.

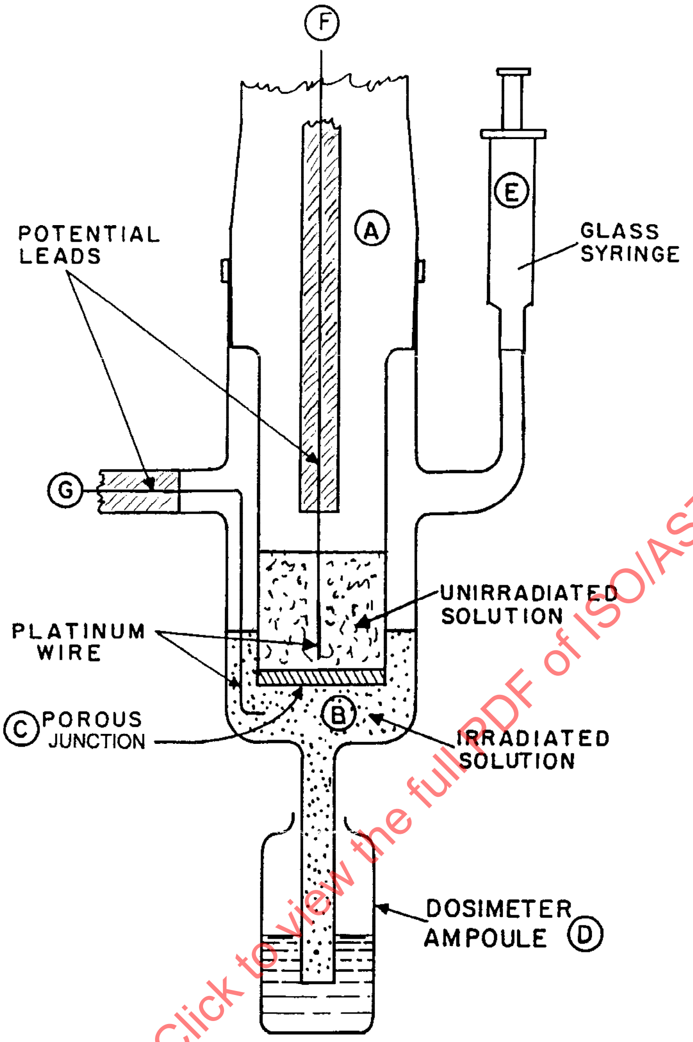

FIG. A1.1 Electrochemical cell

A2. PROCEDURE FOR PREPARING NOMINAL STOCK SOLUTIONS FOR THE DOSIMETER

A2.1 *Nominal 0.4 mol dm^{-3} Sulfuric Acid (H_2SO_4)*:

A2.1.1 Transfer 22.2 mL of 18 mol dm^{-3} H_2SO_4 into a clean, dry 1-L volumetric flask containing about 700 mL of double-distilled water.

A2.1.2 Carefully cool contents and make up to volume with double-distilled water, stopper, and mix well.

A2.1.3 If desired, the resulting solution can be standardized using sodium carbonate primary standard or equivalent.

NOTE A2.1—Add H_2SO_4 cautiously to the water as a considerable amount of heat is released.

A2.2 *Nominal 4 mol dm^{-3} Sulfuric Acid (H_2SO_4)*:

A2.2.1 Transfer 222 mL of 18 mol dm^{-3} H_2SO_4 into a clean, dry 1-L volumetric flask containing about 700 mL of double-distilled water.

A2.2.2 Carefully cool contents and make up to volume with double-distilled water, stopper, and mix well.

A2.2.3 Standardize resulting solution similarly as for 0.4 mol dm^{-3} H_2SO_4.

A2.3 *Nominal 0.1 mol dm^{-3} Ceric Sulfate:*

A2.3.1 In a 1-L volumetric flask, dissolve 58 g of ceric sulfate, $Ce(SO_4)_2 \cdot 4H_2O$, in 600 mL of 0.4 mol dm^{-3} H_2SO_4.

A2.3.2 Shake contents until all $Ce(SO_4)_2 \cdot 4H_2O$ is dissolved. Allow solution to stand at least two weeks in the dark.

A2.3.3 Add enough 0.4 mol dm^{-3} H_2SO_4 solution to dilute to 1 L. Filter if necessary through a clean sintered-glass filter (medium porosity).

A2.3.4 Transfer into a glass bottle provided with a ground glass stopper, and store in a dark place.

A2.4 *Nominal 0.1 mol dm^{-3} Cerous Sulfate:*

A2.4.1 In a 1-L volumetric flask, dissolve 36 g cerous sulfate, $Ce_2(SO_4)_3 \cdot 8H_2O$ in 600 mL of double-distilled water.