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Third edition
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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-céreuse*

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. International Standards are drafted in accordance with the editorial rules of ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by ASTM Committee E61 Radiation Processing and by Technical Committee ISO/TC 85, nuclear energy, nuclear technologies and radiological protection.

This third edition cancels and replaces the second edition (ISO/ASTM 51205:2009), which has been technically revised.

ISO/ASTM 51205:2017(E)



Standard Practice for Use of a Ceric-Cerous Sulfate Dosimetry System¹

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 preparation, testing, and procedure for using the ceric-cerous sulfate dosimetry system to measure absorbed dose to 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 ceric-cerous system. The ceric-cerous dosimeter is classified as a type 1 dosimeter on the basis of the effect of influence quantities. The ceric-cerous system may be used as a reference standard dosimetry system or as a routine dosimetry system.

1.2 This document is one of a set of standards that provides recommendations for properly implementing dosimetry in radiation processing, and describes a means of achieving compliance with the requirements of ISO/ASTM Practice 52628 for the ceric-cerous system. It is intended to be read in conjunction with ISO/ASTM Practice 52628.

1.3 This practice describes both the spectrophotometric and the potentiometric readout procedures for the ceric-cerous 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 conditions are satisfied:

1.5.1 The absorbed-dose range is from 5×10^{-2} to 5×10^4 Gy (1).²

1.5.2 The absorbed-dose rate does not exceed 10^6 Gy s⁻¹ (1).

1.5.3 For radionuclide gamma-ray sources, the initial photon energy is greater than 0.6 MeV. For bremsstrahlung photons, the initial 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.

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, and is also under the jurisdiction of ISO/TC 85/WG 3.

Current edition approved March 8, 2017. Published May 2017. Originally published as ASTM E1205–88. Last previous ASTM edition E1205–99. ASTM E1205–93 was adopted by ISO in 1998 with the intermediate designation ISO 15555:1998(E). The present International Standard ISO/ASTM 51205:2017(E) is a major revision of ISO/ASTM 51205:2009(E).

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

NOTE 1—The lower energy limits are appropriate for a cylindrical dosimeter ampoule of 12-mm diameter. Corrections for dose gradient across the ampoule 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 (see ICRU Report 35).

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

NOTE 2—The temperature coefficient of dosimeter response is known only in this range (see 5.2). Use outside this range requires determination of the temperature coefficient.

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.

1.7 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

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

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.



Ultraviolet-Visible Spectrophotometers

2.2 ISO/ASTM Standards:³

51261 Practice for Calibration of Routine Dosimetry Systems for Radiation Processing

51707 Guide for Estimation of Measurement Uncertainty in Dosimetry for Radiation Processing

52628 Practice for Dosimetry in Radiation Processing

52701 Guide for Performance Characterization of Dosimeters and Dosimetry Systems for Use in Radiation Processing

2.3 ISO Standards:⁴

12749-4 Nuclear energy – Vocabulary – Part 4: Dosimetry for radiation processing

2.4 ISO/IEC Standards:⁴

17025 General Requirements for the Competence of Testing and Calibration Laboratories

2.5 Joint Committee for Guides in Metrology (JCGM) Reports:

JCGM 100:2008, GUM 1995, with minor corrections, Evaluation of measurement data – Guide to the Expression of Uncertainty in Measurement⁵JCGM 200:2012 (JCGM 200:2008 with minor revisions), VIM, International Vocabulary of Metrology – Basis and General Concepts and Associated Terms⁶2.6 International Commission on Radiation Units and Measurements (ICRU) Reports:⁷

ICRU Report 10b (NBS Handbook 85) Physical Aspects of Irradiation

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

ICRU Report 80 Dosimetry Systems for Use in Radiation Processing

ICRU Report 85a Fundamental Quantities and Units for Ionizing Radiation

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 Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

⁵ 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>).

⁶ Document produced by Working Group 2 of the Joint Committee for Guides in Metrology (JCGM WG2), Available free of charge at the BIPM website (<http://www.bipm.org>).

⁷ Available from International Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814, USA.

3.1.2 *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.

3.1.3 *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:

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

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

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

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

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

SI unit: mol J^{-1}

3.1.5 *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.

3.1.6 *type 1 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 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 Definitions of other terms used in this practice that pertain to radiation measurement and dosimetry may be found in ISO 12749-4, ASTM Terminology E170, ICRU 85a, and VIM; these documents, therefore, may be used as alternative references.

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, ICRU Report 80).

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.

5. Effect of influence quantities

5.1 Guidance on the determination of the performance characteristics of dosimeters and dosimetry systems can be



found in ASTM Guide 52701. The relevant quantities that need to be considered when using the ceric-cerous dosimetry system are given below.

5.2 The dosimeter response has a temperature dependence during irradiation that 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 10.6.2).

5.3 The electropotential, E , within the electrochemical cell, has a positive temperature coefficient of 0.33% per $^\circ\text{C}$ between 25°C and 30°C and corrections are required for differences between measurement temperatures and the reference temperature used during calibration (see 10.5.8).

5.4 No effect of ambient light (even direct sunlight) has been observed on dosimetric solutions in glass ampoules.

5.5 The dosimeter response is dependent on the type and energy of the radiation employed. Since cerium is a heavy element from the viewpoint of absorption characteristics of gamma radiation, the response of the dosimetric solution for lower energy degraded radiation during use may be greater than the response in the cobalt-60 radiation during calibration (7). However, studies in an industrial gamma irradiator indicate that this effect is small (8).

5.6 If the dosimetric solution is prepared as described in this document, and steps are taken to avoid contamination, the dosimetric solution stored, or sealed, in glass vessels (for example, ampoules) is stable for several years before and after irradiation.

6. Interferences

6.1 The ceric-cerous dosimetric response is sensitive to impurities, particularly organic impurities. Even in trace quantities, impurities can cause a detectable change in the observed response (9). Organic materials should 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 (10, 11). Water purification methods found to be adequate for use in preparing ceric-cerous dosimeters are described in 8.2.

6.2 Undesirable chemical changes in the dosimetric solution can occur if care is not taken during sealing of the ampoules (see 9.7).

7. Apparatus

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

7.2 *Potentiometric Method*—An electrochemical cell, similar to that described in Annex A1, should be used (see Fig. A1.1). The electropotential across the cell should be measured with a high-precision digital voltmeter that is capable of

measuring dc potentials in the range from 1 to 100 mV within an uncertainty of 1% .

NOTE 4—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 always filled with unirradiated solution. The lower compartment is filled with solution whose response is to be measured (transferred from an irradiated or unirradiated ampoule). The electropotential, E , generated between the platinum electrodes in the two compartments is measured by a digital voltmeter.

7.3 *Glassware*—Borosilicate glass or equivalent chemically resistant glass should be used to store the reagents and the prepared dosimetric solution. All glassware, except ampoules, should be cleaned using chromic acid cleaning solution or an equivalent cleaning agent (see ASTM Practice C912). Glassware should be rinsed at least three times with purified water, dried thoroughly and stored under conditions that will minimize exposure to dust.

7.4 *Glass Ampoules*—If required, glass ampoules should be cleaned in boiling purified water, rinsed twice with purified water, and oven dried.

NOTE 5—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.

8. Reagents

8.1 Analytical reagent grade (or better) chemicals shall be used for preparing all solutions.⁸

8.2 Water quality is very important since it is the major component of the dosimetric solutions, and therefore may be the prime source of contamination. Double-distilled water from coupled all-glass and silica stills or water from a high-quality commercial purification unit capable of achieving Total Oxidizable Carbon (T.O.C.) content below 5 ppb should be used. Use of deionized water is not recommended.

NOTE 6—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.

8.3 Purified water used in this practice should not be stored in plastic containers or in containers with plastic cap liners.

9. Preparation of the dosimeters

9.1 Recommended concentrations for the ceric-cerous dosimeter for measurement of absorbed doses from about 5 to 50 kGy (high-range dosimeter) are 15 mmol dm^{-3} ceric sulfate $[\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}]$ and 15 mmol dm^{-3} cerous sulfate $[\text{Ce}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}]$. For measurement of absorbed doses from about 0.5 to 10 kGy (low-range dosimeter), the recommended concentrations are 3 mmol dm^{-3} $[\text{Ce}(\text{SO}_4)_2 \cdot 4\text{H}_2\text{O}]$ and 3 mmol dm^{-3} $[\text{Ce}_2(\text{SO}_4)_3 \cdot 8\text{H}_2\text{O}]$.

⁸ Reagent specifications are available from American Chemical Society, 1115 16th St., Northwest, Washington, DC 20036, USA.



9.2 The dosimetric solutions specified in 9.1 may be formulated from the following nominal stock solutions: (a) 0.4 mol dm⁻³ and 4 mol dm⁻³ sulfuric acid (H₂SO₄), (b) 0.1 mol dm⁻³ Ce(SO₄)₂ · 4H₂O, and (c) 0.1 mol dm⁻³ Ce₂(SO₄)₃ · 8H₂O. 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.)

9.3 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	
$\frac{V_1}{1000} = \frac{0.015}{c_1}$	$\frac{V_1}{1000} = \frac{0.003}{c_1}$	(3)

$\frac{V_2}{1000} = \frac{0.015}{c_2}$	$\frac{V_2}{1000} = \frac{0.003}{c_2}$	(4)
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$\frac{V_3}{1000 - V_1} = \frac{0.4}{c_3}$	$\frac{V_3}{1000 - V_1} = \frac{0.4}{c_3}$	(5)
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$V_4 = 1000 - V_1 - V_2 - V_3$	$V_4 = 1000 - V_1 - V_2 - V_3$	(6)
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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 purified 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 7—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.

9.4 Determine all of the volumes given in 9.3 using a calibrated volumetric flask that can be read to within ±0.5 mL.

9.5 Transfer the volume of each component of the dosimetric solution into a 1-L or larger glass storage container. Rinse the volumetric flask used for measuring V_1 , V_2 , and V_3 by using some portion of the purified 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 (ICRU 10b).

9.6 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 10.6.4).

9.7 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.

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

10. Calibration of the dosimetry system

10.1 Prior to use, the dosimetry system (consisting of a specific batch of dosimeters and specific measurement instruments) shall be calibrated in accordance with the user's documented procedure that specifies details of the calibration and quality assurance requirements. This calibration shall be repeated at regular intervals to ensure that the accuracy of the absorbed-dose measurement is maintained within required limits. Calibration for routine dosimetry systems are described in ISO/ASTM Practice 51261.

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

10.2.1 When the ceric-cerous dosimeter is used in a reference standard dosimetry system, calibration irradiations shall be performed at an approved laboratory, as defined in 3.1.1.

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

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

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

10.2.5 The dosimeter shall be calibrated in a radiation field of the same type and energy as that in which it is to be used, unless evidence is available to demonstrate equivalence of response. If not, a correction factor has to be applied and its associated uncertainty must be added to the uncertainty budget.

10.2.6 Control (or monitor) the temperature of the dosimeters during irradiation. Calculate or measure the mean irradiation temperature of each dosimeter to an accuracy of ±2°C, or better.

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

10.2.8 Irradiate these sets of dosimeters to at least five known dose values for each factor of ten span of absorbed



doses covering the range of utilization in order to determine the calibration curve for the dosimetry system.

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

10.3.1 Spectrophotometer Performance:

10.3.1.1 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 ASTM Practices E275, E925, and E958.

NOTE 8—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 (12).

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

NOTE 9—Examples of absorbance standards are solutions of various concentrations, such as SRM 931d (13) and SRM 935 (14), and metal-ox-quartz filters, such as SRM 2031 (15,16).

10.3.2 Digital Voltmeter and Electrochemical Cell Performance:

10.3.2.1 For the potentiometric method, correct performance can be demonstrated by showing that the absorbed dose obtained from the measurement of dosimeters given known absorbed doses are in agreement with the given absorbed doses within the limits of the dosimetry system uncertainty (see Section 13).

NOTE 10—This method is only applicable for reference-standard dosimetry systems where the long-term stability of the response has been demonstrated and documented.

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 the average absorbance, $\bar{A}_0$.

10.4.2 For spectrophotometric measurement of both unirradiated and irradiated dosimeters, open dosimeter ampoules (break at neck if quick-break ampoules are used). 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 H₂SO₄ into the flask and make up to volume with 0.4 mol dm⁻³ H₂SO₄.

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 in a 1 cm path length cuvette.

10.4.5 Calculations:

10.4.5.1 Calculate the mean absorbance of the unirradiated dosimeters, $\bar{A}_0$.

10.4.5.2 Calculate the net absorbance, ΔA , for each irradiated dosimeter:

$$\Delta A = \bar{A}_0 - A \quad (7)$$

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.⁹

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 purified 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 voltmeter 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 $\bar{E}_0$.

NOTE 11—If the average electropotential, $\bar{E}_0$, is not equal to zero (greater than 0.2 mV or less than -0.2 mV), rinse cells again with unirradiated dosimetric solution. If still unable to obtain reading within ± 0.2 mV, confirm that cell is operating satisfactorily by reading dosimeters given known doses and confirming that results are within predetermined uncertainty limits.

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 12—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 between the dosimetric solution within the cell and the room temperature near the cell. Subtract the average

⁹ Electrochemical cells can be obtained from Nordion, 447 March Road, Ottawa, Ontario, Canada K2K 1X8.



electropotential, $\bar{E}_0$, to determine ΔE , the net electropotential value. Measure the readout temperature near the electrochemical cell, and apply correction for this temperature.

NOTE 13—The electropotential, E , within the electrochemical cell, has a positive temperature coefficient of 0.33 % per °C between 25°C and 30°C (10).

10.6 Analysis:

10.6.1 Obtain a calibration curve for ΔA or ΔE 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. For example, the data for ceric-cerous dosimeters can be fitted to a third or fourth order polynomial of the form:

$$v = b_0 + b_1 D + b_2 D^2 + b_3 D^3 + b_4 D^4 \quad (8)$$

where:

$v = \Delta A$ or ΔE ,

$b_4 = 0$ for third order polynomial, $b_4 \neq 0$ for fourth order polynomial.

NOTE 14—Computer software is available commercially for performing least-squares fits of data with polynomials or other analytical forms. Further information on mathematical methods for handling calibration data is given in ISO/ASTM Practice 51261 Appendix A2.

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

$$G(\text{Ce}^{3+})_T = (2.33544 - 0.0052 \times T) \times 1.036 \times 10^{-7} \quad (\text{high range}) \quad (9)$$

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

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

10.6.4 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.

10.6.5 The reproducibility (precision) for the individual dosimeter response values, estimated from the results of replicate measurements at a given absorbed-dose level provides a measure of acceptable performance of the dosimetry system. For the high-range dosimeter, the reproducibility, 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 reproducibility 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 E178.

11. Application of dosimetry system

11.1 For use as a transfer-standard dosimeter, a minimum of two dosimeters should be used 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 reproducibility associated with the dosimetry system and the required measurement uncertainty associated with the application. Appendix X3 of ASTM Practice E668 describes a statistical method for determining this number.

11.2 The irradiation and measurement procedures described in Section 10 for the calibration of the dosimetry system should also be followed when performing dosimetry with the ceric-cerous dosimeters.

11.3 The absorbed dose to water is determined from the net absorbance values or net electropotential values and the calibration curve.

NOTE 15—The absorbed dose to materials other than water irradiated under equivalent conditions may be calculated using the procedures given in ASTM Practices E666 and E668.

11.4 Requirements for recording the calculated absorbed dose values and all other relevant data are outlined in Section 12.

12. Minimum documentation requirements

12.1 Calibration:

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 analyze the dosimeters, measurement date, and the temperature during electropotential measurement for each dosimeter.

12.2 Application:

12.2.1 Record the date of irradiation and temperature of dosimeter during irradiation, temperature variation (if any), measurement date, and the temperature during electropotential 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 used to obtain the absorbed-dose values.

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

13.2 All components of uncertainty should be included in the estimate, including those arising from calibration, dosimeter reproducibility, instrument reproducibility, and the effect of influence quantities. A full quantitative analysis of components of uncertainty may be referred to as an uncertainty

budget, and is then often presented in the form of a table. Typically, the uncertainty budget will identify all significant components of uncertainty, together with their methods of estimation, statistical distributions and magnitudes.

13.3 If this practice is followed, the estimate of the expanded uncertainty of an absorbed dose determined by this dosimetry system should be less than 3 % for a coverage factor $k = 2$ for the high-range dosimeter and 4 % for a coverage

factor $k = 2$ for the low-range dosimeter. This corresponds approximately to a 95 % level of confidence for normally distributed data.

14. Keywords

14.1 absorbed dose; absorbed dose measurements; ceric-cerous dosimeter; ceric-cerous dosimetry system; ceric-cerous sulfate dosimeter; ionizing 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,

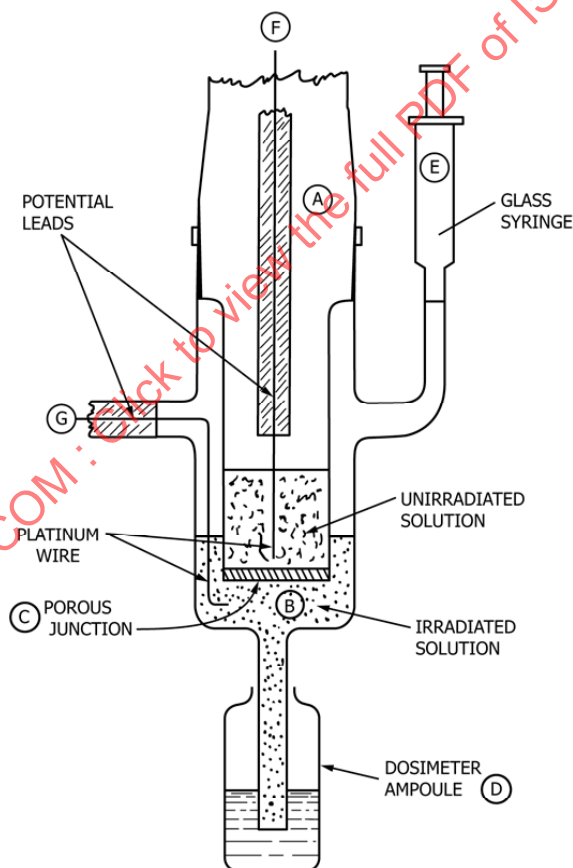


FIG. A1.1 Electrochemical cell



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 should 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 digital voltmeter.

A2. PROCEDURE FOR PREPARING NOMINAL STOCK SOLUTIONS FOR THE DOSIMETER

A2.1 Nominal 0.4 mol dm⁻³ Sulfuric Acid (H₂SO₄):

A2.1.1 Transfer 22.2 mL of 18 mol dm⁻³ H₂SO₄ into a clean, dry 1-L volumetric flask containing about 700 mL of purified water.

A2.1.2 Carefully cool contents and make up to volume with purified 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 H₂SO₄ cautiously to the water as a considerable amount of heat is released.

A2.2 Nominal 4 mol dm⁻³ Sulfuric Acid (H₂SO₄):

A2.2.1 Transfer 222 mL of 18 mol dm⁻³ H₂SO₄ into a clean, dry 1-L volumetric flask containing about 700 mL of purified water.

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

A2.2.3 Standardize resulting solution similarly as for 0.4 mol dm⁻³ H₂SO₄.

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(SO₄)₂ · 4H₂O, in 600 mL of 0.4 mol dm⁻³ H₂SO₄.

A2.3.2 Shake contents until all Ce(SO₄)₂ · 4H₂O is dissolved. Allow solution to stand at least two weeks in the dark.

A2.3.3 Add enough 0.4 mol dm⁻³ H₂SO₄ 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, Ce₂(SO₄)₃ · 8H₂O in 600 mL of purified water.

NOTE A2.2—Cerous sulfate may require recrystallization before use (6).

A2.4.2 Shake contents until all Ce₂(SO₄)₃ · 8H₂O is dissolved. Allow solution to stand at least two weeks in the dark.

A2.4.3 Add enough purified water to dilute to 1 L. Filter if necessary through a clean sintered-glass filter (medium porosity).

A2.4.4 Transfer into a glass bottle provided with a ground glass stopper, mix well, and store in a dark place.

A2.5 Concentration of Cerous-Sulfate Stock Solutions:

A2.5.1 Pipette with a *to contain* pipette 1 mL of the cerous-sulfate stock solution into a 100-mL volumetric flask, followed with a rinse of the pipette with purified water. Make up to volume with purified water, stopper, and mix well.

A2.5.2 Read the absorbance, *A*, at 254 nm in a spectrophotometer with purified water in the reference cuvette. The concentration, *c*₂, of the stock cerous-sulfate solution is given, in mol dm⁻³, by the following equation:

$$c_2 = (0.146)A \quad (\text{A2.1})$$