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Practice for use of a ceric-cerous sulfate dosimetry system

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

Reference number
ISO 15555:1998(E)

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.

International Standard ISO 15555 was prepared by the American Society for Testing and Materials (ASTM) Subcommittee E10.01 (as E 1205-93) and was adopted, under a special "fast-track procedure", by Technical Committee ISO/TC 85, *Nuclear energy*, in parallel with its approval by the ISO member bodies.

A new ISO/TC 85 Working Group WG 3, *High-level dosimetry for radiation processing*, was formed to review the voting comments from the ISO "Fast-track procedure" and to maintain these standards. The USA holds the convenership of this working group.

International Standard ISO 15555 is one of 20 standards developed and published by ASTM. The 20 fast-tracked standards and their associated ASTM designations are listed below:

ISO Designation	ASTM Designation	Title
15554	E 1204-93	*Practice for dosimetry in gamma irradiation facilities for food processing*
15555	E 1205-93	*Practice for use of a ceric-cerous sulfate dosimetry system*
15556	E 1261-94	*Guide for selection and calibration of dosimetry systems for radiation processing*
15557	E 1275-93	*Practice for use of a radiochromic film dosimetry system*
15558	E 1276-96	*Practice for use of a polymethylmethacrylate dosimetry system*
15559	E 1310-94	*Practice for use of a radiochromic optical waveguide dosimetry system*
15560	E 1400-95a	*Practice for characterization and performance of a high-dose radiation dosimetry calibration laboratory*
15561	E 1401-96	*Practice for use of a dichromate dosimetry system*

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15562	E 1431-91	*Practice for dosimetry in electron and bremsstrahlung irradiation facilities for food processing*
15563	E 1538-93	*Practice for use of the ethanol-chlorobenzene dosimetry system*
15564	E 1539-93	*Guide for use of radiation-sensitive indicators*
15565	E 1540-93	*Practice for use of a radiochromic liquid dosimetry system*
15566	E 1607-94	*Practice for use of the alanine-EPR dosimetry system*
15567	E 1608-94	*Practice for dosimetry in an X-ray (bremsstrahlung) facility for radiation processing*
15568	E 1631-96	*Practice for use of calorimetric dosimetry systems for electron beam dose measurements and dosimeter calibrations*
15569	E 1649-94	*Practice for dosimetry in an electron-beam facility for radiation processing at energies between 300 keV and 25 MeV*
15570	E 1650-94	*Practice for use of cellulose acetate dosimetry system*
15571	E 1702-95	*Practice for dosimetry in a gamma irradiation facility for radiation processing*
15572	E 1707-95	*Guide for estimating uncertainties in dosimetry for radiation processing*
15573	E 1818-96	*Practice for dosimetry in an electron-beam facility for radiation processing at energies between 80 keV and 300 keV*

Designation: E 1205 – 93

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

This standard is issued under the fixed designation E 1205; 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.

NOTE—Sections 8 and 10, with regard to low range dosimeters, are currently being reballoted by the subcommittee.

1. Scope

1.1 This practice covers the preparation, testing, and procedure for using the ceric-cerous sulfate dosimetry system to measure absorbed dose in water when exposed to ionizing radiation. For simplicity, the system will be referred to as the ceric-cerous system. It is classified as a reference standard dosimetry system (see Guide E 1261).

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

1.3 This practice applies only to γ rays, X-rays, and high energy electrons.

1.4 This practice applies provided the following are satisfied:

1.4.1 The absorbed-dose range shall be between 5×10^2 and 5×10^4 Gy (1).[2]

1.4.2 The absorbed-dose rate shall be less than 10^6 Gy/s (1).

1.4.3 For radionuclide gamma-ray sources, the initial photon energy shall be greater than 0.6 MeV. For bremsstrahlung photons, the initial energy of the electrons used to produce the bremsstrahlung photons shall be equal to or greater than 2 MeV. For electron beams, the initial electron energy shall be greater than 8 MeV.

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. The ceric-cerous system may be used at lower energies by employing thinner (in the beam direction) dosimeter containers (see ICRU Report 35).

1.4.4 The irradiation temperature of the dosimeter should be between 0 and 62°C.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

C 912 Practice for Designing a Process for Cleaning Technical Glasses[3]

D 941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer[4]

D 1193 Specification for Reagent Water[5]

E 170 Terminology Relating to Radiation Measurements and Dosimetry[6]

E 178 Practice for Dealing with Outlying Observations[7]

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[8]

E 666 Practice for Calculating Absorbed Dose from Gamma or X Radiation[6]

E 668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices[6]

E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers[8]

E 958 Practice for Measuring Practice Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers[8]

E 1026 Practice for Using the Fricke Reference Standard Dosimetry System[6]

E 1261 Guide for Selection and Application of Dosimetry Systems for Radiation Processing of Food[6]

E 1400 Practice for Characterization and Performance of a High-Dose Gamma Radiation Dosimetry Calibration Laboratory[6]

E 1401 Practice for Use of a Dichromate Dosimetry System[6]

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

ICRU Report 10b—Physical Aspects of Irradiation[9]

ICRU Report 14—Radiation Dosimetry: X-Rays and Gamma Rays with Maximum Photon Energies Between 0.6 and 60 MeV[9]

ICRU Report 33—Radiation Quantities and Units[9]

ICRU Report 34—The Dosimetry of Pulsed Radiation[9]

ICRU Report 35—Radiation Dosimetry: Electrons with Initial Energies Between 1 and 50 MeV[9]

[1] This practice is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.01 on Dosimetry for Radiation Processing.

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[2] The boldface numbers in parentheses refer to the list of references appended to this test method.

[3] *Annual Book of ASTM Standards*, Vol 15.02.
[4] *Annual Book of ASTM Standards*, Vol 05.01.
[5] *Annual Book of ASTM Standards*, Vol 11.01.
[6] *Annual Book of ASTM Standards*, Vol 12.02.
[7] *Annual Book of ASTM Standards*, Vol 14.02.
[8] *Annual Book of ASTM Standards*, Vol 14.01.
[9] Available from International Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814.

3. Terminology

3.1 *Definitions:*

3.1.1 *absorbed dose, D*—the quotient of $d\bar{\varepsilon}$ by dm, where $d\bar{\varepsilon}$ is the mean energy imparted by ionizing radiation to the matter of mass dm (see ICRU Report 33).

$$D = \frac{d\bar{\varepsilon}}{dm}$$

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

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

DISCUSSION—Formerly, the special unit for absorbed dose was the rad:

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

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

3.1.3 *electropotential*—difference in potential, ΔE, between irradiated and unirradiated solutions in an electrochemical cell measured in millivolts.

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

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

$$\epsilon = \frac{A}{Md}$$

where:

A = absorbance at a specified wavelength,
M = molar concentration of the ions of interest (that is, ceric or cerous), and
d = optical path length within the solution measured by the spectrophotometer.

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

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

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

$$\Delta A = A_o - A_i$$

3.1.7 *radiation chemical yield, G(x)*—quotient of $n(x)$ by $\bar{\varepsilon}$.

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

where:

$n(x)$ = mean amount of substance of a specified entity, x, produced, destroyed, or changed by the mean energy imparted, $\bar{\varepsilon}$, to matter (see ICRU Reports 14 and 34).

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

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

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

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

3.2 For other relevant terms, see Terminology E 170.

4. Significance and Use

4.1 The ceric-cerous system provides a reliable means for measuring absorbed dose in water. It is based on a process of reduction of ceric ions to cerous ions in acidic aqueous solution by ionizing radiation **(1, 2)**.

4.2 The dosimeter is a solution of ceric sulfate and cerous sulfate in sulphuric 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 change in absorbance and a potentiometer, with a specially designed cell, is used to determine the change in potential in millivolts.

4.3 The dosimeter response has a temperature dependence during irradiation of −0.2 % per degree celsius between 0 and 62°C.

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

4.5 The absorbed dose in other materials irradiated under equivalent conditions may be calculated from the absorbed dose measurement of a ceric-cerous dosimeter. Procedures for making such calculations are given in Practices E 666 and E 668 and Guide E 1261.

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 **(3)**. For high-accuracy results, organic materials shall not be used for any component in contact with the solution. The effect of trace impurities is minimized by the addition of cerous ions to the solution **(4, 5)**.

5.2 Undesirable chemical changes in the dosimetric solution can occur if care is not taken during flame-sealing of the ampoules (see 8.4).

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 2 with an uncertainty of no more than ±1 % in the region from 254 to

320 nm. Use matched quartz cuvettes (for dual-beam instruments) with 10-mm path length for spectrophotometric measurements of absorbance of the solution.

6.2 *Potentiometric Method*—Use an electrochemical cell, similar to that in Appendix X1 (see Fig. X1.1). Measure the electropotential across the cell with a high-precision potentiometer, preferably digital, that is capable of measuring d-c potentials in the range from 1 to 100 mV within an uncertainty of ±1 %.

NOTE 2—The electrochemical cell has two compartments separated by a glass frit. The inner compartment is filled with unirradiated solution. The lower compartment is filled with solution transferred from the irradiated ampoule. The potential difference, ΔE, generated between the platinum electrodes in the two compartments is measured by a digital potentiometer or multimeter.

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 solution or an equivalent cleaning agent. Rinse at least three times with double-distilled water (see Practice C 912). Dry thoroughly and store in a dust-free environment.

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

NOTE 3—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 (or better) chemicals shall be used for preparing all solutions.[10]

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

NOTE 4—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 Dosimetric Solution

8.1 The recommended concentrations for the ceric-cerous dosimeter to measure absorbed doses from about 5 to 50 kGy (high-range dosimeter) are 0.015-*M* ceric sulfate and 0.015-*M* cerous sulfate. For measurement of absorbed doses from about 0.5 to 10 kGy (low-range dosimeter), the recommended concentrations are 0.003-*M* ceric sulfate and 0.003-*M* cerous sulfate.

8.2 The dosimeters specified in 8.1 may be formulated from the following nominal stock solutions: (*a*) 0.4-*M* and 4-*M* sulfuric acid (H_2SO_4), (*b*) 0.1-*M* ceric sulfate [$Ce(SO_4)_2 \cdot 4H_2O$], and (*c*) 0.1-*M* cerous sulfate [$Ce_2(SO_4)_3 \cdot 8H_2O$]. Procedures for preparing these solutions are given in Appendix X1.

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

$$\frac{V_1}{1000} = \frac{0.015}{M_1} \quad (1)$$

$$\frac{V_2}{1000} = \frac{0.015}{M_2} \quad (2)$$

$$\frac{V_3}{1000 - V_1} = \frac{0.4}{M_3} \quad (3)$$

$$V_4 = 1000 - V_1 - V_2 - V_3 \quad (4)$$

where:

V_1 = volume of nominal 0.1-*M* ceric-sulfate stock solution,
V_2 = volume of nominal 0.1-*M* cerous-sulfate stock solution,
V_3 = volume of nominal 4-*M* sulfuric-acid stock solution,
V_4 = volume of distilled water,
M_1 = actual molarity of the ceric-sulfate stock solution,
M_2 = actual molarity of the cerous-sulfate stock solution, and
M_3 = actual molarity of the nominal 4-*M* sulfuric-acid stock solution.

NOTE 5—If the nominal molarities of $M_1 = M_2 = 0.1$, and $M_3 = 4$ are assumed, then $V_1 = V_2 = 150$ mL and $V_3 = 85$ mL. If the molarities of the various stock solutions are significantly different from the nominal values, then use Eqs 1, 2, and 3 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.

9. Spectrophotometer Calibration

9.1 Check the wavelength scale of the spectrophotometer and establish its accuracy. The emission spectrum from a low-pressure mercury arc lamp can be used for this purpose. Such lamps may be obtained from the spectrophotometer manufacturer or other scientific laboratory instrument suppliers. Another appropriate wavelength standard is a holmium-oxide solution sealed in a non-fluorescent fused-silica cuvette. Other wavelength standards also may be appropriate for this purpose. For more details, see Practice E 275.

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

NOTE 6—Holmium-oxide solutions in sealed cuvettes are available as certified wavelength standards for use in the ultraviolet region from the National Institute of Standards and Technology (NIST) as SRM 2034 (7).

9.2 Check the accuracy of the photometric (absorbance) scale of the spectrophotometer in the ultraviolet region. Ultraviolet absorbance standard filters are available for this purpose.

NOTE 7—Examples of absorbance standards available are solutions of potassium dichromate in perchloric acid and mixtures of liquid inorganic compounds. These standards are available from NIST as SRM 935 and SRM 931, respectively (8, 9). Another appropriate absorbance standard consists of a set of metal-on-quartz filters available from NIST as SRM 2031 (10, 11).

9.3 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-*M* nominal in 0.4-*M* H_2SO_4) as described in X2.3 may be used for this measurement. The plot of measured absorbance, *A*, per unit path length versus molar concentration shall be linear. The slope of the line gives, ϵ, the molar linear absorption coefficient.

NOTE 8—A reference value for ϵ is 561 $m^2 \cdot mol^{-1}$ ± 0.4 % at 320 nm (6).

10. Calibration of the Dosimetric Solution

10.1 After the aging period specified in 8.5, quantify the following dosimetric solution parameters: ceric-ion concentration, cerous-ion concentration, acid molarity, ceric-ion molar linear absorption coefficient, radiation chemical yield for the cerous ion, and the density of the dosimetric solution.

10.2 *Ceric-Ion Concentration:*

10.2.1 Use a "to contain" pipette to deliver 0.25 mL of dosimetric solution into a clean, dry 25-mL volumetric flask.

10.2.2 Rinse the pipette with 0.4-*M* sulfuric acid into the flask and make up to volume with 0.4-*M* sulfuric acid.

10.2.3 Stopper the 25-mL flask and mix well.

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

10.2.5 Read the absorbance, *A*, in the spectrophotometer at 320 nm using 0.4-*M* sulfuric acid in the reference cell.

10.2.6 Determine the ceric concentration, M_4, using the following equation:

$$M_4 = (0.01782)A \quad (5)$$

where:

M_4 = ceric-sulfate molarity of the dosimetric solution, $mol \cdot L^{-1}$, and

A = absorbance.

10.3 *Cerous-Ion Concentration:*

10.3.1 Irradiate three dosimeters to an absorbed dose sufficient to completely reduce all ceric ions to cerous ions (for example, 80 kGy for high-range dosimeter).

10.3.2 Pipette 0.25 mL of the irradiated dosimetric solution from each ampoule into separate, clean, dry 25-mL volumetric flasks.

10.3.3 Rinse the pipette with 0.4-*M* H_2SO_4 into the flask and make up to volume with 0.4-*M* H_2SO_4. Stopper flasks and mix well.

10.3.4 Transfer an appropriate amount into a quartz spectrophotometer cuvette.

10.3.5 Read the absorbance, *A*, in the spectrophotometer at 254 nm using 0.4-*M* H_2SO_4 in the reference cell.

10.3.6 Determine the average absorbance, $\bar{A}$.

10.3.7 The following equation gives the resultant total cerous-ion concentration:

$$M_5 = 0.146\,\bar{A} \quad (6)$$

where:

M_5 = cerous-sulfate molarity of the dosimetric solution, $mol \cdot L^{-1}$.

NOTE 9—The total cerous-ion concentration determined for each sample will be slightly greater than the sum of the ceric and cerous ions in the dosimetric solution. The difference is due to the presence of cerous ions in the ceric-sulfate reagents. The cerous-ion concentration is determined by subtracting the ceric-ion concentration from the total cerous-ion concentration.

10.4 *Acid Molarity of the Dosimetric Solution*—Determine the acid molarity of the dosimetric solution using the potentiometric method.

NOTE 10—The molarity should be equal to 0.4 ± 0.01 *M*.

10.5 *Ceric-Ion Molar Linear Absorption Coefficient:*

10.5.1 Pipette 2 mL of the dosimetric solution into a 25-mL volumetric flask to which 0.4-*M* sulfuric acid is added to make up to the volume.

10.5.2 Similarly, pipette 2, 3, 4, 5, and 6 mL of the resultant solution of 10.5.1 respectively into separate 100-mL flasks to which 0.4-*M* sulfuric acid is added to make up to the volume.

10.5.3 Stopper each flask and mix well.

10.5.4 Read the absorbance, *A*, of each sample at 320 nm in the spectrophotometer with 0.4-*M* sulfuric acid in the reference cell.

10.5.5 Using the molarity obtained in 10.2.6, the dilution factors associated with the samples prepared in 10.5.2, and the absorbances determined in 10.5.4, determine the molar linear absorbance coefficient, ϵ, by the slope of the plot of absorbance versus molarity as expressed by the following equation:

$$A_i = 10^3\,\epsilon M_i d \quad (7)$$

where:

A_i = absorbance of sample i,

M_i = molarity of sample i, $mol \cdot L^{-1}$,

d = path length of spectrophotometer sample cell, m, and

i = 1, 2, . . ., 5 (representing samples prepared in 10.5.2).

NOTE 11—The value of ϵ should be equal to 561 $m^2 \cdot mol^{-1}$ ± 0.4 % or 5610 $L \cdot mol^{-1} \cdot cm^{-1}$ ± 0.4 %, at 320 nm (4).

10.6 *Density of Dosimetric Solution*—Determine the density of the dosimetric solution at 25°C using the pycnometer method. (See Test Method D 941.)

NOTE 12—The density should be 1.032 (±0.002) × 10^3 $kg \cdot m^{-3}$ at 25°C (4).

10.7 *Radiation Chemical Yield of Cerous Ion, G(Ce^{3+}), by the Spectrophotometric Method:*

10.7.1 Prepare 33 dosimeters by filling 2-mL ampoules with the dosimetric solution. After filling, flame seal the ampoules.

10.7.2 Irradiate 30 dosimeters, three at a time, at a calibrated position in a cobalt-60 facility to the following

absorbed dose levels: 5, 10, 15, 20, 25, 30, 35, 40, 45, and 50 kGy. Control the temperature of the dosimeter during irradiation to within 26 ± 4°C.

10.7.3 Prepare diluted (by a factor of 100) samples of dosimeters, including the three unirradiated ones. Pipette 0.25 mL from each dosimeter into 25-mL volumetric flasks, and make up to volume with 0.4-*M* sulfuric acid.

10.7.4 Using 0.4-*M* sulfuric acid in the reference cell, read the absorbance, A, of the diluted samples prepared in accordance with 10.7.3 at 320 nm in the spectrophotometer.

10.7.5 Determine the change in absorbance, ΔA, of each irradiated sample as follows:

$$\Delta A_{ij} = A_0 - A_{ij} \tag{8}$$

where:

A_0 = average absorbance of the diluted samples of the unirradiated dosimeters,
i = 1, 2, and 3 (number identifying dosimeters at each absorbed dose level), and
j = 1, 2, . . ., 10 (number identifying absorbed dose levels).

10.7.6 Plot ΔA versus D according to the following equation:

$$\Delta A_{ij} = \beta \cdot D_j \tag{9}$$

where:

ΔA = change in absorbance of irradiated sample,
i = 1, 2, and 3 (number identifying dosimeters at each absorbed dose level),
j = 1, 2, . . ., 10 (number identifying absorbed dose levels),
D = absorbed dose, Gy, and
β = slope, $kg \cdot J^{-1}$, of the plot determined by a least-squares linear regression fit of the data.

10.7.7 Determine $G(Ce^{3+})$, $mol \cdot J^{-1}$, from the following equation:

$$G(Ce^{3+}) = \frac{10^2 \beta}{\epsilon \rho d} \tag{10}$$

where:

ϵ = molar-linear absorption coefficient, $(m^2 \cdot mol^{-1})$,
β = slope, $kg \cdot J^{-1}$, of the plot determined by a least-squares linear regression fit of the data,
ρ = density, $kg \cdot m^{-3}$,
d = path length of spectrophotometer cell, m, and
10^2 = dilution factor for the samples of irradiated dosimeters.

10.8 *Radiation Chemical Yield of the Cerous Ion, $G(Ce^{3+})$, by the Potentiometric Method:*

10.8.1 Prepare dosimeters and irradiate them in accordance with 10.7.1 and 10.7.2.

10.8.2 Place contents of an unirradiated dosimeter (ampoule) into both compartments of the electrochemical cell. See Appendix X1 for a description of the electrochemical cell.

10.8.3 Allow the unirradiated dosimetric solution to remain in the electrochemical cell for about one-half hour in order to establish equilibrium across the glass frit.

10.8.4 Drain the cell and refill it with the contents of another unirradiated dosimeter.

10.8.5 Connect the digital potentiometer across the cell. If the difference in potential is equal to zero within ±0.2 mV, the cell is ready for use.

10.8.6 Expel the unirradiated solution from the lower cell 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 potential difference for any particular dosimeter, rinse the cell with that dosimeter's solution in order to reduce the effects of the previous dosimeter.

10.8.7 Read the potential, ΔE, in millivolts, across the cell for each dosimeter after temperature equilibrium is established within the cell.

NOTE 13—The potential difference, ΔE, within the electrochemical cell, has a positive temperature coefficient of 0.33 % per °C between 25 and 30°C (4). For the best accuracy, normalize measurements to a constant temperature, thereby accounting for this effect.

10.8.8 Average the ΔE values for the three dosimeters irradiated to the same absorbed dose level. Use the average values to calculate $G(Ce^{3+})$ according to the following equation:

$$G(Ce^{3+}) = \frac{10^3}{\rho D_j}\left[M_4 - \left(\frac{M_4 + M_5}{1 + \frac{M_5}{M_4}\,\text{antilog}_{10}\frac{\Delta E_j}{59.16}}\right)\right] \tag{11}$$

where:

M_4 and M_5 = molarities of ceric and cerous ions in the unirradiated dosimetric solution, respectively, $mol \cdot L^{-1}$,
D = absorbed dose, Gy,
ρ = density of the dosimetric solution, $kg \cdot m^{-3}$, and
j = 1, 2, . . ., 10 (number identifying absorbed dose levels).

10.8.9 Calculate the average $G(Ce^{3+})$ from the following equation:

$$G(Ce^{3+}) = \frac{1}{10}\sum_{j=1}^{10} G(Ce^{3+})_j \tag{12}$$

10.8.10 Plot D_j versus $G(Ce^{3+})_j$. The results should be linear with a slope equal to zero. If a significant deviation from zero slope is observed, that is, an apparent dependence of $G(Ce^{3+})$ on absorbed dose, repeat the procedure in 10.8.

NOTE 14—The values of $G(Ce^{3+})$ determined in accordance with 10.7.7 and 10.8.9 should be equal approximately to 2.31×10^{-7} $mol \cdot J^{-1}$ (±1 %) at 25°C for the molarities of ceric and cerous ions specified for the dosimetric solution in this test method. The term in the square bracket in Eq 11 should be equal approximately to 2.38×10^{-3} $mol \cdot L^{-1}$ for an absorbed dose of 10^4 Gy.

11. Dosimetric Procedure

11.1 *Preparation of Dosimeters:*

11.1.1 Meet all specifications of Section 10 before preparing individual dosimeters.

11.1.2 Prepare individual dosimeters by filling 2-mL ampoules up to the constriction in the ampoule's neck. Take care not to contaminate the dosimetric solution with impurities. Flame seal the ampoules.

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

11.1.4 Check the ceric-ion concentration and $G(Ce^{3+})$ value of at least one of the stored dosimeters once per month to ensure the stored dosimeters are within the specified limits

given in 10.2 and 10.7 or 10.8.

11.2 *Irradiation of Dosimeters:*

11.2.1 Before irradiation, enclose each individual dosimeter in a material of thickness sufficient to provide electron equilibrium as specified in 6.4.

11.2.2 Record relevant irradiation conditions, including at least time, position, and temperature.

11.3 *Readout of Dosimeters:*

11.3.1 For spectrophotometric readings, dilute each irradiated dosimeter and three unirradiated dosimeters, that serve as blanks, by 100 (see 10.2). Measure the diluted dosimeters in a spectrophotometer at 320 nm while using 0.4-*M* H_2SO_4 in the reference cell. Determine ΔA for each irradiated dosimeter; that is, the difference between the blank (average of the three) and irradiated absorbance values.

11.3.2 For potentiometric readings, measure ΔE directly for each irradiated dosimeter according to the procedures given in 10.8.2 through 10.8.7.

12. Calculation

12.1 For spectrophotometric readings, calculate the absorbed dose D_s in grays, using the following equation (see Practice 1026):

$$D_s = \frac{10^2 \Delta A}{G(Ce^{3+})\,\epsilon \cdot \rho \cdot d} \qquad (13)$$

where:

ΔA = change in absorbance,

$G(Ce^{3+})$ = radiochemical yield of the cerous ion, $mol \cdot J^{-1}$,

ϵ = molar-linear absorption coefficient for the cerous ion, $m^2 \cdot mol^{-1}$,

ρ = density of the dosimetric solution, $kg \cdot m^{-3}$, and

d = pathlength of the dosimetric solution within the spectrophotometer cell, m.

12.2 For potentiometric readings, calculate the absorbed dose D_p in grays, using the following equation:

$$D_p = \frac{10^3}{\rho \cdot G(Ce^{3+})}\left[M_4 - \left(\frac{M_4 + M_5}{1 + \frac{M_5}{M_4}\,\text{antilog}_{10}\frac{\Delta E}{59.16}}\right)\right] \qquad (14)$$

where:

M_4 and M_5 = molarities of the ceric and cerous ions in the unirradiated dosimetric solutions, respectively, $mol \cdot L^{-1}$,

$G(Ce^{3+})$ = radiochemical yield for the cerous ion, $mol \cdot J^{-1}$,

ρ = density of dosimetric solution, $kg \cdot m^{-3}$, and

ΔE = difference in potential across the electrochemical cell for an unirradiated compared to an irradiated dosimetric solution, mV.

12.3 The value of $G(Ce^{3+})$ varies with irradiation temperature (**12**); therefore, use the following equation to obtain the $G(Ce^{3+})$ value for determining absorbed dose by Eqs 13 and 14:

$$G(Ce^{3+}) = [2.55 - 0.87\,(\bar{M}_5)^{1/3} - 0.0052T]1.036 \times 10^{-7} \qquad (15)$$

where:

$\bar{M}_5$ = mean molarity of the cerous ion in solution during irradiation, $mol \cdot L^{-1}$, and

T = irradiation temperature, °C (within the range from 0 to 62°C).

13. Precision and Bias

13.1 In applying this practice, the random uncertainties and the systematic uncertainties should be estimated to the extent possible. The overall uncertainty in absorbed dose should be obtained from a combination of these uncertainties, and the procedure for combining these uncertainties should be stated explicitly in all results.

13.2 If this practice is carefully applied, the combined uncertainty at the 95 % confidence level should be no more than ±4 % for an absorbed dose value measured in the range of application.

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 rays

E 1205

APPENDIXES

(Nonmandatory Information)

X1. THE ELECTROCHEMICAL CELL

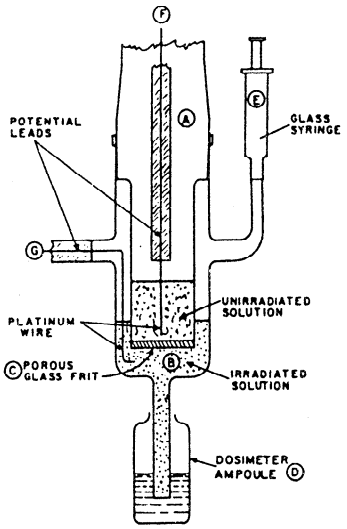

FIG. X1.1 Electrochemical Cell

X1.1 The electrochemical cell shown in Fig. X1.1 has two compartments, *A* and *B*, separated by a glass frit, *C*.

X1.2 Compartment *A* contains the unirradiated dosimetric solution.

X1.3 Compartment *B* contains either unirradiated or irradiated dosimetric solution.

X1.4 The glass frit *C* provides contact between the two solutions and shall have a porosity of less than 2 μm.

X1.5 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.

X1.6 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.

X2. A PROCEDURE FOR PREPARING NOMINAL STOCK SOLUTIONS FOR THE DOSIMETER

X2.1 *Nominal 0.4-M Sulfuric Acid (H_2SO_4):*

X2.1.1 Transfer 22.2 mL of 18-*M* sulfuric acid into a clean, dry 1-L volumetric flask containing about 700 mL of double-distilled water.

X2.1.2 Carefully cool contents and make up to volume with double-distilled water, stopper, and mix well.

X2.1.3 Standardize resulting solution using sodium carbonate primary standard or equivalent.

NOTE X2.1—Add sulfuric acid cautiously to the water as a considerable amount of heat is released.

X2.2 *Nominal 4-M Sulfuric Acid (H_2SO_4):*

X2.2.1 Transfer 222 mL of 18-*M* sulfuric acid into a clean, dry 1-L volumetric flask containing about 700 mL of double-distilled water.

X2.2.2 Carefully cool contents and make up to volume with double-distilled water, stopper, and mix well.

X2.2.3 Standardize resulting solution similarly as for 0.4-*M* sulfuric acid.

X2.3 *Nominal 0.1-M Ceric Sulfate:*

X2.3.1 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-*M* sulfuric acid.

X2.3.2 Shake contents until all ceric sulfate is dissolved. Allow solution to stand at least two weeks in the dark.

X2.3.3 Add enough 0.4-*M* sulfuric acid solution to dilute to 1 L. Filter if necessary through a clean sintered-glass filter (medium porosity).

X2.3.4 Transfer into a glass bottle provided with a ground glass stopper, and store in a dark place.

X2.3.5 Standardize 0.1-*M* ceric sulfate using arsenious oxide, primary standard, osmium-tetroxide catalyst, and feroin-indicator solution.

X2.4 *Nominal 0.1-M Cerous Sulfate:*

X2.4.1 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.

X2.4.2 Shake contents until all cerous sulfate is dissolved. Allow solution to stand at least two weeks in the dark.

X2.4.3 Add enough double-distilled water to dilute to 1 L. Filter if necessary through a clean sintered-glass filter (medium porosity).

X2.4.4 Transfer into a glass bottle provided with a ground glass stopper, mix well, and store in a dark place.

NOTE X2.2—Cerous sulfate may require recrystallization before use (13).

X2.5 *Molarity of Stock Cerous-Sulfate Solutions:*

X2.5.1 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 double-distilled water. Make up to volume with double-distilled water, stopper, and mix well.

X2.5.2 Read the absorbance, *A*, at 254 nm in a spectrophotometer with double-distilled water in the reference cuvette. The molarity, M_2, of the stock cerous sulfate solution is given, in $mol \cdot L^{-1}$, by the following equation:

$$M_2 = (0.146)A$$

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