# INTERNATIONAL STANDARD

ISO 15563

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# Practice for use of the ethanol-chlorobenzene dosimetry system

Pratique de l'utilisation d'un système dosimétrique à l'éthanol chlorobenzène

## iTeh STANDARD PREVIEW (standards.iteh.ai)

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### Foreword

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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 15563 was prepared by the American Society for Testing and Materials (ASTM) Subcommittee E10.01 (as E 1538-93) 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 15563 is one of 20 standards developed and published by ASTM. The 20 fast-tracked standards and their associated ASTM designations are listed below: 618e-d155-4d8c-a317-

ISO Designation	ASTM Designation	12fb0ct8cc/iso-15563-1998 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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#### ISO 15563:1998(E)

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	

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AMERICAN SOCIETY FOR TESTING AND MATERIALS 1916 Race St. Philadelphia, Pa 19103 Reprinted from the Annual Book of ASTM Standards. Copyright ASTM If not listed in the current combined index, will appear in the next edition.

## Standard Practice for Use of the Ethanol-Chlorobenzene Dosimetry System<sup>1</sup>

This standard is issued under the fixed designation E 1538; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

#### 1. Scope

1.1 This practice covers the preparation, handling, testing, and procedure for using the ethanol-chlorobenzene dosimetry system to measure absorbed dose in materials irradiated by photons and electrons in terms of absorbed dose in water. The system consists of a dosimeter and appropriate analytical instrumentation. For simplicity, the system will be referred to as the ECB system. It is classified as a reference standard dosimeter (see Guide E 1261).

1.2 This practice describes the titration analysis as a standard readout procedure for the ECB dosimeter. Other applicable readout methods (spectrophotometric, oscillometric) are described in Appendixes X1 and X2.

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

1.4 This practice applies provided the following are satisfied:

1.4.1 The absorbed dose range shall be from 10 Gy to 2 MGy (1).<sup>2</sup>

1.4.2 The absorbed dose rate does not exceed  $10^6$  Gy s<sup>-1</sup> (2).

1.4.3 For radionuclide gamma-ray sources, the initial photon energy shall be greater than 0.6 MeV<sub>10</sub> 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 4 MeV (see ICRU Reports 34 and 35).

NOTE 1—The lower limits of electromagnetic radiation energy given are appropriate for a cylindrical dosimeter ampoule of 12-mm diameter. Corrections for dose gradients across an ampoule of that diameter or less are not required. The ECB system may be used at energies of incident electrons lower than 8 MeV by employing thinner (in the beam direction) dosimeter containers (see ICRU Report 35).

1.4.4 The irradiation temperature of the dosimeter should be within the range from  $-40^{\circ}$ C to  $80^{\circ}$ 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<sup>3</sup>
- D1193 Specification for Reagent Water<sup>4</sup>
- E 170 Terminology Relating to Radiation Measurements and Dosimetry<sup>5</sup>
- E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers<sup>6</sup>
- E 666 Practice for Calculating Absorbed Dose from Gamma or X-Radiation<sup>5</sup>
- E 668 Practice for Application of Thermoluminescence Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices<sup>5</sup>
- E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers<sup>7</sup>
- E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers<sup>7</sup>
- E 1026 Practice for Using the Fricke Reference Standard Dosimetry System<sup>5</sup>

ISO 15563: E91205 Practice for Use of a Ceric-Cerous Sulfate

- iso-1:E:012619?Guide for the Selection and Application of Dosimetry Systems for Radiation Processing of Food<sup>5</sup>
  - E 1400 Practice for Characterization and Performance of a High-Dose Gamma-Radiation Dosimetry Calibration Laboratory<sup>5</sup>
  - E 1401 Practice for Use of a Dichromate Dosimetry System<sup>5</sup>
  - E 1431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing<sup>5</sup>

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

ICRU Report 14—Radiation Dosimetry: X-Rays and Gamma Rays with Maximum Photon Energies Between 0.6 and 60 MeV<sup>7</sup>

- ICRU Report 34—The Dosimetry of Pulsed Radiation<sup>7</sup>
- ICRU Report 35—Radiation Dosimetry: Electrons with Initial Energies Between 1 and 50 MeV<sup>7</sup>
- ICRU Report 37—Stopping Powers for Electrons and Positrons<sup>7</sup>

- <sup>5</sup> Annual Book of ASTM Standards, Vol 12.02.
- <sup>6</sup> Annual Book of ASTM Standards, Vol 14.01.

<sup>&</sup>lt;sup>1</sup> 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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<sup>&</sup>lt;sup>2</sup> The **boldface** numbers in parentheses refer to the list of references at the end of this practice.

ICRU Report 33—Radiation Quantities and Units<sup>7</sup>

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol 15.02.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vols 02.05, 06.03, 09.01, 10.01, 10.02, 10.05, 11.01, 11.03, and 15.09.

<sup>&</sup>lt;sup>7</sup> Available from the 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  $d\bar{e}$  by dm, where  $d\bar{e}$  is the mean energy imparted by ionizing radiation to the matter of mass dm (see ICRU Report 33).

#### $D = d\bar{e}/dm$

The special unit for absorbed dose is the gray (Gy).

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

3.1.2 *calibration curve*—the graphical or mathematical relationship between the net response and the absorbed dose for a given dosimetry system. This term is also referred to as the response function.

3.1.3 calibration facility—a 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.

3.1.4 conductivity—the conductivity of a solution is usually defined in terms of specific conductivity ( $\kappa$ ), which is given by the conductivity of a solution between electrodes of 1-cm<sup>2</sup> surface area, placed 1 cm from each other.

3.1.5 conductometry—analytical method based on the measurement of conductivity of solutions due to the relationship between concentration and conductivity of electrolytes. The conductivity of a solution depends on the concentration of free ions in the solution.

3.1.6 dosimetry system—the system used for measuring absorbed dose, consisting of dosimeters, measurement instrumentation, the calibration curve or specific values of molar linear absorption coefficient and G-value, reference standards, and procedures for the system's use.

3.1.7 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.

3.1.8 molar linear absorption coefficient (sometimes called molar extinction coefficient),  $\epsilon_m$ —a constant relating the spectrophotometric absorbance,  $A_{\lambda}$ , of an optically absorbing molecular species, x, at a given wavelength,  $\lambda$ , per unit pathlength, d, to the molar concentration, [x], of that species in its host substance:

$$\epsilon_m = \frac{A_\lambda}{d} \times \frac{1}{[x]}$$

(unit:  $L \mod^{-1} \cdot \operatorname{cm}^{-1}$  or  $M^{-1} \cdot \operatorname{cm}^{-1}$ ; SI unit:  $m^2 \cdot \operatorname{mol}^{-1}$ ) where:

 $A_{\lambda}$  = absorbance at a specified wavelength,

- [x] = molar concentration of the species of interest, and
- d = optical pathlength within the solution measured by the spectrophotometer,

3.1.9 oscillometry—an electroanalytical method of conductivity measurements, when high-frequency (1 to 600 MHz) alternating current is applied to measure or follow changes in the composition of chemical systems.

3.1.10 radiation chemical yield (sometimes called G-

value), G(x)—the quotient of the amount n(x), of a substance, usually a specified molecular species (as a solute in solution), x, produced, destroyed, or changed by radiation, by the mean energy  $\overline{e}$ , imparted to the irradiated matter (see ICRU Report 33):

 $G(x) = n(x)/\bar{e}$ 

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

DISCUSSION—The former special unit was  $(100 \text{ eV})^{-1}$ . This quantity is related to the old unit as  $1 \text{ mol} \cdot J^{-1} = 9.65 \ 10^6 \text{ mol} \ (100 \text{ eV})^{-1} \text{ or } 1 \text{ mol} \ (100 \text{ eV})^{-1} = 1.036 \ 10^{-7} \text{ mol} \cdot J^{-1}$ .

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

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

3.2 For other terms, see Terminology E 170.

#### 4. Significance and Use

4.1 The ECB dosimetry system provides a reliable means of measuring absorbed dose in materials. It is based on a process of radiolytic formation of hydrochloric acid (HCl) in aqueous ethanolic solutions of chlorobenzene by ionizing radiation (3).

4.2 The dosimeters are partly deoxygenated solutions of chlorobenzene (CB) in 96 volume % ethanol in an appropriate container, such as a flame-sealed glass ampoule. The solutions indicate absorbed dose by the amount of HCl formed. A number of analytical methods are available for measuring the amount of HCl in ethanol (4).

4.3 The concentration of chlorobenzene in the solution can be varied so as to simulate a number of materials in terms of the photon mass energy-absorption coefficients  $(\mu_{en}/\rho)$  for X and gamma rays, and electron mass collision stopping powers  $(1/\rho)$  (dE/dx), over a broad spectral energy range from  $10^{-2}$  to 100 MeV (5-7).

4.4 The absorbed dose that is measured is the dose absorbed in the dosimeter. Absorbed dose in other materials irradiated under equivalent conditions may be calculated. Procedures for making such calculations are given in Practices E 666 and E 668 and Guide E 1261.

4.5 There are two factors associated with the use of the ECB system at energies below those specified in 1.4.3:

4.5.1 The radiation chemical yield may change at low-photon energies.

4.5.2 Dose gradients across the dosimeter may require corrections in dosimeter response at energies below 8 MeV for electrons.

NOTE 2—For a comprehensive discussion of various dosimetry methods applicable to the radiation types and energies discussed in this practice, see ICRU Reports 14, 17, 34, 35, and 37.

#### 5. Interferences

5.1 The ECB dosimetric solution response is not particularly sensitive to impurities which occur in commercially available components, chlorobenzene and ethanol of the analytical reagent (AR) grade purity or equivalent (pro analysi, p.a., and puriss.). For high-accuracy results, organic © ISO

materials of technical grade purity (or purum) can be purified by distillation.

5.2 Care should be exercised in filling ampoules to avoid depositing solution in the ampoule neck. Subsequent heating during sealing of the ampoule may cause an undesirable chemical change in the dosimetric solution remaining inside the ampoule's neck. Test tubes with ground-glass stoppers are therefore preferred to sealed ampoules for measuring doses below 100 Gy. For the same reason, care should be given to avoid heating the body of the ampoule during sealing.

5.3 The dosimetric solution is somewhat sensitive to ultraviolet light and should be kept in the dark for long-term storage. No special precautions are required during routine handling under normal laboratory lighting conditions, but strong ultraviolet (UV) sources such as sunlight should be avoided (8).

#### 6. Apparatus

6.1 This practice describes mercurimetric titration of radiolytically formed Cl<sup>-</sup> ions as a standard readout procedure.

6.2 For the analysis of the dosimetric solution, use a precision burette capable of measuring volumes with 0.01mL resolution. If necessary, check the original calibration of volumetric glassware and, if necessary, recalibrate to attain 0.1 % relative error. Control the temperature of all solutions during handling at 20°C.

6.3 Use borosilicate glass or equivalent chemically resistant glass to store the reagents, the prepared dosimeter solution, and to perform the titration. Clean all apparatus

6.4 Use a sealed glass ampoule or other appropriate glass /iso-with the indicator diphenylcarbazone in acid medium (11). container to hold the dosimetric solution during irradiation. For photons, surround the container with material of thickness sufficient to produce approximate electron equilibrium conditions during calibration irradiations. For measurement of absorbed dose in water, use materials that have radiationabsorption properties essentially equivalent to water, for example, polystyrene and polyethylene. The appropriate thickness of such material depends on the energy of the photon radiation (see Practices E 666 and E 668).

NOTE 3-The dosimetric ampoule commonly used has a capacity of about 5 mL.

#### 7. Reagents

7.1 Analytical reagent grade chemicals shall be used in this practice for preparing all solutions.<sup>8</sup>

7.2 Use of triply distilled water from coupled all-glass stills is recommended. Type II reagent water as specified in Specification D 1193 is also considered to be of sufficient quality for use in preparing solutions and 96 volume % ethanol.

NOTE 4-High-purity water is commercially available from some suppliers. Such water, labelled HPLC (high-pressure liquid chromatography) grade, is usually sufficiently free of impurities to be used in this practice.

#### 8. Preparation of Dosimeters

8.1 Dosimeter solutions may contain any concentration of CB. For practical reasons, only several characteristic formulations have been thoroughly characterized. Table 1 lists these typical formulations in terms of CB concentrations and radiation chemical yields pertaining to these concentrations.

8.2 Prepare 96 volume % aqueous ethanol first by adding absolute ethanol into a volumetric flask containing the appropriate amount of water. Use this aqueous ethanol for making the dosimeter solutions of the desired concentrations by adding it into volumetric flasks containing appropriate amounts of CB. Store the dosimeter solution in the dark.

NOTE 5: Caution-Chlorobenzene is toxic and a skin irritant. Appropriate precautions should be exercised in handling it.

8.3 Fill the dosimeter ampoules with the dosimeter solution. Bubble the solution in the ampoule with nitrogen for about 1 min at about 1 bubble per second through a 1-mm capillary. Flame-seal immediately after bubbling. Exercise care to avoid depositing solution in the ampoule neck. Store dosimeters in the dark.

NOTE 6-Nitrogen may be saturated by passing it through the ECB solution of the same composition before bubbling the dosimeter ampoules to avoid changing the composition of dosimeter solution by evaporation E VIEW

#### 9. Calibration of the Mercuric Nitrate Solution

9.1 The measurement procedure is based on the titration of chloride ions formed by irradiation. Free chloride is precipitated with mercuric ions as insoluble HgCl<sub>2</sub>, wherethoroughly before use (see Practice /Ct912).ds.iteh.ai/catalog/standardupon/affle excess of Hg2+aions gives a violet-red coloration

> 9.2 Prepare approximately  $5 \times 10^{-4} \text{ mol} \cdot \text{dm}^{-3} \text{ Hg}(\text{NO}_3)_2$ in acidic aqueous ethanol. First dissolve an appropriate amount of  $Hg(NO_3)_2$  in water acidified with sufficient HNO<sub>3</sub> to attain the concentration of the acid in the final solution,  $0.05 \text{ mol} \cdot \text{dm}^{-3}$ .

> NOTE 7: Caution-Mercuric (II) nitrate is highly toxic. Acute exposure of skin and mucous membranes produces violent corrosive effects. Chronic exposure causes many pathological changes. Appropriate precautions should be exercised in handling it. Hazards of mercury poisoning can be avoided by using some of the alternative readout methods described in Appendixes X1 and X2.

TABLE 1 Typical ECB Solution Formulations

Concentration of CB, vol %	Density at 20°C kg ⋅m <sup>-3</sup>	Ratio of coefficients / <sup>A</sup>	Radiation Chemical Yields <sup>₿</sup> (µmol·J <sup>-1</sup> )	
			<sup>60</sup> Co gamma rays (9)	4 to 10 MeV electrons (10)
4	819	0.989	0.42°	
10	839	0.995	0.52	
20	869	1.006	0.59	
24	880	1.011	0.60	0.57 <i>0</i>
40	925	1.027	0.63	

A The ratio of the mass energy-absorption coefficients for water and the dosimeter solution at <sup>60</sup>Co gamma ray energy:

$$f = \frac{(\mu e n / \rho)_w}{(\mu e n / \rho)_0}$$

<sup>a</sup> Radiation chemical yields of HCI in the dose range from 100 Gy to 100 kGy. <sup>c</sup> Upper dose range 20 kGy.

<sup>D</sup> Lower dose range 1 kGy. This formulation also contained 0.04 volume % acetone and 0.04 volume % benzene.

<sup>&</sup>lt;sup>8</sup> Reagent specifications are available from the American Chemical Society, 1115 16th Street, NW, Washington, DC 20036.

9.2.1 Prepare standard solutions of NaCl in water. Make several concentrations to enable cross-checking. Suitable concentrations are  $5 \times 10^{-3}$ ,  $1.0 \times 10^{-2}$ ,  $1.5 \times 10^{-2}$ , and 2.0  $\times 10^{-2}$  mol·dm<sup>-3</sup>. If kept properly in ground-glass stoppered bottles, these solutions are stable for years. Avoid contamination of the standard solutions by using for daily work small portions of these solutions kept in small ground-glass stoppered flasks. Replenish standard solutions in the small flasks as necessary.

9.2.2 Prepare 0.2 mol·dm<sup>-3</sup> HNO<sub>3</sub> in ethanol and 1 % ethanolic solution of diphenylcarbazone (DPC).

9.3 Distribute technical grade ethanol to beakers for titration, 10 mL into each. Pipet standard NaCl solution quantitatively to beakers with ethanol. Add 1 mL of 0.2 M  $HNO_3$  and 7 drops of 1 % DPC and shake. Titrate with  $Hg(NO_3)_2$  solution from the burette. The solution in the beaker which is initially yellow-orange turns to reddish-violet at the end point.

9.4 Construct or calculate the best straight line through the points: (consumption of  $Hg(NO_3)_2$ ) versus (milliequivalents of NaCl). The small positive intercept represents the blank; inverse slope gives concentration of Hg(NO<sub>3</sub>)<sub>2</sub> solution.

NOTE 8-Volumes of the standard NaCl solutions should be such that the consumption of the titrant solution on calibration are similar to the consumptions when analyzing irradiated dosimetric solutions. Take two different volumes of each standard solution to enable crosschecking. The concentration of mercuric nitrate solution should be calibrated daily. standar

#### 10. Irradiation and Measurement Procedures

10.1 Calibration of the Dosimeter Response:

cility that has a dose rate traceable to national standards) and /iso-15/67 11/28. If high doses are to be measured, use appropriate portions that meets the requirements specified in Practices E 1400 and E 1431. Use a reference or transfer dosimetry system to establish this traceability (see Guide E 1261 and Practices E 1026, E 1205, and E 1401).

10.1.2 Specify the dose in terms of absorbed dose in water (for example, see Practices E 1026, E 1205, and E 1401).

10.1.3 Position the dosimeters in the radiation field in a defined, reproducible location (12).

10.1.4 When using a gamma-ray source for irradiation of very thin dosimeters (for example, in capillaries), surround the dosimeters with a sufficient amount of water-equivalent material to achieve approximate electron equilibrium conditions.

NOTE 9-The appropriate thickness of such material depends on the energy of the radiation (see Practices E 666 and E 668). For measurement of absorbed dose in water use materials that have radiationabsorption properties essentially equivalent to those of water. For example, for a <sup>60</sup>Co source, 3 to 5 mm of polystyrene (or equivalent polymeric material) should surround the dosimeter in all directions.

10.1.5 When using an electron beam for irradiation, locate the dosimeters in a well-characterized position within the radiation field (13). In addition, it may be necessary to surround the dosimeter(s) with water-equivalent material.

10.1.6 Make the irradiation field within the volume occupied by the dosimeter(s) as uniform as possible. The variation in dose rate within this volume should be within ±1%.

10.1.7 Control (or monitor) the temperature of the dosim-

eters during irradiation. Take into account any temperature variations beyond the 18 to 30°C range that affect dosimeter response. The temperature dependence of dosimeter response during irradiation between 20 and 80°C is found in Ref (14), and between -40 and  $20^{\circ}$ C in Ref (15).

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

10.1.9 Irradiate these sets of dosimeters to at least five known dose values covering the range of utilization in order to determine the radiation chemical yield of the dosimetry system.

NOTE 10-The observed dose can in principle be determined with the ethanol-chlorobenzene dosimeter without the necessity for calibration of the dosimeter response, if procedures outlined in Sections 5, 6, 7, 8, 9, and 10 are adhered to. However, it is prudent to assure that the dosimeter is behaving as expected. Calibration would also be necessary if a formulation of the dosimetric solution different from any one listed in Table 1 is used. This can be done by irradiating dosimeters in a known radiation field of a calibration facility. Use the procedures of 10.1 to perform the irradiations. Only a few dose levels are needed to check both the absolute response and the linearity of the dosimeter. Analyze the dosimeters and calculate the doses using the procedures of 10.2 and 10.3. Compare the results with the calibration doses. The results from the two methods should not differ by more than the overall uncertainty of the dosimeter response. If the difference is greater, there is an indication of possible contamination of the solution, or some other problem must be resolved.

10.2 Measurement - Transfer the irradiated dosimeter solution quantitatively into a beaker for titration. Rinse the dosimeter ampoule several times with 5 mL of technical grade ethanol, so that the final volume in the beaker is 10

mL. Add 1 mL of 0.2 M HNO<sub>3</sub> and 7 drops of DPC and fitrate to the same color change as that observed during 10.1.1 Calibrate the dosimeters /using an irradiation fandar calibration of the mercuric nitrate solution.

> of irradiated dosimeter solution, taking care that total volume in the beaker is 10 mL.

10.3 Analysis:

10.3.1 Subtract the blank from the amounts of  $Hg(NO_3)_2$ solution consumed and calculate the concentration of radiolytically formed Cl<sup>-</sup> ions in each dosimeter:

$$\begin{pmatrix} \text{equivalents} \\ \text{of } \text{Cl}^- \end{pmatrix} = \begin{pmatrix} \text{equivalents} \\ \text{of } \text{Hg}^{2+} \end{pmatrix} \times \frac{(\text{mL of titrant}) - (\text{blank})}{\text{mL of dosimetric solution}}$$
(1)

Calculate the absorbed dose in the dosimeter as follows:

$$D = \frac{[\text{Cl}^-]}{G(\text{Cl}^-)\rho} \tag{2}$$

The values of G(HCl) and  $\rho$  are found in Table 1 or in the literature (1, 5). Other values can be derived in accordance with 10.1.

10.3.2 To calculate the absorbed dose in water (ICRU Report 14) use the following:

$$D_w = f \cdot D \tag{3}$$

where f is the ratio of photon mass energy-absorption coefficients for water and that for the dosimeter. The values of factor f at the reference energy, 1.25 MeV, are found in Table 1.

#### **11. Minimum Documentation Requirements**

11.1 Calibration of the Dosimeter Response (if Performed):

5

## ∰ E 1538

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

11.1.2 Record or reference the date, irradiation temperature, temperature variation (if any), dose range, radiation source (including dose rate and energy), and associated instrumentation used to calibrate and analyze the dosimeters.

11.2 Application:

11.2.1 Record the date and temperature of irradiation, temperature variation (if any), and the date and temperature of absorbance measurement, for each dosimeter.

11.2.2 Record or reference the radiation source type and characteristics.

11.2.3 Record the consumption of the titrant, net consumption value, temperature correction (if applicable), and resulting absorbed dose for each dosimeter. Reference the calibration curve (if appropriate) or the calculations used to obtain the absorbed-dose values.

11.2.4 Record or reference the precision and bias in the value of the absorbed dose.

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

#### 12. Precision and Bias

shall be accompanied by an estimate of the uncertainty in the measured value. Factors contributing to the total uncertainty may be separated into two types, precision (random) and bias (systematic). Guide E 1261 discusses sources of uncertainty and lists some of the possible factors that affect precision and bias. Additional information is given in Practice E 668 and Refs (16) and (17).

12.2 The random and systematic uncertainties involved in measuring absorbed dose using this dosimetry system should be estimated or determined. The overall uncertainty in absorbed dose should be estimated from a combination of these uncertainties, and the procedure for combining these uncertainties should be specifically stated or referenced for all results.

12.3 If care is taken in carrying out this practice, the combined uncertainty of an absorbed dose evaluated by the dosimetry system should be within  $\pm 3\%$  at a 95% confidence level. This combined uncertainty is based on an uncertainty of  $\pm 2\%$  at a 95% confidence level for the dose rate of the calibration source.

#### 13. Keywords

13.1 absorbed dose; dose measurement; dosimeter; dosimetry system; ECB; electron beam; ethanol-chlorobenzene dosimeter; gamma radiation; ionizing radiation; irradiation; photons; radiation; radiation processing; referasurement of absorbed dose

# 12.1 To be meaningful, a measurement of absorbed dose ence standard do (standards.iteh.ai)

#### APPENDIXES

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#### X1. SPECTROPHOTOMETRIC READOUT

#### X1.1 Apparatus

X1.1.1 For the analysis of the dosimetric solution, use a high-precision spectrophotometer capable of measuring absorbance values up to 2 with an uncertainty of no more than  $\pm 1$  % in the region of 400 to 500 nm. Use quartz cuvettes with 10 to 100-mm pathlength for spectrophotometric measurements of the solution. The cuvette capacity must be small enough to allow it to be thoroughly rinsed by the solution of secondary complex and still leave an adequate amount of that solution to fill the cuvette to the appropriate level for the absorbance measurement. Control the temperature of the room during measurement at  $20 \pm 2^{\circ}C$ .

#### X1.2 Spectrophotometer Calibration

X1.2.1 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 filter or solutions. For more details, see Practices E 275, E 925, and E 958.

NOTE X1.1—For example, holmium-oxide solutions in sealed cuvettes are available as certified wavelength standards (SRM 2034) for

use in the wavelength region of 240 to 650 nm from the National Institute of Standards and Technology (NIST) (formerly the National Bureau of Standards).

X1.2.1 Check the accuracy of the photometric (absorbance) scale of the spectrophotometer. Certified absorbance standard filters or solutions are available for this purpose (18).

NOTE X1.2—Examples of absorbance standards are solutions of various concentrations such as SRM 931d and SRM 935 and metalon-quartz filters such as SRM 2031. These standards are available from the NIST.

#### **X1.3 Irradiation and Measurement Procedures**

X1.3.1 Irradiation—Separate five dosimeters from the remainder of the batch and do not irradiate them. Use them in determining  $A_0$ . Follow 10.1 for the remainder of the procedure.

X1.3.2 Development of the Colored Secondary Complex— Transfer the irradiated dosimeter solution quantitatively into a 25-mL volumetric flask. Add 1.5 mL of 5.25 M aqueous HClO<sub>4</sub>, 50 µL of 0.37 M aqueous Fe(NO<sub>3</sub>)<sub>3</sub>, and 0.5 mL of saturated alcoholic solution of Hg(SCN)<sub>2</sub>, in that order, followed by 96 volume % ethanol to the mark. The color develops for 5 min in the dark and is measured against the blank in a 5-cm spectrophotometric cell.