INTERNATIONAL
STANDARD

ISO
15563

First edition
1998-12-15

Practice for use of the ethanol-chlorobenzene dosimetry system

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

Reference number
ISO 15563:1998(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 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:

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*

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

Printed in Switzerland

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 1538 – 93

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[1]

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 (ϵ) 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)**.[2]

1.4.2 The absorbed dose rate does not exceed 10^6 Gy s^{-1} **(2)**.

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 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°C to 80°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 1193 Specification for Reagent Water[4]
E 170 Terminology Relating to Radiation Measurements and Dosimetry[5]
E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[6]
E 666 Practice for Calculating Absorbed Dose from Gamma or X-Radiation[5]
E 668 Practice for Application of Thermoluminescence Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices[5]
E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers[7]
E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers[7]
E 1026 Practice for Using the Fricke Reference Standard Dosimetry System[5]
E 1205 Practice for Use of a Ceric-Cerous Sulfate Dosimetry System[5]
E 1261 Guide for the Selection and Application of Dosimetry Systems for Radiation Processing of Food[5]
E 1400 Practice for Characterization and Performance of a High-Dose Gamma-Radiation Dosimetry Calibration Laboratory[5]
E 1401 Practice for Use of a Dichromate Dosimetry System[5]
E 1431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing[5]

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[7]
ICRU Report 33—Radiation Quantities and Units[7]
ICRU Report 34—The Dosimetry of Pulsed Radiation[7]
ICRU Report 35—Radiation Dosimetry: Electrons with Initial Energies Between 1 and 50 MeV[7]
ICRU Report 37—Stopping Powers for Electrons and Positrons[7]

---

[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.

Current edition approved April 15, 1993. Published June 1993.

[2] The boldface numbers in parentheses refer to the list of references at the end of this practice.

[3] *Annual Book of ASTM Standards*, Vol 15.02.

[4] *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.

[5] *Annual Book of ASTM Standards*, Vol 12.02.

[6] *Annual Book of ASTM Standards*, Vol 14.01.

[7] 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 $d\bar{\varepsilon}$ by dm, where $d\bar{\varepsilon}$ is the mean energy imparted by ionizing radiation to the matter of mass dm (see ICRU Report 33).

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

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 (κ), which is given by the conductivity of a solution between electrodes of 1-cm^2 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), ϵ_m—a constant relating the spectrophotometric absorbance, A_λ, of an optically absorbing molecular species, x, at a given wavelength, λ, 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 mol$^{-1}\cdot$cm^{-1} or M$^{-1}\cdot$cm^{-1}; SI unit: m$^2\cdot$mol^{-1})

where:

A_λ = 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 $\bar{\varepsilon}$, imparted to the irradiated matter (see ICRU Report 33):

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

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

Discussion—The former special unit was (100 eV)$^{-1}$. This quantity is related to the old unit as 1 mol$\cdot$J^{-1} = 9.65 10^6 mol (100 eV)$^{-1}$ or 1 mol (100 eV)$^{-1}$ = 1.036 10^{-7} 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 (μ_{en}/ρ) 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 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^- ions as a standard readout procedure.

6.2 For the analysis of the dosimetric solution, use a precision burette capable of measuring volumes with 0.01-mL 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 thoroughly before use (see Practice C 912).

6.4 Use a sealed glass ampoule or other appropriate glass 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 radiation-absorption 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.[8]

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.

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 $HgCl_2$, whereupon the excess of Hg^{2+} ions gives a violet-red coloration with the indicator diphenylcarbazone in acid medium (**11**).

9.2 Prepare approximately 5×10^{-4} mol·dm^{-3} $Hg(NO_3)_2$ in acidic aqueous ethanol. First dissolve an appropriate amount of $Hg(NO_3)_2$ in water acidified with sufficient HNO_3 to attain the concentration of the acid in the final solution, 0.05 mol·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^{-3}	Ratio of coefficients f^A	Radiation Chemical Yields^B (μmol·J^{-1})	
			^{60}Co gamma rays (**9**)	4 to 10 MeV electrons (**10**)
4	819	0.989	0.42^C	
10	839	0.995	0.52	
20	869	1.006	0.59	
24	880	1.011	0.60	0.57^D
40	925	1.027	0.63	

^{A} The ratio of the mass energy-absorption coefficients for water and the dosimeter solution at ^{60}Co gamma ray energy:

$$f = \frac{(\mu_{en}/\rho)_w}{(\mu_{en}/\rho)_D}$$

^{B} Radiation chemical yields of HCl in the dose range from 100 Gy to 100 kGy.

^{C} Upper dose range 20 kGy.

^{D} Lower dose range 1 kGy. This formulation also contained 0.04 volume % acetone and 0.04 volume % benzene.

---

[8] 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 5×10^{-3}, 1.0×10^{-2}, 1.5×10^{-2}, and 2.0×10^{-2} $mol \cdot dm^{-3}$. 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 $mol \cdot dm^{-3}$ HNO_3 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 $Hg(NO_3)_2$ 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 cross-checking. The concentration of mercuric nitrate solution should be calibrated daily.

10. Irradiation and Measurement Procedures

10.1 *Calibration of the Dosimeter Response:*

10.1.1 Calibrate the dosimeters using an irradiation facility that has a dose rate traceable to national standards and 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 radiation-absorption properties essentially equivalent to those of water. For example, for a ^{60}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 dosimeters 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°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_3 and 7 drops of DPC and titrate to the same color change as that observed during calibration of the mercuric nitrate solution.

NOTE 11—If high doses are to be measured, use appropriate portions 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^- ions in each dosimeter:

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

Calculate the absorbed dose in the dosimeter as follows:

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

The values of $G(HCl)$ and ρ 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:

$$D_w = f \cdot D \quad (3)$$

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):*

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

12.1 To be meaningful, a measurement of absorbed dose 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 ±3 % at a 95 % confidence level. This combined uncertainty is based on an uncertainty of ±2 % at a 95 % confidence level for the dose rate of the calibration source.

13. Keywords

13.1 absorbed dose; dose; dose measurement; dosimeter; dosimetry system; ECB; electron beam; ethanol-chlorobenzene dosimeter; gamma radiation; ionizing radiation; irradiation; photons; radiation; radiation processing; reference standard dosimeter; X rays

APPENDIXES

(Nonmandatory Information)

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 ±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 ± 2°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 metal-on-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 $HClO_4$, 50 µL of 0.37 *M* aqueous $Fe(NO_3)_3$, and 0.5 mL of saturated alcoholic solution of $Hg(SCN)_2$, 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.

NOTE X1.3—Choice of the cuvette pathlength depends on the maximum absorbance that can be measured accurately by the spectrophotometer and on the dose range and dosimeter formulation chosen for a given calibration.

X1.3.3 *Measurement:*

X1.3.3.1 Set the spectral bandwidth of the spectrophotometer at no more than 1 nm, and maintain the room temperature during measurement at 20 ± 2°C. Determine the exact wavelength of the absorbance peak of the solution by making a spectral scan of an irradiated sample. The peak wavelength is about 485 nm.

X1.3.3.2 Set the balance of the spectrophotometer to zero with air only (no cuvette) in the light path(s).

X1.3.3.3 Fill a clean cuvette of 5-cm pathlength with 96 volume % ethanol. Carefully wipe the cuvette exterior windows through which the light beam passes with a clean, lint-free tissue or cloth. Measure the absorbance with air only in the reference beam of the spectrophotometer. Record this value (A_∞).

X1.3.3.4 Empty the 96 volume % ethanol from the cuvette and rinse it at least once with the solution from a volumetric flask. Discard the rinse solution and fill the cuvette to the appropriate level with more solution from the same flask. Carefully wipe off any solution on the exterior surfaces of the cuvette as instructed in X1.3.3.3. Place the cuvette in the sample holder and measure the absorbance.

NOTE X1.4—Inadequate rinsing of the cuvette between dosimeter solutions can lead to errors due to solution carryover (cross-contamination). Techniques for minimizing this effect are discussed in Ref (**18**).

X1.3.3.5 Check the zero balance after each solution is measured with air only in the light beam(s). Periodically during the measurement process, remeasure the absorbance of 96 volume % ethanol, first rinsing the cuvette with the solvent to detect any drift in the zero balance of the spectrophotometer or contamination of the cuvette, and take appropriate corrective actions if required.

X1.3.4 *Analysis:*

X1.3.4.1 Calculate the mean absorbance of the unirradiated dosimeters, A_0 (see X1.3.1). Calculate the net absorbance, ΔA, for each irradiated dosimeter by subtracting A_0 from its absorbance, A, as follows:

$$\Delta A = A - A_0 \tag{X1.1}$$

X1.3.4.2 The basic equation for calculation of absorbed dose in the dosimetric solution is as follows:

$$D_D = \Delta A \; v/\rho \epsilon G d \tag{X1.2}$$

where:

D_D = absorbed dose in dosimeter solution, Gy,
ΔA = change in absorbance at about 485 nm,
v = dilution factor,
ρ = density of the dosimetric solution, $\text{kg}\cdot\text{m}^{-3}$,
ϵ = molar linear absorption coefficient, $\text{m}^2\cdot\text{mol}^{-1}$,
G = radiation chemical yield of chloride ions, $\text{mol}\cdot\text{J}^{-1}$, and
d = optical pathlength of the dosimetric solution in the cuvette, m.

The values of G and ρ are found in Table 1.

X1.3.4.3 For an irradiation- and absorbance-measurement temperature of 20°C, with a 5-cm pathlength cuvette and dilution factor 5, and using the value of $\epsilon = 399$ $\text{m}^2\cdot\text{mol}^{-1}$ at 485-nm wavelength, Eq (X1.2) reduces to:

$$D_D = 0.2506 \frac{\Delta A}{G\rho} \tag{X1.3}$$

X2. OSCILLOMETRIC READOUT

X2.1 Oscillotitrator

X2.1.1 The oscillotitrator consists of the reader including controls and the oscillator, which is built together with the ampoule holder.

X2.1.2 The unirradiated or the irradiated dosimeter ampoules have to be placed into the ampoule holder for high-frequency conductometric readout. Due to the different concentrations of conducting species in the solution, that is, caused by irradiation, different instrument deflections are shown on the meter (**19, 20**).

X2.1.3 Depending on the concentration of the conducting species, that is, on the absorbed dose, different sensitivity ranges have to be set on the meter unit.

X2.2 Calibration

X2.2.1 *Preparation of Calibration Ampoules:*

X2.2.1.1. Irradiate the dosimeter ampoules in a well-defined, carefully calibrated position of an irradiation facility with different doses in the required dose range.

X2.2.1.2 If a suitable calibration facility is not available, the absorbed dose of the irradiated dosimeters can be determined by titration. The remaining solution can then be filled and sealed in the same size ampoules as those used for routine dose control, thus preparing a series of calibration dosimeter ampoules.

NOTE X2.1—For oscillometric measurements the size of the ampoule, that is, the diameter and the wall thickness of the ampoules, have to be checked so that ampoules can be sorted according to dimension. In order to achieve suitable accuracy (<±5 %) the diameter of the ampoules must be the same within 0.1 mm to that of the calibration ampoules.

X2.2.2 *Preparation of Calibration Curve:*[9]

X2.2.2.1 To establish the calibration curve, that is, the relationship between absorbed dose and instrument deflection, the oscillotitrator can be set in two ways:

(*1*) Set the sensitivity range of the oscillotitrator using the "sensitivity coarse" button. Set the "incr.-decr." direction reversal push button to "incr." position. With an unirradiated dosimeter in the holder, bring the meter pointer to zero by the "compensation coarse" and "compensation fine" knobs. Set the "sensitivity fine" knob to maximum position. Place the dosimeter ampoules of known absorbed doses, in increasing order, into the ampoule holder, and record the instrument deflection shown on the meter. When necessary, that is, when the pointer is off scale, change the sensitivity by using the "sensitivity coarse" buttons. In this way a calibration curve can be obtained in the dose range from 1 to 50 kGy, which can be described by a suitable

---

[9] This procedure applies to the oscillotitrator Model OK-302/1 by Radelkis, Hungary. Refer to pertaining instruction manuals for different models.

mathematical function. In order to set the instrument for later measurements a calibration ampoule of known dose is required to select the suitable sensitivity range. By setting the "compensation coarse" knob to other positions, calibration curves in the 50 to 100-kGy and 100 to 300-kGy dose ranges can be obtained respectively.

(*2*) In order to carry out oscillometric evaluation in narrower dose ranges (that is, 1 to 3 kGy, 3 to 15 kGy, etc.), the instrument deflection can be set by using a compensating circuit and calibration ampoules, which correspond to the lower (1/10) and upper (2/3) range of the given dose range respectively. The series of dosimeter ampoules of known doses are then measured and the instrument deflection recorded. The above mentioned two calibration ampoules are stored and thus the corresponding dose range can repeatedly be set.

X2.3 Dose Evaluation Procedure

X2.3.1 Set the oscillotitrator according to the required dose range as described in X2.2.

X2.3.2 Place the dosimeter ampoule to be evaluated into the ampoule holder and record the instrument deflection. Using the calibration chart or the mathematical function (for example, 3rd order polinomial) the absorbed dose is evaluated.

NOTE X2.2—Due to the effect of temperature on the conductivity of solutions, it is important to note that the temperature of the calibration ampoules and that of the dosimeter ampoules to be measured must be the same. Therefore these ampoules have to be kept together before evaluation at the same place for about half an hour.

In order to avoid the warming up of the ampoules during measurement they should be kept in the ampoule holder only for the duration of the evaluation (1 to 5 s), and they should not be held in the hand for longer times.

In case the temperature of the dose evaluation is significantly different from the temperature at which the calibration curve was taken, a correction is needed (0.17 %/°C).

X3. ADDITIONAL INFORMATION

X3.1 This information is intended to serve as a guide only, since the characteristics of the available readout methods, available sources of dosimeters, and readout instruments may change.

X3.2 The characteristics of some tested readout methods are given in Table X3.1. Only minimum dose is considered here. Maximum dose is not limited by the analytical method because appropriate dilution can be applied to bring the concentration of measured species within the optimum range.

X3.3 Some suppliers of readout instruments are listed in Table X3.2.

X3.4 Some suppliers of ECB dosimeters are listed in Table X3.3.

TABLE X3.1 Characteristics of Some Applicable Methods

Method	Principle of Measurement	Measured Species	Minimum Concentration of Measured Species	Minimum Dose (Gy) where G(HCl) = 0.5 µmol·J^{-1}	References
Spectrophotometry	Absorbance of ferric-SCN complex	Cl^-	3×10^{-6}	10	(**9**)
Potentiometry	Cl^--ion-specific electrode potential	Cl^-	6×10^{-6}	20	(**21**)
Conductometry	Reciprocal resistance	HCl	2×10^{-5}	50	(**22**)
Mercurimetric titration	Diphenylcarbazone color change	Cl^-	4×10^{-5}	100	(**11**)
Alkalimetric titration	Bromophenolblue color change	H^+	4×10^{-5}	100	(**11**)
Coulometry	Time-controlled generation of Ag^+ ions	Cl^-	4×10^{-5}	100	(**4**)
pH Measurement	H^+-ion-specific electrode potential	H^+	4×10^{-5}	100	(**23**)
Oscillometry	High-frequency conductivity	HCl	4×10^{-4}	1000	(**20**)

TABLE X3.2 Some Suppliers of Readout Instruments Suitable for Use with Ethanol-Chlorobenzene (ECB) Dosimetry

Method	Instrument	Supplier
Spectrophotometry	Spectrophotometer	Many suppliers available
Potentiometry	Chlorine analyzer HAG 1	Ströhlein GmbH and Co. Postfach 1460 4044 Kaarst 1 Germany
Conductometry	Conductometer	Many suppliers available
Titration	Burette	Many suppliers available
Coulometry	CCM.1 chloride meter	ChemLab Instruments, Ltd. Hornminster House 129, Upminster Road Hornchuch, Essex RM11 3XJ England
	921 chloride meter	Corning Medical, Corning Ltd. Halstead, Essex CO9 2DX England
	Chloridometer®	Haake Buchler Instruments, Inc. 244 Saddle River Road P.O. Box 549 Saddle Brook, NJ 07662 United States
	ET-1 Microtitre® electrotitrator	World Precision Instruments, Inc. 375 Quinnipiac Ave. New Haven, CT 06513 United States
pH Measurement	pH meter	Many suppliers available
Oscillometry	Oscillotitrator OK-302/1	Radelkis Electrochemical Instruments Laborc utca 1, P.O. Box 106 H-1300 Budapest III Hungary

TABLE X3.3 Some Suppliers of Ethanol-Chlorobenzene (ECB) Dosimeters

Institute of Isotopes
Hungarian Academy of Sciences
P.O. Box 77
H-1525 Budapest
Hungary
Phone: 36 (1) 169-9499
Fax: 36 (1) 156-5045
Telex: (61) 225360 izot h

Radiation Chemistry and Dosimetry Laboratory
"Ruđer Bošković" Institute
Bijenička cesta 54, P.O. Box 1016
41001 Zagreb
Croatia
Phone: 38 (41) 424-794
Fax: 38 (41) 425-497
Telex: 21383 yu irb zg

REFERENCES

(1) Ražem, D., and Dvornik, I., "Application of the Ethanol-Chlorobenzene Dosimeter to Electron-Beam and Gamma-Radiation Dosimetry: II Cobalt-60 Gamma Rays," *Proceedings of IAEA Symposium on Dosimetry in Agriculture, Industry, Biology, and Medicine*, Vienna, 1972, IAEA, Vienna, 1973, pp. 405–419.

(2) Dvornik, I., Ražem, D., and Barić, M., "Application of the Ethanol-Chlorobenzene Dosimeter to Electron-Beam Dosimetry: Pulsed 10 MeV Electrons," *Proceedings of IAEA Symposium on Large Radiation Sources for Industrial Processes*, Munich, 1969; IAEA, Vienna, 1969, pp. 613–622.

(3) Ražem, D., and Dvornik, I., "Scavenging of Electrons Prior to Thermalization in Ethanol," *Journal of Physics and Chemistry*, Vol 84, 1980, pp. 3577–3581.

(4) Ražem, D., and Dvornik, I., "Ethanol-Chlorobenzene Dosimetry for Absorbed Doses Below 1 kGy," *Journal of Applied Radiation and Isotopes*, Vol 38, 1987, pp. 1019–1025.

(5) Ražem, D., and Dvornik, I., "Application of the Ethanol-Chlorobenzene Dosimeter to Electron-Beam and Gamma-Radiation Dosimetry: III. Tissue Equivalent Dosimetry," *Proceedings of IAEA Symposium on Radiation Preservation of Food*, Bombay, 1972; IAEA, Vienna, 1973, pp. 537–547.

(6) Miller, A., and McLaughlin, W. L., "Calculation of the Energy Dependence of Dosimeter Response to Ionizing Photons," *International Journal of Applied Radiation and Isotopes*, Vol 33, 1982, pp. 1299–1310.

(7) Miller, A., "Calculation of Energy Dependence of Some Commonly Used Dosimeters," *Proceedings of IAEA Symposium on High-Dose Dosimetry*, Vienna, 1984; IAEA, Vienna, 1985, pp. 425–436.

(8) Kovács, A., and Stenger, V., "Environmental Effects on the Ethanol-Monochlorobenzene Dosimeter System Before, During and After Irradiation," *Proceedings of Final Research Co-ordination Meeting. Research in Radiation Processing Dosimetry*, Munich, 1983; IAEA-TECDOC-321, IAEA, Vienna, 1984, pp. 91–139.

(9) Ražem, D., Očić, G., Jamičić, J., and Dvornik, I. "Application of the Ethanol-Chlorobenzene Dosimeter to Electron-Beam and Gamma Radiation. IV. Spectrophotometry of Coloured Secondary