ISO/ASTM 51538:2017(E)

Standard Practice for
Use of the Ethanol-Chlorobenzene Dosimetry System[1]

This standard is issued under the fixed designation ISO/ASTM 51538; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision.

1. Scope

1.1 This practice covers the preparation, handling, testing, and procedure for using the ethanol-chlorobenzene (ECB) dosimetry system to measure absorbed dose to water when exposed to ionizing radiation. The system consists of a dosimeter and appropriate analytical instrumentation. For simplicity, the system will be referred to as the ECB system. The ECB dosimeter is classified as a type I dosimeter on the basis of the effect of influence quantities. The ECB dosimetry system may be used as a reference standard dosimetry system or as a routine dosimetry system.

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

1.3 This practice describes the mercurimetric titration analysis as a standard readout procedure for the ECB dosimeter when used as a reference standard dosimetry system. Other readout methods (spectrophotometric, oscillometric) that are applicable when the ECB system is used as a routine dosimetry system are described in Annex A1 and Annex A2.

1.4 This practice applies only to gamma radiation, X-radiation/bremsstrahlung, and high energy electrons.

1.5 This practice applies provided the following conditions are satisfied:

1.5.1 The absorbed dose range is between 10 Gy and 2 MGy for gamma radiation and between 10 Gy and 200 kGy for high current electron accelerators (**1, 2**).[2] (**Warning**—the boiling point of ethanol chlorobenzene solutions is approximately 80 °C. Ampoules may explode if the temperature during irradiation exceeds the boiling point. This boiling point may be exceeded if an absorbed dose greater than 200 kGy is given in a short period of time.)

1.5.2 The absorbed-dose rate is less than 10^6 Gy s^{-1}(**2**).

1.5.3 For radionuclide gamma-ray sources, the initial photon energy is greater than 0.6 MeV. For bremsstrahlung photons, the energy of the electrons used to produce the bremsstrahlung photons is equal to or greater than 2 MeV. For electron beams, the initial electron energy is greater than 8 MeV (**3**).

NOTE 1—The same response relative to ^{60}Co gamma radiation was obtained in high-power bremsstrahlung irradiation produced by a 5 MeV electron accelerator (**4**).

NOTE 2—The lower energy limits are appropriate for a cylindrical dosimeter ampoule of 12-mm diameter. Corrections for dose gradients across the ampoule may be required for electron beams. The ECB system may be used at lower energies by employing thinner (in the beam direction) dosimeters (see ICRU Report 35). The ECB system may also be used at X-ray energies as low as 120 kVp (**5**). However, in this range of photon energies the effect caused by the ampoule wall is considerable.

NOTE 3—The effects of size and shape of the dosimeter on the response of the dosimeter can adequately be taken into account by performing the appropriate calculations using cavity theory (**6**).

1.5.4 The irradiation temperature of the dosimeter is within the range from −30 °C to 80 °C.

NOTE 4—The temperature dependence of dosimeter response is known only in this range (see 5.2). For use outside this range, the dosimetry system should be calibrated for the required range of irradiation temperatures.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* Specific warnings are given in 1.5.1, 9.2 and 10.2.

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

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

2. Referenced documents

2.1 *ASTM Standards:*[3]

C912 Practice for Designing a Process for Cleaning Technical Glasses

D1193 Specification for Reagent Water

E170 Terminology Relating to Radiation Measurements and Dosimetry

E275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers

E666 Practice for Calculating Absorbed Dose From Gamma or X Radiation

E668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices

E925 Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Bandwidth does not Exceed 2 nm

E958 Practice for Estimation of the Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers

2.2 *ISO/ASTM Standards:*[3]

51261 Practice for Calibration of Routine Dosimetry Systems for Radiation Processing

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

52628 Practice for Dosimetry in Radiation Processing

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

2.3 *ISO Standards:*[4]

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

2.4 *ISO/IEC Standards:*[4]

17025 General Requirements for the Competence of Testing and Calibration Laboratories

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

JCGM 100:2008, GUM 1995, with minor correctons Evaluation of measurement data – Guide to the Expression of Uncertainty in Measurement[5]

JCGM 200:2012, (JCGM 200:2008 with minor revisions) VIM, International Vocabulary of Metrology – Basis and General Concepts and Associated Terms[6]

2.6 *International Commission on Radiation Units and Measurements (ICRU) Reports:*[7]

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

ICRU Report 80 Dosimetry Systems for Use in Radiation Processing

ICRU Report 85a Fundamental Quantities and Units for Ionizing Radiation

3. Terminology

3.1 *Definitions:*

3.1.1 *approved laboratory*—laboratory that is a recognized national metrology institute, or has been formally accredited to ISO/IEC 17025, or has a quality system consistent with the requirements of ISO/IEC 17025.

3.1.1.1 *Discussion*—A recognized national metrology institute or other calibration laboratory accredited to ISO/IEC 17025 should be used in order to ensure traceability to a national or international standard. A calibration certificate provided by a laboratory not having formal recognition or accreditation will not necessarily be proof of traceability to a national or international standard.

3.1.2 *calibration*—set of operations that establish, under specified conditions, the relationship between values indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realised by standards.

3.1.2.1 *Discussion*—Calibration conditions include environmental and irradiation conditions present during irradiation, storage and measurement of the dosimeters that are used for the generation of a calibration curve.

3.1.3 *calibration curve*—expression of the relation between indication and corresponding measured quantity value.

3.1.3.1 *Discussion*—In radiation processing standards, the term "dosimeter response" is generally used for "indication".

3.1.4 *dosimetry system*—system used for determining absorbed dose, consisting of dosimeters, measurement instruments and their associated reference standards, and procedures for the system's use.

3.1.5 *ethanol-chlorobenzene dosimeter*—partly deoxygenated solution of chlorobenzene (CB) in 96 volume % ethanol in an appropriate container, such as a flame-sealed glass ampoule, used to indicate absorbed dose by measurement of the amount of HCl formed under irradiation.

3.1.6 *measurement management system*—set of interrelated or interacting elements necessary to achieve metrological confirmation and continual control of measurement processes.

3.1.7 *metrological traceability*—property of a measurement whereby the result can be related to a reference through a documented unbroken chain of comparisons, each contributing to the measurement uncertainty.

3.1.8 *molar linear absorption coefficient ε_m*—constant relating the spectrophotometric absorbance, A_λ, of an optically

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[3] For referenced ASTM and ISO/ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

[5] Document produced by Working Group I of the Joint Committee for Guides in Metrology (JCGM WG1). Available free of charge at the BIPM website (http://www.bipm.org).

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

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

absorbing molecular species at a given wavelength, λ, per unit pathlength, d, to the molar concentration, c, of that species in solution:

$$\epsilon_m = \frac{A_\lambda}{d \times c} \qquad (1)$$

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

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

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

$$G(x) = n(x)/\bar{\varepsilon} \qquad (2)$$

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

3.1.10 *reference standard dosimetry system*—dosimetry system, generally having the highest metrological quality available at a given location, from which measurements made there are derived.

3.1.11 *routine dosimetry system*—dosimetry system calibrated against a reference standard dosimetry system and used for routine absorbed-dose measurements, including dose mapping and process monitoring.

3.1.12 *type 1 dosimeter*—dosimeter of high metrological quality, the response of which is affected by individual influence quantities in a well-defined way that can be expressed in terms of independent correction factors.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *conductometry*—analytical method based on the measurement of conductivity of solutions.

3.2.1.1 *Discussion*—The conductivity of a solution depends on the concentration of free ions in the solution.

3.2.2 *oscillometry*—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.

3.3 Definitions of other terms used in this standard that pertain to radiation measurement and dosimetry may be found in ISO 12749-4, ASTM Terminology E170, ICRU 85a and VIM; these definitions, therefore, may be used as an alternative references.

4. Significance and use

4.1 The ECB dosimetry system provides a reliable means of measuring absorbed dose to water. It is based on a process of radiolytic formation of hydrochloric acid (HCl) in aqueous ethanolic solutions of chlorobenzene by ionizing radiation ((**7, 8**), ICRU 80).

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. Radiation chemical yields (*G*) for the formation of HCl in typical ECB solution formulations are given in Table 1.

4.3 The irradiated 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 (**10**).

TABLE 1 Radiation chemical yields (*G*) for the formation of HCl in typical ECB solution formulations

Concentration of CB, vol %	Density at 20 °C kg · m⁻³	Ratio of Coefficients[A]	Radiation Chemical Yields at 20 °C[B] (µmol · J⁻¹)	
			⁶⁰Co Gamma Radiation (**9**)	4 to 10 MeV Electrons (**3**)
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 photon mass energy-absorption coefficients for water and the dosimeter solution at ⁶⁰Co gamma ray energy:

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

[B] Radiation chemical yield 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 % acetone and 0.04 % benzene.

4.4 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 radiation, and electron mass collision stopping powers (S/ρ), over a broad energy range from 10^{-2} to 100 MeV (**11-14**).

4.5 The ECB dosimetry system may be used with other radiation types, such as neutrons (**15**), and protons (**16**). Meaningful dosimetry of any radiation types and energies novel to the system's use requires that the respective radiation chemical responses applicable under the circumstances be established in advance.

5. Effect of Influence Quantities

5.1 Guidance on the determination of the performance characteristics of dosimeters and dosimetry systems can be found in ISO/ASTM Guide 52701. The relevant influence quantities that need to be considered when using the ECB dosimetry system are given below.

5.2 The irradiation temperature dependence of dosimeter response is a complex function of dose and temperature for each concentration of chlorobenzene (that is, for each formulation). This dependence arises directly from the temperature dependence of radiation chemical yield, *G*. The analysis of the published data (**17**) shows that the irradiation temperature dependence of *G* between 20 °C and 80 °C at any chlorobenzene concentration can be described by a simple exponential expression:

$$G_t = G_0\ exp[k(t - 20)] \qquad (3)$$

where:

G_t = radiation chemical yield in µmol J^{-1} at a given temperature t in °C,

G_0 = radiation chemical yield in µmol J^{-1} at 20 °C (G_0 for different ECB solutions are given in Table 1), and

k = temperature coefficient in $°C^{-1}$ applicable at a given dose.

5.2.1 The values of *k* are given in Table 2.

6. Interferences

6.1 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.

6.2 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 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.

6.3 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 (18).

7. Apparatus

7.1 This practice describes mercurimetric titration of radiolytically formed Cl⁻ ions as a standard readout procedure for the ECB system when used as a reference-standard dosimetry system.

7.2 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 uncertainty. Control the temperature of all solutions during handling at 20 ± 2 °C to ensure correct measurement of volumes.

7.3 Use borosilicate glass or equivalent chemically resistant glass to store the reagents and the prepared dosimetric solution, and to perform the titration. Clean all apparatus thoroughly before use (see ASTM Practice C912).

7.4 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 (see ASTM Practices E666 and E668).

NOTE 5—The dosimetric ampoule commonly used has a capacity of about 5 mL. Quick-break, glass ampoules or "Type 1 glass" colorbreak ampoules or equivalent containers, may be used. Commercially available pharmaceutical ampoules have been found to give reproducible results without requiring additional cleaning.

8. Reagents

8.1 Analytical reagent grade chemicals shall be used in this practice for preparing all solutions.[8]

8.2 Triply distilled water from coupled all-glass stills or water from a high-quality commercial purification unit capable of achieving Total Oxidizable Carbon (T.O.C.) content below 5 ppb should be used. Type II reagent water as specified in ASTM Specification D1193 is also considered to be of sufficient quality for use in preparing solutions and 96 volume % ethanol.

NOTE 6—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.

9. Preparation of dosimeters

9.1 Dosimetric solutions may contain any concentration of CB. For practical reasons, only a few 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.

9.2 Prepare 96 volume % aqueous ethanol first by adding absolute ethanol into a volumetric flask containing the appropriate amount of water. (**Warning—**Ethanol is flammable.) Use this aqueous ethanol for making the dosimetric solutions of the desired concentrations by adding it into volumetric flasks containing appropriate amounts of CB. Store the dosimetric solution in the dark. (**Warning—**Chlorobenzene is toxic and a skin irritant. Appropriate precaution should be taken to avoid contact with the solution during preparation and analysis of the dosimeters. Used solutions should be disposed of as hazardous waste.)

9.3 Fill the dosimeter ampoules with the dosimetric 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.

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[8] Reagent specifications are available from the American Chemical Society, 1115 16th Street, NW, Washington, DC 20036, USA.

TABLE 2 Temperature coefficients k (°C)⁻¹ for typical ECB solution formulations as derived from Ref (17)

Concentration of CB, vol %	2.5 kGy	5 kGy	10 kGy	15 kGy	20 kGy	25 kGy
4	−0.0002	−0.0004	−0.0007	−0.0011	−0.0015	−0.0019
10	0.0018	0.0014	0.0009	0.0002	0.0	0.0
20, 25, 40	0.0037	0.0031	0.0020	0.0013	0.0008	0.0

NOTE 1— For intermediate doses interpolation should be made.

NOTE 7—To minimize the removal of the vapor above the dosimetic solution in the ampoules, the nitrogen is saturated with the vapors of the dosimetric solution by passing it through ECB solution of the same composition before the bubbling of the dosimeter ampoules.

10. Calibration of the mercuric nitrate solution

10.1 The dosimeter 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 (**19**).

10.2 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} . (**Warning**—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. Used solutions should be disposed of as hazardous waste. Hazards of mercury poisoning can be avoided by using some of the alternative readout methods described in Annex A2 and Table A3.1 in Annex A3.)

10.2.1 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 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.

10.2.2 Prepare 0.2 mol dm^{-3} HNO_3 in ethanol and 1 % ethanolic solutions of diphenylcarbazone (DPC).

10.3 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 mol dm^{-3} 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.

10.4 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 consumptions 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.

11. Calibration of the dosimetry system

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

11.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations shall be performed at an approved laboratory.

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

11.2.2 When the ECB dosimeter is used as a routine dosimeter, the calibration irradiation may be performed by irradiating the dosimeters at (*a*) an approved laboratory using criteria specified in ISO/ASTM Practice 51261, (*b*) an in-house calibration facility that provides an absorbed dose (or an absorbed-dose rate) having traceability to nationally or internationally recognized standards, or (*c*) a production irradiator under actual production irradiation conditions, together with reference- or transfer-standard dosimeters issued and read by an approved laboratory.

NOTE 9—If the procedures outlined in Sections 5 – 10 are followed, the radiation chemical yield for the ethanol-chlorobenzene dosimetric solution is expected to be in agreement with the values shown in Table 1 or in the literature and to be approximately constant over the dose range. Quality control testing of the dosimetric solution can be performed by comparing dosimetric solution parameters such as the radiation chemical yield of HCl with accepted values. If the radiation chemical yield is significantly different from that in Table 1 or in the literature, there is an indication of possible contamination of the solution, or some other problem that needs to be resolved.

11.2.3 Calibration shall be performed for all new dosimetric solutions. If the preparation procedure has been demonstrated to give consistently the same radiation chemical yield, the number of absorbed-dose levels required for the calibration irradiations can be reduced to the minimum needed to check the response and to demonstrate its linearity with dose.

11.3 *Measurement Instrument Calibration and Performance Verification*—For the calibration of the instruments, and for the verification of instrument performance between calibrations, see ISO/ASTM Practice 51261 and/or instrument-specific operating manuals.

11.4 *Dosimeter Measurement by Mercurimetric Titration:*

11.4.1 Transfer the irradiated dosimetric solution into a beaker for titration. Rinse the dosimeter ampoule several times with a total volume of 5 mL of technical grade ethanol, so that the final volume in the beaker is 10 mL. Add 1 mL of 0.2 mol dm^{-3} 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 10—If high absorbed doses are to be measured, use appropriate portions of irradiated dosimetric solution, taking care that total volume in the beaker is 10 mL.

11.5 *Analysis:*

11.5.1 Subtract the blank (as defined in 10.4) from the amounts of $Hg(NO_3)_2$ solutions consumed and calculate [Cl^-], the concentration of radiolytically formed Cl^- ions in each dosimeter:

$$(\text{equivalents of } Cl^-) = (\text{equivalents of } Hg^{2+})\times \qquad (4)$$

$$\frac{(\text{mL of titrant}) - (\text{blank})}{\text{mL of dosimetric solution}}$$

11.5.2 Correct response for the irradiation temperature by using Eq 3 (see 5.2).

11.5.3 Obtain a calibration curve for [Cl⁻] as a function of the absorbed dose, D. Fit the data by means of a least-squares method with an appropriate analytical form that best fits the data.

11.6 *Quality control:*

11.6.1 For quality control of the dosimetric solution calculate the radiation chemical yield as follows:

$$G(Cl^-) = \frac{[Cl^-]}{D\rho} \tag{5}$$

where:

D = known absorbed dose to the dosimetric solution, Gy
ρ = density of the dosimetric solution, kg m^{-3}

11.6.2 Published values of $G(Cl^-)$ and ρ are found in Table 1 or in the literature (**3, 9, 20**). The calculated radiation chemical yield should agree with the published values within the measurement uncertainty. If the calculated radiation chemical yield differs from the published value by more than the expanded uncertainty with a coverage factor $k = 2$, there is an indication of possible contamination of the solution or some other problem that needs to be resolved.

NOTE 11—Eq 5 is only to be used for the purpose of quality control of the dosimetric solution, and not for calculating an unknown dose from the dosimeter response [Cl⁻]. The calibration curve obtained in 11.5.3 should be used for determining dose.

12. Application of dosimetry system

12.1 For use as a transfer-standard dosimeter, a minimum of two dosimeters should be used for each absorbed-dose measurement. The number of dosimeters required for the measurement of absorbed dose on or within a material is determined by the reproducibility associated with the dosimetry system and the required measurement uncertainty associated with the application. Appendix X3 of ASTM Practice E668 describes a statistical method for determining this number.

12.2 The measurement and analysis procedures should be performed in accordance with 11.4 and 11.5.

12.3 The absorbed dose to water is determined from the analysis results and the calibration curve.

NOTE 12—The absorbed dose to materials other than water irradiated under equivalent conditions may be calculated using the procedure given in ASTM Practices E666 and E668.

12.4 Requirements for recording the calculated absorbed dose values and all other relevant data are outlined in Section 13.

13. Minimum documentation requirements

13.1 *Calibration of the Dosimetry System:*

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

13.1.2 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.

13.2 *Application:*

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

13.2.2 Record or reference the radiation source type and characteristics.

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

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

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

14. Measurement Uncertainty

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

14.2 All components of uncertainty should be included in the estimate, including those arising from calibration dosimeter reproducibility, instrument reproducibility, and the effect of influence quantities. A full quantitative analysis of components of uncertainty may be referred to as an uncertainty budget, and is then often presented in the form of a table. Typically, the uncertainty budget will identify all significant components of uncertainty, together with their methods of estimation, statistical distributions and magnitudes.

14.3 If this practice is followed, the estimate of the expanded uncertainty of an absorbed dose determined by this dosimetry system should be less than 3 % for a coverage factor $k = 2$ (which corresponds approximately to a 95 % level of confidence for normally distributed data).

15. Keywords

15.1 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-radiation; ICS 17.240

ANNEXES

(informative)

A1. SPECTROPHOTOMETRIC READOUT

A1.1 Apparatus

A1.1.1 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 all reagent solutions, of the glassware and solvents, and of the dosimetric solution at 20 ± 2 °C during absorbance measurements.

A1.2 Spectrophotometer calibration

A1.2.1 Check the wavelength scale of the spectrophotometer. Appropriate wavelength standards are holmium-oxide filters or solutions, and may be obtained from the spectrophotometer manufacturer or other scientific laboratory instrument suppliers. For more details see ASTM Practices E275, E925, and E958.

Note A1.1—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).

A1.2.2 Check the accuracy of the photometric (absorbance) scale of the spectrophotometer. Certified absorbance standard filters or solutions are available for this purpose (21).

Note A1.2—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.

A1.3 Irradiation and measurement procedures

A1.3.1 *Calibration Irradiation of Dosimeters*—Separate five dosimeters from the remainder of the batch and do not irradiate them. Use them in determining A_o. Follow 11.2 for the remainder of the procedure.

A1.3.2 *Development of the Colored Secondary Complex*—Transfer the irradiated dosimetric solution quantitatively into a 25-mL volumetric flask. Add 1.5 mL of 5.25 mol dm^{-3} aqueous HClO$_4$, 50 µL of 0.37 mol dm^{-3} 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 solution prepared in the same way but using unirradiated dosimetric solution (blank) in a 5-cm cuvette.

Note A1.3—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.

A1.3.3 *Measurement:*

A1.3.3.1 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 wevelength is about 485 nm.

A1.3.3.2 Set the balance of the spectrophotometer to zero with air only (no cuvette) in the light path(s).

A1.3.3.3 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_∞).

A1.3.3.4 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 A1.3.3.3. Place the cuvette in the sample holder and measure the absorbance.

Note A1.4—Inadequate rinsing of the cuvette between dosimetric solutions can lead to errors due to solution carryover (cross-contamination). Techniques for minimizing this effect are discussed in Ref (21).

A1.3.3.5 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.

A1.3.4 *Analysis:*

A1.3.4.1 Calculate the mean absorbance of the unirradiated dosimeters, A_o (see A1.3.1). Calculate the net absorbance, ΔA, for each irradiated dosimeter by subtracting A_o from its absorbance, A, as follows:

$$\Delta A = A - A_o \tag{A1.1}$$

A1.3.4.2 Correct response for the irradiation temperature (see 5.2).

A1.3.4.3 Obtain a calibration curve for ΔA as a function of absorbed dose, D. Fit the data by means of a least-squares method with an appropriate analytical form that provides a best fit to the data.

A1.3.4.4 The basic equation for calculation of the radiation chemical yield for the dosimetric solution is as follows:

$$G = \Delta A v / \rho \varepsilon_m D_D d \tag{A1.2}$$

where:

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

Published values of G and ρ are found in Table 1 and in the literature. The calculated radiation chemical yield should agree with the published values within the expanded uncertainty.

A1.3.4.5 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 $\varepsilon_m = 399$ m$^2 \cdot$ mol^{-1} at 485-nm wavelength, Eq A1.2 reduces to:

$$G = 0.2506 \frac{\Delta A}{D_D \rho} \qquad (A1.3)$$

A1.4 Application of the dosimetry system

A1.4.1 For use as a transfer-standard dosimeter, a minimum of two dosimeters should be used for each absorbed-dose measurement. The number of dosimeters required for the measurement of absorbed dose on or within a material is determined by the reproducibility associated with the dosimetry system and the required measurement uncertainty associated with the application. Appendix X3 of ASTM Practice E668 describes a statistical method for determining this number.

A1.4.2 The measurement and analysis procedures should be performed in accordance with A1.3.3 and A1.3.4.

A1.4.3 The absorbed dose to water is determined from the analysis results and the calibration curve.

NOTE A1.5—The absorbed dose to materials other than water irradiated under equivalent conditions may be calculated using the procedures given in ASTM Practices E666 and E668.

A1.4.4 Requirements for recording the calculated absorbed dose values and all other relevant data are outlined in Section 12.

A2. OSCILLOMETRIC READOUT

A2.1 Oscillotitrator

A2.1.1 The oscillotitrator consists of the reader including controls and the oscillator, which is built together with the ampoule holder.

A2.1.2 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 (**22, 23**).

A2.1.3 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.

A2.2 Calibration

A2.2.1 *Calibration Irradiation of Dosimeters*—Separate one dosimeter from the remainder of the batch and do not irradiate it. Use it for zeroing the instrument. Follow Section 11.2 for the remainder of the procedure.

NOTE A2.1—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 in the measured dose value (that is, uncertainty value of less than 5 %) the diameter of the ampoules must be the same within 0.1 mm to that of the calibration ampoules.

A2.2.2 *Preparation of Calibration Curve*[9]—To establish the calibration curve, that is, the relationship between absorbed dose and instrument deflection, set the oscillotitrator according to manufacturer's instructions. For the Model OK-302/1 from Radelkis, Budapest, the oscillotitrator can be set in two ways:

A2.2.2.1 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 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

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[9] This procedure applies to the oscillotitrator Model OK-302/1 by Radelkis, Hungary. Refer to pertaining instruction manuals for different models. A new readout instrument, the oscillometric reader by Sensolab, Hungary, is a programmable digital unit with a slightly different procedure. This procedure is available from the manufacturer: Sensolab Ltd., Török Ignac utca, H-2131, Göd, Hungary.

curves in the 50 to 100-kGy and 100 to 300-kGy dose ranges can be obtained respectively.

A2.2.2.2 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.

A2.3 Dose evaluation procedure

A2.3.1 Set the oscillotitrator according to the required dose range as described in A2.2.

A2.3.2 Place the dosimeter ampoule to be evaluated into the ampoule holder and record the instrument deflection. Using the

calibration curve (for example, 3rd order polynomial) the absorbed dose is evaluated.

A2.3.3 *Temperature dependence:*

A2.3.3.1 For the ECB formulation containing 24 vol % chlorobenzene, linear temperature dependence, represented as 0.05 %/°C, has been reported at 30 kGy for irradiation temperatures between –30 °C and +50 °C (24).

NOTE A2.2—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).

A3. ADDITIONAL INFORMATION

A3.1 This information is intended to serve as a guide only, since the characteristics of the available readout methods, available sources of dosimeters,[10] and readout instruments may change.

A3.2 The characteristics of some tested readout methods are given in Table A3.1. 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.

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[10] The sole source of supply of ethanol-chlorobenzene dosimeters known to the committee at this time is Institute of Isotopes, Centre for Energy Research, Hungarian Academy of Sciences, P.O. Box 49, H-1525 Budapest, Hungary. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

TABLE A3.1 Characteristics of some applicable methods

Method	Principle of Measurement	Measured Species	Minimum Concentration of Measured Species, mol dm^{-3}	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	[25]
Conductometry	Reciprocal resistance	HCl	2×10^{-5}	50	[26]
Mercurimetric titration	Diphenylcarbazone color change	Cl$^-$	4×10^{-5}	100	[19]
Alkalimetric titration	Bromophenolblue color change	H$^+$	4×10^{-5}	100	[19]
Coulometry	Time-controlled generation of Ag$^+$ ions	Cl$^-$	4×10^{-5}	100	[10]
pH Measurement	H$^+$-ion-specific electrode potential	H$^+$	4×10^{-5}	100	[27]
Oscillometry	High-frequency conductivity	HCl	4×10^{-4}	1000	[23]

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