# INTERNATIONAL STANDARD



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# Practice for use of the ethanolchlorobenzene dosimetry system

Pratique de l'utilisation d'un système dosimétrique à l'éthanol iTeh schorobenzere RD PREVIEW (standards.iteh.ai)

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# ISO/ASTM 51538:2009(E)

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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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.

ASTM International is one of the world's largest voluntary standards development organizations with global participation from affected stakeholders. ASTM technical committees follow rigorous due process balloting procedures.

A project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this project, ASTM Subcommittee E10.01, Radiation Processing: Dosimetry and Applications, is responsible for the development and maintenance of these dosimetry standards with unrestricted participation and input from appropriate ISO member bodies.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. Neither ISO nor ASTM International shall be held responsible for identifying any or all such patent rights.

International Standard ISO/ASTM 51538 was developed by ASTM Committee E10, Nuclear Technology and Applications, through Subcommittee E10.01, and by Technical Committee ISO/TC 85, Nuclear energy.

This second edition cancels and replaces the first edition (ISO/ASTM 51538:2002), which has been technically revised.

# ISO/ASTM 51538:2009(E)



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

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 procedure for preparation, handling, testing, and use of the ethanol-chlorobenzene (ECB) dosimetry system to determine absorbed dose (in terms of absorbed dose to water) in materials irradiated by photons (gamma radiation or X-radiation/bremsstrahlung) or high energy electrons. 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 dosimetry system and is also used as a routine dosimetry system (see ISO/ASTM Guide 51261).

1.2 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.

are satisfied:

1.3.1 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). (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.)

1.3.2 The absorbed-dose rate is less than  $10^6$  Gy s<sup>-1</sup>(2).

1.3.3 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 equal to or greater than 4 MeV (3) (see ICRU Reports 34 and 35).

Note 1-The same response relative to 60Co gamma radiation was obtained in high-power bremsstrahlung irradiation produced by a 5 MeV electron accelerator (4). The lower limits of 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 4 MeV by employing thinner (in the beam direction) dosimeters. 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.

1.3.4 The irradiation temperature of the dosimeter is within the range from  $-40^{\circ}$ C to  $80^{\circ}$ C.

NOTE 2-The temperature dependence of dosimeter response is known only in this range (see 4.3). For use outside this range, the dosimetry system should be calibrated for the required range of irradiation temperatures.

1.4 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)**. • **PREVIEW** 1.5 This standard does not purport to address all of the 1.3 This practice applies provided the following conditions 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

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# 2. Referenced documents

- 2.1 ASTM Standards:<sup>3</sup>
- C 912 Practice for Designing a Process for Cleaning Technical Glasses
- D 1193 Specification for Reagent Water
- E 170 Terminology Relating to Radiation Measurements and Dosimetry
- E 275 Practice for Describing and Measuring Performance of Ultraviolet and Visible Spectrophotometers
- E 666 Practice for Calculating Absorbed Dose From Gamma or X Radiation
- E 668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices
- E 925 Practice for Monitoring the Calibration of Ultraviolet-Visible Spectrophotometers whose Spectral Slit Width does not Exceed 2 nm
- E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers

<sup>&</sup>lt;sup>1</sup> This practice is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.01 on Radiation Processing: Dosimetry and Applications, and is also under the jurisdiction of ISO/TC 85/WG 3.

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

<sup>2.2</sup> ISO/ASTM Standards:<sup>3</sup>

<sup>51261</sup> Guide for Selection and Calibration of Dosimetry

<sup>&</sup>lt;sup>3</sup> 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.



Systems for Radiation Processing

- 51400 Practice for Characterization and Performance of a High-Dose Gamma-Radiation Dosimetry Calibration Laboratory
- 51707 Guide for Estimating Uncertainties in Dosimetry for **Radiation Processing**

2.3 International Commission on Radiation Units and Measurements (ICRU) Reports:<sup>4</sup>

- ICRU Report 14 Radiation Dosimetry: X-Rays and Gamma Rays with Maximum Photon Energies Between 0.6 and 60 MeV
- ICRU Report 17 Radiation Dosimetry: X-Rays Generated at Potentials of 5 to 150 kV

ICRU Report 34 The Dosimetry of Pulsed Radiation

- ICRU Report 35 Radiation Dosimetry: Electrons with Initial Energies Between 1 and 50 MeV
- ICRU Report 37 Stopping Powers for Electrons and Positrons
- ICRU Report 60 Fundamental Quantities and Units for **Ionizing Radiation**

# 3. Terminology

3.1 *Definitions:* 

3.1.1 *calibration*—set of operations under specified conditions, which establishes the relationship between values indi-cated by a measuring instrument or measuring system, and the or the value of a standard whereby it can be related to stated corresponding values realised by standards traceable to a nationally or internationally recognized laboratory.

3.1.1.1 Discussion—Calibrations conditions it include a lenviandar diesst/3dcd1b21-712d-4c7c-b881ronmental and irradiation conditions present during inradiation/iso-asti3:21 Definitions of Terms Specific to This Standard: storage and measurement of the dosimeters that are used for the generation of a calibration curve. To achieve stable environmental conditions, it may be necessary to condition the dosimeters before performing the calibration procedure.

3.1.2 calibration curve-graphical representation of the dosimetry system's response function.

3.1.3 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.

3.1.4 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.

3.1.5 measurement quality assurance plan-documented program for the measurement process that ensures that the expanded uncertainty consistently meets the requirements of the specific application. This plan requires traceability to nationally or internationally recognized standards.

3.1.6 molar linear absorption coefficient  $\varepsilon_m$ —constant relating the spectrophotometric absorbance,  $A_{\lambda}$ , of an optically absorbing molecular species at a given wavelength,  $\lambda$ , per unit pathlength, d, to the molar concentration, c, of that species in solution:

$$\varepsilon_m = \frac{A_\lambda}{d \times c} \tag{1}$$

(SI unit: m<sup>2</sup> mol<sup>-1</sup>)

3.1.6.1 Discussion-It is sometimes expressed in units of L mol  $^{-1}$  cm $^{-1}$ .

3.1.7 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,  $\bar{\epsilon}$ imparted to the matter.

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

(SI unit: mol J<sup>-1</sup>)

3.1.8 reference-standard dosimeter-dosimeter of high metrological quality used as a standard to provide measurements traceable to measurements made using primary-standard dosimeters.

3.1.9 response function-mathematical representation of the relationship between dosimeter response and absorbed dose, for a given dosimetry system.

3.1.10 routine dosimeter-dosimeter calibrated against a primary-, reference-, or transfer-standard dosimeter and used for routine absorbed-dose measurements.

references, usually national or international standards, through ISO/ASTM 54n 2mbroken chain of comparisons all having stated uncertain-

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 ASTM Terminology Standard E 170. Definitions in ASTM E 170 are compatible with ICRU 60; that document, therefore, may be used as an alternative reference.

#### 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 (7, 8).

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 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 (9).

<sup>&</sup>lt;sup>4</sup> Available from the Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814, USA.



## 4.3 Effect of Irradiation Temperature:

4.3.1 The temperature dependence of dosimeter response is a complex function of dose and temperature for each concentration of chlorobenzene (that is, for each formulation). The analysis of the published data (10) shows that the temperature dependence between  $20^{\circ}$ C and  $80^{\circ}$ C at any chlorobenzene concentration can be described by a simple exponential expression:

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

where:

- $G_t$  = the radiation chemical yield in µmol J<sup>-1</sup> at a given temperature t in °C,
- $G_0$  = the radiation chemical yield in µmol J<sup>-1</sup> at 20°C (G<sub>0</sub> for different ECB solutions are given in Table 1), and
- k = the temperature coefficient in  $(^{\circ}C)^{-1}$  applicable at a given dose.

4.3.2 The values of k are given in Table 2.

**TABLE 1** Typical ECB solution formulations

Concentration	Donaity at 20°C	Potio of	Radiation 0 at 20°C	Chemical Yields <sup>B</sup> (µmol⋅ J <sup>−1</sup> )	
of CB, vol %	kg · m <sup>−3</sup>	Coefficients <sup>A</sup>	<sup>60</sup> Co Gamma	4 to 10 MeV Electrons (3)	
		iTe	Rays (11)	ANDAI	R
4	819	0.989	0.42 <sup>C</sup>		
10	839	0.995	0.52	andard	
20	869	1.006	0.59	anuaru	D
24	880	1.011	0.60	0.57 <sup>D</sup>	
40	925	1.027	0.63		

<sup>A</sup> The ratio of the mass energy-absorption coefficients for water and the dosimeter solution at <sup>60</sup>Co gamma ray thergy standards. Iteh ai/catalog/standards/violetclight | and should bbe | kept in the dark for long-term  $f = \frac{(\mu_{en}/\rho)_w}{(\mu_{en}/\rho)_D}$  6175495c1ec9/iso-aststorage8-No/special precautions are required during routine handling under normal laboratory lighting conditions but

<sup>B</sup> Radiation chemical yields of HCl 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 % acetone and 0.04 % benzene.

4.3.3 Between  $-30^{\circ}$ C and  $50^{\circ}$ C the temperature coefficient 0.015 kGy/°C applies at 30 kGy dose (**12**). Information on the temperature dependence of dosimeter response during irradiation between 20 and 80°C is found in Ref (**10**), and between -40 and 20°C in Ref (**13**).

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 ( $\mu_{en}/\rho$ ) for X-and gamma radiation, and electron mass collision stopping powers (S/ $\rho$ ), over a broad energy range from  $10^{-2}$  to 100 MeV (14-17).

4.5 The absorbed dose that is determined is the dose absorbed in the water. Absorbed dose in other materials irradiated under equivalent conditions may be calculated.

Procedures for making such calculations are given in ASTM Practices E 666 and E 668 and ISO/ASTM Guide 51261.

NOTE 3—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.

4.6 The ECB dosimetry system may be used with other radiation types, such as neutrons (18), and protons (19). 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. 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.

stationages-No/special precautions are required during routine handling under normal laboratory lighting conditions, but strong ultraviolet (UV) sources such as sunlight should be avoided (20).

### 6. Apparatus

6.1 This practice describes mercurimetric titration of radiolytically formed Cl<sup>-</sup> ions as a standard readout procedure for the ECB system when used as a reference-standard dosimetry system.

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 uncertainty. Control the temperature of all solutions during handling at  $20 \pm 2^{\circ}$ C.

6.3 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 C 912).

TABLE 2 Temperature coefficients k (°C)<sup>-1</sup> for typical ECB solution formulations

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.



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 (see ASTM Practices E 666 and E 668).

NOTE 4—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.

#### 7. Reagents

7.1 Analytical reagent grade chemicals shall be used in this practice for preparing all solutions.<sup>5</sup>

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

NOTE 5—High-purity water is commercially available from some suppliers. Such water, labelled HPLC (high-pressure liquid chromatogra phy) grade, is usually sufficiently free of impurities to be used in this practice.

#### 8. Preparation of dosimeters

8.1 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.

8.2 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.)

8.3 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.

NOTE 6—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.

## 9. Calibration of the mercuric nitrate solution

9.1 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<sub>2</sub>, where-upon the excess of Hg<sup>2+</sup> ions gives a violet-red coloration with the indicator diphenylcarbazone in acid medium (21).

9.2 Prepare approximately  $5 \times 10^{-4}$  mol dm<sup>-3</sup> Hg(NO<sub>3</sub>)<sub>2</sub> in acidic aqueous ethanol. First dissolve an appropriate amount of Hg(NO<sub>3</sub>)<sub>2</sub> in water acidified with sufficient HNO<sub>3</sub> to attain the concentration of the acid in the final solution, 0.05 mol dm<sup>-3</sup>. (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.)

9.2.1 Prepare standard solutions of NaCl in water. Make several concentrations to enable cross-checking. Suitable concentrations are 5 × 10<sup>-3</sup>,  $10 \times 10^{-2}$ ,  $1.5 \times 10^{-2}$ , 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<sub>8-2009</sub>

9.2.2 Prepare 0.2 mol dm<sup>-3</sup> HNO<sub>3</sub> in ethanol and 1 % ethanolic solutions 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 mol dm<sup>-3</sup> HNO<sub>3</sub> 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 7—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.

#### 10. Calibration of the dosimetry system

10.1 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 Guide 51261.

<sup>&</sup>lt;sup>5</sup> Reagent specifications are available from the American Chemical Society, 1115 16th Street, NW, Washington, DC 20036, USA.



10.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations shall be performed at a national or accredited laboratory using criteria specified in ISO/ASTM Practice 51400.

10.2.1 Specify the dose in terms of absorbed dose to water. 10.2.2 When the ECB dosimeter is used as a routine dosimeter, the calibration irradiation may be performed by irradiating the dosimeters at (*a*) a national or accredited laboratory using criteria specified in ISO/ASTM Practice 51400, (*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 that have traceability to nationally or internationally recognized standards.

NOTE 8—If the procedures outlined in Sections 5-9 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 acceptable 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.

10.2.3 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.

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

10.4 Dosimeter Measurement by Mercurimetric Titration:

10.4.1 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<sup>-3</sup> HNO<sub>3</sub> and 7 drops of DPC and titrate to the same color change as that observed during calibration of the mercuric nitrate solution.

Note 9—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.

10.5 Analysis:

10.5.1 Subtract the blank (as defined in 9.4) from the amounts of  $Hg(NO_3)_2$  solutions consumed and calculate [Cl<sup>-</sup>], the concentration of radiolytically formed Cl<sup>-</sup> ions in each dosimeter:

(equivalents of 
$$Cl^{-}$$
) = (equivalents of  $Hg^{2+}$ )×

 $\frac{(mL \text{ of titrant}) - (blank)}{mL \text{ of dosimetric solution}}$ (4)

10.5.2 Obtain a response function 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.

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

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

where:

D = absorbed dose to the dosimetric solution, Gy

 $\rho$  = density of the dosimetric solution, kg m<sup>-3</sup>

10.5.4 Published values of G (Cl<sup>-</sup>) and  $\rho$  are found in Table 1 or in the literature (**3,11,22**). The calculated radiation chemical yield should agree with the published values within the 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.

## 11. Application of dosimetry system

11.1 For use as a transfer-standard dosimeter, use a minimum of two dosimeters 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 estimated uncertainty of the dosimetry system and the required uncertainty associated with the application. Appendix X3 of ASTM Practice E 668 describes a statistical method for determining this number.

11.2 Use the measurement and analysis procedures in accordance with 10.4 and 10.5.

11.3 Determine the absorbed dose from analysis results and the response function.

11.4 Record the calculated absorbed dose values and all other relevant data as outlined in Section 12.

## 12. Minimum documentation requirements

12.1 Calibration of the Dosimetry System:

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

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

12.2 Application:

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

12.2.2 Record or reference the radiation source type and characteristics.

12.2.3 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.