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Practice for use of the ethanol-chlorobenzene dosimetry system

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

Reference number
ISO/ASTM 51538:2002(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 pilot project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this pilot project, ASTM Subcommittee E10.01, Dosimetry for Radiation Processing, 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 International Standard 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.

Annexes A1, A2 and A3 of this International Standard are for information only.

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 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 and is also used as a routine dosimetry system (see ISO/ASTM Guide 51261).

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 Annex A1 and Annex A2.

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 equal to or greater than 4 MeV **(3)** (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 4 MeV by employing thinner (in the beam direction) dosimeter containers (see ICRU Report 35). The ECB system may also be used at X-ray energies as low as 120 kVp **(4)**. In this range of photon energies the effect caused by the wall is considerable.

1.4.4 The irradiation temperature of the dosimeter should be within the range from −40°C to 80°C.

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

1.4.5 The effects of size and shape of the irradiation vessel on the response of the dosimeter can adequately be taken into account by performing the appropriate calculations using cavity theory **(5)**.

1.5 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

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

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

D 1193 Specification for Reagent Water[5]

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

E 177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods[7]

E 178 Practice for Dealing with Outlying Observations[7]

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

E 456 Terminology Relating to Quality and Statistics[7]

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

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

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

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

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

2.2 *ISO/ASTM Standards:*

[1] 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 Dosimetry for Radiation Processing, and is also under the jurisdiction of ISO/TC 85/WG 3.

Current edition approved Jan. 22, 2002. Published March 15, 2002. Originally published as E 1538-93. Last previous ASTM edition E 1538–99$^{\epsilon 1}$. ASTM E 1538–93 was adopted by ISO in 1998 with the intermediate designation ISO 15563:1998(E). The present International Standard ISO/ASTM 51538:2002(E) is a revision of ISO 15563.

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

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

[4] *Annual Book of ASTM Standards*, Vol 15.01.

[5] *Annual Book of ASTM Standards*, Vol 11.01.

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

[7] *Annual Book of ASTM Standards*, Vol 14.02.

[8] *Annual Book of ASTM Standards*, Vol 03.06.

51204 Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing[6]
51205 Practice for Use of a Ceric-Cerous Sulfate Dosimetry System[6]
51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing[6]
51400 Practice for Characterization and Performance of a High-Dose Gamma-Radiation Dosimetry Calibration Laboratory[6]
51401 Practice for Use of a Dichromate Dosimetry System[6]
51431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing[6]
51540 Practice for Use of a Radiochromic Liquid Dosimetry System[6]
51649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV[6]
51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing[6]

2.3 *ISO Standard:*[9]

ISO 11137 Sterilization of Health Care Products—Requirements for Validation and Routine Control—Radiation Sterilization

2.4 *International Commission on Radiation Units and Measurements (ICRU) Reports:*[10]

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 44 Tissue Substitutes in Radiation Dosimetry and Measurements
ICRU Report 60 Radiation Quantities and Units

3. Terminology

3.1 *Definitions:*

3.1.1 *absorbed dose, D*—quantity of ionizing radiation energy imparted per unit mass of a specified material. The SI unit of absorbed dose is the gray (Gy), where 1 gray is equivalent to the absorption of 1 joule per kilogram of the specified material (1 Gy = 1 J/kg). The mathematical relationship is the quotient of $d\bar{\epsilon}$ by dm, where $d\bar{\epsilon}$ is the mean incremental energy imparted by ionizing radiation to matter of incremental mass dm (see ICRU 60).

$$D = d\bar{\epsilon}/dm \quad (1)$$

3.1.1.1 *Discussion*—Absorbed dose is sometimes referred to simply as dose. For a photon source under conditions of charged particle equilibrium, the absorbed dose, D, may be expressed as:

$$D = \Phi \cdot E \cdot \frac{\mu_{en}}{\rho} \quad (2)$$

where:

Φ = particle fluence (particles/m^2),
E = energy of the ionizing radiation (J), and
μ_{en}/ρ = mass energy absorption coefficient (m^2/kg). If bremsstrahlung production within the specified material is negligible, the mass energy absorption coefficient (μ_{en}/ρ) is equal to the mass energy transfer coefficient (μ_{tr}/ρ), and absorbed dose is equal to kerma if, in addition, charged particle equilibrium exists.

3.1.2 *calibration*—the process whereby the response of a measuring system or measuring instrument is characterized through comparison with an appropriate standard that is traceable to and consistent with a nationally or internationally recognized standard.

3.1.3 *calibration curve*—graphical representation of the dosimetry system's response function.

3.1.4 *calibration facility*—combination of an ionizing radiation source and its associated instrumentation that provides a uniform and reproducible absorbed dose, or absorbed-dose rate traceable to national or international standards at a specified location and within a specific material, and that may be used to derive the dosimetry system's response function or calibration curve.

3.1.5 *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.

3.1.6 *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.

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

3.1.8 *measurement quality assurance plan*— a documented program for the measurement process that ensures on a continuing basis that the overall uncertainty meets the requirements of the specific application; this plan requires traceability to, and consistency with, nationally or internationally recognized standards.

3.1.9 *measurement traceability*—the ability to demonstrate by means of an unbroken chain of comparisons that a measurement is in agreement within acceptable limits of uncertainty with comparable nationally or internationally recognized standards.

3.1.10 *molar linear absorption 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:

[9] Available from International Organization for Standardization, 1 Rue de Varembé, Case Postale 56, CH-1211 Geneva 20, Switzerland.
[10] Available from the Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814, USA.

$$\epsilon_m = \frac{A_\lambda}{d} \times \frac{1}{[x]} \quad (3)$$

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

3.1.10.1 *Discussion*—The measurement is sometimes expressed in units of $L\ mol^{-1}\ cm^{-1}$).

3.1.11 *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.

3.1.12 *radiation chemical yield G(x)*— the quotient of *n(x)* by $\bar{\epsilon}$ 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.

$$G(x) = (n(x) / \bar{\epsilon}) \quad (4)$$

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

3.1.13 *reference–standard dosimeter*—a dosimeter of high metrological quality, used as a standard to provide measurements traceable to, and consistent with measurements made using primary standard dosimeters.

3.1.14 *routine dosimeter*—dosimeter calibrated against a primary-, reference-, or transfer-standard dosimeter and used for routine absorbed-dose measurement.

3.1.15 *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 ASTM 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 **(6, 7)**.

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 **(8)**.

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 **(9-12)**.

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 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.

4.5 The ECB dosimetry system may be used with other radiation types, such as neutrons **(13)**, and protons **(14)**. 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.

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 **(15)**.

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 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.[11]

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 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.

TABLE 1 Typical ECB Solution Formulations

Concentration of CB, vol %	Density at 20°C $kg \cdot m^{-3}$	Ratio of Coefficients[A]	Radiation Chemical Yields[B] ($\mu mol \cdot J^{-1}$)	
			^{60}Co Gamma Rays **(16)**	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 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.

8.2 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 6—**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 7—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 $HgCl_2$, where-upon the excess of Hg^{2+} ions gives a violet-red coloration with the indicator diphenylcarbazone in acid medium **(17)**.

9.2 Prepare approximately 5×10^{-4} mol $\cdot$ 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 $\cdot$ dm^{-3}.

NOTE 8—**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 Annex A1 and Annex A2.

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 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 *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 9—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. Calibration of the Dosimetry System

10.1 Prior to use, the dosimetry system shall be calibrated in accordance with the user's documented procedure that specifies details of the calibration process and quality assurance requirements. This calibration procedure shall be repeated at regular intervals to ensure that the accuracy of the absorbed dose measurement is maintained within required limits. Detailed calibration procedures are provided in ISO/ASTM Guide 51261.

10.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations shall be performed by irradiating the dosimeters using a calibration facility that provides an absorbed dose or an absorbed-dose rate having measurement traceability to nationally or internationally recognized standards.

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

10.3 When the ECB dosimeter is to be used as a routine dosimeter, calibration may also be performed using:

10.3.1 An in-house calibration facility that provides an absorbed dose or an absorbed-dose rate having measurement traceability to nationally or internationally recognized standards, or

10.3.2 A production or research irradiation facility together with reference or transfer–standard dosimeters that have measurement traceability to nationally or internationally recognized standards.

10.4 Specify the dose in terms of absorbed dose in water (for example, see ASTM Practice E 1026 and ISO/ASTM Practices 51205 and 51401).

10.5 Position the dosimeters in the radiation field in a defined, reproducible location **(18, 19)**.

10.6 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 10—The appropriate thickness of such material depends on the energy of the radiation (see ASTM 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.

10.7 When using an electron beam for irradiation, locate the dosimeters in a well-characterized position within the radiation field **(20)**. In addition, it may be necessary to surround the dosimeter(s) with water-equivalent material (see ISO/ASTM Guide 51261, subclause 8.8.3.6).

10.8 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 %.

10.9 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. Measurements of the temperature dependence of dosimeter response during irradiation between 20 and 80°C are found in Ref **(21)**, and between −40 and 20°C in Ref **(22)**.

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

10.11 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 11—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 13 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 13.1 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 11.2 and 12. 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.

11. Measurement

11.1 *Analytical Instrument Calibration and Performance Verification*—For the calibration of the individual instruments used in the analysis of the dosimeters, and for the verification of instrument performance between calibrations, see ISO/ASTM Guide 51261.

11.2 *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 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 12—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.

12. Analysis

12.1 Subtract the blank from the amounts of $Hg(NO_3)_2$ solutions consumed and calculate the concentration of radiolytically formed Cl^- ions in each dosimeter:

$$(\text{equivalents of } Cl^-) = (\text{equivalents of } Hg^{2+}) \times \frac{(\text{mL of titrant}) - (\text{blank})}{\text{mL of dosimetric solution}} \quad (5)$$

Calculate the absorbed dose in the dosimeter as follows:

$$D = \frac{[Cl^-]}{G(Cl^-)\rho} \quad (6)$$

The values of G (HCl) and ρ are found in Table 1 or in the literature **(3, 16)**. Other values can be derived in accordance with 10.1.

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

$$D_w = f \cdot D \quad (7)$$

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.

13. Minimum Documentation Requirements

13.1 *Calibration of the Dosimeter Response (if performed)*:

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