INTERNATIONAL STANDARD

ISO/ASTM 51538

Second edition
2009-06-15

Practice for use of the ethanol-chlorobenzene dosimetry system

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

Reference number
ISO/ASTM 51538:2009(E)

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. Neither the ISO Central Secretariat nor ASTM International accepts any liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies and ASTM members. In the unlikely event that a problem relating to it is found, please inform the ISO Central Secretariat or ASTM International at the addresses given below.

STANDARDSISO.COM : Click to view the full PDF of ISO/ASTM 51538:2009

© ISO/ASTM International 2009

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester. In the United States, such requests should be sent to ASTM International.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

ASTM International, 100 Barr Harbor Drive, PO Box C700,
West Conshohocken, PA 19428-2959, USA
Tel. +610 832 9634
Fax +610 832 9635
E-mail khooper@astm.org
Web www.astm.org

Published in the United States

Contents

Page

1 Scope 1
2 Referenced documents 1
3 Terminology 2
4 Significance and use 2
5 Interferences 3
6 Apparatus 3
7 Reagents 4
8 Preparation of dosimeters 4
9 Calibration of the mercuric nitrate solution 4
10 Calibration of the dosimetry system 4
11 Application of dosimetry system 5
12 Minimum documentation requirements 5
13 Measurement Uncertainty 6
14 Keywords 6
Annexes 7
Bibliography 10
Table 1 Typical ECB solution formulations 3
Table 2 Temperature coefficients k $(^\circ C)^{-1}$ for typical ECB solution formulations 3
Table A3.1 Characteristics of some applicable methods 9
Table A3.2 Some suppliers of readout instruments suitable for use with Ethanol-Chlorobenzene (ECB) dosimetry 10
Table A3.3 Some suppliers of Ethanol-Chlorobenzene (ECB) dosimeters 10

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[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 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.

1.3 This practice applies provided the following conditions 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**).[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^{-1}(**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 ^{60}Co 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°C to 80°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**).

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.* Specific warnings are given in 8.2 and 9.2.

2. Referenced documents

2.1 *ASTM Standards:*[3]

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

2.2 *ISO/ASTM Standards:*[3]

51261 Guide for Selection and Calibration of Dosimetry

---

[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 Radiation Processing: Dosimetry and Applications, and is also under the jurisdiction of ISO/TC 85/WG 3.

Current edition approved June 18, 2008. Published June 2009. Originally published as ASTM E 1538-93. Last previous ASTM edition E 1538–99. ASTM E 1538–93 was adopted by ISO in 1998 with the intermediate designation ISO 15563:1998(E). The present International Standard ISO/ASTM 51538:2009 (E) is a major revision of ISO/ASTM 51538:2002(E), which replaced ISO 15563.

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

[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.

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:*[4]

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 indicated by a measuring instrument or measuring system, and the corresponding values realised by standards traceable to a nationally or internationally recognized laboratory.

3.1.1.1 *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. 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* ε_m—constant relating the spectrophotometric absorbance, A_λ, of an optically absorbing molecular species at a given wavelength, λ, 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: $m^2\ mol^{-1}$)

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, $\bar{\varepsilon}$ imparted to the matter.

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

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

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.

3.1.11 *traceability*—property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties.

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 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)**.

---

[4] 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°C and 80°C at any chlorobenzene concentration can be described by a simple exponential expression:

$$G_t = G_0\, exp[k(t - 20)] \tag{3}$$

where:

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

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

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

4.3.2 The values of k are given in Table 2.

TABLE 1 Typical ECB solution formulations

Concentration of CB, vol %	Density at 20°C kg · m^{-3}	Ratio of Coefficients[A]	Radiation Chemical Yields at 20°C[B] (μmol· J^{-1})	
			^{60}Co Gamma Rays **(11)**	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 % acetone and 0.04 % benzene.

4.3.3 Between −30°C and 50°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 (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 **(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.

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 **(20)**.

6. Apparatus

6.1 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.

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 ± 2°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)$^{-1}$ 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.[5]

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 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 $HgCl_2$, whereupon the excess of Hg^{2+} ions gives a violet-red coloration with the indicator diphenylcarbazone in acid medium **(21)**.

9.2 Prepare approximately 5×10^{-4} mol dm^{-3} $Hg(NO_3)_2$ in acidic aqueous ethanol. First dissolve an appropriate amount of $Hg(NO_3)_2$ in water acidified with sufficient HNO_3 to attain the concentration of the acid in the final solution, 0.05 mol dm^{-3}. (**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^{-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.

9.2.2 Prepare 0.2 mol 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 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.

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.

---

[5] 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^{-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 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 $[Cl^-]$, the concentration of radiolytically formed Cl^- ions in each dosimeter:

$$(\text{equivalents of Cl}^-) = (\text{equivalents of Hg}^{2+}) \times \frac{(\text{mL of titrant}) - (\text{blank})}{\text{mL of dosimetric solution}} \quad (4)$$

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:

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

where:

D = absorbed dose to the dosimetric solution, Gy

ρ = density of the dosimetric solution, kg m^{-3}

10.5.4 Published values of G (Cl^-) and ρ 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.

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

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

13. Measurement Uncertainty

13.1 To be meaningful, a measurement of absorbed dose shall be accompanied by an estimate of uncertainty.

13.2 Components of uncertainty shall be identified as belonging to one of two categories:

13.2.1 *Type A*—those evaluated by statistical methods, or

13.2.2 *Type B*—those evaluated by other means.

13.3 Other ways of categorizing uncertainty have been widely used and may be useful for reporting uncertainty. For example, the terms *precision* and *bias* or *random* and *systematic* (non-random) are used to describe different categories of uncertainty.

13.4 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) **(22)**.

NOTE 10—The identification of Type A and Type B uncertainties is based on the methodology published in 1995 by the International Organization for Standardization (ISO) in the Guide to the Expression of Uncertainty in Measurement **(23)**. The purpose of using this kind of characterization is to promote an understanding of how uncertainty statements are arrived at and to provide a basis for the international comparison of measurement results.

NOTE 11—ISO/ASTM Guide 51707 defines possible sources of error in dosimetry performed in radiation processing facilities and offers procedures for estimating the magnitude of the resulting uncertainties in the measurement of absorbed dose using a dosimetry system. The document defines and discusses basic concepts of measurement, including estimation of the measured value of a quantity, "true" value, error and uncertainty. Components of uncertainty are discussed and methods are provided for estimating their values. Methods are also given for calculating the combined standard uncertainty and estimating expanded (overall) uncertainty.

14. Keywords

14.1 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 E 275, E 925, and E 958.

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) (formerly the National Bureau of Standards).

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 **(24)**.

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 10.2 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 **(24)**.

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 Obtain a response function 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.

A1.3.4.3 The basic equation for calculation of the radiation chemical yield for the dosimetric solution is as follows:

$$G = \Delta A v / \rho \varepsilon D_D d \qquad \text{(A1.2)}$$

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 = 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.

A1.3.4.4 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 $\varepsilon = 399\ m^2 \cdot mol^{-1}$ at 485-nm wavelength, Eq A1.2 reduces to:

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

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 **(25, 26)**.

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 10.2 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 (<± 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*[6]—To establish the calibration curve, that is, the relationship between absorbed dose and instrument deflection, 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 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 or the response function (for example, 3rd order polynomial) the absorbed dose is evaluated.

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).

---

[6] 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.