Designation: E 1538 – 99$^{\epsilon 1}$

An American National Standard

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

This standard is issued under the fixed designation E 1538; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ϵ^1 NOTE—Footnote 1 was editorially altered in June 1999.

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 Guide E 1261).

1.2 This practice describes the titration analysis as a standard readout procedure for the ECB dosimeter. Other applicable readout methods (spectrophotometric, oscillometric) are described in Appendix X1 and Appendix X2.

1.3 This practice applies only to gamma rays, X rays, and high-energy electrons.

1.4 This practice applies provided the following are satisfied:

1.4.1 The absorbed dose range shall be from 10 Gy to 2 MGy **(1)**.[2]

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

1.4.3 For radionuclide gamma-ray sources, the initial photon energy shall be greater than 0.6 MeV. For bremsstrahlung photons, the initial energy of the electrons used to produce the bremsstrahlung photons shall be equal to or greater than 2 MeV. For electron beams, the initial electron energy shall be 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]

[1] This practice is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.01 on Dosimetry for Radiation Processing.

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

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

[4] *Annual Book of ASTM Standards*, 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 14.01.

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]
E 1204 Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing[6]
E 1205 Practice for Use of a Ceric-Cerous Sulfate Dosimetry System[6]
E 1261 Guide for the Selection and Application of Dosimetry Systems for Radiation Processing of Food[6]
E 1400 Practice for Characterization and Performance of a High-Dose Gamma-Radiation Dosimetry Calibration Laboratory[6]
E 1401 Practice for Use of a Dichromate Dosimetry System[6]
E 1431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing[6]
E 1540 Practice for Use of a Radiochromic Liquid Dosimetry System[6]
E 1649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV[6]
E 1707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing[6]

2.2 *ISO Standard:*[9]

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

2.3 *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 33—Radiation Quantities and Units
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

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 33).

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

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

[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.

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:

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

(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.

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

(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 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 Practices E 666 and E 668 and Guide E 1261.

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 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