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Standard Practice for Use of the Alanine-EPR Dosimetry System[1]

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

1. Scope

1.1 This practice covers materials description, dosimeter preparation, instrumentation, and procedures for using the alanine-EPR dosimetry system for measuring the absorbed dose in materials irradiated with photons and electrons. The system is based on electron paramagnetic resonance (EPR) spectroscopy of free radicals derived from the amino acid alanine.[2] It is classified as a reference standard dosimetry system (see Guide E 1261).

1.2 This practice covers alanine-EPR dosimetry systems for dose measurements under the following conditions:

1.2.1 The absorbed dose range is between 1 and 10^5 Gy.

1.2.2 The absorbed dose rate is up to 10^2 Gy s^{-1} for continuous radiation fields and up to 5×10^7 Gy s^{-1} for pulsed radiation fields **(1-3)**.[3]

1.2.3 The radiation energy for photons and electrons is between 0.1 and 28 MeV **(1, 2, 4)**.

1.2.4 The irradiation temperature is between − 60 and + 90°C **(2, 5)**.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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:*

E 170 Terminology Relating to Radiation Measurements and Dosimetry[4]

E 178 Practice for Dealing with Outlying Observations[5]

E 456 Terminology Relating to Quality and Statistics[5]

E 668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices[4]

E 1204 Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing[4]

E 1261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing[4]

E 1400 Practice for Characterization and Performance of a High-Dose Gamma Radiation Dosimetry Calibration Laboratory[4]

E 1431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing[4]

E 1707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing[4]

2.2 *ICRU Reports:*[6]

ICRU Report 14 Radiation Dosimetry: X-Rays and Gamma-Rays with Maximum Photon Energies Between 0.6 and 50 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: Electron Beams with Energies between 1 and 50 MeV

ICRU Report 37 Stopping Powers for Electrons and Positrons

ICRU Report 44 Tissue Substitutes in Radiation Dosimetry and Measurement

2.3 *ISO Standard:*

Guide for the Expression of Uncertainty in Measurements[7]

3. Terminology

3.1 *Definitions*—Appropriate terms may be found in Terminology E 170.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *alanine dosimeter*—a specified quantity and physical

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[2] The term "electron spin resonance" (ESR) is used interchangeably with electron paramagnetic resonance (EPR).

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

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

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

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

[7] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

form of the radiation-sensitive material alanine and any added inert substance such as a binder.

3.2.2 *alanine-EPR dosimetry system*—a system used for determining absorbed dose, consisting of the alanine dosimeters, an EPR spectrometer, the calibration curve, reference standards, and procedures for the system's use.

3.2.3 *EPR spectroscopy*—the measurement of resonant absorption of electromagnetic energy resulting from the transition of unpaired electrons between different energy levels, upon application of radiofrequencies to a paramagnetic substance in the presence of a magnetic field.

3.2.4 *EPR spectrum*—the first derivative of the electron paramagnetic absorption spectrum as measured as a function of the magnetic field.

3.2.5 *EPR signal amplitude*—the peak-to-peak amplitude of the main signal of the EPR spectrum. This signal is proportional to the alanine-derived radical concentration in the alanine dosimeter.

3.2.6 *zero dose amplitude*—the EPR signal amplitude measurement of an unirradiated alanine dosimeter with the same EPR spectrometer parameters used for the lowest measurable absorbed dose value.

3.2.7 *calibration curve*—graphical representation of the mathematical relationship between the dosimeter EPR signal amplitude and absorbed dose, for a given type and batch of alanine dosimeters.

4. Significance and Use

4.1 The alanine-EPR dosimetry system provides a reliable means for measuring the absorbed dose. It is based on the generation of specific stable radicals in crystalline alanine by ionizing radiation.

4.2 The dosimeter contains crystalline alanine and registers the absorbed dose by an increase in the alanine-derived radical concentration. Identification and determination of the concentration of the specific alanine radical are performed by EPR spectroscopy.

4.3 Measurement of the concentration of free radicals by EPR spectroscopy is nondestructive. Alanine dosimeters can be read out repeatedly and hence can be used for archival purposes.

NOTE 1—For a comprehensive discussion of various dosimetry methods and materials applicable to the radiation types and energies discussed in this practice, see Practices E 178, E 668, E 1204, E 1400, E 1431, Guide E 1261, and ICRU Reports 14, 17, 33, 34, 35, 37, and 44.

4.4 Alanine-EPR dosimetry systems are used in industrial radiation processing, for example, sterilization of medical devices and pharmaceuticals, preservation of foods, polymer modifications, and radiation damage studies in materials, as reference or transfer standard or routine dosimetry systems.

5. Dosimeter Material

5.1 The dosimeter is prepared using α-alanine, CH_3-$CH(NH_2)$-COOH, in the form of polycrystalline powder.

5.2 Both stereoisomers of α-alanine are suitable for dosimetry; L-alanine is used most commonly.

5.3 The purity of the alanine shall be analytical grade (99 % or better). Alanine of appropriate purity is commercially available. Dopants (a specific trace amount of an element as additive) are not required.

6. Preparation of Dosimeters

6.1 The alanine dosimeter may be used in powdered form or as a solid compressed with a binder.

NOTE 2—Additives used in the preparation of dosimeters should not add any significant intrinsic or radiation-induced EPR signal. Examples of suitable binders are cellulose, ethylene-propylene rubber, gelatin, paraffin, polyethylene, polyethylene vinyl acetate, polystyrene, polyvinylpyrrolidone, polyvinyl propylene, and stearin. Lubricants added in the dosimeter manufacturing process are optional. An example of a suitable lubricant is stearic acid.

6.2 *Powder Dosimeters*:

6.2.1 Alanine powder may be used directly as supplied by the manufacturer.

NOTE 3—Sieving to achieve a narrower range of grain sizes from several tens to several hundreds of μm is recommended to improve the reproducibility of the EPR signal.

6.2.2 The alanine powder is contained in a sachet or capsule for use. From 50 to 200 mg of powder is typically used for a dosimeter.

6.3 *Dosimeters Using Binders*:

6.3.1 Alanine dosimeters can be prepared by compressing, casting, or extruding a mixture of alanine, binder, and lubricant (optional).

6.3.2 Usual physical shapes are pellets, films, cylinders, or cables. The dimensions depend on the inner diameter of the microwave cavity of the EPR spectrometer, the dosimeter holder and, the required precision of the measurement.

6.3.3 The softening point of the binder must be compatible with the temperature during radiation exposure.

6.3.4 The alanine content can vary. Some published values of the alanine content with different binders are polyvinylpyrrolidone (95 %) **(6)**, paraffin wax (80 to 90 %) **(2, 7, 8)**, polystyrene (70 %) **(9)**, ethylene-propylene rubber (67 %) **(10)**, and low-density polyethylene (60 to 90 %) **(11, 12)**. The sensitivity of the dosimeter is proportional to the alanine content.

6.3.5 The manufacturing process involves a number of operations, for example, mortaring, sieving, binder and lubricant (optional) addition, homogenization, pressing, or extruding. The introduction of radicals from even small amounts of paramagnetic material or from mechanical force must be avoided during the manufacturing process. Several fabrication techniques are described in Refs **(12)** and **(13)**.

6.4 *Preparation Quality Assurance*:

6.4.1 Care shall be exercised in conducting dosimeter preparation. Preparation shall be performed under clean laboratory conditions and with high-quality fabrication procedures as specified in the literature **(7, 14)**. Measurement repeatability, interspecimen variation, and batch sensitivity may be affected by each process step.

6.4.2 Important factors for measurement precision are homogeneity, reproducibility of mass, density, size, and shape of the dosimeters.

6.4.3 Representative sampling of dosimeters shall be performed for each production batch and subjected to quality control tests, for example, visual tests of surface conditions, impact tests, weight tests, and dimensional and density checks.

6.4.4 Dosimetric quality control for each production batch includes the mean batch sensitivity and interspecimen scattering of the zero-dose-signal dosimeter response.

6.4.5 To achieve the accuracy described in 13.2, the interspecimen variation of the radiation-induced response should be within± 1 % at a 95 % confidence level.

7. Apparatus

7.1 The following equipment and instruments are necessary to determine the radiation-induced response of the alanine-EPR dosimetry system:

7.1.1 The apparatus comprises an X-band EPR spectrometer capable of determining the alanine-derived radical concentration in a dosimeter by measurement of the EPR spectrum. A spectrometer capable of attaining the uncertainty limits described in 13.2 over the dose range of 1 to 10^5 Gy should be capable of the following settings: microwave frequency 9 to 10 GHz with automatic frequency locking (AFC); corresponding magnetic field to set a g-factor of 2.0 (at 9.8 GHz, this equals 350 mT; see Note 4) with a field scan range of 20 mT about the center field; RF modulation amplitude 0.1 to 1 mT; microwave power 0.1 to 10 mW (levelled); variable sweep time, time constant, and receiver gain according to absorbed dose. The sensitivity of the spectrometer should be at least 2×10^{11} spins/mT. The cavity should have a sample access diameter of at least 1 mm greater than the diameter of the dosimeter to be analyzed.

NOTE 4—The relationship between microwave frequency (Hz) and the magnetic field (T) is given by:

$$h\nu = g\mu_B B \quad (1)$$

where:
h = Planck's constant,
ν = microwave frequency,
g = the spectroscopic splitting factor (typically 2.0),
μ_B = the Bohr magneton, and
B = magnetic field.

7.1.2 There shall be some mechanical means of positioning the dosimeter accurately and reproducibly, in terms of both height and centricity in the cavity. The dosimeter holder is usually made of fused quartz and should be of such quality and cleanliness to contribute no interfering EPR signal.

7.2 If precise assessment of alanine dosimeter mass is required and is not provided by the manufacturer, a balance with the appropriate resolution shall be used.

8. Calibration Procedures

8.1 Calibration of the instrument and dosimeter used in the measurement of absorbed dose is a four-step process consisting of the following:

(1) Irradiation of reference alanine dosimeters;
(2) Instrument setup;
(3) Routine spectrometer performance checks; and
(4) Establishment of the calibration curve.

8.2 *Irradiation of Reference Dosimeters*—Prior to the calibration and use of the dosimetry system, the effects (if any) of temperature, humidity, absorbed dose rate, incident energy spectrum, and ultraviolet radiation on the dosimeter response shall be determined (see Section 11). These shall be taken into account during calibration and use.

8.2.1 To calibrate the alanine dosimeters, use a calibration facility that has an absorbed dose rate traceable to national standards.

8.2.2 Establish the calibration absorbed doses in terms of absorbed dose in water.

8.2.3 Absorbed dose in materials other than water may be calculated by applying conversion factors in accordance with Guide E 1261.

8.2.4 Select a location in the calibration field in which the absorbed dose rate within the volume occupied by the alanine dosimeter has been demonstrated to be uniform to within ±0.5 % **(1)**.

8.2.5 When using photons for calibration (gamma rays or bremsstrahlung), surround the alanine dosimeter with a thickness of alanine-equivalent material to achieve approximate electron equilibrium conditions.

NOTE 5—As an example, for ^{60}Co gamma-ray sources, approximately 3 to 5 mm of polystyrene, an equivalent polymeric material or alanine surrounding the alanine dosimeters in all directions, effectively approximates electron equilibrium conditions.

8.2.6 Monitor and control, if possible, environmental factors such as temperature and humidity during irradiation of the alanine dosimeters. If possible, these should be held approximately constant throughout irradiation.

8.2.7 Calibrate each batch of alanine dosimeters prior to use. Use sufficient alanine dosimeters for each absorbed dose value (see Section 9 of Practice E 668).

8.2.8 The number of sets of alanine dosimeters required to establish the calibration curve of the alanine-EPR dosimetry system depends on the dose range of utilization. Use at least one set per decade of absorbed dose in the linear range. More sets may be necessary in the non-linear range (see 10.1).

8.3 *Instrument Setup*—Follow manufacturer's procedures for the setup and instrument calibration of salient parameters, either by reading the appropriate calibration files or making the appropriate electromechanical adjustments.

8.4 *Routine Spectrometer Performance Checks*—Verify proper operation of the instrument by comparing the measurement of a suitable spin standard (which might be an irradiated alanine dosimeter stored under controlled conditions, a pitch sample, or Mn(II) in CaO). If there is not agreement within an acceptable range (±1 % at 95 % confidence), repeat the steps given in 8.2 and 8.3 to ascertain any obvious faults, for example, sample position error. Sensitivity changes > 1 % can be compensated for by normalizing the dosimeter response to the value of the spin standard.

8.5 *Establishment of Calibration Curve*:

8.5.1 Obtain EPR spectra for each dosimeter irradiated according to 8.1.

8.5.2 Use the procedure described in Section 9 to measure the EPR signal amplitude.

8.5.3 Calculate and document the mean EPR signal amplitude $\bar{k}$ *and the dosimeter standard deviation* (s_{n-1}) for each set of n dosimeters at each dose value.

NOTE 6—The sample standard deviation, s_{n-1}, is calculated from the set of n measurements ($n < 30$) as follows:

$$s_{n-1} = \sqrt{\frac{\Sigma(k_i - \bar{k})^2}{n-1}} \quad (2)$$