



Standard Practice for Use of an Alanine-EPR Dosimetry System¹

This standard is issued under the fixed designation ISO/ASTM 51607; 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 dosimeter materials, instrumentation, and procedures for using the alanine-EPR dosimetry system for measuring the absorbed dose in the photon and electron radiation processing of materials. The system is based on electron paramagnetic resonance (EPR) spectroscopy of free radicals derived from the amino acid alanine.²

1.2 The alanine dosimeter is classified as a type I dosimeter as it is affected by individual influence quantities in a well-defined way that can be expressed in terms of independent correction factors (see ASTM Practice E2628). The alanine dosimeter may be used in either a reference standard dosimetry system or in a routine dosimetry system.

1.3 This document is one of a set of standards that provides recommendations for properly implementing dosimetry in radiation processing, and describes a means of achieving compliance with the requirements of ASTM E2628 “Practice for Dosimetry in Radiation Processing” for alanine dosimetry system. It should be read in conjunction with ASTM E2628.

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

1.4.1 The absorbed dose range is between 1 and 1.5×10^5 Gy.

1.4.2 The absorbed dose rate is up to 10^2 Gy s⁻¹ for continuous radiation fields and up to 3×10^{10} Gy s⁻¹ for pulsed radiation fields **(1-4)**.³

1.4.3 The radiation energy for photons and electrons is between 0.1 and 30 MeV **(1, 2, 5-8)**.

1.4.4 The irradiation temperature is between -78 °C and +70 °C **(2, 9-12)**.

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:⁴

E170 Terminology Relating to Radiation Measurements and Dosimetry

E2628 Practice for Dosimetry in Radiation Processing

E2701 Guide for Performance Characterization of Dosimeters and Dosimetry Systems for Use in Radiation Processing

2.2 ISO/ASTM Standards:⁴

51261 Practice for Calibration of Routine Dosimetry Systems for Radiation Processing

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

2.3 ICRU Reports:⁵

ICRU Report 85a Fundamental Quantities and Units for Ionizing Radiation

ICRU Report 80 Dosimetry Systems for Use in Radiation Processing

2.4 Joint Committee for Guides in Metrology (JCGM) Reports:

JCGM 100:2008, GUM 1995, with minor corrections, Evaluation of measurement data – Guide to the Expression of Uncertainty in Measurement⁶

JCGM 100:2008, VIM, International vocabulary of metrology – Basis and general concepts and associated terms⁷

3. Terminology

3.1 Definitions:

3.1.1 *alanine dosimeter*—specified quantity and physical form of the radiation-sensitive material alanine and any added inert substance such as a binder.

3.1.2 *alanine-EPR dosimetry system*—system used for determining absorbed dose, consisting of alanine dosimeters, an EPR spectrometer and its associated reference materials, and procedures for the system’s use.

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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 bibliography at the end of this standard.

⁴ 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.

⁵ Available from International Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814, U.S.A.

⁶ Document produced by Working Group 1 of the Joint Committee for Guides in Metrology (JCGM/WG 1). Available free of charge at the BIPM website (<http://www.bipm.org>).

⁷ Document produced by Working Group 2 of the Joint Committee for Guides in Metrology (JCGM/WG 2). Available free of charge at the BIPM website (<http://www.bipm.org>).

3.1.3 *alanine-EPR dosimeter response*—value resulting from applied adjustments to the EPR signal amplitude.

3.1.4 *check standard*—a standard prepared independently of the calibration standards that is measured to verify the performance of a dosimetry system.

3.1.5 *EPR intensity reference material*—a stable paramagnetic material whose measurement by EPR is applied to the dosimeter EPR signal amplitude as part of the dosimeter response determination.

3.1.6 *EPR signal amplitude*—peak-to-peak amplitude of the central signal of the EPR spectrum.

3.1.6.1 *Discussion*—This signal is proportional to the alanine-derived free radical concentration in the alanine dosimeter.

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

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

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

3.2 Definitions of other terms used in this standard that pertain to radiation measurement and dosimetry may be found in ASTM Terminology E170. Definitions in E170 are compatible with ICRU Report 85a; that document, therefore, may be used as an alternative reference.

4. Significance and use

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

4.2 Alanine-EPR dosimetry systems are used in reference- or transfer-standard or routine dosimetry systems in radiation applications that include: sterilization of medical devices and pharmaceuticals, food irradiation, polymer modifications, medical therapy and radiation damage studies in materials (1, 13-15).

5. Overview

5.1 The dosimeter is prepared using α -alanine, $\text{CH}_3\text{-CH}(\text{NH}_2)\text{-COOH}$, in the form of polycrystalline powder.

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

5.3 Usual physical shapes are films or pellets (cylinders).

NOTE 1—Additives, capsules, or film support materials used in the preparation of dosimeters should not add any significant intrinsic or radiation-induced EPR signal. Examples of suitable binders are 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 (16-21).

5.4 The dosimeter contains crystalline alanine and registers the absorbed dose by the formation of alanine-derived free radicals (22). Identification and measurement of alanine-derived free radicals are performed by EPR spectroscopy. ICRU Report 80 provides information on the scientific basis and historical development of this dosimetry system.

5.5 The measurement of free radicals by EPR spectroscopy is nondestructive. This can be repeated and hence can be used for archival purposes (23-25).

6. Influence quantities

6.1 Factors other than absorbed dose which influence the dosimeter response are referred to as influence quantities, and are discussed in the following sections (see also ASTM Guide E2701). Examples of such influence quantities are temperature and dose rate.

6.2 Pre-Irradiation Conditions:

6.2.1 *Dosimeter Conditioning and Packaging*—Alanine dosimeter conditioning and packaging may be important under certain conditions (see 6.2.4).

NOTE 2—The sorting of alanine pellet dosimeters by mass into sub-lots will improve the measurement uncertainty.

6.2.2 *Time Since Manufacture*—There is no known influence of time since manufacture on alanine dosimeters when stored under recommended conditions.

6.2.3 *Temperature*—There is no known influence of pre-irradiation temperature. However, it is recommended that alanine dosimeters be stored at manufacturer recommended temperatures. Exposure to temperatures outside the manufacturer's recommended range should be avoided to reduce the potential for adverse effects on dosimeter response.

6.2.4 *Relative Humidity*—The humidity during pre-irradiation storage may influence the EPR signal amplitude of alanine dosimeters (24, 25). The effect of humidity may be reduced by sealing dosimeters in a material impervious to water.

6.2.5 *Exposure to Light*—There is no known influence of ambient light.

6.3 Conditions During Irradiation:

6.3.1 *Irradiation Temperature*—The irradiation temperature influences the EPR signal amplitude of alanine dosimeters.

NOTE 3—The effect of irradiation temperature on the dosimeter EPR signal amplitude may be dependent on the dosimeter type. The temperature coefficient, R_t (K^{-1}) is described by the relationship, $(\Delta m/m)/\Delta T$, where m is the EPR signal amplitude (in arbitrary units) and T is the irradiation temperature (in K). For dosimeters with L-alanine, a positive temperature coefficient, expressed in percent, in the range of +0.1 to +0.2 % $^\circ\text{C}^{-1}$ is typical for irradiation temperatures from -10°C to $+70^\circ\text{C}$ (10, 11, 26-28); refer to Ref (9, 12) for irradiation temperatures below -10°C . The temperature coefficient for dosimeters prepared with the DL stereoisomer of alanine is more than 50 % higher than one prepared with L-alanine (29). A summary of published temperature coefficients is tabulated in Ref (26, 29).

6.3.2 *Absorbed-Dose Rate*—Under normal radiation processing conditions there is no measurable effect of absorbed dose rate; however, a dose dependent effect has been characterized for alanine dosimeters irradiated to high doses at low dose rates (30).

NOTE 4—The dose-rate effect is absorbed-dose dependent. Alanine dosimeters irradiated with gamma radiation to absorbed doses > 5 kGy at low dose rates (< 2 Gy/s) show a progressive decrease in EPR signal amplitudes relative to that found at dose rates greater than 2 Gy/s (30). This combined dose/dose rate effect may reach several percent and is irradiation temperature dependent; though relatively constant above 0 °C, no rate effect was measured at –10 °C and –40 °C (31).

6.3.3 *Dose Fractionation*—There is no known influence of dose fractionation.

NOTE 5—In some instances the fractionation of dose to alanine dosimeters may not be straightforward. Certain influence quantities that contribute to the dosimeter response may not be equivalent for the fractionated and non-fractionated irradiations. For example, the fractionation of dose imposes multiple temperature changes to the dosimeter that may not be equivalent to the irradiation temperature experienced by a dosimeter irradiated to a single dose (equal to the sum of the fractionated doses). An accurate comparison of fractionated and non-fractionated doses will depend greatly on an accurate knowledge of the irradiation temperature for the irradiations (see 6.3.1).

6.3.4 *Relative Humidity*—The humidity during irradiation may influence the EPR signal amplitude of alanine dosimeters. The effect of humidity may be reduced by sealing dosimeters in a material impervious to water.

6.3.5 *Exposure to Light*—There is no known influence of ambient light.

6.3.6 *Radiation Energy*—For most radiation processing applications there is no influence of radiation energy for photons and electrons.

NOTE 6—Differences have been reported between the absorbed dose to water response of alanine dosimeters irradiated by photons and electrons over a range of energies (4, 6-8). The response in electron beams has been reported to be 1–2 % lower than in Co-60 beams (8) and the response in 150 kV X-ray beams has been reported to be ~15 % lower (7). The response to ~100 keV electrons was found to be equivalent to the response to high energy electrons (32, 33).

6.4 *Post-Irradiation Conditions:*

6.4.1 *Time*—The interval between irradiation and dosimeter reading shall be standardized and should conform to the manufacturer's recommendations (see 6.4.4). Alanine dosimeters are commonly regarded as stable over time periods as long as weeks or months. However, the degree of stability may be influenced by, but not limited to, absorbed dose, relative humidity, dosimeter composition and this should be characterized by the end user.

6.4.2 *Temperature*—There is no known influence of storage temperature on alanine dosimeters. However, it is recommended that alanine dosimeters be stored according to manufacturer's recommendations.

6.4.3 *Conditioning Treatment*—Post-irradiation treatment is not applicable.

6.4.4 *Storage Relative Humidity*—The humidity during post-irradiation storage can influence the EPR signal amplitude of alanine dosimeters. Sufficient time should be allowed for dosimeters to equilibrate with ambient conditions before measurement (25).

6.4.5 *Exposure to Light*—There is no significant influence of ambient light.

6.5 *Response Measurement Conditions:*

6.5.1 *Exposure to Light*—There is no significant influence of ambient light.

6.5.2 *Temperature*—Controlled temperatures are recommended for measuring alanine dosimeters. Avoid exposure to temperatures outside the manufacturer's recommended range.

6.5.3 *Relative Humidity*—The humidity during measurement can influence the EPR signal amplitude of alanine dosimeters. During measurement, the effects of humidity can be compensated by measuring the ratio of the alanine signal to that of a humidity insensitive EPR intensity reference material (see 7.3.1). If a humidity sensitive EPR reference material is used (e.g. an irradiation-calibrated alanine dosimeter) compensate for changes in humidity by standardizing the time of measurement (see 6.4.4) between the reference and dosimeter reading (see Note 8).

NOTE 7—Some commercial EPR instrumentation may automatically compensate for nominal changes in temperature and ambient humidity.

NOTE 8—The historical data for humidity effects on alanine dosimeters and quantitative EPR measurements have been compiled (25, 34-36).

7. Dosimetry system and its verification

7.1 The following are components of the Alanine-EPR Dosimetry System:

7.1.1 Alanine Dosimeters.

7.1.2 EPR Spectrometer.

7.1.2.1 An X-band EPR spectrometer is used to measure the EPR signal amplitude of an alanine dosimeter. To obtain the expanded uncertainty cited in 12.3, an EPR spectrometer should be capable of the following settings:

(1) microwave frequency 9 to 10 GHz with automatic frequency locking (AFC);

(2) corresponding magnetic field to set a g-factor of 2.0 (at 9.8 GHz, this equals 350 mT) with a field scan range of 20 mT about the center field;

(3) magnetic field modulation amplitude 0.1 to 1.5 mT;

(4) microwave power 0.1 to 10 mW (leveled);

(5) adjustable sweep time, time constant, and receiver gain according to absorbed dose.

7.1.2.2 The sensitivity of the spectrometer should be at least 2×10^{11} spins for EPR line width of 0.1 mT (37).

7.1.3 Dosimeter Holder.

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

7.1.4 Analytical Balance (Optional).

7.1.4.1 For certain types of dosimeters, the measurement reproducibility may be improved by normalizing the EPR signal amplitude to the dosimeter mass. To attain the uncertainty cited in 12.3, an analytical balance capable of measuring masses to within ± 0.1 mg should be used. The analytical balance shall be calibrated according to the manufacturer's guidelines.

7.2 *Measurement Management System:*



7.2.1 The measurement management system includes the dosimeter system calibration curve resulting from calibration according to ISO/ASTM 51261 and the procedures for use.

7.3 Performance Verification of Instrumentation:

7.3.1 EPR spectrometer performance can be verified by routinely measuring a suitable EPR intensity reference material; examples of these include Cr(III) in Al₂O₃ (ruby), or Mn(II) in CaO or MgO (34, 37). If the EPR intensity reference material does not agree with its established value within an acceptable range, ascertain any obvious faults, for example, an EPR intensity reference material position error.

NOTE 9—EPR intensity reference materials traceable to National Metrology Institutes are currently unavailable. The suitability of an EPR intensity reference material to verify and compensate for EPR spectrometer performance variation should be established either based on publication, manufacturer-supplied data, or measurement. The acceptable range for the EPR intensity reference material measurements is dependent on the measurement precision of the equipment used. Typically this is about $\pm 0.5\%$ (1 σ). Compensation for the specific performance changes may be applied if the changes are greater than the measurement uncertainty requirements.

7.4 Dosimetry System Verification:

7.4.1 Calibrated alanine dosimeters are suitable check standards for dosimetry system verification. At prescribed time intervals, and whenever there are indications of poor performance during periods of use, the performance of the dosimetry system shall be checked through the measurement of check standards, and the results documented. This information should be compared with the prescribed acceptance limits to verify adequate performance, and the result documented. Check standard measurements that measure outside of set limits must be resolved through check-standard measurement repetition, the acquisition of new check standards if necessary, or reconfiguration of spectrometer settings or dosimeter holder, or both (this reconfiguration may require a total recalibration of the dosimetry system).

NOTE 10—Drift of the dosimetry system performance that exceeds prescribed acceptance limits may result from two primary causes, a change in the check standard (e.g., signal fade) or a change in the spectrometer measurement system that includes the spectrometer electronics or its associated components, or both (e.g., dosimeter holder). The source of the system drift may be determined through the procurement of new calibrated dosimeters or the measurement of in-house check standards. In-house check standards may be prepared from dosimeters irradiated in a reproducible well-characterized irradiation geometry with a stated uncertainty. The in-house check standards should be compared to the calibrated dosimeters upon initial calibration of the dosimetry system so as to provide a reference for future comparisons.

NOTE 11—Normalizing the EPR signal amplitude to the value of the EPR intensity reference material can compensate for performance changes. The effectiveness of compensation for these changes depends on the choice of EPR intensity reference material ranging from *in situ* materials (e.g., ruby) measured with the test alanine dosimeter in place, to *ex situ* materials (e.g., reference alanine dosimeter) measured in series with substitution of the test alanine dosimeter. An *in situ* EPR intensity reference material can be used to compensate for electronic instabilities internal to the EPR spectrometer that negatively affect measurement repeatability and its associated uncertainty (23, 34, 38). If the alanine dosimeters are susceptible to humidity, significant errors can be introduced when the alanine dosimeter storage humidity differs significantly from the measurement humidity (25). An *in situ* EPR intensity reference

material can also be used to compensate for environmental humidity effects during the measurement of alanine dosimeters as well as the related effect that results from alanine-dosimeter-dependent differences in moisture content (25, 34-36).

NOTE 12—If an alanine dosimeter is chosen for use as an EPR intensity reference material, environmental humidity effects can be minimized if the time of measurement (after removal from the storage environment) is held constant and the difference in the storage and environmental humidity is minimized and controlled (19). As alanine is not an *in situ* reference material, additional uncertainties should be included to compensate for unaccountable differences in relative spectrometer sensitivity resulting from removal/replacement of alanine dosimeters between measurements.

8. Incoming dosimeter stock assessment

8.1 A process shall be established for the purchase, receipt, acceptance and storage of dosimeters.

8.2 For dosimeters received, the user shall perform an incoming inspection of a representative sample to verify, for example, batch designation against the manufacturer's certification, packaging integrity (if any), and that the sample's mass range (if appropriate) are within documented specifications.

8.3 Retain sufficient dosimeters for additional investigations or for use during verification, or recalibration.

8.4 Store dosimeters according to the manufacturer's written recommendations, or as justified by published data or experience.

9. Calibration procedures

9.1 Prior to initial use of each batch of dosimeters, the dosimetry system shall be calibrated in accordance with the user's procedures, which shall detail the calibration process and quality assurance requirements in compliance with ISO/ASTM 51261.

9.2 The user's dosimetry system calibration procedures shall take into account the influence quantities associated with pre-irradiation, irradiation, and post-irradiation conditions applicable to the process in the user's facility (see Section 6).

NOTE 13—If prior experience, manufacturer's recommendations, or scientific literature (see Refs 1-39), suggest that the conditions experienced by the dosimeters are likely to influence dosimeter response and increase the uncertainties significantly, the calibration irradiation of the dosimeters should be performed under conditions similar to those in routine use (39).

9.3 Multiple calibration curves may be required to accommodate particular dose ranges or post-irradiation measurement intervals.

10. Routine use

10.1 Pre-Irradiation:

10.1.1 Store alanine dosimeters as recommended by the manufacturer.

10.1.2 Inspect each dosimeter or dosimeter package for external imperfections. Discard any that show unacceptable imperfections. Handle alanine dosimeters with suitable care to avoid physical damage.

10.1.3 Identify each dosimeter appropriately in terms of batch and number.

10.2 Post-Irradiation Analysis Procedure:

10.2.1 Retrieve the dosimeters.



10.2.2 Maintain the dosimeters in their original packaging, if applicable, in an approved location under specified conditions prior to measurement. See 6.4 and 6.5.

10.2.3 The dosimeters should be measured within a specified time interval (see 6.4.1 and 6.4.4) and under conditions (6.5) that take account of potential post-irradiation changes (23, 25).

10.2.4 Place the alanine dosimeter precisely within the holder inside the microwave cavity of the EPR spectrometer.

NOTE 14—EPR signal amplitude measurements from repeated positioning of the same dosimeter should show agreement within $\pm 0.5\%$ (1σ).

NOTE 15—The EPR signal amplitude is dependent on the orientation of the dosimeter along its cylindrical axis; typically this dependence is less than 0.5% (1σ).

10.2.5 Record the EPR spectrum of the alanine dosimeter.

10.2.6 Determine the EPR signal amplitude of each alanine dosimeter after irradiation.

10.2.6.1 The amplitude is measured in terms of relative units and its measurement can be performed manually or automatically.

NOTE 16—Other corrections may be necessary, for example it may be necessary to subtract the zero-dose amplitude from the measured EPR amplitude, depending on their relative magnitudes and the desired precision of measurement (40).

10.2.6.2 An estimate of the dosimeter temperature during irradiation can be used to correct for its influence on the dosimeter EPR signal amplitude as necessary (see 6.3.1).

10.2.6.3 Normalizing the EPR amplitude to the dosimeter mass for alanine pellet dosimeters can be used to improve the precision.

NOTE 17—The linearity of the EPR signal amplitude with dosimeter mass may have to be established, and a mass correction applied, depending on the dosimeter type and desired precision of measurement.

10.2.7 Normalize to EPR reference material.

NOTE 18—EPR spectrometer sensitivity changes $> 0.5\%$ can be compensated for by normalizing the dosimeter EPR signal amplitude to the value of the EPR intensity reference material (see Notes 11 and 12).

10.2.8 Determine the absorbed dose from the normalized dosimeter response and the appropriate calibration curve (see Section 9).

11. Minimum documentation requirements

11.1 Record details of the measurements in accordance with the user's measurement management system.

12. Measurement uncertainty

12.1 All dose measurements need to be accompanied by an estimate of uncertainty. Appropriate procedures are recommended in ISO/ASTM 51707 and 51261 (see also GUM).

12.2 All components of uncertainty should be included in the estimate, including those arising from calibration, dosimeter variability, instrument reproducibility, and the effect of influence quantities. A full quantitative analysis of components of uncertainty is referred to as an uncertainty budget, and is then often presented in the form of a table. Typically, the uncertainty budget will identify all significant components of uncertainty, together with their methods of estimation, statistical distributions and magnitudes (41).

12.3 Uncertainty:

12.3.1 The estimate of the expanded uncertainty achievable with measurements made using alanine-EPR as a reference standard dosimetry system is typically of the order of $\pm 2\text{--}4\%$ for a coverage factor $k = 2$ (which corresponds approximately to a 95 % level of confidence for normally distributed data).

12.3.2 The estimate of the expanded uncertainty achievable with measurements made using alanine-EPR as a routine dosimetry system is typically of the order of $\pm 4\text{--}6\%$ for a coverage factor $k = 2$ (which corresponds approximately to a 95 % level of confidence for normally distributed data).

13. Keywords

13.1 absorbed dose; alanine dosimetry; dose measurements; dosimeter; dosimetry system; electron beam; electron paramagnetic resonance; electron spin resonance; EPR dosimeter; EPR dosimetry; ESR dosimeter; gamma radiation; ionizing radiation; irradiation; photons; radiation; radiation processing; reference standard dosimeter; X radiation; ICS 17.240

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