INTERNATIONAL STANDARD

ISO/ASTM 51607

Second edition
2004-08-15

Practice for use of an alanine-EPR dosimetry system

Pratique pour l'utilisation d'un système dosimétrique à l'alanine utilisant la résonance paramagnétique électronique

Reference number
ISO/ASTM 51607:2004(E)

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Published in the United States

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75% of the member bodies casting a vote.

ASTM International is one of the world's largest voluntary standards development organizations with global participation from affected stakeholders. ASTM technical committees follow rigorous due process balloting procedures.

A 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, Dosimetry for Radiation Processing, is responsible for the development and maintenance of these dosimetry standards with unrestricted participation and input from appropriate ISO member bodies.

Attention is drawn to the possibility that some of the elements of this 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 51607 was developed by ASTM Committee E10, Nuclear Technology and Applications, through Subcommittee E10.01, and by Technical Committee ISO/TC 85, Nuclear energy.

ISO/ASTM 51607:2004(E)

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

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 materials description, dosimeter preparation, instrumentation, and procedures for using the alanine-EPR dosimetry system for measuring the absorbed dose in the photon and electron irradiation processing of materials. 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 ISO/ASTM Guide 51261)).

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:* [4]

E 170 Terminology Relating to Radiation Measurements and Dosimetry

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

2.2 *ISO/ASTM Standards:*[4]

51204 Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing

51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing

51400 Practice for Characterization and Performance of a High-Dose Gamma Radiation Dosimetry Calibration Laboratory

51431 Practice for Dosimetry in Electron and bremsstrahlung Irradiation Facilities for Food Processing

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

2.3 *ICRU Reports:*[5]

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 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

ICRU Report 60 Fundamental Quantities and Units for Ionizing Radiation

2.4 *ISO Document:*[6]

Guide to the Expression of Uncertainty in Measurement

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.

[1] This practice is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.01 on Dosimetry for Radiation Processing, and is also under the jurisdiction of ISO/TC 85/WG 3.

Current edition approved June 30, 2004. Published August 15, 2004. Originally published as ASTM E 1607 – 94. Last previous ASTM edition E 1607 – 96$^{\epsilon 1}$. ASTM E 1607 – 94 was adopted by ISO in 1998 with the intermediate designation ISO 15566:1998(E). The present International Standard ISO/ASTM 51607:2004(E) replaces ISO 15566 and is a major revision of the last previous edition ISO/ASTM 51607–2002(E).

[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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036 or the International Organization for Standardization, 1 rue de Varembé, Case Postal 56, CH-1211, Geneva 20, Switzerland.

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

3.1.4 *EPR spectroscopy*—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.

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

3.1.6 *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 E 170. Definitions in E 170 are compatible with ICRU 60; 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 the absorbed dose. It is based on the measurement of specific stable free radicals in crystalline alanine generated by ionizing radiation.

4.2 The dosimeter contains crystalline alanine and registers the absorbed dose by the formation of alanine-derived free radicals. Identification and measurement of alanine-derived free radicals are performed by EPR spectroscopy.

4.3 The measurement 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 ASTM Practice E 668, ISO/ASTM Practices 51204, 51400, 51431, ISO/ASTM Guide 51261, and ICRU Reports 14, 17, 34, 35, 37, 44 and 60.

4.4 Alanine-EPR dosimetry systems are used as 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.

4.5 The EPR signal amplitudes of irradiated alanine dosimeters have been shown to be equivalent for photon and electron absorbed doses **(4)**.

5. Alanine characteristics

5.1 The dosimeter is prepared using α-alanine, CH_3-$CH(NH_2)$-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 The purity of the alanine shall be analytical grade (99 % or better). Alanine of this purity is commercially available.

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, 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 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 expected maximum temperature experienced by the dosimeter must be considered in relation to the softening point of the binder.

6.3.4 The alanine content can vary. Some published values of the alanine content (with various binders) are 95 % (polyvinylpyrrolidone) **(6)**, 60 to 95 % (polyethylene) **(2, 7-10)**, 70 % (polystyrene) **(11)**, and 67 % (ethylene-propylene rubber) **(12)**.

6.3.5 The manufacturing process involves a number of operations, for example, mortaring, sieving, binder and lubricant (optional) addition, homogenization, pressing, or extruding.

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, 13)**. The introduction of free 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 **(10)** and **(14)**. Measurement repeatability, batch radiation sensitivity and the related interspecimen variation may be affected by each process step.

6.4.2 Important factors for measurement precision are alanine/binder homogeneity, reproducibility of mass, density, size, and shape of the dosimeters. The environmental influences discussed in Section 11 shall be considered.

6.4.3 Representative samples of dosimeters shall be selected from each dosimeter batch and subjected to quality control

tests which may include, for example, visual inspection and mass and dimensional consistency.

6.4.4 Dosimetric quality control for each production batch includes the mean batch radiation sensitivity and the related interspecimen variation.

6.4.5 To achieve the expanded uncertainty cited in 13.4, the interspecimen variation of the radiation-induced EPR signal amplitude should be within ±1.0 % (1σ).

7. Instrumentation

7.1 An X-band EPR spectrometer is used to measure the EPR signal amplitude of an alanine dosimeter. To obtain the expanded uncertainty cited in 13.4, an EPR spectrometer 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; magnetic field modulation amplitude 0.1 to 1.5 mT; microwave power 0.1 to 10 mW (leveled); adjustable 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 microwave 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 and the magnetic field 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.

7.1.1 There shall be some mechanical means of positioning the dosimeter accurately and reproducibly, in terms of both vertical position 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 The overall precision of the dosimetry system may be improved by normalizing the EPR signal amplitude to the dosimeter mass. To attain the uncertainty cited in 13.4, an analytical balance capable of measuring masses to within ±0.1 mg should be used. The analytical balance should be calibrated according to the manufacturer's guidelines.

8. Calibration procedures

8.1 *Calibration of the Dosimetry System*:

8.1.1 Prior to use, the dosimetry system (consisting of a specific batch of dosimeters and specific measurement instruments) shall be calibrated in accordance with the user's documented procedure that specifies details of the calibration process and quality assurance requirements. This calibration process shall be repeated at regular intervals to ensure that the accuracy of the absorbed dose measurement is maintained within required limits. Calibration methods are described in ISO/ASTM Guide 51261.

8.1.2 Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations shall be performed at an accredited calibration laboratory, or at an in-house calibration facility meeting the requirements in ISO/ASTM Practice 51400, that provides an absorbed dose (or absorbed-dose rate) having measurement traceability to nationally or internationally recognized standards.

8.1.3 When the alanine dosimeter is used as a routine dosimeter, the calibration irradiation may be performed as per 8.1.2, or at a production or research irradiation facility together with reference- or transfer-standard dosimeters that have measurement traceability to nationally or internationally recognized standards.

8.1.4 *Measurement Instrument Performance Verification*—Spectrometer performance can be verified by routinely measuring a suitable EPR intensity reference material (examples include an irradiated alanine dosimeter stored under controlled conditions, a pitch sample, Cr(III) in Al_2O_3 (ruby) or Mn(II) in CaO or MgO). The EPR intensity reference material can be positioned external to the dosimeter **(15)** or incorporated into the dosimeter itself **(16)**. 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. Normalizing the EPR signal amplitude to the value of the EPR intensity reference material can compensate for performance changes.

NOTE 5—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 should be established either through 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 ± 0.5 % (1 σ). Compensation for the specific performance changes need only be applied if the changes are greater than the measurement precision requirements.

NOTE 6—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 **(17)**. In situ EPR intensity reference materials can be used to compensate for environmental humidity effects during the measurement of alanine dosimeters **(15, 16)**. 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.

9. Measurement of the EPR spectrum

9.1 The following procedures are used for obtaining and evaluating the EPR spectrum of an irradiated alanine dosimeter:

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

NOTE 7—The dosimeter should be positioned precisely in the microwave cavity. EPR signal amplitude measurements from a check of repeated positioning of the same dosimeter should show agreement within ± 0.5 % (1 σ). The EPR signal amplitude is dependent on the orientation of the dosimeter along its cylindrical axis; typically this dependence is less than 0.5 % (1 σ).

9.1.2 Measure the EPR spectrum.

9.1.3 Measure the EPR signal amplitude, *m*, of the EPR spectrum (see Fig. 1). The amplitude is measured in terms of arbitrary units and its measurement can be performed manually or automatically.

NOTE 8—The peak-to-peak procedure is faster and more precise than double integration of the EPR spectrum **(2)**.

9.1.4 The microwave power and modulation amplitude shall be held constant throughout the establishment of the calibration curve and for all unknown dosimeter measurements.

9.1.5 Normalize *m* for the following: sweep time, receiver gain, and number of sweeps, unless this is accomplished automatically by the EPR spectrometer.

NOTE 9—Other corrections may be necessary, for example: *(1)* linearity of *m* with dosimeter mass may have to be established, and a mass correction applied, depending on the dosimeter type and desired precision of measurement; *(2)* It may be necessary to subtract the zero-dose amplitude from *m*, depending on their relative magnitudes and the desired precision of measurement; and *(3)* EPR spectrometer sensitivity changes >1 % can be compensated for by normalizing the dosimeter EPR signal amplitude to the value of the EPR intensity reference material (see 8.1.4).

10. General dosimetry practice

10.1 Store alanine dosimeters as recommended by manufacturer.

10.2 Handle alanine dosimeters with suitable care to avoid physical damage.

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

10.4 The number of dosimeters required for a measurement of absorbed dose is determined by the precision of the dosimetry system and the application. Appendix X3 of ASTM Practice E 668 describes a statistical method for determining this number.

10.5 Use the irradiation and measurement procedures in accordance with Sections 8 and 9.

10.6 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 11.1).

10.7 Determine the EPR signal amplitude of each alanine dosimeter after irradiation, and evaluate the absorbed dose from the normalized EPR signal amplitude and the appropriate calibration curve.

10.8 Record the absorbed dose values and all relevant data as outlined in Section 12.

11. Environmental interferences

11.1 The irradiation temperature influences the EPR signal amplitude of alanine dosimeters.

NOTE 10—For alanine dosimeters that contain binders, the effect of irradiation temperature on the dosimeter EPR signal amplitude may be influenced by the binder type. The temperature coefficient, R_t (% °C^{-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 °C). A positive temperature coefficient in the range of +0.1 to +0.2 % °C^{-1} is typical for irradiation temperatures from −10 °C to +50 °C; refer to Ref **(5)** for irradiation temperatures below −10 °C. A summary of published temperature coefficients is tabulated in Ref **(18)**.

11.2 The humidity during pre-irradiation storage, irradiation, measurement, and post-irradiation storage can influence the EPR signal amplitude of alanine dosimeters. The effect of humidity may be reduced by sealing dosimeters in a material

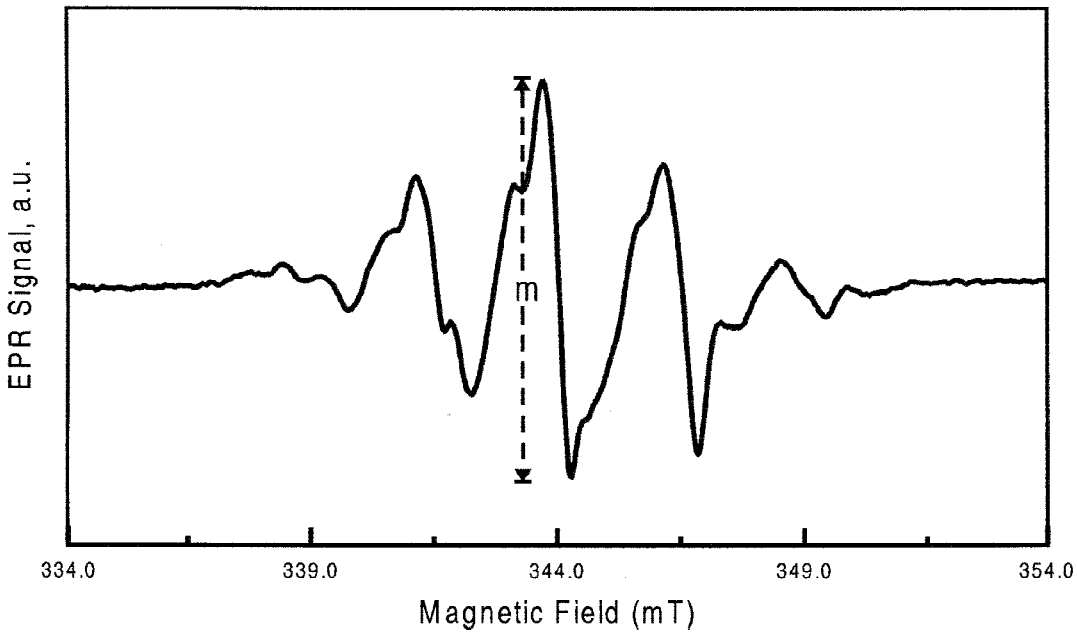

FIG. 1 EPR spectrum of an alanine dosimeter irradiated to an absorbed dose of 1 kGy; the amplitude, *m*, of the central peak is used for dose evaluation

impervious to water. During measurement, the effects of humidity can be compensated by measuring the ratio of the alanine signal to that of an EPR intensity reference material.

NOTE 11—For a summary of published information of humidity effects on alanine dosimeters, see Ref **(17)**.

11.3 For most alanine dosimeters light has little influence on the radiation-induced EPR signal amplitude. However, if the effect is not known, prolonged exposure is not recommended **(2, 19)**.

11.4 Ambient temperature and relative humidity conditions of the EPR laboratory shall be monitored, and controlled if possible, at all stages of analysis.

12. Minimum documentation requirements

12.1 *Calibration*:

12.1.1 Record the type, batch number, and manufacturer of the alanine dosimeters.

12.1.2 Record or reference the date and temperature of irradiation, dose range, radiation source, and associated instrumentation used to calibrate the alanine-EPR dosimetry system.

12.2 *Use*:

12.2.1 Record the date of irradiation and date of EPR measurement for each dosimeter.

12.2.2 Record the estimated or measured irradiation temperature, applied temperature correction, and resulting absorbed dose for each dosimeter. Reference the calibration curve used to obtain the absorbed dose values.

12.2.3 Record or reference the radiation source type characteristics.

12.2.4 Record the EPR signal amplitude and relevant EPR spectrometer settings (microwave frequency, microwave power, magnetic field strength and sweep width, modulation amplitude, and gain factor settings).

12.2.5 Record or reference the components of the measurement uncertainty associated with the absorbed dose value (see Section 13).

12.2.6 Record or reference the measurement quality assurance plan used for the alanine-EPR dosimetry system application.

12.2.7 Record and/or control the temperature and relative humidity conditions during storage, irradiation, and analysis. Use of an instrument with an EPR intensity reference material compensates for humidity conditions and makes recording of relative humidity optional.

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 approximately 3 % for a coverage factor $k = 2$ (which corresponds approximately to a 95 % level of confidence for normally distributed data).

NOTE 12—The identification of Type A and Type B uncertainties is based on methodology for estimating uncertainty published in 1995 by the International Organization for Standardization (ISO) in the Guide to the Expression of Uncertainty in Measurement **(20)**. The purpose of using this type of characterization is to promote an understanding of how uncertainty statements are developed and to provide a basis for the international comparison of measurement results.

NOTE 13—ISO/ASTM Guide 51707 defines possible sources of uncertainty 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 provided for calculating the combined standard uncertainty and estimating expanded (overall) uncertainty.

14. Keywords

14.1 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 rays; ICS 17.240