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Practice for calibration of routine dosimetry systems for radiation processing

Practique d'étalonnage des appareils de mesure dosimétrique routinier pour le traitement par irradiation

Reference number
ISO/ASTM 51261:2013(E)

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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 pilot project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this pilot project, ASTM Committee E61, 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 51261 was developed by ASTM Committee E61, Radiation Processing, through Subcommittee E61.01, Dosimetry, and by Technical Committee ISO/TC 85, Nuclear energy, nuclear technologies and radiological protection.

ISO/ASTM 51261:2013(E)

An American National Standard

Standard Practice for Calibration of Routine Dosimetry Systems for Radiation Processing[1]

This standard is issued under the fixed designation ISO/ASTM 51261; 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 specifies the requirements for calibrating routine dosimetry systems for use in radiation processing, including establishing measurement traceability and estimating uncertainty in the measured dose using the calibrated dosimetry system.

NOTE 1—Regulations or other directives exist in many countries that govern certain radiation processing applications such as sterilization of healthcare products and radiation processing of food requiring that absorbed-dose measurements be traceable to national or international standards (ISO 11137-1, Refs (**1-3**)[2]).

1.2 The absorbed-dose range covered is up to 1 MGy.

1.3 The radiation types covered are photons and electrons with energies from 80 keV to 25 MeV.

1.4 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 the calibration of routine dosimetry systems. It is intended to be read in conjunction with ASTM E2628 and the relevant ASTM or ISO/ASTM standard practice for the dosimetry system being calibrated referenced in Section 2.

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:*[3]

E170 Terminology Relating to Radiation Measurements and Dosimetry

E178 Practice for Dealing With Outlying Observations

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:*[3]

51607 Practice for Use of an Alanine-EPR Dosimetry System

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

2.3 *International Commission on Radiation Units and Measurements Reports:*[4]

ICRU Report 85a Fundamental Quantities and Units for Ionizing Radiation

2.4 *ISO Standards:*[5]

ISO 11137-1 Sterilization of health care products—Radiation—Requirements for the development, validation and routine control of a sterilization process for medical devices

2.5 *ISO/IEC Standards:*[5]

17025 General Requirements for the Competence of Testing and Calibration Laboratories

2.6 *Joint Committee for Guides in Metrology (JCGM) Reports:*[6]

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

3. Terminology

3.1 *Definitions:*

3.1.1 *approved laboratory*—laboratory that is a recognized national metrology institute; or has been formally accredited to ISO/IEC 17025; or has a quality system consistent with the requirements of ISO/IEC 17025.

3.1.1.1 *Discussion*—A recognized national metrology institute or other calibration laboratory accredited to ISO/IEC 17025 should be used in order to ensure traceability to a national or international standard. A calibration certificate

[1] This guide is under the jurisdiction of ASTM Committee E61 on Radiation Processing and is the direct responsibility of Subcommittee E61.01 on Dosimetry, and is also under the jurisdiction of ISO/TC 85/WG 3.

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

[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.

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

[5] Available from International Organization for Standardization (ISO), 1, ch. de la Voie-Creuse, Case postale 56, CH-1211, Geneva 20, Switzerland, http://www.iso.ch.

[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).

provided by a laboratory not having formal recognition or accreditation will not necessarily be proof of traceability to a national or international standard.

3.1.2 *calibration*—set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.

3.1.3 *calibration curve*—expression of the relation between indication and the corresponding measured quantity value.

3.1.4 *charged-particle equilibrium* (referred to as *electron equilibrium* in the case of electrons set in motion by photon beam irradiation of a material)—condition in which the kinetic energy of charged particles (or electrons), excluding rest mass, entering an infinitesimal volume of the irradiated material equals the kinetic energy of charged particles (or electrons) emerging from it.

3.1.5 *dosimeter batch*—quantity of dosimeters made from a specific mass of material with uniform composition, fabricated in a single production run under controlled, consistent conditions, and having a unique identification code.

3.1.6 *dosimeter stock*—part of a dosimeter batch held by the user.

3.1.7 *dosimetry system*—system used for measuring absorbed dose, consisting of dosimeters, measurement instruments and their associated reference standards, and procedures for the system's use.

3.1.8 *electron equilibrium*—charged particle equilibrium for electrons. (See *charged-particle equilibrium*.)

3.1.9 *influence quantity*—quantity that is not the measurand but that affects the result of the measurement.

3.1.10 *in-situ/in-plant calibration*—calibration where the dosimeter irradiation is performed in the place of use of the routine dosimeters.

3.1.10.1 *Discussion*—In-situ/in-plant calibration of dosimetry systems refers to irradiation of dosimeters along with reference or transfer standard dosimeters, under operating conditions that are representative of the routine processing environment, for the purpose of developing a calibration curve for the routine dosimetry systems.

3.1.11 *measurand*—specific quantity subject to measurement.

3.1.12 *measurement management system*—set of interrelated or interacting elements necessary to achieve metrological confirmation and continual control of measurement processes.

3.1.13 *primary standard dosimetry system*—dosimetry system that is designated or widely acknowledged as having the highest metrologial qualities and whose value is accepted without reference to other standards of the same quantity.

3.1.14 *reference standard dosimetry system*—dosimetry system, generally having the highest metrological quality available at a given location or in a given organization, from which measurements made there are derived.

3.1.15 *routine dosimetry system*—dosimetry system calibrated against a reference standard dosimetry system and used for routine absorbed dose measurements, including dose mapping and process monitoring.

3.1.16 *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.

3.1.16.1 *Discussion*—Measurement traceability is a requirement of any measurement management system (see Annex A4).

3.1.17 *transfer standard dosimetry system*—dosimetry system used as an intermediary to calibrate other dosimetry systems.

3.1.18 *type I dosimeter*—dosimeter of high metrological quality, the response of which is affected by individual influence quantities in a well-defined way that can be expressed in terms of independent correction factors.

3.1.19 *type II dosimeter*—dosimeter, the response of which is affected by influence quantities in a complex way that cannot practically be expressed in terms of independent correction factors.

3.1.20 *uncertainty (of measurement)*—parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand or derived quantity.

3.1.21 *uncertainty budget*—quantitative analysis of the component terms contributing to the uncertainty of a measurement, including their statistical distribution, mathematical manipulation and summation.

3.2 *validation (of a process)*—establishment of documented evidence, which provides a high degree of assurance that a specified process will consistently produce a product meeting its predetermined specifications and quality attributes.

3.3 *verification*—confirmation by examination of objective evidence that specified requirements have been met.

3.3.1 *Discussion*—In the case of measuring equipment, the result of verification leads to a decision either to restore to service or to perform adjustments, repair, downgrade, or declare obsolete. In all cases it is required that a written trace of the verification performed be kept on the instrument's individual record.

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

4. Significance and use

4.1 Ionizing radiation is used to produce various desired effects in products. Examples of applications include the sterilization of medical products, microbial reduction, modification of polymers and electronic devices, and curing of inks, coatings, and adhesives **(4)**.

4.2 Absorbed-dose measurements, with statistical controls and documentation, are necessary to ensure that products receive the desired absorbed dose. These controls include a program that addresses requirements for calibration of routine dosimetry system.

4.3 A routine dosimetry system calibration procedure as described in this document provides the user with a dosimetry system whose dose measurements are traceable to national or international standards for the conditions of use (see Annex A4). The dosimetry system calibration is part of the user's measurement management system.

5. Dosimeter system calibration overview

5.1 Calibration of a routine dosimetry system consists of the following:

5.1.1 Selection of the calibration dosimeters from the user stock (see Section 8).

5.1.2 Irradiation of the calibration dosimeters (see 9.1 and 9.2).

5.1.3 Calibration and/or performance verification of measurement instruments (see Section 7).

5.1.4 Measurement of the calibration dosimeters response (see 9.1.6 and 9.2.5.1).

5.1.5 Analysis of the calibration dosimeter response data (see 9.1.7 and 9.2.6).

5.1.6 Verification of the calibration curve for conditions of use, if appropriate (see 9.1.8 and Note 2).

5.1.7 Estimation of the combined uncertainty for the conditions of use (see 9.1.10 and 9.2.7).

5.1.8 Verification of the calibration curve at a time other than calibration for assessment of continuing validity of the calibration curve (see 9.1.11, 9.2.9, and Note 2).

NOTE 2—Calibration verification is conducted as part of the calibration when the calibration irradiation conditions are different from the conditions of use (5.1.6). Calibration verification is also conducted between calibrations to ensure continued suitability of the calibration curve for the conditions of use (5.1.8).

5.2 *Calibration Irradiation Methods*—There are two methods for irradiating dosimeters for calibration:

5.2.1 Calibration irradiations performed at an approved laboratory followed by a calibration verification exercise.

5.2.2 In-situ/in-plant calibration irradiations of routine dosimeters along with transfer standard dosimeters issued and analyzed by an approved laboratory.

NOTE 3—Valid in-situ/in-plant calibration irradiations result in a calibration curve generated under conditions that are representative of the routine processing environment. An in-situ/in-plant calibration may not be valid or may require calibration verification if the calibration conditions can not be maintained during routine use. For example, the calibration irradiations are carried out as a single exposure, but the dosimeter is used for dose measurement of fractionated irradiations.

5.3 *Uncertainties*:

5.3.1 All measurements of absorbed dose need to be accompanied by an estimate of uncertainty (see ISO/ASTM 51707, Refs **(5,6)** and GUM).

5.3.2 All components of uncertainty should be included in the estimate, including those arising from calibration, dosimeter reproducibility, instrument stability and the effect of influence quantities. A full quantitative analysis of components of uncertainty is referred to as an *uncertainty budget* and is 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.

5.3.3 Examples of components of uncertainty in the dosimetry system calibration include inherent variation in dosimeter response, uncertainty in the calibration irradiation dose, uncertainty in the calibration curve fit and uncertainty in dosimeter response correction parameters such as dosimeter thickness, dosimeter mass, unirradiated response and irradiation temperature.

5.3.4 Additional components of uncertainty might be present when the conditions of use are different than the conditions of calibration. In these instances, a calibration verification is conducted to quantify a component of uncertainty to account for these differences (see 9.1.8 and 9.2.9).

6. Requirements for a routine dosimetry system calibration

6.1 Dosimetry system calibration shall be conducted for each new dosimeter batch.

NOTE 4—The response of different dosimeter stocks purchased at different times from a given dosimeter batch should be verified to ensure equivalent response. A statistical test should be used to determine if there is any significant difference between the stocks. This should be repeated at several doses over the calibration dose range.

6.2 Routine dosimetry systems shall be calibrated using one of the methods described in 9.1 and 9.2.

6.3 The rationale for selecting a method for calibration shall be documented (see 9.1.4 and 9.2.3).

6.4 Recalibration of an existing batch or stock shall be conducted at a frequency specified by the user based on the known characteristics of the dosimetry system.

6.4.1 Additional calibration or calibration verification may be required to determine if changes have occurred that affect the calibration. Examples are changes in the values of influence quantities, such as temperature or humidity, changes in the use of the dosimetry system and change in response due to dosimeter aging. Changes in influence quantities can result from seasonal changes in ambient conditions or changes in source activity or distribution.

6.5 Calibration curves are specific to the measurement instrument used to generate them. They shall not be used with other instruments unless it has been demonstrated that the dose measurements agree within user defined limits.

6.6 All software associated with dosimetry system and calibration data analysis shall be validated for its intended use.

7. Requirements for measurement instruments calibration and performance verification

7.1 All measurement instrumentation associated with the dosimetry system shall either be calibrated, or have its performance verified, before use. Performance checks and/or recalibration shall be carried out at user-specified intervals, based on the known characteristics of the instrument.

7.1.1 Where recognized standards exist, the calibration of the instrument shall be traceable to national or international standards.

7.1.2 Where recognized standards do not exist, the performance of the instrument shall be verified in accordance with industry or manufacturer recommended practices and procedures.

NOTE 5—For example, the Alanine-EPR dosimetry system employs electron paramagnetic resonance (EPR) spectroscopy for analysis. The proper operation of the EPR spectrometer is verified with appropriate EPR spin reference such as irradiated alanine dosimeters, pitch sample, or Mn(II) in CaO (see ISO/ASTM Practice 51607).

7.1.3 When maintenance or modification of the measurement instrumentation has occurred that may affect its performance, instrument performance shall be verified and, if necessary, the instrument shall be re-calibrated.

8. Requirements for the sampling of calibration dosimeters

8.1 Dosimeters selected for the calibration shall constitute a representative sample of the dosimeter stock held by the user to be used in routine processing. These dosimeters are referred to as 'calibration dosimeters'.

8.2 Calibration dosimeters shall be labelled to ensure segregation and identification throughout the calibration exercise.

8.3 The number of dose levels required for developing the calibration curve depends on the range of utilization. At least five dose levels shall be used for each factor of ten span of absorbed dose (for example, choose five dose levels for a 5 to 50 kGy range).

8.3.1 The minimum number of dose levels to be used in the calibration can be determined as follows: divide the maximum dose (D_{max}) of the dose range by the minimum dose (D_{min}) of the dose range; calculate log (base 10) of this ratio: $Q = \log(D_{max}/D_{min})$. If Q is equal to or greater than 1, calculate $5 \times Q$, and round this up to the nearest integer value. This value represents the minimum number of dose levels to be used. If Q is less than 1 use five dose levels.

8.4 A minimum of four dosimeters for each dose level shall be used. However, using a larger number of dosimeters per dose level may reduce the uncertainty associated with the calibration.

9. Calibration of dosimetry systems

9.1 *Calibration of Dosimetry Systems using irradiations at an approved laboratory*:

9.1.1 *Overview* —The routine dosimeter may be a Type I or Type II dosimeter. The calibration irradiation at an approved laboratory has the advantage that the dosimeters are irradiated to known doses under well-controlled and documented conditions. However, when conditions of use (in-situ/in-plant) differ from calibration conditions, significant uncertainties may be introduced in the combined uncertainty of the routine absorbed dose measurement. Transport of the dosimeters to and from the approved laboratory may also introduce uncertainties from pre- and post-irradiation influence quantities that are difficult to characterize.

9.1.2 *Post Irradiation Response*—Post-irradiation response characteristics of the routine dosimeter shall be determined prior to calibration irradiation and incorporated into the calibration procedure.

9.1.3 *Transport of Calibration Samples*—The effect of intended transportation on dosimeter response shall be evaluated to establish criteria for acceptable packaging and transportation of calibration dosimeters. The evaluation should be based on characterization data of the routine dosimetry system (see ASTM E2701).

9.1.4 *Irradiation Conditions*—A rationale shall be prepared for the calibration target dose levels, their spacing and irradiation conditions, for example, dose rate and irradiation temperature specified to the approved laboratory. Document the allowable variation from these conditions.

9.1.4.1 For example, for dose ranges of less than one decade (factor of ten), dose levels should be distributed arithmetically uniformly (for example, 10, 20, 30, 40, 50 kGy). For dose ranges of more than one decade, dose levels should be distributed geometrically uniformly (for example, 1.0, 1.5, 2.3, 3.4, 5.1, 7.6, 11.4, 17.1, 25.6, 38.4, 57.7, 86.5 kGy).

9.1.5 *Dosimeter Irradiation*—The dosimeters shall be irradiated at an approved laboratory to the specified absorbed doses. The absorbed dose is usually specified in terms of absorbed dose to water.

9.1.5.1 The approved laboratory shall report deviations from the conditions specified by the user (see 9.1.4).

9.1.6 *Dosimeter Response Measurement*—The performance of measurement instrumentation shall be verified (see 7.1).

9.1.6.1 Measure the calibration dosimeter response upon return from the approved laboratory in accordance with the users calibration and measurement procedures.

9.1.7 *Analysis of Dosimetry Data*:

9.1.7.1 If required, each dosimeter response shall be adjusted for dosimeter parameters such as dosimeter thickness, mass or unirradiated dosimeter response following established measurement practice.

9.1.7.2 The individual dosimeter response, the sample standard deviation and the coefficient of variation of the replicate measurements at each dose level shall be determined and documented.

NOTE 6—In general, if the coefficient of variation at any dose level is greater than a user-defined limit, a re-determination of the data should be considered (for example, perform a visual inspection to identify potential dosimeter damage, repeat the calibration irradiation at the dose level or perform an outlier test).

9.1.7.3 Derive the calibration curve in mathematical form, $y = f(x)$, where dosimeter response is the dependent variable (y) and absorbed dose is the independent variable (x). Choose an analytical form (for example, linear, polynomial, or exponential) that provides an appropriate fit to the measured data. The ease of deriving dose from measured dosimeter response (the mathematical inverse of the analytical form) may also be a consideration in selecting the analytical form (see Annex A2 and Annex A3).

9.1.7.3.1 The resulting calibration curve shall be evaluated for goodness of fit within user defined limits.

9.1.8 *Calibration Verification (as part of calibration)*—Prior to implementation of a calibration curve, a calibration verification shall be performed to assess the suitability of the calibration curve for the conditions of use. This is usually

achieved by in-situ/in-plant irradiation of transfer standard dosimeters supplied by an approved laboratory alongside representative samples from the routine dosimeter stock under the conditions of use. The dosimetry system being calibrated and the transfer standard dosimetry system used for calibration verification should, if possible, be based on different types of dosimeters. For example, if the dosimetry system being calibrated is based on alanine dosimeters, and the transfer standard dosimetry system is also based on alanine dosimeters, then the effect of an inappropriate correction for influence quantities, such as temperature, will not be apparent as both systems will respond in the same way.

9.1.8.1 The calibration verification shall be conducted at a minimum of three dose levels targeted near the extremes and near the center of the calibration dose range.

9.1.8.2 The routine dosimeters for the calibration verification shall be selected from the same dosimeter stock as the calibration dosimeters.

9.1.8.3 The irradiation of the routine dosimeters and transfer standard dosimeters shall consist of complete pathways through the irradiator.

9.1.8.4 The routine and transfer dosimeters shall be irradiated so that it is ensured that they receive the same dose within predetermined limits (see Annex A1).

NOTE 7—The temperatures associated with the calibration verification irradiations should be similar to those expected to be encountered during routine use of the dosimetry system.

9.1.8.5 In a few instances it may be impossible to conduct the calibration verification as described. In these instances, the user shall develop a verification method and rationale that is capable of demonstrating that the calibration curve of the routine dosimetry system is suitable for the conditions of use. The rationale for the need to use this alternative method shall be documented.

9.1.8.6 The calibration verification results shall be evaluated to identify difference between the measured dose values of the routine and transfer standard dosimetry systems and to provide an estimate of one of the components of calibration uncertainty (see Annex A3).

9.1.9 *Corrective Action*—If the calibration verification result exceeds a user defined acceptable limit, corrective action in accordance with the measurement management system shall be implemented.

9.1.9.1 Corrective action may include: repeating the calibration using more appropriate influence quantity conditions during calibration irradiation, reducing the dose range of the calibration curve, developing calibration curves for specific irradiator pathways, applying a correction factor to the routine dosimeter response in cases where a single factor is applicable over the entire calibration curve, or calibrating using an in-situ/in-plant calibration method (see 9.2).

9.1.10 *Dosimetry System Measurement Uncertainty*—Prepare an estimate of the combined uncertainty in the measured dose using the calibrated dosimetry system for the conditions of use (see Annex A3 and ISO/ASTM 51707).

9.1.11 *Stability Verification*—The suitability of the calibration curve shall be verified over its period of use in accordance with the requirements of 6.4.1.

9.2 *In-situ/In-plant Calibration of Routine Dosimetry Systems in a Production Irradiator Using Transfer Standard Dosimetry System*:

9.2.1 *Overview*—The routine dosimeter may be a Type I or Type II dosimeter. The calibration irradiation of the routine dosimeters together with the transfer standard dosimeters in the production irradiator has the advantage that the influence quantity value ranges will be very similar in routine application and calibration, provided the calibration irradiation conditions are chosen appropriately. This method takes into account the effect of the influence quantities of the conditions of use to the extent that the transfer standard dosimeter response can be corrected for the difference between the fixed influence quantity values of its calibration and the production irradiation influence quantities profile by the approved laboratory issuing and analyzing the transfer standard dosimeters. Care must be taken to ensure that the routine dosimeters and transfer standard dosimeters irradiated together receive the same absorbed dose.

9.2.2 *Post-Irradiation Response*—Post-irradiation response characteristics of the dosimeter shall be determined prior to calibration irradiation and incorporated into the calibration procedure.

9.2.3 *Irradiation Conditions*—A rationale for target dose levels and irradiation conditions for calibration irradiation shall be prepared and documented. The irradiation conditions selected for calibration irradiation should be such that the irradiation conditions are similar to those expected during the intended use of the irradiator, for example, during performance qualification and routine process monitoring.

9.2.3.1 For example, for dose ranges of less than one decade (factor of ten): dose levels should be distributed arithmetically uniformly (for example, 10, 20, 30, 40, 50 kGy). For dose ranges of more than one decade, dose levels should be distributed geometrically uniformly (for example, 1.0, 1.5, 2.3, 3.4, 5.1, 7.6, 11.4, 17.1, 25.6, 38.4, 57.7, 86.5 kGy).

9.2.4 *Dosimeter Irradiation*—The calibration dosimeters shall be irradiated with transfer standard dosimeters issued and analyzed by an approved laboratory. The irradiation phantom used to co-locate the calibration dosimeters and the transfer standard dosimeters shall be characterized to ensure both the calibration dosimeters and the transfer standard dosimeters receive the same absorbed dose (see Annex A1). The absorbed dose is usually specified in terms of absorbed dose to water.

NOTE 8—The temperatures of the routine dosimeters during calibration irradiation should be similar to those expected to be encountered during routine use of the dosimetry system.

9.2.5 *Dosimeter Response Measurement*—Verify the performance of the measurement instrumentation (see 7.1-7.1.3).

9.2.5.1 The calibration dosimeter response shall be measured in accordance with the users' calibration and measurement procedures.

9.2.6 *Analysis of Dosimetry Data*—If required, each dosimeter response shall be adjusted for response parameters such as

dosimeter thickness, mass or unirradiated dosimeter response following established measurement practice.

9.2.6.1 The individual dosimeter response, the sample standard deviation and the coefficient of variation of the replicate measurements at each dose level shall be determined and documented.

NOTE 9—In general, if any coefficient of variation is greater than a user-defined limit, a re-determination of the data should be considered (for example, perform a visual inspection to identify potential dosimeter damage, repeat the calibration irradiation at the dose level or perform an outlier test).

9.2.6.2 Derive the calibration curve in mathematical form, $y = f(x)$, where dosimeter response is the dependent variable (y) and absorbed dose is the independent variable (x). Choose an analytical form (for example, linear, polynomial, or exponential) that provides an appropriate fit to the measured data. The ease of deriving dose from measured dosimeter response (the mathematical inverse of the analytical form) may also be a consideration in selecting the analytical form (see Annex A2 and Annex A3).

9.2.6.3 The resulting calibration curve shall be evaluated for goodness of fit within user defined limits

9.2.7 *Dosimetry System Measurement Uncertainty*—Prepare an estimate of the combined uncertainty in the measured dose using the calibrated dosimetry system for the conditions of use (see Annex A3 and ISO/ASTM 51707).

9.2.8 *Corrective Action*—If the combined uncertainty exceeds a user defined acceptable limit, corrective action in accordance with the measurement management system shall be implemented.

9.2.8.1 Corrective action may include: repeating the calibration using more appropriate calibration irradiation conditions, reducing the dose range of the calibration curve, developing calibration curves for specific irradiator pathways.

9.2.9 *Stability Verification*—The suitability of the calibration curve shall be verified over the period of use in accordance with the requirements of 6.4.1.

9.2.9.1 Changes to the intended conditions of use of the routine dosimetry system may render the calibration curve unsuitable. An example of such a change is that of dose fractioning during the intended use when the calibration irradiation consists of a single exposure. In such instances, the effect of the change shall be evaluated.

NOTE 10—Performing a calibration verification is one method of evaluating the effect of changes to the conditions of intended use, reference 9.1.8 and Note 7.

10. Minimum documentation requirements

10.1 Document the dosimetry system being calibrated including the dosimeter manufacturer, type and batch number, and measurement instrumentation.

10.2 Document the rationale for the calibration method.

10.3 Document the dosimetry system calibration data, irradiation parameters, irradiation date, transfer standard dosimeters, and description of the irradiation facility used.

10.4 Document or reference a description of the radiation source(s) used in calibration and processing, including the type, nominal activity or beam parameters, and any available information on the energy spectrum.

10.5 Document irradiation temperatures and, if necessary, the relative humidity.

10.6 Document the combined uncertainty in the measured dose using the calibrated dosimetry system.

10.7 Reference the measurement management system at the radiation facility.

11. Keywords

11.1 absorbed dose; accredited laboratory; dosimeter; dosimetry system calibration; dosimetry system; electron beam; gamma radiation; ionizing radiation; measurement traceability; radiation processing; reference standard dosimetry system; routine dosimeter; transfer standard dosimetry system; Type I dosimeter; Type II dosimeter; X-ray; X-radiation

ANNEXES

(informative)

A1. PHANTOM GEOMETRY

A1.1 A phantom of known homogenous material is used for the irradiation of the dosimeters in order to minimize the difference between the absorbed doses received by the routine and transfer standard dosimeters. The phantom design should hold the two types of dosimeters so that they do not significantly influence each other and provide a geometry that is appropriate for the radiation source employed (see Fig. A1.1 and Fig. A1.2 for examples of such phantoms employed for gamma or X-ray irradiation; see Fig. A1.3 for an example of a phantom suitable for high energy electron-beam irradiation).

A1.2 The use of a phantom can result in different irradiation temperature and temperature profile than the conditions of use of the routine dosimeter without a phantom. The effect of these differences should be evaluated as part of the calibration procedure.

A1.3 When thick and thin dosimeters are irradiated together, the thin dosimeters should be surrounded by sufficient polymeric material to ensure that the attenuation characteristics are similar to the thick dosimeters and that the dosimeters receive the same dose.

A1.4 Dose variation within the phantom can be characterized by irradiating the phantom with the same type of dosimeter in all the dosimeter positions within the calibration irradiation phantom. However, difference in geometry between the routine dosimeters and transfer standard dosimeters must be taken into account.

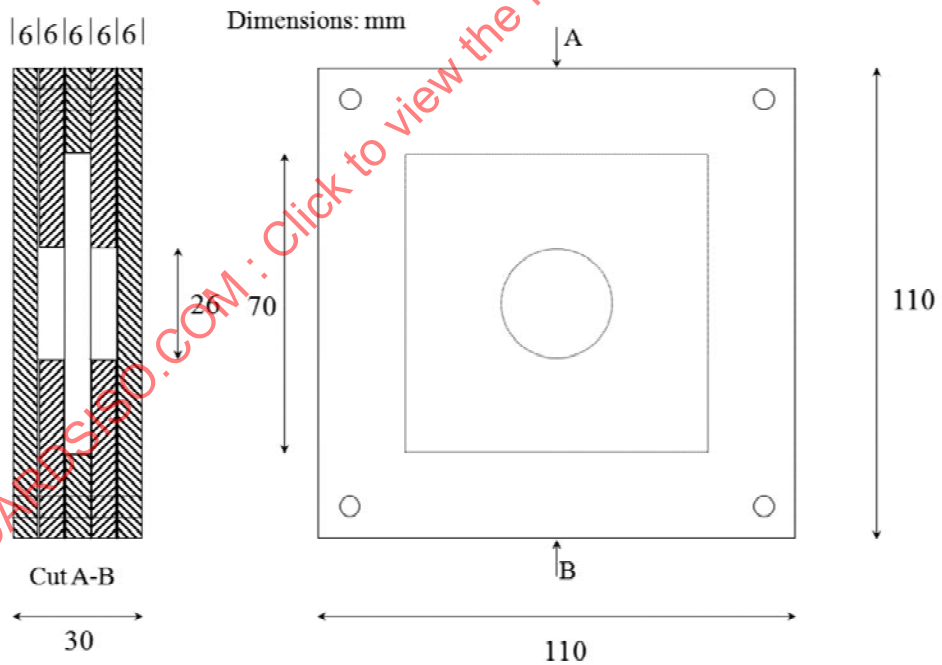

FIG. A1.1 Example of calibration phantom allowing alanine dosimeters to be placed on either side of thin film routine dosimetry system dosimeter

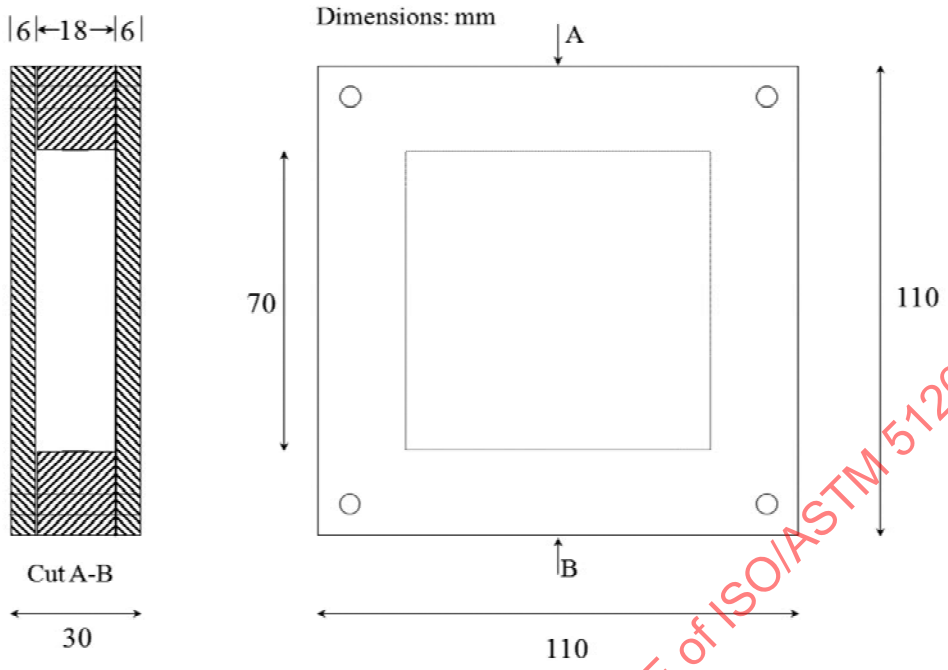

FIG. A1.2 Example of calibration phantom allowing reference standard dosimeter ampoules and routine dosimetry system dosimeter to be placed adjacent to each other

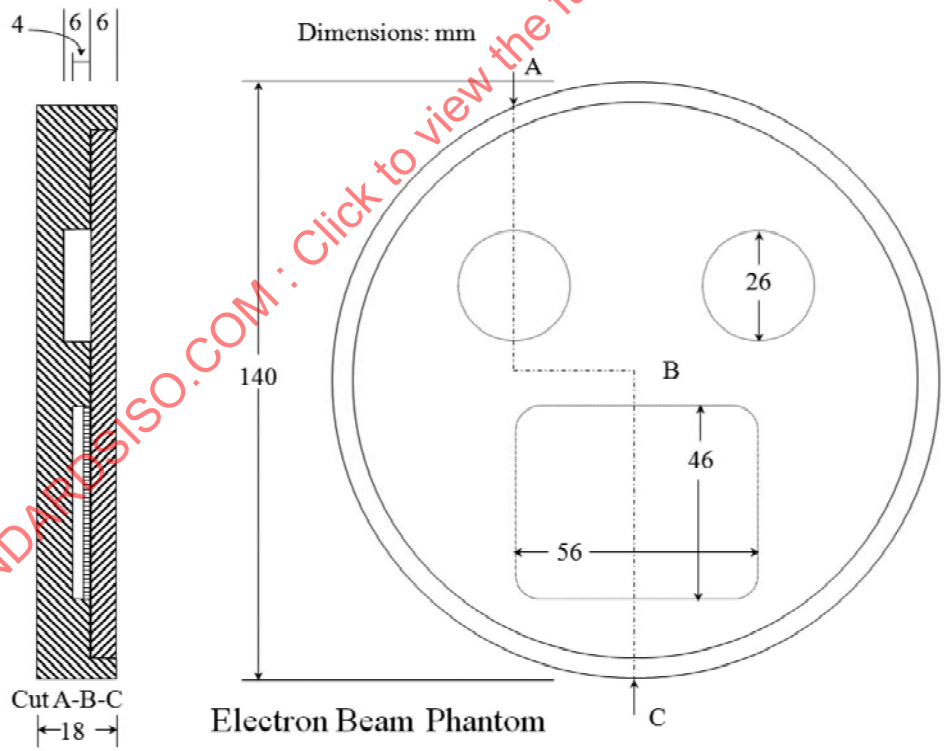

FIG. A1.3 Example of 10 MeV calibration phantom allowing alanine dosimeters and thin-film routine dosimetry system dosimeters to be irradiated at the same position on the depth-dose curve

A2. CURVE FITTING

A2.1 Curve fitting is the application of regression analysis techniques to a set of data where by the selected mathematical form (model) defines the dependent variable (Y) in terms of the independent variable (X). Regression analysis is used to fit data to a model and provide estimates of the fit parameters (coefficients) based on a minimization technique.

A2.2 Regression models are either an empirical or a mechanistic model. The empirical model describes the general shape of the data set. The parameters of the empirical model do not correspond to an underlying biological, chemical or physical process. The mechanistic model is formulated to provide insight or description of the process under study.

A2.3 The two basic types of regression analysis are linear regression and non-linear regression. Linear regression is where the unknown parameters (coefficients) appear linearly in the expression as in Eq A2.1. Non-linear regression is where the unknown parameters (coefficients) appear in a non-linear or nested fashion as in Eq A2.2.

$$y = a + bx + cx^2 + dx^3 \tag{A2.1}$$

$$y = \frac{a}{1 + \left(\frac{x}{b}\right)^c} \tag{A2.2}$$

NOTE A2.1—In the context of regression analysis, the terms linear and non-linear do not refer to the shape of the plotted curve, for example, both Eq A2.1 and Eq A2.2 represent curved plots.

A2.3.1 In both types of regression analysis (linear and nonlinear) several assumptions are made:

A2.3.1.1 X is known precisely and all error is in Y. (It is sufficient that imprecision in measuring X is very small compared to the variability in Y. Error refers to deviation from the average.)

A2.3.1.2 Variability of Y at any X follows a known distribution, typically assumed to be Gaussian or near Gaussian.

A2.3.1.3 The standard deviation of the residuals is the same along the curve (homoscedasticity).

NOTE A2.2— In some dosimetric calibration data, homoscedasticity does not exist and is corrected with the use of a weighting factor, see Eq A2.3 and Eq A2.4.

A2.3.1.4 Observations (Y) are independent (whether one point is above or below the regression analysis model curve is a matter of chance and does not influence whether another point is above or below the regression analysis model curve).

A2.4 A minimization technique is used to determine the coefficients of the regression model form that provides the best fit. The most common technique for linear fitting is a least squares algorithm which minimizes the sum of the squares of the residuals (SSE) where a residual is the vertical distance between the data point and regression model curve (reference Eq A2.3). The most common technique for non-linear fitting is the Levenberg-Marquardt algorithm. Most commercially available regression software will provide linear and non-linear regression and multiple minimization algorithms.

$$SSE = \sum_{i=1}^{n} w_i (y_i - \hat{y}_i)^2 \tag{A2.3}$$

where:

y_i = the observed dependent variable at an independent variable value,
$\hat{y}_i$ = the model predicted value of the dependent variable at the corresponding independent variable, and
w_i = assigned weight which in most cases is assumed to be 1 unless a weighting is applied to compensate for a deviation of homoscedasticity (A2.3.1.3).

NOTE A2.3—When the Gaussian distribution of error assumption is invalid due to appreciable tails in the residuals distribution, the assumption that least squares provides the maximum likelihood fit is also invalid. In these instances a robust method of minimization may be used. The essence of robust fitting is to use a minimization technique that is less influenced by potential outliers and the range of the dependent variable. Several examples of nonlinear robust minimization are Least Absolute Deviation, Lorentzian, and Pearson.

A2.5 Goodness of fit describes how well the model fits a set of data. Measures of goodness of fit typically summarize the discrepancy between observed values (y_i) and the values predicted by the model ($\hat{y}$). A review of a plot of the residuals is critical when assessing goodness of fit. The most commonly used statistics for assessing goodness of fit are the coefficient of determination, lack of fit sum of squares (F statistic), confidence intervals of the fit coefficients, and the F test when comparing fits between different models. Another powerful non-statistical evaluation method is a review of the plot of the residuals.

A2.5.1 A plot of the residuals can reveal behaviour in the data that is otherwise difficult to see in the curve fit. A plot of the residuals should not demonstrate a form or trend. A residuals plot may also indicate potential or suspect outliers (see A2.6).

A2.5.2 The coefficient of determination (r^2) has no units and ranges in value between 0 and 1 which is computed as shown in Eq A2.4. A value of 1.0 indicates the curve passes through all the data points. The coefficient of determination can be interpreted as the fraction of the total variance in y that is explained by the model. A common mistake is using the coefficient of determination solely as the gauge of goodness of fit; this may lead to the selection of a model that may fluctuate wildly with very large confidence intervals.

$$r^2 = 1 - \frac{SSE}{SSM} = 1 - \frac{\sum_{i=1}^{n} w_i (y_i - \hat{y}_i)^2}{\sum_{i=1}^{n} w_i (y_i - \bar{y}_i)^2} \tag{A2.4}$$

where:

SSE = sum of the squares of the residuals,
SSM = sum of the squares deviation about the mean,
$\bar{y}_i$ = average response at dose level i, and

w_i = assigned weight which in most cases is assumed to be 1 unless a weighting is applied to compensate for a deviation of homoscedasticity (A2.3.1.3).

A2.5.3 The *F*-statistic is a measure of the extent to which the given model represents the data. The *F*-statistic is calculated as the ratio of the mean square error of the regression to the mean square error:

$$F = \frac{MSR}{MSE} = \frac{\left(\frac{SSM - SSE}{m - 1}\right)}{\left(\frac{SSE}{DF}\right)} \quad \text{(A2.5)}$$

where:

SSM = $\sum_{i=1}^{n} w_i (y_i - \bar{y}_i)^2$
SSE = $\sum_{i=1}^{n} w_i (y_i - \bar{y}_i)^2$
m = number of coefficients fitted,
n = number of data points, and
DF = n·m

A larger *F* ratio indicates the model fits the data well.

A2.5.4 The regression analysis estimates coefficients of the model for the fit of the data. Most commercially available regression software provides an estimate of the standard error for each coefficient and the 95 % confidence interval about the coefficient estimate. The value of the standard error and the 95 % confidence interval provides a means to gauge how well the regression has determined the coefficients. If the assumptions of A2.3 are not significantly violated, the 95 % confidence interval is considered to be an interval that has a 95 % chance of containing the 'true' value of the coefficient. If the confidence intervals are wide, the coefficient has not been determined precisely. If the confidence intervals are narrow, the coefficients have been determined precisely.

A2.6 Suspect outlying observations can typically be identified from a review of the residuals plots (reference A2.5.1). Generally, a dosimetry system calibration consists of relatively few dependent replicate observations (y_i) for any given independent value (x). As a result of relatively few replicate observations, it is likely that variation in dependent response may express a value that appears to be significantly different than other observations even when the observation is from the same population with a Gaussian distribution of error. When a suspect outlier is proven to be an outlier it should be removed from the data set prior to regression analysis.

A2.6.1 Although not rigorously defined, an outlier is an observation from a population other than the population under study. Thus, a suspect outlier must be proven to come from a different population before it can be removed. An outlier then is the result of:

A2.6.1.1 An extreme observation that is part of the population under study (false discovery).

A2.6.1.2 An observation from a population other than the one under study (true discovery).

A2.6.1.3 An incorrect assumption of the population distribution of error (usually results in false discovery).

A2.6.2 Extreme observation values are probable in a Gaussian distribution of error although they are highly unlikely. Statistical outlier tests are the application of statistical inference which is based on an assumed probability distribution. Most statistical outlier tests are applied at a 95 % level of significance. This means that 5 % of the true population (either in a single-sided or double-sided test) will be identified by the statistical outlier test as significant. Unless it can be identified that the suspect observation is the result of an experimental error or the sample is in violation of criterion applied qualifying it as a viable sample, it can not be conclusively proven to come from a different population by a statistical outlier test. Although not conclusive in and of themselves, several methods are used to identify suspect outliers:

A2.6.2.1 Visual inspection of plot of the residuals (qualitative).

A2.6.2.2 Confidence Intervals (quantitative).

A2.6.2.3 Prediction Limits (quantitative).

A2.6.2.4 Statistical test such as a *t*-test (quantitative).

A2.6.3 A visual inspection of the residuals plot is a qualitative means of quickly identifying suspect outliers.

A2.6.4 Confidence intervals make use of the assumptions of linear and non-linear regression about the population distribution of the observations used to identify a measure estimate, specifically the assumption of a Gaussian distribution of error. The confidence interval is a range of values where at a specified confidence coefficient (95 or 99 %) the 'true' value exists. For regression analysis, this is an interval wherein the 'true' best fit curve lies for a specified level of confidence, for example, 95 % probability for the given model. This is not the same as inferring a 95 % confidence interval contains 95 % of the observations. Given this, a confidence interval is not a suitable measure for identifying suspect outliers.

A2.6.5 Prediction intervals, similarly to confidence intervals, assume a Gaussian distribution of error. The prediction interval describes error about the curve or scatter associated with the individual observations. In this case a 95 % prediction interval is expected to contain 95 % of the observations from the single experiment. Thus, prediction intervals are a useful tool in identifying suspect outliers. For example, a 99.9 % prediction interval would be expected to contain 99.9 % of the observations. Observations outside of this interval are then considered highly probable suspect outliers.

NOTE A2.4—A distinction between confidence intervals and prediction intervals is if the number of replicates is significantly increased, the confidence interval would become smaller while the prediction interval would not change appreciably provided the assumption of a Gaussian error distribution is valid.

A2.6.6 Statistical tests are routinely used to identify suspect outliers (see ASTM Practice E178). As identified, any statistical test in and of itself is not conclusive evidence of an outlier. The suspect outlier must be identified through investigation to be a sample from a population other than that population under study.

A2.6.7 The uncertainty of the regression curve describes the quality of the selected model and regression analysis in characterizing the relationship of the dependent and independent variables. The confidence interval about the regression curve is used to quantify the uncertainty of the curve fit. The confidence interval is not constant over the curve range and is

generally wider at the upper and lower extremes of the curve (see Fig. A3.6). The confidence interval represents an interval in which the true value of the curve exists at the identified confidence coefficient (95 or 99 %). The uncertainty of the dose estimate ($\hat{x}$) can be estimated at any single dose as the ratio of the one half the dose range defined by the confidence interval to the dose estimate ($\hat{x}$), reference Fig. A2.1 and Eq A2.6.

$$U_{\hat{x}fit}\% = \left(\frac{D_{UCL} - D_{LCL}/2}{\hat{x}}\right) \times 100 \quad \text{(A2.6)}$$

D_{UCL} = dose at the upper confidence level,
D_{LCL} = dose at the lower confidence level.

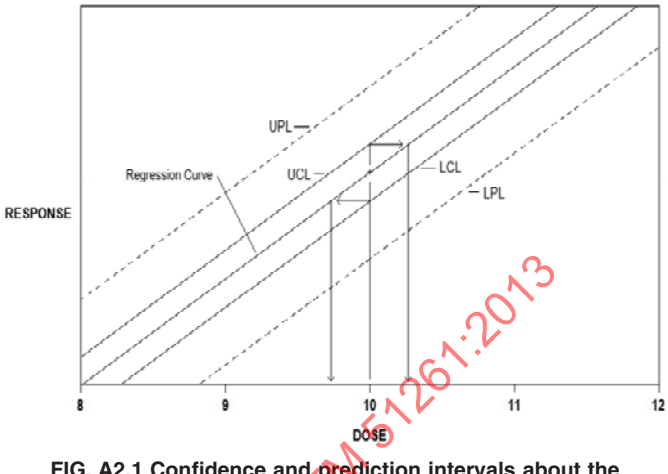

FIG. A2.1 Confidence and prediction intervals about the regression curve

A3. CALIBRATION EXAMPLE

A3.1 The following is an example of a laboratory calibration of a routine dosimetry system based on a type II film dosimeter. This example is a simplified treatment of a calibration and focuses on the mechanics of computation, and does not address the measurement management system specifications and procedures or design of experiment that are required for both a laboratory calibration and in-situ calibration.

A3.2 Prior to the selection of calibration dosimeters, inspect the dosimeter stock for suitability in accordance with a measurement management system. Characteristics that are evaluated are those that impact the routine performance of the routine dosimetry system but also characteristics that affect the laboratory calibration method such as post-irradiation development.

A3.3 Upon dosimeter stock inspection and approval, calibration dosimeters are drawn from the stock. The number of dosimeters for each dose level and the number of dose levels are selected and the total number of dosimeters drawn.

A3.4 The calibration dosimeters are then irradiated.

A3.4.1 For a laboratory calibration method, calibration dosimeters are sent to an approved calibration laboratory for calibration irradiation. Specify and document the irradiation dose rate and irradiation temperature provided to the approved calibration laboratory. These parameters are critical for the success of the laboratory calibration method. Values for dose rate and irradiation temperature should be selected based on the knowledge of the routine measurement conditions and knowledge of the routine type II dosimeter response to the routine measurement influence quantity conditions. Calibration irradiation response data for a type II dosimeter are given in Table A3.1.

A3.5 Regression analysis is applied to the data set for the model below:

$$y = cx^2 + bx + a \quad \text{(A3.1)}$$

A3.5.1 A review of the residuals of the fit model identifies a suspect outlier at the 40 kGy dose level (reference Fig. A3.1). The suspect outlier can be statistically tested, however, the statistical test alone should not be used as the sole basis for the datum omission.

A3.5.1.1 Removing the outlier, the data is re-fitted with the resulting residual plots; Fig. A3.2.

A3.5.1.2 An inspection of the residuals identifies an "oscillating" form. A more complex form should be evaluated for better fit. The more complex form is:

$$y = dx^3 + cx^2 + bx + a \quad \text{(A3.2)}$$

A3.5.2 Using the F test to evaluate the more complex 3rd order polynomial model to the 2nd order polynomial gives:

$$F = \frac{(SS_{null} - SS_{alt})/(DF_{null} - DF_{alt})}{SS_{alt}/DF_{alt}}$$
$$= \frac{4.8714326 \times 10^{-3} - 6.2227065 \times 10^{-4}/(28 - 27)}{6.2227065 \times 10^{-4}/27}$$
$$= 184.369 \quad \text{(A3.3)}$$

where:

SS_{null} = sum of squares of the null hypothesis model (simple model),
SS_{alt} = sum of squares of the alternate hypothesis model (complex model),
DF_{null} = degrees of freedom of the null hypothesis model (simple model), and
DF_{alt} = degrees of freedom of the alternate hypothesis model (complex model).

A3.5.2.1 Solving the F distribution for an F value of 184.369 with 1 degree of freedom in the numerator and 27 degrees of freedom in the denominator) gives a p value of $<<0.001$.

NOTE A3.1—Microsoft Excel will calculate the p value with the

TABLE A3.1 Calibration sample response data

Dose Level	Replicate	Response	Thickness	*k*, (response/thick) x (Norm. Constant)
3 kGy	1	0.188	30.1	0.187
	2	0.188	29.9	0.189
	3	0.186	30.0	0.186
	4	0.186	30.2	0.185
5 kGy	1	0.318	30.1	0.317
	2	0.313	29.8	0.315
	3	0.314	30.0	0.314
	4	0.309	29.9	0.310
10 kGy	1	0.590	29.5	0.600
	2	0.605	30.3	0.599
	3	0.598	30.4	0.590
	4	0.593	30.0	0.593
15 kGy	1	0.842	30.0	0.842
	2	0.842	30.1	0.839
	3	0.829	29.7	0.837
	4	0.831	29.9	0.834
20 kGy	1	1.075	30.6	1.054
	2	1.063	30.3	1.052
	3	1.036	29.7	1.046
	4	1.041	29.8	1.048
30 kGy	1	1.373	29.9	1.378
	2	1.390	30.1	1.385
	3	1.401	30.2	1.392
	4	1.390	29.9	1.395
40 kGy	1	1.641	30.1	1.636
	2	1.705	30.5	1.677
	3	1.629	30.0	1.629
	4	1.600	29.6	1.622
50 kGy	1	1.764	29.4	1.800
	2	1.801	29.9	1.807
	3	1.798	29.8	1.810
	4	1.816	30.0	1.816

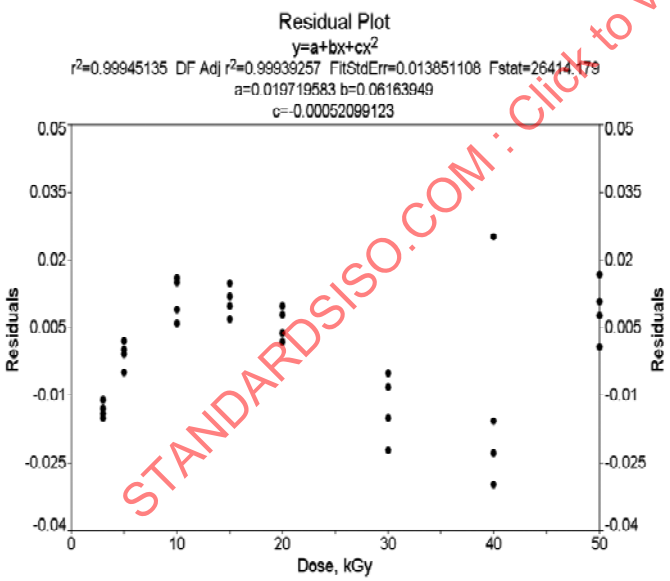

FIG. A3.1 Residual plots

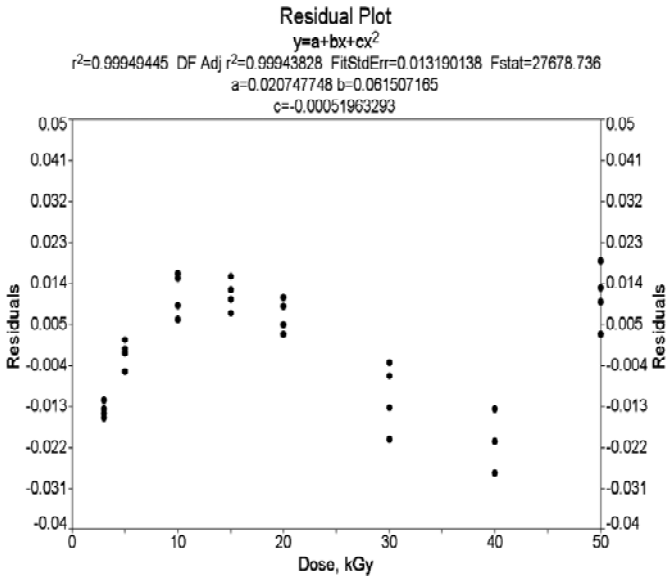

FIG. A3.2 Residuals plot

following formula syntax: *Fdist* (F, DF_n, DF_d), where DF_n is the degrees of freedom in the numerator and DF_d is the degrees of freedom in the denominator.

A3.5.2.2 The extremely small p value warrants testing a more complex model, a 4th order against the 3rd order.

$$F = \frac{(SS_{null} - SS_{alt})/(DF_{null} - DF_{alt}}{SS_{alt}/DF_{alt}}$$
$$= \frac{6.2227065 \times 10^{-4} - (6.2069049 \times 10^{-4})/(27 - 26)}{6.2069049 \times 10^{-4}/26}$$
$$= 0.066191058 \quad \text{(A3.4)}$$

A3.5.2.3 Solving the F distribution for an F value of 0.066191058 and degrees of freedom of 1 (DF_n) and 26 (DF_d) for a p value of $p = 0.799$, which indicates the less complex model is the better fit.

A3.5.2.4 A large p value means the relative increase in the sum of squares is approximately equal to the relative increase in degrees of freedom, i.e. nothing substantial is gained in the fit with the extra degree of freedom used to fit the additional fit coefficient and the less complex model is the better fit. Typically a p value above 0.05 indicates acceptance of the less complex model and a p value below 0.05 indicates acceptance of the more complex model. In the case of the 4th order polynomial and the 3rd order polynomial in the example, the 3rd order provides a better fit ($p = 0.799$).

A3.5.3 The objective of regression analysis is to determine the best fit values of the parameters of the selected model. However, a statement must be made about the parameter estimate, specifically how precisely have the fit coefficients been determined. The standard error and confidence interval of the fit coefficient value is an estimate of how precisely the fit coefficient has been determined. The standard error of a fit coefficient is the expected value of the standard deviation of that coefficient. The construction of a confidence interval at a desired level of confidence about the parameter value is based on the standard error. The confidence interval identifies a range within which the 'true' value of the fit coefficient to be at a stated level of confidence. Thus, the smaller the confidence interval of a fit coefficient, the better the coefficient has been determined.

A3.5.3.1 Review of the parameter estimates of the 3rd order polynomial shown in Table A3.2, the values of standard error and confidence intervals for each parameter.

A3.5.3.2 The t value can also provide a degree of certainty with which the fit parameters are determined. The highest t value indicates the greatest contribution to the fit but is also determined to the greatest level of certainty. A positive t value indicates a direct relationship between the coefficient and the dependent variable (y) where a negative value indicates an inverse relationship.

A3.5.3.3 As shown in Table A3.2 results, the 'b' coefficient is the best determined parameter and has a direct relationship with the dependent variable. The 'a' coefficient is the least well determined coefficient and has an inverse relationship with the dependent variable. The confidence intervals for each coefficient show relatively small intervals indicating the coefficients are well determined. Plots of the 3rd order regression curve and residuals plots are shown in Fig. A3.3, Fig. A3.4, and Fig. A3.5.

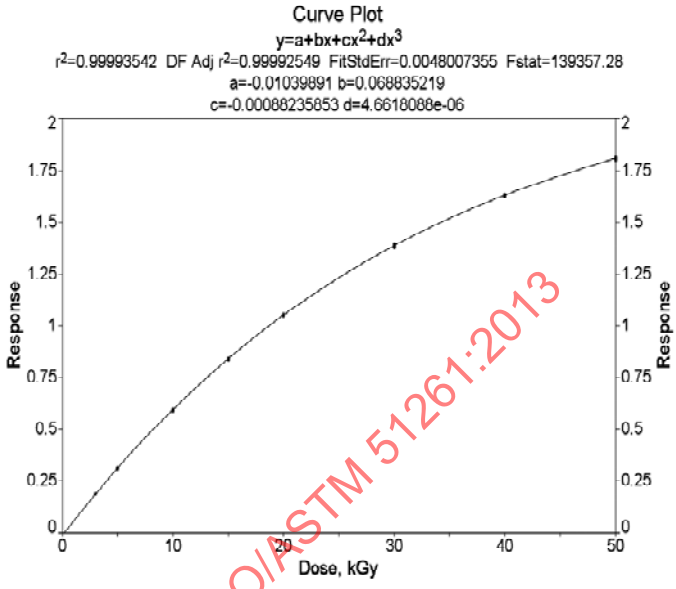

FIG. A3.3 Regression curve

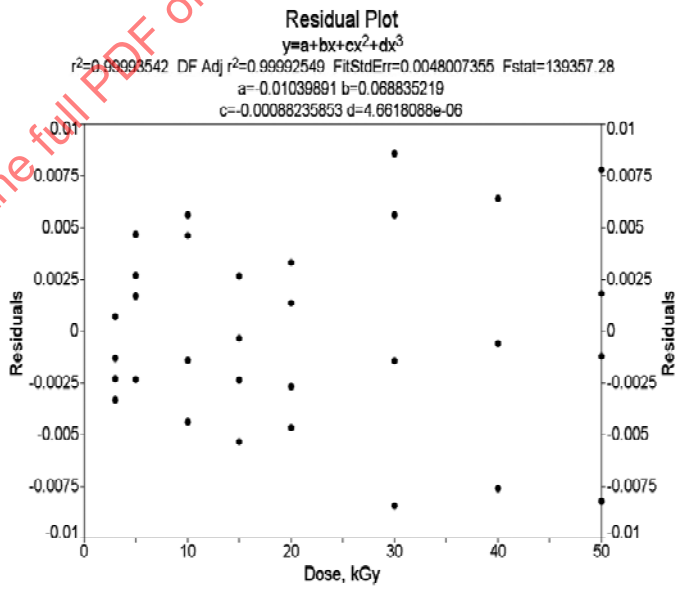

FIG. A3.4 Residuals plot

A3.6 The regression curve is fitted as $y = f(x)$, however the inverse $x = f^{-1}(y)$ is used to estimate absorbed dose for a given dosimeter response value. Directly observable in Fig. A3.4 and Fig. A3.5, variation in the dosimeter response is expected. Well

TABLE A3.2 Third order polynomial coefficient standard error and confidence intervals

Coefficient	Estimate	Standard Error	t value	Upper 95 % Confidence Interval	Lower 95 % Confidence Interval
a	−0.01039891	0.003164916	−3.28568331	−0.01689278	−0.00390504
b	0.068835219	0.000583230	118.0241762	0.067638530	0.070031908
c	−0.00088236	2.7033e-5	−32.6396921	−0.00093783	−0.00082689
d	4.66181e-6	3.43329e-7	13.57825192	3.95736e-6	5.36626e-6

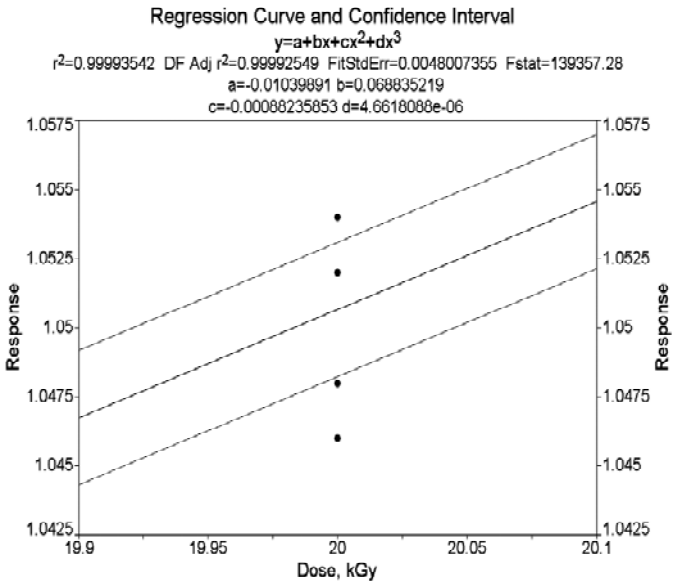

FIG. A3.5 20 kGy dose level regression curve and 95 % confidence intervals

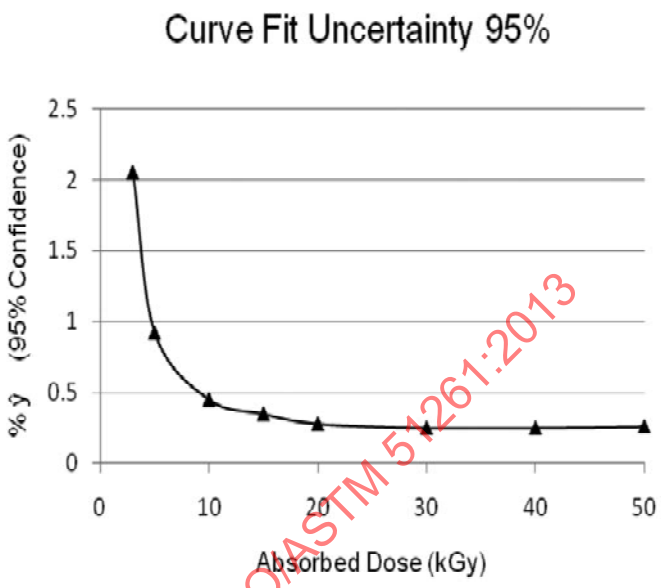

FIG. A3.6 95 % Confidence interval (5 of y)

controlled and monitored radiation processing requires knowledge and an accurate estimate of the repeatability of the routine dosimetry system absorbed dose measurement. Repeatability of the absorbed dose measurement is estimated using the inverse of the fit regression curve and the calibration sample response. The estimate of measurement repeatability is calculated as a pooled relative variance given by Eq A3.5. The '*k*' values from Table A3.1 are used to calculate the dose for each calibration sample replicate. A summary of the components of Eq A3.5 are given in Table A3.3.

$$Precision = k\left(\frac{\sum_{i=1}^{m}(n-1)_i\left(\frac{s^2}{\bar{d}^2}\right)}{(\sum_{i=1}^{m}n_i)-m}\right)^{\frac{1}{2}} \quad \text{(A3.5)}$$

where:

s^2 = the variance of the measurement estimate (x) of the model inverse, $x = f^{-1}(y)$,
$\bar{d}^2$ = the square of the average replicate observation estimates ($\hat{x}$) of the model inverse, $x = f^{-1}(y)$,
n = the number of replicate estimates ($\hat{x}$) at the dose level m,
m = the number of dose levels, and
k = coverage factor (k=2 approximates a 95 % confidence level, or 2σ)

A3.6.1 The repeatability associated with absorbed dose measurements from the 3rd order polynomial is given by Eq A3.6:

$$\left[k\left(\frac{\sum_{i=1}^{m}(n-1)_i\left(\frac{s^2}{\bar{d}^2}\right)}{(\sum_{i=1}^{m}n_i)-m}\right)^{\frac{1}{2}}\right] \quad \text{(A3.6)}$$

$$=\left[1\left(\frac{3(8.1172\times10^{-5})+3(9.4169\times10^{-5})+3(8.2886\times10^{-5})+3(2.4372\times10^{-5})+3(2.1778\times10^{-5})+3(7.8801\times10^{-5})+2(7.1979\times10^{-5})+3(7.3000\times10^{-5})}{(31)-8}\right)^{\frac{1}{2}}\right]$$

$$=\left[1\left(\frac{1.1512492\times10^{-3}}{23}\right)^{\frac{1}{2}}\right]=\left[1\left(\frac{1.151249\times10^{-3}}{23}\right)^{\frac{1}{2}}\right]=[1(8.109286\times10^{-3})]=0.008109286$$

Which when reported as a percent is ±0.81 % at 1σ.

A3.7 Calibration verification for the conditions of use establishes the measurement traceability for the use of the routine dosimetry system within the routine measurement application. Testing consists of co-location of replicates of the routine dosimeters and transfer standard dosimeters at a minimum of three dose levels over the calibration curve range (see 9.1.8). The specific irradiation pathways and parameters are part of the design of experiment. They should be selected so that the validity of the calibration curve near the extremes of expected routine use conditions is tested. For an in-situ/in-plant calibration, verification is only performed when an event such as those identified in 9.2.9.1 have occurred.

TABLE A3.3 Component for the estimate of measurement repeatability

Dose Level kGy	Dose Level Average (kGy)	Standard Deviation (s)	Variance (s^2)	Relative Variance ($s^2/\bar{d}^2$)	Number of Replicates (n)
3	2.975804	2.6810721×10^{-2}	7.18815×10^{-4}	8.1172×10^{-5}	4
5	5.028182	4.8793782×10^{-2}	2.380833×10^{-3}	9.4169×10^{-5}	4
10	10.021406	9.1236514×10^{-2}	8.324101×10^{-3}	8.2886×10^{-5}	4
15	14.970798	7.3907351×10^{-2}	5.462297×10^{-3}	2.4372×10^{-5}	4
20	19.983328	9.3256357×10^{-2}	8.696748×10^{-3}	2.1778×10^{-5}	4
30	30.039379	$2.66658922 \times 10^{-1}$	7.1106981×10^{-2}	7.8801×10^{-5}	4
40	39.972510	$3.39129695 \times 10^{-1}$	$1.15008950 \times 10^{-1}$	7.1979×10^{-5}	3
50	50.005350	$4.27245238 \times 10^{-1}$	$1.82538493 \times 10^{-1}$	7.3000×10^{-5}	4

TABLE A3.4 Calibration verification test results

Dose Target	Routine Dosimeter Dose (d_R)	Transfer Standard Dose ($\bar{d}_t$)	$\bar{d}_t^2$	$s^2(\mathrm{Var_m})$	$s^2/\bar{d}_t^2$	Number of routine dosimeter replicates, n
15 kGy	13.9	14.2	201.64	6.500×10^{-2}	3.22357×10^{-4}	4
	13.9					
	14.0					
	14.0					
25 kGy	24.3	23.9	571.21	4.9000×10^{-1}	8.87279×10^{-4}	4
	24.3					
	23.7					
	23.7					
40 kGy	40.5	40.2	1616.04	1.8500×10^{-1}	1.14477×10^{-4}	4
	39.7					
	40.4					
	39.6					

A3.7.1 The absorbed dose results of the routine dosimeters and transfer standard dosimeters are evaluated as a pooled relative variance sum of squares (see Eq A3.7). For the example, Table A3.4 shows absorbed dose results for the routine type II dosimeters and the co-located transfer standard dosimeters at three dose levels.

$$\left[k\left(\frac{\sum_{i=1}^{m}(n-1)_i\left(\frac{(Var_m)}{(\bar{d}_{tm}^2)}\right)}{(\sum_{i=1}^{m}n_i)-m}\right)^{\frac{1}{2}}\right] \quad \text{(A3.7)}$$

$$=\left[1\left(\frac{3.(3.22357\times10^{-4})+3(8.87279\times10^{-4})+3(1.4477\times10^{-4})}{(12-3)}\right)^{\frac{1}{2}}\right]$$

$$=\left[1\left(\frac{(3.972339\times10^{-3}}{(9)}\right)^{\frac{1}{2}}\right]=[1(0.021008831)]=0.021008831$$

When reported as a percent is ±2.10 % at 1σ.

A3.7.2 Several components of the overall expanded estimate of uncertainty are also expressed in the calibration verification test result of ±2.10%. In order to isolate the component of uncertainty of the absorbed dose measurement of routine dosimetry system for conditions of use, other components expressed in the calibration verification test result need to be 'backed out' of the verification result. Using assigned values for components which have not been directly solved in the example:

u_{CF} = 0.75 (curve fit)
u_{Lab} = 0.60 (uncertainty of the transfer standard temperature correction)
u_{GP} = 0.60 (positioning or dose gradients)
u_{IN} = unknown

and components that have been solved in the example:

u_{CV} = 2.10 (calibration verification test result)
u_{Re} = 0.81 (Repeatability – Precision)
u_{TM} = 0.5 (Calibration verification thickness)

gives the following: