

INTERNATIONAL
STANDARD

ISO/ASTM
51956

Second edition
2005-05-15

**Practice for use of thermoluminescence-
dosimetry (TLD) systems for radiation
processing**

*Pratique de l'utilisation des systèmes dosimétriques à
thermoluminescence pour le traitement par irradiation*

STANDARDSISO.COM : Click to view the full PDF of ISO/ASTM 51956:2005



Reference number
ISO/ASTM 51956:2005(E)

© ISO/ASTM International 2005

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. Neither the ISO Central Secretariat nor ASTM International accepts any liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies and ASTM members. In the unlikely event that a problem relating to it is found, please inform the ISO Central Secretariat or ASTM International at the addresses given below.

STANDARDSISO.COM : Click to view the full PDF of ISO/ASTM 51956:2005

© ISO/ASTM International 2005

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester. In the United States, such requests should be sent to ASTM International.

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

ASTM International, 100 Barr Harbor Drive, PO Box C700,
West Conshohocken, PA 19428-2959, USA
Tel. +610 832 9634
Fax +610 832 9635
E-mail khooper@astm.org
Web www.astm.org

Published in the United States

Contents

	Page
1 Scope	1
2 Referenced documents	1
3 Terminology	1
4 Significance and use	3
5 Apparatus	3
6 Handling and readout procedures	3
7 Summary of requirements for performance testing of a TLD system	4
8 Calibration of the dosimetry system	4
9 Procedures for monitoring the radiation process	4
10 Minimum documentation requirements	5
11 Measurement uncertainty	5
12 Keywords	5
Bibliography	8
Table 1 Types of TLDs and applicable dose ranges	3
Table A1.1 Estimates of uncertainties for typical LiF system utilized as individual chips	7
Table A1.2 Estimates of uncertainties for typical LiF system utilized in batch mode	8

STANDARDSISO.COM : Click to view the full PDF of ISO/ASTM 51956:2005

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 51956 was developed by ASTM Committee E10, Nuclear Technology and Applications, through Subcommittee E10.01, and by Technical Committee ISO/TC 85, Nuclear energy.

This second edition cancels and replaces the first edition (ISO/ASTM 51956:2002), which has been technically revised.



Standard Practice for Use of Thermoluminescence Dosimetry (TLD) Systems for Radiation Processing¹

This standard is issued under the fixed designation ISO/ASTM 51956; 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 procedures for the use of thermoluminescence dosimeters (TLDs) to determine the absorbed dose in materials irradiated by photons or electrons in terms of absorbed dose to water.

1.2 This practice covers systems that permit absorbed dose measurements under the following conditions:

1.2.1 The absorbed-dose range is from 1 Gy to 100 kGy.

1.2.2 The absorbed-dose rate is between 1×10^{-2} and 1×10^{10} Gy s⁻¹.

1.2.3 The radiation-energy range for photons and electrons is from 0.1 to 50 MeV.

1.3 Absorbed dose and absorbed-dose rate measurements in materials subjected to neutron irradiation are not covered in this practice.

1.4 Procedures for the use of TLDs for determining absorbed dose in radiation-hardness testing of electronic devices are given in ASTM Practice E 668.

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:²

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

E 2303 Guide for Absorbed-Dose Mapping in Radiation Processing Facilities

2.2 ISO/ASTM Standards:²

51204 Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing

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

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

51431 Practice for Dosimetry in Electron Beam and X-ray (Bremsstrahlung) Irradiation Facilities for Food Processing

51608 Practice for Dosimetry in an X-Ray (Bremsstrahlung) Facility for Radiation Processing

51649 Practice for Dosimetry in an Electron-Beam Facility for Radiation Processing at Energies Between 300 keV and 25 MeV

51702 Practice for Dosimetry in Gamma Irradiation Facilities for Radiation Processing

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

51939 Practice for Blood Irradiation Dosimetry

51940 Guide for Dosimetry for Sterile Insect Release Programs

52116 Practice for Dosimetry for a Self-Contained Dry-Storage Gamma-Ray Irradiator

2.3 *International Commission on Radiation Units and Measurements (ICRU) Reports:*³

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 60 Fundamental Quantities and Units for Ionizing Radiation

3. Terminology

3.1 Definitions:

3.1.1 *absorbed-dose rate* (\dot{D})—absorbed dose in a material per incremental time interval, that is, the quotient of dD by dt .

$$\dot{D} = \frac{dD}{dt} \quad (1)$$

Unit: Gy · s⁻¹.

3.1.1.1 *Discussion*—(1) The absorbed-dose rate is often specified in terms of its average value over longer time intervals, for example, in units of Gy · min⁻¹ or Gy · h⁻¹. (2) In gamma industrial irradiators, dose rate may be significantly

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 by ASTM Jan. 1, 2005. Published May 15, 2005. Originally published as ASTM E 1956–98. The present International Standard ISO/ASTM 51956:2005(E) is a minor revision of the last previous edition ISO/ASTM 51956:2002(E), which replaced ASTM E 1956–98.

² 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.

³ Available from International Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814, USA.



different at different locations. (3) In electron-beam facilities with pulsed or scanned beam, there are two types of dose rate; average value over several pulses (scans) and instantaneous value within a pulse (scan). These two values can be significantly different.

3.1.2 *annealing*—thermal treatment of a TLD prior to irradiation or prior to readout.

3.1.2.1 *Discussion*—Pre-irradiation annealing of TLDs is usually done to erase the effects of previous irradiation and to readjust the sensitivity of the phosphor; pre-readout annealing usually is done to reduce low-temperature TLD response.

3.1.3 *calibration*—set of operations under specified conditions, which establishes the relationship between values indicated by a measuring instrument or measuring system and the corresponding values realized by standards traceable to a nationally or internationally recognized laboratory.

3.1.3.1 *Discussion*—Calibration conditions include environmental and irradiation conditions present during irradiation, storage and measurement of the dosimeters that are used for the generation of a calibration curve. To achieve stable environmental conditions, it may be necessary to condition the dosimeters before performing the calibration procedure.

3.1.4 *calibration curve*—graphical representation of the dosimetry system's response function.

3.1.5 *calibration facility*—combination of an ionizing radiation source and its associated instrumentation that provides, at a specified location and within a specific material, a uniform and reproducible absorbed dose, or absorbed-dose rate, traceable to national or international standards, and that may be used to derive the dosimetry system's response function or calibration curve.

3.1.6 *charged-particle equilibrium*—condition that exists in an incremental volume within a material under irradiation if the kinetic energies and number of charged particles (of each type) entering that volume are equal to those leaving that volume.

3.1.6.1 *Discussion*—When electrons are the predominant charged particles, the term “electron equilibrium” is often used to describe charged-particle equilibrium.

3.1.7 *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.

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

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

3.1.10 *electron equilibrium*—charged-particle equilibrium for electrons. See *charged-particle equilibrium*.

3.1.11 *measurement quality assurance plan*—documented program for the measurement process that ensures that the expanded uncertainty consistently meets the requirements of the specific application. This plan requires measurement traceability to nationally or internationally recognized standards.

3.1.12 *measurement traceability*—ability to demonstrate by means of an unbroken chain of comparisons that a measure-

ment is in agreement within acceptable limits of uncertainty with comparable nationally or internationally recognized standards.

3.1.13 *primary-standard dosimeter*—dosimeter of the highest metrological quality, established and maintained as an absorbed-dose standard by a national or international standards organization.

3.1.14 *process load*—volume of material with a specified product loading configuration irradiated as a single entity.

3.1.15 *quality assurance*—all systematic actions necessary to provide adequate confidence that a calibration, measurement, or process is performed to a predefined level of quality.

3.1.16 *reference-dose location*—position (on, in or near the process load) where the absorbed-dose value has a reproducible and documented relationship relative to the maximum or minimum absorbed-dose value in the process load.

3.1.16.1 *Discussion*—A dosimeter may be placed at this location for process monitoring during routine product processing.

3.1.17 *reference-standard dosimeter*—dosimeter of high metrological quality, used as a standard to provide measurements traceable to measurements made using primary-standard dosimeters.

3.1.18 *response function*—mathematical representation of the relationship between dosimeter response and absorbed dose for a given dosimetry system.

3.1.19 *routine dosimeter*—dosimeter calibrated against a primary-, reference-, or transfer-standard dosimeter and used for routine absorbed-dose measurements.

3.1.20 *simulated product*—material with radiation attenuation and scattering properties similar to those of the product, material or substance to be irradiated.

3.1.20.1 *Discussion*—Simulated product is used during irradiator characterization as a substitute for the actual product, material or substance to be irradiated. When used in routine production runs in order to compensate for the absence of product, simulated product is sometimes referred to as compensating dummy. When used for absorbed-dose mapping, simulated product is sometimes referred to as phantom material.

3.1.21 *stock*—see *dosimeter stock*.

3.1.22 *thermoluminescence dosimeter (TLD)*—TL phosphor, alone or incorporated in a material, used for determining the absorbed dose to materials.

3.1.22.1 *Discussion*—For example, the TL phosphor is sometimes incorporated in a TFE-fluorocarbon matrix.

3.1.23 *thermoluminescence dosimeter (TLD) reader*—instrument used to measure the light emitted from a TLD consisting essentially of a heating element, a light-measuring device, and appropriate electronics.

3.1.24 *thermoluminescence dosimeter (TLD) response*—measured light emitted by the TLD and read out during its heating cycle consisting of one of the following: (a) the total light output over the entire heating cycle, (b) a part of that total light output, or (c) the peak amplitude of the light output.

3.1.25 *thermoluminescence (TL) phosphor*—material that stores, upon irradiation, a fraction of its absorbed dose in



various excited energy states. When thermally stimulated, the material emits this stored energy in the form of photons in the ultraviolet, visible, and infrared regions.

3.1.26 *TLD preparation*—procedure of cleaning, annealing, and encapsulating the TL phosphor prior to irradiation.

3.1.27 *transfer-standard dosimeter*—dosimeter, often a reference-standard dosimeter, suitable for transport between different locations, used to compare absorbed-dose measurements.

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 ASTM Terminology E 170 are compatible with ICRU Report 60; that document, therefore, may be used as an alternative reference.

4. Significance and use

4.1 During the irradiation of certain crystalline materials, for example, LiF, CaF₂, CaSO₄, Li₂B₄O₇, and Al₂O₃, the filling of electron and hole traps between the ground state and the conduction band results in stored energy that can be released as fluorescence during heating. Thermoluminescence dosimetry systems provide a means of determining absorbed dose to materials by measuring this fluorescence by the controlled heating of the irradiated crystalline material.

4.2 Thermoluminescence dosimeters (TLDs) are available from different commercial suppliers in different forms such as loose powder, chips or crystals encapsulated in glass or plastic.

4.3 TLDs can be reused by subjecting the irradiated TLDs to an annealing process at a higher temperature to release all the electron and hole traps.

4.4 In radiation processing, TLDs are mainly used in the irradiation of blood products (see ISO/ASTM Practice 51939), food (see ISO/ASTM Practices 51204 and 51431), and insects for sterile insect release programs (see ISO/ASTM Guide 51940). TLDs may also be used in other radiation processing applications such as the sterilization of medical products, modification of polymers, irradiation of electronic devices, and curing of inks, coatings and adhesives. (See Refs (1, 2)⁴ and ISO/ASTM Practices 51608, 51649, and 51702).

4.5 For radiation processing, the absorbed-dose range of interest is from 1 Gy to 100 kGy. Some TLDs can be used in applications requiring much lower absorbed doses (for example, for personnel dosimetry), but such applications are outside the scope of this practice. Examples of TLDs and applicable dose ranges are given in Table 1. Further information on various types of TLDs and their applications can be found in Refs (3-8).

4.6 Regulations for sterilization of medical products, irradiation of blood products, and radiation processing of food exist in many countries. These regulations may require that the dosimetry system be calibrated with traceability to national standards (9, 10, 11, 12). Adequate dosimetry, with proper statistical controls and documentation, is necessary to ensure that the products are properly processed.

TABLE 1 Types of TLDs and applicable dose ranges^a

Type of TLD	Linear Dose Range, Gy	Supralinear Dose Range, Gy
LiF: Mg, Ti	10 ⁻⁵ – 1	1 – 10 ³
LiF:Mg, Cu, P	10 ⁻⁶ – 10	NA
CaF ₂ : Mn	10 ⁻⁵ – 10	10 – 10 ³
CaF ₂ :Dy	10 ⁻⁵ – 6	6 – 5 × 10 ²
CaF ₂ :Tm	10 ⁻⁵ – 1	1 – 10 ⁴
Al ₂ O ₃ :C	10 ⁻⁶ – 1	1 – 30
Al ₂ O ₃ :Mg, Y	10 ⁻³ – 10 ⁴	NA
BeO	10 ⁻⁴ – 1	1 – 10 ²
MgO	10 ⁻⁴ – 10 ⁴	NA
CaSO ₄ : Dy and CaSO ₄ :Tm	10 ⁻⁵ – 10	10 – 5 × 10 ³
Li ₂ B ₄ O ₇ : Mn	10 ⁻⁴ – 10 ²	10 ² – 10 ⁴
Li ₂ B ₄ O ₇ : Cu	10 ⁻⁵ – 10 ³	NA
MgB ₄ O ₇ :Dy and MgB ₄ O ₇ :Tm	10 ⁻⁵ – 50	50 – 5 × 10 ³

^aThis table is taken from Ref (13). Ranges are approximate, and may vary with batch. Supralinearity refers to a region where the slope of the response versus dose curve is greater than that for the linear region.

4.7 Proper dosimetric measurements shall be employed to ensure that the product receives the desired absorbed dose. The dosimeters shall be calibrated as outlined in Section 8. All possible factors that may affect the response of dosimeters, including environmental conditions and variations of such conditions within a processing facility, should be known and taken into account. The associated measurement instrumentation shall also be calibrated.

NOTE 1—For comprehensive discussions of various applicable dosimetry methods see ISO/ASTM Guide 51261 and ICRU Reports 14, 17, and 34.

5. Apparatus

5.1 *Components of the Dosimetry System*—The following shall be used to determine absorbed dose with thermoluminescence dosimetry systems:

5.1.1 *Thermoluminescence Dosimeters*

5.1.2 *Thermoluminescence Dosimeter Reader*—The instrument used to measure TLDs consists of a heating element that subjects the TLD to a carefully controlled heating program that allows the freed electrons and holes from traps to recombine with the emission of characteristic light. The emission of light as a function of temperature produces a glow curve that is related to the absorbed dose.

6. Handling and readout procedures

6.1 Bare TLDs should not be handled with the bare fingers; dirt or grease on their surfaces can affect their response and can contaminate the heating chamber of the TLD reader. A vacuum pen or tweezers coated with TFE-fluorocarbon should be used in handling. If required, the TLDs can be cleaned by using the procedures in accordance with Annex A1.

6.2 TLDs, especially those with high sensitivity, should be protected from light such as sunlight or fluorescent light which have an appreciable ultraviolet component. Prolonged exposure to ultraviolet light, either before or after irradiation, can cause spurious TLD response or enhanced post-irradiation fading. Incandescent lighting should be used for the TLD preparation and readout areas. However, brief exposures of a few minutes to normal room fluorescent lighting is not likely to

⁴ The boldface numbers in parentheses refer to the bibliography at the end of this standard.



significantly affect the TLD response except for low dose measurements (<1 Gy) or measurements with high-sensitivity TLDs.

6.3 Preparation of the TLDs for irradiation consists of cleaning the TL phosphor (if required), annealing (if reusable TLDs are employed), and encapsulating the TL phosphor. Reusable TLDs require careful treatment during annealing in order to obtain the best results in dose measurements. The annealing procedure should include a reproducible temperature cycle of the annealing oven, accurate timing of the annealing period, and a reproducible cooling rate.

6.4 If the TLD reader uses hot gas to heat the TLDs, nitrogen should be used.

6.5 Calibration-irradiated TLDs and all subsequent application-irradiated TLDs from the same batch shall be read out with the same reader using the same readout techniques and reader parameters. The calibration is valid only for that batch used in that particular reader. Readers that are different from the one used for calibration, including those of the same make and model, do not necessarily indicate the same response for TLDs irradiated to the same absorbed dose.

6.6 TLDs may be used either as reusable or as single-use dosimeters. Single-use dosimeters are irradiated once, read out, and then discarded; they are generally used as received from the manufacturer. Dosimeters that are reused are cycled repeatedly through an anneal-irradiation-readout procedure.

7. Summary of requirements for performance testing of a TLD system

7.1 The performance of a specific TLD system should be evaluated to determine its suitability for use in a specific radiation processing application. Acceptable performance of the TLD system should be verified before applying the system in a particular application. Specific performance criteria are discussed in ASTM Practice E 668.

7.2 Performance tests should be repeated whenever a significant change is made in the TLD system or in the specific application. Examples of such changes are: a change in the physical form or type of phosphor in the TLD, a change in any critical component or in any adjustable readout factor of the TLD reader, or a change in the irradiation source characteristics.

7.3 A particular performance test may be omitted if widely accepted documentation exists in the scientific and technical literature to show that the performance of the TLD system is satisfactory for that specific requirement. For example, if previously accepted studies document that a particular TLD has no absorbed-dose rate dependence for the expected conditions of irradiation, then performance testing for absorbed-dose rate dependence of that TLD system is unnecessary. All reports of test results should include appropriate references that substantiate the performance of the system and thereby justify the omission of such performance tests.

7.4 If a particular TLD system fails to meet the performance specification of any performance test, then use of that TLD system is not recommended. Such a system may be used only if appropriate corrections to the TLD response can be deter-

mined sufficiently well in order that the results of the specific processing application can be determined within the required uncertainty.

7.5 The number of TLDs, or the number of replicates of measurements with a single TLD, used for each test should be sufficient to ensure that the test results are significant at the 95 % confidence level. See Ref (14) and ASTM Practice E 688 for details of the procedures used to select random samples and to determine the sample size required.

8. Calibration of the dosimetry system

8.1 The dosimetry system shall be calibrated prior to use and at intervals thereafter in accordance with the user's documented procedure that specifies details of the calibration process and quality assurance requirements. Calibration requirements are given in ISO/ASTM Guide 51261.

8.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations shall be performed in one of three ways by irradiating the dosimeters at:

8.2.1 a national or accredited laboratory using criteria specified in ISO/ASTM Practice 51400, with the resulting calibration curve verified for the actual conditions of use, or

8.2.2 an in-house calibration facility that provides an absorbed dose (or an absorbed-dose rate) having measurement traceability to nationally or internationally recognized standards, with the resulting calibration curve verified for the actual conditions of use, or

8.2.3 a production irradiator under actual production irradiation conditions, together with reference- or transfer-standard dosimeters that have measurement traceability to nationally or internationally recognized standards.

8.3 *Measurement Instrument Calibration and Performance Verification*—For the calibration of the instruments, and for the verification of instrument performance between calibrations, see ISO/ASTM Guide 51261 and/or instrument-specific operating manuals.

8.4 When the TLD is used as a reference- or transfer-standard dosimeter, the calibration irradiation shall be performed at a national or accredited laboratory using criteria specified in ISO/ASTM Practice 51400.

9. Procedures for monitoring the radiation process

9.1 Use TLDs to perform dose mapping within the product or simulated product in order to determine the locations of the maximum dose, D_{\max} , and the minimum dose, D_{\min} . Use more TLDs located at the D_{\max} or D_{\min} positions or at a reference dose location to monitor the dose delivered during routine production irradiations.

NOTE 2—Discussion of reference dose locations and general guidance in absorbed-dose mapping in radiation processing facilities is given in ASTM Guide E 2303. Procedures for absorbed-dose mapping in self-contained dry-storage gamma-ray irradiators are given in ISO/ASTM Practice 52116.

9.2 Select the TLDs to be used in monitoring the production irradiations from a batch that has been calibrated previously. The number of TLDs required for determining a specific



absorbed dose during the production irradiations may be obtained from the procedures in ASTM Practice E 668.

9.2.1 From the same batch, select several TLDs to be used as calibration-check TLDs during the analysis of the TLDs. At a time as close as possible to that of the production irradiations, irradiate these TLDs in the calibration facility to two or more known absorbed-dose levels within the absorbed-dose range expected for the production irradiations. Read out these calibrated TLDs along with the TLDs used in the production irradiations. The calibration-check TLDs serve as checks on the stability of the TLD system.

9.2.2 If it is not convenient to use the procedure given in 9.2.1, an alternative procedure may be used. At some time before the production irradiations occur, irradiate a number of TLDs that will be used as calibration checks in the calibration facility to two or more known absorbed-dose levels within the expected absorbed-dose range of the production irradiations. Place these calibration-check TLDs in the storage facility until production irradiations are performed. Remove a few calibration-check TLDs from storage and read them out along with the production-irradiated TLDs. The other calibration-check TLDs remain in storage until the next production irradiations are performed, when a few more should be read out with the production TLDs. The disadvantage of this method compared to that of 9.2.1, is that different fading (and possibly temperature dependence) corrections must be applied to each group of calibration-check TLDs. In addition, the fading correction is different for the calibrated TLDs than for the production TLDs. If the fading correction is excessively large (>25 %) for the calibration-check TLDs, irradiate another group for readout with the production-irradiated TLDs.

9.2.3 If reusable TLDs are irradiated (for either calibration or production use) to high single or accumulated absorbed-dose levels (>10² Gy) re-calibration may be required after each anneal-irradiation cycle because of possible changes in absorbed-dose sensitivity (15). If the TLD system being used is subject to this effect, it is recommended that each TLD in the batch be irradiated only once until the entire batch has been used after which the entire batch can be annealed and a new calibration performed. In addition, because of possible changes in batch response uniformity due to high absorbed-dose irradiations, periodically repeat the tests in accordance with the procedures in ASTM Practice E 668.

10. Minimum documentation requirements

10.1 Document the routine TLD dosimetry system used with each product irradiated. Identify the dosimeter manufacturer, type and batch number, and instruments used for analysis.

10.2 Document the dosimeter calibration data, including date, reference standard or transfer standard, and description of the facility used.

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

10.4 Document the irradiation temperature and relative humidity for routine dosimeters whose performance is affected by these environmental conditions.

10.5 Document the value and the assigned uncertainty of the absorbed dose to the product for each irradiation.

10.6 Document or reference the measurement quality assurance plan used for the routine dosimetry.

11. Measurement uncertainty

11.1 To be meaningful, a measurement of absorbed dose shall be accompanied by an estimate of uncertainty.

11.2 Components of uncertainty shall be identified as belonging to one of two categories:

11.2.1 Type *A*—those evaluated by statistical methods, or

11.2.2 Type *B*—those evaluated by other means.

11.3 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.

11.4 *Calibration and Dose Measurement Uncertainty*—The uncertainties in the calibration and absorbed-dose measurement of a routine TLD dosimetry system depend on the specific dosimetry system employed and on the specific application. See Annex A1 for an example of the use of LiF chips.

NOTE 3—The identification of Type A and Type B uncertainties is based on methodology for estimating uncertainties published in 1995 by the International Organization for Standardization (ISO) in the Guide to the Expression of Uncertainty in Measurement (16). 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 4—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.

12. Keywords

12.1 absorbed dose; dosimeter measurement; radiation processing; thermoluminescence dosimeter; thermoluminescence dosimetry system; TLD; ICS 17.240



ANNEX

(informative)

A1. RECOMMENDED PROCEDURES FOR APPLICATION OF LiF CHIPS

A1.1 Scope

A1.1.1 The procedures in this annex cover the use of lithium fluoride TLDs in the form of reusable solid chips. This is done for illustrative purposes only and is not meant to imply that other types of phosphors, and physical forms of this or other phosphors, are not suitable for use in radiation processing dosimetry. Each type and form of TLD requires a somewhat different application procedure. See Refs (3, 17, 18, 19) for descriptions of various types of TLDs. LiF chips do have some significant advantages over some other types and forms of TLDs. Some of these advantages include radiation absorption characteristics reasonably similar to water and ease of handling compared to powders. One disadvantage in using LiF TLDs is a moderate fading of the TLD response after irradiation. The TLDs discussed here are for the natural isotopic ratio of ^6Li and ^7Li . Single isotope ^6Li and ^7Li type TLDs are generally used in neutron dosimetry and are not addressed in this practice.

A1.2 Dosimeter preparation

A1.2.1 Always handle chips gently and in a manner that will minimize mechanical stress as well as the possibility of scratching or chipping the dosimeter. Never touch the chips with bare fingers to avoid getting dirt or oils on them. The recommended handling tool is a vacuum pen; however, tweezers may be used. The contact points of all handling tools should be coated with TFE-fluorocarbon if possible.

A1.2.2 Between normal uses, the TLDs should be rinsed with analytical-grade anhydrous methyl alcohol and allowed to dry by evaporation (20). More thorough cleaning of the TLDs should not be necessary under normal use. Water should not be used.

A1.2.3 Keep the chips as clean as possible at all times so that additional cleaning can be avoided. Clean the chips only if necessary since the process can contribute to the aging (decrease in sensitivity) of the phosphor. If additional cleaning is necessary, the following procedure is recommended (20).

A1.2.3.1 Wash the chips in approximately 50°C trichloroethylene for 2 min. An ultrasonic cleaner may be used.

A1.2.3.2 Wash the chips in reagent grade anhydrous methyl alcohol for 2 min. An ultrasonic cleaner may be used.

A1.2.3.3 Place the chips between two layers of paper towels and allow to dry by evaporation.

A1.2.4 Anneal the chips for 1 h at 400°C followed by rapid cooling. This annealing is essential after irradiation at high absorbed doses to avoid changes in dose sensitivity. For annealing, place the chips in a tray or container of a material that will not react with them at the annealing temperature, such as high-temperature borosilicate glass. Do not use aluminum.

A1.2.5 For photon irradiation, encapsulate the chips so as to provide electron equilibrium conditions in the dosimeter. (See ISO/ASTM Guide 51261.)

A1.3 Effects of storage and transportation

A1.3.1 Minimize the storage and transportation of the dosimeters either between preparation and irradiation or between irradiation and readout. Protect the dosimeters from ultraviolet light and elevated temperatures during storage or transit. Apply corrections for any effects on dosimeter response caused by the duration and conditions of the storage or transit periods, or both. Correction factors for fading during the storage periods before and after irradiation and for any temperature effects can be determined in accordance with procedures in ASTM Practice E 668. Changes in humidity have not been shown to affect the response of LiF chips.

A1.4 Irradiation procedures

A1.4.1 Procedures for using the TLDs during calibration or production irradiations depend on conditions within each individual facility and on the requirements of the radiation processing application. However, precautions on handling, exposure to light, and exposure to temperature variations apply. The procedures described in Section 9 are applicable.

A1.5 Readout

A1.5.1 Pre-readout cleaning of the chips should be done only if necessary (see A1.2.3). LiF chips may require annealing at low temperatures (approximately 100°C) between irradiation and readout to remove unstable low temperature peaks in the response output. This procedure is necessary only if the entire response output glow curve (current versus temperature) is used. For readers with adjustable temperature discrimination levels or when using the peak-height response, the pre-readout annealing procedure is not needed.

A1.5.2 Reader parameters should be adjusted to give reproducible responses over the absorbed doses measured. For readers that use resistively heated planchets to heat the TLDs, a heating rate of approximately 30°C/s should be satisfactory. The TLD chips should have been heated to a temperature of about 350°C at the end of the heating cycle. For readers that use hot (nitrogen) gas to heat the TLDs, a gas temperature of about 350°C and heating times between 15 and 30 s should be satisfactory.

A1.5.3 TLD response can be measured as the peak height of the light output versus temperature curve, or as integrated light output over the heating cycle. For heating cycles that are very reproducible, the peak height of the light output versus temperature curve may be used. However, the integrated light output is usually conveniently obtained and is satisfactory in most cases. When hot gas readers are used, integrated light



output should be used; the heating profile (and therefore the peak light output) depends on the orientation of the TLD in the reader chamber, which usually cannot be controlled. For readers in which the digital data (charge or current vs temperature) can be obtained, then the data may be analyzed offline and various methods may be used to compare results.

A1.5.4 Most TLD readers are furnished with some type of light source that may be used to check the stability of the reader. This procedure provides a check of the reader stability only for the light measuring section and its associated electronics; it does not test the performance and stability of the heating and temperature measuring section. Therefore, the use of the calibration-check TLDs, as described in 9.2, during each readout session also is recommended.

A1.6 Absorbed dose measurement uncertainty

A1.6.1 An example of the uncertainty analysis of a typical LiF chip system employed in radiation processing is given in Table A1.1 and Table A1.2. These tables identify the sources of uncertainties and give estimates of their magnitudes. A basic assumption for these data is that the TLD system has been characterized and used in accordance with the recommended procedures in this practice. Therefore, as indicated in Footnote A in Table A1.1, certain potential sources of uncertainty are expected to be insignificant in this case.

A1.6.2 The uncertainties are estimated by the methods discussed in Section 11, that is, by classification as Type A and Type B, according to how they are evaluated. The values in the tables are given at the one standard deviation level (Type A; determined by standard statistical methods) or the equivalent one standard deviation level (Type B; determined by all other methods). Table A1.1 gives uncertainties for the TLDs used as individual chips, that is, the identity and calibration response history is maintained for the entire period of use of each chip.

TABLE A1.1 Estimates of uncertainties for typical LiF system utilized as individual chips

Source of Uncertainty	Type A (%)	Type B (%)
⁶⁰ Co source calibrated dose value	0.74	0.47
Determination of calibration curve	0.10	1.00
Time between irradiation and readout: fading correction	0.5	1.00
Correction for attenuation in equilibrium material	...	2.00
Reproducibility of individual dosimeter response	1.00	...
Interspecimen scatter	1.00	...
Absorbed dose rate dependence	A	A
Energy dependence	A	A
Effect of time between preparation and readout	A	A
Directional dependence	A	A
Temperature before, during, and after irradiation	A	A
Humidity dependence	A	A
Effect of size of TLD	A	A
Combined separately in quadrature	1.68	2.49
Total combined in quadrature		3.00
Total combined X 2		6.0

^A For purposes of this uncertainty analysis, it is assumed that the TLD system is utilized in such a way as to make these uncertainties negligible. However, this assumption may not be valid under all conditions of use for radiation processing dosimetry. A careful examination of all possible sources of uncertainty must be made for the irradiation conditions and TLD system employed in each specific application.

Table A1.2 gives uncertainties for the TLDs used in a batch mode with no chip identity maintained and a group calibration response utilized. The difference between individual and batch mode is discussed in ASTM Practice E 668.

A1.6.3 The uncertainties are all assumed to be uncorrelated. They are combined in quadrature and multiplied by a coverage factor of two to provide an expanded (or overall) uncertainty that is approximately equivalent to a 95 % confidence level. If there are known correlations among any of the uncertainties, then that must be accounted for (see Ref (16)). Whatever method of combining uncertainties is used, it should always be reported in the dosimetry measurement results.