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Standard Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices[1]

This standard is issued under the fixed designation E668; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This standard has been approved for use by agencies of the U.S. Department of Defense.

1. Scope

1.1 This practice covers procedures for the use of thermoluminescence dosimeters (TLDs) to determine the absorbed dose in a material irradiated by ionizing radiation. Although some elements of the procedures have broader application, the specific area of concern is radiation-hardness testing of electronic devices. This practice is applicable to the measurement of absorbed dose in materials irradiated by gamma rays, X rays, and electrons of energies from 12 to 60 MeV. Specific energy limits are covered in appropriate sections describing specific applications of the procedures. The range of absorbed dose covered is approximately from 10^{-2} to 10^{4} Gy (1 to 10^{6} rad), and the range of absorbed dose rates is approximately from 10^{-2} to 10^{10} Gy/s (1 to 10^{12} rad/s). Absorbed dose and absorbed dose-rate measurements in materials subjected to neutron irradiation are not covered in this practice. (See Practice E2450 for guidance in mixed fields.) Further, the portion of these procedures that deal with electron irradiation are primarily intended for use in parts testing. Testing of devices as a part of more massive components such as electronics boards or boxes may require techniques outside the scope of this practice.

NOTE 1—The purpose of the upper and lower limits on the energy for electron irradiation is to approach a limiting case where dosimetry is simplified. Specifically, the dosimetry methodology specified requires that the following three limiting conditions be approached: (*a*) energy loss of the primary electrons is small, (*b*) secondary electrons are largely stopped within the dosimeter, and (*c*) bremsstrahlung radiation generated by the primary electrons is largely lost.

1.2 *This standard ~~dose~~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~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E170 Terminology Relating to Radiation Measurements and Dosimetry

E380 Practice for Use of the International System of Units (SI) (the Modernized Metric System) (Withdrawn 1997)[3]

E666 Practice for Calculating Absorbed Dose From Gamma or X Radiation

E2450 Practice for Application of CaF_2(Mn) Thermoluminescence Dosimeters in Mixed Neutron-Photon Environments

2.2 *International Commission on Radiation Units and Measurements (ICRU) Reports:*[4]

ICRU Report 10e Radiobiological Dosimetry

ICRU Report 14 ~~14 Radiation~~Radiation Dosimetry: X Rays and Gamma Rays with Maximum Photon Energies Between 0.6 and 50 MeV

[1] This practice is under the jurisdiction of ASTM Committee E10 on Nuclear Technology and Applicationsand is the direct responsibility of Subcommittee E10.07 on Radiation Dosimetry for Radiation Effects on Materials and Devices on Materials and Devices.

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[2] For referenced 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] The last approved version of this historical standard is referenced on www.astm.org.

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

ICRU Report 17 ~~17—Radiation~~Radiation Dosimetry: X Rays Generated at Potentials of 5 to 150 keV
ICRU Report 21 ~~21—Radiation~~Radiation Dosimetry: Electrons with Initial Energies Between 1 and 50 MeV
~~ICRU Report 31—Average Energy Required to Produce an Ion Pair~~
ICRU Report 33 ~~33—Radiation~~Radiation Quantities and Units
ICRU Report 34 ~~34—The~~The Dosimetry of Pulsed Radiation
ICRU Report 37 ~~37—Stopping~~Stopping Powers for Electrons and Positrons
ICRU Report 90 Key Data for Ionizing-Radiation Dosimetry: Measurement Standards and Applications

3. Terminology

3.1 *Definitions:*

3.1.1 *absorbed dose, D*—the quotient of ~~d~~d$\bar{\varepsilon}$ by dm, where ~~d~~d$\bar{\varepsilon}$ is the mean incremental energy imparted by ionizing radiation to ~~the matter in a volume element and~~ matter of incremental mass dm~~m~~. ~~is the mass of matter in that volume element.~~Thus:

$$D = \frac{\mathrm{d}\bar{\varepsilon}}{\mathrm{d}m} \tag{1}$$

~~Previously, the special unit of absorbed dose was the rad; however, the gray (Gy) has been adopted as the official SI unit (see Practice E380).~~

~~1 Gy = 1 J·kg^{-1} = 10^2 rad~~ (2)

3.1.1.1 *Discussion—*

Previously, the special unit of absorbed dose was the rad; however, the gray (Gy) has been adopted as the official SI unit (see Practice E380).

1 Gy = 1 J·kg^{-1} = 10^2 rad (2)

3.1.2 *absorbed-dose rate*—the absorbed dose per unit time interval.

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

3.1.3.1 *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.

3.1.4 *calibration conditions*—the normal environmental conditions prevailing during routine calibration irradiations such as the ambient temperature, humidity, and lighting.

3.1.5 *equilibrium absorbed dose*—the absorbed dose at some incremental volume within the material which the condition of electron equilibrium (as many electrons of a given energy enter as leave the volume) exists **(1)**[5] (see Appendix X1).

3.1.6 *exposure, X*—the quotient of dQ by dm, where dQ is the absolute value of the total charge of the ions of one sign produced in air when all the electrons (negatrons and positrons) liberated by photons in a volume element of air having mass dm are completely stopped in air.

$$X = \frac{\mathrm{d}Q}{\mathrm{d}m} \tag{3}$$

Unit C · kg^{-1}

3.1.6.1 *Discussion—*

Formerly the special unit of exposure was the roentgen (R).

$$1\,R = 2.58 \times 10^{-4} C \cdot \mathrm{kg}^{-1}\ \text{(exactly)} \tag{4}$$

3.1.7 *primary electrons—for the case of electron irradiation*, the electrons introduced into the device under test by the irradiation source.

3.1.8 *secondary-electron equilibrium—for the case of electron irradiation*, the condition where as many secondary electrons of a given energy enter a given volume as leave it.

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

3.1.9 *secondary-electron equilibrium absorbed dose—for the case of electron irradiation*, the absorbed dose at some incremental volume within the material in which the condition of secondary-electron equilibrium exists.

3.1.9.1 *Discussion—*

Additional definitions can be found in ICRU Report 33.

3.1.10 *secondary electrons— for the case of electron irradiation*, electrons knocked out of the electron shells of the material being irradiated by the primary electron. *For the case of photon irradiation*, energetic electrons (photoelectrons, Auger electrons, and Compton electrons) produced within the material being irradiated by the action of the incident photons.

3.1.10.1 *Discussion—*

Secondary electrons are produced by the interaction of the primary electrons with the atoms of the material being irradiated. This interaction is a principal means of energy loss for the primary electrons. The kinetic energy of a secondary electron is typically much lower than that of the primary electron which creates it.

3.1.11 *test conditions*—the normal environmental conditions prevailing during routine hardness-test irradiations such as the ambient temperature, humidity, and lighting.

3.1.12 *thermoluminescence dosimeter (TLD)*—~~a TL phosphor, alone, or incorporated in a material, used for determining the absorbed dose in materials. For example, the TL phosphor is sometimes incorporated in a TFE-fluorocarbon matrix.~~dosimeter made of a material that stores energy when irradiated by ionizing radiation and then releases that energy in the form of visible light when heated.

3.1.12.1 *Discussion—*

The TL phosphor can be used alone or incorporated into a material such as a TFE-fluorocarbon matrix.

3.1.13 *thermoluminescence dosimeter (TLD) batch*—a group of TLDs, generally originating from a single mix or lot of TL phosphor, having similar TL responses and similar thermal and irradiation histories.

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

3.1.15 *thermoluminescence dosimeter (TLD) response*—the 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.

3.1.16 *thermoluminescence (TL) phosphor*—a 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.

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

3.2 For units and terminology in reports of data, Terminology E170 may be used as a guide.

4. Significance and Use

4.1 Absorbed dose in a material is an important parameter that can be correlated with radiation effects produced in electronic components and devices that are exposed to ionizing radiation. Reasonable estimates of this parameter can be calculated if knowledge of the source radiation field (that is, energy spectrum and particle fluence) is available. Sufficiently detailed information about the radiation field is generally not available. However, measurements of absorbed dose with passive dosimeters in a radiation test facility can provide information from which the absorbed dose in a material of interest can be inferred. Under certain prescribed conditions, TLDs are quite suitable for performing such measurements.

NOTE 2—For comprehensive discussions of various dosimetry methods applicable to the radiation types and energy and absorbed dose-rate range discussed in this practice, see ICRU Reports 14, 17, 21, and 34.

5. Apparatus

5.1 The *TLD System* consists of the TLDs, the equipment used for preparation of the TLDs, and the TLD reader.

5.2 *Calibration Facility* delivers a known quantity of radiation to materials under certain prescribed environmental and geometrical conditions. Its radiation source is usually a radioactive isotope, commonly either ^{60}Co or ^{137}Cs, whose radiation output has been calibrated by specific techniques to some specified uncertainty (usually to within ±5 %) and is traceable to national standards.

5.3 *Storage Facility* provides an environment for the TLDs before and after irradiation, that is light tight and that has a negligible background absorbed-dose rate. A TLD stored in the facility for the longest expected storage period should absorb no more than 1 % of the lowest absorbed dose expected to be measured in hardness-testing applications.

5.4 *Environmental Chamber* is used in testing the effects of temperature and humidity on TLD response. The chamber should be capable of controlling the temperature and humidity within ±5 % over the range expected under both calibration and test conditions.

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 PTFE should be used in handling. If required, the TLDs can be cleaned by using the procedures in accordance with Appendix X2.

6.2 TLDs, especially those with high sensitivity, should be protected from light having an appreciable ultraviolet component, such as sunlight or fluorescent light. 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 significantly affect the TLD response except for low absorbed-dose measurements (<1 Gy or <100 rad) 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 For low absorbed-dose measurements (<1 Gy (100 rad)), dry nitrogen should be flowed through the heating chamber of the TLD reader during readout. This suppresses the spurious TLD response that occurs in most forms of TLDs as a result of absorbed oxygen on the phosphor surface. If the TLD reader uses hot gas to heat the TLDs, nitrogen should be used.

6.5 Calibration-irradiated TLDs and all subsequent test-irradiated TLDs from the same batch should 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.

6.7 The statistical methods specified in the following sections are optimal if the response of a batch of TLDs to a given radiation dose is normally distributed. However, it has been demonstrated that TLD distributions can be severely skewed, so that the sample mean may not be a suitable metric for small sample sizes(**2**). In this case TLDs should be fielded in groups of three, with either the lowest reading or the two extremes discarded. Whatever procedure is adopted, it must be applied consistently for all calibrations and routine measurements.

NOTE 3—Adequately determining the normality of a TLD distribution requires a large sample size.

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-hardness test. Acceptable performance of the TLD system should be verified before applying the system in a particular radiation-hardness-testing facility. Specific performance criteria are discussed in Section 8.

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 determined sufficiently well in order that the results of the specific radiation-hardness test 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 assure that the test results are significant at the 95 % confidence level. See Ref (**3**) for details of the procedures used to select random samples and to determine the sample size required.

NOTE 4—If a sample of n measurements $Y_1, Y_2, \ldots, Y_n$ is taken, the best estimate of the population mean, m, of a normal distribution is given by the mean value, $\bar{Y}$, of the sample:

$$\bar{Y} = \frac{1}{n}\sum_{i=1}^{n} Y_i \tag{5}$$

The best estimate of the variance, σ^2, of the distribution is given by the variance, s^2, of the sample:

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n} \left(\bar{Y} - Y_i\right)^2 \tag{6}$$

The quantity $\sigma\left(=\sqrt{s^2}\right)$ is called the standard deviation of the distribution. The degree to which s is a best estimate of σ depends on the sample size and, as might be expected, s becomes a better estimate of σ as the sample size increases.

8. Specific Performance Tests and Correction Factors

8.1 *Uniformity of TLD Response Within a Batch:*

8.1.1 Select a random sample of 30 TLDs from a batch. Treating all of the sample TLDs in an identical manner, prepare them, irradiate them in the calibration facility to the same absorbed-dose level, and read them out. Determine the variance, s^2, of the sample and estimate the standard deviation of the TLD response distribution $\left(\sigma=\sqrt{s^2}\right)$. the standard deviation, σ, should not exceed 8 % of the sample mean value, $\bar{Y}_0$; that is, $\sigma \leq (0.08)\bar{Y}_0$. The sample size specified (30) is the number of measurements required to estimate the standard deviation, σ, of the TLD response distribution within 25 % of its true value at a 95 % confidence level (see 2.4 of Ref **3**).

8.1.2 For reusable TLDs that have been subjected to a number of anneal-irradiation cycles, the uniformity of the batch response should be verified periodically by repeating the test in accordance with 8.1.1. The frequency required for the test depends on the type of TLD and on its previous anneal-irradiation history. Retesting of the batch uniformity becomes particularly important for TLDs irradiated to high-dose levels ($>10^2$ Gy (10^4 rad)). See, however, X2.2.2.

8.2 *Reproducibility of TLD Response of Individual Reusable Dosimeters*—Certain types of TLDs may be utilized as individual reusable dosimeters. In this case, the identity of each individual dosimeter is maintained during repeated measurement cycles throughout its useful life. This is in contrast to utilization in the batch mode where individual dosimeters within the batch are not identified. To test the reproducibility of the response of an individual reusable dosimeter, the following procedures should be followed:

8.2.1 Select the individual TLD to be tested, prepare it, irradiate it in the calibration facility to a specific absorbed-dose level (for example, at the midpoint of the absorbed-dose range of interest), and read it out. In an identical manner, repeat this procedure 30 times. Determine the variance, s^2, of the responses and estimate the standard deviation of the TLD response distribution $\left(\sigma=\sqrt{s^2}\right)$. The standard deviation, σ, should not exceed 5 % of the mean response value, $\bar{Y}_0$, that is $\sigma \leq (0.05)\bar{Y}_0$.

8.2.2 Some types of TLDs may exhibit a change in sensitivity (that is, response per unit absorbed dose) with repeated anneal-irradiation-readout cycling. This effect is most pronounced if the TLD is not annealed thoroughly. The test results in accordance with 8.2.1 may not show such a change in response sensitivity. However, if such a change is shown in that test or if it appears after a larger number of cycles than specified in that test, then a different analysis of the data is required. In this case, a curve should be fitted to the data of response versus number of cycles by a least-squares method. A measure of reproducibility would then be given by the average standard deviation of the data points from the least-squares curve. The performance criterion is the same as in 8.2.1.

8.2.3 Since the identity of each TLD is maintained when it is utilized as an individual dosimeter, it is not necessary that groups of such individual TLDs meet the batch requirements in accordance with 8.1. However, for the other performance tests and correction factors discussed in Section 8, it is assumed that such tests and factors are evaluated by utilizing TLDs in a batch mode.

8.3 *Dependence of TLD Response on Absorbed-Dose Rate:*

8.3.1 From a TLD batch meeting the requirements in accordance with 8.1.1, select a number of TLDs. Divide the TLDs into x number of groups, each group containing n samples. Determine the absorbed-dose-rate range of interest for the intended application and divide this range into x intervals (for example, one interval per decade). Prepare all the TLDs in an identical manner and irradiate each group to the same dose level, but at a different absorbed-dose rate for each x group, covering the absorbed-dose-rate range of interest. Read out the TLDs. Determine the mean response, $\bar{Y}_i$, for each x group of n samples. Determine an overall mean value, $\bar{Y}_0$, for all x group means. Then the absolute difference between any group mean and the overall mean should not exceed 20 % of the overall mean. That is,

$$\left|\bar{Y}_i - \bar{Y}_0\right| \leq (0.2)\bar{Y}_0 \tag{7}$$

8.3.2 If $|\bar{Y}_i - \bar{Y}_0| > (0.05)\bar{Y}_0$, then appropriate correction factors to the TLD response as a function of absorbed-dose rate should be determined by the procedures that follow.

8.3.3 Determine the number of samples n required in each x group in order to detect a difference of $\delta = (0.05)\bar{Y}_0$ between a group mean and the overall mean for a confidence level of 95 % and a probability of 0.50 of failing to detect such a difference. It is assumed that the variance, σ^2, of the TLD response determined in accordance with 8.1.1, does not vary with the absorbed-dose rate. Calculate the following parameter:

$$d = \frac{\delta}{\sqrt{2\sigma^2}} = \frac{\delta}{\sigma\sqrt{2}} \tag{8}$$

Then the sample size, n, is required for each x group to satisfy the above parameters is read off the graph of n versus d (see Fig. X3.1).

8.3.4 *Example of Sample Number Determination*—If $\sigma = 0.03\ \bar{Y}_0$ (determined in 8.1.1),

$$d = \frac{0.05\ \bar{Y}_0}{\sqrt{2}\,0.03\ \bar{Y}_0} = 1.18 \tag{9}$$

From Fig. X3.1, the sample size required is $n = 4.4$. The sample size should be 5, obtained by rounding up to the nearest integer.

NOTE 5—One method by which this test requirement can be carried out is by comparing the TLD responses with the response of another radiation dosimeter whose absorbed-dose-rate dependence is known. A suitable type of dosimeter for use in most cases would be a calorimeter whose response is absorbed-dose-rate independent and whose radiation absorption properties are similar to the TLD under test.

8.4 *Dependence of TLD Response on Energy:*

8.4.1 The radiation absorption properties of the TLDs employed in radiation-hardness testing should be similar to those of the material in which the dose is to be estimated. Calculations can be made to determine the effects of a broad incident energy spectrum on the response of the TLDs compared to that of the material of interest (usually silicon). The requirements of 7.5 are not applicable to this section.

8.4.2 If the ratios $[(\mu_{en}/\rho)_{TLD}]/[(\mu_{en}/\rho)_{mat}]$ and $[(S/\rho)_{TLD}]/[(S/\rho)_{mat}]$ are equal to 1.0 within ±10 % over a significant range of energy spectrum (for both calibration and test irradiations) incident upon both the TLD and the material of interest, then the energy-response performance of the TLD system is acceptable. Here, μ_{en}/ρ is the mass photon energy absorption coefficient and S/ρ is the mass collision electron stopping power. Tables of values of μ_{en}/ρ and S/ρ for several materials may be found in Appendix X4. The phrase "significant range of the energy spectrum" means the minimum and maximum energy limits containing those incident radiation particles (either photons or electrons) that contribute at least 90 % of the absorbed dose. In this case, detailed energy spectral information is not required; the incident particle fluence (either photons or electrons) between the energy limits is sufficient.

8.4.3 If the energy spectrum of the radiation incident upon the TLD (under both calibration and test conditions) and the material of interest (under test conditions) is well known, then the conversion from absorbed dose in the TLD to absorbed dose in the material of interest can be calculated from such data. If this conversion can be made to an uncertainty of ±10 % or less, then the performance of the TLD system is acceptable. In this case, the criteria concerning the ratios of μ_{en}/ρ and S/ρ in 8.4.2 need not be met. (See Practice E666 for more specific guidelines.)

8.5 *Dependence of TLD Response on Direction of Incident Radiation:*

8.5.1 If the geometrical orientation of the TLD with respect to the radiation-hardness test field is significantly different than its orientation with respect to the calibration radiation field, then any dependency of the TLD response on the direction of the incident radiation should be determined. Select a number of TLDs from a batch meeting the requirements in accordance with 8.1.1. Divide the TLDs into x number of groups, each group containing n samples. Prepare the TLDs in an identical manner, and irradiate each group to the same absorbed-dose level in the following manner: (*a*) group g_0, in the usually oriented direction used for routine calibration, and (*b*) groups $g_1, g_2, \ldots, g_x$ oriented, respectively, at angles $\theta_1, \theta_2, \ldots, \theta_x$, relative to the usually oriented direction with the center of the group at the same distance from the source. These angles should divide, in equal intervals of no more than 30° each, the angle between the normal and the maximum possible angle of incidence of the radiation-hardness test field. Read out all the TLDs. Determine the mean response, $\bar{Y}_i$, for each x group of n samples. Then the absolute difference between the mean, $\bar{Y}_0$, for the normally used calibration orientation and the mean for any other orientation should not exceed 5 % of the mean $\bar{Y}_0$. Thus:

$$\left|\bar{Y}_i - \bar{Y}_0\right| \le 0.05\ \bar{Y}_0 \tag{10}$$

To determine the sample size n required for each x group, use the procedures in accordance with 8.3.3.

NOTE 6—This test applies only to a collimated-beam type source geometry. If the angle of incidence of the radiation from the source is nearly isotropic, then it is recommended that the TLDs and their encapsulation material should be as nearly spherical as possible.

8.6 *Dependence of TLD Response on Time Between Preparation and Irradiation:*

8.6.1 A change in TLD sensitivity can occur during the storage period between preparation and irradiation. This may be a significant effect if a wide range of storage periods is used. Use the following procedure to test for this effect. From a TLD batch meeting the requirements in accordance with 8.1.1, select two equal groups of n samples each. Prepare the first group of TLDs and place them in the storage facility for a time interval equal to the maximum time interval expected between preparation and

irradiation during routine application in either calibration or hardness testing. At a later time, prepare the second group of TLDs, and place them in the storage facility for the minimum time interval expected between preparation and irradiation. Time the procedures so that the ends of the storage period for both groups occur simultaneously. Then irradiate both groups to the same absorbed-dose level in the calibration facility and read them all out. The difference between the mean TLD response, $\bar{Y}_1$, of the first group and the mean response, $\bar{Y}_2$, of the second group is a measure of the effect of storage time between preparation and irradiation. This difference should not exceed 20 % of the average of the means of the two groups. Thus:

$$\left|\bar{Y}_1 - \bar{Y}_2\right| \leq (0.2)\,\frac{\bar{Y}_1+\bar{Y}_2}{2} \tag{11}$$

8.6.2 If the effect tested for in accordance with 8.6.1 exceeds 5 % of the average of the group means, then the functional dependence of the TLD response on the storage period should be determined in order that appropriate correction factors may be applied. This functional dependence may be determined by the procedures that follow.

8.6.3 The range of the elapsed time intervals between preparation and irradiation of interest is determined from the minimum and maximum intervals utilized in 8.6.1. Tests should be performed at a minimum of two intervals per decade of elapsed time over the entire range. For example, if the minimum elapsed time is 0.1 h and maximum elapsed time is 100 h, then an appropriate set of tests would be at elapsed times of 0.1, 0.3, 1, 3, 10, 30, and 100 h. From a TLD batch meeting the requirements in accordance with 8.1.1, select as many groups of *n* samples each as there are elapsed time intervals as determined above. Prepare a group of TLDs, and place it in the storage facility for the appropriate preselected test-time interval. Repeat this procedure for all preselected storage time intervals from the maximum to the minimum elapsed time. Arrange the storage times so that the ends of all procedures occur simultaneously. Then irradiate all groups to the same dose level in the calibration facility and read them all out as quickly as possible thereafter. This procedure is designed to minimize effects on dosimeter response caused by fading and variation in reader output. Determine the mean response for each group of TLDs. A plot of mean TLD response versus elapsed time provides a correction factor for a change in TLD sensitivity as a function of storage period. The number of samples *n* required for each group of TLDs should be determined by the procedure in accordance with 8.3.3.

8.7 *Dependence of TLD Response on Time Between Irradiation and Readout:*

8.7.1 Significant fading of the TLD response may occur during the storage period between the end of irradiation and readout. Use the following procedure to test for this effect. From a TLD batch meeting the requirements in accordance with 8.1.1, select two equal groups of *n* samples each. Prepare the first group of TLDs, irradiate them in the calibration facility to a specific dose level, then place them in the storage facility for an interval equal to the maximum time interval expected during routine application (for either calibration or hardness testing) between the end of the irradiation period and readout. Prepare the second group of TLDs, irradiate them in the calibration facility to the same dose level as the first group, then place them in the storage facility for an interval equal to the minimum time interval expected between the end of irradiation and readout. Time the procedures so the ends of the storage periods for both groups occur simultaneously. Read out all of the TLDs. The absolute difference between the mean TLD response, $\bar{Y}_1$, of the first group and the mean response, $\bar{Y}_2$, of the second group is a measure of the effect of storage time between the end of irradiation and readout. The difference should not exceed 20 % of the average of the means of the two groups. Thus:

$$\left|\bar{Y}_1 - \bar{Y}_2\right| \leq (0.2)\,\frac{\bar{Y}_1+\bar{Y}_2}{2} \tag{12}$$

8.7.2 If the fading effect is greater than $(0.05)(\bar{Y}_1 + \bar{Y}_2)/2$, then either a correction should be made to the TLD response or a procedure used that eliminates the need for a correction. A procedure that achieves the latter would be one in which all TLDs are read out at the same elapsed time after the end or irradiation. Such a procedure is often inconvenient or impractical. Therefore, it is usually necessary to apply a fading correction to the TLD response. The fading characteristics of the TLD system may be determined by the test procedures that follow.

8.7.3 Determine the minimum and maximum elapsed times between the end of the irradiation period and readout. Tests should be performed at a minimum of two time intervals per decade of elapsed time over the entire period in accordance with 8.6.3. From a TLD batch meeting the requirements in accordance with 8.1.1, select as many groups of *n* samples each as there are elapsed time intervals as determined above. Each group of TLDs should undergo identical preparation and then should be irradiated in the calibration facility to the same dose level. The groups of TLDs are placed in the storage facility for all preselected appropriate time intervals from the maximum to the minimum elapsed time. Arrange the time of irradiations for all the groups so that the ends of their storage periods occur simultaneously. Read out all the TLDs. Determine the mean response for each group of TLDs. A plot of mean TLD response versus elapsed time provides the fading correction factor. The number of samples *n* required for each group of TLDs should be determined by the procedures in accordance with 8.3.3.

8.8 *Dependence of TLD Response on Temperature During Storage or Irradiation:*

8.8.1 If the storage temperature experienced by the TLDs between preparation and irradiation during routine radiation-hardness testing differs from the temperature during routine calibration by more than 10°C, the test in accordance with 8.6 should be repeated over the range of temperatures expected using the environmental chamber instead of the storage facility. The performance criteria in accordance with 8.6 are applicable to this section.

8.8.2 If the storage temperature experienced by the TLDs between irradiation and readout during routine radiation-hardness testing differs from the temperature during routine calibration by more than 10°C, the test in accordance with 8.7 should be repeated over the range of temperatures expected using the environmental chamber instead of the storage facility. The performance criteria in accordance with 8.7 are applicable to this section.

8.8.3 If the temperature experienced by the TLDs during the irradiation period during routine radiation-hardness testing differs from the temperature during routine calibration by more than 10°C, then the effect on TLD response should be determined by the following procedure: Select a number of TLDs from a batch meeting the requirements in accordance with 8.1.1, prepare them in an identical manner, and separate them into two equal groups of n samples each. Irradiate the first group in the calibration facility to a specific dose level, maintaining the temperature of the TLDs at the minimum temperature expected during routine hardness-test irradiation. Irradiate the second group in the calibration facility to the same dose level, maintaining the temperature of the TLDs at the maximum temperature expected during routine hardness-test irradiations. Readout all of the TLDs. The difference between the mean TLD response, $\bar{Y}_1$, of the first group and the mean response, $\bar{Y}_2$, of the second group is a measure of the effect of temperature variation during irradiation. This difference should not exceed 20 % of the average of the means of the two groups. If the magnitude of the effect is greater than 5 % of the average of the means, then appropriate corrections to the TLD responses should be determined by procedures analogous to those in accordance with 8.6.

8.9 *Dependence of TLD Response on Humidity*—In general, the responses of the most widely used TLDs have not been shown to be sensitive to changes in relative humidity. However, if a TLD that is hygroscopic is being considered for application in radiation-hardness testing, then the performance tests in accordance with 8.8 should be repeated with the humidity as the variable parameter and the temperature maintained at the maximum value used in the temperature tests.

NOTE 7—Once a TLD system of a particular TL-phosphor type and physical configuration has met the performance requirements of Section 8, new batches of the same type need only be tested for the requirements of 8.1 (batch uniformity) and 8.7 (post-irradiation fading). See also 7.2 and 7.3.

9. Calibration of the TLD System

9.1 Calibrate the TLD system in a manner such that the TLD response can be related directly to the absorbed dose in the TLD phosphor. Use a suitable, well-characterized radiation source in the calibration. Radioactive isotope sources such as ^{60}Co or ^{137}Cs are generally used for this purpose. Exposure rates (or absorbed-dose rates) from such sources should be known to be better than ±5 % at all locations normally used for calibration irradiations. The methods used for determining the output rates from such calibration sources include the use of secondary standard radiation measuring instruments, such as air-ionization chambers or transfer dosimeters, whose calibration is traceable to the National Institute of Standards and Technology (NIST) or other recognized calibration laboratory. Other types of dosimeters whose responses are absolute (require no calibration), such as calorimeters, may also be employed for calibration of such sources.

9.2 The response of most types of TLDs generally is not linear as a function of absorbed dose (**4**). The response of a typical TLD is nearly linear from low-absorbed-dose levels to approximately 10 Gy(TLD) (10^3 rad (TLD)), then becomes supralinear up to approximately 10^2 to 10^3 Gy(TLD) (10^4 to 10^5 rad (TLD)) where saturation effects become evident. Exercise care in the use of the TLD system for absorbed-dose levels of approximately 10^3 Gy(TLD) (10^5 rad(TLD)) or higher to ensure that the change in the system response per unit absorbed dose is adequate in order that the absorbed dose can be determined within the required uncertainty.

9.3 The absorbed-dose range of calibration should cover the maximum absorbed-dose range of interest for the intended application. Measure a minimum of three absorbed-dose levels per decade of absorbed dose covered. Since the TLD response versus absorbed dose for most types of TLDs generally is not linear, make a sufficient number of measurements at each absorbed-dose level to define accurately the shape of the characteristic response curve. The number of TLD samples required to determine the mean response at each absorbed-dose level is given by the following procedures:

9.3.1 In order to determine the mean TLD response, $\bar{Y}_0$, within ±5 % at a 95 % confidence level, the number of TLD samples required for a given absorbed-dose level is as follows:

$$n = \frac{(2.045)^2 s^2}{\left(0.05\ \bar{Y}_0\right)^2} \tag{13}$$

where s is the estimate of the standard deviation σ of the TLD response distribution as determined by the procedures in accordance with 8.1.1. For example , if $s = (0.06)\ \bar{Y}_0$, then:

$$n = \frac{(2.045)^2\left(0.06\ \bar{Y}_0\right)^2}{\left(0.05\ \bar{Y}_0\right)^2} = 6.0 \tag{14}$$

(See Section 2.3.2 of Ref (**3**) for more details.)

9.3.2 The procedures described in 9.3.1 assume that the standard deviation of the TLD response distribution is constant for all absorbed-dose levels measured. This assumption generally is valid over most of the usable absorbed-dose range for most TLDs but may not be correct for very high-absorbed-dose levels of approximately 10^3 Gy(TLD) (10^5 rad(TLD)) or higher. If the TLD

system is used at these absorbed-dose levels, then redetermine the standard deviation of the response distribution at these levels by repeating the procedures in accordance with 8.1.1.

9.4 During a calibration irradiation, encapsulate the TL phosphor in a material with a thickness just sufficient to product electron equilibrium in the phosphor (see Appendix X1). If possible, the encapsulation material should have the same thickness on all sides of the dosimeter.

NOTE 8—The encapsulation material should resemble the phosphor material as closely as possible with respect to radiation absorption properties. For example, if the TL phosphor is CaF_2, acceptable encapsulation material would be calcium fluoride, 1000-series aluminum, or silicon (see Appendix X2). If the calibration source is ^{60}Co, then a thickness of 2.2 mm of aluminum (equal to the practical range of the highest-energy secondary electrons produced) could establish electron equilibrium in the CaF_2 phosphor. This thickness is sufficient to stop secondary electrons that might be generated by the source photons in material other than the encapsulation material.

9.5 Correct for attenuation of the photons from the source by the layer of material used to establish electron equilibrium, using the following formula:

$$X = X_0 \exp\left[-\left(\frac{\mu_{en}}{\rho}\right)\rho x\right] \tag{15}$$

where:

X = unattenuated exposure at the position of the TLD phosphor, roentgens,
X_0 = unattenuated exposure, roentgens,
μ_{en}/ρ = mass energy absorption coefficient of the encapsulation material for the effective source photon energy, cm^2/g,
ρ = density of the encapsulation material, g/cm^3, and
x = thickness of the encapsulation material, cm.

Values of μ_{en}/ρ may be found in Appendix X4 and Ref (**5**).

NOTE 9—The attenuation formula given is not rigorously correct for a broad-beam geometry as it does not include a buildup factor. Buildup factors generally are not available for a wide range of energies, materials, and geometries. However, the formula gives the results that are in reasonable agreement with more rigorous treatments for materials of low to medium atomic number of relatively thin sections over the range of photon energies that are applicable to this practice

9.6 Once the exposure has been determined, the absorbed dose (grays) to the encapsulated TL phosphor is found from the formula:

$$D_{TLD} = \frac{(\mu_{en}/\rho)_{TLD}}{(\mu_{en}/\rho)_{air}}(0.873 \times 10^{-2})X \tag{16}$$

$$D_{TLD} = \frac{(\mu_{en}/\rho)_{TLD}}{(\mu_{en}/\rho)_{air}}(0.877 \times 10^{-2})X \tag{16}$$

The factor ~~(0.873~~(0.877 × 10^{-2}) is used to convert exposure (roentgens) to absorbed dose in air (grays). The subscripts refer to the material of interest. As in 9.5, the μ_{en}/ρ values are evaluated at the effective calibration source photon energy. This formula is valid only if electron equilibrium exists in the TL phosphor. It is assumed that the incident photon fluence is essentially monoenergetic. If this is not the case, then average all of the energy-dependent energy absorption coefficients of 9.5 and 9.6 over the appropriate energy spectrum.

NOTE 10—The value of ~~0.873~~0.877 × 10^{-2} Gy(air)/roentgen is based on an average energy of ~~33.85~~33.97 ± ~~0.15~~0.20 eV required to produce an ion pair in dry air (see ICRU Report ~~31)).~~90).

9.7 The absorbed dose calibration results of the procedures of 9.3 – 9.6 are valid only for a given batch of TLDs. A different batch generally will have a different radiation sensitivity. However, this difference is usually a constant factor over the entire absorbed-dose range. Therefore, it may not be necessary to generate a new calibration curve over the entire absorbed-dose range covered. Measurements at a minimum of two points in the linear region and at a minimum of three points in the supralinear and saturation regions should be used to verify that the shape of the calibration curve is the same for the new batch. If it is not, a new calibration over the entire absorbed-dose range is required.

9.8 Because of possible long-term effects in the TLD reader, recalibrate the TLD system (as specified in 9.3 – 9.6) at periodic intervals over the entire absorbed-dose range of application. The time interval between calibrations depends on the long-term stability of the TLD reader and on how much it is used. For a reader that is used daily with a heavy work load, monthly calibration is probably required. The interval between calibrations should not exceed 12 months.

9.9 Calibration of the TLD reader system shall be checked as described in 9.7 if any maintenance on the system is performed that could affect the calibration, or if any reader parameters are adjusted.

10. Procedures for Characterizing and Monitoring a Test Radiation Field and for Determining Dose in a Device Under Test Using TLD Systems

10.1 A variety of sources are used to produce the radiation fields that are appropriate for radiation-hardness testing of electronic devices. The most widely used fields are ^{60}Co gamma rays, X-ray (bremsstrahlung) photons from fast-pulse (flash) accelerators,

and high-energy electron beams from linear accelerators (linacs). Maximum absorbed-dose rates range from about 10 Gy(Si)/s (10^3 rad (Si)/s) to about 10^{10} Gy(Si)/s (10^{12} rad(Si)/s).

10.2 *Characterization of Radiation Field*—TLDs irradiated in various locations in the test facility under free-field conditions can be used to characterize the radiation field. In addition, it may be desirable and practical to monitor the radiation field of the source during actual radiation-hardness testing of electronic devices. When there is a significant variation of the source output from irradiation to irradiation, use TLDs as monitors.

10.2.1 *TLD Use for Irradiation with Gamma Sources with Energies Above 300 keV*—For irradiation by relatively high energy gamma rays (e.g. ^{60}Co gamma rays), encapsulate the TL phosphor in material with sufficient thickness to produce electron equilibrium conditions in the TL phosphor (see 9.4 and Appendix X1 for details). The equilibrium material should have radiation absorption properties similar to the TLD. When the TLD material is CaF_2:Mn, 1000-series aluminum is an acceptable equilibrium material.

10.2.2 *TLD Use for Irradiation with Pulsed X-Ray Sources:*

10.2.2.1 Pulsed X-ray sources provide a particularly difficult problem because they have a wide range of photon energies. The appropriate treatment of dosimetry for sources depends on the peak electron energy which is used to generate the X-rays. A summary of some relevant considerations for dose in the dosimeter (and dose in the device under test (DUT) is provided in Table 1 for flash X-ray sources lying in three energy bands.

10.2.2.2 For irradiation with pulsed X rays, encapsulate the TL phosphor in material with sufficient thickness to produce electron equilibrium conditions in the TL phosphor (see 9.4, Table 1, and Appendix X1 for details).

10.2.2.3 The combined thickness for the TLD and its protective layer shall be small in comparison to the characteristic absorption lengths of the incident radiation. Since the incident radiation has many wavelengths, this can only be achieved in an approximate sense. A reasonable criterion for the upper limit on the combined thickness is

$$(t_{TLD}+t_{prot})<0.5\,\frac{\int[\mu(E)]^{-1}N(E)\,dE}{\int N(E)\,dE} \tag{17}$$

where $\mu(E)$ is the linear absorption coefficient for photons of energy E, $N(E)$ is the number of photons of energy E per unit energy interval, and t_{TLD} and t_{prot} are the thickness of the TLD and the protective layer, respectively. It will be noted that the application of Eq 17 requires some knowledge of the incident spectrum. However, an approximate knowledge of the spectral distribution will be adequate.

NOTE 11—For many flash X-ray sources, the primary impediment to passing the criterion of Eq 17 come from the low energy end of the spectrum. The problem of the low energy portion of the spectrum is typically negligible for flash X-ray sources with endpoint energies of 10 MeV and above.

NOTE 12—The problems caused by the low energy portion of the flash X-ray spectrum may be ameliorated in some cases by the use of filtration to remove some portion of the photons below approximately 200 keV.

NOTE 13—When the criterion of Eq 17 cannot be passed, then appropriate use of the given source will require the use of an appropriate electron-photon transport code (see Appendix X5). The use of such a code is outside the scope of this document.

10.2.2.4 The equilibrium material should have radiation absorption properties similar to the TLD. When the TLD material is CaF_2:Mn, 1000-series aluminum is an acceptable equilibrium material.

10.2.3 *TLD Use for Electron Irradiation:*

10.2.3.1 For irradiation with electrons, the combined thickness for the TLD and its protective layer shall be small in comparison to the primary electron range. (See 1.1 for a statement of minimum primary electron energy. See also 10.2.2.3.)

TABLE 1 Flash X-Ray Dosimetry Characteristics for Three Energy Bands

Flash X-Ray Type	Absorbed Dose in Dosimeter	Absorbed Dose in DUT
Type I: low energy (most probable photon energy = 20-100 keV)	Can get electron equilibrium[A] May need photon transport correction[B]	Depends on DUT: May need an electron transport calculation[C] May need a photon transport calculation[B]
Type II: medium energy (most probable photon energy = 100-300 keV)	Often cannot get electron equilibrium and therefore need electron transport calculation[D,C]	Depends on DUT: May need electron transport calculation (especially without use of beam filtration)[C,E] May need a photon transport calculation[B]
Type III: high energy (most probable photon energy >300 keV)	Can get electron equilibrium with proper equilibrium layer[D,F]	Depends on DUT: May need an electron transport calculation[C] Usually no photon transport calibration needed[D]

[A]The dosimeter or region of interest is large compared to the electron range.
[B]The dosimeter or region of interest is large compared to the photon range.
[C]The dosimeter or region of interest is of comparable size to the electron range.
[D]The dosimeter or region of interest is small compared to the photon range.
[E]A filter may be used to essentially eliminate the lower energy portions of the flash X-ray spectrum. This makes the spectrum more nearly monochromatic and may simplify dosimetry.
[F]The dosimeter or region of interest is small compared to the electron range.

10.2.3.2 In addition, the combined TLD and protective layer thickness should be large in comparison to the range of secondary electrons. Since there is a wide range of secondary electrons, this requirement can only be met in an approximate sense. (See 10.2.2.3 and 10.2.2.4.)

10.2.3.3 A reasonable criterion for the upper limit on the combined thickness is as follows:

$$(t_{TLD}+t_{prot})<0.05\,R_{csda} \tag{18}$$

where:

R_{csda} = electron range for the primary electron in the continuous slowing down approximation, and
t_{TLD} and t_{prot} = thicknesses of the TLD and the protective layer, respectively.

In this case, scattering of the primary electrons within the TLD is small and thus may be assumed to have a straight-line path through the TLD. Note that Eq 18 sets a lower limit on the primary electron energy which can be used for electron irradiation. It may be necessary under some circumstances to increase the primary electron beam energy in order to meet the requirement of Eq 18.

NOTE 14—Tables of electron range in the continuous slowing-down approximation are available in the published literature. See, for example, ICRU Report 37.

10.2.3.4 When the range of secondary electrons is small in comparison to the thickness of the TLD then it is a good approximation that the dose in the TLD is proportional to the electron stopping power for the TLD, as follows:

$$D_{TLD}=k\times\Phi\times(S/\rho)^{prim}_{TLD} \tag{19}$$

where:

the dose is in Gy,

Φ = electron fluence, cm^{-2},

$(S/\rho)^{prim}_{TLD}$ collision electron mass stopping power in the TLD for the primary electrons, in MeV· cm^2/g,

and

$$k=1.602\times10^{-10}\ \text{Gy·MeV/g} \tag{20}$$

For guidance on the application of Eq 19 see 10.2.2.5, 10.3.2.4, and 10.3.2.5.

NOTE 15—The form of Eq 19 assumes that the primary electron beam is approximately monoenergetic. This is commonly the case for LINAC irradiations.

10.2.3.5 One commonly used practice for electron-beam dosimetry involves using a TLD wrapped with 20 to 40 μm of aluminum. The TLD dose in such a structure can be estimated using Eq 19. Detailed transport calculations using primary electron energies between 6 and 60 MeV suggest that such a procedure overestimates the dose in the TLD. The error is less than about 15 % (**6**). However, what is usually needed is the ratio of the TLD dose to the dose in the region of interest within the device under test (DUT). Using methods parallel to that of Eq 19 to obtain the ratio of TLD dose to DUT dose usually results in smaller errors (see 10.3.2).

NOTE 16—The dose obtained as specified in 10.2.2 for electron irradiation is, strictly speaking, not an equilibrium dose as defined for photon irradiation in 4.4 and Appendix X2. Specifically, the primary electrons are not in equilibrium. However, the dose as defined in this manner may be characterized as secondary-electron equilibrium absorbed dose. That is, the secondary electrons are in approximate equilibrium. In this sense, the case of electron irradiation is parallel to the case of photon irradiation where it is the secondary particles (electrons) which approach an equilibrium condition. As a result, in this practice the term "equilibrium dose" may be read as "secondary-electron equilibrium dose" for the case of electron irradiation.

NOTE 17—Tables of the collision stopping power for electrons can be found in the literature. See, as examples, Appendix X4 and ICRU Report 37.

10.2.4 Select the TLDs to be used in characterizing or monitoring the test radiation field from a batch that has been calibrated previously. From the same batch, select several TLDs to be used a calibration-check TLDs. The number of TLDs required for determining a specific absorbed dose during the test irradiation may be obtained from the procedures in accordance with 9.3.

10.2.4.1 At a time as close as possible to that of the hardness-testing irradiations, irradiate several TLDs in the calibration facility to two or more absorbed-dose levels within the absorbed-dose range expected for the test irradiations. Read out these calibrated TLDs along with the TLDs used in the hardness-testing irradiations. The calibrated TLDs serve as checks on the stability of the TLD system.

10.2.4.2 If it is not convenient to use the procedure in accordance with 10.2.3.1, an alternative procedure may be used. At some time before the hardness-testing irradiations occur, irradiate a number of TLDs that will be used as calibration checks in the calibration facility to two or more absorbed-dose levels within the expected absorbed-dose range of the test irradiations. Place these calibrated TLDs in the storage facility until hardness-testing irradiations are performed. Remove a few calibrated TLDs from storage and read them out along with the test TLDs. The other calibrated TLDs remain in storage until the next test irradiations are performed, when a few more should be read out with the test TLDs. The disadvantage of this method compared to that of 10.2.3.1, is that different fading (and possibly temperature dependence) corrections must be applied to each group of calibrated TLDs. In addition, the fading correction is different for the calibrated TLDs than for the test TLDs. If the fading correction is excessively large (>25 %) for the calibrated TLDs, calibrate another group for readout with the test-irradiated TLDs.