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Standard Procedure for Use of Thermoluminescence Dosimetry in Radiation-Hardness Testing of Electronic Devices

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National Measurement Laboratory
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Interim Report

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U.S. DEPARTMENT OF COMMERCE, Juanita M. Kreps, *Secretary*

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NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Director*

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STANDARD PROCEDURE FOR USE OF THERMOLUMINESCENCE DOSIMETRY
IN RADIATION-HARDNESS TESTING OF ELECTRONIC DEVICES

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1. INTRODUCTION

Electronic systems of many types are employed in the nation's
tactical and strategic weapons systems as well as in deep-space
probes. These systems must be able to function reliably in the
radiation environments they are expected to encounter in carrying
out their missions. There has been a lack of consistency in the
experimental results of radiation dosimetry measurements made at
the various facilities that are testing the radiation hardness of
these electronic systems. It became apparent that there was a need
for improvement in the dosimetry procedures employed by the hardness-
testing facilities. Since thermoluminescence dosimetry (TLD) systems
are widely used by the hardness-testing facilities, it was clear that
a standard TLD procedure would improve the reliability, reproducibility,
and uniformity of dosimetry measurements at these facilities. To this
end, a standard recommended practice was developed for the use of TLDs
in measuring absorbed dose in a medium as a result of photon or elec-
tron irradiation. This recommended practice is in the form of an
American Society for Testing and Materials (ASTM) standard. The
latest draft of this standard is contained in this report.

It should be noted that this draft standard has not received final approval from ASTM (although approval is expected within the next few months); therefore, it should not be cited as a reference or published in another form without the expressed approval of the E10 Committee Chairman or the Managing Director of ASTM. When the standard has been approved and published by ASTM, it may be obtained from that organization and referenced.

This report includes a selected bibliography of TLD character- istics. This listing is not represented as being comprehensive, but is intended as a guide for those workers getting started in the field of thermoluminescence dosimetry.

The reference citations in the body of the draft standard are for the references listed within that standard (pp. 35 and 36) and are not to be confused with the bibliography.

ACKNOWLEDGMENTS

E668-78

Standard Recommended Practice for

THE APPLICATION OF THERMOLUMINESCENCE-DOSIMETRY (TLD) SYSTEMS FOR

DETERMINING ABSORBED DOSE IN RADIATION-HARDNESS TESTING OF ELECTRONIC DEVICES

1. Scope

1.1 This practice covers procedures for the use of thermo-
luminescence 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 in which the material
of interest is usually silicon. This practice is applicable to the measure-
ment of absorbed dose in materials irradiated by gamma rays, x rays, and
electrons of energies up 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
(one to 10^6 rad) and the range of absorbed dose rates is approximately from
10^{-2} to 10^{10} Gy/s (one to 10^{12} rad/s). Absorbed dose and absorbed dose-
rate measurements in materials subjected to neutron irradiation are not
covered in this standard.

2. Significance

2.1 Absorbed dose in a material is an important parameter that
can be correlated with radiation effects produced in electronic com-
ponents and devices that are exposed to ionizing radiation. Reason-
able estimates of this parameter can be calculated if knowledge
of the source radiation field (i.e., 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.

3. Applicable documents

   3.1 ASTM standards

      E 170. Definitions of Terms Relating to Dosimetry.

      E 380. Metric Practice

      E 665. Practice for Determining Absorbed Dose vs. Depth
              in Materials Exposed to the X-Ray Output of Flash
              X-Ray Machines.

      E 666. Method for the Calculation of Absorbed Dose from
              Gamma- or X-Radiation.

   3.2 International Commission on Radiation Units and Measurements
        (ICRU) Reports*

        ICRU Report 10b. Physical Aspects of Irradiation

        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 19. Radiation Quantities and Units

        ICRU Report 21. Radiation Dosimetry: Electrons with Initial
              Energies Between 1 and 50 MeV

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* Available from ICRU, 7910 Woodmont Avenue, Washington, D.C. 20014.

4. Definitions

4.1 <u>absorbed dose</u> - D, is the quotient of $d\bar{\epsilon}$ by dm, where $d\bar{\epsilon}$ is the mean energy imparted by ionizing radiation to the matter in a volume element and dm is the mass of matter in that volume element.

$$D = \frac{d\bar{\epsilon}}{dm}$$

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

$$1 \text{ Gy} = 1 \text{ J} \quad kg^{-1} = 10^2 \text{ rad}$$

4.2 <u>exposure</u>- X, is 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.

$$X = \frac{dQ}{dm}$$

The special unit of exposure is the roentgen (R).

$$1 \text{ R} = 2.58 \times 10^{-4} \text{ C} \cdot kg^{-1}$$

4.3 <u>absorbed-dose rate</u> - the absorbed dose per unit time interval.

4.4 <u>equilibrium absorbed dose</u> - the absorbed dose at some incremental volume within the material in which the condition of electron equilibrium (as many electrons of a given energy enter as leave the volume) exists. (See reference 1 and Appendix A3.)

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4.5 thermoluminescence (TL) phosphor - a material which 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.

4.6 thermoluminescence dosimeter (TLD) - a TL phosphor, alone, or incorporated in a material, used for determining absorbed dose in materials. For example, the TL phosphor is sometimes incorporated in a Teflon matrix.

4.7 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.

4.8 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.

4.9 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.

4.10 TLD preparation - the procedure of cleaning, annealing, and encapsulating the TL phosphor prior to irradiation.

4.11 annealing - thermal treatment of a TLD prior to irradiation or prior to readout.

NOTE 1 - 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.

4.12 calibration conditions - the normal environmental conditions prevailing during routine calibration irradiations such as the ambient temperature, humidity, and lighting.

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

5. Apparatus

5.1 A TLD system consists of the TLDs, the equipment used for preparation of the TLDs, and the TLD reader.

5.2 A 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%).

5.3 A 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 An 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, such as those not sealed in glass, should not be handled with the bare fingers; dirt or grease on their surfaces can affect their response. It is recommended that tweezers or vacuum pick-up tools be used in handling. If required, the TLDs can be cleaned by using the procedures given in Appendix Al.

6.2 TLDs should be protected as much as possible from light having an appreciable ultraviolet component such as sunlight or fluorescent lighting. Prolonged exposure to uv light, either before or after irradiation, can cause spurious TLD response and enhanced post-irradiation fading. Incandescent lighting is recommended for the TLD preparation and readout areas. However, brief exposures of a few minutes under normal room fluorescent lights should not significantly affect TLD response except for low absorbed-dose measurements (<1 Gy or <100 rad).

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 or <100 rad), dry nitrogen should be flowed through the heating pan area of the TLD reader during readout. This technique suppresses the spurious TL response that occurs in most forms of TLDs as a result of adsorbed oxygen on the phosphor surface.

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 TLD response for TLDs irradiated to the same absorbed dose.

6.6 TLDs are utilized in two basic ways: as reusable or as single-use "throw-away" dosimeters. Dosimeters employed in a reusable mode are cycled repeatedly through an anneal-irradiation-readout procedure. The single-use dosimeters are irradiated once, read out, and then discarded; they are generally used as received from the manufacturer.

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 ascertained before applying the system in a particular radiation-hardness-testing facility. Specific performance criteria will be 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. However, such a system may be employed, if necessary, but 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.

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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. The number of measurements required is specified in each test procedure and is based on the assumption of random samples drawn from a TLD batch whose responses have a normal population distribution. See reference 2 for details of the procedures used to select random samples and to determine the sample size required.

NOTE 2 - If a sample of n measurements Y_1, Y_2, ..., Y_n is taken, the best estimate of the <u>population mean</u>, m, of a normal distribution is given by the <u>mean value</u>, $\overline{Y}$, of the sample:

$$\overline{Y} = \frac{1}{n} \sum_{i=1}^{n} Y_i \ .$$

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

$$s^2 = \frac{1}{n-1} \sum_{i=1}^{n} (Y_i - \overline{Y})^2$$

The quantity $\sigma (\approx \sqrt{s^2})$ is called the <u>standard</u> <u>deviation</u> of the distribution. It should be noted that 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. Treat 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 ($\sigma = \sqrt{s^2}$). The standard deviation σ should not exceed 8% of the sample mean value, $\overline{Y}_0$; i.e., $\sigma \leq (0.08)\, \overline{Y}_0$. 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 section 2.4 of reference 2).

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 of 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)).

8.2 Reproducibility of TLD response of individual reusable dosimeters

Certain types of TLDs may be utilized as individual reusable do-simeters. 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.

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

8.2.2   Some types of TLDs may exhibit a change in sensitivity
(i.e., 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 of 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 vs. 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 of 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.

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8.3   Dependence of TLD response on absorbed-dose rate

8.3.1.   From a TLD batch meeting the requirements of 8.1.1, select a number of TLDs. Divide the TLDs into a number of groups, x, 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, $\overline{Y}_i$, for each x group of n samples. Determine an overall mean value, $\overline{Y}_o$, 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,

$$| \overline{Y}_i - \overline{Y}_o | \leq (0.2) \overline{Y}_o \quad .$$

8.3.2   If $| \overline{Y}_i - \overline{Y}_o | > (0.05) \overline{Y}_o$, 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 $\delta = (0.05)\overline{Y}_o$ between a group mean and the overall mean for a confidence level of 95% and a probability of 0.05 of failing to detect such a difference. It is assumed that the variance (σ^2) of the TLD response, determined in 8.1.1, does not vary with absorbed-dose rate. Calculate the following parameter:

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

Then the sample size, n, required for each x group to satisfy the above parameters is read off the graph of n vs. d (Fig. A4.1) in Appendix A4.

8.3.4   Example of sample number determination.

If $\sigma = 0.03\ \overline{Y}_0$ (determined in 8.1.1),

$$d = \frac{0.05\ \overline{Y}_0}{\sqrt{2}\ \ 0.03\ \overline{Y}_0} = 1.18.$$

From Fig. A4.1, the sample size required is n = 4.4. The sample size should be 5, obtained by rounding up to the nearest integer.

NOTE 3 - 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 $\dfrac{(\mu_{en}/\rho)_{TLD}}{(\mu_{en}/\rho)_{mat}}$ and $\dfrac{(S/\rho)_{TLD}}{(S/\rho)_{mat}}$ are equal to 1.0 within ±10% over a significant range of the 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

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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 A2. 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 ASTM Method E 666 for more specific guidelines.)

8.5   <u>Dependence of TLD response on direction of incident radiation.</u>

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 of 8.1.1. Divide the TLDs into a number of groups, x, 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,....., g_x oriented, respectively, at

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angles θ_1, θ_2,...., θ_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 normal and the maximum possible angle of incidence of the radiation-hardness test field. Read out all the TLDs. Determine the mean response, $\overline{Y}_i$, for each x group of n samples. Then the absolute difference between the mean, $\overline{Y}_o$, for the normally used calibration orientation and the mean for any other orientation should not exceed 5% of the mean. $\overline{Y}_o$. That is,

$$| \overline{Y}_i - \overline{Y}_o | \leq 0.05 \, \overline{Y}_o \quad .$$

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

NOTE 4: CAUTION - This test applies only to a collimated-beam type calibration source geometry. If the angle of incidence of the radiation from the calibration 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 of 8.1.1, select two equal groups of n samples each. Prepare the first group of TLDs and place them in the storage facility

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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 periods 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, $\overline{Y}_1$, of the first group and the mean response, $\overline{Y}_2$, 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. That is,

$$| \overline{Y}_1 - \overline{Y}_2 | \leq (0.2)\frac{\overline{Y}_1 + \overline{Y}_2}{2} .$$

8.6.2 If the effect tested for in 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 hour and maximum elapsed time is 100 hours, than an appropriate set of tests would be at elapsed times of 0.1, 0.3, 1, 3, 10, 30, and 100 hours. From a TLD batch meeting the requirements of 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 vs. 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 procedures of 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 of 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

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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, $\overline{Y}_1$, of the first group and the mean response, $\overline{Y}_2$, of the second group is a measure of the effect of storage time between the end of irradiation and readout. This difference should not exceed 20% of the average of the means of the two groups. That is,

$$| \overline{Y}_1 - \overline{Y}_2 | \leq (0.2) \frac{\overline{Y}_1 + \overline{Y}_2}{2} \quad .$$

8.7.2   If the fading effect is greater than $(0.05) \frac{\overline{Y}_1 + \overline{Y}_2}{2}$, 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 of 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 as discussed in 8.6.3. From a TLD batch meeting the requirements of 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

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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 simul-

taneously. Read out all the TLDs. Determine the mean response for each

group of TLDs. A plot of mean TLD response vs. elapsed time provides the

fading correction factor. The number of samples n required for each group

of TLDs should be determined by the procedures of 8.3.3.

    8.8   <u>Dependence of TLD response on temperature during storage or</u>

           <u>irradiation</u>

    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, test 8.6 should be repeated over the range of temperatures expected

using the environmental chamber instead of the storage facility. The per-

formance criteria of 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, test 8.7

should be repeated over the range of temperatures expected using the environ-

mental chamber instead of the storage facility. The performance criteria

of 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 of 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 irradiations. 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, $\overline{Y}_1$, of the first group and the mean response, $\overline{Y}_2$, 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 used in 8.6.

8.9 Dependence of TLD response on humidity

8.9.1   In general, the responses of the most widely used TLDs have not been shown to be sensitive to changes in relative humidity [3]. However, if a TLD that is hygroscopic (such as lithium borate) is being considered for application in radiation-hardness testing, then the perfomance tests of 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 5.   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).

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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. The exposure rate (or absorbed-dose rate) produced by the source should be known to better than ±5% at all locations normally used for calibration irradiations. The methods used for determining the output rates of such sources include the use of secondary standard radiation measuring instruments, such as air-ionization chambers, whose calibration is traceable to the National Bureau of Standards or other recognized calibration laboratories. Other types of dosimeters, whose responses are absolute (require no calibration), such as ferrous sulfate dosimeters and calorimeters, may also be employed for source calibration.

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 usually linear from low absorbed-dose levels (millirad region) to approximately 10 Gy (10^3 rad), then becomes supralinear up to approximately 10^2 - 10^3 Gy (10^4 - 10^5 rad) where saturation effects become evident. Exercise care in the use of the TLD system for absorbed dose levels of approximately 10^3 Gy (10^5 rad) 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 vs. absorbed dose for most types of TLDs generally is

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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, $\overline{Y}_0$, within ±5% at a 95% confidence level, the number of TLD samples required for a given absorbed-dose level is

$$n = \frac{(2.045)^2 \ s^2}{(0.05 \ \overline{Y}_0)^2} \quad ,$$

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

$$n = \frac{(2.045)^2 \ (0.06 \ \overline{Y}_0)^2}{(0.05 \ \overline{Y}_0)^2} = 6.0$$

(See Section 2-3.2 of reference 2 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 (10^5 rad) 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 of 8.1.1.

9.4 During a calibration irradiation, encapsulate the TL phosphor in a material with a thickness just sufficient to produce electron equili-

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brium in the phosphor (see Appendix A3). If possible, the encapsulation material should have the same thickness on all sides of the dosimeter.

NOTE 6. 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 CaF_2, Al, or Si. If the calibration source is ^{60}Co, then a thickness of 2.2 mm of Al (equal to the practical range of the highest energy secondary electrons produced) would 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 It is necessary to correct for attenuation of the photons from the source by the layer of material used to establish electron equilibrium. This should be done using the following formula:

$$X = X_0 e^{[-(\mu_{en}/\rho)\rho x]}$$

where X is the attenuated exposure at the position of the TLD phosphor in roentgens,

X_0 is the unattenuated exposure in roentgens,

μ_{en}/ρ is the mass energy absorption coefficient of the encapsulation material for the effective source photon energy in cm^2/g,

ρ is the density of the encapsulation material in g/cm^3, and

x is the thickness of the encapsulation material in cm.

Values of (μ_{en}/ρ) may be found in Appendix A2.

NOTE 7. 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 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 standard.

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

$$D_{TLD} = \frac{(\mu_{en}/\rho)_{TLD}}{(\mu_{en}/\rho)_{air}} \ (0.869 \times 10^{-2})X$$

The factor (0.869×10^{-2}) is used to convert exposure (R) to absorbed dose in air (Gy). Conversion of exposure (R) to absorbed dose in air (rad) would require a factor of 0.869. 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.

9.7 The absorbed dose calibration results of the procedures of 9.3 to 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

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range. Therefore, it is usually not 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 of the response curve normally is adequate to characterize the absorbed-dose sensitivity of a different batch.

9.8 Because of possible long term aging effects in the TLD reader, recalibrate the TLD system (as specified in 9.3 to 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. If the reader is used only a few hours a week, then recalibration at 6 or 12 months intervals should be adequate. For a reader that is used daily with a heavy work load, monthly calibration is probably required.

10. Procedures for characterizing and monitoring a test radiation
field with 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 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 For irradiation by gamma rays or pulsed x rays, encapsulate
the TL phosphor in material with sufficient thickness to produce electron
equilibrium conditions in the TL phosphor. See Section 9.4 and Appendix A3
for details. The equilibrium material should have radiation absorption
properties similar to the material in which the absorbed dose is to be
determined. Since silicon is usually the material of interest, aluminum
is an acceptable equilibrium material.

10.2.2 For irradiation with electrons, the absorbed dose as a
function of depth for normally incident monoenergetic electrons has the
characteristic shape shown in Fig. 1 [5]. For the electron energy spectrum
appropriate to the test source being employed, encapsulate the TL phosphor

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in a material with a thickness of approximately 0.1 the practical range (R_p in Fig. 1) of the maximum-energy electrons on the incident radiation side and with a thickness greater than the practical range on the side opposite the incident electrons. By placing the TLD at this depth in the material, possibly anomalous response caused by interface effects at the material surface is avoided. As in the case for photons, the encapsulation material should have radiation absorption properties similar to the material of interest. In all cases in which the TLD is encapsulated, the combined thickness of the encapsulation material in front of the TLD and the TLD itself should be such that the back surface of the TLD is located at a depth not much greater than the peak of the absorbed dose vs. depth curve. For normal incidence, this depth is approximately one half of the practical range of the electrons.

10.3 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 as calibration-check TLDs. The number of TLDs required for determining a specific absorbed dose during the test irradiation may be obtained from the procedures of 9.3.

10.3.1   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. These calibrated TLDs serve as checks on the stability of the TLD system.

10.3.2   If it is not convenient to use the procedure described in 10.3.1, an alternate procedure may be used. At some time before the

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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 10.3.1, 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.

10.3.3   If reusable TLDs are irradiated (for either calibration
or testing) to high single or accumulated absorbed-dose levels (>10^2 Gy
(10^4 rad)), recalibration may be required after each anneal-irradiation cycle
because of possible changes in absorbed-dose sensitivity [6]. 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 per-
formed. In addition, because of possible changes in batch response uni-
formity due to high absorbed-dose irradiations, repeat the tests of 8.1.1
periodically.

10.4 The equilibrium absorbed dose in a material of interest can
be estimated from the absorbed dose in a TLD exposed to the same radiation

field. This involves a conversion of the absorbed dose in a TLD irradiated within a material under electron equilibrium conditions to absorbed dose in a volume element of the material of interest having comparable dimensions to the TLD, the volume element being surrounded by an equilibrium thickness of the same material of interest. It has been shown that the TLD response per unit absorbed dose in the TLD material is independent of the type (photons or electrons) or the energy spectrum of the incident radiation for the range of energies considered in this standard.

10.4.1   In a material undergoing photon irradiation, the presence of the TLD may perturb the spectrum of the secondary electrons generated by the primary photons. If the TLD is very thin compared to the range of the secondary electrons, most of the energy deposited in the TLD and in the material surrounding it come from secondary electrons produced outside the TLD (i.e., in the equilibrium layer of material). Thus, the absorbed dose in the material is given by

$$D_{mat.} = \frac{(S/\rho)_{mat.}}{(S/\rho)_{TLD}} \cdot D_{TLD} \tag{1}$$

If the TLD has a thickness much greater than the range of the secondary electrons, most of the energy deposited in it comes from secondary electrons produced within the TLD itself. Thus, the absorbed dose in the material is given by

$$D_{mat.} = \frac{(\mu_{en}/\rho)_{mat.}}{(\mu_{en}/\rho)_{TLD}} \cdot D_{TLD} \tag{2}$$

If the TLD thickness is intermediate between the two limits given above, then the two equations may be combined with appropriate weighting factors to reflect the relative contributions of each term [7]. In

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general, for low atomic number material and for photon energies above about 0.2 MeV, the difference in the absorbed dose determined by equations (1) and (2) is usually less than 10%. If the equilibrium buildup material surrounding the TLD is not silicon, then the equilibrium absorbed dose in silicon is given by

$$D_{Si} = \frac{(\mu_{en}/\rho)_{Si}}{(\mu_{en}/\rho)_{mat.}} \cdot D_{mat.} \tag{3}$$

10.4.2   The effects of dosimeter size on the absorbed-dose conversion for electron irradiation are not as clearly understood as for photon irradiation [8,9]. However, reasonable estimates of the absorbed dose in the material of interest can be made using equation (1) in 10.4.1 if the initial incident electron energy is greater than 5 MeV and the TLD is less than two thirds of the practical electron range in thickness. If the material surrounding the TLD is not silicon, then the absorbed dose in silicon is given by

$$D_{Si} = \frac{(S/\rho)_{Si}}{(S/\rho)_{mat.}} \cdot D_{mat.} \tag{4}$$

10.5 Limitations of interpretation. Caution must be used in interpreting the results of using the procedures of 10.4 for converting the absorbed dose in the TLD to absorbed dose in the material of interest (assumed to be silicon). Reasons for this are given in the following sections.

10.5.1   The absorbed-dose conversions are most reliable when the TLD and the equilibrium material surrounding it are similar to silicon in radiation absorption properties.

10.5.2   The absorbed dose in the material is interpreted from an integrated or average absorbed dose in the TLD at its location in the

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surrounding material and does not necessarily represent the actual absorbed dose at any other point within the volume of the material.

10.5.3   The evaluated equilibrium absorbed dose in silicon does not necessarily represent the absorbed dose in an electronic device irradiated in the same test field. A number of factors complicate a straightforward interpretation of the absorbed dose distribution within an irradiated device. Examples of such perturbing factors include attenuation of the radiation by the packaging material surrounding the device chip, variations in absorbed dose near interfaces of the thin insulation and metallized layers on or near the front surface of the chip, and changes in radiation energy spectrum due to scattered radiation from adjacent hardware.

10.5.4   These absorbed dose interpretations are valid only if the ratios of the energy absorption coefficients and stopping powers of silicon relative to the TLD are fairly constant over a significant range of the incident photon or electron spectra. Otherwise, the incident energy spectra must be known and the uncertainty in the results of the absorbed dose conversion depends on the accuracy of the spectra data.

11. Report of results

11.1 Reports of radiation-hardness testing of electronic devices should include information that fully describes the following:

11.1.1 The TLD system employed should be given, including the type and physical form of the TLDs, the type of TLD reader, and the annealing procedure used, if any.

11.1.2 The results of all performance tests carried out or reference to relevant published studies of the TLD system should be given. Such test results should include, as a minimum, the sample size, the mean value of the sample responses, the absorbed-dose level, and the standard deviation of the sample response distribution.

11.1.3 The procedure for calibrating the absorbed-dose response of the TLD system should be described, including the radiation source type, irradiation geometry, and conditions (e.g., absorbed-dose level, absorbed-dose rate, and equilibrium material).

11.1.4 A description of the radiation-hardness-test irradiations should be given, including radiation source type, geometry, and conditions, (e.g., absorbed-dose level, absorbed-dose rate, and equilibrium material) as well as any useful supplemental data (e.g., energy spectra).

11.1.5 A description of the conversion of TLD response to absorbed dose in the material of interest should be given, including calibration factors, correction factors, and absorbed-dose conversion factors. The absorbed-dose conversion factors would include information such as the radiation absorption characteristics of the material of interest and assumptions or data about the source energy spectrum.

11.1.6 An estimate of the overall uncertainty of the results should be given as well as an error analysis of the factors contributing to the random and systematic uncertainties. (For an example, see A1.6 of Appendix A1.)

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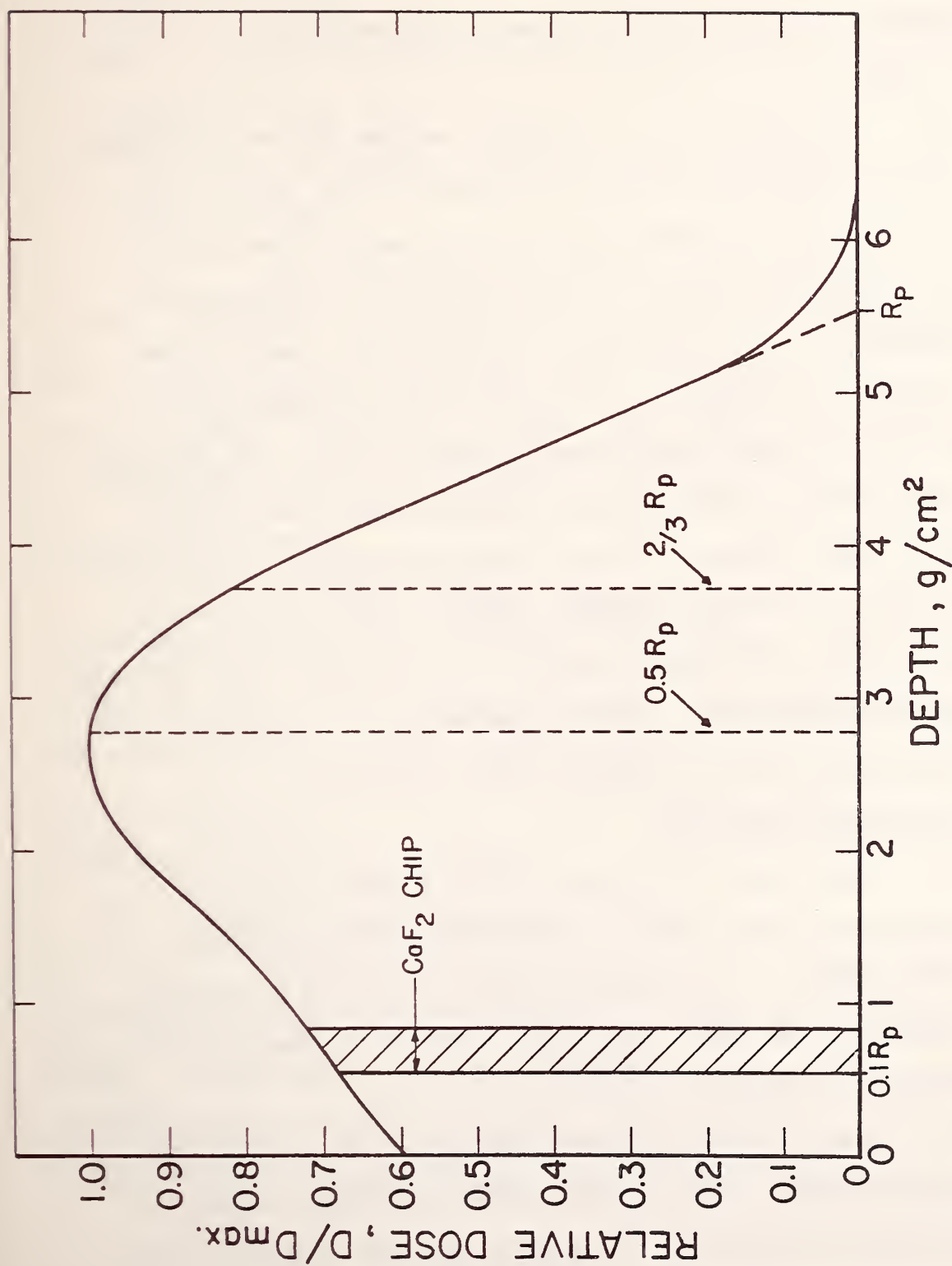   Institute of Radiology, London, 1972.

Fig. 1. Typical depth-dose curve for broad-beam 10-MeV electrons incident on aluminum. R_p is the practical (extrapolated) range. Shaded area illustrates a CaF_2 TLD of 0.089-cm thickness.

APPENDIXES

A1. Recommended Procedures for Application of CaF_2:Mn Chips

A1.1 Scope

A1.1.1   The procedures in this Appendix address the use of manganese doped calcium 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-hardness testing. Each type and form of TLD requires a somewhat different application procedure. See references 10-12 for descriptions of various types of TLDs. CaF_2:Mn 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 silicon, a simple annealing schedule (compared to LiF), ease of handling compared to powders, and relatively linear absorbed-dose response characteristics. The only disadvantage in using CaF_2:Mn TLDs is a moderate fading of the TLD response after irradiation.

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. The recommended handling tool is a vacuum pen; however, tweezers may be used. The contact points of all handling tools should be coated with Teflon if possible.

A1.2.2   Keep the chips as clean as possible at all times so that cleaning can be avoided. Clean the chips only if absolutely necessary since the process can contribute to the aging (decrease in sensitivity) of the phosphor. If cleaning is necessary, the following procedure may be used:

1. bathe the chips in cool trichloroethylene for about 10 minutes in an ultrasonic cleaner,

2. bathe the chips in distilled water for about 10 minutes in the ultrasonic cleaner, and

3. dry the chips as rapidly as possible (dry nitrogen may be used to hasten the drying).

A1.2.3   Anneal the chips for one hour at 500°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. Platinum-plated silver or pyrex glass should be satisfactory.

A1.2.4   For photon irradiation, encapsulate the chips so as to provide electron equilibrium conditions in the dosimeter. See 9.4 and Appendix A3. The required thickness of material surrounding the chip is approximately equal to the range of the highest energy secondary electrons that are generated by the incident photons. See 10.2.2 for encapsulation of the chips for electron irradiation.

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. The dosimeters should be protected from uv light and elevated temperatures during storage or transit as much as possible. 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 by the procedures of Section 8. Changes in humidity have not been shown to affect the response of CaF_2:Mn chips.

A1.4 Irradiation procedures

Procedures for using the TLDs during calibration or test irradiations depend on conditions within each individual facility and on the requirements of the radiation-hardness tests. However, precautions on handling, exposure to light, and exposure to temperature variations apply. The procedures described in Sections 9 and 10 are applicable.

A1.5 Readout

A1.5.1 Pre-readout cleaning of the chips should be done only if necessary (see A1.2.2). Some types of TLDs, such as LiF, may require pre-readout annealing. This is not required for CaF_2:Mn.

A1.5.2 A heating rate of approximately 30°C/s should be satisfactory. The TLD chips should have been heated to a temperature of at least 450°C and preferably to 500°C at the end of the heating cycle.

A1.5.3 In general, the preferred measure of the TLD response is the peak height of the light output vs. temperature curve. However, the integrated light output is usually more conveniently obtained and is satisfactory in most cases. When trade-offs are to be made with regard to efficiency and accuracy, experience with a particular dosimeter and reader combination usually determines which parameter gives the most satisfactory measure of TLD response.

A1.5.4   Most TLD readers are furnished with some type of check 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 calibrated TLDs, as described in 10.3, during each readout session also is recommended.

A1.6 Precision and accuracy

A1.6.1   An example of the error analysis of a typical $CaF_2:Mn$ chip system employed in radiation-hardness testing is given in Tables A1.1 and A1.2. These tables identify the sources of error and give estimated magnitudes of the upper bounds of the errors. A basic assumption for these data is that the TLD system has been characterized and used in accordance with the recommended procedures in this standard. Therefore, as pointed out in a footnote in Table A1.1, certain potential sources of error are expected to be insignificant in this case.

A1.6.2   Table A1.1 lists systematic errors and Table A1.2 lists random errors. The systematic errors are estimates of the upper limits of the errors for the particular factors identified. Since, by their very nature, systematic errors cannot be known with great accuracy, they are estimated from observation of the long-term behavior of a given TLD system. On the other hand, random errors are obtained by standard statistical techniques. The values given in Table A1.2 are equal to one standard deviation (σ) of a batch or individual TLD response distribution.

A1.6.3   A further distinction is made in the analysis between whether the absorbed dose is determined from a TLD system utilizing dosimeters in an individual mode or in a batch mode. The difference between individual and batch mode is discussed in Section 8.

A1.6.4   A universally accepted procedure for combining syste-
matic errors does not exist. Generally, these errors are combined
either by simple addition or by a combination in quadrature (i.e., the
square root of the sum of the squares). In this analysis, the syste-
matic errors in Table A1.1 are combined in quadrature and the result
is given as the total systematic uncertainty, E_s. Whatever method of
combining errors is used should always be reported in the radiation-
hardness test results.

A1.6.5   The random errors listed in Table A1.2 are combined in
quadrature and the result given as a value of σ_T. For the purposes of
this analysis, five dosimeters are assumed to be used in a specific radi-
ation hardness test. In this case, a standard error of the mean (SEOM)
of the absorbed-dose response of the five dosimeters is found by dividing
the combined standard deviation, σ_T, by the square root of the number, n,
of dosimeters employed; that is

$$SEOM = \frac{\sigma_T}{\sqrt{n}}$$

The total random uncertainty is taken to be equal to three times the
SEOM.

A1.6.6   The overall uncertainty of the mean absorbed dose de-
termined by five dosimeters for the conditions described is taken as
the algebraic sum of the total systematic uncertainty, E_s, and the
total random uncertainty, E_R. For this example, the overall uncertainty
is equal to the following:

(for individual dosimeters), $E_s + E_R = 6.7\%$

(for batch), $E_s + E_R = 13\%$

Table A1.1

Estimates of Systematic Uncertainties for Typical CaF$_2$:Mn Chip System

Source of Systematic Error	Individual Dosimeters	Batch
1. ^{60}Co source calibration	3%	3%
2. TLD absorbed dose calibration		
a. Determination of calibration curve	1%	2%
b. Conversion of exposure to dose in TLD	2%	2%
3. Time between irradiation and readout: fading correction factor	1%	3%
4. Conversion of dose in TLD to dose in Si for device test irradiation	2%	2%
5. Correction for attenuation in equilibrium material	2%	2%
6. Absorbed dose rate dependence	*	*
7. Energy dependence	*	*
8. Time between preparation and readout	*	*
9. Directional dependence	*	*
10. Temperature before, during, and after irradiation	*	*
11. Humidity dependence	*	*
12. Effect of size of TLD	*	*
Total systematic uncertainty, all errors combined in quadrature, E$_s$	4.8%	5.8%

* For purposes of this error analysis, it is assumed that the TLD system is utilized in such a way as to make these errors negligible. However, this assumption is not valid under all conditions of radiation-hardness testing. A careful examination of all possible sources of error must be made for the irradiation conditions and TLD system employed in each specific test.

Table A1.2

Estimates of Random Uncertainties for Typical CaF_2:Mn Chip System

Sources of Random Error	Individual Dosimeters	Batch
1. Reproducibility of individual dosimeter response, σ	1%	
2. Correction for sensitivity variation between dosimeters, σ	1%	
3. Uniformity of batch response, σ		5%
Total, combined in quadrature, σ_T	1.4%	5%
Standard error of mean of dose response of five dosimeters, $\sigma_T/\sqrt{n}$	0.63%	2.2%
Total random uncertainty, $E_R = 3(\sigma_T/\sqrt{n})$	1.9%	6.7%

A2. Energy Absorption Coefficients and Collision Stopping Powers

A2.1 Values of photon mass energy absorption coefficients and electron mass collision stopping powers for several materials of interest in radiation-hardness testing are shown in Table A2.1. All values for the energy absorption coefficients are derived from reference 13, except those values for air that are from reference 14. Values for the stopping powers are from reference 15. Energy absorption co-efficient values for chemical compounds not listed directly were evaluated from the coefficients μ_i/ρ_i for the constituent elements according to the weighted average

$$\mu/\rho = \sum_i w_i\left(\mu_i/\rho_i\right)$$

where w_i is the proportion by weight of the i-th constituent [14]. Ratios of the energy absorption coefficients for the various materials in Table A2.1 relative to silicon as a function of incident photon energy are shown in Figure A2.1. Similarly, ratios of stopping powers are shown in Figure A2.2.

Table A2.1.   Mass Energy Absorption Coefficients: μ_{en}/ρ (cm²/g) and
Mass Collision Stopping Powers: S/ρ (MeV·cm²/g)

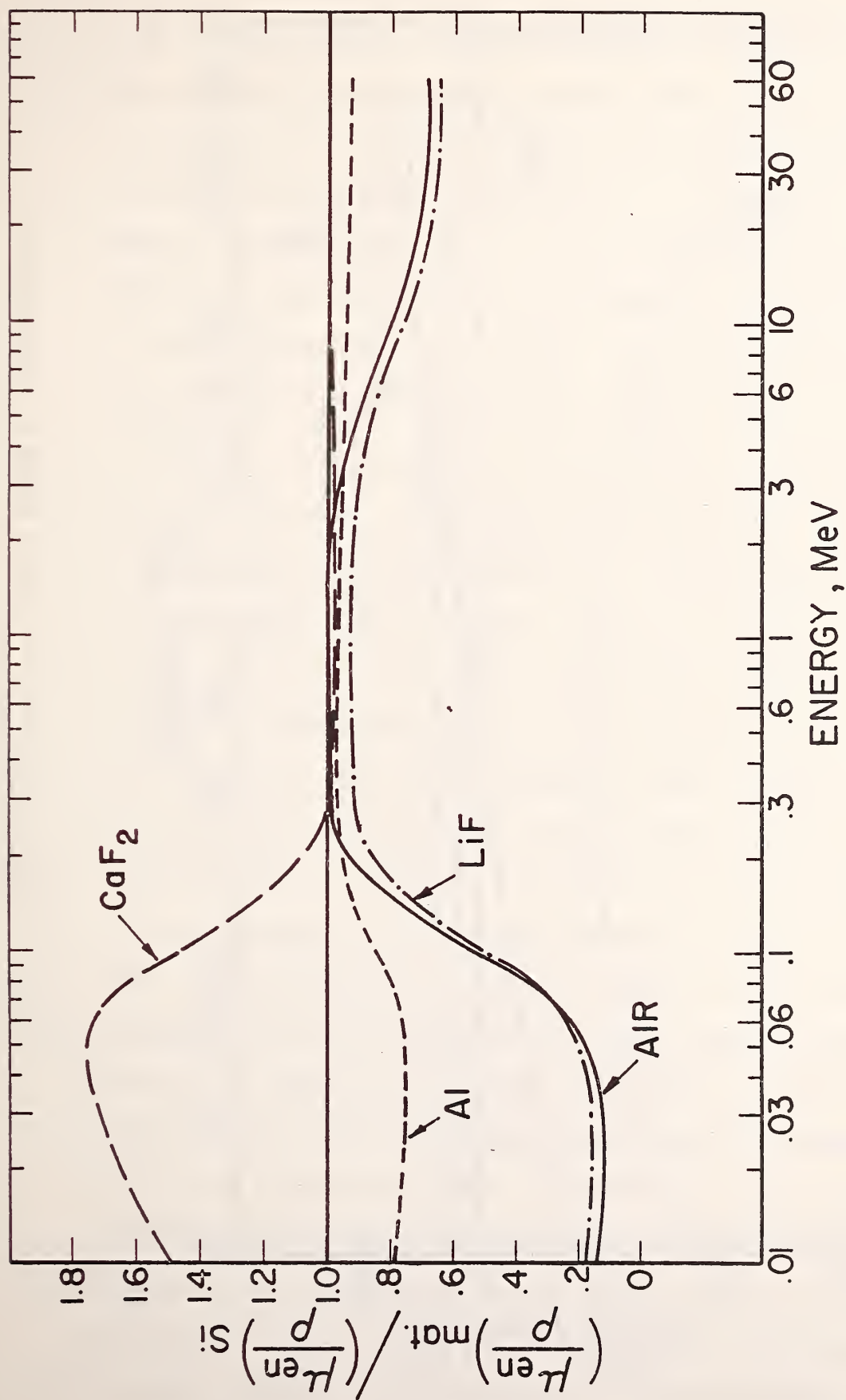| Energy (MeV)	Air		LiF		CaF$_2$		Al		Si	
	μ_{en}/ρ	S/ρ	μ_{en}/ρ	S/ρ	μ_{en}/ρ	S/ρ	μ_{en}/ρ	S/ρ	μ_{en}/ρ	S/ρ
0.01	4.61	19.7	5.74	18.2	48.3	16.6	25.4	16.4	32.4	16.9
0.02	0.511	11.5	0.632	10.7	6.58	9.93	3.06	9.79	3.99	10.1
0.04	0.0669	6.83	0.0769	6.31	0.819	5.96	0.353	5.88	0.470	6.07
0.06	0.0305	5.10	0.0317	4.71	0.247	4.48	0.108	4.42	0.142	4.56
0.08	0.0243	4.19	0.0234	3.87	0.111	3.70	0.0542	3.64	0.0682	3.76
0.1	0.0234	3.62	0.0219	3.35	0.0658	3.21	0.0373	3.16	0.0442	3.27
0.2	0.0268	2.46	0.0247	2.28	0.0309	2.20	0.0272	2.16	0.0287	2.24
0.4	0.0295	1.90	0.0273	1.74	0.0293	1.71	0.0286	1.67	0.0298	1.73
0.6	0.0295	1.74	0.0272	1.59	0.0289	1.57	0.0284	1.53	0.0296	1.58
0.8	0.0289	1.68	0.0266	1.52	0.0279	1.51	0.0277	1.48	0.0285	1.53
1.0	0.0278	1.66	0.0257	1.49	0.0271	1.49	0.0268	1.46	0.0277	1.51
2.0	0.0234	1.68	0.0217	1.48	0.0231	1.49	0.0225	1.47	0.0236	1.52
4.0	0.0186	1.79	0.0174	1.52	0.0194	1.56	0.0188	1.54	0.0197	1.60
6.0	0.0164	1.87	0.0153	1.55	0.0182	1.61	0.0175	1.58	0.0185	1.65
8.0	0.0152	1.93	0.0142	1.58	0.0178	1.64	0.0170	1.62	0.0180	1.69
10	0.0145	1.98	0.0137	1.60	0.0177	1.67	0.0168	1.64	0.0179	1.72
20	0.0131	2.13	0.0123	1.66	0.0180	1.75	0.0167	1.71	0.0180	1.80
40	0.0124	2.25	0.0119	1.72	0.0181	1.83	0.0168	1.77	0.0182	1.87
60	0.0122	2.31	0.0116	1.75	0.0177	1.87	0.0165	1.81	0.0178	1.90

Fig. A2.1. Ratios of mass energy absorption coefficients of various materials relative to silicon.
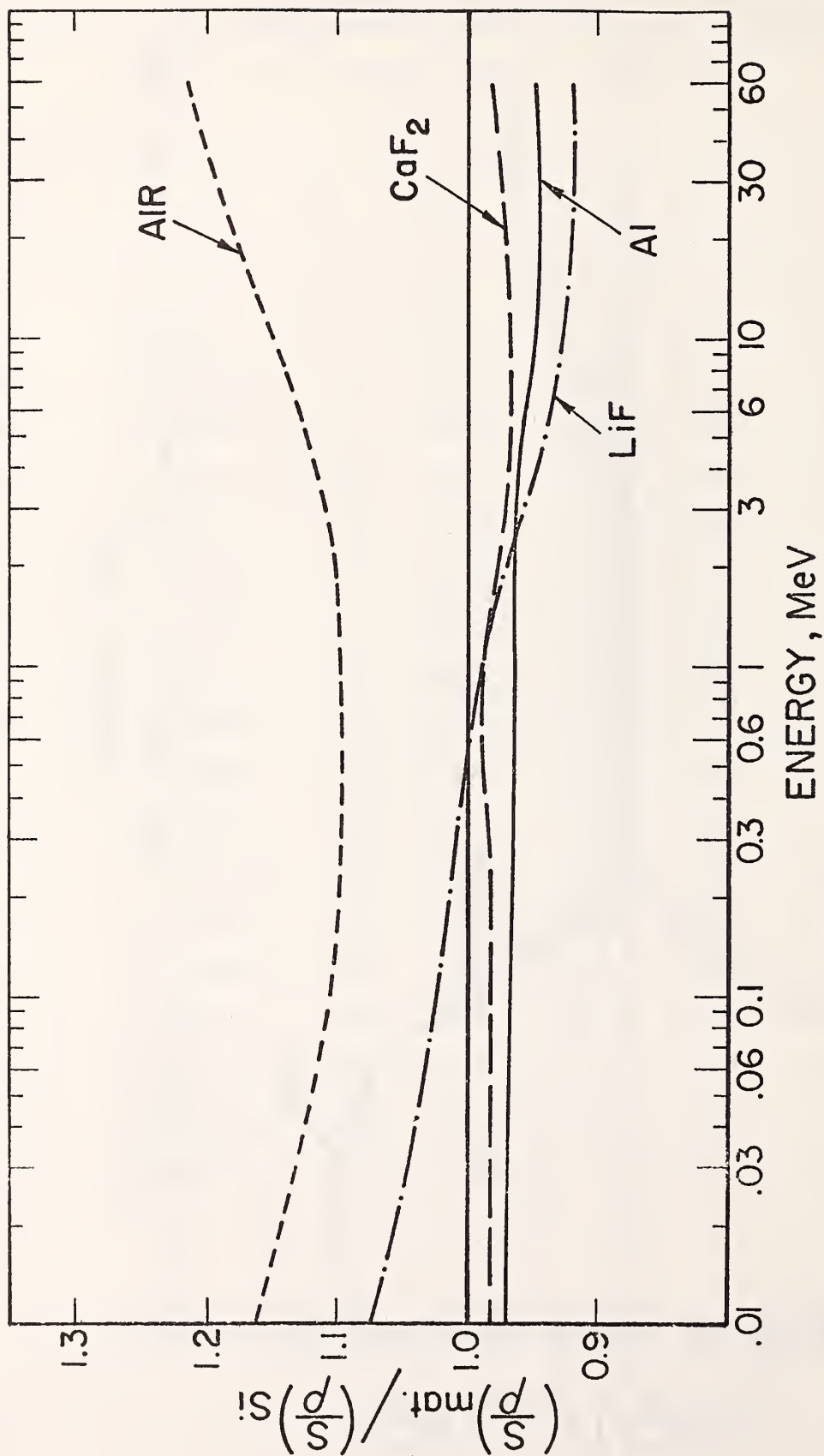
Fig. A2.2. Ratios of mass collision stopping powers of various materials relative to silicon.

A3.   Determination of Electron Equilibrium Thickness

A3.1   When a material is irradiated by a photon beam, secondary electrons
are generated in the material by interaction of the photons with
the atoms of the material. At some depth in the material, the
number of secondary electrons of a given energy entering a small
volume of the material is equal to the number of secondary electrons
of the same energy leaving the volume. Within that volume, electron
(charged particle) equilibrium is said to exist [1,16].

A3.2   The thickness of material required to approximate electron equilibrium
is equal to the range of the maximum-energy secondary electron that
can be generated by the primary photons. This thickness as a function
of maximum photon energy is shown as curve A in Fig. A3.1 [17]. It
has been found that for all practical purposes electron equilibrium
is achieved within a few percent of its true condition by a thick-
ness considerably less than the maximum secondary electron range
[18,19]. This lesser thickness is given by curve B of Fig. A3.1
and approximately corresponds to the depth at which the peak of the
depth vs. absorbed dose buildup curve occurs for a given incident photon
energy spectrum [18,20]. It should be noted that curve B has been
determined from data for bremsstrahlung beams with broad energy
spectra. The depth of this absorbed-dose peak to some extent
depends on the incident photon energy spectrum and the determina-
tion of that depth on the method of measurement. Thus, it should be
determined experimentally for a particular radiation source.

A3.3   Obviously, it is an advantage to use the least amount of material
practical to achieve equilibrium conditions since the intensity of
the primary photons is attenuated by this material thickness.

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Correction should be made for this attenuation (as in 9.5) since the dose is being determined for the photon fluence at the point of measurement.

A3.4   A significant error in absorbed-dose determination can occur if the thickness given by curve B is used when an appreciable number of near-maximum-energy secondary electrons are generated by the primary photon beam <u>outside</u> the material of interest. These electrons might come directly from an x-ray converter or from direct interaction of the primary photon beam with collimators or other material structures within the vicinity of the measurement area. One method of removing such unwanted electrons from the photon beam would be the use of a transverse magnetic field. However, if this technique is not practical, and it is known or suspected that the primary photon beam contains a significant number of high-energy secondary electrons, then the minimum equilibrium thickness chosen should be equal to the secondary electron range given by curve A of Fig. A3.1.

NOTE A3.1   Fig. A3.1 is based on data calculated or experimentally determined for water. However, equilibrium thickness values obtained from these curves should be within 25% of the thicknesses required for most materials of low to medium atomic number (up to Z=26).
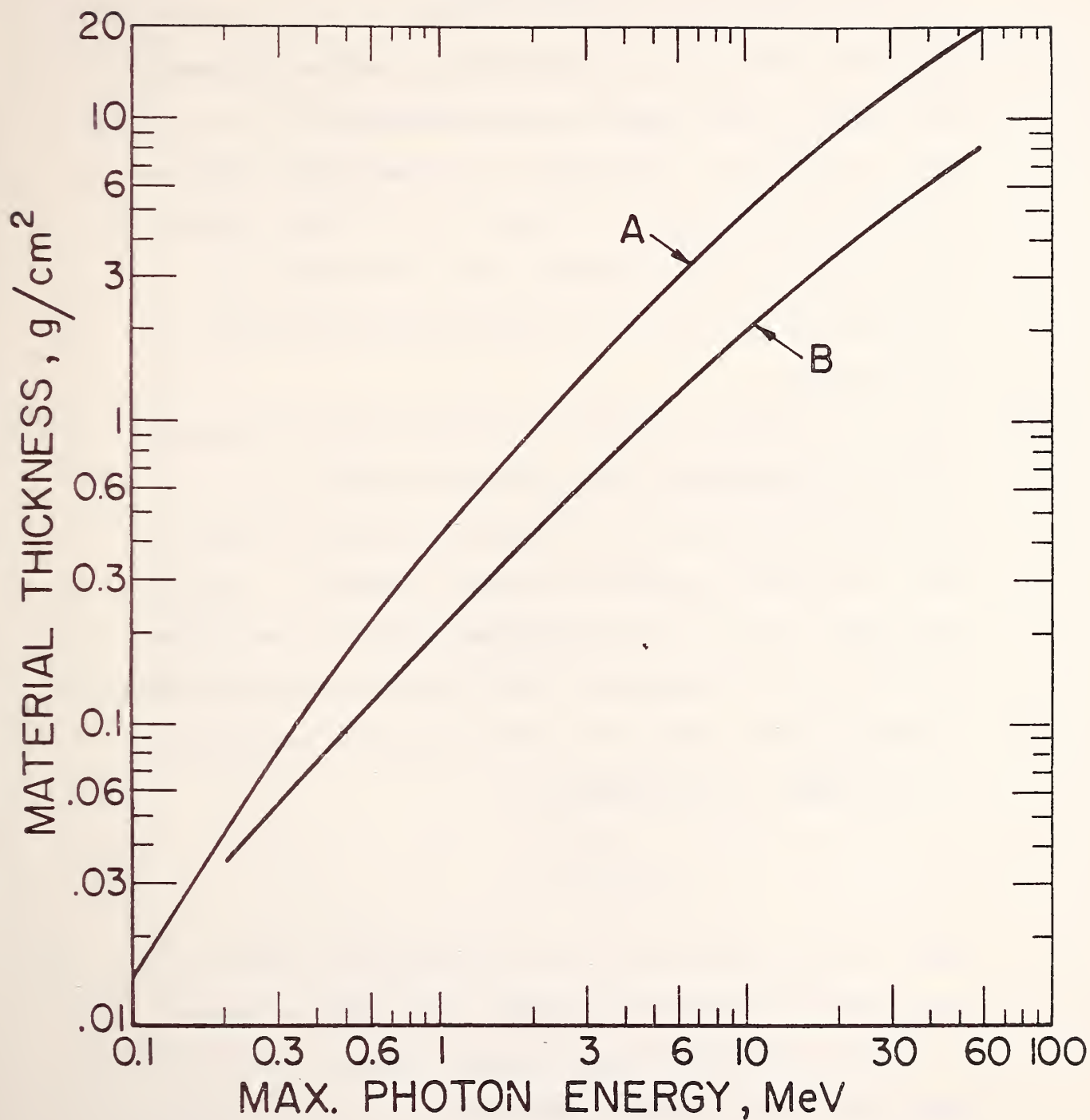
Fig. A3.1 Material thickness required for electron equilibrium. A: electron range. B: depth of peak dose.

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A4. Determination of Test Sample Size

A4.1   The number of TLDs (i.e., the sample size n) required for each
test group in 8.3.3 is based on a two-sided t-test to
detect a difference δ between means of two test groups with a
confidence level of 95% and a probability of failing to detect
such a difference of 0.50 (see Sec. 3-3.1.1 of reference 2).
The graph of n vs. d in Fig. A4.1 was derived from table A-8
of reference 2.

A4.2   The number of TLDs, n, required to estimate the mean TLD response
at a given absorbed-dose level as described in 9.3.1 is
based on the determination of a two-sided confidence interval
that is expected to bracket the true mean response, m, $100(1-\alpha)$%
of the time.   In this case, the confidence level has been chosen
as 95% (i.e., $1-\alpha$ = 0.95 and α = 0.05) and the confidence interval
has been assigned a value of d = ±5% of the sample mean response,
$\overline{Y}_o$.   The number of TLDs required is

$$n = \frac{t^2 \ s^2}{d^2} \quad ,$$

where t is the percentile of the t distribution at a 95% confi-
dence level for 29 degrees of freedom.   This number of degrees
of freedom is determined from the number of samples used for ob-
taining the estimated standard deviation, s, in 8.1.1   (see
sec. 2-3.2 of reference 2).

A4.3   The statistical test methods included here are those generally
accepted for product testing.   The significance levels chosen are
somewhat arbitrary but were selected on the basis of being

adequate for the performance tests specified. Other more or less
stringent acceptable statistical requirements should be assigned
upon practical assessment of the overall objectives of the hard-
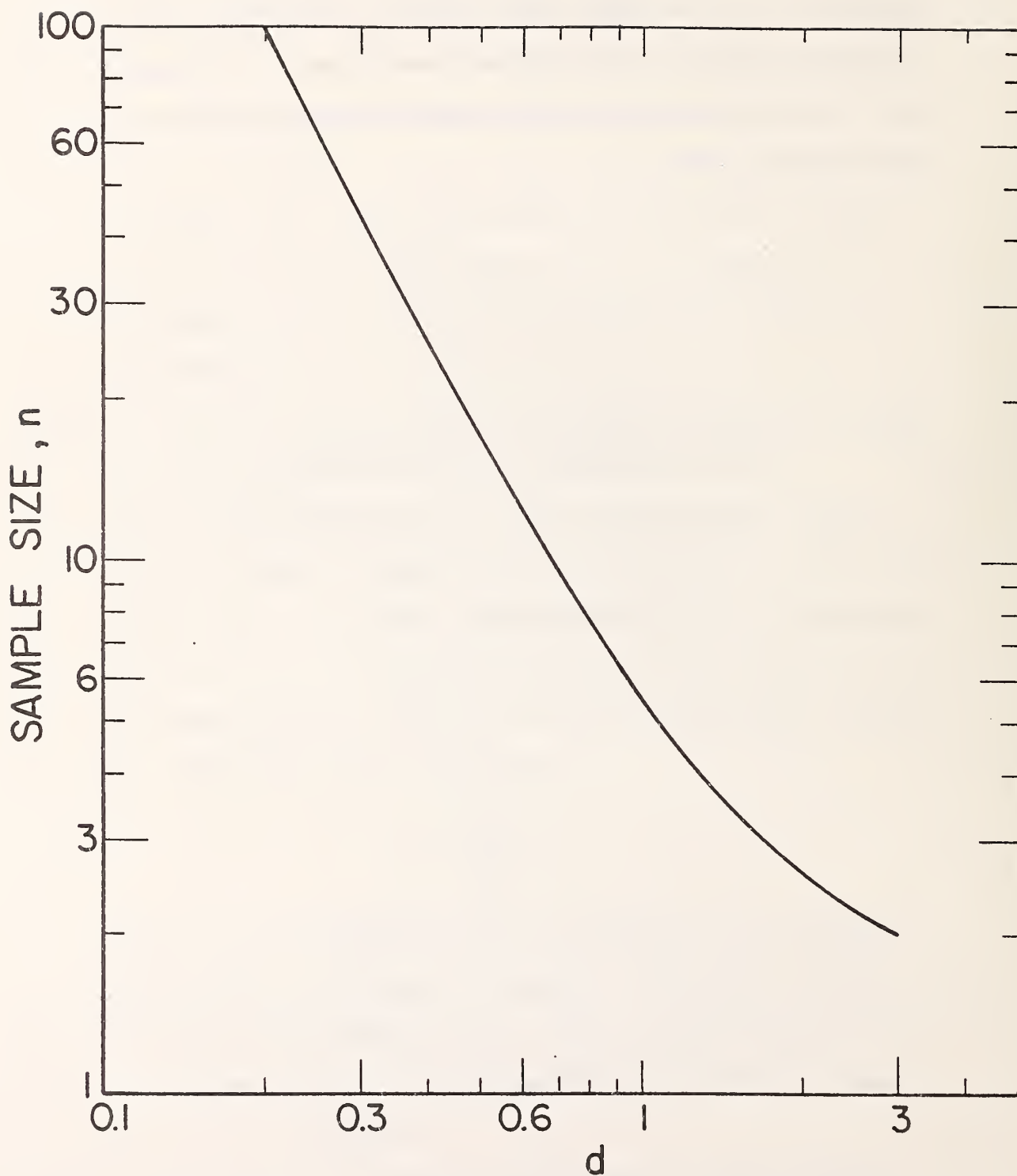ness assurance tests.

Fig. A4.1 Sample size required to detect
difference of two means.

$$d = \frac{|m_A - m_B|}{\sqrt{2\,\sigma^2}}$$

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15. SUPPLEMENTARY NOTES

16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

A standard recommended practice for the use of thermoluminescence dosimetry (TLD) systems has been developed. It is intended for the use of facilities that are engaged in the radiation-hardness testing of electronic components and systems. The practice has been written in the form of an ASTM standard for determination of absorbed dose in a medium as a result of photon or electron irradiation. It covers various aspects of TLD systems such as performance testing, dose calibration, and use procedures and precautions. A selected bibliography of TLD characteristics is included.

17. KEY WORDS (six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)

Electronic devices; electrons, photons, radiation hardness; standard practice; thermoluminescence dosimeters

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