INTERNATIONAL STANDARD

ISO/ASTM 51650

Second edition
2005-05-15

Practice for use of a cellulose triacetate dosimetry system

Pratique de l'utilisation d'un système dosimétrique au triacétate de cellulose

Reference number
ISO/ASTM 51650:2005(E)

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Published in the United States

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Foreword

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

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

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

A project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this project, ASTM Subcommittee E10.01, Dosimetry for Radiation Processing, is responsible for the development and maintenance of these dosimetry standards with unrestricted participation and input from appropriate ISO member bodies.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. Neither ISO nor ASTM International shall be held responsible for identifying any or all such patent rights.

International Standard ISO/ASTM 51650 was developed by ASTM Committee E10, Nuclear Technology and Applications, through Subcommittee E10.01, and by Technical Committee ISO/TC 85, Nuclear energy.

This second edition cancels and replaces the first edition (ISO/ASTM 51650:2002), which has been technically revised.

Standard Practice for Use of a Cellulose Triacetate Dosimetry System[1]

This standard is issued under the fixed designation ISO/ASTM 51650; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision.

1. Scope

1.1 This practice covers procedures for using the cellulose triacetate (CTA) dosimetry system for measuring absorbed dose and dose profile in materials irradiated by electrons and photons in terms of absorbed dose to water. The CTA dosimeter is a routine dosimeter especially useful for measurement of dose distribution.

NOTE 1—Cellulose triacetate dosimeter refers to untinted film dosimeter.

1.2 This practice applies provided the following conditions are satisfied.

1.2.1 The absorbed-dose range is 10 kGy to 300 kGy for electrons and photons.

1.2.2 The absorbed-dose rate range is 3 Gy/s to 4×10^{10} Gy/s (**1**).[2]

1.2.3 The radiation-energy range for electrons is 0.2 to 50 MeV.

1.2.4 The radiation-energy range for photons is 0.1 to 50 MeV.

1.2.5 The irradiation-temperature range of the dosimeter is −10 to 70°C.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced documents

2.1 *ASTM Standards:* [3]

E 170 Terminology Relating to Radiation Measurements and Dosimetry

E 177 Practice for Use of the Terms Precision and Accuracy as Applied to Measurement of a Property of a Material

E 178 Practice for Dealing With Outlying Observations

E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers

E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers

2.2 *ISO/ASTM Standards:*[3]

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

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

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

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

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

ICRU Report 17 Radiation Dosimetry: X Rays Generated at Potentials of 5 to 150 kV

ICRU Report 34 The Dosimetry of Pulsed Radiation

ICRU Report 35 Radiation Dosimetry: Electron Beams with Energies Between 1 and 50 MeV

ICRU Report 37 Stopping Powers for Electrons and Positrons

ICRU Report 60 Fundamental Quantities and Units for Ionizing Radiation

3. Terminology

3.1 *Definitions:*

3.1.1 *absorbed-dose mapping*—measurement of absorbed dose within a process load using dosimeters placed at specified locations to produce a one-, two- or three-dimensional distribution of absorbed dose, thus rendering a map of absorbed-dose values.

3.1.1.1 *Discussion*—The CTA dosimeter strip with appropriate length provides continuous measurement of one-dimensional dose distribution.

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

$$\dot{D} = \frac{dD}{dt} \tag{1}$$

Unit: $\text{Gy} \cdot \text{s}^{-1}$.

3.1.2.1 *Discussion*—(1) The absorbed-dose rate is often specified in terms of its average value over longer time intervals, for example, in units of $\text{Gy} \cdot \text{min}^{-1}$ or $\text{Gy} \cdot \text{h}^{-1}$. (2) In

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

Current edition approved by ASTM June 1, 2004. Published May 15, 2005. Originally published as ASTM E 1650–94 with title: Practice for Use of Cellulose Acetate Dosimetry Systems. ASTM E 1650–94 was adopted by ISO in 1998 with the intermediate designation ISO 15570:1998(E). The present International Standard ISO/ASTM 51650:2005(E) is a major revision of the last previous edition ISO/ASTM 51650:2002(E), which replaced ISO 15570.

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

[3] For referenced ASTM and ISO/ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

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

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

3.1.3 *analysis wavelength*—wavelength used in a spectrophotometric instrument for the measurement of optical absorbance or reflectance.

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

3.1.5 *cellulose triacetate dosimeter*—untinted CTA film that undergoes change in optical absorbance under irradiation.

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

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

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

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

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

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

3.1.11 *measurement traceability*—ability to demonstrate by means of an unbroken chain of comparisons that a measurement is in agreement within acceptable limits of uncertainty with comparable nationally or internationally recognized standards.

3.1.12 *net absorbance* (Δ*A*)—change in measured optical absorbance at a selected wavelength determined as the absolute difference between the pre-irradiation absorbance, A_0, and the post-irradiation absorbance, *A* as follows:

$$\Delta A = |A - A_0| \tag{2}$$

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

3.1.14 *specific net absorbance* (Δ*k*)—net absorbance, ΔA_λ, at a selected wavelength, λ, divided by the optical pathlength, *d*, through the dosimeter as follows:

$$\Delta k = \Delta A_\lambda / d \tag{3}$$

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

3.1.16 *traceability*—see *measurement traceability*.

3.1.17 Definitions of other terms used in this practice that pertain to radiation measurement and dosimetry may be found in Terminology E 170. Definitions in E 170 are compatible with ICRU 60; that document, therefore, may be used as an alternative reference.

4. Significance and use

4.1 The cellulose triacetate (CTA) dosimetry system provides a means for measuring absorbed dose in materials **(2-15)**. The dosimeter is a film containing cellulose triacetate and plasticizer. The dosimetry is based on chemical reactions in the film resulting in changes in the optical absorption properties in near ultraviolet region. The absorbance is measured at a specific wavelength using a spectrophotometer or equivalent photometric instruments.

4.2 Absorbed dose is evaluated by use of a calibration curve or response function traceable to nationally or internationally recognized standards.

4.3 Absorbed dose is usually specified in terms of absorbed dose to water. Absorbed dose to other materials may be evaluated by applying the conversion factors discussed in ISO/ASTM Guide 51261.

NOTE 2—For a comprehensive discussion of various dosimetry methods applicable to the radiation types and energies discussed in this practice, see ICRU Reports 14, 17, 34, 35, and 37.

4.4 This dosimetry system may be used in industrial radiation processing of various products, for example radiation effect tests, polymer modifications, and sterilization of medical devices.

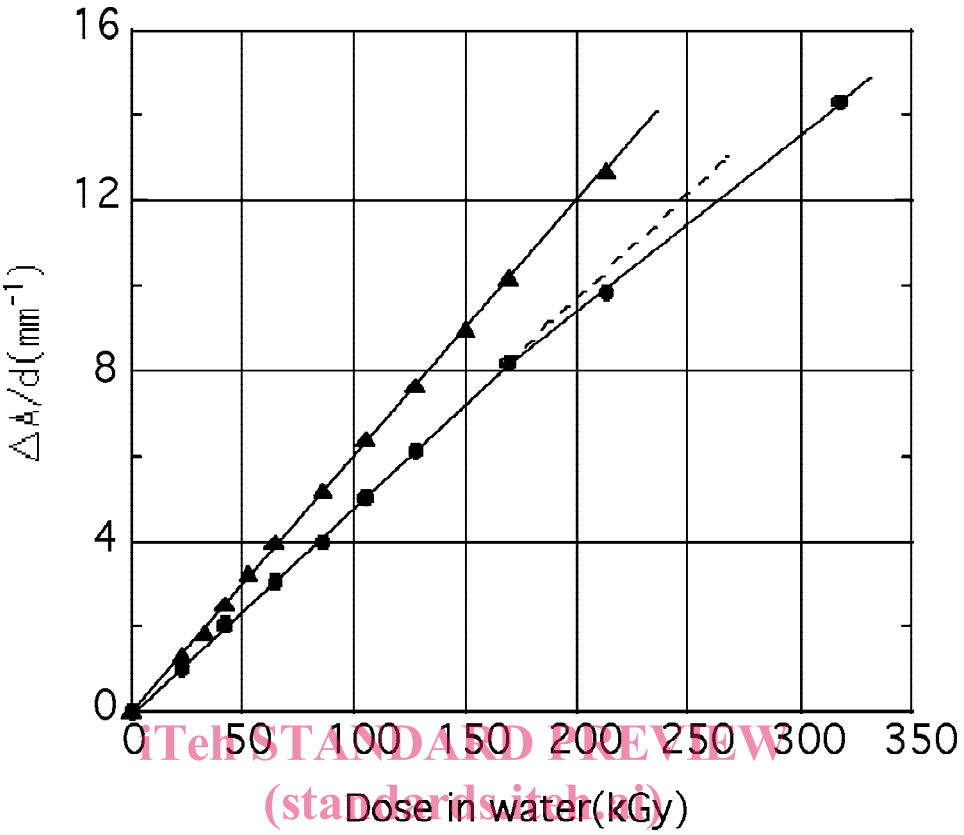
4.5 A one-dimensional absorbed-dose mapping on or within a product may be obtained by irradiating a dosimeter strip of appropriate length.

4.6 The absorbed-dose range indicated in 1.2.1 may be achieved by using triphenylphosphate (TPP) as the sole plasticizer in the dosimeter.

4.7 The effect on the dosimeter response due to changes in the irradiation conditions, such as absorbed-dose rate, temperature, humidity, and atmosphere should be considered when these are different from the calibration conditions.

4.7.1 The dosimeter has different responses to electron beams at relatively high dose rates than to gamma radiation at relatively low dose rates (see Fig. 1). Calibration should be carried out separately for each type of radiation.

4.7.2 The dosimeter response increases linearly with temperature from –10 to 60°C at 7 kGy/h, and from –10 to 40°C at 1.2 kGy/h **(7)**, and with relative humidity (up to 80 %) during irradiation when irradiated at lower dose rates (<10 kGy/h) typical of gamma irradiators. The effects are not appreciable at higher absorbed-dose rates (>100 kGy/h). All these effects need to be considered before the dosimeter can be used routinely for processing **(4,7,12,15)**. For high-dose-rate dosimetry (for example, electron beams) the influence of humidity on dosimeter response is not appreciable **(4,7)**. For other conditions it is recommended to calibrate the dosimetry system by the final user under specific environmental conditions.



▲ Gamma-rays (dose rate: 10^4 Gy/h, temperature: 25° C, in air, relative humidity: 50 to 60 %)
● Electron beam average dose rate: 10^7 Gy/h, temperature: 15°C, in air, relative humidity: (60 %)

FIG. 1 The relation between specific net absorbance ΔA /d at 280 nm and absorbed dose in water for 1 MeV electron beam and ^{60}Co gamma-ray irradiation

5. Instrument required

5.1 *Components of Dosimetry System*—The following shall be used to determine absorbed dose with the film dosimetry system.

5.1.1 *Cellulose Triacetate Dosimeter*—The CTA dosimeters are in the form of pieces and strips with appropriate width, length and thickness. For most applications, the width and the thickness of the dosimeter are 8 mm and 0.125 mm, respectively.

5.1.2 *Spectrophotometer* (or equivalent instrument), capable of measuring optical absorbance values up to 2 with an uncertainty of no more than ±1 % at the analysis wavelength (280 nm) and having documentation covering analysis wavelength range, absorbance determination, spectral bandwidth and the reproducibility (See ASTM Practices E 925 and E 958).

5.1.3 *Film Holder* (or equivalent device), to position the dosimeter piece reproducibly in, and perpendicular to the analysis light path. A built-in automatic dosimeter strip feeder with the same capability as the static film holder is applicable for automatic one-dimensional dose distribution measurement.

5.1.4 *Thickness Gauge*, calibrated with a precision of ±1 μm for the typical dosimeter having nominal 125 μm thickness.

6. Preparation of dosimeter

6.1 The dosimeter strip of 8 mm width and 100 m length rolled on a spool is commercially available, and is described in the informative annex of this practice.

6.2 In-house preparation has an advantage that the film thickness can be adjusted according to the intended application. In making long dosimeter strips, which is required for dose mapping, the uniformity in thickness is necessary.

6.3 The dosimeter can be cast by pouring a prescribed recipe solution consisting of cellulose triacetate, plasticizer, and solvent onto an optically flat horizontal plate and evaporating the solvent slowly. The thickness of the film can be controlled by the concentration of solutes or by the amount of solution poured on to a given area of the horizontal plate.

6.4 The recommended recipe is 70 to 85 weight % of cellulose triacetate and balance of triphenylphosphate (TPP) as a sole plasticizer, and solvents, for example, methylenechloride-methanol mixture **(13)**.

7. Calibration of the dosimetry system

7.1 The dosimetry system shall be calibrated prior to use and at intervals thereafter in accordance with the user's documented procedure that specifies details of the calibration

process and quality assurance requirements. Calibration requirements are given in ISO/ASTM Guide 51261.

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

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

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

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

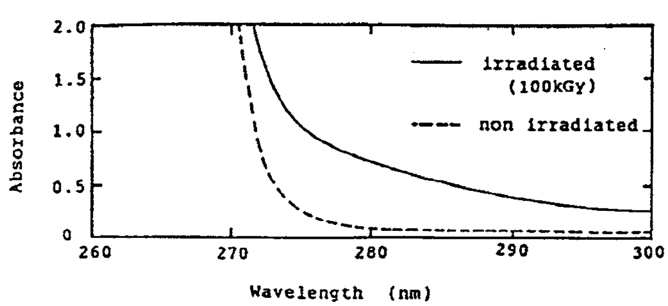
7.3 *Measurement Instrument Calibration and Performance Verification*—For the calibration of the instruments, and for the verification of instrument performance between calibrations, see ISO/ASTM Guide 51261 and/or instrument-specific operating manuals.

8. Measurement

8.1 *Analysis Wavelength Selection*—Use a 280 nm light-beam for A and A_o measurements (see Fig. 2). This wavelength is chosen due to low absorbance before irradiation and linear absorbance increase with dose. For CTA dosimetry, measurement wavelength affects results markedly, there is a ±3 % change in absorbance per 1 nm change of analysis wavelength. Precise wavelength setting is important.

8.1.1 Set the spectrophotometer to the appropriate wavelength at bandwidth no more than 2 nm, or use photometer equipped with a manganese (Mn) hollow cathode lamp and appropriate band-pass filters.

8.1.2 Set the balance of the spectrophotometer, or photometer equipped with Mn hollow cathode lamp to zero absorbance, without a CTA dosimeter (with only air) in the analysis light path.



NOTE—The suggested wavelength of 280 nm is chosen due to low absorbance before irradiation (A_o), and linear increase in absorbance (A) with dose. (Original drawing by the author of Refs **13** and **16**).

FIG. 2 Absorption spectra before and after irradiation of CTA dosimeter film with 2 MeV electron beam

8.1.3 For dosimeter pieces, insert the non-irradiated dosimeter in the holder and place it in the analysis light path of the spectrophotometer, or equivalent instruments. Measure the absorbance and record this value (A_o). Insert the irradiated dosimeter piece in the light path of spectrophotometer, or the equivalent instruments and measure the absorbance (A). Record this value.

8.1.4 For dosimeter strips, insert an unirradiated dosimeter strip into the built-in automatic dosimeter strip feeder and pass through the analysis light path of the scanning spectrophotometer, or the equivalent instrument. Measure the average value of absorbance as the strip is fed automatically through the analysis light path, and record this average value (A_o). Feed automatically the irradiated dosimeter strip in the analysis light path of the scanning spectrophotometer or the equivalent instruments and record the values of absorbance along the dosimeter strip (A).

NOTE 3—The absorbance first decreases and then slowly increases with storage time longer than fifteen minutes after high dose-rate electron beam irradiation. The readings will become almost stable about 2 h after irradiation. Therefore, it is recommended that the absorbance of the dosimeter be measured at a constant time period, for example, 2 h after the irradiation **(7,14)**.

8.1.5 Measure the thickness, d, of each dosimeter piece or the average thickness, $\bar{d}$ of the dosimeter strip. For dosimeter pieces, calculate specific net absorbance $\Delta A/d$ or $\Delta A/\bar{d}$. For a dosimeter strip, calculate the specific net absorbance along the strip.

NOTE 4—Net absorbance corrected by nominal thickness (quoted by the manufacturer d_n, see Table A1.1), $\Delta A/(d_n/d)$, is often used to calculate dose when the calibration curve is given for the nominal thickness quoted by the manufacturer.

8.1.6 Prepare a calibration curve by plotting specific net absorbance, $\Delta A/d$ versus absorbed dose. Examples of calibration curves are shown in Fig. 1.

8.1.7 Fit the data with an appropriate analytical function that provides an acceptable fit to the data. Linear regression best fits the data in a practically applied absorbed dose range (see Fig. 1).

8.1.8 Calculate the coefficients of variation (CV) of the individual dosimeter readings of the A_o values and the A values at each dose from replicate measurements (see 11.2). Suspected data outliers should be tested and eliminated using statistical procedures found in ASTM Practices E 177 and E 178.

9. General practice

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

9.2 Use the irradiation and measurement procedures in accordance with Sections 7 and 8.

9.3 When using photons (gamma radiation or bremsstrahlung) for calibration, surround the dosimeter with appropriate thickness of CTA–equivalent materials such as polystyrene, or polymethylmethacrylate to achieve approximate electron equilibrium conditions.

9.4 Determine the specific net absorbance of each CTA dosimeter after irradiation, and evaluate the absorbed dose based on the appropriate calibration curve taking into account interferences outlined in Section 10.

10. Environmental and other interferences

10.1 *Effect of Environment During Irradiation*:

10.1.1 The dosimeter response is influenced by dosimeter temperature and humidity especially for gamma irradiation. For the dosimeter in Table A1.1 the temperature coefficient dependence of dosimeter response is for example about +0.5 % per °C (–10 to 60°C) at an absorbed-dose rate of 7 kGy/h and relative humidity of about 60 % for gamma irradiation. The humidity dependence is for example about +0.2 % per 1 % change of relative humidity at dose rates ≤10 kGy/h and in the temperature range of 25 to 40°C **(7,15)**.

10.1.2 The dosimeter response is influenced by absorbed-dose rate. The response changes gradually with the absorbed-dose rate in the range of 10^4 to 10^6 Gy/h **(6)**, while it is mostly constant in the range of 10^6 to 4×10^{10} Gy/h **(1,7,16)**. The difference in response is caused by the difference in dose-rate rather than different types of radiation. As shown in Fig. 3 **(17)**, studies with X-rays (bremsstrahlung) demonstrate that the dosimeter response changes gradually with the absorbed-dose rate in the range of 10^4 to 10^6 Gy/h. The dosimeter response agrees with that for electron beam in the higher dose-rate range and for gamma radiation in the lower dose-rate range.

NOTE 5—The effect of dose rate on the dosimeter response can be eliminated when the dosimeter is irradiated under vacuum or in a nitrogen atmosphere; the dosimeter response at low absorbed-dose rate typically of gamma irradiation is similar to that at high absorbed-dose rate typically of electron beams. The dose rate effect is due to the effect of oxygen and NO_x diffusion into CTA film **(14)**. See Fig. 1.

10.1.3 In case of photon irradiation, surround the CTA dosimeter with a appropriate thickness of CTA-equivalent material, for example, polystyrene or polymethylmethacrylate, to achieve approximate electron equilibrium conditions.

10.2 *Effect of Environment During Storage*:

10.2.1 The dosimeter is insensitive to visible light and intermediate humidity conditions during storage. It is a common practice to store them in a conventional laboratory storage area or in an instrumentation room.

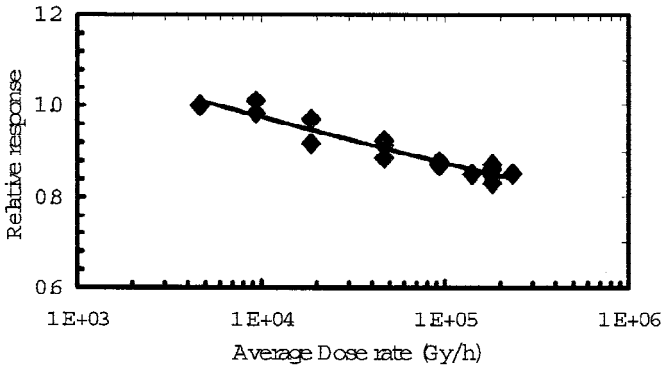


FIG. 3 Relative dose response of CTA dosimeter (FTR-125) in air, as a function of average absorbed-dose rate

10.2.2 The dosimeter material is quite stable under normal conditions. However, exposure to UV light or extreme temperature change should be avoided. Pre-irradiation absorbance A_o may change with time of storage by such influences, mostly for the outer-most layer of the dosimeter strip rolled on a spool, even when the dosimeter has been protected from the environmental influences in the sealed pouch. Measurement of A_o values prior to irradiation is recommended to check for such changes.

10.3 *Handling of Dosimeter*:

10.3.1 The dosimeter film irradiated to high dose exceeding 200 kGy becomes brittle to some degree and has to be handled with care. During the measurement procedures do not touch dosimeter surface with bare fingers to avoid finger-print, dusting, water marks, etc. These kinds of surface contaminations will affect optical density.

11. Minimum documentation required

11.1 *Calibration*:

11.1.1 Record the batch number (code) with dosimeter type.

11.1.2 Record or reference the irradiation date, irradiation temperature, humidity, temperature and humidity variation (if any), radiation source and approximate dose-rate range, and the associated instrumentation used to calibrate the dosimeters.

11.2 *Application*:

11.2.1 Record the date, temperature and humidity, temperature and humidity variation (if any), radiation type, approximate dose-rate range, dosimeter position / irradiation geometry, and the associated instrumentations used to analyze the dosimeters.

11.2.2 Record the specific net absorbance (absorbance before and after irradiation), temperature correction, film thickness, and specific net absorbance, and resulting absorbed dose for each dosimeter. Reference the calibration curve used to obtain absorbed dose.

11.2.3 Record or reference the Type A and Type B uncertainties in the values of absorbed dose.

11.2.4 Record or reference the measurement quality assurance plan used for the dosimetry system application.

12. Measurement uncertainty

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

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

12.2.1 *Type A*—Those evaluated by statistical methods, or

12.2.2 *Type B*—Those evaluated by other means.

12.3 Other ways of categorizing uncertainty have been widely used and may be useful for reporting uncertainty. For example, terms *precision* and *bias* or *random* and *systematic* (non-random) are used to describe different categories of uncertainty.

12.4 If this practice is followed, the estimate of the expanded uncertainty of an absorbed dose determined by this dosimetry system should be within about ±6 % for the dose rates higher than 10^6 Gy/h for a coverage factor $k = 2$ (which corresponds approximately to 95 % level of confidence for normally distributed data).