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Practice for use of cellulose acetate dosimetry systems

Pratique de l'utilisation des systèmes dosimétriques à l'acétate de cellulose

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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.

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A pilot project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this pilot project, ASTM 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 International Standard 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.

Annex A1 of this International Standard is for information only.

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Standard Practice for Use of Cellulose Acetate Dosimetry Systems[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 the preparation, handling, testing and procedures for the use of cellulose acetate dosimetry systems, and the spectrometric, densitometric, or photometric readout equipment for measuring absorbed dose in materials irradiated by photons and electrons in terms of absorbed dose in water.

NOTE 1—Cellulose acetate dosimeter refers to untinted and tinted cellulose triacetate (CTA) or cellulose diacetate (CDA) film dosimeter.

1.2 This practice applies to cellulose acetate film dosimeters that can be used within part or all of the specified ranges as follows:

1.2.1 The absorbed dose range for untinted CTA is 5×10^3 to 3×10^5 Gy for photons and electrons,

1.2.2 The absorbed dose range for tinted CDA is 1×10^4 to 1×10^6 Gy for photons and electrons,

1.2.3 The absorbed dose rate for both CTA and CDA is from 0.03 to 4×10^{10} Gy/s,

1.2.4 The radiation energy range for photons is from 0.1 to 50 MeV, and

1.2.5 The radiation energy range for electrons is from 0.2 to 50 MeV.

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NOTE 2—In cases where low-energy electrons and charged particles cannot completely penetrate the thickness of standard CTA and DCA films, thin films may be used **(1,2)**.[2]

1.2.6 The irradiation temperature range is from − 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:*

E 170 Terminology Relating to Radiation Measurements and Dosimetry[3]

E 275 Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near Infrared Spectrophotometers[4]

E 666 Practice for Calculating Absorbed Dose from Gamma or X Radiation[3]

E 925 Practice for the Periodic Calibration of Narrow Band-Pass Spectrophotometers[4]

E 958 Practice for Measuring Practical Spectral Bandwidth of Ultraviolet-Visible Spectrophotometers[4]

E 1026 Practice for Using the Fricke Reference Standard Dosimetry System[3]

2.2 *ISO/ASTM Standards:*

51205 Practice for Use of a Ceric-Cerous Sulfate Dosimetry System[3]

51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing[3]

51275 Practice for Use of a Radiochromic Film Dosimetry System[3]

51276 Practice for Use of a Polymethylmethacrylate Dosimetry System[3]

51310 Practice for Use of a Radiochromic Optical Waveguide Dosimetry System[3]

51400 Practice for Characterization and Performance of a High-Dose Gamma Radiation Dosimetry Calibration Laboratory[3]

51401 Practice for Use of a Dichromate Dosimetry System[3]

51538 Practice for Use of the Ethanol-Chlorobenzene Dosimetry System[3]

51540 Practice for Use of a Radiochromic Liquid Dosimetry System[3]

51607 Practice for Use of the Alanine-EPR Dosimetry System[3]

51608 Practice for Dosimetry in an X-Ray (Bremsstrahlung) Irradiation Facility for Radiation Processing[3]

51631 Practice for Use of Calorimetric Dosimetry Systems for Electron Beam Dose Measurements and Dosimeter Calibrations[3]

51649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV[3]

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing[3]

51818 Practice for Dosimetry in an Electron Beam Facility

[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 Jan. 22, 2002. Published March 15, 2002. Originally published as ASTM E 1650–94. Last previous ASTM edition E 1650–97[e1]. ASTM E 1650–94 was adopted by ISO in 1998 with the intermediate designation ISO 15570:1998(E). The present International Standard ISO/ASTM 51650:2002 (E) is a revision of ISO 15570.

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

[3] *Annual Book of ASTM Standards*, Vol 12.02.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

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for Radiation Processing at Energies Between 80 and 300 keV[3]

2.3 *International Commission on Radiation Units and Measurements (ICRU) Reports:*[5]

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 and Gamma Rays 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 44 Tissue Substitutes in Radiation Dosimetry and Measurement

ICRU Report 60 Radiation Quantities and Units

3. Terminology

3.1 *absorbed dose*, (*D*)—quantity of ionizing radiation energy imparted per unit mass of a specified material. The SI unit of absorbed dose is the gray (Gy), where 1 gray is equivalent to the absorption of 1 joule per kilogram of the specified material (Gy = 1 J/kg). The mathematical relationship is the quotient of $d\bar{\epsilon}$ by *dm*, where $d\bar{\epsilon}$ is the mean incremental energy imparted by ionizing radiation to matter of incremental mass *dm* (see ICRU 60).

$$D = d\bar{\epsilon}/dm \quad (1)$$

3.1.1 *Discussion*—The discontinued unit for absorbed dose is the rad (rad = 100 erg/g = 0.01 Gy). Absorbed dose is sometimes referred to simply as dose.

For a photon source under conditions of charged particle equilibrium (see definition), the absorbed dose, *D*, may be expressed as:

$$D = \Phi\,[E\,(\mu_{en}/\rho)] \quad (2)$$

where:

Φ = particle fluence (see definition),

E = energy of the ionizing radiation, and

μ_{en}/ρ = mass energy absorption coefficient (see definition).

If bremsstrahlung production within the specified material is negligible, the mass energy absorption coefficient (μ_{en}/ρ) is equal to the mass energy transfer coefficient (μ_{tr}/ρ), and absorbed dose is equal to air kerma.

3.2 *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.

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

$$\dot{D} = dD/dt \quad (3)$$

$$SI \text{ unit} : \text{Gy} \cdot \text{s}^{-1} \quad (4)$$

3.3.1 *Discussion*—The absorbed-dose rate is often specified in terms of the average value of $\dot{D}$ over long-time interval, for example, in units of $\text{Gy} \cdot \text{min}^{-1}$ or $\text{Gy} \cdot \text{h}^{-1}$.

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

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

3.6 *cellulose acetate dosimeter*—untinted and tinted cellulose triacetate (CTA) or cellulose diacetate (CDA) film dosimeter that undergoes change in optical absorbance or optical density under ionizing radiation.

3.6.1 *Discussion*—This change in absorbance or optical density is related to radiation chemical change in cellulose acetate, plasticizer and tinted dyes, and can be related to absorbed dose in water.

3.7 *charged particle equilibrium*—a condition that exists in a material under irradiation if the kinetic energies, number, and direction of charged particles induced by the radiation are uniform throughout the measurement volume of interest. Thus, the sum of the kinetic energies of the charged particles entering the volume equals the sum of the kinetic energies of the charged particles leaving the volume.

3.7.1 *Discussion*—Electron equilibrium is often referred to as charged-particle equilibrium.

3.8 *dosimeter batch*—quantities 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.

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

3.10 *electron equilibrium*—charged-particle equilibrium for secondary electrons.

3.11 *measurement quality assurance plan*—a documented program for the measurement process that assures on a continuing basis that the overall uncertainty meets the requirements of the specific application. This plan requires traceability to, and consistency with, nationally or internationally recognized standards.

3.12 *measurement traceability*—the 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.

3.13 *net absorbance (ΔA)*—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:

$$\Delta A = |A - A_0| \quad (5)$$

3.14 *net optical density, ΔOD*—another expression for "net absorbance."

3.14.1 *Discussion*—This expression is more commonly used for film and plastic dosimeters than for liquid dosimeters.

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

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3.15 *specific net absorbance* (Δk)—net absorbance, ΔA, at a selected wavelength divided by the optical pathlength, d, through the dosimeter as follows:

$$\Delta k = \Delta A/d \quad (6)$$

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

3.17 *traceabilty*—see measurement traceability.

3.18 Other appropriate terms may be found in Terminology E 170.

4. Significance and Use

4.1 The cellulose acetate (CTA and CDA) dosimetry systems provide a means of measuring absorbed dose in materials **(3-17)**. Under the influence of radiation, chemical reactions take place in the cellulose acetate, plasticizer or dyes in the matrix, changing the optical absorption properties (absorption wavelength (band) and density) **(18)**. Absorbance or optical density values are measured at the selected wavelength using a spectrophotometer, densitometer, or photometer.

4.2 In the use of a specific dosimetry system, absorbed dose is evaluated by the use of a calibration curve or response function traceable to nationally or internationally recognized standards.

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

NOTE 3—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 These dosimetry systems may be used in the industrial radiation processing of various products, for example radiation effects tests, polymer modifications, and sterilization of medical devices.

4.5 The available dynamic ranges indicated in 1.2.1 and 1.2.2 are achieved by using a variety of plasticizer and dye concentrations in the CTA and CDA systems.

4.6 The difference in dose response due to changes in the parameters of the irradiation conditions, such as dose rate, temperature, humidity, and atmosphere should be considered when these are different from the parameters of the calibration.

NOTE 4—The dose response of the CTA dosimeter increases linearly with temperature (−10 to 40°C) and relative humidity (20 to 80 %) when irradiated at lower dose-rates (<10 kGy/h) typical of gamma-irradiators. The effects are found to be less severe at the higher dose-rates for electron irradiators (>100 kGy/h). Moreover, as mentioned in Ref **16**, these effects are known to vary from batch to batch. All these effects need to be considered before CTA dosimeters can be used routinely for processing **(6,9,11,12,16,17, and 20)**.

5. Apparatus [6]

5.1 The following shall be used to evaluate absorbed dose with cellulose acetate dosimetry systems:

5.1.1 A batch or portion of a batch of cellulose acetate film.

5.1.2 UV/visible spectrophotometer or an equivalent instrument having documentation covering: (*1*) the analytical wavelength at which absorbance or optical density is measured (see 8.2); (*2*) the accuracy of wavelength selection, absorbance or optical density reading (see Figs. 1-4 for suitable wavelength—for example, 280 nm for untinted CTA and 390 nm for dyed CDA); and (*3*) the spectral wavelength range (in the case of absorption spectral readout as discussed in Ref **16**). In addition, stray light rejection is needed. The spectrophotometer or the equivalent instrument should also be able to read the absorbance up to a value of 2.0 at a suitable wavelength with an uncertainty of no more than ±1 %.

5.1.3 A film holder for spectrophotometer, or equivalent device, should keep the film perpendicular to the analytical beam, or a built-in automatic film feeder at a speed of the order of 0.1 to 1 cm/s with the same specifications of the film holder used for automatic one-dimensional dose profile measurement.

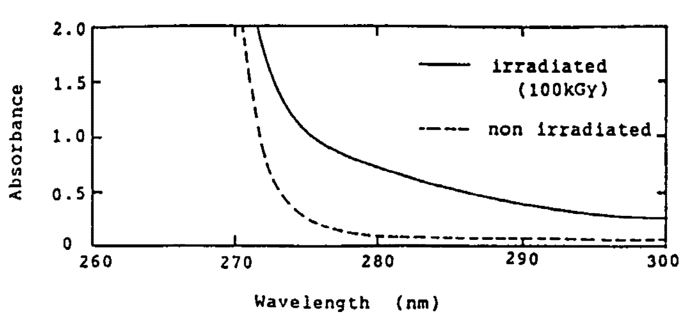
5.1.4 The thickness gage shall be calibrated and traceable to nationally or internationally recognized standards within a precision of ±1 % of the film thickness at the 95 % confidence level.

6. Preparation of Dosimeters

6.1 Cellulose acetate dosimeters can be prepared by pouring a prescribed recipe solution (for example, see Ref. **5**) consisting of cellulose diacetate or triacetate, plasticizer, dye, and solvent onto an optical flat plate and evaporating the solvent slowly and gently. 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.

6.1.1 For both untinted and tinted CTA dosimeter films, the recommended recipe is 85 weight % of cellulose triacetate and balance of triphenyl phosphate (TPP) as a sole plasticizer, plus compatible kinds and amounts of solvents, for example, methylenechloride-methanol mixture **(18)**.

6.2 In-house preparation of cellulose acetate dosimeters has an advantage that the film thickness can be adjusted according to the intended application, the measurable dose range and the range of the electron beam. The disadvantage lies in the difficulty in making a large size film of constant thickness. Such film may be used for small size dosimeters but, unless the



NOTE—The suggested wavelength of 280 nm is chosen due to low absorbance before irradiation (A_o), and linear absorbance (A) increase with dose. (Original drawing by the author of Refs **1,2, and 19**).

FIG. 1 Absorption Spectra Before and After Irradiation of Untinted Cellulose Triacetate (CTA) Film with a 2 MeV Electron Beam

[6] Corning S5-58, available from Corning, Inc., Technical Products Division, Advanced Materials Dept., Main Plant 21-3, Corning, NY 14831, USA, has been found satisfactory.

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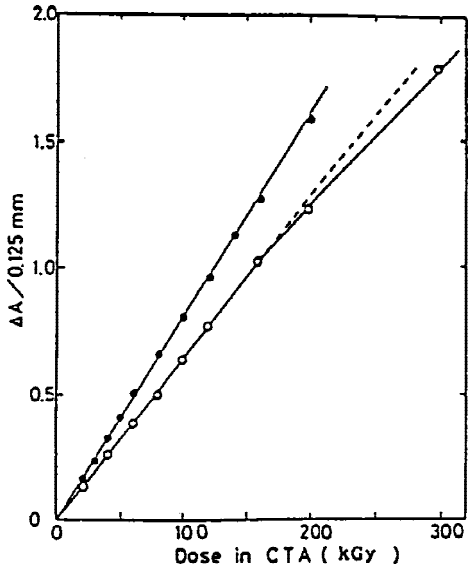

NOTE 1—O: electron beam (dose rate: 10^7/Gy/h, temperature: 15°C, relative humidity: 60 %). •: gamma-rays (dose rate: 10^4/Gy/h, temperature 25°C, relative humidity: 50 to 60 %).

NOTE 2—The ΔA values were measured 2 h after irradiation **(19)**.

FIG. 2 The Relation Between the Increment of Absorbance ΔA/ Nominal Thickness (0.125 mm) at 280 nm and Dose in Untinted CTA by Electron and Gamma-Radiation

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thickness is uniform, may not be used as long strips or large size films for continuous dose mapping purposes.

6.3 Some CTA and CDA films are commercially available, and are described in the nonmandatory annex of this practice.

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7. Calibration of the Dosimetry System

7.1 Prior to use, the dosimetry system shall be calibrated in accordance with the user's documented procedure that specifies details of the calibration process and quality assurance requirements. This calibration procedure shall be repeated at regular intervals to ensure that the accuracy of the absorbed dose measurement is maintained within required limits. Detailed calibration procedures are provided in ISO/ASTM Guide 51261.

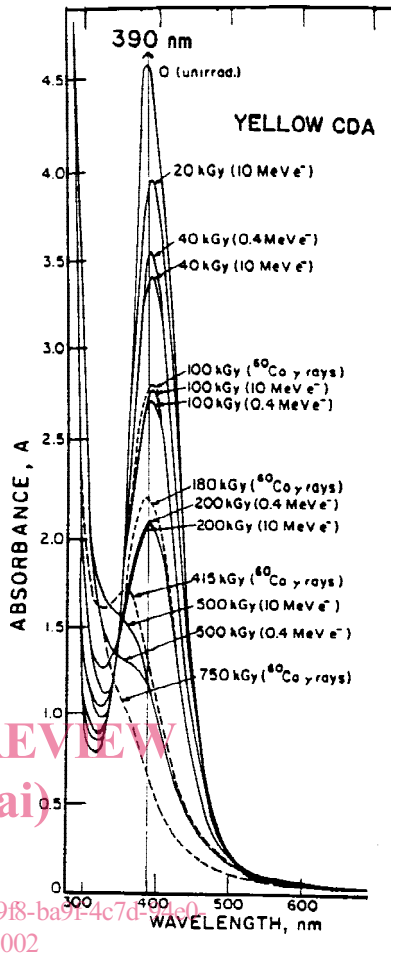
7.2 *Calibration Irradiation of Dosimeters*—Irradiation is a critical component of the calibration of the dosimetry system. Calibration irradiations may be performed in several ways, including irradiating the dosimeters using:

7.2.1 a calibration facility that provides an absorbed dose or an absorbed-dose rate having measurement traceability to nationally or internationally recognized standards, 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, or

7.2.3 a production or research irradiation facility together with reference or transfer–standard dosimeters that have measurement traceability to nationally or internationally recognized standards.

7.3 *Instrument Calibration*—Calibrations of the individual instruments used in the analysis of the dosimeters shall be



NOTE—The suggested wavelength for spectrophotometric analysis for dosimetry is indicated by the vertical arrow. If the spectrophotometer or densitometer is not able to measure very high absorbance ($A_{390\ nm} = 4.55$), measurement may be made at a higher wavelength on a shoulder of the absorption spectrum (for example, at 410 nm) or using a broad band-pass filter[6] with a densitometer **(16)**.

FIG. 3 Absorption Spectra Before and After Irradiation to High Doses (Using ^{60}Co γ Rays, 0.4- and 10-MeV Electron Beams) of Yellow Cellulose Diacetate (CDA) Film

verified at periodic intervals. These calibrations shall be traceable to nationally or internationally recognized standards. For example, if an optical absorbance-measuring instrument such as a spectrophotometer or densitometer is used, then appropriate standards shall be used to verify the accuracy of the optical absorbance at a specified wavelength(s). See ASTM Practices E 275, E 925, and E 958.

8. Calibration of Cellulose Acetate Dosimeter

8.1 *Irradiation*:

8.1.1 Randomly select five dosimeters from the batch or stock and do not irradiate them. Use them for determining A_o (see 8.3.1).

8.1.2 Select a set of at least four dosimeters for each absorbed dose value.

8.1.3 Irradiate these sets of dosimeters to at least five known dose values per decade covering the range of utilization, or at least four sets if the range of use is less than one decade.

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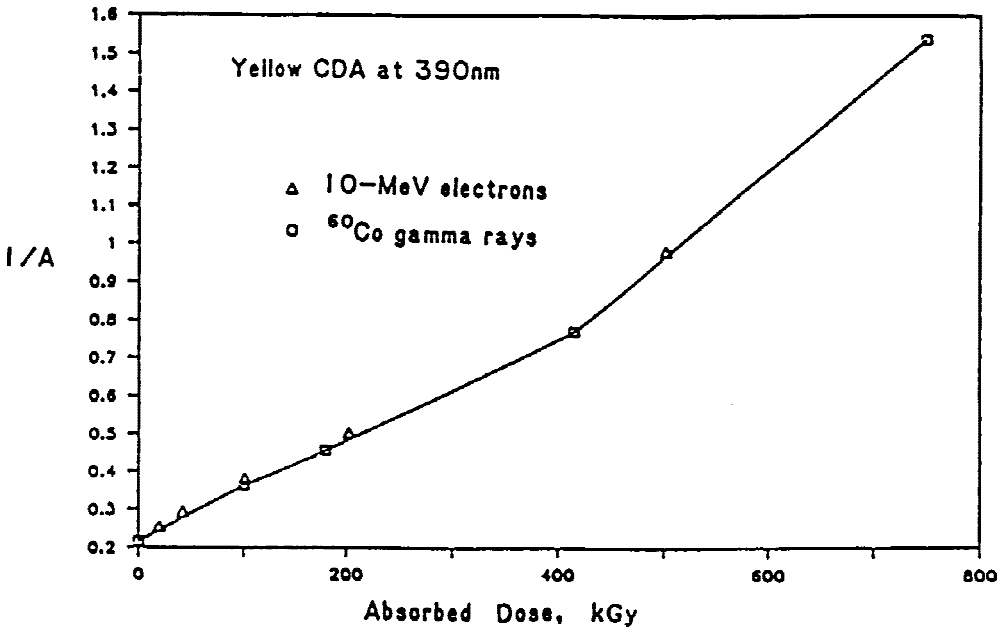

FIG. 4 Increase in the Reciprocal of Absorbance (I/A), at 390 nm Wavelength, as a Function of Absorbed Dose (in Water), when Yellow CDA is Irradiated with Electrons and Gamma Rays (16)

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NOTE 5—If the dose range exceeds a decade, the number of values of dose, N, is calculated by the following equation:

$$N = \text{Nearest integer}\,[5 \times \log_{10}(D_{max}/D_{min})] \quad (7)$$

For example, if the maximum dose range (D_{max}) is 200 kGy, and the minimum dose range (D_{min}) is 10 kGy,

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$$N = \text{Nearest integer}\,[5 \times \log(200/10)] \quad (8)$$

$$= \text{Nearest integer}\,[(5 \times 1.301)]$$

$$= \text{Nearest integer}\,[6.505]$$

$$= 7$$

8.1.4 Specify the calibration dose in terms of absorbed dose in water or in another material appropriate for the specific application (for example, see ASTM Practice E 1026 and ISO/ASTM Practice 51205).

8.1.5 Position the dosimeter in the calibration radiation field in a defined, reproducible location.

8.1.6 When using photon radiation for calibration, surround the dosimeters with a sufficient amount of water-equivalent material to ensure approximate electron equilibrium condition.

NOTE 6—For example, for a ^{60}Co gamma-ray source, this could be accomplished by surrounding the dosimeter with 3 to 5 mm of polymeric material, (for example, polystyrene) in all directions.

8.1.7 When using an electron beam for the calibration, locate the dosimeter in a well characterized position within the radiation field **(17,21)**.

8.1.8 Make the calibration field within the volume occupied by the dosimeter(s) as uniform as possible. The variation in dose rate within the occupied volume should be within ±1 %.

8.1.9 Control (or monitor) the temperature and humidity of the dosimeters during irradiation. Take into account any temperature and humidity variation that can affect dosimeter response (that is, specific net absorbance). For the dosimeter systems in Table A1.1 the temperature dependence of dosimeter response during gamma-irradiation is about + 0.5 % per °C.

NOTE 7—Untinted CTA (FTR-125)[7] does not show appreciable temperature dependence when used at dose rates of 10^6 Gy/h and higher **(22)**. Extremes in relative humidity affect the sensitivity. Therefore, avoid very low (<20 %) and very high humidity conditions (>80 %). For high dose-rate dosimetry (for example, electron beams), the effect of humidity differences on dosimeter response is less severe than at low dose rates (for example, gamma radiation) **(6,9)**. For high doses exceeding 200 kGy, CTA film becomes brittle and must be handled with care.

8.1.10 Calibrate each batch or stock of dosimeters prior to routine use, and at least once per year.

8.2 *Measurement*:

8.2.1 Depending on the cellulose acetate dosimeter used (see Table A1.1), set the spectrophotometer at the appropriate wavelength at a band width of no more than 1 nm, or use photometer or densitometer equipped with an appropriate band-pass filter or hollow cathode lamp or light-emitting diode (LED) of appropriate wavelength.

8.2.2 Set the balance of the spectrophotometer, densitometer, or photometer to zero absorbance, without a film dosimeter (with only air) in the analytical light beam.

8.2.3 Insert the non-irradiated film dosimeter in the holder and insert it in the analytical light beam of the spectrophotometer, densitometer, or photometer. Measure the absorbance with only air in the reference light beam. Record this value (A_o). With use of the scanning spectrophotometer, densitometer, or photometer, read the average value of absorbance with air as the reference. Record these values (A_o).

8.2.4 Insert the irradiated dosimeter film in the analytical light beam of the spectrophotometer, densitometer, or photometer and measure the absorbance (A). Using a scanning

[7] See the annex for the availability address.

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