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**Practice for use of calorimetric dosimetry  
systems for electron beam dose  
measurements and dosimeter calibrations**

*Pratique de l'utilisation de systèmes dosimétriques calorimétriques pour des  
mesures de dose délivrée par un faisceau d'électrons et l'étalonnage de  
dosimètres*



## 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.

International Standard ISO 15568 was prepared by the American Society for Testing and Materials (ASTM) Subcommittee E10.01 (as E 1631-96) and was adopted, under a special "fast-track procedure", by Technical Committee ISO/TC 85, *Nuclear energy*, in parallel with its approval by the ISO member bodies.

A new ISO/TC 85 Working Group WG 3, *High-level dosimetry for radiation processing*, was formed to review the voting comments from the ISO "Fast-track procedure" and to maintain these standards. The USA holds the convenership of this working group.

International Standard ISO 15568 is one of 20 standards developed and published by ASTM. The 20 fast-tracked standards and their associated ASTM designations are listed below:

| ISO Designation | ASTM Designation | Title                                                                                                          |
|-----------------|------------------|----------------------------------------------------------------------------------------------------------------|
| 15554           | E 1204-93        | <i>Practice for dosimetry in gamma irradiation facilities for food processing</i>                              |
| 15555           | E 1205-93        | <i>Practice for use of a ceric-cerous sulfate dosimetry system</i>                                             |
| 15556           | E 1261-94        | <i>Guide for selection and calibration of dosimetry systems for radiation processing</i>                       |
| 15557           | E 1275-93        | <i>Practice for use of a radiochromic film dosimetry system</i>                                                |
| 15558           | E 1276-96        | <i>Practice for use of a polymethylmethacrylate dosimetry system</i>                                           |
| 15559           | E 1310-94        | <i>Practice for use of a radiochromic optical waveguide dosimetry system</i>                                   |
| 15560           | E 1400-95a       | <i>Practice for characterization and performance of a high-dose radiation dosimetry calibration laboratory</i> |
| 15561           | E 1401-96        | <i>Practice for use of a dichromate dosimetry system</i>                                                       |

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|       |           |                                                                                                                            |
|-------|-----------|----------------------------------------------------------------------------------------------------------------------------|
| 15562 | E 1431-91 | <i>Practice for dosimetry in electron and bremsstrahlung irradiation facilities for food processing</i>                    |
| 15563 | E 1538-93 | <i>Practice for use of the ethanol-chlorobenzene dosimetry system</i>                                                      |
| 15564 | E 1539-93 | <i>Guide for use of radiation-sensitive indicators</i>                                                                     |
| 15565 | E 1540-93 | <i>Practice for use of a radiochromic liquid dosimetry system</i>                                                          |
| 15566 | E 1607-94 | <i>Practice for use of the alanine-EPR dosimetry system</i>                                                                |
| 15567 | E 1608-94 | <i>Practice for dosimetry in an X-ray (bremsstrahlung) facility for radiation processing</i>                               |
| 15568 | E 1631-96 | <i>Practice for use of calorimetric dosimetry systems for electron beam dose measurements and dosimeter calibrations</i>   |
| 15569 | E 1649-94 | <i>Practice for dosimetry in an electron-beam facility for radiation processing at energies between 300 keV and 25 MeV</i> |
| 15570 | E 1650-94 | <i>Practice for use of cellulose acetate dosimetry system</i>                                                              |
| 15571 | E 1702-95 | <i>Practice for dosimetry in a gamma irradiation facility for radiation processing</i>                                     |
| 15572 | E 1707-95 | <i>Guide for estimating uncertainties in dosimetry for radiation processing</i>                                            |
| 15573 | E 1818-96 | <i>Practice for dosimetry in an electron-beam facility for radiation processing at energies between 80 keV and 300 keV</i> |

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Designation: E 1631 – 96

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An American National Standard

## Standard Practice for Use of Calorimetric Dosimetry Systems for Electron Beam Dose Measurements and Dosimeter Calibrations<sup>1</sup>

This standard is issued under the fixed designation E 1631; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers the preparation and use of semi-adiabatic calorimeters for measurement of absorbed dose in graphite, water, or polystyrene when irradiated with electrons. The calorimeters are either transported by a conveyor past a scanned electron beam or are stationary in a broadened beam. It also covers the use of these calorimeters to calibrate dosimeter systems in electron beams intended for radiation processing applications.

1.2 This practice applies to electron beams in the energy range from 4 to 12 MeV.

1.3 The absorbed dose range depends on the absorbing material and the irradiation and measurement conditions. Minimum dose is approximately 100 Gy and maximum dose is approximately 50 kGy.

1.4 The averaged absorbed dose rate range shall generally be greater than  $10 \text{ Gy} \cdot \text{s}^{-1}$ , but depends on the same conditions as above.

1.5 The temperature range for use of these calorimeters depends on the thermal resistance of the materials and on the calibration range of the temperature sensor.

1.6 *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<sup>2</sup>
- E 666 Practice for Calculating Absorbed Dose from Gamma or X Radiation<sup>2</sup>
- E 668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices<sup>2</sup>
- E 1261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing<sup>2</sup>
- E 1431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing<sup>2</sup>

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee E-10 on Nuclear Technology and Applications and is the direct responsibility of Subcommittee E10.01 on Dosimetry for Radiation Processing.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 12.02.

E 1649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies Between 300 keV and 25 MeV<sup>2</sup>

E 1707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing<sup>2</sup>

2.2 International Commission on Radiation Units and Measurements (ICRU) Reports:<sup>3</sup>

ICRU Report 33 Radiation Quantities and Units

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 Measurements

### 3. Terminology

#### 3.1 Definitions:

3.1.1 *adiabatic, adj*—no heat exchange with the surroundings.

3.1.2 *calorimeter, n*—assembly consisting of calorimetric body (absorber), thermal insulation, and temperature sensor with wiring.

3.1.3 *calorimetric body, n*—the mass of material absorbing radiation energy and whose temperature is measured.

3.1.4 *endothermic reaction, n*—a chemical reaction that consumes energy.

3.1.5 *exothermic reaction, n*—a chemical reaction that releases energy.

3.1.6 *heat defect (thermal defect), n*—the amount of energy released or consumed by chemical reactions caused by the absorption of radiation energy.

3.1.7 *specific heat capacity, n*—the amount of energy required to raise a specified mass of material by a specified temperature.

3.1.8 *thermistor, n*—electrical resistor with a well-defined relationship between resistance and temperature.

3.1.9 *thermocouple, n*—a junction of two metals producing an electrical voltage with a well-defined relationship to temperature.

3.2 For additional terms, see Terminology E 170 and ICRU Report 33.

### 4. Significance and Use

4.1 This practice is applicable to the standardization of

<sup>3</sup> Available from the Commission on Radiation Units and Measurements, 7910 Woodmont Ave., Suite 800, Bethesda, MD 20814.

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absorbed dose in electron beams, the qualification of electron irradiation facilities, dosimetry intercomparisons between laboratories, periodic checks of operating parameters of electron processing facilities, and calibration of other dosimeters in electron beams.

NOTE 1—For additional information of the use of dosimetry in electron accelerator facilities, see Practices E 1431 and E 1641, ICRU Reports 34 and 35, and Refs 1–3.<sup>4</sup>

4.2 Graphite calorimeters provide a reliable means of measuring absorbed dose in graphite. The dose measurement is based on the measurement of the temperature increase in a graphite absorber irradiated by an electron beam.

4.2.1 For graphite for which the specific heat capacity is known, no calibration of the graphite calorimeter is needed.

4.2.2 The absorbed dose in other materials irradiated under equivalent conditions may be calculated. Procedures for making such calculations are given in Practices E 666 and E 668, Guide E 1261, and Reference (1).

4.2.3 The average absorbed dose in the graphite volume is measured. Dose gradients may occur in this volume and may have to be considered when estimating dose in other materials.

4.3 Water calorimeters provide a reliable means of measuring absorbed dose in water. The dose measurement is based on the measurement of the temperature increase in a volume of water, for example, a water-filled polystyrene petri dish.

4.3.1 The response of the water calorimeters should be calibrated by comparison with graphite calorimeters irradiated under precisely the same conditions.

4.3.2 The average dose in the water calorimeter is evaluated. Dose gradients may occur in this volume and may need to be considered when estimating dose in other materials.

4.4 Polystyrene calorimeters provide a reliable means of measuring absorbed dose in polystyrene. The dose measurement is based on the measurement of the temperature increase in a volume of polystyrene.

4.4.1 The response of the polystyrene calorimeters should be calibrated by comparison with graphite calorimeters irradiated under precisely the same conditions.

4.4.2 The average dose in the polystyrene volume is evaluated. Dose gradients may occur in this volume and may need to be considered when estimating dose in other materials.

4.4.3 Polymeric materials other than polystyrene may be used for calorimetric measurements. Polystyrene is used because it is known to be resistant to radiation (4) and because no exo- or endothermic reactions are taking place (5).

## 5. Interferences

5.1 *Extrapolation*—The calorimeter designs described in this practice are usually not strictly adiabatic, because of the exchange of heat with the surroundings or within the calorimeter assembly. The maximum temperature reached by the calorimetric body is different from the temperature that would have been reached in the absence of that heat

exchange. The temperature drifts before and after irradiation are extrapolated to the midpoint of the irradiation period in order to determine the true temperature increase due to the absorption of radiation energy.

5.2 *Heat defect*—Chemical reactions in irradiated water and other materials (resulting in what is called the heat defect or thermal defect) may be endo- or exothermic and may lead to measurable temperature changes. They are respectively deficient or excessive with respect to the temperature increase due directly to the absorption of radiation energy in the water. The extent of these effects depends on the purity or the gas content of the water and on any chemical effects arising from the container of the water. At the absorbed doses and dose rates usually encountered by these calorimeters, these effects are not significant (3).

5.3 *Temperature effects from accelerator structure*—The calorimeters are often irradiated on a conveyor used for passing products and samples past the irradiation zone. Radiated heat from the mechanical structures of the irradiation facility and from the conveyor may contribute to the measured temperature increase in the calorimeters.

5.4 *Thermal equilibrium*—The most reproducible results are obtained when the calorimeters are in thermal equilibrium before irradiation.

5.5 *Other materials*—The temperature sensors, wires, etc. of the calorimeter represent foreign materials, which may influence the total temperature rise. These components should be as small as possible.

5.6 *Dose gradients*—Dose gradients will exist within the calorimetric body when it is irradiated with 4 to 12 MeV electrons. These gradients must be taken into account, for example, when other dosimeters are calibrated by intercomparison with calorimeters.

## 6. Apparatus

6.1 *One Type of Graphite Calorimeter*, is a disc of graphite placed in a thermally-insulating material such as foamed plastic (6–8). A calibrated thermistor or thermocouple is embedded inside the disc. See Fig. 1 for an example of such a calorimeter. Some typical examples of graphite disc thicknesses and masses are listed in Table 1 (2).

6.2 *A Typical Water Calorimeter*, is a sealed polystyrene petri dish filled with water and placed in thermally-insulating foamed plastic (6). A calibrated temperature sensor

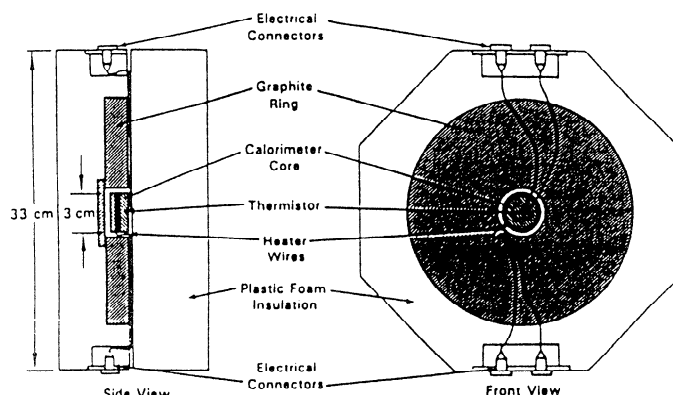


FIG. 1 Example of a Graphite Calorimeter Used at a 10-MeV Industrial Electron Accelerator (7)

<sup>4</sup> The boldface numbers in parentheses refer to the list of references at the end of this practice.

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**TABLE 1** Thickness and Size of Several Graphite Calorimeters Designed at NIST for Use at Specific Electron Energies

| Electron Energy<br>MeV | Electron Range<br>in Graphite <sup>A</sup><br>density: 1.7 g·cm <sup>-3</sup> |      | Calorimeter Disc (30 mm diameter) |      | Mass,<br>g |
|------------------------|-------------------------------------------------------------------------------|------|-----------------------------------|------|------------|
|                        | g cm <sup>-2</sup>                                                            | cm   | Thickness <sup>B</sup>            |      |            |
|                        |                                                                               |      | g cm <sup>2</sup>                 | cm   |            |
| 4                      | 2.32                                                                          | 1.36 | 0.84                              | 0.49 | 5.9        |
| 5                      | 2.91                                                                          | 1.71 | 1.05                              | 0.62 | 7.5        |
| 6                      | 3.48                                                                          | 2.05 | 1.25                              | 0.74 | 8.9        |
| 8                      | 4.59                                                                          | 2.70 | 1.65                              | 0.97 | 11.7       |
| 10                     | 5.66                                                                          | 3.33 | 2.04                              | 1.20 | 14.4       |
| 11                     | 6.17                                                                          | 3.63 | 2.22                              | 1.31 | 15.7       |
| 12                     | 6.68                                                                          | 3.93 | 2.40                              | 1.41 | 16.9       |

<sup>A</sup> This is the continuous-slowing-down-approximation (CSDA) range  $r_o$  of electrons for a broad beam incident on a semi-infinite absorber. It is calculated from:

$$r_o = \int_0^{E_o} \frac{1}{(S/\rho)_{\text{tot}}} \cdot dE$$

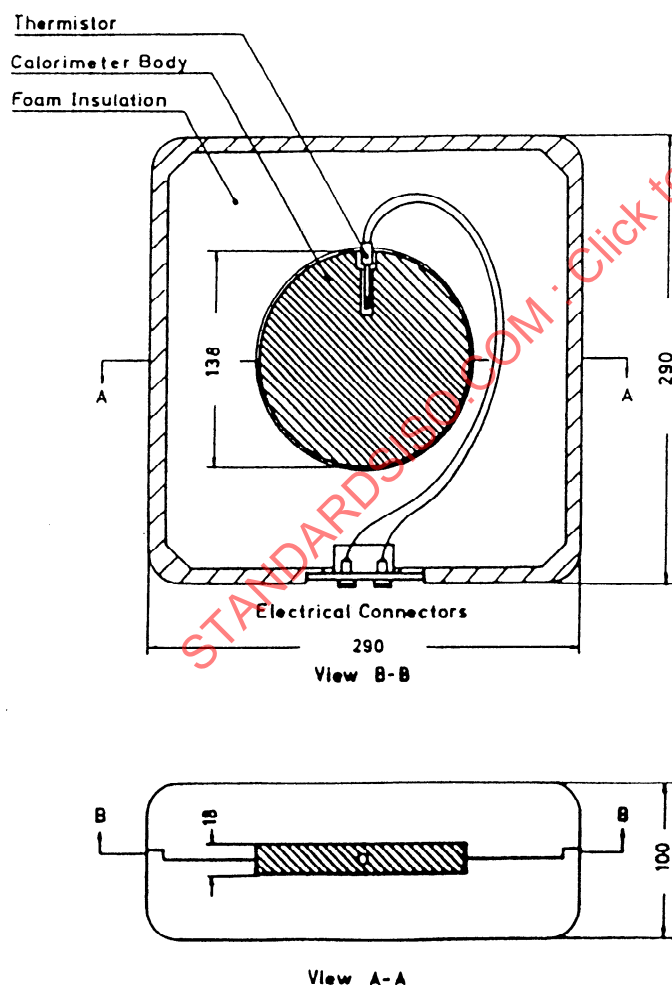
where:

$E_o$  = the primary electron energy, and  
 $(S/\rho)_{\text{tot}}$  = the total mass stopping power at a given electron energy (1).

<sup>B</sup> The thicknesses specified are equal to 0.36 ( $r_o$ ).

(thermistor) is placed through the side of the dish into the water. See Fig. 2 as an example of such a calorimeter.

6.3 *A Typical Polystyrene Calorimeter*, is a polystyrene

**FIG. 2** Example of a Water Calorimeter Used for Routine Measurements at a 10-MeV Industrial Electron Accelerator (6)

disc placed in thermally-insulating foamed plastic. A calibrated thermistor or thermocouple is imbedded inside the disc. The dimension of the polystyrene disc may be similar to that of the graphite and water calorimeters.

6.4 Radiation-resistant components should be used for the parts of the calorimeter that are exposed to the electron beam. This also applies to insulation of electrical wires.<sup>5</sup>

6.5 Good thermal contact must exist between the temperature sensor and the calorimetric body. For graphite and polystyrene calorimeters, this can be assured by adding a small amount of heat-conducting compound when mounting the temperature sensor.

6.6 *Read-Out*—The calorimeters are read by measuring the temperature of the calorimetric body. This temperature is registered by thermistors or thermocouples.

6.6.1 *Thermistor*—Use a high-precision ohm-meter for measurement of thermistor resistance. The meter should have a resolution of better than  $\pm 0.1\%$  and an accuracy of better than  $\pm 0.2\%$ . It should preferably be equipped for four-wire type resistance measurements, especially if the thermistor resistance is less than 10 k $\Omega$ . With the four-wire measurement technique, the effects of resistance in the measurement wires and electrical contacts are minimized.

6.6.2 Other appropriate instrumentation may be used for the thermistor resistance measurement, for example, a resistance bridge or commercial calibrated thermistor readers (7). It is important for both ohm-meters and resistance bridge measurements to minimize the dissipated power in the thermistor, preferably below 0.1 mW.

6.6.3 *Thermocouple*—Use a high-precision digital voltmeter, or commercial reader (2). The sensitivity of the voltmeter should be better than 0.1  $\mu$ V.

## 7. Calibration Procedures

7.1 The graphite calorimeters may be considered *either* as primary standard dosimetry systems *or* as routine dosimetry systems requiring calibration against other standards, depending on how they are used for dose measurement, while water and polystyrene calorimeters typically are used as routine dosimeters.

7.2 *Primary Standard Dosimeter*—In order to consider the graphite calorimeter as a primary standard dosimeter, the specific heat capacity of the graphite and its temperature dependence must be known and the temperature sensors and the measuring equipment must be accurately calibrated. Any influence of the irradiation conditions must be evaluated and any possible influence on the uncertainty of the dose reading must be taken into account.

7.2.1 The specific heat capacity of the graphite of the calorimetric body and its functional dependence on temperature may be determined by several techniques. One method employs a built-in electrical heater in the calorimetric body to dissipate a known amount of electrical energy (see 7.2.3 and Appendix X1). Another method uses a separate adiabatic calorimeter to measure specific heat of a sample of the graphite material (8). Adiabatic calorimeters that use differential scanning calorimetry techniques for specific heat

<sup>5</sup> Radiation resistant wiring is available, for example, from Huber und Suhner, Pfäffikon, Switzerland, under the brand name Radox.

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measurement are commercially available.

7.2.2 Calibrate the temperature sensors and their associated readout instrumentation by placing the sensors in a well-controlled environment with a precision, high-accuracy thermometer whose response is traceable to national standards. If possible, place the entire calorimetric body containing the temperature sensors in this environment in good thermal contact with the calibration thermometer. An appropriate environment could be a stirred oil or water bath or a well-insulated metal block. Slowly vary the temperature of the environment over the range of expected use, allowing ample time for all components to come to thermal equilibrium. Record the temperature sensor readings as a function of the calibration thermometer readings.

7.2.3 If the specific heat capacity of the graphite is not known or cannot be obtained conveniently, then the calorimetric body may be equipped with a built-in electrical heater for calibration. This, in effect, determines the mean specific heat capacity for a particular initial temperature and temperature increase.

7.2.3.1 The heater may consist of a resistance wire that is placed in the graphite calorimetric body in such a way that its heat is dissipated evenly in the graphite disc. The mass of the heater wire inside the graphite disc should be only a small fraction of the total mass of the two combined, preferably less than 1 %.

7.2.3.2 A known amount of energy is dissipated in the graphite disc if a known electrical current,  $I$ , (unit: A) is allowed to flow for a known time,  $t$ , (unit: s) through the wire with resistance  $R$  (unit: ohm). The mean specific heat capacity,  $c_G$ , may be calculated from

$$c_G = \frac{I^2 \cdot R \cdot t}{\Delta T \cdot m} \quad (\text{J} \cdot \text{kg}^{-1} \cdot ^\circ\text{C}^{-1}) \quad (1)$$

where:

$\Delta T$  = the observed temperature (unit:  $^\circ\text{C}$ ) increase from the initial temperature,  $T_0$  to the maximum temperature,  $T_{\text{max}}$ , and

$m$  = the mass (unit: kg) of the graphite disc.

Only the resistance wire which is actually inside the graphite disc should be considered when determining the resistance  $R$ . The mean specific heat capacity determined is valid only for the particular values of  $T_0$  and  $T_{\text{max}}$  employed. Thus, a series of electrical calibrations are needed to cover the expected temperature ranges of operation.

7.2.3.3 To determine  $\Delta T$ , plot the temperature versus time before and after switching on the electrical current. Extrapolate the curves to the midpoint of the heating time. The two values of temperature obtained from the extrapolations are used to calculate  $\Delta T = T_2 - T_1$  that would occur in the absence of heat exchange with the surroundings.

7.2.4 If the specific heat capacity is determined by other means, then it shall be known over the expected temperature range of operation.

NOTE 2—Repeated measurements of specific heat of various types of graphite have been carried out over the range of 0 to 50 $^\circ\text{C}$ , indicating a value for  $c_G$  of  $644.2 + 2.86 \cdot T$  ( $\text{J} \cdot \text{kg}^{-1} \cdot ^\circ\text{C}^{-1}$ ), where  $T$  is the mean temperature ( $^\circ\text{C}$ ) of the graphite. This value must, however, not be considered a universal value. (8).

7.3 *Routine Dosimeter*—Without knowledge of the specific heat of graphite, the graphite calorimeter may be used as

a routine dosimeter. Its response shall be calibrated against another reference standard dosimeter.

7.3.1 Calibration may be obtained in two ways:

7.3.1.1 Irradiation at a calibration laboratory together with reference standard dosimeters.

7.3.1.2 Irradiation at the user's facility together with transfer standard dosimeters from a calibration laboratory.

7.3.2 For irradiation in a calibration laboratory, usually the procedure in 8.3 may be used. Any effect on the calorimeter response in changing from the calibration laboratory to the user's facility must be evaluated and taken into account.

7.3.3 For irradiation together with transfer dosimeters at the user's facility, the procedure given in Section 9 may be used.

7.4 Water or polystyrene calorimeters may be calibrated against graphite calorimeters or by comparison with transfer standard dosimeters from an accredited calibration laboratory by irradiation sequentially (or simultaneously) at an electron accelerator. The radiation field over the cross-sectional area of the calorimetric body shall be uniform to within  $\pm 2\%$  and constant over the time required to irradiate both calorimeters. The irradiation conditions should be arranged so that the electron fluence is equal in the two calorimeters. If that is not the case, corrections or adjustments must be made.

7.4.1 The specific heat capacity of polystyrene is a function of temperature. The calibration must therefore be carried out at a range of temperatures, so that a relationship between the calibration factor (expressed in  $\text{kGy} \cdot ^\circ\text{C}^{-1}$ ) and the average temperature of the calorimetric body can be determined.

7.4.2 The calibration factor for water calorimeters is approximately  $3.4 \text{ kGy} \cdot ^\circ\text{C}^{-1}$  and for polystyrene calorimeters it is approximately  $1.4 \text{ kGy} \cdot ^\circ\text{C}^{-1}$ . For graphite, the relationship is approximately  $0.75 \text{ kGy} \cdot ^\circ\text{C}^{-1}$  (see Note 2). These values apply for 10 MeV irradiation of calorimeters with thickness approximately  $1.7 \text{ g} \cdot \text{cm}^{-2}$ .

7.5 Calibration of all types of calorimeters used as routine dosimeters should be checked by comparison with reference standard or transfer standard dosimeters at a frequency determined by the user.

## 8. Dose Measurement Procedures

8.1 *Conveyor Irradiation*—For calorimeters carried on conveyors past scanned electron beams, the calorimeter is usually disconnected from the temperature measurement system just prior to irradiation and reconnected for readout just after irradiation (9).

8.1.1 Before irradiation, measure the temperature of the calorimetric body and check that the temperature remains stable for a period of at least ten min (typically less than  $0.1^\circ\text{C}$  change).

8.1.2 Disconnect the measurement wires and place the calorimeter on the conveyor for transport through the irradiation zone.

8.1.3 Transport the calorimeter through the irradiation zone on the conveyor system.

8.1.4 During irradiation, record the time of irradiation, and the irradiation parameters (electron energy, electron current, scanned beam width, and conveyor speed).

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8.1.5 After passage of the irradiation zone, reconnect the wires for measurement of temperature, and record the time from the end of irradiation to the first temperature measurement. Record the temperature as a function of time for 10 to 20 min after irradiation, enough to establish the thermal decay characteristics of the calorimeter.

8.1.6 Plot the temperature values as a function of time before and after irradiation.

8.1.7 Extrapolate the curves before and after irradiation to the midpoint of the irradiation time. The two values of temperature obtained from the extrapolations are used as the temperature before irradiation ( $T_1$ ) and after irradiation ( $T_2$ ) that would occur in the absence of heat exchange with the surroundings. An example of data obtained by this measurement technique is shown in Fig. 3.

8.1.8 For the graphite calorimeter, the average absorbed dose in the graphite disc,  $D_G$ , is given by:

$$D_G = c_G \cdot (T_2 - T_1) \quad (2)$$

where:

$c_G$  = the specific heat capacity of the graphite at the mean temperature during irradiation,  $(T_1 + T_2)/2$ .

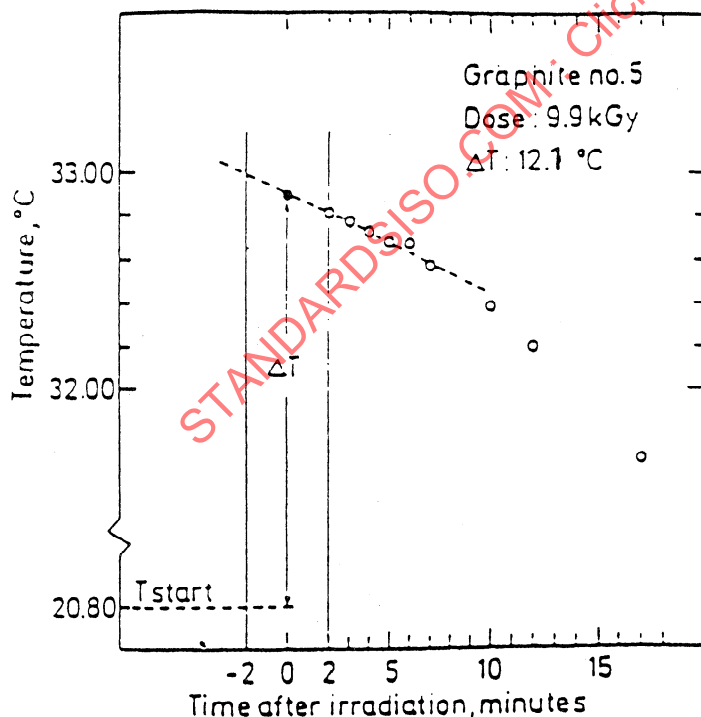
8.1.9 The dose,  $D_M$ , in another material of the same dimensions irradiated under the same conditions is given by:

$$D_M = D_G \cdot S_M / S_G \quad (3)$$

where:

$S_M$  and  $S_G$  = mass collision stopping powers of the other material and graphite, respectively (see Guide E 1261 and ICRU Reports 37 and 44).

8.1.9.1 This equation is valid only when the electron fluences in the two absorbers of interest are equal, which has



NOTE— $\Delta T$  is the temperature rise found by extrapolation and used for dose calculation (8.1.8). Wires were disconnected during irradiation.

FIG. 3 Example of Measurements of Temperature of a Graphite Calorimeter Before and After Irradiation Only (9)

been found to be the case for graphite and water, but not for aluminum and water (2).

8.1.10 For the water and polystyrene calorimeters, multiply the temperature difference,  $T_2 - T_1$ , by the calibration factor previously determined by calibration against graphite calorimeters (see Section 7), to evaluate the average absorbed dose in water or polystyrene, respectively.

8.1.11 For well-established, reproducible irradiation conditions the extrapolation procedure of 8.1.7 may not be needed. One measurement of temperature before and one after irradiation may suffice, and the temperature difference at the time of irradiation is found by use of a correction factor derived during the establishment of the irradiation procedures (6, 7, 9, 10).

8.2 *On-line Irradiation on Conveyor*—It is possible to measure the calorimeters' temperature during irradiation as the calorimeters are transported through the irradiation zone on the conveyor with measurement wires attached. Four-wire measurement (see 6.6.1) may be preferred in order to increase measurement precision.

8.3 *Stationary Irradiation*—The calorimeters described in this practice may also be used in a stationary configuration instead of being transported on a conveyor system through the electron beam. In this arrangement the beam is made uniform over the area of the calorimeter disc either by the use of metallic scattering foils or by raster scanning. The irradiation period is controlled by turning the electron beam on and off.

8.3.1 The readout of the temperature of the calorimeters in a stationary configuration may be done during irradiation rather than measuring before and after irradiation as described in 8.1.

8.3.2 With the electron beam turned off, locate the calorimeter on the beam axis at an appropriate distance from the accelerator beam exit window such that the beam profile is uniform to within  $\pm 2\%$  across the diameter of the calorimeter disc. The beam profile should be measured and if it varies more than  $\pm 2\%$  across the calorimeters, corrections for the non-uniformity may have to be carried out. Connect the temperature sensor wires to the calorimeter. The temperature readout system is located outside of the irradiation area, and the long connecting wires make four-wire measurements essential.

8.3.3 Measure the temperature of the calorimetric body as a function of time to ensure that the initial drift rate is less than the desired limits (see 8.1.1).

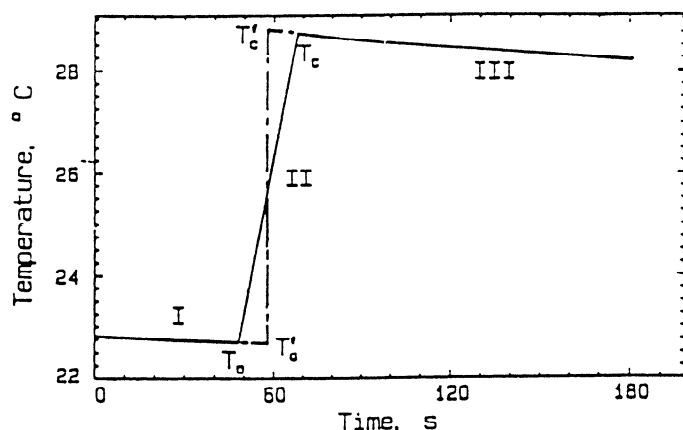
8.3.4 With the calorimetric body temperature being recorded continuously, turn on the electron beam for the desired irradiation time. After the beam is turned off, continue recording the temperature for enough time to establish the thermal decay characteristics of the particular calorimeter being used.

8.3.5 Extrapolate the time versus temperature curves before and after irradiation to the midpoint of the irradiation time in the same manner as in 8.1.7. The average dose in the calorimeter body is calculated in the same manner as in 8.1.8 or 8.1.10. An example of data obtained by this measurement technique is shown in Fig. 4 (2, 7).

## 9. Calibration of Other Dosimeters

9.1 The absorbed dose measured by the calorimeter is the

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NOTE—The regions I is before irradiation, II during irradiation and III after irradiation. The temperature curves were extrapolated from  $T_o$  and  $T_c$  to the midpoint of the irradiation time at  $T_o'$  and  $T_c'$ , respectively.  $\Delta T = T_c' - T_o'$  is used for dose calculation.

FIG. 4 Example of On-Line Measurements of a Graphite Calorimeter (7)

average dose in the calorimetric body.

9.2 For the purpose of calibration, other dosimeters are irradiated so as to receive the same average absorbed dose when placed at specific depths in phantoms which simulate the calorimetric body. These dosimeters shall be small enough to fit inside the thickness of the phantom. In addition the dosimeters should be small in order that the stopping power ratio (see 8.1.9) may be applied correctly (1, 7).

9.3 By successive (or simultaneous) irradiation of the phantom and the calorimeter under identical conditions, the dosimeters being calibrated are irradiated to accurately known doses.

9.4 A relation between the average dose in the calorimetric body and the dose at the midpoint of the dosimeter being calibrated may be established, if the dose distribution throughout the calorimetric body is known. This dose distribution can be measured by irradiating a dummy calorimeter disc containing a calibrated radiochromic film placed at an angle to the beam direction, and analyzing the film by scanning densitometry (9, 11, 12).

9.5 The mean temperature of the dosimeters during irradiation for calibration shall be known (7). This is necessary if temperature corrections are to be applied.

## 10. Documentation

10.1 For the calibration and application of the graphite calorimeters, record or reference the temperature sensor calibration data and the graphite specific heat capacity measurement data.

10.2 For the calibration and application of the water and polystyrene calorimeters, record or reference the temperature sensor calibration data and the data for the graphite calorimeters used to calibrate the water and polystyrene calorimeters.

10.3 Record or reference the irradiation date, temperature and time data analysis instrumentation, dose interpretation of calorimetry data, electron beam characteristics, and accelerator operating parameters.

## 11. Precision and Bias

NOTE 3—Measurement Uncertainty is a more accurate description of Section 11.

11.1 To be meaningful, a measurement of dose shall be accompanied by an estimate of uncertainty. Components of uncertainty shall be identified as either Type A or Type B according to their method of evaluation. Type A evaluation of standard uncertainty is based on the statistical analysis of a series of observations, and Type B is based on all other methods of analysis. Additional information is given in Guide E 1707 and Refs 14 and 15. In addition to Type A and Type B classification, other classifications may be useful.

NOTE 4—This practice recommends using the methodology adopted in 1993 by the International Organization for Standardization (ISO) for estimating uncertainty. This is different from the way that uncertainty has been traditionally expressed in terms of “precision” and “bias” where precision is a measure of the extent to which replicate measurements made under specified conditions are in agreement, and bias is a systematic error (see Practices E 170, E 177, and E 456). This new method for treatment of uncertainties is in conformance with current internationally accepted practice (15).

NOTE 5—Guide E 1707 defines possible sources of error in dosimetry performed in radiation processing facilities and offers procedures for estimating the resultant magnitude of the uncertainties in the measurement results. Basic concepts of measurement, estimate of the measured value of a quantity, “true” value, error, and uncertainty are defined and discussed. Components of uncertainty are discussed and methods are given for evaluating and estimating their values. Their contributions to the standard uncertainty in the reported values of absorbed dose are considered, and methods are given for calculating the combined standard uncertainty and an estimate of overall (expanded) uncertainty.

11.2 The components of uncertainty involved in measuring absorbed dose using this dosimetry system shall be estimated or determined. The overall uncertainty in absorbed dose may be estimated from a combination of these components, and the procedure for combining these components shall be specifically stated or referenced in all results.

11.3 If care is taken in carrying out this practice, the overall uncertainty of an absorbed dose determined by the system should be within  $\pm 5\%$  at a 95 % confidence level. Tables 2 (1, 7) and 3 (13) give examples of uncertainty

TABLE 2 Factors Contributing to Uncertainties in the Absorbed Dose Reading of the NIST Reference Graphite Calorimeter (In Percent, at a 95 % Confidence Level) (1, 7)

NOTE—The calculation of dose in a material other than the calorimetric body material will add an additional uncertainty of about 1 to 3 %, depending on the electron energy and the material.

| Source of Uncertainty                                                                 | Method of Evaluation of Uncertainty |                 |
|---------------------------------------------------------------------------------------|-------------------------------------|-----------------|
|                                                                                       | Statistical (%)                     | Other Means (%) |
| Calibration of temperature sensor and measuring equipment                             | 0.1                                 | 0.2             |
| Measurement of specific heat capacity                                                 | 0.5                                 | 2.0             |
| Effects of foreign material (thermistor, heater wire, etc.) on specific heat capacity |                                     | 0.2             |
| Non-uniformity of electron beam across calorimetric body                              |                                     | 0.5             |
| Temperature measurements                                                              | 0.2                                 | 0.5             |
| Time measurements                                                                     | 0.2                                 | 0.1             |
| Extrapolation of response curves to midpoint of irradiation period                    | 0.5                                 | 1.0             |
| Electrical power calibration (if used)                                                | 0.1                                 | 0.5             |
| Combined, added in quadrature                                                         | 0.77                                | 2.42            |
| Overall, added in quadrature                                                          |                                     | 2.5             |

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**TABLE 3 Factors Contributing to Uncertainties in the Absorbed Dose Reading of Routine Polystyrene Calorimeters from Risø High Dose Reference Laboratory (In Percent, at a 95 % Confidence Level) (13)**

| Source of Uncertainty                                 | Method of Evaluation of Uncertainty |                 |
|-------------------------------------------------------|-------------------------------------|-----------------|
|                                                       | Statistical (%)                     | Other Means (%) |
| Calibration                                           | 2.3                                 | 2.4             |
| Measurement of temperature at 3 kGy                   | 1.0                                 |                 |
| Specific heat of polystyrene                          |                                     | 0.5             |
| Influence of irradiation conditions                   | 0.5                                 |                 |
| Stability after irradiation at 3 kGy                  | 1.0                                 |                 |
| Conversion to dose absorbed in water (stopping power) |                                     | 0.5             |
| Combined, added in quadrature                         | 2.7                                 | 2.5             |
| Overall, added in quadrature                          |                                     | 3.7             |

factors for specific calorimeter systems. The values in these tables are not meant to be representative of other calorimeter systems or types and are only for illustrative purposes.

## 12. Keywords

12.1 absorbed dose; calibration; calorimeter; calorimetry; dosimeter; dosimetry; electron accelerator; electron beam; graphite; ionizing radiation; irradiation; polystyrene; radiation; radiation processing; water

## APPENDIXES

### (Nonmandatory Information)

#### X1. DETERMINATION OF SPECIFIC HEAT CAPACITY

X1.1 One method of determining the specific heat capacity involves the use of electrical power applied to the calorimetric body to produce a temperature increase (1). This technique is similar to that discussed in 7.1.3.

X1.1.1 The resistance wire used for the heater should be as small in diameter as practical to ensure the smallest mass and, thus, the least amount of perturbing material (that is, materials different from that of the calorimetric body) in the calorimetric body. The wire should have a low temperature coefficient of resistance to minimize the change in resistance when heated. The wire should be attached firmly to the graphite of the body (for example, glued on with epoxy) to ensure good thermal contact. The heater wire should be distributed within the body (such as in a spiral groove) to ensure even dissipation of the electrical power. The heater wires should be extended some distance from the calorimetric body before being attached to heavier wires to lead to the power supply and electrical measurement instrumentation. This will ensure that there are no significant heat losses from the calorimetric body through the heavy wires. Place the calorimetric body in good thermal-insulating material such as several centimeters of polystyrene foam. All electrical measurements shall be traceable to national standards.

X1.1.2 Provide a known amount of energy to the calorimetric body by supplying a known current,  $I$ , to the heater for a known period of time,  $t$ . This may be done by use of a

precision, stable, constant-current power supply that is controlled with an electronic timer. As noted in 7.2.3.2, the resistance,  $R$ , of the heater wire to be used is only that actually embedded in the calorimetric body, and should not include the resistance of the external leads. The time,  $t$ , should be as short as practical, and certainly no longer than the expected irradiation time in the electron beam. Adjust the power input to the heater to give the smallest temperature change that can be measured accurately. Record the temperature increase of the calorimetric body as a function of time. Calculate the mean specific heat capacity by use of the equation in 7.2.3.2.

X1.1.3 Perform a series of specific heat capacity measurements starting with the lowest initial temperature that it is anticipated will be encountered in actual application. Raise the temperature of the entire calorimeter assembly a small amount (somewhat greater than the initial  $\Delta T$  interval) and maintain the temperature at that level until thermal equilibrium is established as indicated by a low temperature drift rate. This will probably require the use of a temperature-controlled environment. Perform another measurement at this new initial temperature. Continue this series of measurements over the entire range of temperature that is expected to be encountered in actual application. Calculate the specific heat capacity for the midpoint of each  $\Delta T$  interval. Plot the specific heat capacity data as a function of temperature. It should be essentially linear.

#### X2. CALORIMETER SUPPLIERS

X2.1 Two calorimeter suppliers are listed in X2.1.1 and X2.1.2.:

X2.1.1 Risø National Laboratory  
High Dose Reference Laboratory  
Environmental Research Department  
DK 4000 Roskilde  
Denmark

X2.1.2 Radiation Interactions and Dosimetry  
Radiation Physics C229  
National Institute of Standards and Technology  
Gaithersburg, MD 20899-0001  
USA

X2.1.3 Reference to these suppliers does not imply endorsement by ASTM of the suppliers or their products.

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