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

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

Reference number
ISO/ASTM 51631:2002(E)

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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 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 51631 was developed by ASTM Committee E10, Nuclear Technology and Applications, through Subcommittee E10.01, and by Technical Committee ISO/TC 85, Nuclear Energy.

Annexes A1 and A2 of this International Standard are for information only.

Standard Practice for Use of Calorimetric Dosimetry Systems for Electron Beam Dose Measurements and Dosimeter Calibrations[1]

This standard is issued under the fixed designation ISO/ASTM 51631; 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 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 $Gy \cdot s^{-1}$, 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[2]

E 177 Practice for the Use of the Terms Precision and Bias in ASTM Test Methods[3]

E 456 Terminology Relating to Quality and Statistics[3]

E 666 Practice for Calculating Absorbed Dose from Gamma or X Radiation[2]

E 668 Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices[2]

2.2 *ISO/ASTM Standards:*

51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing[2]

51431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing[2]

51649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies Between 300 keV and 25 MeV[2]

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing[2]

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

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

ICRU Report 60 Radiation Quantities and Units

3. Terminology

3.1 *Definitions:*

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

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

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

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

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

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

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

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

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

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

Current edition approved Jan. 22, 2002. Published March 2002. Originally published as E 1631 – 94. Last previous ASTM edition E 1631 – 96$^{\epsilon 1}$. ASTM E 1631 – 96$^{\epsilon 1}$ was adopted by ISO in 1998 with the intermediate designation ISO 15568:1998(E). The present International Standard ISO/ASTM 51631:2002(E) is a revision of ISO 15568.

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

[3] *Annual Book of ASTM Standards*, Vol 14.02.

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

3.2 For additional terms, see ASTM Terminology E 170 and ICRU Report 60.

4. Significance and Use

4.1 This practice is applicable to the standardization of 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 ISO/ASTM Practices 51431 and 51649, and ICRU Reports 34 and 35, and Refs **1-3**.[5]

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 ASTM Practices E 666 and E 668, ISO/ASTM Guide 51261, and Ref **(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 (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 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.

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

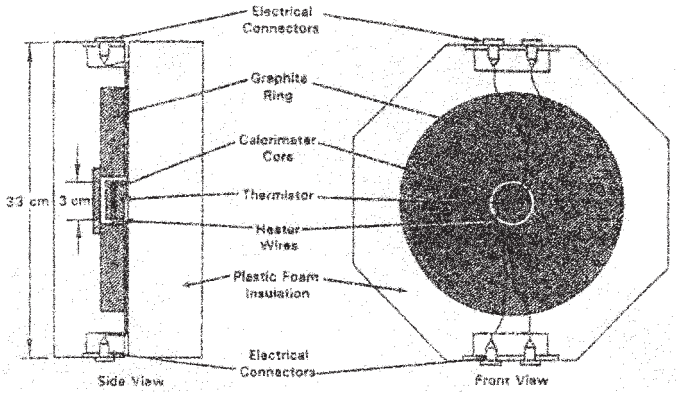


FIG. 1 Example of a Graphite Calorimeter Used at a 10–MeV Industrial Electron Accelerator (7)

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

Electron Energy MeV	Electron Range in Graphite[A] density: 1.7 g·cm^{-3}		Calorimeter Disc (30 mm diameter)		
			Thickness[B]		Mass, g
	g cm^{-2}	cm	g cm^{2}	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

[A]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:

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

where:

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

[B]The thicknesses specified are equal to 0.36 (r_o).

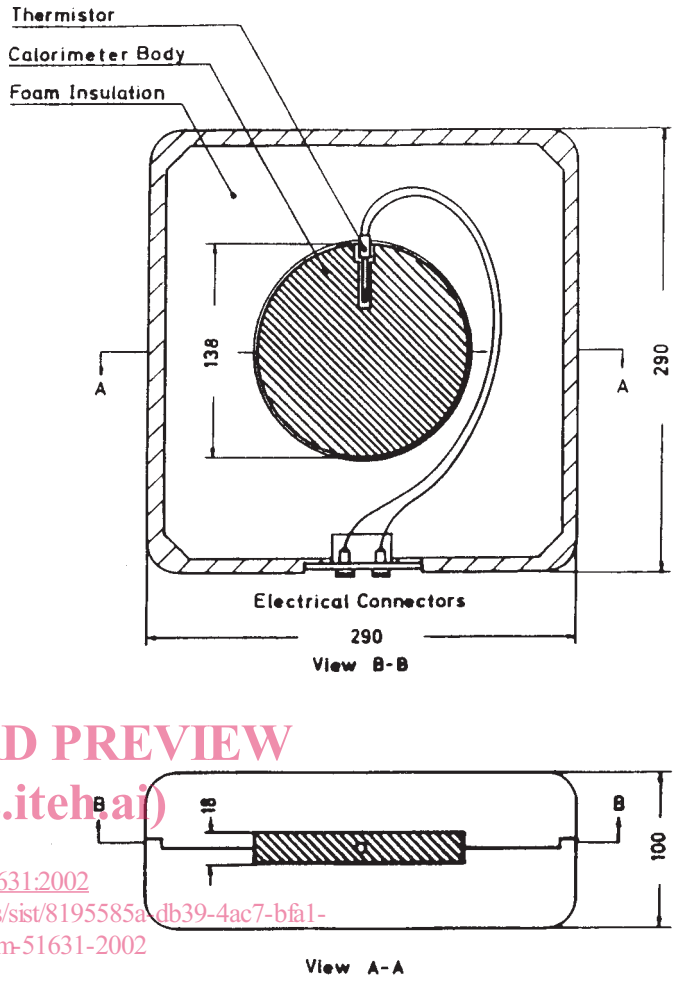


FIG. 2 Example of a Water Calorimeter Used for Routine Measurements at a 10–MeV Industrial Electron Accelerator (6)

This also applies to insulation of electrical wires.[6]

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 ± 0.1 % and an accuracy of better than ± 0.2 %. It should preferably be equipped for four-wire type resistance measurements, especially if the thermistor resistance is less than 10 kΩ. 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 µV.

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.

[6] Radiation resistant wiring is available, for example, from Huber und Suhner, Pfäffikon, Switzerland, under the brand name Radox.

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 Annex A1). 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 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

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

where:

ΔT = the observed temperature (unit: °C) increase from the initial temperature, T_o to the maximum temperature, T_{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_{max} employed. Thus, a series of electrical calibrations are needed to cover the expected temperature ranges of operation.

7.2.3.3 To determine ΔT, 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°C, indicating a value for c_G of $644.2 + 2.86 \cdot T$ ($J \cdot kg^{-1} \cdot °C^{-1}$), where T is the mean temperature (°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 ± 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 $kGy \cdot °C^{-1}$) and the average temperature of the calorimetric body can be determined.

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

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°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.

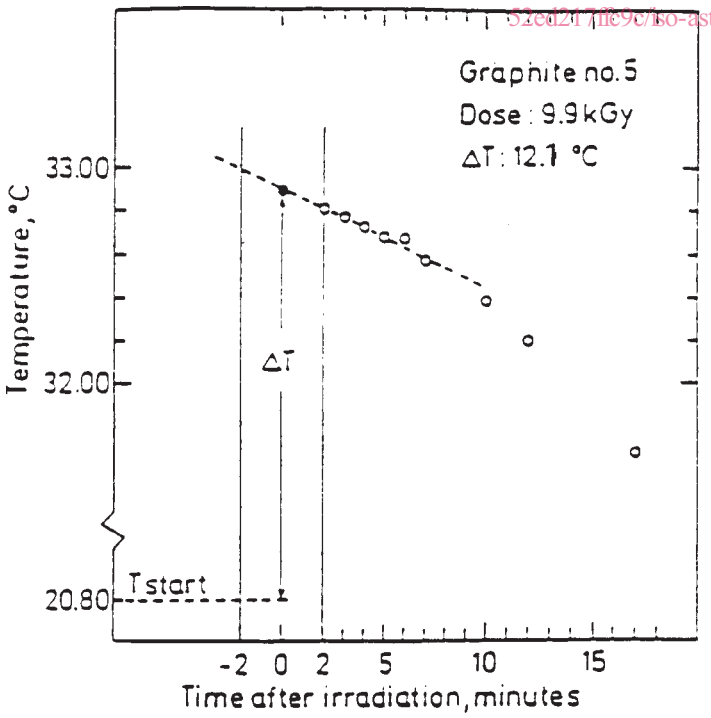
8.1.4 During irradiation, record the time of irradiation, and the irradiation parameters (electron energy, electron current, scanned beam width, and conveyor speed).

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:



NOTE—ΔT 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)

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

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 ISO/ASTM Guide 51261 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 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 ± 2 % across the diameter of the calorimeter disc. The beam profile should be measured and if it varies more than ± 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