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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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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 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 (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Footnote 1 was editorially altered in June 1999.

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 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]

E 1261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing[2]

E 1431 Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing[2]

E 1649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies Between 300 keV and 25 MeV[2]

E 1707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing[2]

2.2 *International Commission on Radiation Units and Measurements (ICRU) Reports:*[3]

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.

[1] 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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[2] *Annual Book of ASTM Standards*, Vol 12.02.

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

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 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**.[4]

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)**.

[4] The boldfaced numbers in parentheses refer to the list of references at the end of this practice.

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.

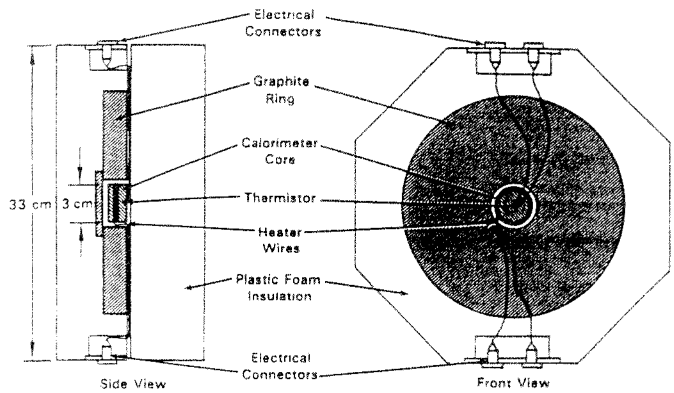

FIG. 1 Example of a Graphite Calorimeter Used at a 10–MeV Industrial Electron Accelerator (7)

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:

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

where:

E_o = the primary electron energy, and

$(S/p)_{tot}$ = the total mass stopping power at a given electron energy **(1)**.

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

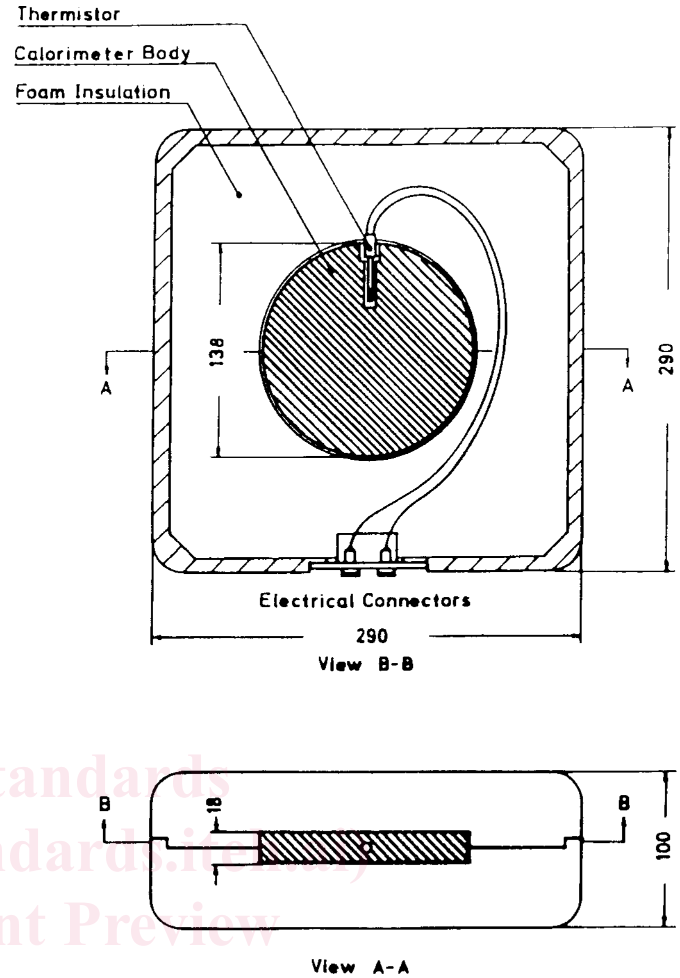

FIG. 2 Example of a Water Calorimeter Used for Routine Measurements at a 10–MeV Industrial Electron Accelerator (6)

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.[5]

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.

[5] 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 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 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

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

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