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Technical Note 163

THE CONSTRUCTION OF CALORIMETERS FOR THE MEASUREMENT OF ABSORBED DOSE

BEN PETREE AND GEORGE WARD

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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B. Petree and G. Ward

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The Construction of Calorimeters for the Measurement of Absorbed Dose

Ben Petree and George Ward

Direct measurements of energy locally absorbed in irradiated materials can be made with adiabatic calorimeters of suitable design. Design criteria imposed by requirements of accuracy include limitations on size and complexity. Small calorimeters of simple design with precision better than one percent at dose rates above one rad per second have been developed. Details of fabrication, auxiliary equipment and performance are described.

1. Introduction

It is generally conceded[1] that an important parameter to correlate with the effect of radiation on matter is the energy locally absorbed. In 1953[2] the International Commission on Radiological Units and Measurements called this quantity absorbed dose and its unit the rad (equal to 100 ergs per gram in the material of interest). Methods for determining the magnitude of the absorbed dose have been outlined by this group in later publications[3,4]. The ionization method, because of its high sensitivity and its long usefulness has been treated in some detail by this group. However, the calorimetric method is an alternative and more direct technique, if the dose rate is high enough.

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[1] Report of International Commission on Radiological Units and Measurements, NBS Handb. 47.

[2] Recommendations of International Commission on Radiological Units and Measurements (1953), Am. J. of Roentgenol. 71, p. 139 (1954).

[3] Report of International Commission on Radiological Units and Measurements (1956), NBS Handb. 62.

[4] Report of International Commission on Radiological Units and Measurements (1959), NBS Handb. 78.

Absorbed dose calorimetry measurements have been reported by several authors[5,6,7,8] but the desired criteria and complete construction details are often lacking. The present paper discusses the criteria that need to be satisfied and the details of construction of two absorbed dose calorimeters.

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2. Requirements for Calorimeters Used for Absorbed Dose Measurement

When calorimetric methods are used for the experimental determination of absorbed dose, certain general features of radiation absorption must be considered if the measurements are to be accurate, useful and easily interpreted. The following restrictions on calorimeter design are thus imposed in addition to those necessary for accurate calorimetry alone.

Size: Dose rate gradients are generally present in irradiated media. Consequently small calorimeters rather than large ones are generally required for measuring effectively the absorbed dose at a given point in the medium. This size limitation appears to be a serious difficulty in the calorimetric dosimetry of soft X-rays, where the half-value-layer in aluminum is less than a few millimeters.

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[5] Ben Petree, Absorbed Dose Calorimeters for High Dose Rate, Radiation Res. 9, No. 1, p. 166, July 1958. E. J. Hart, H. W. Koch, B. Petree, J. H. Schulman, S. I. Taimuty and H. O. Wyckoff, Measurement Systems for High-level Dosimetry, Paper No. 1927, Proceedings of the Second United Nations International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, 21, p. 188, United Nations, Geneva (1958).

[6] P. Milvy, S. Genna, N. Barr and J. S. Laughlin, Calorimetric Determination of Local Absorbed Dose, Paper No. 744, Proceedings of the Second United Nations International Conference on the Peaceful Uses of Atomic Energy, Geneva, 1958, 21, p. 142.

[7] W. B. Reid and H. E. Johns, Measurement of Absorbed Dose with Calorimeter and Determination of W, Radiation Res. 14, No. 1, p. 1, January 1961.

[8] J. S. Laughlin and S. Genna, Calorimetric Methods, Chapter 9 in Radiation Dosimetry, ed. Gerald J. Hine and Gordon L. Brownell, (Academic Press, New York, N. Y., 1956).

Homogeneity: The dose rate at a point in a medium is affected by nearby voids or imbedded masses of a different atomic composition. Consequently the calorimeter should be of a simple compact design. Foreign matter, as for example, thermocouple wire, should be minimized.

Chemical Stability: Radiation energy absorbed by a molecular substance generally has noticeable chemical effects in which energy is absorbed or released. When this occurs the thermal effect, to which the calorimeter responds, does not correspond precisely to the quantity of energy initially deposited by the radiation. More than 2 percent of Co^{60} energy absorbed in polystyrene fails to appear as heat and is apparently stored in the altered chemical structure of some of the molecules[6]. On the other hand non-molecular substances, such as metals, store energy only by atomic dislocations, and this amounts to only about 0.001 percent of the radiation energy absorbed. Such material should be used for calorimeters where possible.

3. General Description and Performance

3.1. Structure

The basic structure of the calorimeters to be described is shown in figure 1. The calorimeter consists of two principal parts; a calorimetric body or core, in which the absorbed dose is measured, and a shield that prevents heat exchange with the surroundings by conduction and radiation. Two thermocouples are attached to the core and one to the shield. They permit the indication of core temperature and core-shield temperature differences. Electrical heaters are attached to both core and shield. The core's heater permits the addition of accurately measured quantities of energy to the core for the purpose of calibrating the core temperature indications in energy units. The shield's heater permits the adjustment of shield temperature, as required during a measurement, to prevent heat transfer to the core.

Figures 2-4 of a spherical graphite calorimeter and figures 5-7 of a cylindrical aluminum calorimeter indicate the sizes involved and show some of the construction details. The cores are made in halves that are cemented together with a film of electrically conductive epoxy resin that constitutes the core heating element. The thermocouples are cemented with epoxy resin into small holes drilled in the core and shield. The shields are made in two pieces that fit tightly together and around their cores. The outer surfaces of the shields are wound with fine resistance heating wire. The graphite calorimeter was mounted in Styrofoam (fig. 4) and, when used to measure the dose rate in carbon from a Co^{60} source, was irradiated from all sides. The aluminum calorimeter was mounted in an aluminum block (fig. 7) and, when used to measure the heat developed in aluminum by high energy X-rays, was irradiated axially. Both calorimeters were ordinarily operated in vacua of the order of ten microns of mercury to reduce conductive and convective heat transfer to the core through air.

3.2. Performance

Examples of the response of these instruments are shown in figures 8 and 9. Figure 8 is a graph of data obtained from the graphite calorimeter during a measurement of the energy deposited in carbon by the gamma rays from a Co^{60} irradiator. Figure 9 is a reproduction of a chart recording of the response of the aluminum calorimeter before, during and after an irradiation in the X-ray beam of a synchrotron. The steps at 6 minutes and 7-1/2 minutes were produced by adjustments in the potentiometer with which the thermocouple voltage was measured. The noise on this trace was generated partly by fluctuating temperature gradients in the laboratory air and partly by the amplifiers used in the measuring and controlling circuits.

a. Precision

A series of electrical calibrations gave the following results which are typical of the performance of these simple instruments.

Calorimeter	Graphite	Aluminum
Power, watts	1.2×10^{-3}	13×10^{-6}
Equivalent dose rate, rads per second	130	1
Time of runs, seconds	900	900
Number of runs	7	5
Sensitivity, microvolts per 1000 rads	0.814	0.710
Standard deviation of mean sensitivity	0.14 percent	0.5 percent

b. Accuracy

Systematic errors are introduced in adiabatic calorimeters when the temperature gradients in core and shield during calibration runs are not precisely proportional to the gradients during measurement runs. Such differing gradients affect the apparent temperature of core and shield as registered by the thermocouples, and in consequence, the heat transfer to the core from the shield during a measurement will in general not equal that during a calibration. Upper limits of less than 0.24 and 0.15 percent have been calculated for errors of that origin in the graphite and aluminum calorimeters respectively.

Data in agreement with the calculation for the graphite calorimeter have been obtained by the ionization method mentioned in the introduction. Measurements with an ion chamber were made of exposure rate from a Co^{60} source, and at the same position measurements with the graphite calorimeter were made of the rate of heat evolution. After allowing for attenuation of the gamma rays in the graphite, the dose rate predicted from the exposure rate could be compared with the dose rate measured directly by the calorimeter as follows:

Exposure Dose Rate	Roentgens per Second
Measured	157.5 ± 1 percent
Effective (157.5 x .974)	153.4 ± 1 percent
Heating in Carbon	Milliwatts per Gram
Predicted ($0.868 \times 153.4 \times 10^{-2}$)	1.332 ± 1 percent
Measured (calorimeter)	1.323
(Predicted) - (Measured)	0.009 (0.7 percent)

c. Resistance to Radiation Damage

The graphite calorimeter was not affected in its electrical or thermal characteristics by a total absorbed dose of about 10^7 rads accumulated during test and use. However, some darkening of the epoxy resin was observed, and possibly a slight tendency to become brittle.

The aluminum calorimeter received a total dose of about 10^5 rads. No signs of damage resulted.

4. Construction of Calorimeters

The various parts will be taken up in order in the following sections:

4.1. Core

a. Dimensions and Composition

There appear to be four principal considerations in choosing the size of the core:

Stability of core temperature
Homogeneity of core temperature
Homogeneity of core composition
Degree of space resolution

The first two affect the accuracy of the calorimetric determinations made by the instrument; the last two affect the usefulness of those determinations to the particular dosimetric measurement that is desired. These four requirements are conflicting in the matter of core size, as the first and third lead to large cores while the second and last lead to small cores.

(1) Stability of Core Temperature

The rate at which the core temperature, θ, drifts in response to a unit core-shield temperature differential is:

$$\frac{d\theta}{dt} = \frac{h}{C}$$

where h is the heat transfer coefficient in watts $°C^{-1}$ of all heat paths from core to shield, including air, wires, spacers and thermal radiation[8], and C is the heat capacity of the core in watt-seconds $°C^{-1}$. Since it is desirable to minimize the drifts in temperature caused by unavoidable differentials, the largest core mass compatible with other requirements is desirable to give the largest value of C. Increasing the mass of the core does not affect the sensitivity of the calorimeter if, as is most frequent, it is completely immersed in the radiation field. In designs where thermal radiation accounts for an important fraction of h, it is also desirable to adopt a spherical or nearly spherical shape to minimize the surface area of the core relative to the volume. The quantity $\frac{C}{h}$ is approximately the time constant of the relaxation of core-shield temperature differences, and is readily measured in an assembled calorimeter. For both calorimeters the calculated and measured time constants are about 1000 seconds.

(2) Homogeneity of Core Temperature

It is desirable to minimize by design the temperature inhomogeneity of the core so as to minimize systematic changes in heat transfer to the core which accompany core temperature gradients. An upper limit to the fractional error, δ, caused by inhomogeneities of core and shield temperature is given by:

$$\delta = \frac{1}{(1 + \frac{1}{N})} \left(\frac{T_c + T_s}{T_o}\right) \text{ where}$$

T_c and T_s are the time constants for the relaxation of temperature gradients in core and shield, respectively,

T_o is the time constant for the relaxation of temperature differences between core and shield, and

N = heat capacity of shield ÷ heat capacity of core.
(N=4 for graphite calorimeter; N=1 for aluminum calorimeter)

The time constants in this expression can be calculated with fair accuracy for simple designs, and can be measured directly for greater accuracy.

The value of T_c is related to the core dimensions as follows, provided the core is homogeneous and convex:

$$T_c \lessapprox \frac{\rho c}{k} \left(\frac{L}{\pi}\right)^2 \text{ where}$$

π = 3.14 ....
ρ = density, g cm^{-3}
c = specific heat, joules $°C^{-1}$ g^{-1}
k = thermal conductivity, watt cm^{-1} $°C^{-1}$
L = largest dimension of core, cm
T_c = core time constant, sec.

Larger values of T_c are to be expected for the split cores, but the increase was slight for the two calorimeters described. For both aluminum and graphite cores the calculated and measured time constants were about one second.

Since the value of T_o was about 1000 seconds for both instruments, the errors attributable to temperature gradients in the cores do not exceed 0.08 percent and 0.05 percent for the graphite and aluminum calorimeters respectively. The corresponding contributions of the shields to the systematic error are treated in a later section where it is found that they do not exceed 0.16 percent and 0.1 percent respectively.

(3) Homogeneity of Core Composition

The thermocouple, heating element, and connecting wires which are fastened to the core may receive a different dose than the core material proper because of their differing atomic numbers. The extent to which the calorimeter response is affected by the presence of those foreign materials depends upon the energy of the radiation, and is least for photon energies near 1 Mev where the Compton process predominates. At lower energy where the photoelectric process is important and at higher energy where pair-production is important, the effect is more pronounced. Corrections may not be very precise, depending as they do upon a knowledge of the radiation spectrum. It is therefore desirable to minimize the effect by utilizing the largest possible core and the smallest possible mass of foreign material.

The composition of the graphite and aluminum cores and their intended applications were as follows:

	Graphite	Aluminum
Total Mass	0.9 g	1.4 g
Percent composition:		
core proper	97	99.9
foreign materials	3	0.1
Radiation spectrum	1.25 Mev (Co^{60})	X-rays Ep>50 Mev

(4) Degree of Resolution

The dose values determined by the calorimeter are averages over the volume of the core, and as such do not give directly the precise value at any given point. The expected dose gradients in the medium and the required accuracy of measurement should both be considered in deciding upon the largest tolerable core dimensions. The gradients depend greatly upon the configuration and energy spectrum of the source as well as upon the depth in and atomic number of the medium. Therefore the maximum core dimensions will depend very specifically upon the measurement that is to be made.

b. Core Heaters

Conducting film heaters were used in both graphite and aluminum cores. The mass of foreign material thus added to the cores was considerably smaller than if a separate heating element had been installed inside of or attached to the core. The film heater has the advantage also of distributing well the generated heat, a desirable method of increasing the uniformity of core temperature during calibrations.

(1) Choice of Resistance

There appear to be two principal considerations affecting the choice of core heater resistance:

The value should be large enough compared to the resistance of the heater lead-in wires that the measured calibrating power can be accounted for precisely; and small enough to permit the required power to be developed with a reasonably small applied voltage.

The values shown below were satisfactory.

Core	Graphite	Aluminum
Core resistance, ohms	250	8000
Lead-in resistance, ohms	0.5	0.5
Calibration power, watts	10^{-3}	10^{-5}
Potential, volts	0.5	0.3

(2) Film Preparation

A resin or ink[9] with the following composition by weight was used for the core heaters:

Epoxy Resin	100
Hardener (BF_3 piperdidine)	7.5
Carbon Black	5-10

The ink is a paste at room temperature and has a shelf life of about six months. Hardening is induced by baking for about one hour at about 200 °C. The cores were assembled with ink and spacers as shown in figure 10. They were clamped to prevent slipping, baked, and allowed to cool. The clamps were then removed and the spacers were pulled out.

The cores thus prepared were very strongly united but the resistance values obtained show considerable spread. Resistances ranging from about 40 ohms to 1300 ohms were obtained.for a dozen graphite cores. Half of those were above 200 ohms and were considered acceptable.

The aluminum cores were less satisfactory, the resistances ranging from less than an ohm to more than 1000 megohms. Although the values were quite unpredictable, several acceptable cores were obtained having resistances between one and ten kilohms. It seems likely that the aluminum tends to remain insulated from the ink, perhaps because of its oxidized surface. Thus, the heating in such films may be concentrated at a small spot rather than well distributed.

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[9]The ink was obtained from the Diamond Ordnance Fuze Laboratories, Washington, D.C., where it is ordinarily used to form resistors on printed circuits. We are indebted to Dr. Norman Doctor for suggesting its use and to Miss Marian Davies and Miss Mary Lee Hebb for advice concerning its properties and for the preparation of the graphite core heaters. The preparation of the ink requires special equipment and techniques.

(3) Power Lead-in

The heating current was brought to the core through enameled copper wires of 0.00175 in. diameter. Connection was made to the aluminum core by wedging the wires into holes with aluminum pegs, and to the graphite core by soldering the wires to electro-deposited copper spots at the poles of the core. Wood's alloy was used for the solder in an effort to avoid overheating. Figure 11 shows the details. These or other simple methods of connection are possible for cores of any material having sufficient electrical conductivity. Connections for non-conducting cores would require different and possibly more elaborate construction.

The use of some material other than copper for the power lead-in will reduce heat transfer to the core, and is therefore desirable provided the increased electrical resistance does not prevent accurate calibration.

c. Thermocouples

(1) Choice of Wire

Chromel P-constantan thermocouples were used in both calorimeters. That combination is well suited to this application for two reasons: the thermoelectric power, 60 microvolts/°C, is the largest available from common thermocouple materials; the thermal conductivity of both metals is small. The wires are available in many sizes and with various kinds of insulation. Glass fiber insulation with silicone binder was used in both calorimeters, and appears to be quite satisfactory.

It is desirable, for small heat leakage, to use small size thermocouple wires. However, if excessively fine wires with consequent high electrical resistance are used, then the signal-to-noise ratio at the detector will be poor. The choice of wire size should be influenced by the total thermocouple circuit resistance, which in turn depends upon the length of the leads. For the graphite calorimeter, the experimental arrangement required leads about 14 ft long and wires of 0.005 in. diameter were used. In the aluminum calorimeter, for which leads only 6 ft long were necessary, wires of 0.003 in. diameter were used.

(2) Welding

Couples were produced by a capacitor discharge process, the apparatus for which is shown in figure 12. The wires to be welded were held in place on the bottom block with tape so as to cross on the electrode, 1. The top block was closed down and held with about 150 grams weight. S_1 was closed to charge C to the desired voltage, usually about 20 volts, then S_2 was closed to produce the weld[10]. The couples so formed were trimmed and tied and given a narrow hairpin shape by pulling with a wire looped through the couple and pressing between thumb and finger as shown in figure 13A.

(3) Embedding

The thermocouples need to be embedded in such a way as to be thermally attached to, but electrically insulated from the core. Our efforts to insulate the thermocouples by painting on resins and enamels were not very satisfactory, as all of those tried tended to form beads before hardening and left the wires largely bare. Several coatings were necessary to insulate the couples used in the graphite cores.

More reliable results were obtained by another process pictured in figures 13B-D. An epoxy resin plug was cast around the couple for insulation (fig. 13B). This plug was then pressed out of the Teflon mold, its insulation tested in mercury, and embedded with more resin in the core. The liquid resin was inserted into the holes with the help of a hypodermic syringe with a ground-down needle. It is desirable for the hole diameter to be small so as to minimize the proportion of foreign material in the core, and when the resin conducts heat less readily than the core, to improve the speed and accuracy of temperature indication.

4.2. Shield

a. Dimensions and Composition

It is desirable, as for the core, to minimize the quantity of foreign material contained in or attached to the shield in order that the distribution of radiation that would exist in a homogeneous medium may be perturbed as little as possible. For the same reason, the spacing allowed between core and shield should also be minimized. The shield should be limited in thickness to a value sufficient to accomodate

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[10] S_2 should be of a type having low resistive losses under the conditions of use so that the energy available for welding cannot fluctuate from one weld to the next. An ordinary copper single-pole knife switch was used. It was closed rapidly by striking it with a light wooden stick. The electrodes of figure 10 were number 12 copper wires secured to the hinged plates with epoxy resin. They were flattened initially by sandpapering them while the blocks were held closed. Sandpapering was also done to clean the electrodes before each weld, and is apparently needed for consistent results.

thermocouple and heater wires and to satisfy special requirements[11] of the particular measurement. Unnecessary thickness is not desirable because the time lag for the propagation of temperature changes from the heater on the outside of the core to the thermocouple inside, is detrimental to accurate control of shield temperature. The time lag varies as the thickness squared.

The dimensions of the shields were as follows:

	Graphite cm	Aluminum in.
Wall thickness	0.4	0.040
Diameter (outer)	2.2	0.518
Height	(spherical)	0.346
Spacing, core to shield:		
Radial	0.2	0.020
Top and bottom	(spherical)	0.008

b. Isothermal Requirements

It was mentioned in the preceding section on core dimensions that systematic errors result from temperature inhomogeneities in the shield. These errors can be minimized by the following means without complicating the calorimeter structure:

(1) Reduce the size of the shield.

The value of T_s is proportional to the area of thin shields of a fixed shape. For example on a spherical shield:

$$T_s = \frac{\rho c}{k} \frac{D^2}{8} \text{ where}$$

ρ = density, g cm^{-3}

c = specific heat, joules °C^{-1} g^{-1}

k = thermal conductivity, watts cm^{-1} °C^{-1}

D = diameter of shield, cm

T_s = shield time constant, sec.

The values of T_s for both calorimeters were calculated to be about two seconds. Thus, the fractional errors attributable to

[11]As an example, the thickness of the graphite shield, 0.4 cm, was selected to prevent electrons originating outside the calorimeter in materials other than carbon from reaching the core.

temperature gradients in the shield do not exceed 0.16 percent and 0.1 percent for the graphite and aluminum calorimeters respectively.

(2) Apply heater windings with uniformly spaced turns over the whole exterior surface of the shield. That will give uniform average surface density of heating power. Uniformly good thermal contact for the whole length of heater is equally important.

(3) Space the heater windings closely. Periodic temperature variations on the inner surface set up by the heater wires outside can be reduced to any arbitrarily small amplitude by the choice of small enough spacing. The amplitude will not exceed A degrees C where:

$$A = \frac{1}{2} \frac{I^2 \rho}{k} \frac{1}{\sinh 2\pi \frac{T}{L}}$$

I = heater current, amperes

ρ = resistance of heater wire, ohm cm^{-1}

k = thermal conductivity of shield, watts cm^{-1} $°C^{-1}$

T = thickness of shield, heater to thermocouple, cm

L = heater winding spacing, center-to-center, cm

This expression also shows that A is proportional to the total shield heating power, which is in turn approximately proportional to the number of degrees above ambient temperature that the shield is operating. Hence,

(4) Operate the shield as close to ambient temperature as the experiment permits.

(5) Design the shield so that its separate parts fit together tightly for good thermal connection.

c. Thermocouple

(1) Location

The shield's thermocouple should indicate the temperature of the inside surface of the shield, since it is with that surface that the core exchanges heat. Therefore the shield's thermocouple should be embedded as close as possible to the inner surface of the shield.

(2) Orientation

The differential thermocouple circuit may give false indications of temperature difference if the two opposing couples differ in thermoelectric power. We have generally found that effect to be least if the pair of couples is made from adjacent segments of wire as shown in figure 14, and not from widely separated or totally unrelated segments.[12]

d. Heaters

The shield heaters of both calorimeters consisted of #43 nylon-insulated resistance wire wound on the outer surfaces of the shields and held in place with epoxy resin. Figures 3 and 4 show the graphite shield heater. The winding was applied to the ungrooved surface and secured by tape until the resin holding it was hard. Figures 5 and 6 show the spiral winding grooves on the aluminum shield, and figure 7, a portion of the completed heater winding. The center-to-center spacing of turns was 0.028 in. The groove was square and just large enough for the wire to slip in.

The heater resistance of both shields was about 250 ohms. The power requirements of the heaters were a few milliwatts, the precise value depending upon both ambient and core temperatures.

e. Core Spacers

The core of the graphite calorimeter was supported on tapered polystyrene pegs that were glued to the inner surface of the shield with epoxy resin. The manufacture of these small pegs was avoided when the aluminum calorimeter was constructed by substituting polystyrene spheres. Spheres of the required sizes were obtained by sieving polystyrene Expandible Beads,[13] a molding resin consisting of remarkably accurate spheres with a distribution of diameters from a few thousandths to about one tenth inch. Spheres of that kind were also used to support the calorimeter in its aluminum mounting block.

f. Wire Arrangement

The wires entering the calorimeters were bonded to the shields with epoxy resin to improve the thermal contact, so as to prevent or

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[12]Methods for testing thermocouple wire for homogeneity are given by Wm. F. Roeser and H. T. Wensel, Methods of Testing Thermocouples and Thermocouple Materials, p. 284, Temperature, its Measurement and Control in Science and Industry (Reinhold Publ. Corp., New York, N. Y., 1941).

[13]Koppers Corporation, Pittsburgh, Pennsylvania.

reduce the exchange of heat between the core and the outside by way of the wires. The graphite shield was sufficiently thick to permit the machining of a slot in which the wires were embedded around a fraction of the circumference of the shield. The aluminum shield was not thick enough to permit that construction, however, and the bundle of wires entering the shield was simply cemented in place in the entry hole.

As a further step to reduce conduction of heat to the core along the wires, considerable excess length of the wires was allowed in the gap between the core and shield. Even so, conduction along the copper wires was nearly one half of the total heat leakage to the cores in both calorimeters.

5. Auxiliary Equipment

Three separate circuits shown on figure 15 are required for the operation of these simple calorimeters. They permit, A, control of shield temperature, B, indication of core temperature, C, calibration of core temperature indication in units of energy. The functioning of these circuits will be described and their components discussed in the following sections.

5.1. Shield Temperature Control Circuit (fig. 15A)

For an adiabatic condition of the core, the shield and core temperatures are equal and the voltage V_D generated by the opposed thermocouples C' and C" is zero. If a temperature difference develops, it is indicated in magnitude and polarity by a corresponding V_D. V_D is amplified, and, acting through a controller, changes the shield heater current so as to reduce V_D and restore the adiabatic condition. The entire calorimeter is operated above the temperature of the surroundings, so that heat can be lost from the shield when necessary.

a. Thermocouple Leads

The following precautions may be necessary when a relatively high degree of stability is required, as in a calorimeter for small dose rates. In both circuits the thermocouple wires were as short as possible and were protected by thermal insulation from the randomly changing temperature gradients of laboratory air. The thermocouple wires were handled carefully to avoid producing inhomogeneities by mechanical stress. Mechanical vibration during use was guarded against, and the entire thermocouple circuit was carefully shielded against electrical disturbances.

b. Reference Junctions

A melting ice reference temperature was used with the graphite calorimeter, which operated at 15 °C. It was necessary to use a desiccant to prevent moisture condensation on the cold wires.

A re-entrant glass cell containing solidifying diphenyl ether, which provides a temperature of 26.8 °C was used for the aluminum calorimeter's reference junctions.[14] The calorimeter was operated at a temperature of 29.3 °C. The basis for choosing the reference temperature is mentioned in a following section on core temperature indication.

c. Amplifiers

The amplifiers of figure 15 were high gain contact modulated d.c. amplifiers with low equivalent input noise and drift. Each contains an internal adjustable voltage source in series with the input. The voltage can be used to control the drift rate of the core temperature if, as invariably occurs, the drift rate is not precisely zero when $V_D = 0$.

d. Controller

The block marked CONTROL on figure 15 consisted of a recording millivoltmeter followed by a three-mode current adjusting controller equipped with a magnetic amplifier. That equipment, which was used with the aluminum calorimeter, provided a continuous controlling action and a pen-and-ink graph of the core-shield temperature differences.

The graphite calorimeter was operated with manual control of the shield temperature, an operator adjusting a multiple turn rheostat as required to keep V_D equal to zero. Manual control is not recommended because it requires the constant and exacting attention of an operator.

5.2. Core Temperature Indication Circuit (fig. 15B)

A single couple C''' produces a signal V_C corresponding to the core temperature which is measured by comparison to a potentiometer. An amplifier is used to secure the necessary accuracy of comparison. Changes in V_C correspond to energy deposited in or removed from the core.

a. Reference Junctions

In principle the junctions J of the thermocouple wires to copper can be maintained at any steady reference temperature. However, there is an advantage to the use of a reference temperature close to the core temperature. The total voltage V_C will then be relatively small, and excessive stability will not be required of the potentiometer with which changes in V_C are measured. When changes in V_C are very small

[14] Furnished by Dr. Chas. P. Saylor and Mr. Delmo Enagonio of N.B.S.

that advantage may be important. If experimental conditions permit, the core and reference temperatures should both approximate the room temperature, as gradients along the wires will then be small and the effect of wire inhomogeneity correspondingly reduced.

b. Potentiometer

The potentiometers used in this circuit need not be calibrated, but instead are required only to furnish a set of stable and reproducible potentials. The Lindeck constant resistance potentiometer circuit was used, as it is well suited to meet these requirements. It can be made relatively free from parasitic thermal emf's [15,16] which are of serious concern at microvolt levels.

5.3. Core Calibration Circuits (fig. 15C)

The purpose of these circuits is to permit the addition of accurately measured quantities of energy to the core so that the indications of core temperature will be calibrated directly in absolute energy units. If that procedure is followed then the quantities of energy deposited in the core by radiation are obtained directly from the core temperature indications with no dependence whatever upon knowledge of the heat capacity of the core, or upon the thermoelectric power of the measuring junction.

Accurate energy measurement was complicated by the tendency of the core heater resistance to drift slightly with temperature and also, in the aluminum calorimeter, to fluctuate slightly. The following methods were used to suppress the resulting power fluctuations.

a. Constant Voltage Circuit

In the circuit of figure 16A[17] the power to the core is supplied by a battery through a known resistor equal to the initial or nominal core resistance, R. Measurements of battery voltage, E, and core voltage, V, with an ordinary potentiometer then permit the calculation of power:

$$P = VI = V \frac{E-V}{R}$$

Since V is nearly equal to E/2, put

$$V = E/2 + v$$

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[15] Forest K. Harris, Electrical Measurements, p. 176 (John Wiley and Sons, New York, N. Y., 1952).

[16] Walter H. Wood, Rev. Sci. Instr. 28, No. 3, 202 (1957).

[17] Harold Hoge, Rev. Sci. Instr. 20, No. 1, 59 (1949).

Then $$P = (E/2+v)\ \frac{E/2-V}{R} = \frac{(E/2)^2-v^2}{R}$$

so that fractional changes in core power are equal to the squares of the fractional changes observed in core voltage. The battery voltage, E, is not disturbed by the changing core resistance, and can therefore be measured precisely; hence the power determination is accurate in spite of fluctuating core voltage. The fractional changes in core voltage observed for either calorimeter including drift and fluctuations were less than 0.01, so the fractional power uncertainty from that cause was less than 10^{-4}.

b. Constant Current Circuit

An adjustable constant power source that is more stable is shown in figure 16B. The basis for that circuit is the constancy of the collector current, J, of the transistor. The current is approximately equal to E_b/r, and is only very slightly affected by changes in the main battery voltage, Ec, or by changes in load resistance. The core is shunted by a known resistor that is equal to the initial or nominal core resistance R; for convenience, the series resistor is adjusted to half that value. Measured values of E and V permit the power to be calculated:

$$P = VI = V\ \frac{2E-V}{R}$$

Since V is very nearly equal to E, put

$$V = E + v.$$

Then $$P = (E+v) \cdot \frac{E-v}{R} = \frac{E^2-v^2}{R} \quad ,$$

so that fractional changes in power are equal to the squares of the fractional changes observed in core voltage. The values E and V are nearly equal for this circuit, so that the measurements of potential need not require range changes on the potentiometer. The power level can be continuously adjusted by varying r.

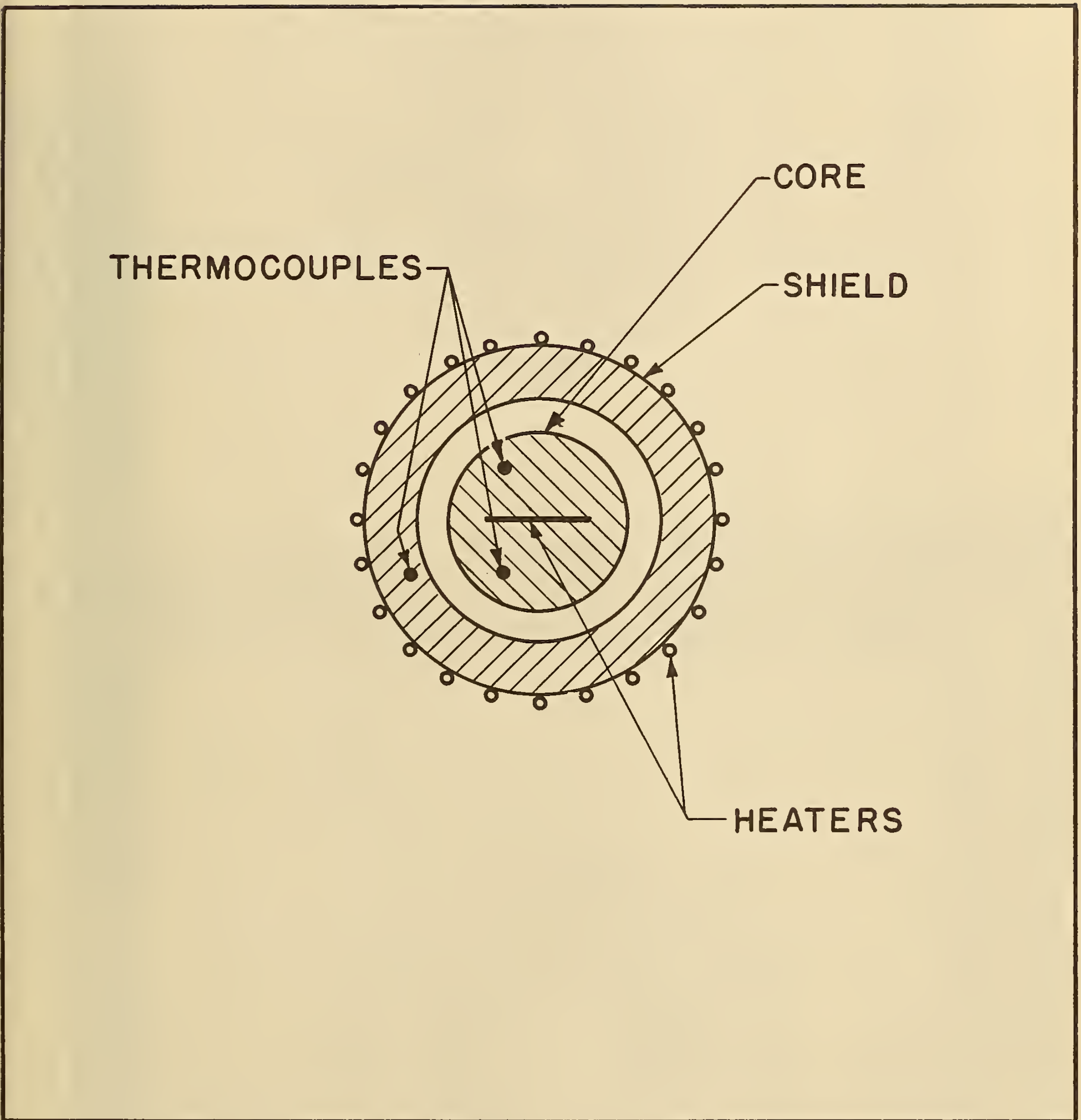

Fig. 1. Basic structure of adiabatic calorimeters.

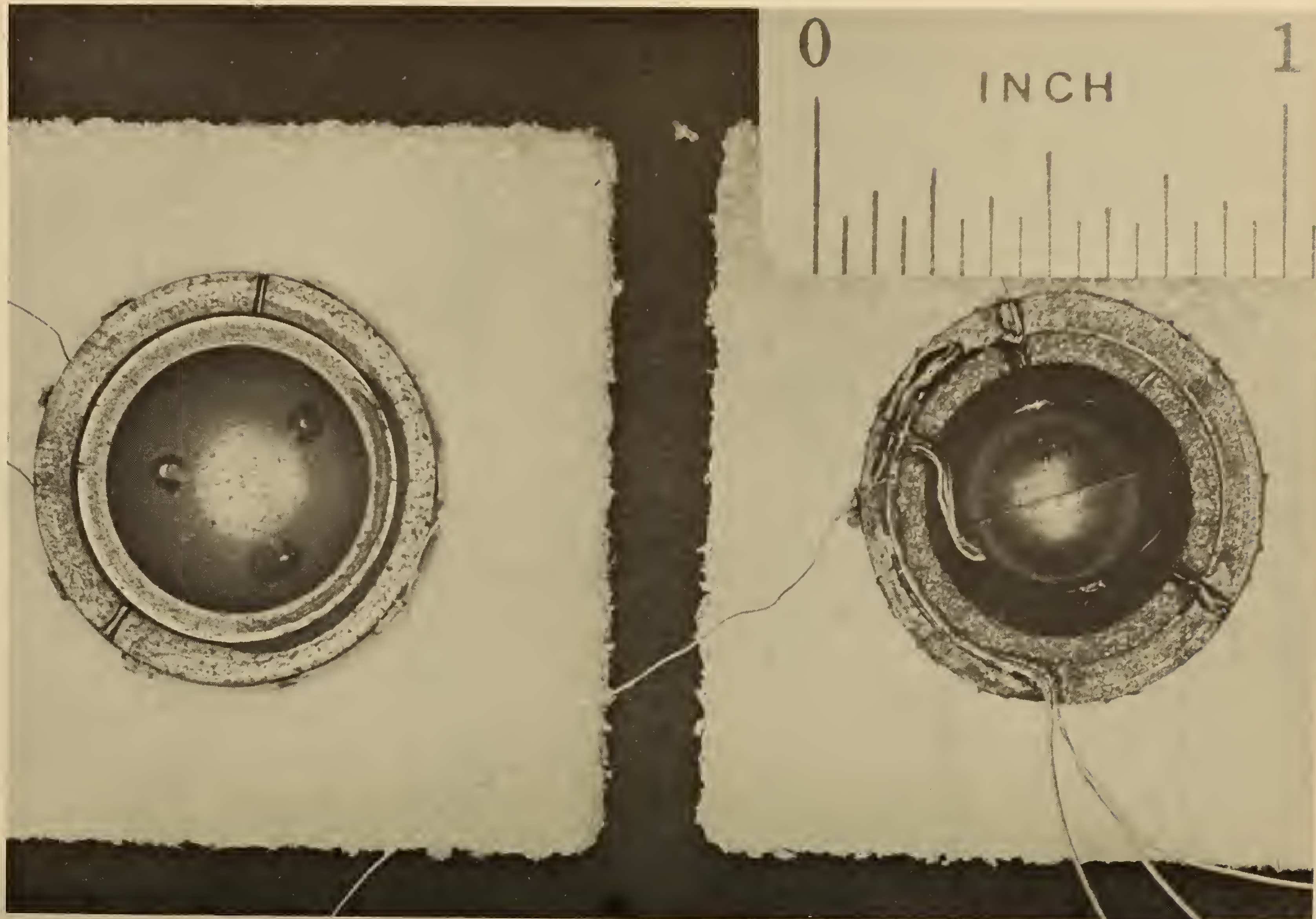

Fig. 2. Graphite calorimeter. Right, core in half of shield. Left, inside surface of remainder of shield, showing supporting pegs.

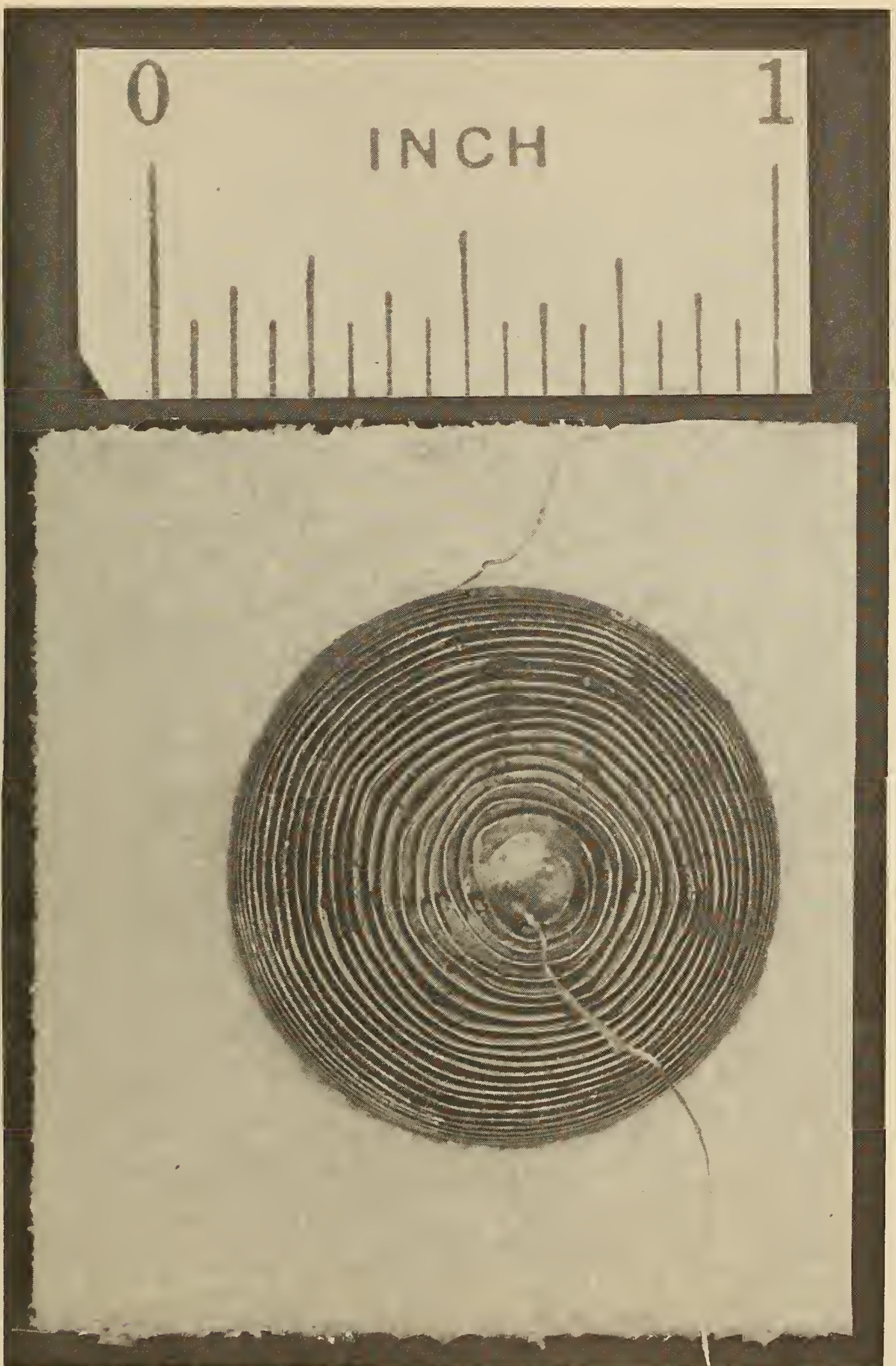

Fig. 3. Graphite calorimeter. Outside surface of shield showing heater wires.

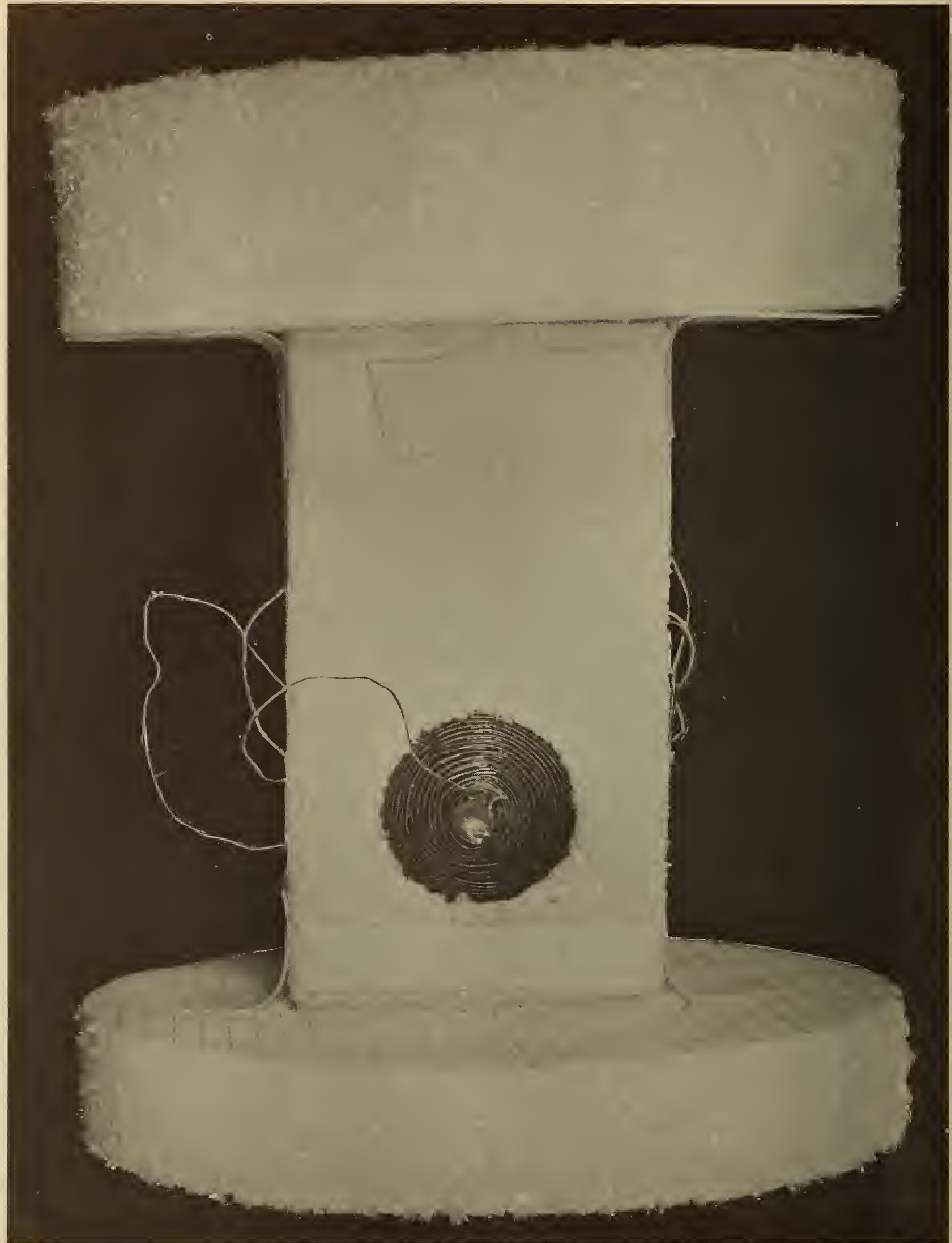

Fig. 4. Graphite calorimeter mounted in Styrofoam holder.

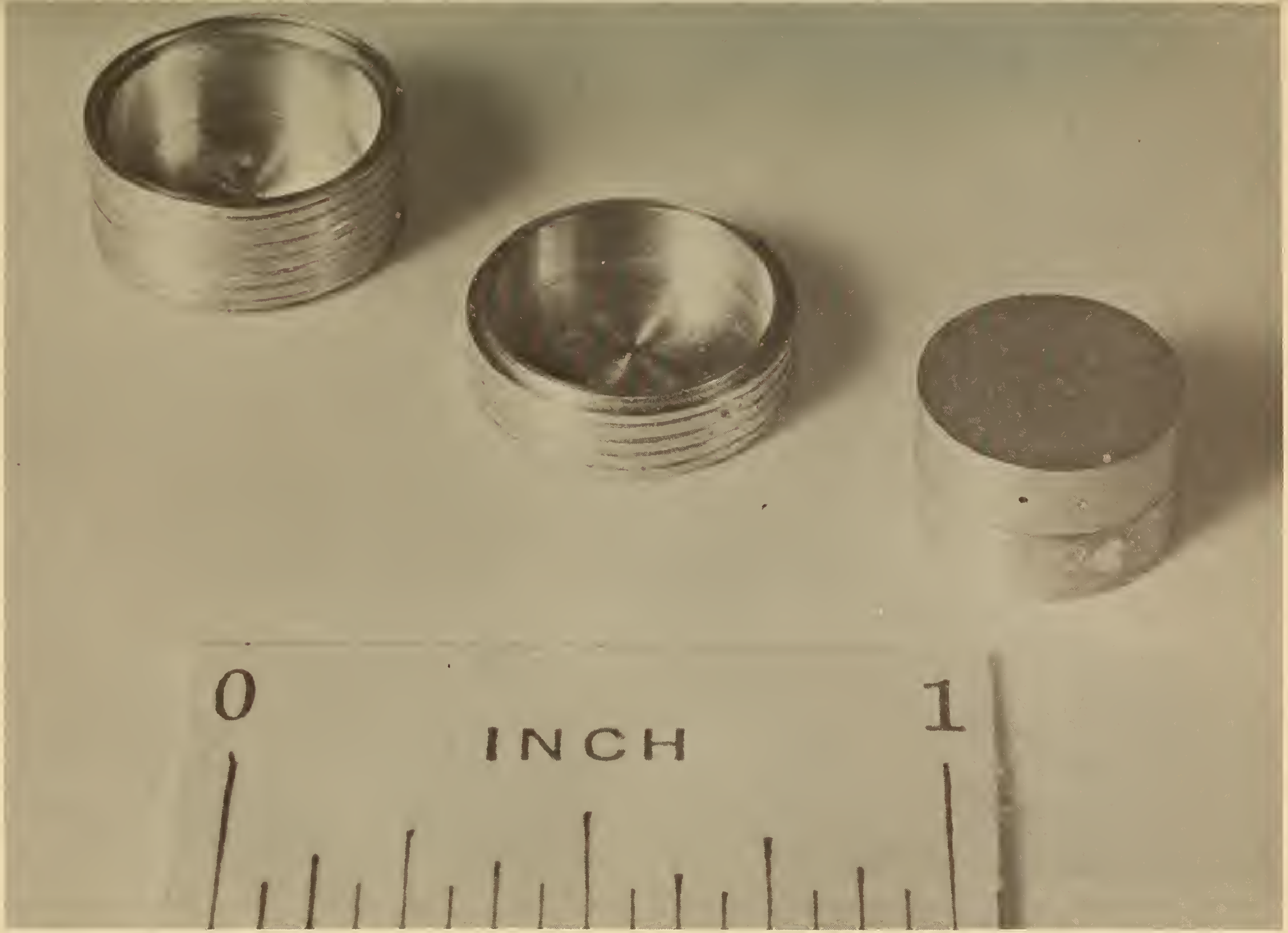

Fig. 5. Aluminum calorimeter. Shield and core before assembly.

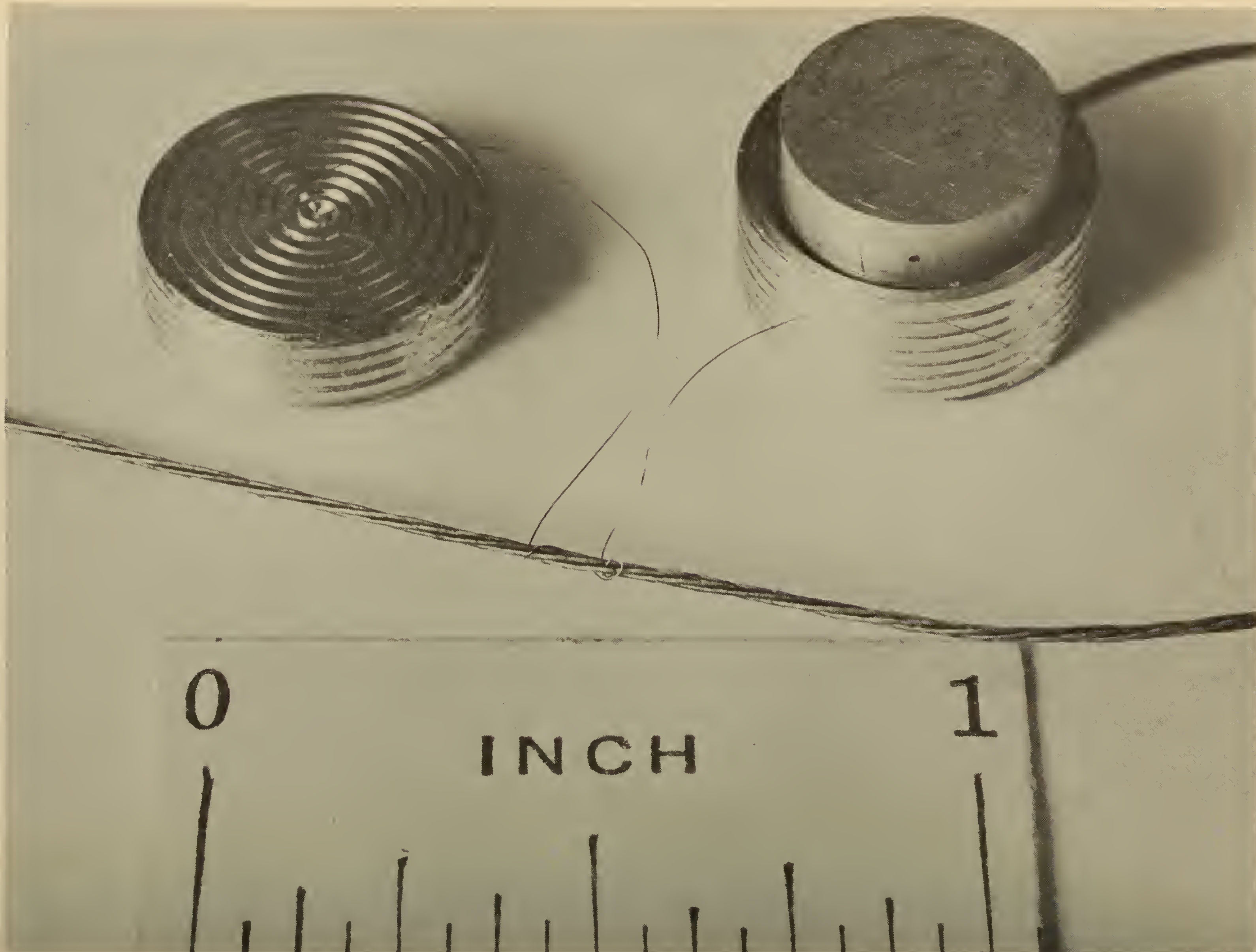

Fig. 6. Aluminum calorimeter, partly assembled. The cable contains four thermocouple wires and the pair of copper leads for the core heater.

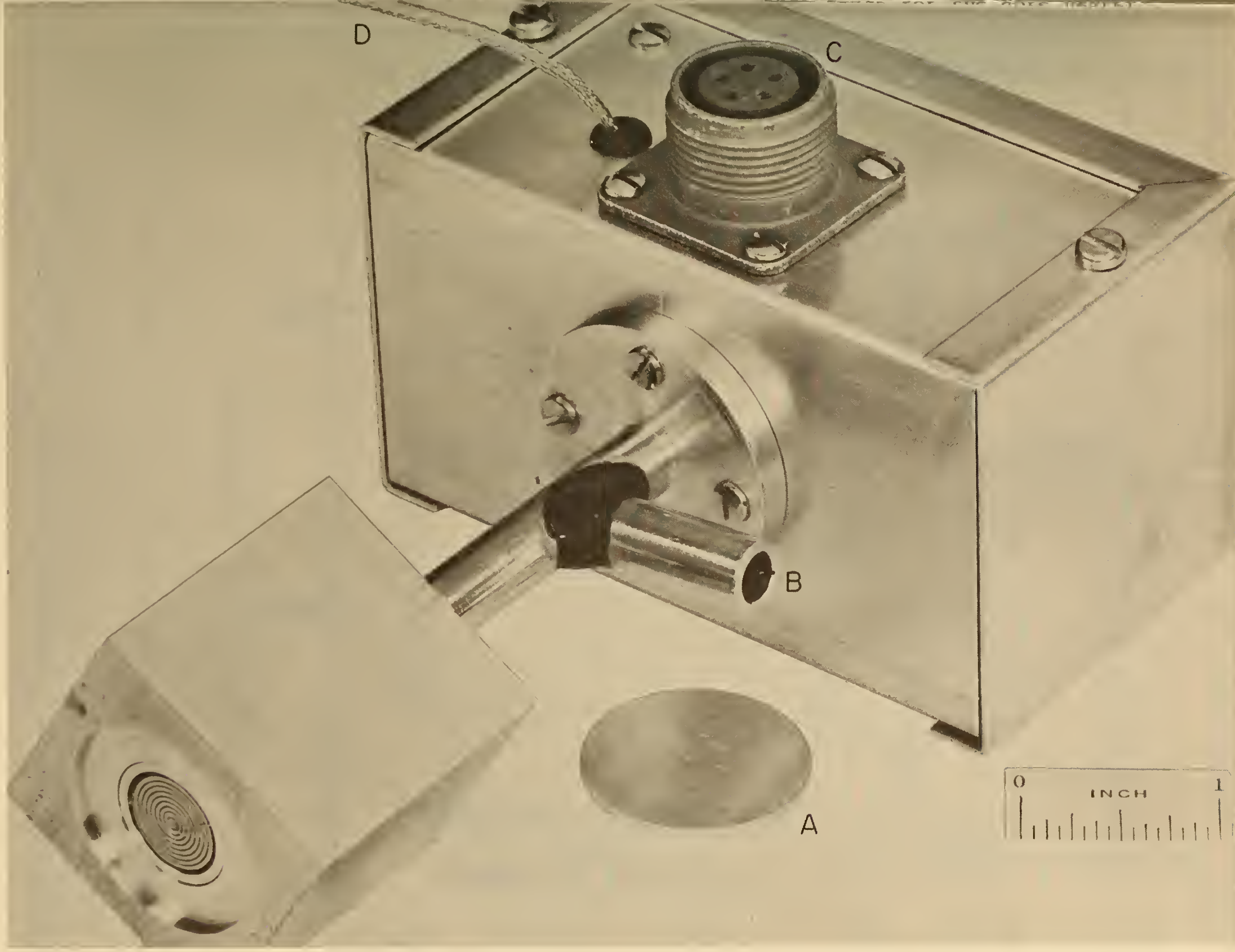

Fig. 7. Aluminum calorimeter mounted in aluminum block. A, aluminum lid. B, pumping stem. C, connections for shield and core heaters. D, thermocouple wires in shield.

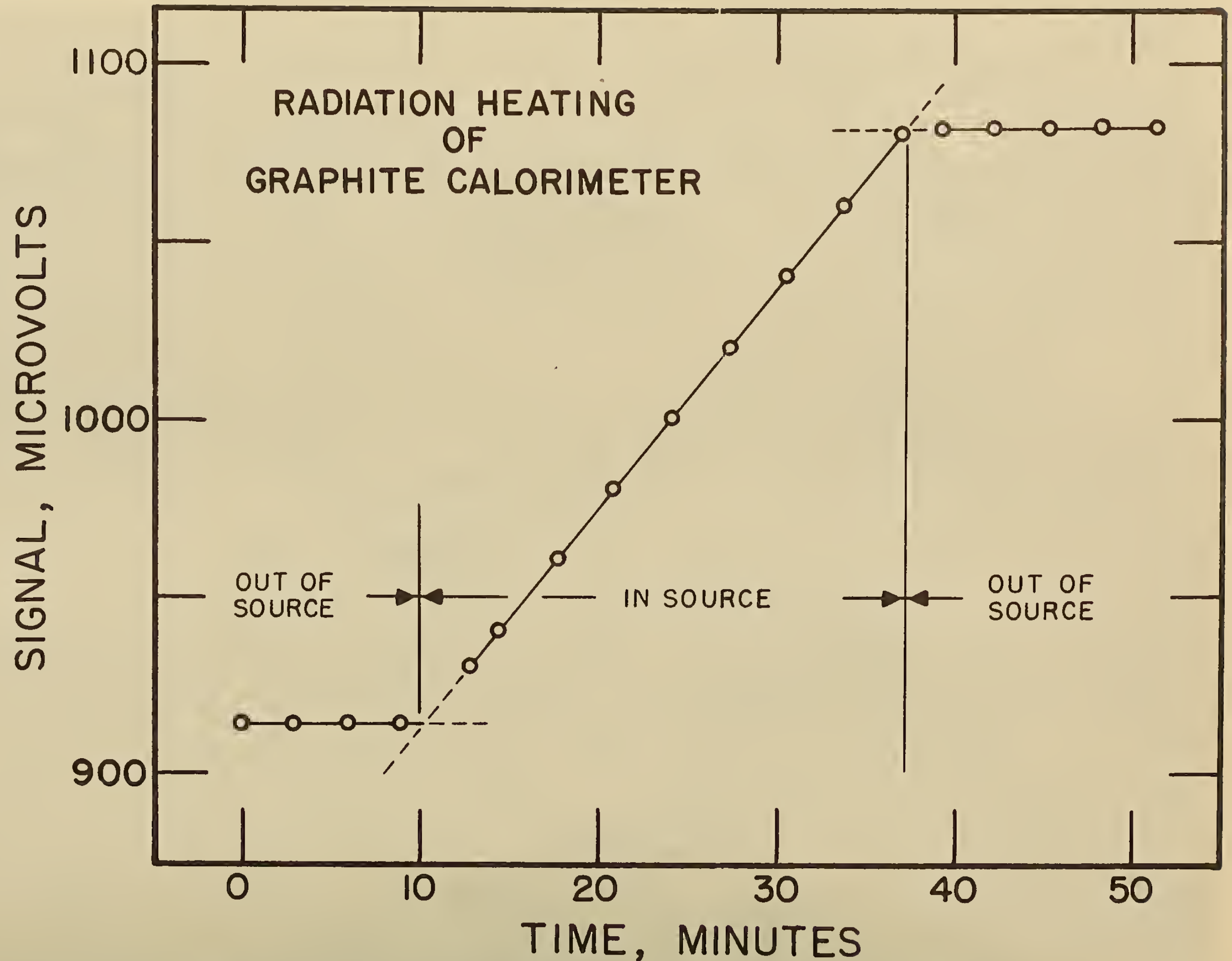

Fig. 8. Response of graphite calorimeter to heating by Co^{60} γ-rays. Dose rate, 135 rad/sec.

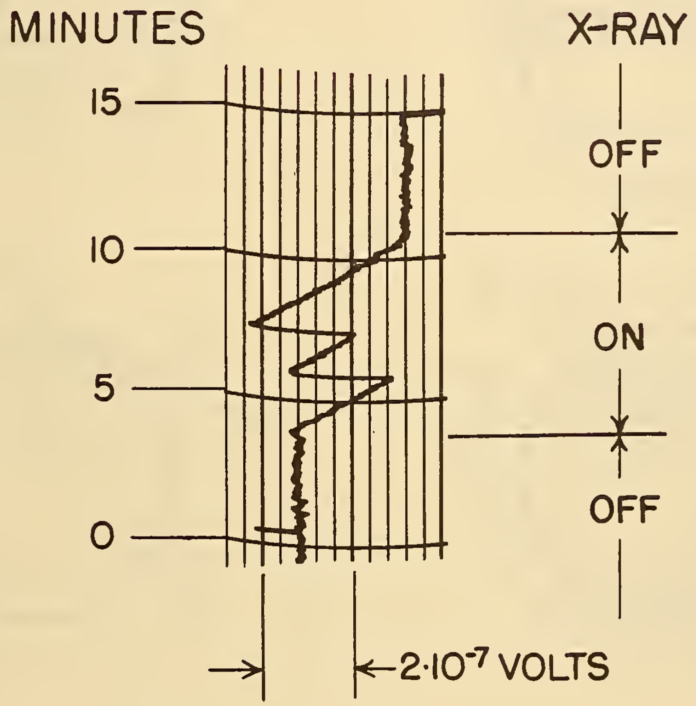

Fig. 9. Response of aluminum calorimeter to heating by X-rays (90 Mev peak). Dose rate, 0.86 rad/sec. The steps at 6 minutes and 7-1/2 minutes were produced by adjustments of the potentiometer with which the thermocouple voltage was measured.

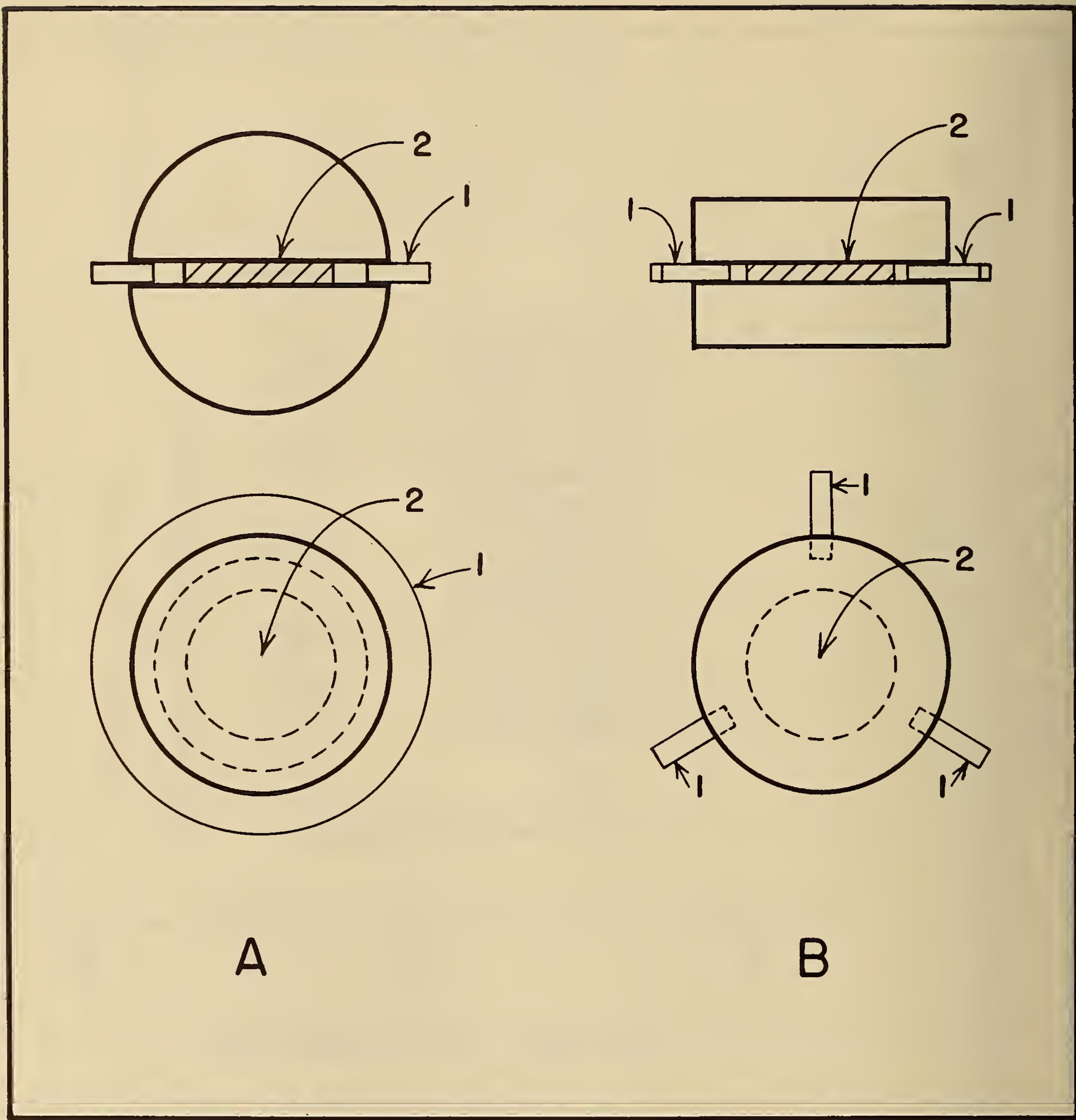

Fig. 10. Preparation of cores. A, graphite core: 1, Teflon washer 0.002" thick. 2, ink (10 parts carbon). B, aluminum core: 1, Mylar strips 0.0004" thick wiped with silicone grease. 2, ink (5 parts carbon).

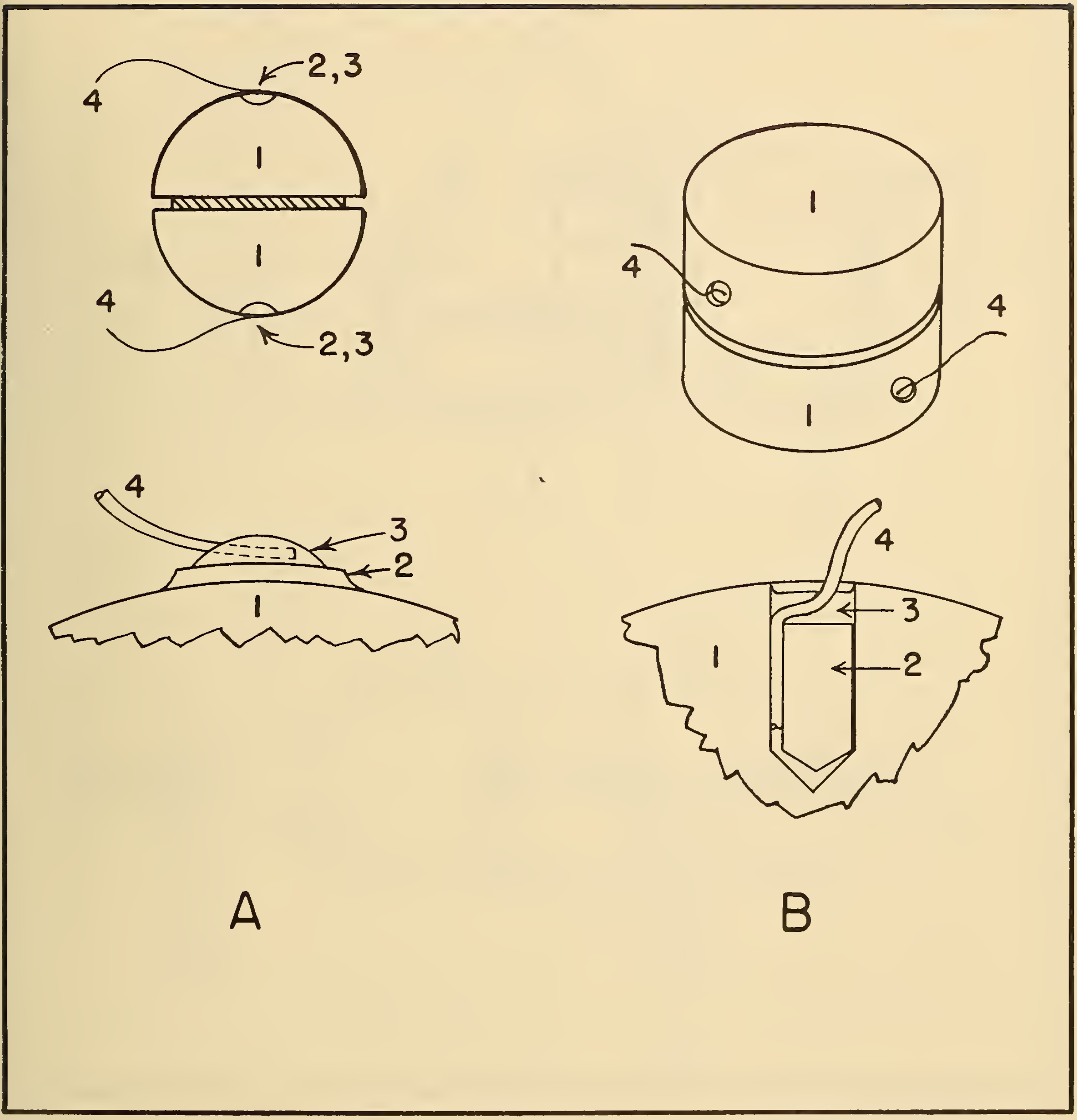

Fig. 11. Heater connections. A, graphite core: 1, graphite. 2, copper spot. 3, Wood's alloy. 4, copper power lead. B, aluminum core: 1, aluminum. 2, aluminum peg. 3, epoxy resin used to strengthen power lead. 4, copper power lead.

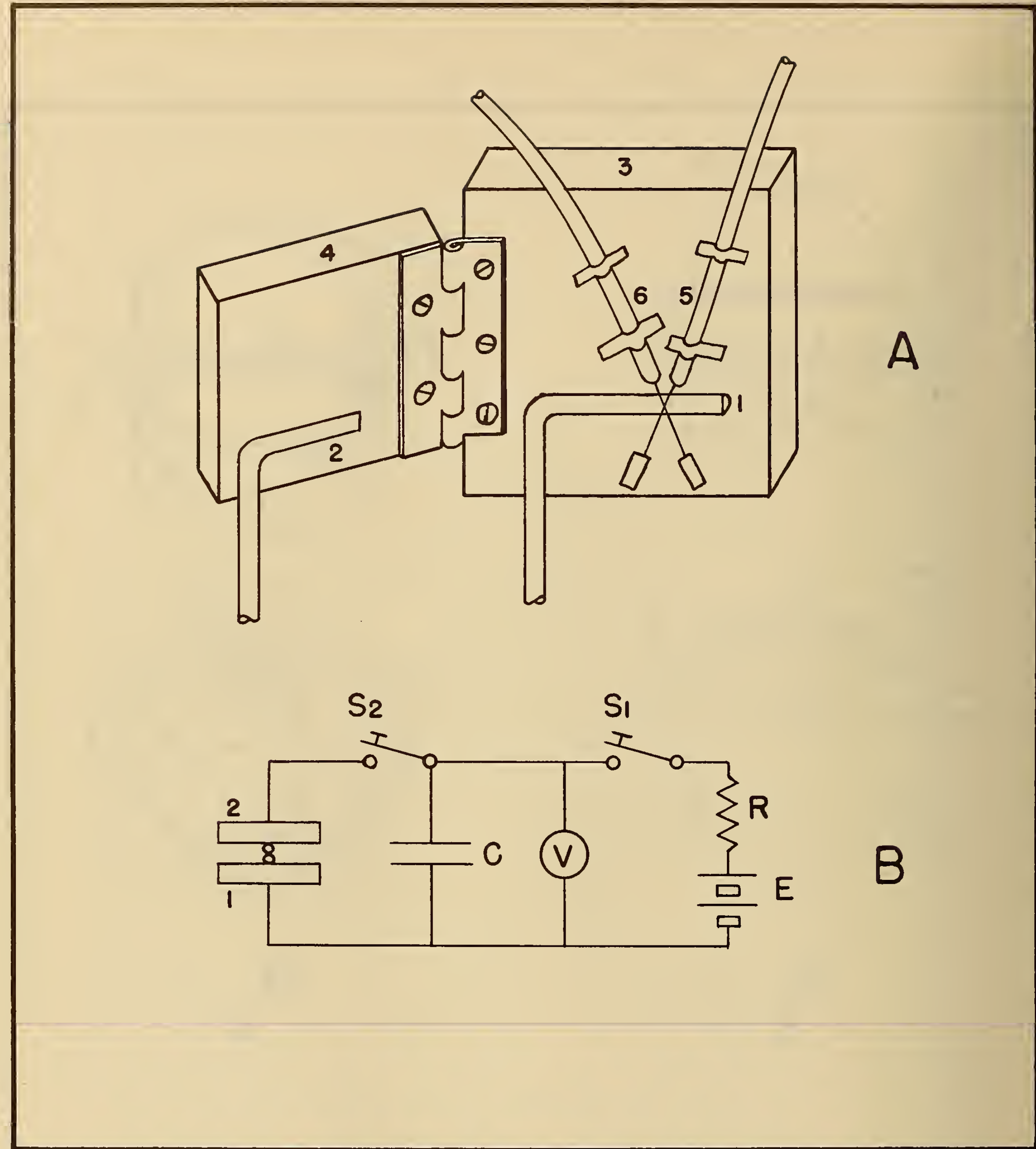

Fig. 12. Thermocouple welder. A, holder: 1-2, flat copper electrodes cemented to hinged blocks 3-4. 5-6, thermocouple wires. B, welding circuit: 1-2, electrodes. S_1, charging switch. S_2, welding switch. C, 200 µf oil capacitor. V, voltmeter. R, 100,000 ohms. E, 22 1/2 volts.

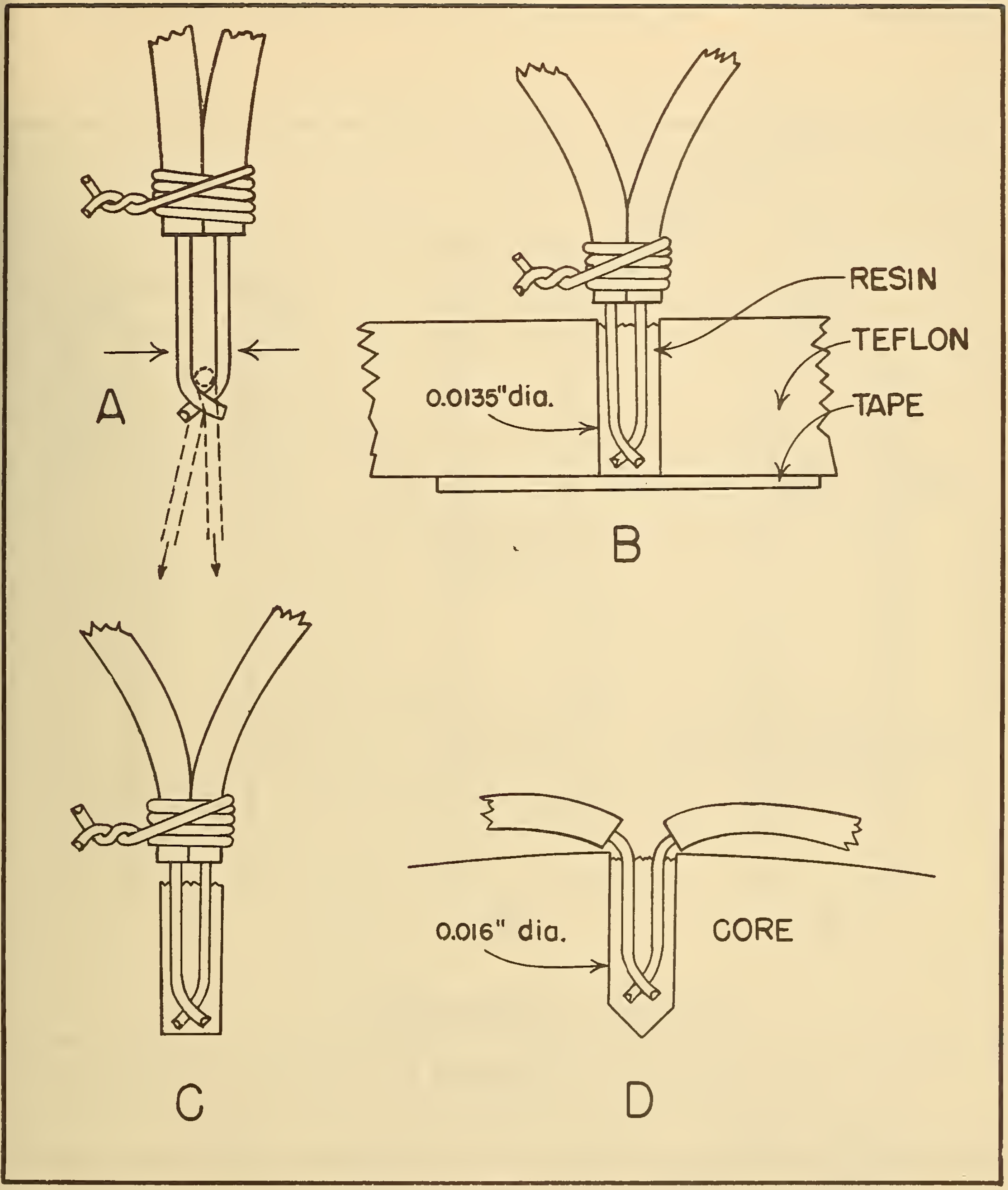

Fig. 13. Shaping and embedding thermocouples. A, shaping. B, method of preparing insulation. C, couple with insulation. D, insulated couple embedded in core.

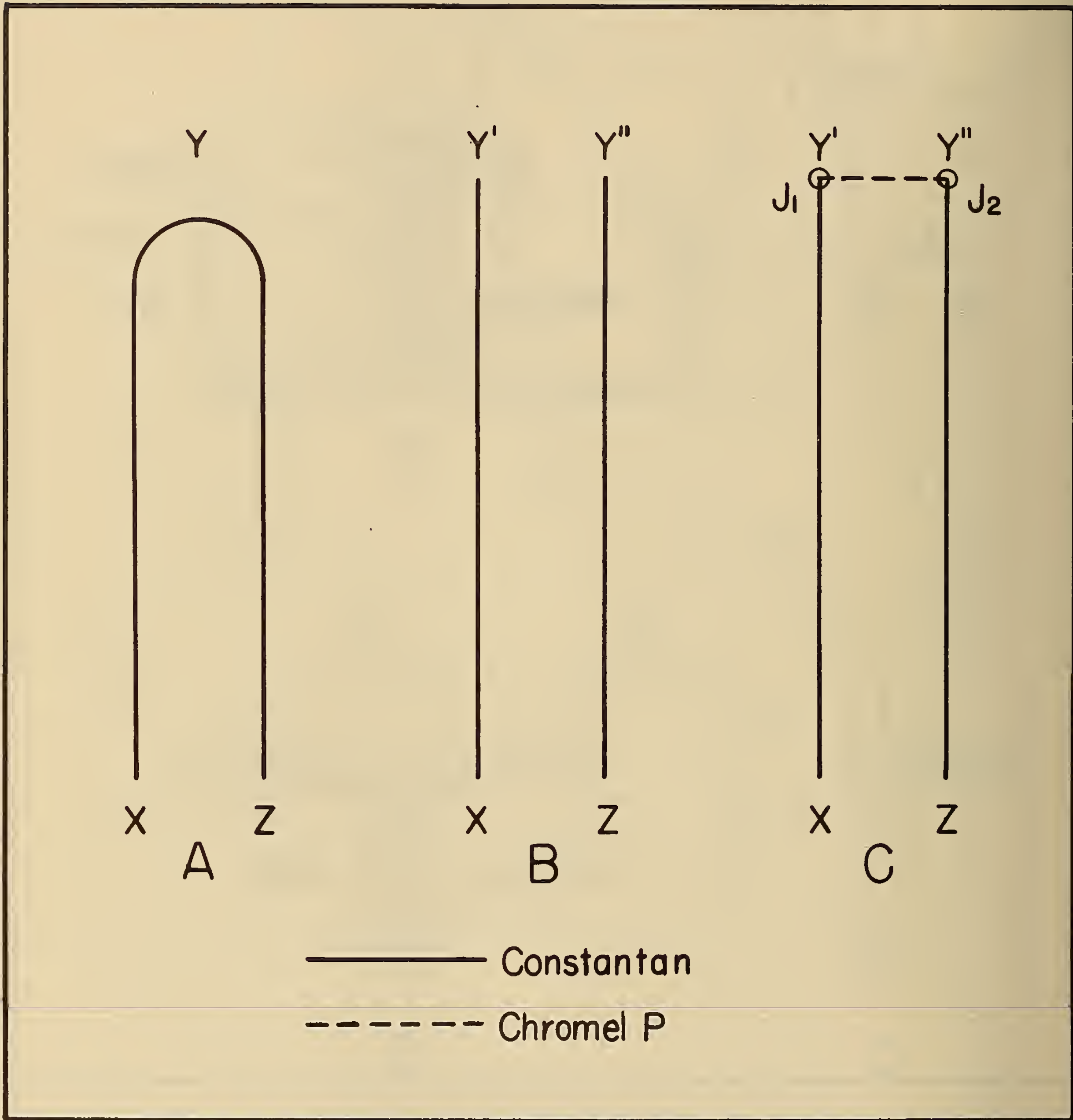

Fig. 14. Orientation of differential thermocouple wires. A, wire from spool long enough for both leads. B, wire cut in half. C, wire made into thermocouple pair with original ends, x-$\frac{y}{z}$, still free.

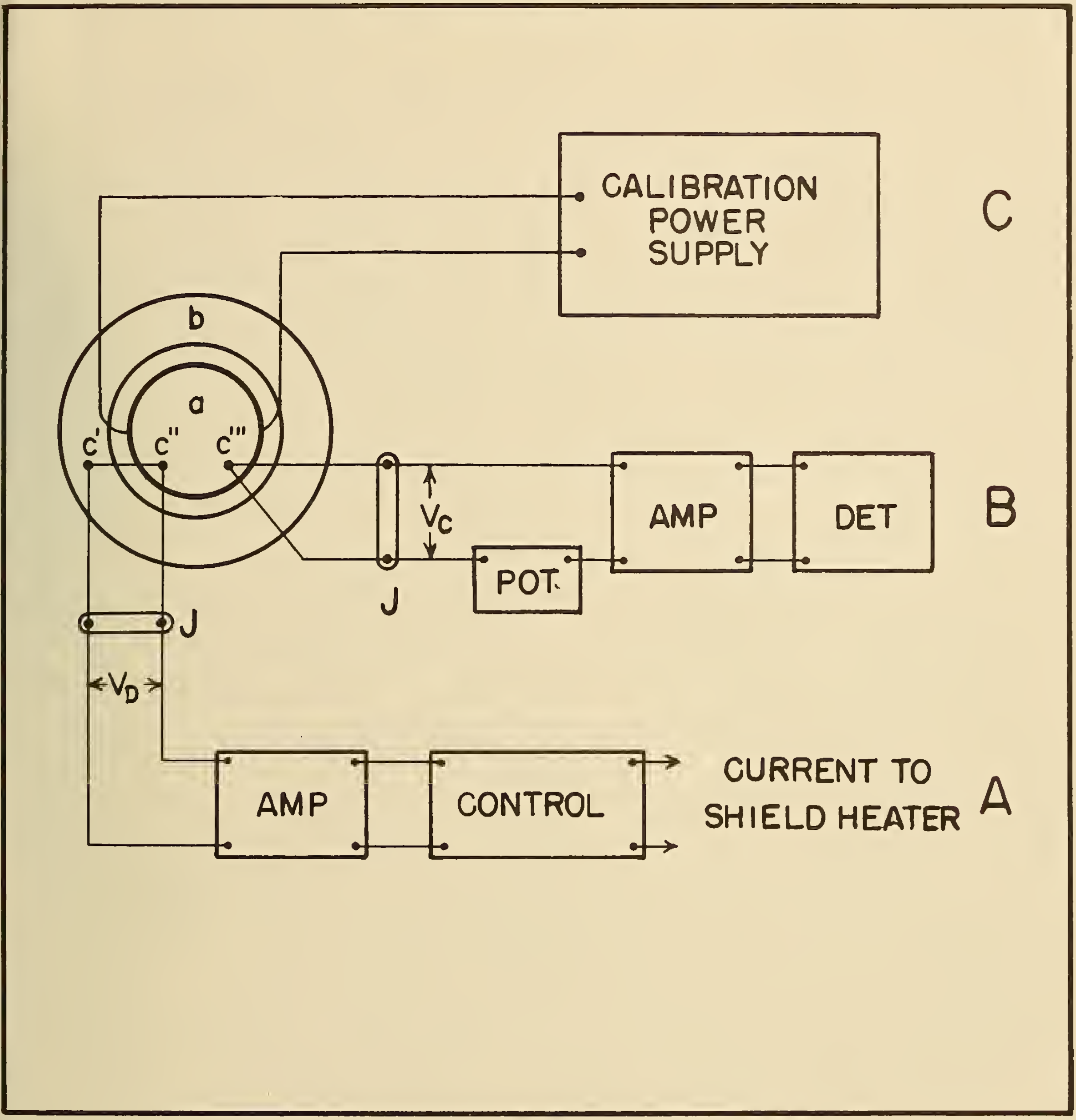

Fig. 15. Absorbed dose calorimeter with auxiliary circuits. A, shield temperature control. B, core temperature indication. C, calibration. a, core. b, shield, C'-C''-C''', thermocouples. J, constant temperature reference junctions. AMP, dc breaker type amplifier. CONTROL, three-mode current adjusting control. POT, Lindeck potentiometer. DET, chart recorder. CALIBRATION POWER SUPPLY, constant power source.

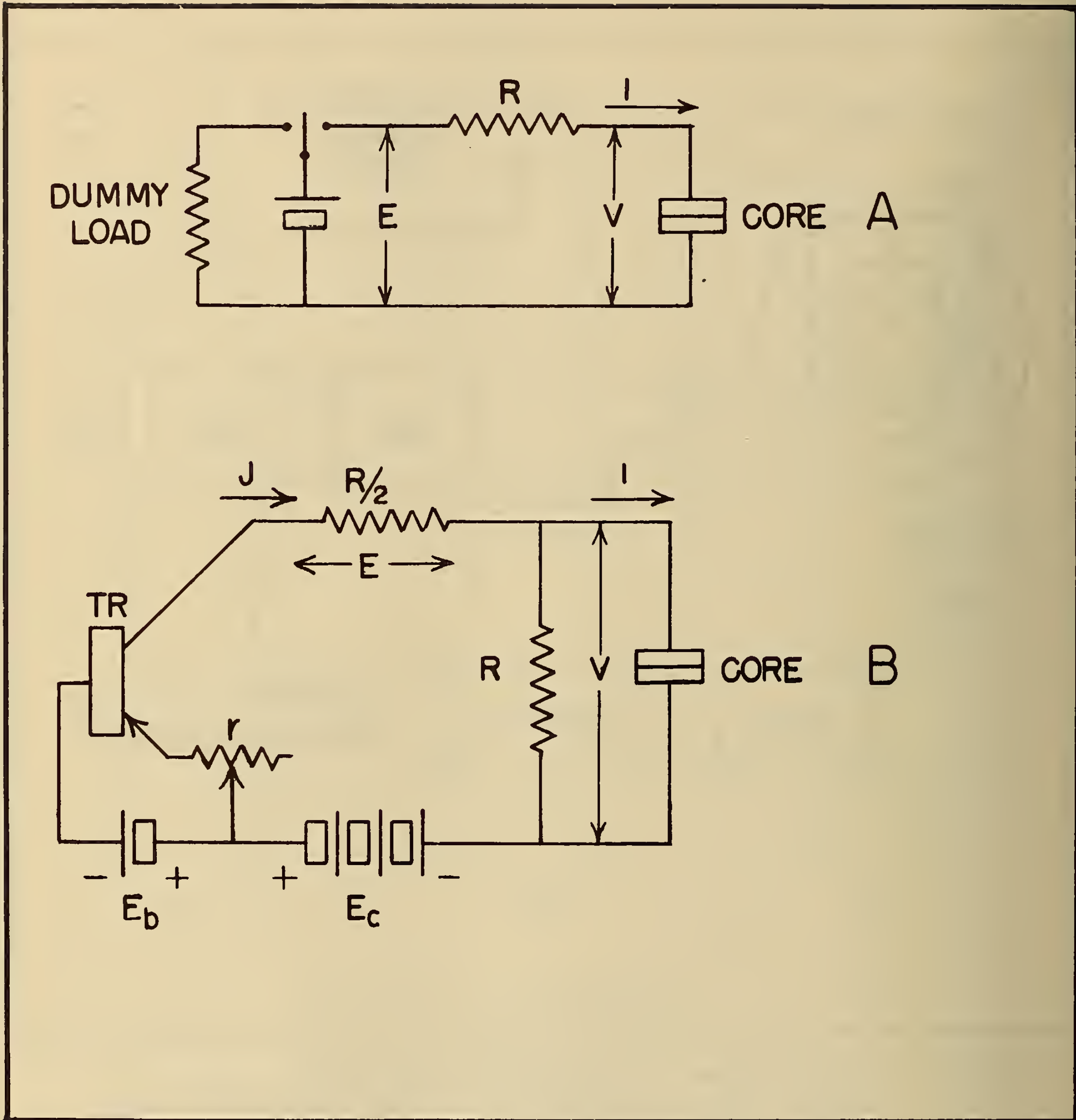

Fig. 16. Stabilized power sources. A, constant voltage type: R, stabilizing series resistance equal to core resistance. B, constant current type: TR, transistor type 2N404. E_b, 1.5 volt reference cell. Ec, 4.5 volt collector battery. R, stabilizing shunt resistor equal to core resistance. r, 50,000 ohm ten-turn rheostat.

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