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Twin-Tube Conduction Radionuclide Calorimeter

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ABSTRACT

The complete calorimeter system used to measure powers from 0.1 W to 2 W of samples 1.5 in. in diameter by 5 in. in length is described. Calorimeter readout is by digital voltmeter. A sensitivity of 1501 mV/W at 1.327 W with an accuracy of 99.9% was obtained. Sample equilibrium time is 4 h. The twin-tube temperature measurement system compensates for thermostat temperature fluctuations. Thermistors measure the temperature gradient produced by heat flow through a 5-in. length of 1.75-in.-o.d., 0.035-in.-wall brass tube. The thermistors are supplied with a constant operating current by current regulator diodes to simplify and improve operation. An integral calibration heater surrounds the sample chamber. Reliability is enhanced by all-solid-state design.

I. INTRODUCTION

This calorimeter is an isothermally jacketed twin-tube calorimeter of the heat flow type. It provides a nondestructive assay of radioactive samples. The design was aided by information contained in a review paper by Gunn,[1] and by a Mound Laboratory report on their calorimeters.[2]

The design of this radionuclide calorimeter was influenced by many interrelated requirements and special considerations. It was necessary to produce a satisfactory operating system in a short time using components on hand or available from the LASL stock room. Reliability, sensitivity, ease of operation and low cost were major considerations. The calorimeter was required to measure the power of samples of 0.1 W to 2 W with an accuracy of 99.9% in a reasonable time. The sample was a 1.5-in.-o.d., 5-in.-long cylinder. The calorimeter output must be sufficient for readout on a digital voltmeter with adequate accuracy and precision. It is believed that all the requirements have been fulfilled by this design, which has been operating satisfactorily for over 1-1/2 yrs.

The twin-tube design was adopted to minimize thermostat temperature fluctuations. The use of the most sensitive temperature sensing devices, thermistors,[3] was dictated by the requirement for a large output voltage which could be easily read on a digital voltmeter. An operational amplifier connected between the differential thermistor circuits and the digital voltmeter provides isolation and necessary amplification at low sample powers.

The main calorimeter tube has a sample cavity at the top end. This cavity has a nonremovable heater tube which is used to calibrate the calorimeter over a range of electrical power levels.

Four series-connected equally spaced thermistors are cemented to the calorimeter tube at the bottom of the sample cavity and 4 others are similarly cemented to the base of the calorimeter tube, a spacing of 5 in. These measure the temperature gradient produced by heat flow from the sample or calibration heater to the thermostat bath.

Current regulator diodes supply a stable current to the thermistors of each identical tube. The voltage produced across the two thermistor circuits is cancelled at the input of the summing operational amplifier. Heat flow in a tube assembly will lower the thermistor resistance and produce a corresponding output millivoltage from the amplifier which is read on the DVM. Equilibrium time for the heater is 2-1/2 h. A typical sample reaches equilibrium in 4 h.

II. DESIGN OF CALORIMETER

A. Physical and Thermal Design

1. Calorimeter Assembly. Figure 1 shows the twin-tube calorimeter assembly which contains the two calorimeter tubes. The assembly is immersed in an 88-gal thermostat water bath 22 in. deep and 36 in. in diameter, which is kept at a constant temperature of 28.3°C by a commercial temperature controller.*

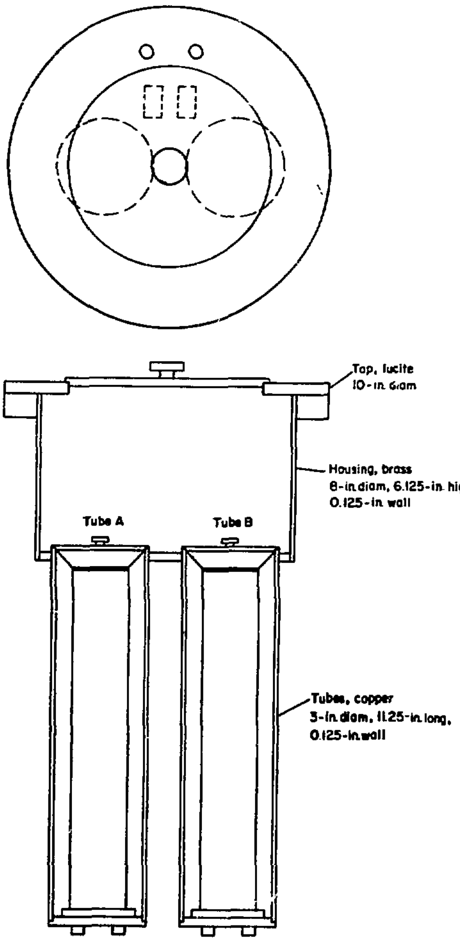

Fig. 1. Twin-tube calorimeter assembly.

*Precision Temperature Controller Model 40, Tronac Inc., Orem, Utah.

The air-conditioned room is maintained at 25°C. A 1/20 hp stirrer vigorously agitates the water.* A 14-W Rotron coaxial muffin fan directs a stream of air onto the stirrer motor to remove heat which would otherwise heat the thermostat water above the control range of the temperature controller. A safety switch in series with the thermostat heater turns power off if the bath temperature exceeds 29.3°C.

2. Calorimeter Main Tube. Figure 2 shows the organization and dimensions of the calorimeter main tube. The 1.5-in. sample diameter determined the i.d. of the copper heater tube in which it fits. The heater tube fits inside the brass main calorimeter tube. The thin 0.035-in.-wall main tube permits a maximum temperature gradient to be developed by sample or heater power, which flows down to the copper base of the main tube and then out to the thermostat bath. The main tube was insulated with a 1/2-in.-thick tube of styrofoam to reduce convec-

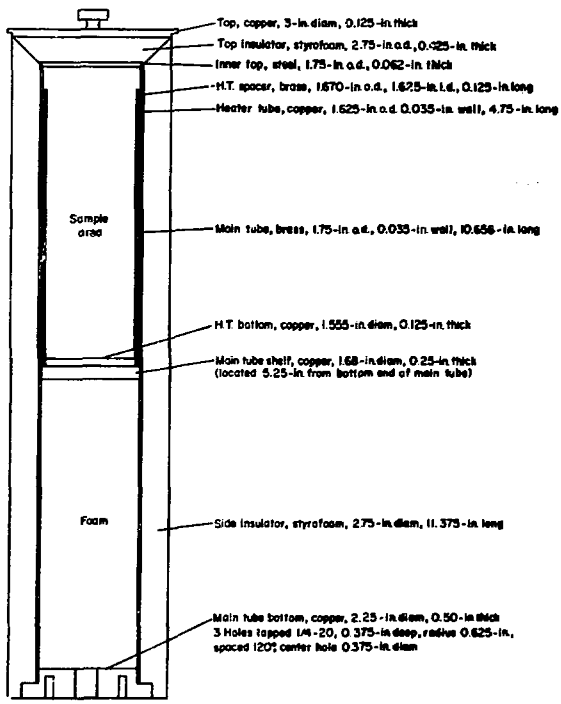

Fig. 2. Calorimeter main tube.

*Stirrer Type NSI-53, Central Scientific Co.

tion air currents. The lower cavity of the main tube was filled with solidifying liquid foam for the same reason.

B. Electrical Design

The twin-tube differential thermistor design was adopted to minimize the effects of thermostat temperature fluctuations. Refer to Fig. 3, the temperature measurement circuit, to see how this operates. Assume equal currents through the 4 groups of thermistors, R_{AU}, R_{AL}, R_{BU} and R_{BL}. When the resistances of the 4 groups of thermistors are equal, the voltage drops will be equal, and the differential voltage will be zero. See Eq. (1).

$$(E_{AU} + E_{AL}) - (E_{BU} + E_{BL}) = 0 \quad (1)$$

Should the bath temperature change, it would affect R_{AL} and R_{BL} equally. E_{AL} and E_{BL} would change in the same direction and cancel. Therefore, there would be no change in the differential output voltage.

1. Temperature Measurement Circuit. Disc-type thermistors,* 5 k Ω at 25°C, stock items at LASL, were selected to sense the temperature gradient produced by heat flowing through a 5 in. length of the main calorimeter tube. Four series-connected thermistors equally spaced around the circumference of the main calorimeter tube at the height of the 1/4-in.-thick copper shelf respond to the temperature at that level. These correspond to the resistor labeled R_{AU} of Tube A in Fig. 3. These are connected in series with a second group of 4 series-connected thermistors cemented to the end of the main tube. An identical configuration was duplicated as closely as possible in constructing Tube B.

Each of the 2 groups of series-connected thermistors is supplied with a constant current of approximately 0.47 mA furnished by a separate current regulator diode.

The 2 groups of thermistors in each calorimeter tube circuit were selected and matched equally in resistance, 17.4 k Ω. A voltage drop of 16.4 V is produced across the 8 thermistors of each circuit.

The output of the 2 thermistor circuits is connected to a summing operational amplifier, A. The positive and negative voltage levels from the twin-tube thermistor circuits effectively cancel at the input of the amplifier. The amplifier is generally used with a gain of X1.

2. Calibration Power Measurement Circuit. The power measurement circuit for the 2 calibration heaters is shown in Fig. 4. Heater selector switch, SW2, allows either heater to be supplied with regulated power from adjustable power supply No. 4. Heater current switch, SW1, selects either a 10-Ω or 100-Ω precision resistor for measurement of high or low heater currents. Heater voltage is measured across separate leads connected to the 900-Ω heater element.

3. Current Regulator Diodes. To eliminate current adjustments, current regulator diodes* are used to supply a constant current of approximately 0.47 mA to each of the differential thermistor circuits. The diodes, D1 and D2, are powered by 2 regulated

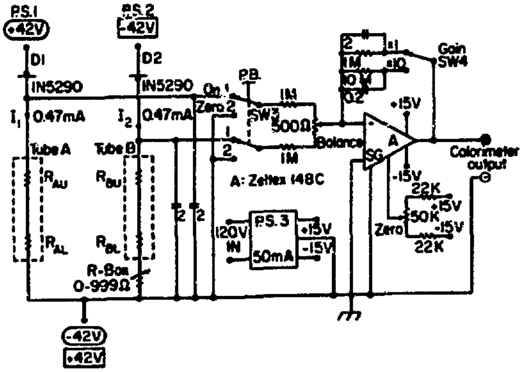

Fig. 3. Temperature measurement circuit.

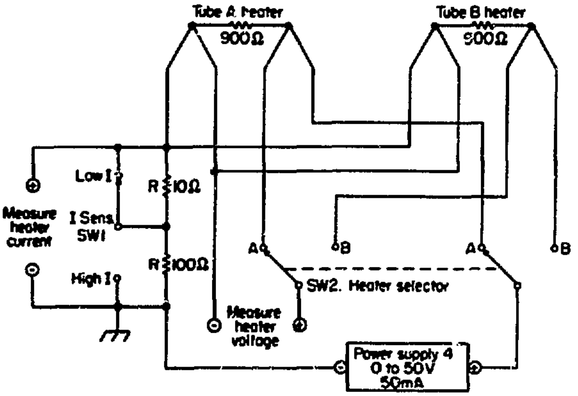

Fig. 4. Calibration power measurement circuit.

*General Electric thermistor No. 1D-103, 10% tol., 0.100-in. diam by 0.048 in. thick, 4-mW dissipation constant, 8-sec thermal time constant, $.35 each.

*Mfg. by Motorola Semiconductor Products.

power supplies of opposite polarity. The diodes were selected for approximately equal regulated currents and low noise characteristics. The 2-μF capacitors eliminate high frequency noise. The diodes operate with 25-V bias.

III. ASSEMBLY AND CONSTRUCTION

The successful completion of the calorimeter depends on careful attention given to many details. The most important of these are described below.

A. Calorimeter Assembly

The calorimeter assembly consists of an 8-in.-diam brass container which has 2 copper down tubes attached to the bottom plate. Construction was by silver soldering. Access to the calorimeter tops is through a removable Lucite top. Eight wires from each calorimeter tube terminate at 2 small receptacles mounted on the inner horizontal surface. Cables with attached plugs lead out through holes in the outer Lucite ring of the calorimeter housing. The entire assembly is supported by a 1/2-in.-thick segment of Lucite, with an 8-1/4-in.-diam hole, resting on a stainless steel frame.

B. Calorimeter Main Tube

Soft soldering was used to minimize warping when assembling the bottom and shelf to the main tube. The 2 spacer rings and bottom of the heater tube were also assembled with soft solder.

After each main tube was completed with the electrical components and wires attached, it was bolted to the bottom of the down tube with three 1/4-20 bolts. Dow Corning 340 Silicone heat sink compound was used at the base of the main tube to improve heat conduction. Dow Corning Silastic 732 RTV sealant was used around the bolts to make them water tight.

1. Heater Element. The 900-Ω heater element was made of Manganin wire, Alloy A, SNC No. 36, 0.005 in. in diameter, 27.1 Ω per ft. Sixteen feet, 7-1/4 in., of this wire was bifilar wound on the outside of the heater tube by hand. The winding was sprayed with enamel when completed. Two pairs of No. 30 stranded-copper nylon-insulated wire attach to the 2 element leads at the top end of the heater tube. They pass through a slot 5/16 in. wide, which was removed from the upper spacer. The 4 wires then pass through a horizontal slot cut through the main tube, down the main tube where they combine with the 4 wires from the 2 groups of thermistors. Then they travel upward on the outside of the styrofoam side insulation, through a slot machined in the top edge of the copper down tube and to the receptacle, a Cannon subminiature type D, 9 contact item.

2. Thermistors. A group of 40 thermistors, which were uninsulated, were sprayed with 3 coats of enamel. Their individual resistance was measured with a low-current ohmmeter while they were immersed in the thermostat bath. Four groups of 4 thermistors of equal total resistance were selected from these. They were cemented to the outside of the 2 main tubes with 5-min Devcon epoxy cement. Four were cemented equally spaced around the circumference at the height of the shelf, and the second set of four was cemented 1/8 in. from the bottom end of the main tube. The 4 thermistors in each group were connected in series. The thermistor wires joined with the heater leads, then traveled up along the outside of the main tube insulation to the receptacle.

C. Current Regulator Diodes

Current regulator diodes maintain a constant current in a series circuit when they are kept at constant temperature. The diodes, D1 and D2, are mounted in a silicone-oil-filled metal container, which is partially submerged in the thermostat bath.

IV. OPERATION OF CALORIMETER

The calorimeter is usually operated by the replacement method. In this method the sample at equilibrium causes a temperature rise in the calorimeter which produces a proportional change in the calorimeter output voltage, ΔE_S. The sample is then removed from the calorimeter, and the calibration heater voltage is adjusted to produce a nearly equivalent amount of electrical power, P_H, which produces a calorimeter output voltage, ΔE_H. The last proportionality, $\Delta E_H/P_H$, is the sensitivity of the calorimeter at that particular power. It decreases with increasing power, because the resistance of the thermistors decreases with increasing temperature. The sample power is found by dividing the calorimeter output sample voltage by the sensitivity.

$$P_S = \Delta E_S P_H/\Delta E_H \quad . \qquad (2)$$

Either Tube A or Tube B may be used to measure a sample by the replacement method. The tubes must be individually calibrated, because they are not exactly equal in sensitivity.

A. Measurement of Sample Power

The output of the amplifier is adjusted to zero by pressing switch, SW3, and using the Zero adjustment. This is an infrequent adjustment. The Balance control is used to compensate for differences in the two 1-M Ω input resistors, and need be adjusted only once after construction.

The R-box is adjusted to bring the output of the amplifier to zero voltage. The sample is then placed in the Tube A sample chamber. A magnet is handy for removing the steel inner top. After the calorimeter output has stabilized, time required is approximately 4 h, the output reading is taken and recorded. The total output voltage change divided by the sensitivity at this output voltage will give the value of sample power. If no sensitivity factor is available near the sample millivoltage, the calibration heater may be run to obtain one.

Table I lists representative sensitivity factors for Tube A.

B. Measurement of Calibration Heater Power

Figure 4 shows the power measurement system for the calibration heaters. Measurement of the power supplied by the calibration heater consists of measuring the heater voltage and the heater current separately, and obtaining the product, $E_H I_H$. The heater current is obtained by measuring the voltage developed across the series resistance, R, and dividing by the proper value of resistance. The heater voltage is measured directly at the heater voltage leads. The calorimeter output divided by the heater power gives the sensitivity at that power.

TABLE I

TYPICAL SENSITIVITY OF CALORIMETER TUBE A

Power (W)	Sensitivity (mV/W)
0.100	1737
0.251	1704
0.500	1650
0.750	1602
1.000	1555
1.303	1505
1.503	1471
1.730	1435
2.000	1396

ACKNOWLEDGMENT

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2. "Precision Calorimetry at Mound Laboratory," Director, Nuclear Operations, Monsanto Research Corporation, Mound Laboratory, Miamisburg, Ohio 45342.
3. "Capsule Thermistor Course," Fenwal Electronics, Inc., 63 Fountain St., Framingham, Mass.