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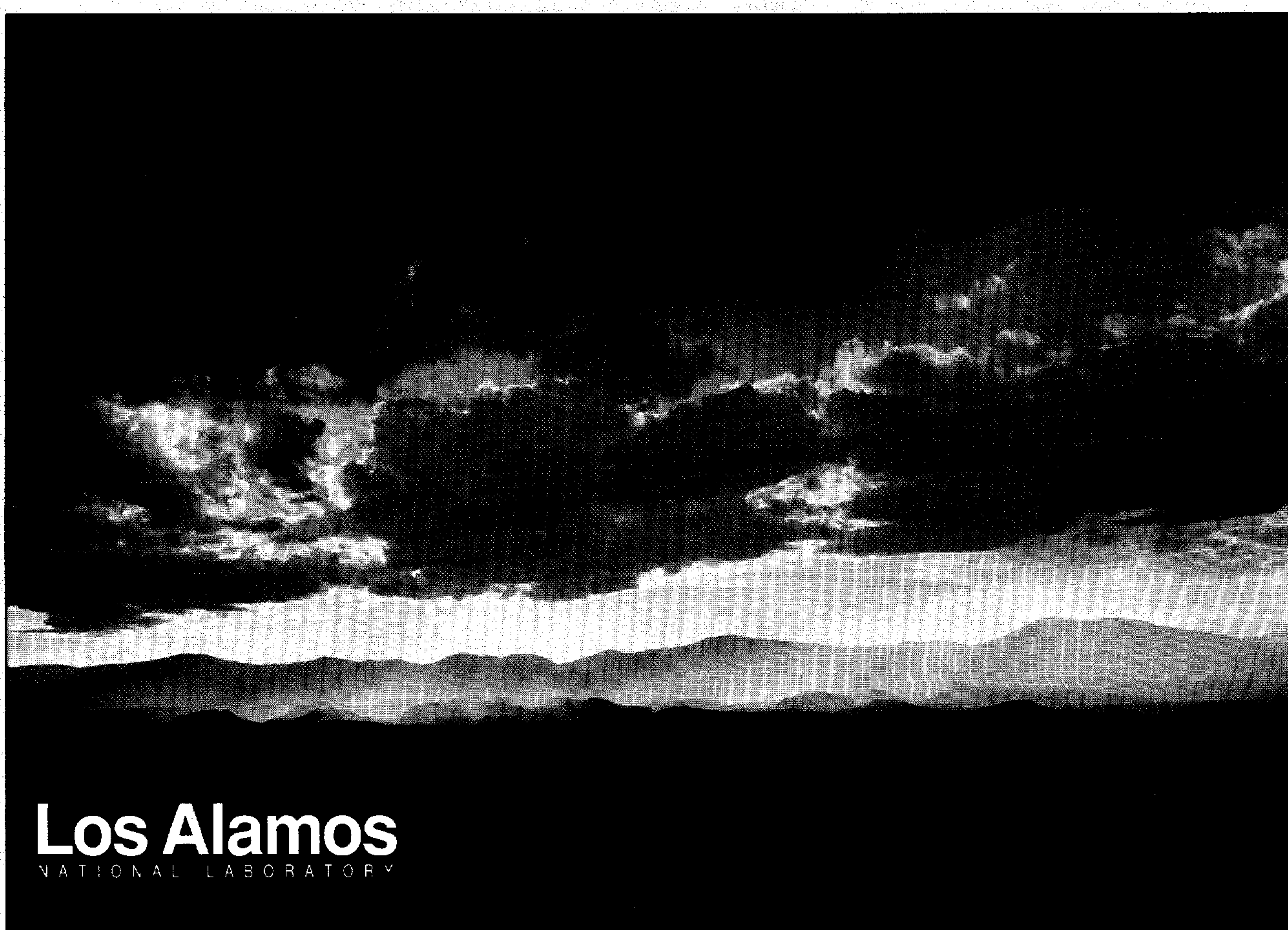

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Design and Performance of a Vacuum-Bottle Solid-State Calorimeter*

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Abstract

EG&G Mound Applied Technologies calorimetry personnel have developed a small, thermos-bottle solid-state calorimeter, which is now undergoing performance testing at Los Alamos National Laboratory. The thermos-bottle solid-state calorimeter is an evaluation prototype for characterizing the heat output of small heat standards and other homogenous heat sources. The current maximum sample size is 3.5 in. long with a diameter of 0.8 in. The overall size of the thermos bottle and thermoelectric cooling device is 9.25 in. high by 3.75 in. diameter and less than 3 lb. Coupling this unit with compact electronics and a laptop computer makes this calorimeter easily hand carried by a single individual. This compactness was achieved by servo controlling the reference temperature below room temperature and replacing the water bath used in conventional calorimeter design with the thermos-bottle insulator. Other design features will also be discussed. The performance of the calorimeter will be presented.

Introduction

The goal of this work is to expand the in-plant measurement techniques for small samples and heat standards using calorimeter measurements. Continuing advances in the precision, stability, accuracy and interchangeability of solid-state temperature measuring devices (i.e., thermocouples, thermopiles and thermistors) has prompted the investigation of these devices as sensors within calorimeters. These solid-state devices can be extremely compact and are readily available at reasonable prices. High sensitivities can be obtained in a rugged design package.

Taking advantage of the above mentioned solid-state temperature advances, a small calorimeter 9.25 in. long by 3.75 in. diameter and weighing less than 3 lb was designed and built by EG&G Mound Applied Technologies using a thermopile[1] and thermistors[2] as the active temperature-sensing devices. This calorimeter accepts samples up to 3.5 in. long and with a diameter of up to 0.8 in. The calorimeter is operated with a reference temperature below room temperature. Constant reference temperature control is obtained via a thermoelectric cooling device and a digital servo control algorithm. The compact design allows the calorimeter to be transported easily by measurement personnel.

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Hardware Design

A schematic of the vacuum-bottle solid-state calorimeter is presented in Fig. 1. The overall size of the calorimeter is 9.25 in. high by 3.75 in. diameter. Shielding from external temperature fluctuations is obtained via a stainless steel vacuum bottle[3] filled with an expanding foam insulating material. Extending 3.5 in. into the center of the vacuum bottle and insulation is a 1-in. diameter aluminum rod with a center bore of 0.8 in. This aluminum rod contains the sample during measurements and is used to direct heat flow towards the solid state sensors. Because of the insulation and vacuum bottle surrounding the aluminum rod, most of the heat flow is directed along the rod towards the solid-state temperature sensors.

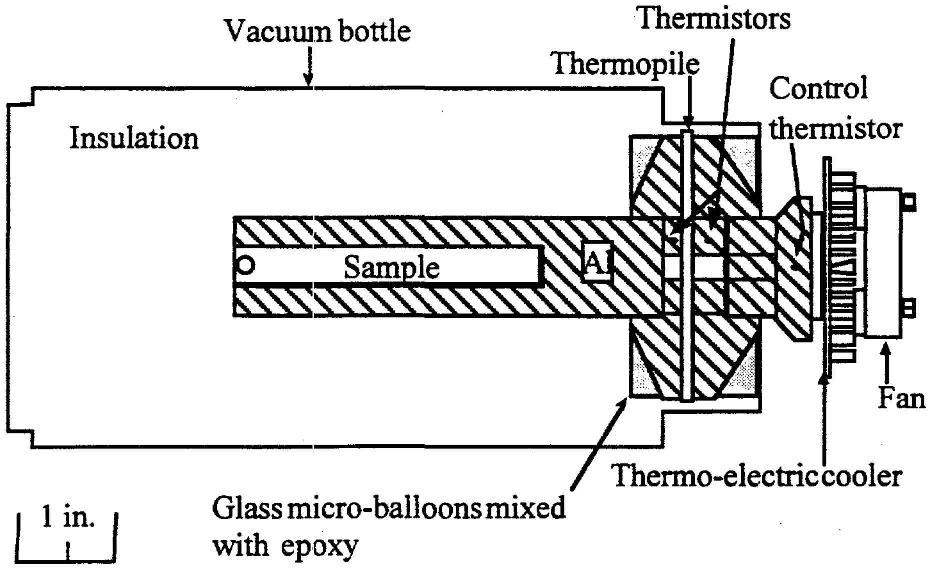

Fig. 1. Crossectional schematic view of solid-state calorimeter.

The smaller diameter region at the top of the vacuum bottle contains two glass encapsulated thermistors separated by a 2-in. by 2-in. square thermopile heat-flux transducer. The thermistor closest to the sample, below the thermopile, is used to measure the sample temperature, T_s, while the thermistor on the opposite side of the thermopile is used to measure the reference temperature, T_r. Therefore, the thermal gap used to define the constant heat flow region between the two thermistors is the thermopile. Each thermistor is permanently bonded within a small hole near the center of an 2.5 in. diameter aluminum disk. Each disk is convex on one side and flat on the other. The thickness at the center of the disk is 0.5 in. A 1-in. diameter hole is bored along the centerline of each disk perpendicular to its diameter starting from the convex side. One hole is 0.75 in. deep and the other is 0.33 in. The bottom of each hole is threaded to accept a threaded rod.

The two convex disks are placed on each side of a thermopile, flat side down, and all three are encapsulated in a two-part epoxy mixed with glass micro-balloons. The final shape is a 1.33 in. thick by 2.8 in. diameter puck. The glass micro-balloons are added to increase the thermal resistance of the epoxy. The epoxy puck fits into the top of the vacuum bottle to reduce the effect of room temperature fluctuations from entering the measurement cell and solid-state sensors. The sensor signals are brought from inside the puck via gold-plated pins protruding perpendicular to the flat surface of the puck. These pins define the top of the puck. The 0.75 in. deep hole is on the top side of the puck.

The aluminum sample rod is threaded into the bottom aluminum disk encapsulated in the epoxy. A short 1-in. long by 1-in. diameter rod which flares at the top to a diameter of 1.5 in. is threaded into the upper disk within the epoxy puck. The flare on the top of the rod is used to increase the surface area contacted to the thermoelectric cooling unit. The thermoelectric cooling unit and the associated cooling fan are placed on top of the aluminum flare with thermally conductive grease. Within the flared aluminum rod is another glass encapsulated thermistor used for feedback control of the thermoelectric cooling device.

Electronics, Data Collection, and Digital Servo Control

Commercially available digital multimeters[4] are used to readout the solid-state temperature sensors. The resistance of the thermistors are measured to 0.1 ohms resolution. The heat flow sensor is measured in volts to 0.1 mV resolution. System performance was not limited by meter resolution.

Data collection and servo control are performed using a personal computer interfaced to a general purpose interface bus (GPIB). The instrument control software used is HP Vee which is commercially available from Hewlett-Packard. This software is an object-oriented programming language with a large instrument driver library and the ability for direct input/output (I/O) of instruments that are not included in the library. The versatility and ease of programming with this software is well suited for application in a research and development laboratory.

A digital proportional-integral (PI) servo control algorithm was developed using HP Vee software. A small, 2-in. by 2-in., thermoelectric cooling device[5] was used to maintain a constant reference temperature of approximately 16.5 °C. The voltage supplied to the thermoelectric unit was controlled by the digital PI servo controller and implemented via the GPIB by a Kepco programmable power supply[6] and a Kepco digital programmer.[7] The optimized control parameters were found to be a proportional band width of 600 ohms and an integration time of approximately 7 sec. Control precision was found to be dependent on sampling rate. Because of the low thermal mass of the system, the response time of the calorimeter is short, thus for good control, a sampling rate of more than one reading per second was needed. The control thermistor sampling rate was two readings per second.

System Performance

In general the system performed acceptably relative to its size and simplicity of design. This was due in part to good feedback control of the control thermistor. The PI digital servo controller maintained thermistor resistance fluctuations to ± 0.35 ohm (one sigma) at a setpoint of 29,000 ohms with room temperature fluctuations of approximately ± 2-3 °C. This 0.35 ohm fluctuation is equivalent to approximately $\pm 0.26 \times 10^{-3}$ °C temperature control. By placing the solid-state calorimeter within the controlled environment of a water bath, controlled to $\pm 0.5 \times 10^{-3}$ °C, the feedback resistance was controlled to the resolution of the multimeter, ± 0.1 ohm. The setpoint control could have been improved by using a higher resolution meter, but nothing would have been gained because of the intrinsic noise of the calorimeter itself.

Plutonium-238 heat standards were measured in the solid-state calorimeter under two basic operating conditions. In one configuration the calorimeter was subject to room temperature variations. In the other configuration the calorimeter was within the controlled environment of a large water bath. Sensitivity and measurement uncertainties for each of the operating conditions are presented in Table I. Heat standards ranging from 0.0007 to 0.43 W were used to determine the calorimeter sensitivities in the two configurations. Sensitivities were based on least squared linear fits to several different measurement parameters versus the heat standard calculated watts. The linear fit was found to be adequate with the small wattage ranges measured. The flexibility of this research calorimeter allowed the determination of the sensitivity by a number of different techniques. Column two of Table I lists sensitivities based on thermopile readings. The thermopile output for each source is equal to the average of baseline voltages directly preceding and following the source run subtracted from the source voltage. The third column in Table I is as listed where T_r and T_s are the reference and sample thermistor, respectively, and "baseline ave" is as listed above the average of the baselines directly preceding and following the source run. The final column in Table I does not use a differential temperature measurement but absolute. The sample side thermistor reading from the source run is reduced by the average baseline thermistor reading.

TABLE I.: Sensitivities for solid-state calorimeter based on least squared linear fits to several different measurement parameters. The sensitivity is determined from plots of ^{238}Pu heat standard watts versus the listed parameter. The listed uncertainties are the one sigma uncertainty of the slope of the linear fit.

	Thermopile (V/W)	$(T_r - T_s)_{sample} - (T_r - T_s)_{baseline\ ave}$ (ohms/W)	$T_{s\ sample} - T_{s\ baseline\ ave}$ (ohms/W)
In room sensitivity	-0.243	2005	-4267
In room uncertainty	0.66%	0.69%	1.1%
In bath sensitivity	-0.249	2104	---
In bath uncertainty	0.31%	0.17%	---

Baseline stability and reproducibility of sample measurements are presented in Table II. Based on the data in Tables I and II it is apparent that the calorimeter performs better in a temperature-controlled environment. This is due to room temperature fluctuations affecting the temperature-sensing elements of the calorimeter. The standard deviation of the average for T_r and T_s, individually, with the calorimeter in the room environment is 6 to 7 ohms while this same number is an order of magnitude smaller while in the water bath environment. The 6 to 7-ohm variation cannot be due to the servo control because the standard deviation on that thermistor in the room is less than half an ohm. The room temperature variations are radiating into the solid-state sensors through the gold-plated signal pins in the epoxy puck and/or through the epoxy puck itself.

TABLE II. Baseline stability and ^{238}Pu heat standard data in ohms for thermistors and milivolts for thermopile. The ^{238}Pu heat source had a heat output of 0.43 W. T_r - T_s for the heat source and baseline runs are the same as column three in Table I.

Measurement Condition	Number of Measurements	Average (ohm or mV)	Standard Deviation	One sigma (%)
Baselines				
In room (T_r - T_s)	22	229	13	5.9
In room (thermopile)	22	-29.2	1.6	5.3
In bath (T_r - T_s)	6	127.0	1.6	1.3
In bath (thermopile)	6	-24.0	0.20	0.82
Heat standard (0.43 W)				
In room (T_r - T_s)	4	858	9.5	1.1
In room (thermopile)	4	-103.9	1.1	1.1
In bath (T_r - T_s)	1	904	--	--
In bath (thermopile)	1	-106.6	--	--

Conclusions

The small vacuum-bottle solid-state calorimeter developed and built by EG&G Mound Applied Technologies when operated in servo controlled mode is capable of measuring a 0.5 W source to 1% (one relative standard deviation) in an uncontrolled environment and to 0.2% in a controlled environment. Improvements in the mechanical design of the calorimeter would lessen the room effects on measurement precision. Two design improvements would be to make a full twin design to significantly reduce temperature fluctuation effects and to shield a portion of the signal leads and the solid-state temperature sensors with material that is under servo control, in effect creating a self-contained controlled environment. Sensitivity can be increased for thermistor readout operation by increasing the resistance of the thermal gap between the thermistors.

Implementation of the design changes could produce a rugged-lightweight small-sample calorimeter capable of calibrating Plutonium-238 heat sources in plant, eliminating the costly shipping of heat sources to a standards calibration facility.

Reference

1. HyCal, Heat flow sensor, El Monte, CA 91713

2. Wavelength Electronics, Glass encapsulated thermistor model TCS620.

3. Campmor, Inc., Thermos-bottle brand vacuum bottle.

4. Hewlett-Packard company, 3478A digital multimeter, Palo Alto, CA 94304.

5. JDR Microdevices, Active CPU cooler model P54C-FN-R, San Jose, CA 95112.

6. Kepco, Programmable power supply model JQE 25-2 (M), Flushing, NY 11352.

7. Kepco, Digital programmer model SN 488-032, Flushing, NY 11352.