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A microcomputer-controlled vacuum adiabatic calorimeter for the temperature range 4·2–300 K

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Abstract. In this paper, we describe the operation of a vacuum adiabatic calorimeter controlled by a Rockwell AIM 65 microcomputer, which is suitable for specific heat measurements in the temperature interval 4·2–300 K. The system measures and calculates the specific heat and the results are printed out on a thermal tape. The cryostat, the electronic circuitry and the software are described. Results obtained using a pulsed heat technique on a specimen of high purity copper are given and compared with values reported in the literature.

Keywords. Calorimetry; specific heats at low temperatures; microcomputer-controlled instrumentation.

1. Introduction

The technique of vacuum adiabatic calorimetry for low temperature specific heat measurements is well established (Hall *et al* 1975, Moses *et al* 1977; Junod 1979). We have made use of a microcomputer for the automatic measurement and print-out of specific heat data, and the details of this system are described here.

2. The cryostat

The entire cryostat assembly and the electronic instrumentation system are shown in figure 1. The specimen whose specific heat is to be determined is suspended inside an adiabatic shield and this assembly is enclosed inside a vacuum can by a stainless steel strip connected to the top flange of this can. An indium O-ring ensures a sufficiently small leak rate so that a high vacuum can be maintained inside the can when it is immersed in liquid helium (LHe). The specimen is normally cooled down overnight by slow conduction through various electrical leads which pass through the top flange as well as by radiation. However, when required, rapid cooling can also be carried out by using helium exchange gas. The details of the sample holder, thermometers, heater assemblies, etc. are shown in figure 2. The specimen is sandwiched between two thin copper plates, one of which carries a constantan and wire heater (82Ω) while the other carries the chromel versus Au-0·07 at% Fe thermocouple which measures the absolute temperture of the specimen. Besides, a

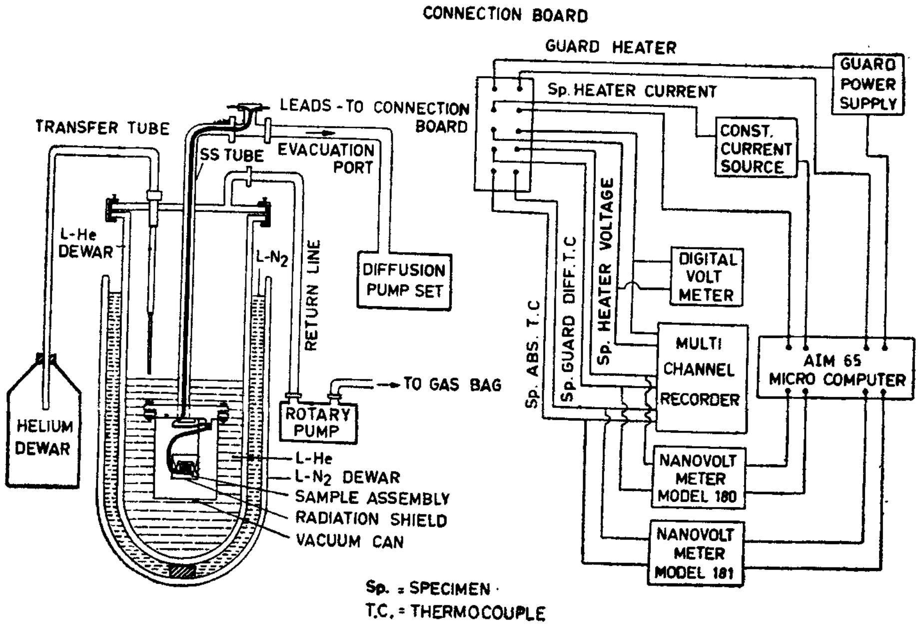

Figure 1. Cryostat and the measurement system.

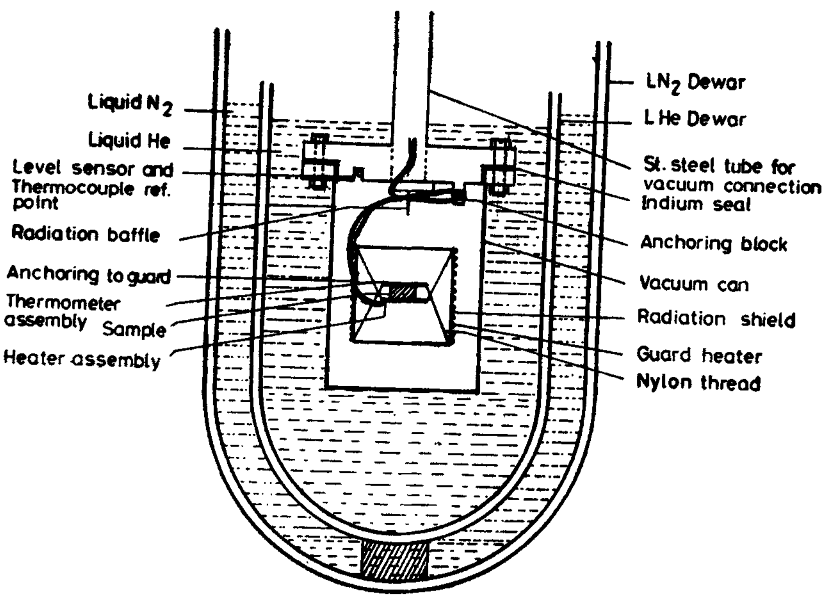

Figure 2. Sample assembly.

differential thermocouple fixed at suitable points on the specimen assembly and the copper adiabatic shield (guard) enables the temperature difference between them to be continuously monitored. The electrical leads are thermally anchored to the adiabatic shield as well as to the top flange of the vacuum can. The guard can be heated by means of a constantan wire heater of 32 Ω wound non-inductively over its surface.

3. Operation

After cooling down the specimen-cum-guard assembly to 4·2 K using liquid helium, the sample space is evacuated. A heat pulse is then given to the specimen by a Keithley model 225 constant current source for a duration of 60 seconds. The resultant temperature rise of the sample is observed while maintaining nearly perfect adiabatic conditions. The microcomputer automates the control and the measurement part of the experiment so as to improve the overall accuracy and speed of measurement and also eliminates the need for taking readings during the measurement. The main functions performed by the microcomputer are:

(i) precise control of the duration of the heat pulse given to the specimen by feeding the heating current to the specimen heater *via* a relay which is controlled by the microcomputer
(ii) control and maintenance of the temperatures of the specimen and the guard at the same value within predetermined narrow limits of variation (0·1 K).
(iii) maintenance of the zero temperature differential between the specimen and guard, when the specimen is heated by a pulse
(iv) determination of the rise in temperature of the specimen due to the heat pulse and
(v) calculation and display of the specific heat value at a given temperature.

4. Details of the microcomputer system

The computer system is shown schematically in figure 3. The Rockwell AIM 65

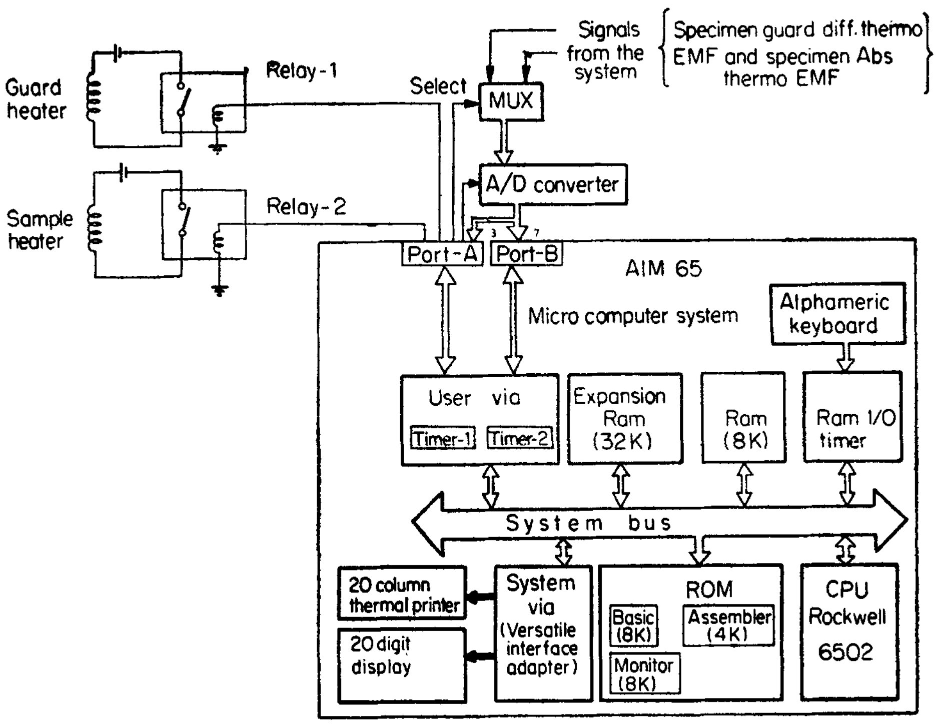

Figure 3. Block diagram of the microcomputer.

microcomputer is an 8-bit microcomputer built around an R 6500 microprocessor. It has an ASCII keyboard, a 20-digit, 16-segment alphanumeric display, a 20-column thermal printer and interfaces for TTY and cassette tape recorder. It includes an R 6522 versatile interface adaptor (VIA) which provides two 8-bit input/output, bit-wise programmable ports, two 16-bit timers and handshake signals for the same. Random access memory (RAM) of 4 K and read only memoiy (ROM) of 20 K containing software for text-editor, assembler, monitor and BASIC are available on board. For the direct print-out of specific heat data, it was necessary to store the thermo e.m.f. *vs* temperature data for the thermocouples over the desired temperature range in the memory. For this purpose, an additional 32 K of RAM was added on. The inputs to the microcomputer system are provided by interfacing an A/D converter sub-system to the microcomputer. Output signals are applied, through port A of the computer, to two relays which control the specimen and guard heaters respectively.

The 10-bit A/D converter has a conversion time of 25 μsec and an accuracy of 1 mV in 1 volt. Of the 10 bits of the A/D converter, the 7 most significant (MS) bits are interfaced to the 7 least significant (LS) bits of port B. The remaining 3 bits of the A/D converter are connected to the 3 LS bits of port A. The other bits of port A are used for A/D start signal, channel selection and for sending the pulses to the relays.

5. Software

The software can be functionally divided into three segments (Figure 4)

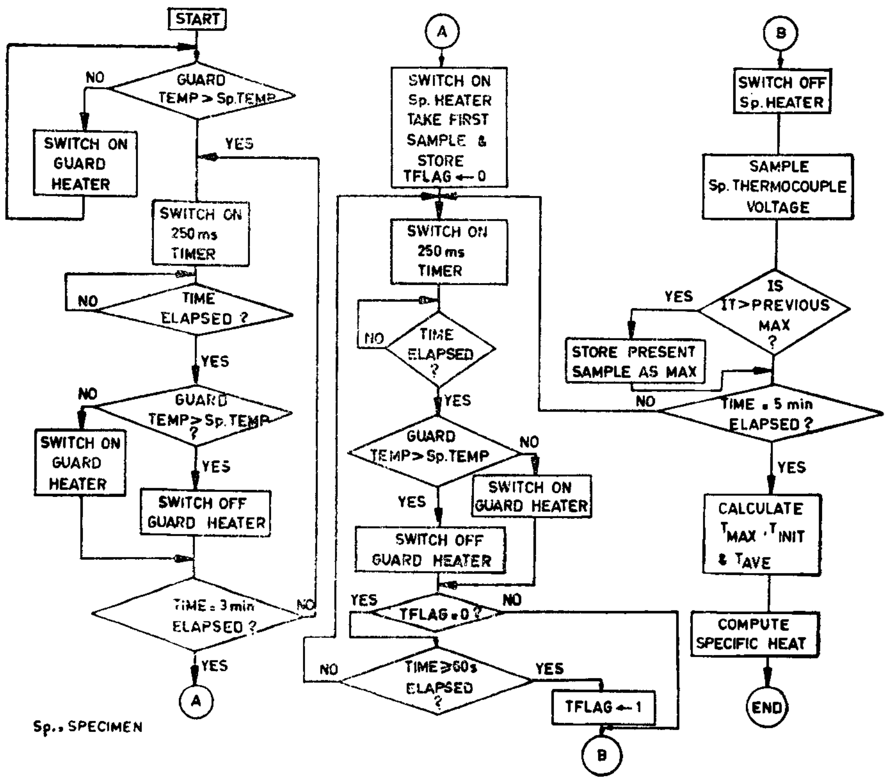

Figure 4. Software for the operation of the microcomputer.

(1) segment 1 for raising the guard temperature to that of the sample;

(2) segment 2 for giving a heat pulse of 60 ($\pm$ 0·01) second duration to the sample; (during this period, zero temperature differential is maintained between the guard and the specimen and samples of specimen temperature are taken and stored at regular intervals;

(3) segment 3 for calculating the specific heat from the measured rise in temperature and the constants of the apparatus, using BASIC.

These three segments are discussed in some detail below.

5.1 *Segment* 1

Initially, due to its proximity to liquid helium, the guard is colder than the specimen. The guard heater is switched on till its temperature equals that of the specimen. Thereafter, this zero temperature differential is maintained by simple on-off control of the guard heater. The on-off time is 250 msec which is obtained by computer-generated interrupts spaced at 50 msec each. A 3-min wait period is allowed to ensure steady-state conditions.

5.2 *Segment* 2

The initial specimen temperature is sampled and stored. Then the specimen heater is switched on and after 60 sec the heater is switched off. However, the sample continues to heat up to a maximum value due to the thermal time constant. This saturation value is finally sampled and stored. Throughout this period, the temperature of the guard is made to track the specimen temperature.

5.3 *Segment* 3

During this phase of off-line computation, BASIC programmes are run to calculate the temperature difference from the initial and final values obtained in sequence and hence the specific heat. Because of the non-linearity of the temperature voltage characteristic of the thermocouple this data is stored in the memory and used to transform thermo e.m.f. values into temperatures.

6. Test results

Typical data obtained, using this system and a high purity specimen of copper, and printed out on a thermal tape after subtraction of the background values of the heat capacity, are shown in figure 5. The results thus obtained over the temperature interval 10-300 K are shown graphically in figure 6 along with the values calculated using a Debye temperature of 310 K for copper. The two sets of data agree over the entire temperature interval within 3%. The value obtained for the Debye temperature from the specific heat data in the temperature interval 4·2-20 K is also in good agreement with the values reported in the literature (Kok and Keesom 1936).

```
RUN
E(1)=268.5 MV
E(2)=281.7 MV
T(1)=20.94 K
T(2)=21.71 K
T(AV)=21.3 K
MS(EMPTYCALORIMETER)
    =0.016 J/K
S(SPECIFIC HEAT)
 =0.1309 CAL/MOLE-K
...................
RUN
E(1)=473.5 MV
E(2)=492.1 MV
T(1)=33.22 K
T(2)=34.34 K
T(AV)=33.8 K
MS(EMPTYCALORIMETER)
    =0.038 J/K
S(SPECIFIC HEAT)
 =0.6494 CAL/MOLE-K
...................
RUN
E(1)=707 MV
E(2)=733.5 MV
T(1)=47.36 K
T(2)=48.96 K
T(AV)=48.2 K
MS(EMPTYCALORIMETER)
    =0.071 J/K
S(SPECIFIC HEAT)
 =1.525 CAL/MOLE-K
...................
RUN
E(1)=868 MV
E(2)=889.5 MV
T(1)=56.94 K
T(2)=58.21 K
T(AV)=57.6 K
MS(EMPTYCALORIMETER)
    =0.104 J/K
S(SPECIFIC HEAT)
 =1.868 CAL/MOLE-K
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Figure 5. Typical data print out on thermal tape at different temperatures.

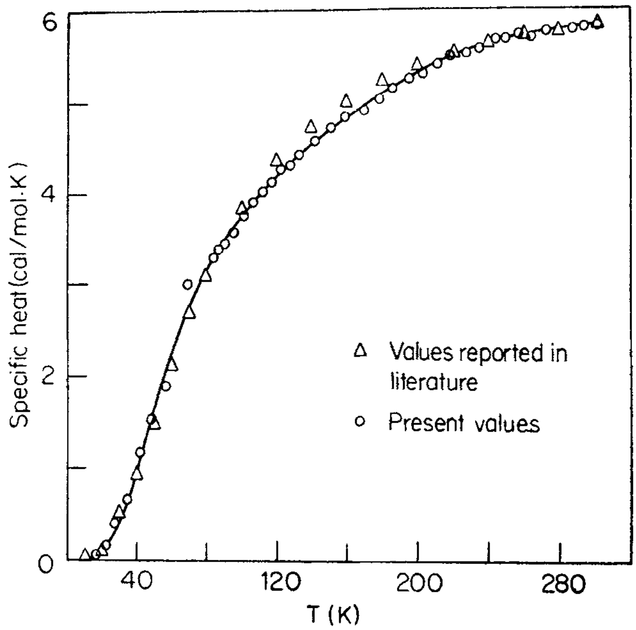

Figure 6. Temperature variation of specific heat for pure copper.

Acknowledgements

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