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A low cost PC based card for heat-capacity measurements at low temperatures

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Abstract. We describe a simple, low cost pc based card for the measurement of heat capacity using adiabatic calorimetry at low temperatures. This card provides the control pulse to the sample heater as well as trigger pulse to the nano-voltmeter which monitors the sensor voltage (Ge sensor, Lake Shore Inc., USA). We have also added a 12 bit DAC on this card and this is used for remote setting of the heater current of an old SHE (now Biomagnetic, Inc., USA, model CCS) analog constant current source. Although this card is used here for heat-capacity measurements, the same can also be used for thermo-power and thermal-conductivity measurements.

Keywords. Low temperature; automation; heat-capacity.

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1. Introduction

Low temperature heat-capacity measurements are always very important to the solid state physicists [1, 2]. Many of the phase transitions (such as, magnetism or superconductivity) can be confirmed only after performing the heat-capacity measurements at low temperatures. Usually, the variation of the heat-capacity of solids with temperatures is very large at low temperatures. Depending on the nature of the samples, the mass of the sample used in the heat-capacity measurements may vary as much as two orders in magnitude. It is therefore essential that the apparatus used for this measurement should allow flexible control of the heating power applied to the sample at low temperatures. One normally uses a programmable current source (Keithley, model 220, USA) which has a GPIB interface and this is used to control the value of the heater current as well as the duration of the heater pulse in the heat-capacity measurements. In this paper, we describe a PC based (compatible with IBM PC) card which can be used along with the analog current source in place of the programmable current source which is generally used for heater current supply. Moreover, the card provides a trigger pulse for every nth ($n = 1$ to 999) second to the nanovoltmeter which monitor the sensor voltage within 10 μsec after the heater is turned off. This helps us to measure accurately the sample temperature as well as the time to determine its thermal relaxation time of the sample which is related to its heat-capacity at that temperature. Further, this circuit provides automatic control for current reversal for temperature sensor current which eliminates the use of another programmable current source.

2. Circuit details

Figure 1 shows how the card and relay drivers are connected in the system. We have used a commercial card (from M/s Dynalog Microsystems Ltd., India) which provides the basic *I/O* facilities like addresses, chip-select lines, read, write and other *I/O* control lines. This is connected to an *I/O* slot of an IBM compatible personal computer and uses the power supply of the host computer. On this card, we have built the necessary interfaces for the microvoltmeter HP, model 34401A, and the constant current sources (M/s Keithley model 225 and M/s Biomagnetic Inc., model CCS). However, one can use

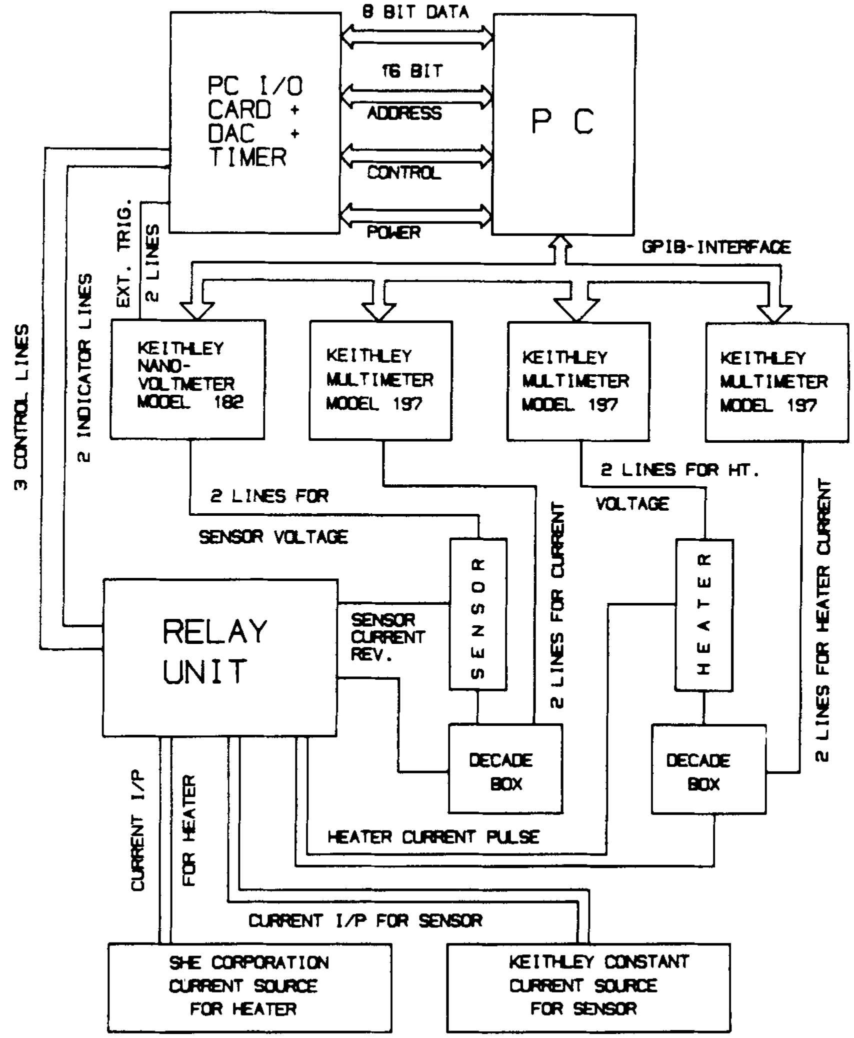

Figure 1. Schematic diagram of the connection of the interface card and relay unit to the host computer, microvoltmeter and the analog dc current source.

any IEEE-488 programmable microvoltmeter and any analog dc current source with voltage programmable facility with this interface card.

The voltage output from a calibrated Germanium sensor (M/s Lake Shore Inc., USA) is connected to the nanovoltmeter (Keithley, model 182, USA) for monitoring the temperature. The card provides a facility to measure the voltage for the forward and reverse current to eliminate the error due to thermo-emfs. The sensor voltage is monitored every two seconds (selectable through software) for a period of 2–3 min (programmable using software). After the sensor temperature is stabilized, a heat pulse is given using the timer and DAC built on the *I/O* card. At the end of the timer pulse to the heater, trigger pulses are generated from the card. The sensor voltage data are sent to the computer every one second by this external trigger pulse to the nanovoltmeter. The data are acquired by the PC using a IEEE-488 interface bus. There is a very short time delay ($< 10\,\mu$sec) between the first trigger pulse and the end of heater current pulse.

Figures 2 and 3 give the details about the timer interfaces of the card along with the DAC for heater current control and also the sensor current reversal. With the help of software (written in Quick Basic, 4.5, Microsoft, USA), a current reversal pulse is sent using the *C* port pin 10 of IC11 [3]. This IC is configured with all ports as output ports. The *A* and *B* ports are used for outputting 12 bit data to the DAC. The *C* port of the IC11 also provides a stop pulse option for switching off the current pulse. The timer gate is open for clock pulses with the start pulse for the heater current source. The clock frequency is 100 kHz. Along with the 0·1 s pulse from pin 16 of IC11, Q1 of IC2 goes high and Q2 of IC2 gives 0·1 s pulse. This opens one of the gates of IC3 and gives 100 kHz clock pulses to first counter in IC1 [3]. This IC1 has three 16 bit counters. The first and second counters are configured as rate generators. These clock pulses are used in generating the trigger pulses for nanovoltmeter and current pulse for the heater. These pulses are given to third counter in IC1 and this is configured as a monoshot. The period of the monoshot is preselectable. This period should have integer values in order to synchronize with trigger pulses and this period is obtained by loading the counter. The output of the monoshot goes low (negative pulse) with the first clock pulse provided the gate pulse is already received. The same gate pulse is used to reset the "divide by 2 counter" (first flip-flop of IC6). The input to IC6 is 1 s (selectable using software) pulses. The output of the IC6 (two second pulses) are prevented during the heater current pulse by the monoshot output of IC1. At the end of this current pulse, the two second pulses come out of one of the gates of IC3, which are given to the first monoshot of IC7. This monoshot gives two second negative pulses ($\bar{Q}1$) for triggering the voltmeter. The monoshot pulse of IC1 is inverted and used to drive the reed relays, which supply the heater current pulse. The relays have 2 NO (normally open) contacts. The relay RL1 is active with the monoshot pulse of IC1 and this gives current to the heater. The relay RL2 keeps the heater current source short circuited when IC1 monoshot pulse is absent. For sensor current reversal, we have another set of relay RL3 and RL4 as shown in figure 3. The driving pulses to all the relays are fed via two opto-isolators (MCT2E) and this ensures complete isolation for these pulses.

The heater current amplitude is controllable using an external dc voltage in an old SHE's (model CCS) constant current source. We have modified this current source so that the output current can be programmed using an external dc voltage. The unit requires 1 V dc for the full amplitude in any current range. However, one can use any

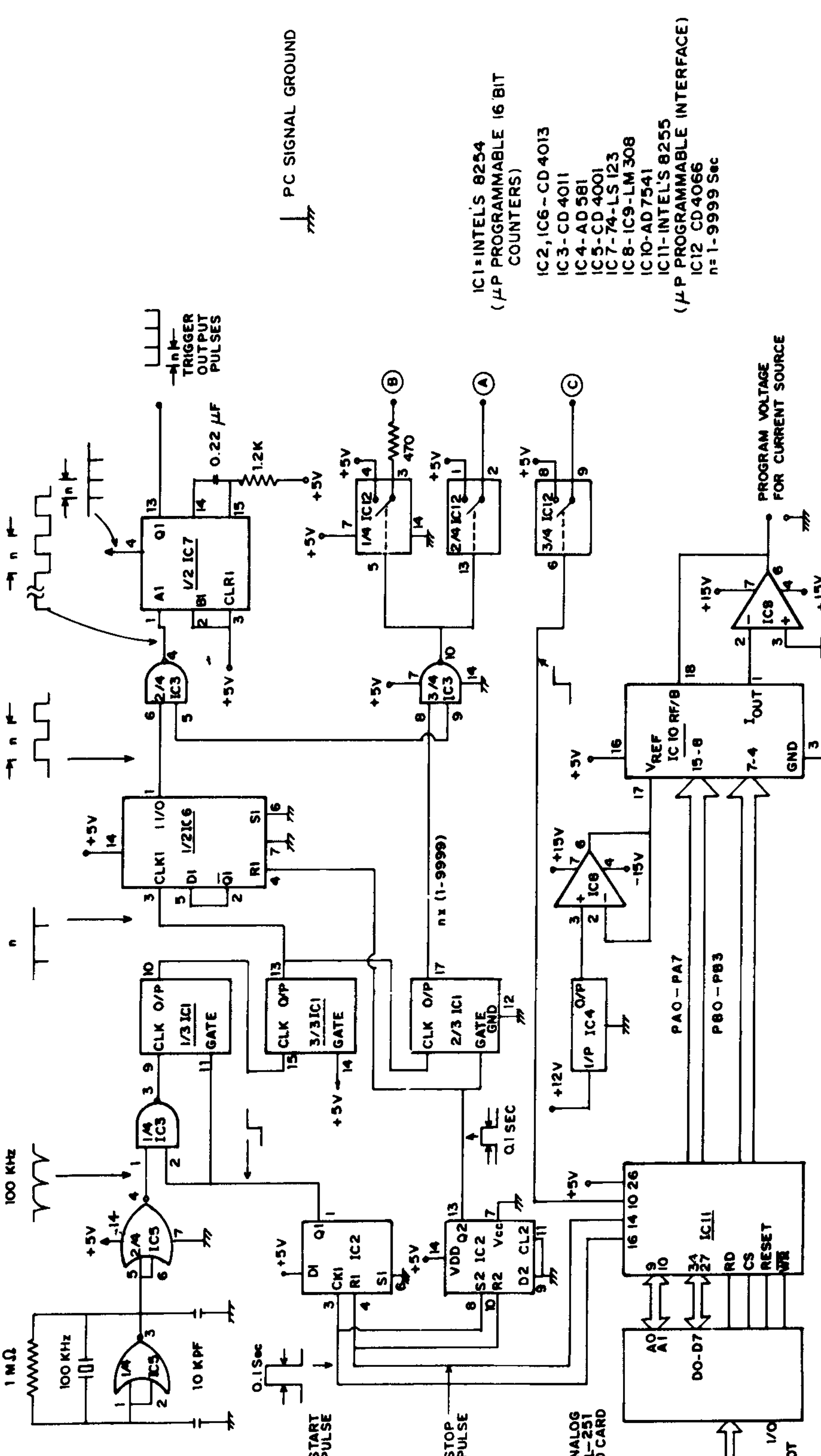

Figure 2. Circuit diagram of the interface card. The pulses are derived from 100 kHz quartz clock.

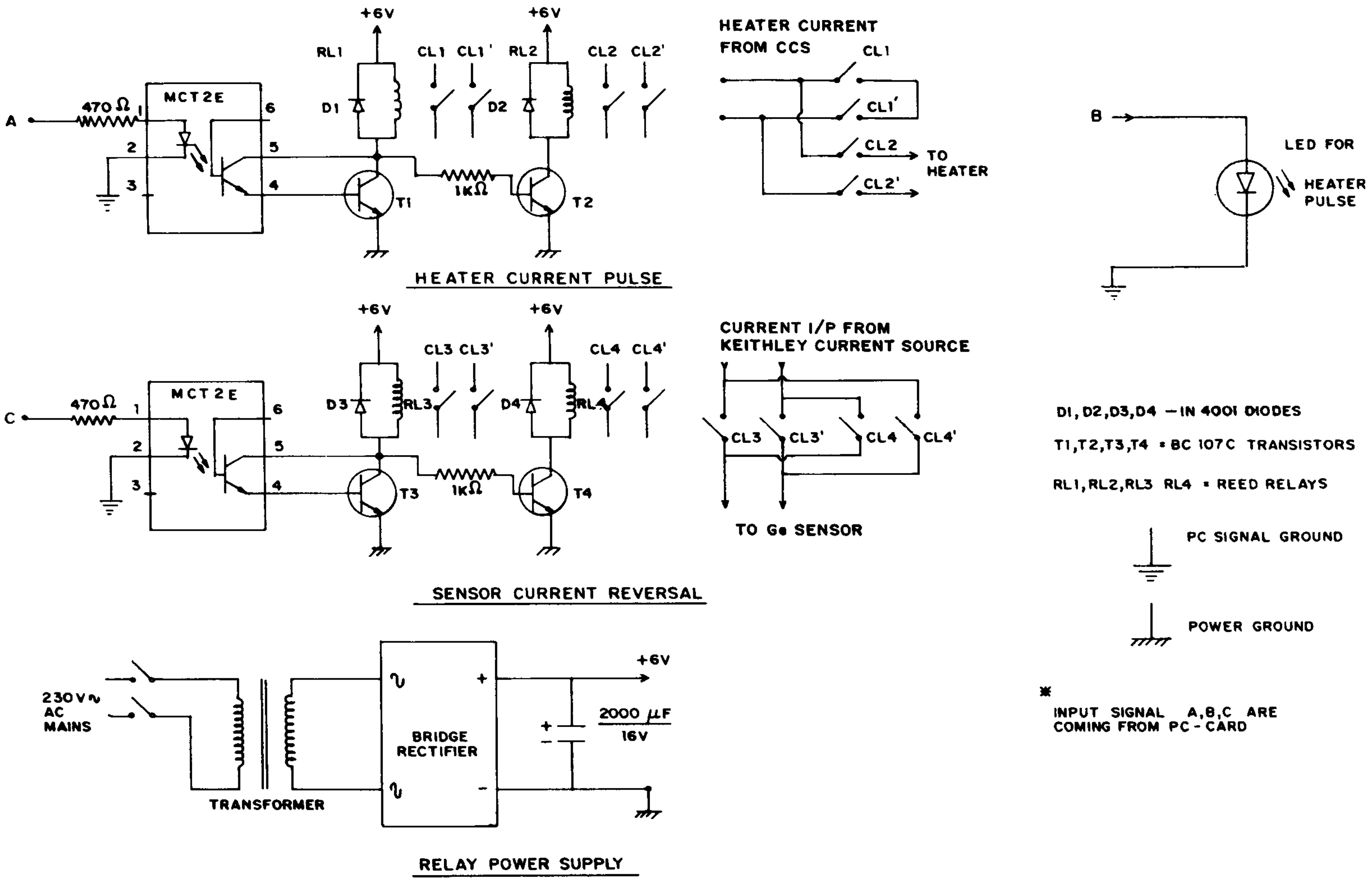

Figure 3. The circuit diagram of the relay unit.

analog voltage programmable current source with this card. The DAC IC is AD 7541 from Analog Devices, USA [4]. The 12 bit binary O/P from A and B ports of IC11 is connected to this DAC. The external reference to this DAC is derived by using an AD 581 (Analog Devices) and a buffer which gives a 10 V reference. The DAC output is connected to an operational amplifier LM308 [5] which gives a voltage output. The feedback path is completed by the resistors inside the DAC. This output is used for heater current control.

3. Heat capacity measurements

Heat-capacity measurements are performed in a standard glass cryostat which consists of outer liquid N_2 dewar and an inner dewar where liquid He is transferred. The typical capacity of N_2 dewar is 3 litres and that of He is 1·3 litres. The hold time for He dewar is 16 hours which is enough to perform heat-capacity measurements from 1·5 K to 70 K. We have used adiabatic heat pulse technique to measure the heat-capacity of materials in the above mentioned temperature range. In this technique, the base temperature line is sampled via measuring the output from the nanovoltmeter (sensor voltage) every one second for 3 minutes and if the drift rate is less than 5 μK/sec, a heat pulse is applied to the sample. After a preselected time duration (software selectable) of this heater pulse at a given temperature range, the nanovoltmeter is triggered every second to measure the time vs temperature data for at least two minutes (software selectable) [7]. All the triggering operations, current settings (heater and sensor currents) and current reversal (for sensor current) are preformed by the pc card. The heat-capacity is calculated using the expression,

$$C_p\left(\frac{\mathrm{d}T_S}{\mathrm{d}t}\right)=P-k(T_S-T_0) \tag{1}$$

where P is the heating power of the heat pulse, T_S is the sample temperature and T_0 denotes the sample temperature in the absence of external heating and is determined by extrapolating the pre-heating drift curve as shown in figure 4. It can be shown that for the post-heating period, $U(t)=T_S-T_0$ is given by,

$$U(t)=\left(\frac{P}{C_p\beta}\right)[1-e^{-\beta t_h}]e^{-\beta(t-t_h)} \tag{2}$$

where t_h denotes the heating time and $\beta=\tau^{-1}$ and the heating starts at $t=0$. The relaxation time is defined as $\tau=C/\kappa$ where C is the heat-capacity and κ is the thermal conductance of the heat leak. Figure 4 shows T vs t measurement cycle of the heat pulse method, when τ is comparable to t_h. The pre- and post-heating drift curves are measured during a time t_m. The post-heating drift curve is registered after a thermal delay time t_d following the heating interval t_h in order to allow for internal temperature equilibrium of the sample and the sample holder assembly. The post-heating temperature drift curve is fitted to eq. (2). At low temperatures ($T<20$ K), a linear version of eq. (2) is used (retaining only the first order terms) and at high temperatures, the full non-linear expression is used. The fitted expression is then extrapolated to the middle of the heating period, $t=t_h/2$ to obtain $U(t_h/2)$. The heat-capacity itself is calculated

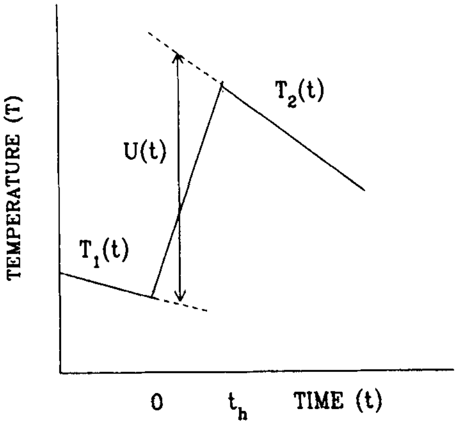

Figure 4. A schematic plot of temperature (T) vs time (t) in the pre- and post-heating interval of the heat-capacity measurements.

iteratively from the expression,

$$U(t_h/2)=\frac{2P}{C\beta}\sinh\left(\frac{1}{2}\beta t_h\right)=\frac{Pt_h}{C}\left[1+\frac{1}{24}(\beta t_h)^2\right]. \tag{3}$$

The third term is used for the evaluation of C at low temperatures. A mixed language [6] (BASIC-FORTRAN-BASIC) has been used for controlling the instruments (via IEEE-488 interface) and real time evaluation of heat-capacity data.

4. Results

The pc card is used in a standard adiabatic calorimeter to measure the heat-capacity of single crystal copper. The temperature dependence of the heat-capacity (C_p) is shown in figure 5. The heat-capacity from 1·5 K to 10 K is fitted with the expression,

$$C_p=\gamma T+\beta T^3. \tag{4}$$

The inset shows the C_p/T vs T^2 plot at low temperatures. Our values (C_p and γ) agree with the literature values [7]. We have also studied the heat capacity of a superconductor $Y_5Os_4Ge_{10}$ and the data are shown in figure 6. The inset shows the superconducting transition at 8·9 K [8,9] in this compound. The heat-capacity of this sample is given by,

$$C_p=\gamma T+\beta T^3+\delta T^5 \tag{5}$$

where the first term is due to electronic contribution, the second and third terms are lattice and anharmonic contribution to the heat capacity from 10 K to 30 K. The values of γ, β and δ for this sample and for single crystal copper are given in table 1.

This card can also be used in the measurement of thermo-power of metals and alloys. The onboard DAC and timer can be used for giving heater pulse to the auxiliary heater in standard thermo-power setup and the voltmeters can be triggered at the end of this

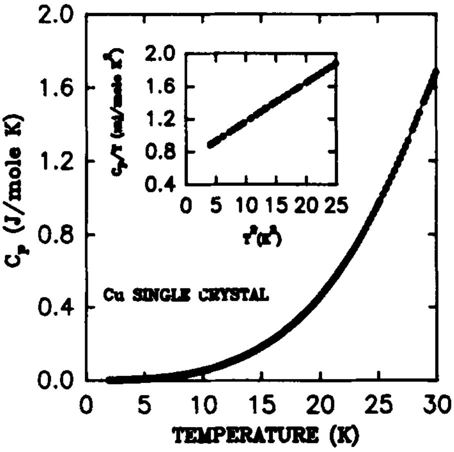

Figure 5. Temperature dependence of the heat-capacity of single crystal copper from 1·5 K to 30 K. The inset shows C_p/T vs T^2 at low temperatures. The solid line is a fit to the eq. (4) (see text).

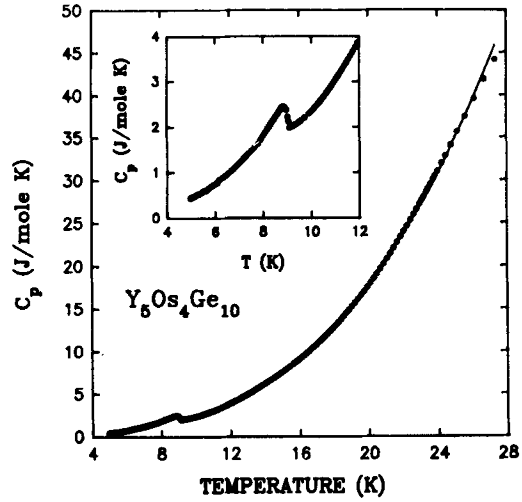

Figure 6. Temperature dependence of the heat-capacity of $Y_5Os_4Ge_{10}$ from 4·5 K to 25 K. The inset shows C_p vs T at low temperatures. The solid line is a fit to the eq. (5) (see text).

heat pulse. The time dependence of the voltage (dV/dt) and the time dependence of temperature (dT/dt) can be computed and the ratio (dV/dT) yields the thermo-power. In the case of thermal-conductivity measurements, one gives known value of the heat pulse (selectable using software) to the auxiliary heater using this card and by noting the

Table 1. Heat-capacity data of single crystal copper and poly-crystalline $Y_5Os_4Ge_{10}$.

Sample	Range	γ	β	δ
	K	mJ/mole K^2	mJ/mole K^4	mJ/mole K^6
Copper	1·5–10	0·7	0·0476	—
$Y_5Os_4Ge_{10}$	10–30	24·4	2·1	0·00013

temperature difference across the sample. Then one can calculate the thermal-conductivity of the specimen.

5. Conclusion

We have described a simple substitute for a programmable/timer current source using PC based card in conjunction with an analog dc current source. The card eliminates the use of two such programmable current sources in the heat-capacity experiments. The simple design with accurate timer and very low cost (< 200 US Dollars) are the main features of this interface card. Further, this card can also be used in other measurements like thermo-power which require both timer and programmable current sources.

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