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## SHORT COMMUNICATION

# Arguments for making use of a differential gas thermometer in cryogenic microcalorimetry

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## Abstract

This communication invites attention to a simple differential gas thermometer at liquid helium temperatures for detecting the heat power released at either end of some heat resistor. In conjunction with the most sensitive models of commercially available differential pressure sensors, this type of cryogenic calorimeter promises sub-nanowatt sensitivity with a time constant of heat relaxation of the order of 60 s.

For a number of calorimetric problems, sensitivity to small amounts of energy is enhanced if the system's temperature can be lowered to cryogenic levels, where both heat capacity and thermodynamic noise are small. The subject of this communication is calorimetric problems, where the heat power resolution must be as high as possible; the measurand can remain constant for a relatively long period, but measurements of moderate accuracy are adequate. A cryogenic microcalorimeter [1] illustrates such a measurement situation. The very quantity to be measured (the nuclear heating due to tritium decay) renders a measurement uncertainty of less than 0.3% unreasonable. The experimental set-up embodies, however, a dual-compensated method. There are two temperature-controlled stages in this case. These operate at constant temperatures separated from each other by about 0.1 K. The stages are connected through weak thermal links, with a constant heat flux from one stage to the other. The temperature of each stage is monitored with Ge-resistor thermometers and controlled through a power-feedback loop with a resistance heater. The measurand is the feedback power needed for compensating any heat power released as a result of tritium  $\beta$ -decay from the tritium-contaminated sample. The measurement resolution is limited by fluctuations of the electric feedback power, which depend on the total noise of all input circuits. The total noise consists mainly of the temperature sensors' noise, the Johnson noise of the bridges' resistors and the preamplifiers' noise.

We suggest an alternative set-up that was in general use in the middle of the last century to measure heat conductivity at cryogenic temperatures [2]. That set-up involved a simple differential gas thermometer to measure the temperature drop along the sample, provided the heat flux through the sample is known. Such a thermometer consists of two reservoirs filled with <sup>4</sup>He gas and connected by capillary tubes to either input of a differential pressure meter. With this arrangement any difference in gas temperatures of the reservoirs causes a pressure difference to be measured by the meter. To provide the only way for the heat flux a high vacuum thermal isolation and isothermal radiation screens are commonly used. A similar set-up can apparently be used for detecting the heat flux through the heat resistor of known conductance.

In this case one of the gas reservoirs is thermally well connected with a sample, in which some amount of energy is released as heat. Along with the sample and its holder, this reservoir forms a system with overall heat capacity  $C$  (the sample assembly). The other reservoir is thermally connected to an isothermal heat sink (the base block). The heat then flows between the sample assembly and the base block through the thermal resistor, resistance  $R_h$ , producing a temperature difference proportional to the heat flux. At present, for the pressure measurement, it is natural to take advantage of commercially available differential pressure sensors. For example, the most sensitive model of differential pressure

sensors (LP9000 series) of Druck Limited has a full scale range of 10 Pa. It has a guaranteed  $\pm 0.1\%$  full scale accuracy. This value combines non-linearity, hysteresis and repeatability. So, one can deal with devices of resolution  $\delta p = 0.01$  Pa or even better.

For an estimation of the calorimeter sensitivity, consider the single arm of the gas thermometer where the pressure meter is held at room temperature  $T_r$ . Neglecting the small amount of gas inside the capillary, the gas column of the arm is divided into two volumes: the working volume  $V_t$  having a gas temperature  $T_1$  and the dead volume  $V_d$  of the pressure sensor. We assume the  $^4\text{He}$  gas to be ideal<sup>3</sup>. The equation of state of the gas inside the column can then be written as:

$$p \left( \frac{V_d}{T_r} + \frac{V_t}{T_1} \right) = Rn, \quad (1)$$

where  $p$  is the gas pressure,  $R$  is the molar gas constant and  $n$  is the total amount of substance. The small change  $\Delta T_1$  of the gas temperature in  $V_t$  causes a change of pressure  $\Delta p = (\partial p / \partial T_1) \Delta T_1$  with  $\partial p / \partial T_1$  derived from (1) given by  $\partial p / \partial T_1 = (p / T_1) (1 + n_r / n_t)^{-1}$ , where  $n_r$  and  $n_t$  are the amounts of substance in  $V_d$  and in  $V_t$  correspondingly, so that  $n_r + n_t = n$ .

If  $\Delta T_1$  results from a heat power  $P$  produced in the sample,  $\Delta T_1 = P R_h = P \tau / C$ , where  $\tau = R_h C$  is the time constant of heat relaxation of the sample assembly. For  $\Delta p$ , one can then write

$$\Delta p = \left( \frac{p}{T_1} \right) \left( 1 + \frac{n_r}{n_t} \right)^{-1} \left( \frac{\tau}{C} \right) P. \quad (2)$$

We introduce the relative values  $v = V_t / V_d$  and  $c = C / (C_p n_t)$ , where  $C_p = 2.5R$  is the molar heat capacity of a monatomic ideal gas at constant pressure  $p$ . Taking  $C = c C_p n_t = 2.5 c p V_t / T_1$  one can derive from (2) the heat power resolution  $\delta P$  as

$$\delta P = (\delta p V_d) 2.5 \tau^{-1} c \left( v + \frac{T_1}{T_r} \right). \quad (3)$$

The factor in the first parentheses of (3) is related to the pressure sensor and can be defined as a sensor energy resolution, whereas the other quantities can be optimized by the calorimeter design to minimize  $\delta P$ .

For the above pressure sensor the dead volume  $V_d$  is equal to 7 ml [3], and its energy resolution is 70 nJ. It is reasonable to take  $v$  to be of the order of  $T_1 / T_r$ , which for a 4.2 K working temperature is equal to 0.014. Also, to make  $c$  as close to unity as possible one should choose low mass samples and calorimeter construction materials of low specific heat capacities. This is feasible for a variety of calorimetric problems, particularly by using single crystal sapphire for the sample holder and walls of the thermometer reservoir<sup>4</sup>. Where this is the case, and  $\tau$  is chosen to be, say, 60 s the estimate for  $\delta P$  from (3) is about 0.1 nW.

Such an ultimate sensitivity is the main argument for applying gas thermometry. This limit could probably be achieved in practice with a thorough calorimeter design. Note

<sup>3</sup> The first virial coefficient,  $B(T)$ , for  $^4\text{He}$  gas at 4.2 K is  $B \cong -3 \times 10^{-3}$ .

<sup>4</sup> Compare specific heat capacities for (1)  $^4\text{He}$  gas at  $p = 5 \times 10^4$  Pa and  $T = 4.2$  K equal to  $30 \text{ mJ cm}^{-3} \text{ K}^{-1}$ ; (2) Nb at 4.2 K equal to  $2.5 \text{ mJ cm}^{-3} \text{ K}^{-1}$ ; (3) Cu at 4.2 K equal to  $0.75 \text{ mJ cm}^{-3} \text{ K}^{-1}$  and (4) sapphire at 4.2 K equal to  $0.02 \text{ mJ cm}^{-3} \text{ K}^{-1}$ .

that for the cryogenic calorimeter having a dual-compensated structure with resistive thermometers of  $470 \Omega \text{ K}^{-1}$  sensitivity,  $\tau = 200$  s and  $R_h = 2 \times 10^4 \text{ K W}^{-1}$ , the optimal estimate gives a 1 nW measurement uncertainty [4].

We also mention the proportionality between pressure and heat flux so that the latter is readily characterized by the differential pressure sensor output. This neglects a small non-linearity caused by the influence of radiation and some respiration volume of the pressure sensor.

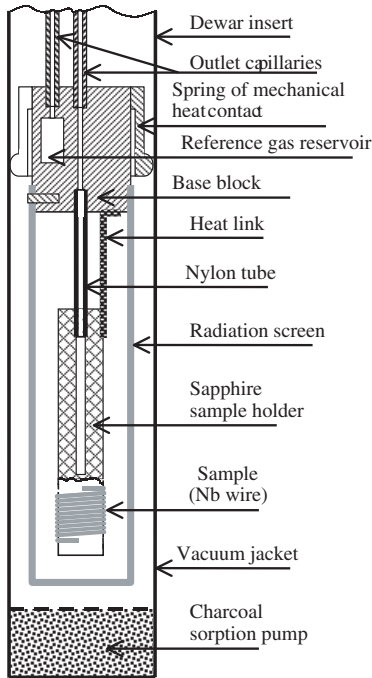
Using the above values of  $\tau$  and  $v$ , further estimates can be derived. The working volume of the gas thermometer is  $V_t = V_d T_1 / T_r = 0.1$  ml. Assume for simplicity that the system is filled with  $^4\text{He}$  gas at room temperature and atmospheric pressure  $p_a$ . Then, the resulting pressure in the gas thermometer reservoir cooled to 4.2 K is determined by [2]

$$p = p_a \frac{(V_t + V_d) T_1}{(V_t T_r + V_d T_1)} = 0.5 p_a. \quad (4)$$

The heat capacity of this amount of  $^4\text{He}$  gas at constant pressure is  $C_p n_t = 2.5 R n_t = 2.5 p V_t / T_1 = 2.5 \times 0.5 \times 10^5 \text{ Pa} \times 10^{-7} \text{ m}^3 / 4.2 \text{ K} \approx 3 \text{ mJ K}^{-1}$ . The heat resistance  $R_h$  of the weak heat link between the sample assembly and the base block can then be evaluated as  $R_h = \tau / (C_p n_t) = 60 \text{ s} / (3 \text{ mJ K}^{-1}) \approx 2 \times 10^4 \text{ K W}^{-1}$ . The full scale of the above pressure sensor (10 Pa) corresponds to the 100 nW heat flux through  $R_h$  consistent with a maximum temperature drop of 2 mK. So the maximum heat flux radiated by every square centimetre of the sample assembly to the isothermal screen held at the base block temperature can be estimated from the Stefan–Boltzmann law to be  $\sim 3 \text{ pW cm}^{-2}$ .

For the base block to have a temperature close to that of the surrounding liquid helium bath, the heat link between them should have a heat resistance  $r \ll R_h$ —a value of  $r = 200 \text{ K W}^{-1}$  suffices. Heat conductivity of this order can be provided by a mechanical contact between the base block and a surface held at the ambient temperature of the liquid helium bath [2]. Such an approach allows the cryogenic calorimeter probe to be simplified. Importantly, high vacuum sealing at cryogenic temperatures can be avoided. For top loading into a liquid helium storage Dewar, the probe must be designed to have the conventional oblong shape with the lower end dipped into the helium bath. A vacuum jacket over the calorimeter can then be implemented in the form of a long tube, blind at its lower end and having a conventional o-ring vacuum flange at the upper end. A sketch of the possible design of the lower end of the calorimeter probe is shown in figure 1. This calorimeter design is appropriate for measuring hysteresis losses in superconductor samples under re-magnetization. As seen, the base heat block has a spring (assumed to be of rather good thermal conductivity) providing a ring-shaped heat contact with the smooth cylindrical inner surface of the probe vacuum jacket. Before air evacuation, as the sample and the radiation screen are fixed, the calorimeter assembly is inserted into its working position through an upper hole in the vacuum jacket by means of a rod (not shown in the figure). A proper lubricant can be used to enhance the heat transfer and reduce friction.

The equilibrium temperature of a liquid helium bath of a Dewar freely vented to the atmosphere typically drifts over a range of 50 mK because of atmospheric pressure variations.



**Figure 1.** Sketch of a possible design of the lower end of the microcalorimeter cryo-probe that is to be dipped into the liquid helium bath.

Though the temperature dependence of relevant heat quantities can be ignored for this variation of temperature, the drift is 25 times the maximum value of the temperature drop across  $R_h$ . So, variations of ambient pressure in time of the order of  $\tau$  and shorter would be felt by the measurement system. Therefore, stabilization of the helium bath temperature by the usual means of outlet vapour pressure stabilization is suggested.

The capillary connectors to the differential pressure meter, especially those parts subject to room temperature, have to be thermally balanced. A gas thermometer is sensitive to temperature changes in any part of a common volume. It can be inferred from the above two-volume model that  $\Delta p = (\partial p / \partial T_x) \Delta T_x = (p / T_x) (n_x / n) \Delta T_x$ , where  $T_x$  and  $n_x$  characterize, respectively, the absolute temperature and amount of substance in any part  $V_x$  of the common gas volume in a single arm of the gas thermometer. The predominant parts of the common volumes, the dead volumes  $V_d$ , are

fairly well balanced. This is characterized in part by the thermal coefficient of the above pressure sensor (less than  $10^{-4}$  of the full scale per degree Celsius). As to the capillary parts subject to the room temperature  $T_r$ , taking into account the above condition of filling the gas thermometer, one can estimate  $n \simeq (p_a V_d) / (RT_r)$ ,  $n_x = (0.5 p_a V_x) / (RT_r)$ , and  $\Delta p \simeq (0.25 p_a / T_r) (V_x / V_d) \Delta T_r = 86 \text{ Pa K}^{-1} (V_x \Delta T_r / V_d)$ . With the sensitivity of the differential pressure meter better than 0.01 Pa, one needs to have the value in parentheses to be of the order of  $10^{-4}$  K. This appears feasible for a dead volume  $V_d$  of the pressure sensor that is not too small. Decreasing the working gas pressure in the thermometer could further reduce this requirement, because  $\delta P$  according to (3) does not depend directly on this pressure.

In conclusion, for the problems of cryogenic calorimetry, as sensitivity is a matter of more importance than accuracy, we have described a simple differential gas thermometer based on a modern differential pressure sensor. The approach promises sub-nanowatt sensitivity with only a few minutes measurement time and may prove to be a good alternative to resistive thermometers that are in common use. The power detected is proportional to the pressure difference, so that the output scale of the pressure meter can be used directly. The well-known immunity of a gas thermometer to magnetic fields makes this approach especially good for applications where magnetic fields are inherently present.

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## References

- [1] Richardson J M 2000 *IEEE Trans. Nucl. Sci.* **47** 854–9
- [2] White G K 1959 *Experimental Techniques in Low-Temperature Physics* (Oxford: Clarendon Press)
- [3] Druck Limited, Fir Tree Lane, Groby, Leicester LE6 0FN, UK private communication
- [4] Nummila K K, Riski K, Kajastie H and Manninen J 2002 *CPEM 2002 Digest 02CH37279C (Ottawa, Canada, 16–21 June 2002)* (New York: IEEE) pp 510–11