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T.A. Alvesalo, T. Haavasoja, P.C. Main, L.M. Rehn, K.M. Saloheimo. A magnetically diluted CMN thermometer. Journal de Physique Lettres, 1978, 39 (23), pp.459-460. 10.1051/jphyslet:019780039023045900 . jpa-00231544

HAL Id: jpa-00231544

<https://hal.science/jpa-00231544>

Submitted on 1 Jan 1978

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Classification
 Physics Abstracts
 07.20D — 07.55

A MAGNETICALLY DILUTED CMN THERMOMETER (*)

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(Reçu le 23 août 1978, accepté le 6 octobre 1978)

Résumé. — Un thermomètre à poudre de CMN dilué ($\text{Ce}_{0.03}\text{La}_{0.97}$) et un thermomètre à poudre de platine destinés à mesurer la chaleur spécifique de l'hélium-3 superfluide ont été réalisés. Leur comparaison suggère que la température magnétique T^* du CMN dilué est reliée à la température absolue T par la relation $T = T^* + \Delta$ où $\Delta = -0.16 \pm 0.01$ mK.

Abstract. — A powdered $\text{Ce}_{0.03}\text{La}_{0.97}$ magnesium nitrate thermometer has been developed to facilitate specific heat measurements in liquid ^3He . Comparison with a platinum NMR thermometer suggests that the magnetic temperature T^* is related to the absolute temperature T by $T = T^* + \Delta$, where $\Delta = -0.16 \pm 0.01$ mK.

Recent work on ^3He at very low temperatures has emphasized the need for a reliable thermometer in the millikelvin region. Of particular urgency is an accurate measurement of the specific heat of liquid ^3He in both its normal and superfluid phases. With this end in mind, we have developed a thermometer based on the magnetic susceptibility of cerous magnesium nitrate (CMN) diluted to 3 % molar solution in the corresponding lanthanum salt.

Figure 1 shows the experimental cell designed to measure the heat capacity of ^3He . The silver chamber and support are thermally connected to the inner bundle of a double-bundle nuclear refrigerator [1] via a tin heat switch.

The salt was prepared by first separately crystallizing the double nitrates $\text{La}_2\text{Mg}_3(\text{NO}_3)_{12} \cdot 24 \text{H}_2\text{O}$ and $\text{Ce}_2\text{Mg}_3(\text{NO}_3)_{12} \cdot 24 \text{H}_2\text{O}$: the metal content of the salts was checked by complexometric analysis. Then the salts were weighed out in the stoichiometric ratio and dissolved in the minimal amount of distilled water. The solution was evaporated at room temperature and the crystals dried with diethyl ether. Analysis yielded that the ratio of La : Ce ions was correct to within 1 %. The crystals were then ground with a pestle and mortar and passed through a fine sieve to give a maximum particle size of $37 \mu\text{m}$, and packed to about 70 % filling factor in the shape of a cylinder with diameter equal to its height of 5 mm. This pill was inserted into a thin epoxy coil form onto which was wound, in series

FIG. 1. — Schematic drawing of the experimental cell.

opposition, two single-layer 54 turn coils of 0.08 mm diameter niobium wire 8 mm apart. The coils formed one part of a superconducting flux transformer, the other part coupled to an R.F. biased thin-film SQUID operating in a flux locked loop [2]. A single layer 56 turn coil of Niomax-CN 0.05 mm filamentary superconducting wire was wound directly over each pick-up coil and this formed one arm of a low frequency (33 Hz) impedance bridge [3] with an excitation field of a few milligauss. The other external arm of the bridge was a coil inductively coupled to the

(*) This paper has been presented at the LT 15 Conference as a post-deadline paper.

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SQUID which then acted as a very sensitive null detector.

The whole pick-up coil assembly fitted inside a 9 mm inner diameter niobium shield which was sited inside the liquid ^3He in a cylindrical appendage to the main cell (see Fig. 1). Nine 1.2 mm holes were drilled in the shield to provide a thermal link to the helium in the main body of the cell; an adjacent 4 mm hole was drilled in the coil form. The thermal time constant after a heat pulse was typically around ten seconds at 1 mK and ten minutes at 10 mK. No attempt was made to externally compensate the earth's magnetic field and the variation with temperature of the d.c. component through the flux transformer would indicate a trapped field of order 1 G.

For calibration and comparison we placed a standard platinum NMR thermometer in a hole cut into the sinter about 35 mm from the magnetic salt. Despite the proximity of the niobium shield and indium seals, the homogeneity of the applied field did not seem badly affected. We observed an effective platinum spin-spin relaxation time τ_2^* of around 800 μs , which compares well with previous experiments on the same batch of platinum powder [1]; the applied field was 27 mT.

For pure CMN above its ordering temperature of 1.8 mK a useful empirical expression for the susceptibility, or equivalently the magnetometer output S_m , is

$$S_m = \frac{C}{T - \Delta} + S_{\text{ind}} \quad (1)$$

where S_{ind} is the temperature independent part, T the absolute temperature, C a constant and Δ a constant of order a few millikelvin. Assuming that this is true for the diluted salt, and that the integrated signal from the pulsed platinum thermometer, S_{Pt} , is inversely proportional to temperature, *viz.*

$$S_{\text{Pt}} = \frac{K}{T}$$

with K a constant, then plotting $(S_m - S_{\text{ind}})^{-1}$ against $1/S_{\text{Pt}}$ should yield a straight line of slope K/C and intercept $-\Delta/C$. Such a plot is shown in figure 2 confirming eq. (1) as a suitable description. If we now use the Korringa relationship between the spin-lattice relaxation time of the platinum, τ_1 , and the absolute temperature, $\tau_1 T = 29.9 \text{ msK}$ [1], then we can evaluate K and C separately and hence calibrate the thermometer. Such an analysis yields

$$\Delta = -0.16 \pm 0.01 \text{ mK}.$$

FIG. 2. — The inverse temperature-dependent magnetometer signal, $(S_m - S_{\text{ind}})^{-1}$ in arbitrary units, plotted against the inverse integrated platinum NMR signal, $(S_{\text{Pt}})^{-1}$. The units of $(S_{\text{Pt}})^{-1}$ are mK referred to a calibration using the Korringa relationship. The measurements were made at the vapour pressure of ^3He .

A negative value of Δ would affect the proposed phase diagram of Paulson *et al.* [4], who used a similar thermometer and assumed $\Delta = +0.10 \text{ mK}$, and might even remove the discrepancy between their temperatures and the Helsinki scale [5]. However, there is no reason why Δ should be the same for different samples and in our case at least trapped flux may be causing the salt to behave anomalously. The experiment should be repeated with compensation of the earth's field.

Generally the diluted CMN thermometer was far more precise than the platinum susceptibility device, which has a maximum resolution worse than one part in 10^3 compared with an observed 5 parts in 10^5 for the former. In fact the precision in this case was limited by noise and since we observed no heating effects associated with increasing the bridge excitation the ultimate precision will probably be determined by our impedance bridge, about 1 part in 10^5 . The two thermometers agreed with each other reasonably well from run to run, but since the platinum signal is particularly sensitive to stray fields, direct comparison is not possible without a comprehensive τ_1 calibration for each run.

Unfortunately, due to an unidentified and fluctuating heat leak, we were not able to extend the comparison below 1 mK.

Acknowledgments. — We are grateful to Ms P. Riikonen for her help in preparing the diluted salt, and to M. Manninen and Y. Takano for assistance at various times.

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