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Author
Fogle, W.E.

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EXPERIMENTS WITH POWDERED CMN THERMOMETERS BETWEEN 10 mK AND 4K, AND A COMPARISON WITH AN NBS SRM 768 FIXED POINT DEVICE

W. E. Fogle, E. W. Hornung, M. C. Mayberry and Norman E. Phillips

Department of Chemistry, University of California, Berkeley California 94720
and Materials and Molecular Research Division
Lawrence Berkeley Laboratory, Berkeley, California 94720

Comparison of a powdered CMN thermometer with an NBS fixed point device demonstrates an internal inconsistency in the T_c's assigned to the fixed point device. T_c's between 100 and 200 mK are in excellent agreement with a temperature scale interpolated between He vapor pressure temperatures and nuclear orientation temperatures, but there is a discrepancy of 8% at the 15 mK point. Evidence for different susceptibility-temperature relations for superficially similar CMN thermometers is also presented.

Temperatures in the mK region are usually derived from He vapor pressure temperatures by extrapolation of the susceptibility, $\chi = c(T-\Delta)^{-1}$, of CMN, or by absolute measurements with, e.g., nuclear orientation (NO) or noise thermometers. The former are generally more precise, but the value of Δ must be derived independently because the high-temperature fits are not sensitive to changes in Δ of the order of 1 mK. Recent improvements in the accuracy and precision of noise and NO thermometers[1] have been combined with the use of the T_c's of superconductors to produce the NBS SRM 768 fixed point device.[2] This device makes possible a comparison of a laboratory temperature scale with absolute measurements at NBS at five temepratures between 15 and 200 mK, and shows promise of becoming a key element in the intercomparison of different temperature scales. For example, Lhota et al.[3] have compared the Helsinki Pt-nmr temperature scale with two of the T_c's and interpreted the results as support for ^{3}He specific heat measurements on that scale.

We report the results of calibrations of several CMN thermometers with He vapor pressures and a ^{60}Co NO thermometer that show that the value of Δ can be significantly different for powdered CMN thermometers of similar shape and particle size. Comparison with an SRM 768 device shows an internal inconsistency in the T_c's and that, while the three highest T_c's are in excellent agreement with an extrapolation of T_{76}[4] from above 1K, the lowest T_c, at approximately 15 mK, is in error by about 8%.

Four different powdered CMN thermometers were used. In A and B the CMN was mixed with silicone oil and packed around Ag wires in an expoxy holder. χ was measured with a low-frequency ac bridge using a SQUID detector. Silicone oil was also used as a thermal contacting agent in C, but the CMN-oil mixture was contained in an Au-plated Cu holder and χ was measured by a dc SQUID technique. For D, ^{3}He was used to provide thermal contact to a sintered Cu heat exchanger and Cu container. The CMN and the coils were shielded from the Cu by a Nb shell, and the ac bridge was used to measure χ. In every case the CMN was material that passed through a #400 NBS sieve (37μ) and the sample was in the shape of a right circular cylinder with height equal to diameter; background signals were measured and used to correct the data as necessary.

Thermometer A was calibrated against He vapor pressures betwen 1.5 and 3.5 K taking, initially, $\Delta = 0$. The calibration was corrected to T_{76}, extrapolated to lower temperatures, and used to calibrate a Ge thermometer to 70 mK. That Ge thermometer was in turn compared with the other CMN thermometers and B and C were also directly intercompared. The precision of these comparisons was highest for B and C in the 0.2 to 0.8 K region. In that region they fit a Curie law ($\Delta = 0$) with maximum deviations of 0.012 and 0.011%, and rms deviations of 0.005 and 0.004%, respectively. These results do not show that $\Delta = 0$, but only that it is the same, to within about 0.1 mK, for the three thermometers.

Thermometer C was also compared with the NO thermometer at temperatures between 16 and 20 mK. The NO thermometer gave temperatures that were higher by about 6% with a precision of about 1%. On this basis the temperature scale was adjusted by refitting to the He vapor pressure temperatures, constraining the fit to reproduce the NO temperature, and allowing Δ to be determined by the fit. The result was $\Delta = 0.95$ mK, with essentially no effect on the quality of the high-temperature fit. The resulting temperature scale, which was transferred to the GE thermometer, is the one used throughout the following discussion.

Thermometer D was calibrated against the Ge thermometer and compared with the fixed point device on two separate runs. For both runs the calibration of the CMN thermometer gave $\Delta = 0$ with an uncertainty of about 0.1 mK. Extrapolation of the calibrations to lower temperatures gave agreement with the NO thermometer to within about 2.5% at 8 mK and 2% at 20 mK which is satisfactory in view of the somewhat lower precision of both measurements in this case.

We see no obvious reasons for the difference in Δ's for the two types of CMN thermometer. A, B, and C were pumped for only a few minutes at room temperature and then cooled rapidly; D was pumped for about an hour at room temperature, but through a 0.04" ID×12" capillary tube. Furthermore, no evidence of aging from run to run was noticed in repeated experiments. The differences are, however, similar to some published results: Greywall[5] has reported Δ=0.7 mK for a CMN-stopcock grease thermometer, and a value of essentially zero, for temperatures above 5 mK, has been reported[6] for CMN in ^{3}He. It is evident that these differences must be better understood before CMN can be used to make a reliable comparison of T_{76} with mK temperature scales, particularly if Δ is not determined by an accurate low-temperature calibration point.

The results of the two comparisons with the fixed point device are shown in Table I. In run 1, the recommended measuring currents were used. Under these conditions the Be transition was not observed at all. At 20% of the recommended current, the Be transition was not observed on cooling to 16 mK, but the broad transition reported in Table I was observed on warming after interrupting the bridge current. On run 2 the effect of varying the measuring current was investigated; the transition reported in Table I was observed at measuring currents of 5 to 10% of the recommended values, and appears to represent the zero-current limit. The W transition was surprisingly sharp in our work, but in this case the transition consists of a very steep center region with long, ill-defined tails. The difference between our result and the NBS result may be simply a matter of uncertainty in estimating the 80%-complete limits of transition.

The NBS values for the $AuIn_2$, $AuAl_2$, and Ir T_c's are in excellent agreement with those reported here. The Be T_c obtained in run 2 is also in very satisfactory agreement with the NBS value, but its width and problems with supercooling make it less suitable for a fixed point device. The disagreement betweeen our W T_c and the NBS value is about 8% and indicative of a serious problem. Greywall[5] has reported a similar discrepancy with the NBS T_c. Other similar comparisons give various results, but some are in approximate agreement with our observations.[7]

The internal inconsistency of the NBS T_c's can be demonstrated to some degree independently of our CMN temperature scale by fitting our CMN bridge readings to their T_c's. With $\Delta = 0$ and the three highest T_c's fitted to the bridge readings, the rms deviation is 0.11% but the W T_c is 6.6% high; if all five T_c's are fit, the rms deviation is 1.5% with the Ir T_c 1.7% low and the W T_c 2.4% high. With Δ determined by the fit, the rms deviation is 0.5% but $\Delta = -1.3$ mK, a value that seems unreasonable[6] for a right-circular cylinder of CMN and ^{3}He.

We conclude that the NBS SRM 768 can play an important role in interlaboratory temperature scale comparisons, but that Be is unsuited for use in such a device and should be replaced if another better material can be found. Furthermore, the W T_c is in serious error. Its value should be redetermined, and in the meantime temperature scales based on it must be regarded with suspicion.

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REFERENCES

[1] Soulen, R. J., Marshak, H., to be published, Metrologia.
[2] Soulen, R. J., Dove, R.B., NBS Special Publication 260-62 (1979). (USGPO, Washington, D.C. Stock No. 002-002-02047-8).
[3] Lhota, E., Manninen, M.T., Pekola, J.P., Soinne, A.T., Soulen, R.J., to be published in Phys. Rev. Lett. and to be presented at this conference.
[4] Metrologia 15, 57 (1979).
[5] Greywall, D.S., Busch, P.A., preprint.
[6] Webb, R.A., Giffard, R.P., Wheatley, J.C., Journal of Low Temperature Physics, 13, 383 (1973).
[7] Soulen, R.J., private communication, and to be presented at this conference.

Table I. Comparison of T_c's determined by CMN thermometry with those assigned at the NBS for SRM 768 #44. All temperatures are in mK.

	$AuIn_2$		$AuAl_2$		Ir		Be		W	
	T_c	Width	T_c	Width	T_c	Width	T_c	Width	T_c	Width
Run 1	205.13	0.4	162.47	0.1	99.19	0.8	23.76	1.8	16.32	0.01
Run 2	205.16	0.4	162.49	0.1	99.26	0.8	22.67	0.4	16.33	0.01
NBS values	205.57	0.4	162.57	0.3	98.88	0.8	22.96	0.2	15.15	0.7