
# NBS TECHNICAL NOTE 830

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

# NBS Cryogenic Thermometry and the Proposed Cryogenic Extension of the IPTS

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This article outlines a comprehensive and long-term program being carried out by NBS scientists of the Cryogenic Physics and Temperature Sections. The goals of the program are the extension of IPTS 68 below 13.81 K and the development of devices which make practical realizations of that scale convenient and reliable. We propose to contribute to international adoption of a thermodynamically accurate scale below 13.81 K by analyzing the results of three thermometers: the NBS Acoustical Thermometer (already in operation for several years), noise thermometry using the Josephson effect (recently developed), and $\gamma$-ray anisotropy thermometry (recently studied in detail at NBS). Such a temperature scale will most likely be disseminated by the use of certain superconductors as thermometric reference points. Practical interpolation devices will be based on the principles of nuclear and electronic paramagnetism, nuclear quadrupole resonance, and nuclear magnetic resonance. Details of operation, measurement schemes and experimental progress made to date are included in nine appendices.

*Key words:* Acoustical thermometry; $\gamma$-ray anisotropy thermometry; noise thermometry; nuclear magnetic resonance; nuclear quadrupole resonance; paramagnetism; superconductivity; temperature.

## A. Introduction

With the increasing world-wide use of cryogenic technology in transportation, in energy generation, storage and transmission, and in scientific research, there is a growing need for international agreement on the temperature scale below 13.8 K. Although the IPTS-68 recommends the vapor pressure-temperature relations of liquid $^3$He and $^4$He for use in thermometry between 0.2 K and 5.2 K, the difficulty involved in obtaining vapor pressure measurements which are sufficiently precise to define temperature in that range to within $\pm$ 1 mK, as well as the advent of the $^3$He-$^4$He dilution refrigerator (wherein vapor pressure thermometry is not at all convenient), have led us to re-examine the basis for a low temperature extension of the scale. In concert with the national laboratories of other countries, the U.S. National Bureau of Standards has been carrying out a comprehensive experimental program directed toward an adequate definition of the temperature scale from 0.001 K to 30 K and the practical realization of that scale.

The aim of this paper is to summarize the work being done at NBS, to note those problems which are still unsolved and which thus remain as an impediment to the extension of the IPTS, and finally to suggest possible forms which this extension might take.

## B. Thermodynamic Temperatures

In thermometry, great importance is attached to the evaluation of the thermodynamic temperatures of physical phenomena. It should be a prime responsibility of the Advisory Committee to obtain and to promulgate a reasonable estimate of the uncertainty of the IPTS with respect to thermodynamic temperatures, irrespective of whether the IPTS itself can be changed to reduce a known error at any given time.

Experiments under way at NBS can be expected to play a significant role in evaluating the uncertainty with which the low temperature scale reflects thermodynamic temperatures.

The acoustic interferometer experiment of H.H. Plumb and G. Cataland is well known to the thermometric community. It is the source of the NBS Provisional Temperature Scale 2-20 (1965), which has been

disseminated world-wide by the calibration of germanium resistance thermometers. A subsequent discussion in Appendix 1 will describe changes made in the NBS apparatus for the purpose of evaluating possible systematic errors, and the results of recent measurements.

The possibility of realizing the thermodynamic temperature scale by means of noise thermometry has existed since 1927 when Nyquist derived his familiar equation. Prior to development of Josephson junctions, the difficulties encountered in measuring the very small noise voltages developed acros the source resistor frustrated all attempts to establish a low temperature scale based upon this principle. The high sensitivity and low intrinsic noise of Josephson junction devices, however, have for the first time provided a solution to these measurement problems. The noise thermometer program at NBS is expected to provide a thermodynamic temperature scale from 0.010 K to 20 K. Details of this work are discussed in Appendix 2.

A nuclear $\gamma$-ray anisotropy thermometer also has the potential of yielding thermodynamic temperatures. When all the parameters of the nuclear spin system and the decay properties are known, a measurement of the $\gamma$-ray anisotropy will yield the thermodynamic temperatures. The disadvantage of a $\gamma$-ray thermometer is that usually it is sensitive over a limited temperature region. Although its use is thereby restricted, it can be used to test more widely ranging thermometers, e.g. the noise thermometer mentioned above. In Appendix 3 we describe our recent work comparing temperatures obtained with a single-crystal $^{60}Co$ $\gamma$-ray anisotropy thermometer with the noise thermometer from 0.02 K to 0.04 K.

## C. Scale Definition

The IPTS-68 is defined from the triple point of hydrogen to the gold point by assigned values for the temperatures of several fixed points and the properties of standard interpolating instruments. The continued use of this rationale below 13.81 K has much to recommend it; the vapor pressure-temperature relation of liquid helium covers only a limited portion of the cryogenic range and, as we have already pointed out, and the method itself no longer has general utility in cryo-thermometry.

It appears that physical phenomena exist which will provide conveniently spaced and readily realizable fixed points over the entire cryogenic temperature range. The most promising scheme, in our view, is based on superconductive transition temperatures; a device employing elemental superconductors has been developed at NBS, and it offers the possibility of five thermometric fixed points between 0.5 K and 7.2 K. The progress of our program in superconductive fixed point research will be discussed in Appendix 4.

Magnetic phase transitions can be considered as possible alternate candidates for fixed points. We have considered the prospects of utilizing cooperative transitions in ferromagnets, antiferromagnets, and "Jahn-Teller salts" as thermometric fixed points. Experiments to date by B.W. Mangum, however, have indicated that a temperature uncertainty of several millikelvins might ensue from magnetic susceptibility measurements of such salts, due primarily to the fact that the susceptibility change at the transition does not occur over a sufficiently small temperature range.

The status of a standard interpolating device appears much less satisfactory at this time. The cryogenic sensor most commonly used from 0.01 K to 20 K is the doped-germanium resistance thermometer. This thermometer is capable of millikelvin precision, is compact, and can be read easily with standard electronic bridges. However, several defects offset these advantages. For one, the resistance-temperature relation is not analytic and thus the user must calibrate the germanium resistor versus some other thermometer. Moreover, many calibration points and a complicated polynomial fitting technique are required in order to use the resistor for accurate thermometry. To cover the full range from 0.01 K to 20 K, a two step process is often used: a paramagnetic salt is first calibrated versus a vapor pressure scale or by fixed points, and then the resistor is calibrated versus the salt. Another problem stemming from the non-linear dependence of the resistance on temperature is that the user must generally employ two or three separate resistors to cover the full range. Finally, the resistors suffer occasional

and unpredictable shifts in calibration and this necessitates rechecking, recalibration or possible replacement.

In contrast to the situation with cryoecious fixed points, there is no ready solution to the problem of inadequate standard interpolating instruments. We believe that a number of possible sensors merit investigation, however. One of these is the Rh-0.5%Fe resistance thermometer developed at NPL. Its chief advantage lies in the fact that the R-T calibration may need fewer calibration points than the germanium thermometer, so that only a few fixed points may be adequate to provide accurate interpolation at the 1 mK level. Since this thermometer has only recently been developed, more data as to stability, reproducibility and the functional R-T dependence will be needed before its usefulness can be judged.

Paramagnetism in several materials has been used quite effectively in interpolative thermometry. The salt cerous magnesium nitrate, (CMN), obeys the Curie Law to 1 mK precision from 2 K down to 0.05 K and, in powder form, down to 0.002 K. Thermal contact, however, becomes increasingly more difficult below 0.05 K. For this reason, below 0.1 K, pulsed nuclear magnetic resonance techniques precise to 0.3% are being used by D.B. Utton in this laboratory to examine copper and platinum as Curie-law thermometers. The progress of such experiments will be noted in Appendix 9.

Other possible schemes which appear promising for interpolation from 2 K to 30 K are based on the paramagnetic susceptibilities of the salts neodymium ethylsulfate, chromic methylammonium alum and gadolinium metaphosphate; on nuclear magnetic resonance frequencies of ferromagnetic and antiferromagnetic salts below their transition temperatures; and on nuclear quadrupole resonance frequencies of "Jahn-Teller salts." Each of these topics will be discussed briefly in Appendices 5-9.

## Appendix 1
## NBS Acoustical Thermometry
H. H. Plumb and G. Cataland

### A. Introduction

This report is principally concerned with the determination of the thermodynamic values of temperature for some low temperature fixed points using the NBS acoustical thermometer.

In the first reports [1, 2][1] on the acoustical thermometer the results were preliminary but did indicate that the instrument merited additional effort and was at least competitive with other methods (gas thermometry and P-V, isotherms) for determining values of temperature on the thermodynamic temperature scale. As an example, the value of the boiling point of equilibrium hydrogen was reported to be 20.265 K, with isotherm data reproducibility of $\pm$ 0.007 K. That experiment was not conducted under closely controlled conditions but did indicate that acoustical thermometry presented the possibility of more accurate determinations than was evidenced by the results reported by Van Itterbeek [3]. He reported comparisons of temperature values between hydrogen vapor pressure scales and acoustical thermometry that differed between 0.120 K and 0.170 K.

Our research which yielded 20.265 K for the hydrogen boiling point was termed "preliminary" because the experimental conditions were not closely controlled. The low temperature components of the acoustical thermometer were not vacuum-shielded or instrumented to the extent of more recent experiments. Rather, the components were located in a liquid hydrogen bath that was contained in a three-inch-neck, commercial, metal liquid hydrogen storage dewar. Although the vapor pressure was closely controlled, large temperature gradients can exist in such a bath. Furthermore, germanium thermometers were not yet available for use, and the vapor pressure was read from a less than precise mercury U-tube column. No special efforts were taken to insure that a complete conversion to equilibrium hydrogen had occurred.

[1] Figures in brackets indicate the literature references found at the end of each independent appendix in this paper.

The resulting isotherm, and value of temperature that was derived from it, were preliminary albeit encouraging. Comparable measurement conditions existed for the reported [1, 2] difference between acoustically determined values of temperature and those associated with the liquid $^4$He vapor pressures below the lambda point.

After the aforementioned measurements, the results of more sophisticated measurements, a description of the apparatus and the NBS Provisional Temperature Scale 2-20 (1965) were reported in *Metrologia* [4]. It is apparent that systematic deviations occur for some of the isotherms and that the linear representation was extended into pressure ranges that were too high to be so characterized. At the time of the experimentation it was not expected that millidegree accuracies were being achieved -- the principal need was to achieve for the United States' scientific and industrial community a reasonably accurate temperature scale that covered the range 4 to 14 kelvins. Five millikelvin uncertainties were then being reported for low temperature gas thermometry endeavours and discrepancies which greatly exceeded 5 millikelvins were observed when gas thermometer scales were intercompared. Additional information and remarks will made later in the paper concerning current results on the determination of the e-$H_2$ normal boiling point.

B. Design Changes and Operational Procedures

Further work indicated that more attention must be paid to the possible existence of thermal gradients in the acoustical thermometer components and to the attainment of thermal equilibrium. These problems appear to be dependent upon the amount of electrical power that must be applied to heaters in the apparatus and one must allow sufficient time to elapse to achieve thermal equilibrium. It is highly probable that these factors unduly affected at least some of the isotherms that were determined, e.g., the 20.051 K isotherm of the NBS 2-20 (1965) Scale, the reported 20.284 K isotherm for the e-$H_2$ NBP and probably the 21 K isotherm (reported respectively in *Metrologia*, [4] paper #27 of CCT [5] and at the 5th Temperature Symposium [6]). In certain circumstances in which "proper" procedures were followed it has been possible to maintain the acoustical thermometer at temperatures and have all of the sensing germanium resistors indicate temperatures that agreed within about 0.2 mK. These conditions were achieved with the instrument that employed a fused quartz rod and a micrometer head for measuring piston displacements. Early in 1972 an additional radiation shield, another heater coil and additional germanium sensors were installed as is illustrated in Figure 1. Thus the thermometer can be operated with two sensing resistors and two controlled heaters as well as six resistors that provide for monitoring not only the temperature of the gas-space chamber wall but also the temperature of the outer radiation shield.

Recently the acoustical thermometer has been redesigned to eliminate the possible error that may be caused by employing the extended fused quartz rod to measure the displacement of the reflecting piston. A laser interferometer and fringe counter have been incorporated in the design. While the values of temperature that are derived from employing this new design do not appear to differ substantially from those of the fused quartz apparatus, thermal equilibrium problems that require resolution have arisen. The substitution of an evacuated stainless steel tube for the fused quartz rod appears to have altered the temperature control and attainment of thermal equilibrium for the apparatus. It is believed that the severity of the problem is probably related to the difference between the temperature of the apparatus above that of the helium bath.

C. Fixed Point Temperature Values

1. Normal Boiling Point of e-$H_2$

In the 1967 submission to CCT [5] and at the 5th Temperature Symposium [6] it was indicated that

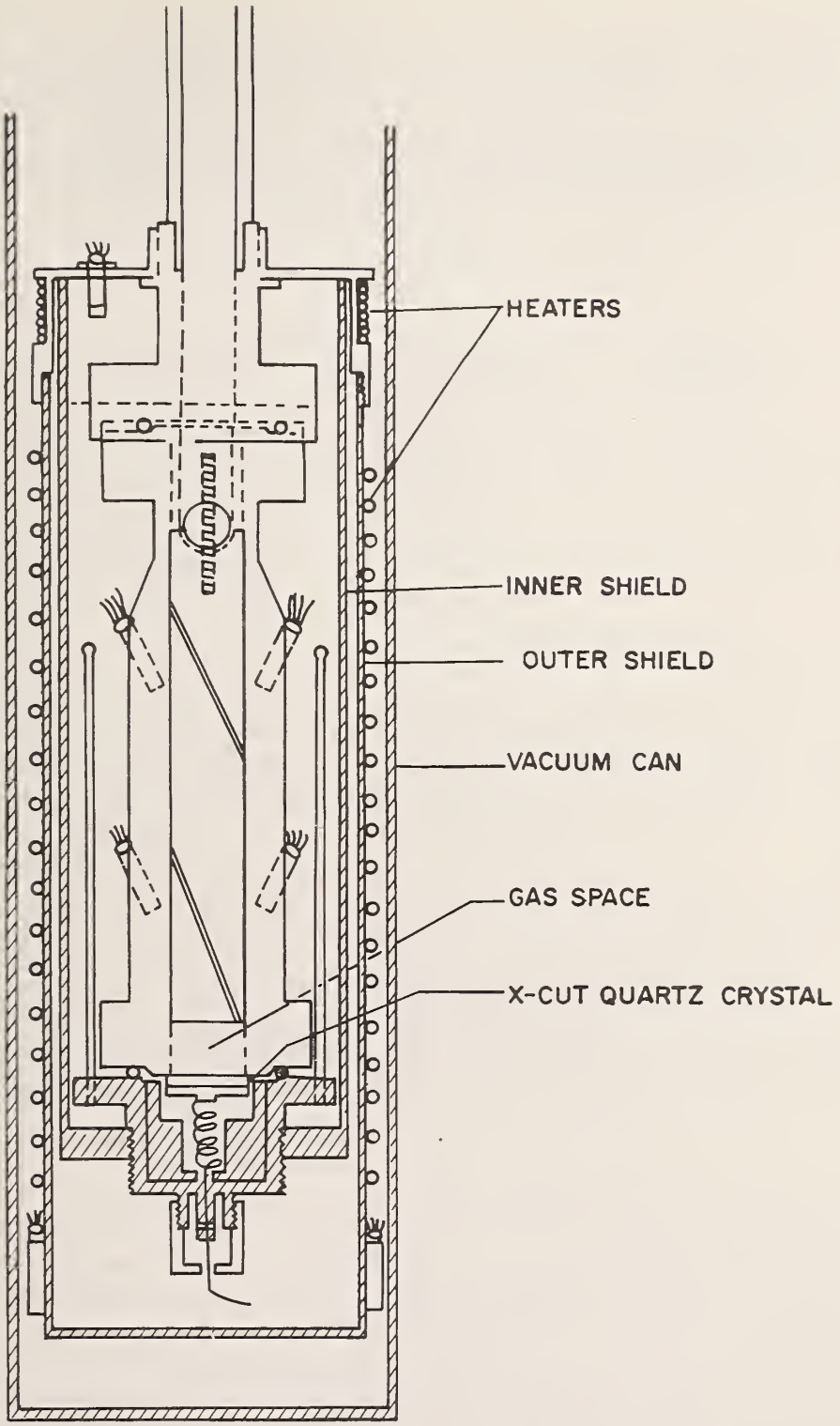


Figure 1. Schematic of the low temperature section of the NBS Acoustical Thermometer.

our acoustically determined value for an isotherm at the NBP of e-$H_2$ was 20.284 K, but the recommended value (see ERRATA for page 189 of *Temperature, Vol. 4,* Instrument Society of America, Pittsburgh, Pa., 1972) was 20.2786 K $\pm$ .0050 K. The value 20.2786 was derived from smoothing through a number of isotherms with germanium and platinum resistance thermometers (assuming the resistances are smooth and continuous functions of temperature) and assigning an appropriate temperature to the germanium-thermometer resistance value that had been measured when the resistor was exposed to the NBP of e-$H_2$.

TABLE I

| Date | Number of Data Points | Pressure Range ($kNm^{-2}$) | NBP Temperature (K) | Refrigerant |
|---|---|---|---|---|
| 1966-1967 | 15 | 30-90 | 20.2840 $\pm$ .0014[a] | liquid $^4$He |
| 1968 | 10 | 30-120 | 20.2752 $\pm$ .0007[a] | liquid $^4$He |
| 1971 | 7 | 30-80 | 20.2747 $\pm$ .0003[a] | liquid $H_2$ |
| 1973 | 13 | 13-100 | 20.2756 $\pm$ .0006[a] | liquid $^4$He |

[a]Statistical uncertainty of the linear fit.

In Table I there is listed information regarding four separate isotherms that were determined at the NBP of e-$H_2$. For each isotherm the data appeared to be represented linearily over the indicated pressure range with data points exhibiting random deviations from the computer fitting of a straight line. We believe that the temperature values given in the lower two rows are more accurate than that indicated in the first row. The data of the first three rows were taken with the fused quartz rod acoustical thermometer while that of 1973 was obtained using the laser interferometer instrument. Unfortunately the slopes of the four isotherms are not in precise agreement; there appears to be a variation of the isotherm slope with the amount of electrical power that is required to maintain the acoustical thermometer's temperature above that of the refrigerant bath. Not unexpectedly, the required power is about a factor of ten less for the $H_2$ refrigerant bath than that for the helium bath. For reasons that are not yet completely understood, larger helium gas pressures within the acoustical thermometer require greater electrical powers to maintain a constant temperature of the instrument. Presumably this is caused by convection currents of helium gas within the annular space of the instrument or conduction down a stainless steel tube. This problem naturally merits further attention. At the present time we feel that the values 20.2747 K and 20.2756 K probably best represent the temperature value for our acoustical thermometry determinations of the NBP of e-$H_2$; this feeling is based upon the fact that with low electrical powers thermal equilibrium of the instrument was more likely achieved in the $H_2$ bath and upon the use of the second radiation shield and laser. Because the maximum estimated uncertainty in realizing the e-$H_2$ NBP was 1.4 mK [6], it can be presumed that the acoustical thermometry determinations of the NBP are 20.275 K $\pm$ 0.002 K. This certainly is in good agreement with the result of K.H. Berry's measurements [7] -- P-V Isotherms of $^4$He at Low Temperatures.

In view of the previous discussion and the above value for the NBP of e-$H_2$ it may be quite possible that the reported [4] isotherm temperature determinations involve a systematic error, as indicated by Cetas and Swenson [8] and Swenson [9].

2. Normal Boiling Point of Ne of Natural Composition

In June and July of 1972 an isotherm, consisting of 22 data points, for helium gas was measured at

a temperature that was close to the NBP of natural neon. The pressure range covered 10 to 130 $kNm^{-2}$. A straight line was fitted by the method of least squares to the data, and deviations of data points from the linear representation appeared to be random. The resulting intercept of the isotherm yielded a temperature value of 27.0953 K ± .0005 K. Truncation of the higher pressure data points down to 70 $kNm^{-2}$ (15 data points) yielded temperature values that varied between 27.0953 K and 20.0955 K. Two of the data points were obtained from the use of a gas cavity diameter of 2 cm and both were within the statistical fitting (equivalent to 1 mK) of the data points.

It is possible to obtain the acoustical value of temperature for the NBP of Ne (nat. comp.) because Furukawa's determination [10] was on the NBS-1955 scale and the germanium resistor #561 that we used has been calibrated on the NBS-1955 scale at 27.0935 K at a resistance of 30.5115 (dR/dT = .0022 Ω/mK). Thus the resistance of #561 can be normalized to the temperature 27.096 K (NBS-1955) that Furukawa obtained, and the appropriate resistance for #561 is 30.506 Ω. The acoustical isotherm, for a resistance value of 30.507 Ω for #561, yielded a temperature 27.0953 K and therefore if the resistance of #561 had been maintained at 30.506 the corresponding temperature would be 27.0958 K. For all practical purposes the value of the NBP of Neon (nat. comp.) that was determined with the NBS acoustical thermometer is identical with the value that Furukawa determined on the NBS-1955 scale. Compton [11] found a value of 27.0961 K (NBS-1955) for the NBP of Neon of natural composition. The agreement among these three values is undoubtedly fortuitous. Furukawa's temperature uncertainty was stated as 1 mK and the standard deviation for fitting the acoustical isotherm was 0.5 mK.

In view of our experience with unknown systematic errors that can occur when relatively large powers are applied to the acoustical thermometer (the refrigerant for this work was liquid helium) it seems preferable to consider the value 27.0958 K for the NBP of Ne as preliminary and await the results of further experiments that will be performed with liquid neon as a refrigerant liquid. This work will be done in the near future with the laser interferometer apparatus.

### 3. Superconducting Fixed Points

Initial steps towards determining temperature values for the superconductive transition fixed points [12] of lead and indium have been undertaken. Measurements have been made, in the germanium thermometer calibration apparatus, [13, 14] of the resistance values for several germanium thermometers during the traverse of the transitions. One or more of these germanium thermometers will be mounted in the acoustical thermometer and the two appropriate isotherms will be determined. It is expected that this experimentation will be completed by the date of the CCT meeting and the results will be communicated to the group.

## D. Discussion

Although a boiling point (not the normal boiling boint) of liquid helium has not been redetermined, additional data were acquired on the 4.2 K isotherm in May 1972. The acoustical thermometer was equipped with a second radiation shield in this work and the germanium resistors indicated that the same temperature was attained as for the "4.2 K isotherm" reported earlier [4]. The determined temperature was 4.2114 K ± .002 K which is within the limits of the previously reported [4] value 4.212 K ± .001 K. In the new isotherm determination nine data points were acquired at pressures between 4 $kNm^{-2}$ and 40 $kNm^{-2}$ but it was necessary to truncate the upper pressure limit to 16 $kNm^{-2}$ (6 data points) to achieve a random distribution of data-point deviations for a linear fitting. Again this indicates a boiling point of liquid $^4He$ (close to the normal boiling point) that is 9 or 10 mK greater than that associated with the $T_{58}$ helium-4 vapor pressure-temperature scale. This result also is consistent with the reports of the $^4He$ normal boiling point by K.H. Berry [7] 4.2240 K and A.R. Colclough [15] 4.2218 K ± 2.5 mK. ($T_{58}$ NBP is 4.215 K).

We have no new data to indicate a value of the e-$H_2$ triple point different from that listed in the Errata for Low Temperature Fixed Points of the 5th Temperature Symposium [6]. There the T (acoustical-smooth) for the e-$H_2$ triple point is 13.8047 K whereas the actual temperature of the isotherm yielded 13.0857 K.

In some instances in the preceding report [6] the statistical uncertainties of representing the data by a linear fitting have been indicated. These should not be construed to be estimates of the total uncertainties. As was indicated earlier, it is felt that the effect of temperature gradients and the thermal equilibrium of the apparatus components constitute problems in our experiments. Appropriate steps are being taken to minimize or control these effects. It is probably desirable that all isotherms between 10 and 24 kelvins be measured using a refrigerant bath of liquid or solid hydrogen. This may not be possible due to laboratory safety regulations, and there is always the concern of the possible contamination of the working helium gas by hydrogen gas. The results reported in row 3 of Table I involved the use of liquid hydrogen as a refrigerant. After the isotherm was completed the helium gas was analyzed and found not to contain hydrogen gas within the resolution of the mass spectrometer that was employed.

We are aware of the reports by Colclough [16] and Quinn [17] and recognize that there are possible high frequency acoustical thermometer errors that are difficult to detect. We have reported attempts to investigate the effect of the Helmholtz-Kirchhoff [18, 19] correction as it might be applied to our instrument. In addition to not observing any effect due to changing the gas cavity diameter (note previous remarks on the 27 K isotherm), we, as yet, have not observed any systematic departures of the data points from linearity at low pressures such as the above mentioned correction predicts. If we have effectively resolved problems with the e-$H_2$ NBP isotherm and if 20.275 K is the temperature value of the point, then it would appear that serious acoustical thermometer errors which can not be evaluated are not of serious significance in our experimentation. It will be particularly interesting to determine the value of the normal boiling point of neon, with liquid neon as a refrigerant, and compare this result with those obtained from other types of measurements, P-V isotherms and gas thermometry.

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## Appendix 2
## Noise Thermometry
R. J. Soulen, Jr.

### A. Introduction

Experimental studies by Johnson [1] led Nyquist [2] to propose the following law relating the noise power of a resistor to absolute temperature

$$\langle V^2 \rangle = 4kRTB, \tag{1}$$

where $\langle V^2 \rangle$ is the average of the square of the noise voltage, k is Boltzmann's constant, R the value of the resistance, and B the bandwidth of the detector. This equation has shown to be a limiting form of a more general result [3], but for temperatures above 0.001 K, we can consider Equation 1 as rigorous. We thus have an ideal and simple law of physics at our disposal for use in thermometry. Despite its simplicity and age, thermometry based on this principle has not been pursued for temperature work below 20 K until very recently. The reason is that accurate measurement of $\langle V^2 \rangle$ has posed a severe problem. A consideration of Figure 1(A) will help demonstrate the problems. The resistor at temperature, T, provides a time-varying noise voltage $V_n$ to a room-temperature amplifier of gain G. The amplifier has an input noise voltage, $V_a$. The output voltage of the amplifier, $V_o = G(V_a + V_n)$ is filtered by a band pass filter of width B. The filter is followed by a circuit to square this voltage, so that the output at this point in the circuit is $MG^2(V_a + V_n)^2B$, where M is gain of the squaring circuit. An averaging circuit yields the final output of the device,

$$\langle V_{out}^2 \rangle = M\ B\ G^2(\langle V_a^2 \rangle + \langle V_n^2 \rangle), \tag{2}$$

where cross-product voltages, if assumed to be uncorrelated, will average to zero. We can now understand the measurement problems from equation (2). The best room temperature amplifiers have amplifier noise roughly equivalent to a resistor at 20 K, so that if the noise thermometer is at 20 K, we have to apply a 100% correction to the temperature measurement! Of course, at even lower temperatures this problem is proportionally worse. Since $\langle V_a^2 \rangle$ may vary, application of such a correction is intolerable. A scheme using two amplifiers at room temperature and a cross-correlation procedure has been used to suppress the noise of the amplifier but there remained a residual amplifier noise contribution equivalent to 0.1 K [4]. The problem of the contribution of the amplifier noise to the output was recently solved using a very sensitive Josephson junction device, called a SQUID magnetometer, for which $V_a \simeq 0$. In this scheme, shown in 1(B), the noise resistor is connected to a superconducting inductive circuit, thus converting the voltage noise to flux noise by the relation

(3)

$$\phi_n = Li_n = \frac{LV_n}{R}, \tag{3}$$

where $\phi_n$ is the magnetic flux noise in the inductor, L, caused by the noise currents from the resistor. The SQUID magnetometer, generally maintained at 4.2 K, acts as a very sensitive and extremely low-noise preamplifier. The RL circuit is coupled to the magnetometer by the mutual inductance $M_o$. The post-detection chain is identical to the one in Fig. 1(A), so that the final output is

$$\langle V_{out}^2 \rangle = M_o M B G^2 \left(\frac{L}{R}\right)^2 \left(\langle V_n^2 \rangle + \langle V_a^2 \rangle\right) \tag{4}$$

The amplifier noise contribution is so small that it contributes a temperature error of $\sim$ 0.0001 K, and thus is negligible. Though this circuit eliminates the amplifier noise contribution, and has been used for low temperature experiments [5], it does suffer from one significant problem. The various multiplicative factors in Equation 4 must be carefully calibrated in order to use the device as an absolute thermometer.

B. Noise Thermometer

NBS is conducting experiments on yet another noise thermometer which uses a Josephson junction. Instead of inserting the resistance into an inductive circuit and then inductively coupling the output to a Josephson junction magnetometer, the Josephson junction is connected in parallel to R. An elementary analysis of the junction shows that the instantaneous noise voltage will be directly converted into a signal of frequency f determined by the well-known Josephson junction relation [6]

$$V_n = \frac{h}{2e} f_n = \phi_o f_n \tag{5}$$

where h is Planck's constant, e the electronic charge, and $\phi_o = h/2e$. This signal at frequency $f_n$ is amplified with a high-frequency amplifier, then $f_n$ is measured with a frequency counter. Successive frequency counts will vary, because the noise voltage is varying. The remarkable aspect of this form of noise thermometer is that it directly yields values of thermodynamic temperature. Because the noise voltage is directly converted to frequency and the V-f conversion involves fundamental constants (see Equation 5), no arbitrary coefficients creep into the final equation relating the output to temperature. In fact, by calculating the variance, $\sigma$, of the frequency counts, it can be shown that [7]

$$\sigma^2 = \sum_{i=1}^{N} \frac{(f_i - f_{i+1})^2}{2N} = \frac{4RT}{\phi_0^2 \tau} \tag{6}$$

where $\tau$ is the gate time of the frequency counter and N is the total number of frequency counts taken. To reemphasize the point, there are no arbitrary coefficients relating the measured quantity $\sigma^2$ to T which need a separate calibration. Further details about how this thermometer can be used to obtain an in situ calibration of R, and how other moments of the frequency counts can be used to look for spurious noise, can be found elsewhere [8].

This noise thermometer is capable of high accuracy and should span the full temperature range from 0.001 K to 20 K (the present upper limit of superconductivity). Because the variance is a statistical quantity, the accuracy of a temperature measurement is given by

$$\Delta T/T = \sqrt{2/N} \tag{7}$$

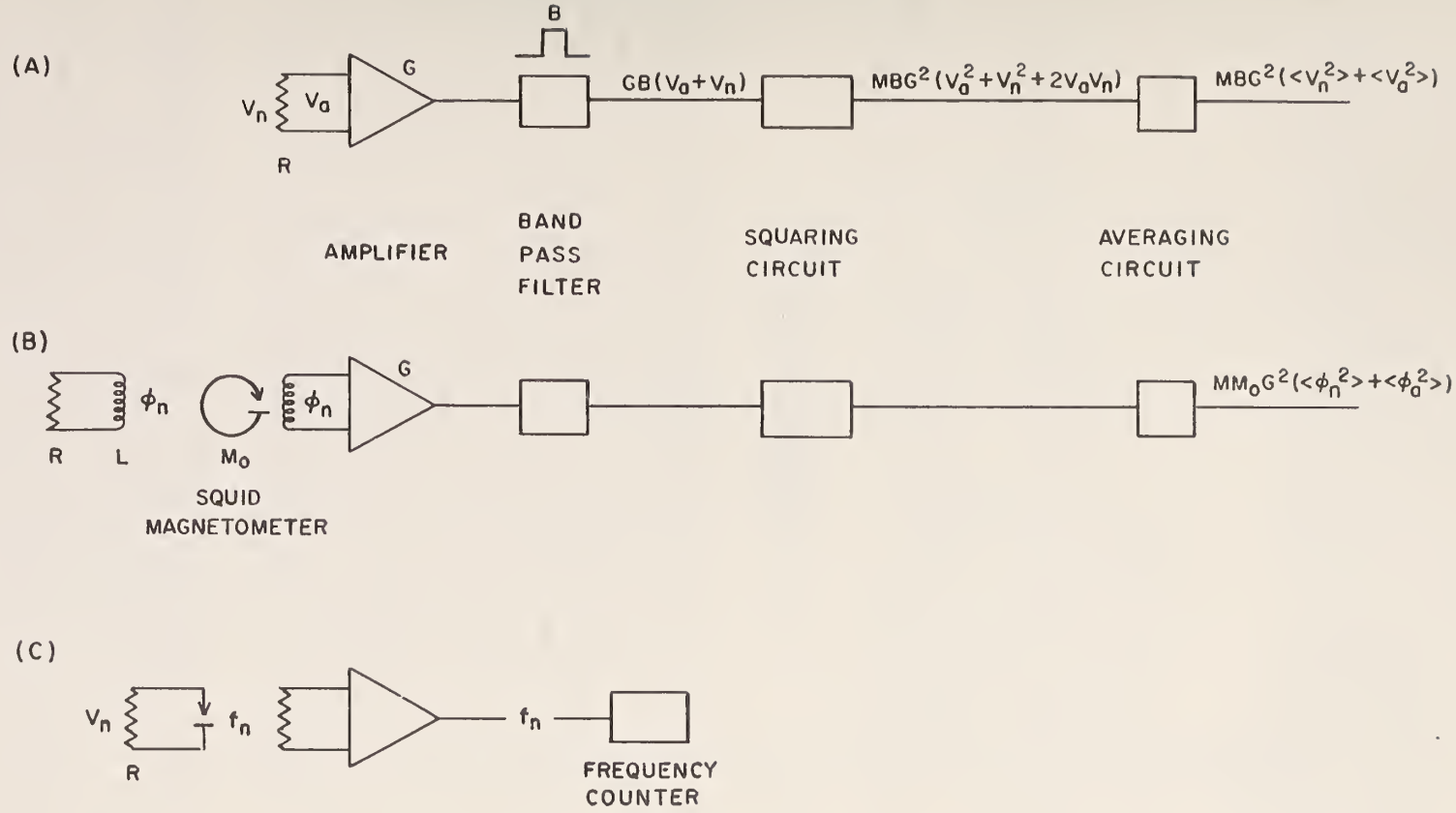


Figure 1. (A). Conventional noise thermometry.
(B). Noise thermometry using a SQUID magnetometer.
(C). NBS noise thermometer.

The noise thermometer has been compared at 0.020 K, 0.030 K, 0.050 K, 0.100 K, 0.410 K, 0.475 K and 0.700 K with a temperature scale obtained by calibration of a CMN sphere against the $^3$He vapor pressure scale. The agreement was within the 5% statistical uncertainty of the noise thermometer measurements.

The most rigorous testing has been made by comparing the noise temperature scale to one obtained from a $^{60}$Co γ-ray anisotropy thermometer. More detailed information about this comparison can be found in Appendix 3. Once this low temperature comparison is completed from 0.010 K to 0.040 K, the $T_c$ of Be and W will be determined. The noise thermometer will then be used to assign thermodynamic temperature values to the remaining superconductive fixed points; and will ultimately be compared with the acoustical thermometer scale from 2 K to 20 K.

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## Appendix 3
## $^{60}$Co γ-Ray Anisotropy Thermometry
H. Marshak

Single-crystal (hcp) $^{59}$Co$^{60}$Co γ-ray anisotropy thermometers have been used and evaluated in various experimental situations [1-4]. The main advantage of using this particular γ-ray anisotropy thermometer is that all the parameters of the nuclear spin system along with its decay properties have been extensively studied. Thus, one can calculate the effect of any small uncertainties in these quantities on the thermodynamic temperature. Another advantage in using this thermometer is that no magnetic field is needed to achieve ordering of the nuclear spin system -- only low temperatures. The presence of an applied magnetic field would, of course, perturb the noise thermometer with which we are comparing.

We have carefully examined the possible sources of error in using a single-crystal $^{60}$Co γ-ray anisotropy thermometer. The reproducibility of the anisotropy at a fixed temperature is within the statistical uncertainty of the measurements; systematic errors arise from the uncertainty in the value of the hyperfine interaction, neglect of a possible quadrupole interaction and/or a small admixture of M3 radiation, mosaic spread of the crystal, solid angle correction factor, effects of scattered γ-rays. The effects of these errors have been calculated and some experiments have been performed to check these calculations.

Preliminary results of our comparison measurement between the Josephson junction noise thermometer and the $^{60}$Co γ-ray anisotropy thermometer have been reported previously [5] and are also shown in Figure 1. The error bars shown are the combined errors of both thermometers, and in several cases reflect the larger scatter of noise temperature. From this figure it appears that there is a discrepancy of about 1 mK between the two thermometers. Part of this discrepancy can be explained by maximizing all the possible errors mentioned above in the $^{60}$Co thermometer. When this is done the dashed line in Figure 1 replaces the solid one. Although the two thermometers are then in better agreement, the results still show a slight discrepancy. Further work is continuing which will reduce the error bars and should show whether or not there is a real discrepancy between these two absolute

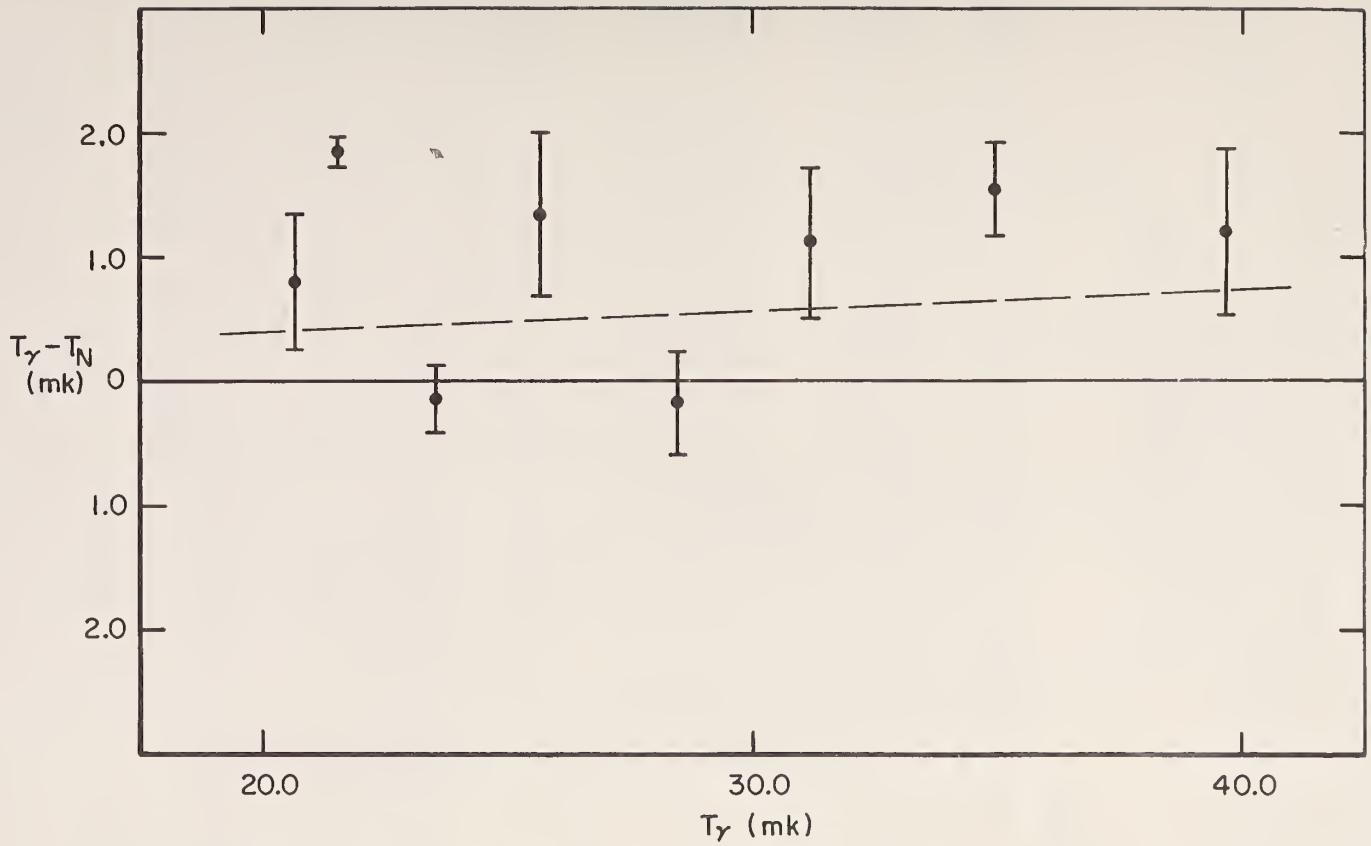


Figure 1. The difference between the temperature obtained from the $^{60}Co$ γ-ray anisotropy thermometer ($T_\gamma$) and that obtained from the noise thermometer ($T_N$) plotted versus $T_\gamma$. The dashed line is explained in the text.

thermometers.

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## Appendix 4
## Superconductive Thermometric Fixed Points
J. F. Schooley and R. J. Soulen, Jr.

### A. The NBS SRM 767 Unit

Experimental studies carried out over the past five years at NBS have shown that the superconductive transitions of five elements (lead, indium, aluminum, zinc and cadmium), extending from 0.5 K to 7.2 K, were sufficiently narrow and reproducible to be used as thermometric fixed points with a precision of ± 1 mK [1]. Presently, the National Bureau of Standards is offering for sale a unit, referred to as SRM 767, which consists of samples of these five superconductors surrounded by a coil set [2]. The superconductive transition of each sample causes a change in mutual inductance of the coils which is easily detected by a simple electronic circuit [3].

As of this writing, thirty of these devices have been sold, many to scientific and government laboratories around the world. More importantly, in this context, at least one unit is in the possession of each of the national standards laboratories of the USA, UK, Australia, Canada and the Netherlands. It is our expectation that the values for the transition temperatures ($T_c$) of these units will be evaluated in terms of the national temperature scales and that the units can be used as the basis for intercomparison of these scales.

To this end, work has already begun at NBS which will lead to a more direct determination of the superconductive transitions of lead and indium on the NBS Provisional Temperature Scale 2-20 (1965). We would like to take "acoustic isotherms" at the superconductive transitions. Unfortunately the NBS Acoustical Thermometer from which this temperature scale is derived is not equipped with the capability of making mutual inductance measurements used to detect the superconductive transitions. Thus the following alternate scheme was used. A SRM 767 unit was installed in a second apparatus which contains two germanium resistors which were calibrated on the acoustic thermometer apparatus and thus bear the NBS Provisional Temperature Scale 2-20 (1965). This second apparatus is used to calibrate all germanium resistors sent to NBS for calibration on this scale, and was easily altered to accept a SRM unit. The superconductive transitions were measured with the usual attention given to the elimination of dc magnetic fields [2]. Thus the transition temperatures of lead and indium were recorded on these germanium resistors (the superconducting transition temperatures of the other elements in SRM 767 fall outside the 2-20 K range of the acoustic scale).

Table I summarizes the values of the two calibration resistors, the corresponding temperatures calculated from the usual eighth-order interpolation equation (since the transitions do not fall one one of the acoustic isotherms) and, for comparison, the transition temperatures quoted in the SRM 767 certificate. The agreement is reassuring, for the SRM 767 certificate values were derived by calibrating a paramagnetic material, chromium methylammonium alum, with a germanium resistor at the acoustic

isotherm values and then using the paramagnet to interpolate to the values of the transition temperatures. This procedure demonstrates the ease with which the SRM 767 can be used and also tests the self-consistency of our thermometry. The experiment also served to transfer the transition temperatures of Pb and In to the calibration apparatus so that all resistors subsequently calibrated in this cryostat will bear this extra information. For instance, three other germanium resistors will be installed into this calibration apparatus, their resistance values noted at the superconductive transitions of these two materials, and they will be submitted to the international "round robin" on germanium resistors.

TABLE I

Determination of the Transition Temperatures of Lead and Indium on the NBS Provisional Temperature Scale 2-20 (1965).

| Date | $R_{565}$ (Ω) | $T_{565}$ (K) | $R_{540}$ (Ω) | $T_{540}$ (K) | Assigned Value To SRM 767 (K) |
|---|---|---|---|---|---|
| January 21, 1974 | 789.444 | $7.201_9$ | 851.507 | $7.202_3$ | 7.201 |
| January 22, 1974 | 789.451 | $7.201_8$ | 851.539 | $7.202_2$ | 7.201 |
| January 25, 1974 | 789.505 | $7.201_6$ | 851.578 | $7.202_1$ | 7.201 |
| January 25, 1974 | 789.507 | $7.201_6$ | 851.516 | $7.202_3$ | 7.201 |
| January 22, 1974 | 4181.00 | $3.415_8$ | 4448.2 | $3.415_5$ | $3.416_7$ |
| January 25, 1974 | 4182.18 | $3.415_3$ | 4447.3 | $3.415_8$ | $3.416_7$ |
| January 25, 1974 | 4182.31 | $3.415_3$ | 4447.4 | $3.415_8$ | $3.416_7$ |

Finally, one or more of these resistors will be transferred to the acoustic interferometer apparatus so that isotherms can be taken at the In and Pb fixed points.

B. Other Superconductive Fixed Points

If current experience shows this approach to cryogenic thermometry to be useful and convenient, it is quite possible that a wider choice of superconductive transition temperatures will become desirable. Anticipating this possibility, work is proceeding at the National Bureau of Standards to examine the characteristics of W, Be, Ir, and $Nb_3Sn$ (the respective transition temperatures are approximately 0.01 K, 0.02 K, 0.10 K, and 18 K). These materials are considerably more difficult to prepare in pure, unstrained samples than those in the present National Bureau of Standards device, but present preliminary results offer the possibility that each may be useful. Since the materials differ in purity and behavior, each material will be discussed under a separate heading.

1. $Nb_3Sn$

Niobium and tin form a broad set of alloys, as well as several compounds [4]. The compound $Nb_3Sn$ was chosen as a possible superconductive fixed-point material because its transition temperature is close both to the IPTS-68 hydrogen boiling points and to the upper temperature limit for superconductivity, and because the work of DeSorbo [5] indicated that transition widths in this material might be

as small as 30 mK. Although DeSorbo actually found narrower transitions in samples prepared from Nb with approximately 770 ppm Zr impurity, we used the purest niobium commercially available in preparing our samples. We used DeSorbo's technique of baking 1 mm-diameter Nb wires in an atmosphere of Sn vapor at approximately 960 °C for 72 hours. Presently our procedure is to outgas the Nb and Sn (99.999% pure powder, commercially available) at progressively higher temperatures, and to interpose a niobium scavenger between the Nb and Sn to remove oxygen before melting the Sn.

A group of four samples was prepared by this technique. The superconductive transition temperatures were obtained, as usual, by mutual inductance measurements and calibrated germanium resistance thermometers. All four transition midpoint temperatures occurred within 2 mK of 18.009 K, and each of the four transition widths was 20 $\pm$ 5 mK.

The present cryostat temperature control technique involves a visual observation of the transition midpoint. This is a very convenient method for general use, but it introduces a set-point uncertainty of perhaps 10% of the transition width. Thus a $\pm$ 2 mK uncertainty in the transition temperature is not unexpected in the present case.

While a 20 mK transition width is larger than desired, it is substantially narrower than DeSorbo's experiments gave, and we find the present results encouraging. We plan to continue the study, varying the preparation in an attempt to prepare samples with still narrower transitions.

2. Iridium

Progressively poorer thermal contact at low temperatures required a new design of the sample holder for the low temperature superconductors W, Be, and Ir. The holder used for low temperatures was made by brazing 200 #43 AWG bare copper wires to one end of a threaded copper stud (2-56). This assembly was gold plated, and then the samples were bound tightly with thread and glued into position. A primary and secondary coil set was placed around the samples and anchored to the stud to form a complete unit. The transitions were detected in the usual way with a mutual inductance bridge.

The lower-$T_c$ materials require much more careful elimination of ambient magnetic fields in order to prevent the occurrence of the metastable states of "supercooling" (i.e., the sample remains in the normal state when the temperature is lowered below $T_c$) and "superheating" (i.e., the sample remains in the superconducting state when the temperature is raised above $T_c$). We encountered no serious superheating problems, and we did develop experimental procedures for reducing supercooling effects to a manageable level. The best procedure was to insert a high permeability magnetic shield inside the usual Helmholtz coil used to cancel ambient magnetic fields.

We obtained several iridium samples from different sources and determined from residual resistivity measurements that they were not of uniform purity. We list the results for the determination of the superconductive transition temperatures of five samples in Table II; several other samples were unsatisfactory for one or more reasons. For example, we found that only those samples which had been annealed and presumably purified by the process of electron-beam zone refining possessed sufficiently narrow and reproducible transitions. A germanium resistance thermometer previously calibrated with a CMN thermometer which had in turn been calibrated with a SRM 767 unit provided the temperature scale used in these experiments. The estimated accuracy of this procedure is approximately $\pm$ 1 mK. All the samples shown in Table II had transitions which were no broader than 1.5 mK and, by defining the midpoint of the transitions as $T_c$, we found that the transition of each sample was quite reproducible despite several thermal cycles from 300 K to 0.020 K over a time span of one year. We conclude that each sample must be individually calibrated until a preparation technique is found which produces samples of uniform chemical quality.

TABLE II

Superconductive Transition Temperatures for Iridium

| Sample Designation | $T_c$(K) Experiment 1 | $T_c$(K) Experiment 2 | $T_c$(K) Experiment 3 | $T_c$(K) Experiment 4 |
|---|---|---|---|---|
| Ir 1 | $0.112_5$ | $0.113_0$ | | |
| Ir 2 | $0.108_5$ | $0.108_5$ | $0.109_0$ | |
| Ir 3 | $0.099_7$ | $0.100_0$ | $0.100_3$ | $0.100_2$ |
| Ir 4 | $0.099_3$ | $0.099_0$ | $0.098_7$ | |
| Ir 5 | $0.099_2$ | | | |

3. Beryllium

The results for several beryllium samples are shown in Table III. Samples Be 1, 2, 3, 4 came from a 2 kg lot of vapor-deposited material which we hope is sufficiently homogeneous to provide several reference units. Clearly, more data are needed on the reproducibility of individual samples. For the one experiment in which all four samples were examined, the transitions fell at the same temperature. These data are encouraging in the sense that they imply that this large lot of beryllium is homogeneous insofar as a measurement of the $T_c$ is concerned. The transitions of all four samples are quite sharp ($\sim$ 0.1 mK) and satisfy this criterion for superconductive fixed points.

The other two samples, BeBT and BeFI are unique since they were not cut from a larger lot of material. They have higher purities and suggest that the $T_c$ of the four samples do not represent that of pure Be. Also their superconductive transitions are extremely narrow ($\sim$ 0.003 mK). In conclusion, we plan to carry out further experiments on samples taken from the 2 kg lot to determine their suitability as superconductive reference temperatures.

TABLE III

Superconductive Transition Temperatures of Beryllium

| Sample Designation | $T_c$(K) Experiment 1 | $T_c$(K) Experiment 2 | $T_c$(K) Experiment 3 |
|---|---|---|---|
| Be 1 | $0.023_2$ | | |
| Be 2 | $0.023_2$ | | |
| Be 3 | $0.023_0$ | $0.022_4$ | $0.022_3$ |
| Be 4 | $0.023_0$ | | |
| BeBT | $0.024_3$ | | |
| BeFI | $0.023_6$ | $0.023_8$ | |

4. Tungsten

We have measured only one sample of W, and find that it has a very sharp transition ($\sim$ 0.01 mK) at

approximately 0.015 K (± 0.002 K). The sample is of high quality and, since tungsten is easy to obtain in high purity from commercial sources, we are optimistic that W will soon qualify as a superconductive fixed point.

C. Conclusions

The superconductors Pb, In, Al, Zn and Cd provide in a complete device five fixed points extending from 0.5 K to 7.2 K. They have already been circulated and are actively being evaluated at other independent laboratories. We hope that the new candidates, W, Be, Ir, and $Nb_3Sn$ will also qualify as fixed points so that we may ultimately have a set of well-spaced reference temperatures extending from 0.020 K to 20 K.

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## Appendix 5
## A Magnetic Thermometer for 1 K - 20 K
R. P. Hudson

The magnetic thermometer is well-known as a useful sensor for the cryophysics region and many paramagnetic "working substances" exhibit Curie Law (or nearly so) behavior, $\chi = cT^{-1}$, over one or more decades of the temperature scale between 0.01 K and 100 K. In principle, this device is especially useful for checking the inter- or intra-consistencies of other scales; it is usually not rendered "absolute" because it is so much easier to measure relative susceptibilities. Even then, its usefulness as a "smoother" or interpolator may be limited by deviations from Curie Law behavior and also by a lack of sensitivity. For example, if the Curie Law is applicable, a typical magnetic thermometer may be calibrated at two temperatures determined by other means, whereafter "all" temperatures are known. If the Curie Law is *not* applicable, more calibration points are needed and the form of $\chi(T)$ takes on a certain arbitrariness--usually rationalized, at best, by appeal to theoretical considerations. Such a "bootstrap" type of operation inevitably suffers from diminished credibility--at, say, mK levels of precision--no matter how cogent and thorough the analysis. Secondly, one will almost inevitably have to strike a compromise between ideal (Curie Law) behavior and best sensitivity via the largest Curie constant.

In addressing the question of finding a truly superior paramagnetic salt for scale-smoothing operations in the 1 K to 20 K region (also for bridging the annoying gap between 4.2 K and 13.81 K), it occurred to me that neodymium ethylsulfate, NES, was an obvious and yet untried choice! The salt is a reasonably strong paramagnet ($S = 1/2$, $g_{||} = 3.5$); hyperfine effects [1] markedly modify $\chi$ only below 1 K; crystal field anomalies are negligibly small below 20 K with the first excited state lying as it does [2] at 150 $cm^{-1}$; the salt is magnetically very dilute and any exchange effects must be extremely small--none is detectable in heat capacity determinations [3] near 1 K and the "ordering temperature" lies no higher than 0.01 K, [4] as could be explained in terms of effectively dipolar interaction coupling only.

The usefulness of any magnetic thermometer diminishes with rising temperature, primarily because the sensitivity $\chi^{-1}(d\chi/dT)$ varies as $T^{-1}$, and also because, eventually, the effects of thermal population of the excited states of the magnetic ion will be manifested in detectable departures from the Curie Law behavior. The excited states will have negligible effect until the quantity $\exp(-\Delta/kT)$ arises above insignificance, say, $10^{-5}$; for NES where $\Delta/k \sim 220$ K, no related problem is to be anticipated below $T \sim 20$ K. Assuming the usual relation $N = AT^{-1}$ where N is the output signal which is proportional to the temperature-dependent part of $\chi$, and A is a constant, the minimum detectable $\Delta T$ is equal to $-(T^2/A).\Delta N$, where $\Delta N$ is the minimum detectable signal for the apparatus. The proportionality constant depends upon the Curie constant of the salt, the amount of material employed, and the size of the encompassing mutual inductance. Let us take $\Delta N = \pm 10^{-3}$ and, considering a 2 cm. diameter sphere of NES, $A = 400$.[†] Then the measurement precision, $\Delta T$, will be equal to $2.5 \times 10^{-6}T^2$ and, hence, $\pm$ 1 mK at 20 K.

At the low temperature end of the range under consideration we must pay heed to other departures from ideal behavior, namely, those arising from nuclear hfs effects and dipole-dipole coupling in the case of NES. For a spherical specimen[‡] one has [1]

$$\chi = \frac{1.716 \times 10^{-3}}{T} \left[1 + \frac{1.32 \times 10^{-2}}{T} - \frac{2.34 \times 10^{-4}}{T^2} - \cdots\right]$$

and it is seen that, while the cubic term is negligible above 2 K (and "manageable" at 1 K), the quadratic term is large enough to cause concern in the type of measurements under discussion. Two courses lie open: as the quadratic term stands on a very sound theoretical footing it can be accommodated within the analysis of the data without controversy; or, on the same grounds, a specimen of ellipsoidal shape may be employed, of eccentricity calculated to reduce the correction term to zero [5]. One finds that the required shape is oblate, of axial ratio 0.3.

Salts such as manganous ammonium sulfate, MAS, and gadolinium sulfate, GS, are well suited to the range 20 K to 90 K, having Curie constants [0.0205 and 0.0629 $cm^{-3}$, respectively] much larger then that of NES [0.00321 $cm^{-3}$]. They are also isotropic and, hence, may be used in powdered form. Chromic methylammonium alum, CMA, is also isotropic but has "intermediate" sensitivity and cannot be easily used above 30 K; unlike other alums, it is reasonably stable. Departures from Curie Law behavior in these three salts, which are not very firmly established, are small for that region but signigicantly large for $T \leq 20$ K. Cerous magnesium nitrate, CMN, is a relatively weak paramagnet [$c = 8.7 \times 10^{-4}$ $cm^{-3}$], very useful for temperatures below 2 K but, above that temperature, is too insensitive and perturbed by the effect of excited states. Any other known and commonly available salts, for various reasons, are even more inferior to NES for high-quality thermometry in the range 2 K to 20 K.

In summary, neodymium sulfate offers adequate measurement sensitivity and will only depart significantly from Curie Law behavior by dipolar-interaction effects which can be easily handled analytically or eliminated by suitable choice of specimen shape. It thus appears to be the outstanding choice for for magnetic thermometry in the range 1 K to 20 K.

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[†]As are achievable (see Appendix 6) using a conventional ac mutual induction bridge arrangement; SQUID detectors, capable of greatly enhanced sensitivity, could be used with smaller samples and/or for better resolution.

[‡]For a spherical specimen of a cubic material, the quadratic term is identically zero.

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## Appendix 6
## Magnetic Thermometry Between 1 K and 30 K using Neodymium Ethylsulfate, Gadolinium Metaphosphate Glass, and Chromic Methylammonium Alum

B. W. Mangum

We are in the preliminary stages of testing neodymium ethylsulfate, chromic methylammonium alum, and gadolinium metaphosphate glass for use as magnetic thermometers in the temperature range 1 K - 30 K. It is anticipated that these can then be used as interpolating devices between thermometric fixed points.

The approach being used is the measurement of the salt's temperature-dependent magnetic susceptibility by the ac mutual inductance technique (i.e., the Hartshorn bridge) where the mutual inductance is related to this susceptibility as follows: $\chi = a(N - N_\infty) = A/(T + \Delta + \delta/T)$

where $\chi$ = susceptibility

$N$ = mutual inductance at temperature T

$N_\infty$ = empty-coil constant, or mutual inductance at $T = \infty$

$a, A$ = constants which are related to the Curie constant of the salt by multiplication factors which depend on the coil geometry, etc.

$\Delta$ = a constant determined by demagnetizing factor, exchange interactions, etc.

$\delta$ = a constant determined by crystal field and nuclear hyperfine effects and second order dipole-dipole interactions.

The salt under test is in good thermal contact with two copper blocks, which are located some 10 cm from the ends of the coil system, and four germanium resistors are located in one of the blocks. The germanium resistors have been calibrated against the NBS Provisional Temperature Scale 2-20 (1965); [1] one of them is used as the sensor in the temperature control system. The commonly used coil-foil shield surrounds the salt and extends from one copper block to the other.

A highly stable audio oscillator provides the voltage source for the bridge circuit and a filtering network plus a phase-sensitive detector are used in the detection circuit.

The chromic methylammonium alum (CMA) has been used previously in this temperature range as a magnetic thermometer [2] and the interaction constants and crystal field effects in this salt have been measured [3]. We want to use the CMA in our experiment in order to confirm the previous measurements. One very useful property of CMA is that its g-factor is isotropic and, hence, single crystals are not required in the experiment.

The neodymium ethylsulfate (NES) salt has not, to my knowledge, been used previously as a magnetic thermometer in this temperature region. It is, however, similar to chromic methylammonium alum and chromic potassium alum in that its magnetic interactions are weak, resulting in an ordered magnetic state only at a temperature less than 20 mK [4]. One disadvantage of this salt is its magnetic anisotropy [5] but this does not create much of a hardship. It requires only that single crystals be used in the experiments. Unlike that of most other neodymium salts in which the first excited state is very low, the first excited state in NES is approximately 150 $cm^{-1}$ above the ground state doublet [6].

This is sufficiently high that its contribution to the magnetic susceptibility is negligible at temperatures below 20 K.

The gadolinium metaphosphate (GMP) glass has not previously, to my knowledge, been used as a magnetic thermometer. Its magnetic ordering temperature is sufficiently low ($\sim$ 100 mK) [7] to permit its use as a thermometer above 1 K. This material has many desirable properties: it can be cut into any desired shape; its density is relatively high ($\rho \gtrsim 4$); there is no preferred direction and thus there is only one Weiss constant. Furthermore, since the magnetic ion is gadolinium, which is an S-state ion, the g-factor is isotropic and, also, the first excited level is some $10^4$ $cm^{-1}$ above the ground state. The latter can be neglected as far as its contribution to the magnetic susceptibility is concerned.

Now, let us talk about sensitivity. First of all, the ground state in NES is a Kramers doublet, effective spin S = 1/2, with $g_{||} = 3.535$ and $g_{\perp} = 2.072$ [5]. Thus, $g^2 S(S + 1) = 9.37$. In CMA, the ground state in the temperature region above 1 K can be considered a quartet, i.e., S = 3/2, and g = 1.98 [8]. Thus for CMA we have $g^2S(S + 1) = 14.70$ or approximately 1.5 times that for NES. So for the same number of moles of NES and CMA, the CMA is approximately 1.5 times more sensitive than NES. For gadolinium metaphosphate (GMP) glass, the ground state is an $^8S$ state with g = 2. Then $g^2S(S + 1) = 63$ or 6.7 times that for NES and approximately 4.3 times that for CMA. Again, for the same number of moles of GMP, NES and CMA, the GMP is $\sim$ 4.3 times as sensitive as CMA and $\sim$ 6.7 times that of NES. Taking into account the difference in densities of the salts, the relative sensitivities of the three salts, for the same size (volume) sample, are GMP $\sim$ 13.4 times as sensitive to temperature as NES and $\sim$ 8.6 times as sensitive as CMA.

Preliminary measurements on NES show that for an ac field of 1.25 gauss, and a crystal volume of 4 $cm^3$, the sensitivity of our system is 1 - 2 mK at 20 K and would be 2 - 4 mK at 30 K. From this we may calculate that with CMA we would have, for the same size sample and a 1.25 gauss field, a sensitivity of $\sim$ 0.7 - 1.3 mK at 20 K and $\sim$ 1.3 - 2.7 mK at 30 K. For GMP we calculate, for a 4 $cm^3$ size sample (the same as that assumed for NES and CMA) and an ac field of 1.25 gauss, a sensitivity of $\sim$ 0.1 mK at 20 K and $\sim$ 0.1 - 0.3 mK at 30 K.

As far as sensitivity to temperature, then, all three salts are suitable for use as interpolating devices or for use for smoothing of the scale. The GMP provides the highest sensitivity but NES probably requires the smallest correction terms, $\Delta$ and $\delta$. Work toward experimental verification of the suitability of these salts as thermometers from 2 K to 20 K is in progress.

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Appendix 7
A Proposal for a Very Simple Thermometric Device for Use From 0.01 K to 20 K.
J. F. Schooley

## A. Introduction

The lack of a convenient, precise thermometer in the range 2 K to 20 K has done much to inhibit the extension of the IPTS below 13.8 K.

The use of mutual inductance measurements of the paramagnetic susceptibility of various salts is well known in cryogenic thermometry [1]. Indeed, for laboratory thermometry from 0.01 K to 2 K, the cerous magnesium nitrate (CMN)--mutual inductance thermometer has no peer. This system can be made quite small, and its obedience to the Curie-Weiss Law permits *in situ* calibration at only three temperatures to yield millikelvin precision over its useful range. The susceptibility is usually measured with a Hartshorn mutual inductance bridge circuit; unfortunately changes in the resistivity of the copper coils are reflected in the measured mutual inductance. This complication has led to great difficulty in applying this measurement technique to other paramagnetic salts usable above 2 K.

This report notes an experiment which is barely underway at the National Bureau of Standards in which an attempt is made to circumvent the most troublesome of the problems just mentioned.

## B. Mutual Inductance Measurement

In order to avoid the influence of changes in the resistivity of the inductance coil on the measured inductance values, the NBS experiment utilizes the circuit shown in Figure 1. In this circuit, no attempt is made to measure the imaginary component of susceptibility. Rather, the identical secondary coils, S, are connected across a ratio arm transformer, R, in the manner shown. Thus, resistive changes in the two secondary coils (both contained, with the primary coil, P, in the cryostat, C) tend to cancel, so that the ratio arm balance will reflect only the sample susceptibility.

## C. Sample and Coil Construction

A second cause of the large spurious temperature dependence of mutual inductance found in empty linear coil inductors is the change in coil geometry which necessarily accompanies thermal expansion of the coil itself.

To alleviate this problem, the NBS experimental configuration is in the shape of a torus, with four equally spaced secondary coils surrounding a toroidal primary coil, as shown in Figure 2. Here $S_1$ denotes a pair of secondary coils to be used from 0.5 K - 20 K with spherical sample of chromic methylammonium alum (CMA) connected as in Figure 1. A brush of fine, copper wires fills the hollow torus, and forms the thermal link, L, which establishes thermal contact between the CMA sphere and a thermometer calibration block (which is not shown in Figure 2). $S_2$ in Figure 2 denotes a similar pair of secondary coils, one of which surrounds a CMN sphere. This portion of the system can be used from 2 K to 0.01 K; at the latter temperature, the CMA system is virtually useless.

## D. Discussion

The thermometer described above will be considered successful if the mutual inductance - temperature relation follows the Curie-Weiss relation sufficiently closely to permit millikelvin-level thermometry from 0.01 K to 20 K following an in-situ calibration with the NBS SRM 767 device (perhaps including a calibration point near 18 K as well).

If the device were to exhibit a reproducibility of its mutual inductance -- temperature relation

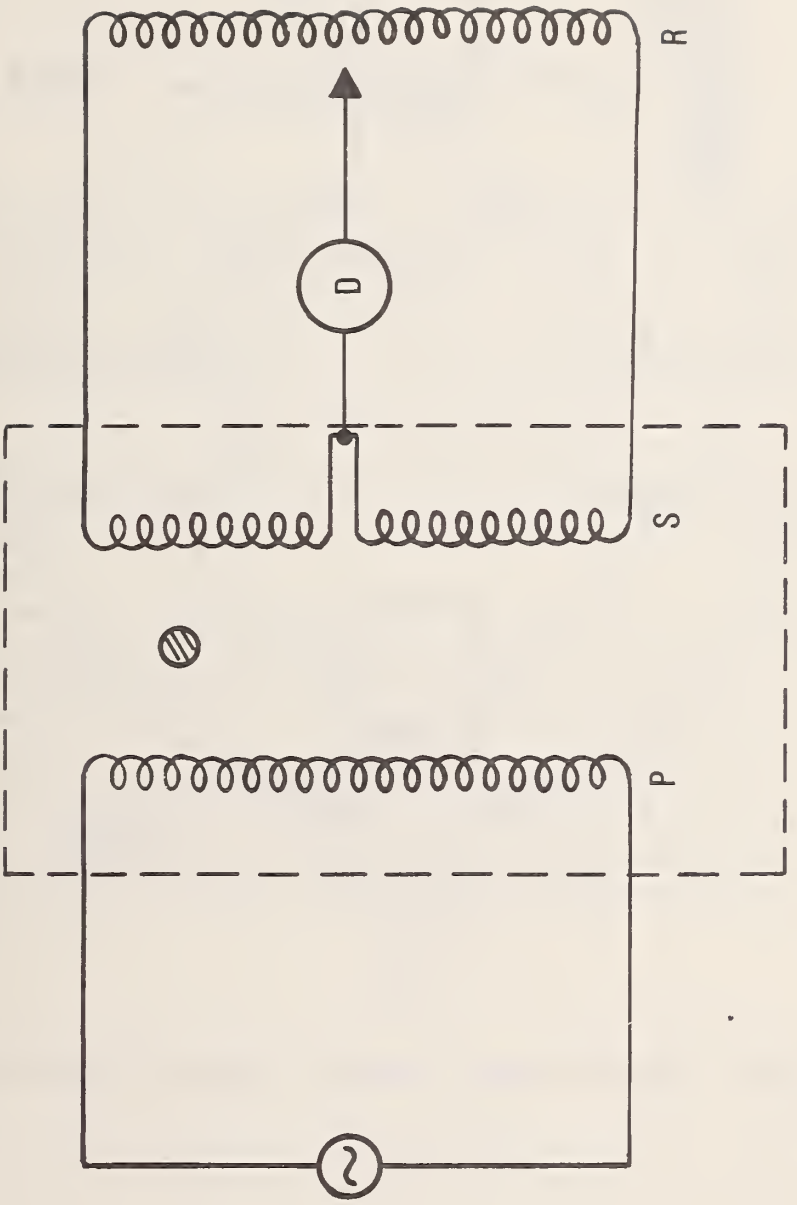


Figure 1. Mutual inductance circuit used to eliminate the effects of temperature-dependent resistivity. D is a phase-sensitive detector, P is the primary, S the secondary, and R an ac voltage divider. The section of the apparatus maintained at low temperatures is enclosed in the dashed box.

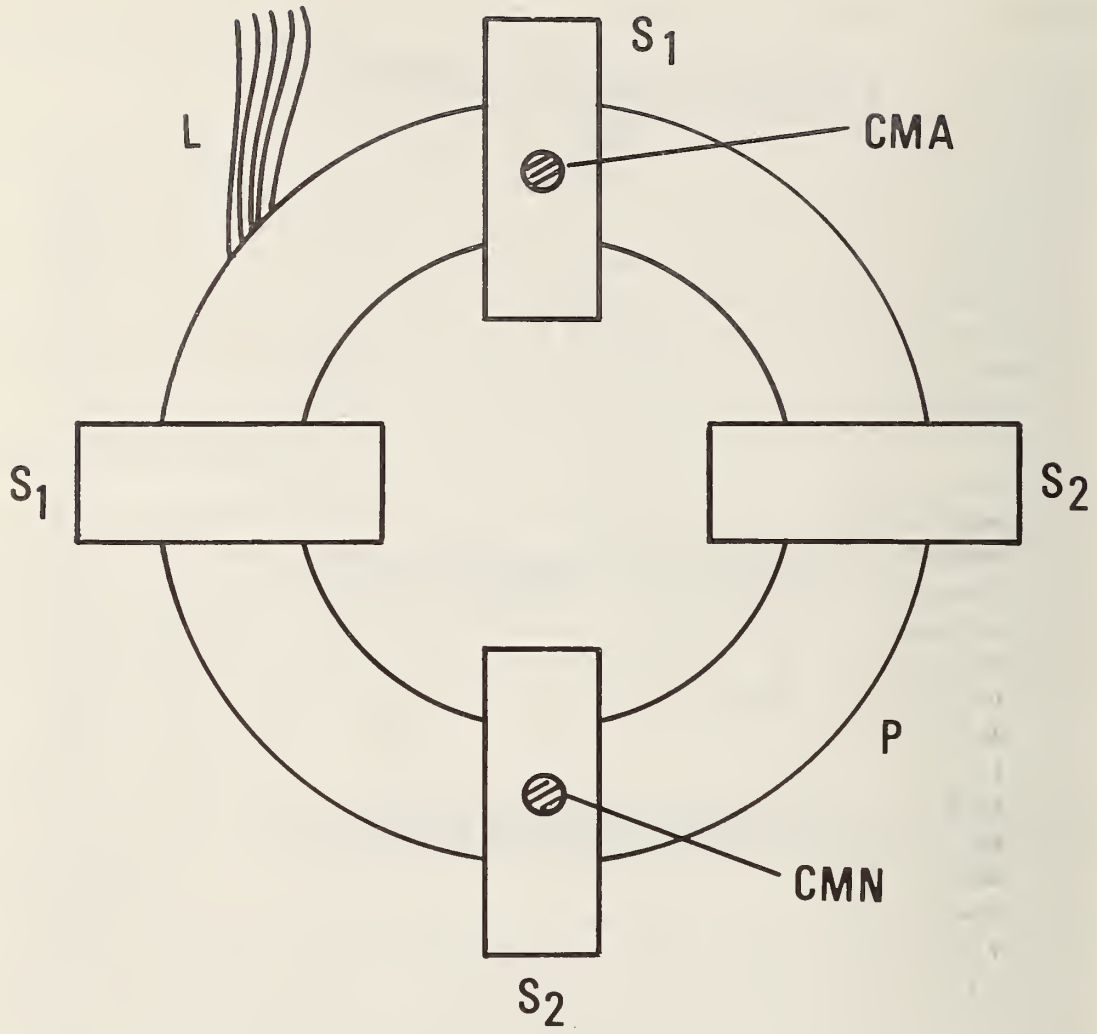


Figure 2. The susceptibility probe containing two paramagnetic materials, CMN and CMA.

following thermal cycling to room temperature, substantial deviation from the Curie-Weiss law would be tolerable.

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1. See, for example, R.P. Hudson, *Principles and Application of Magnetic Cooling*, North-Holland, Amsterdam, 1972.

## Appendix 8
## Nuclear Quadrupole Resonance Thermometry
B. W. Mangum

It has been previously shown [1] that NQR frequencies can be used as a good secondary thermometric standard at temperatures in the range 20 K - 300 K. In that experiment the shift in NQR frequency was due to lattice vibrations which are diminished at lower temperatures and, hence reduces the thermometric sensitivity. NQR has two features worth emphasizing. One is that no static magnetic field is required and the other is that it is a molecular effect independent of sample size and shape. It follows that, provided the material is of sufficient purity, the frequency-temperature relationship is unique to the material and, at the same time, provides a universal calibration curve. We would like to suggest that at temperatures below 50 K there is another phenomenon which gives rise to strongly temperature dependent NQR frequency shifts. That phenomenon is the cooperative Jahn-Teller effect. This produces a distortion of the lattice, [2, 3] which modifies the electric field gradient at the nucleus and, thus, causes the NQR frequency to change. This then provides the basis of possible thermometric fixed points and of thermometers.

Cooperative Jahn-Teller distortions have been observed in several rare earth compounds [4-12] with their transition temperatures, $T_D$, lying between 0.4 K and 33 K. One feature of such distortions is that the NQR is observable throughout the transition region, [4] thereby making such compounds candidates for thermometric fixed points. (This is not the case for cooperative magnetic transitions). It has also been shown that the distortion may be described by the molecular field approximation [6, 8, 13] and assuming this to be the case, the NQR frequencies may then be used as a thermometer over the temperature interval $T_D/3 \leq T < T_D$. The compounds which we have specifically in mind for possible thermometric fixed points as well as thermometers as just discussed are the vanadates and arsenates of Tb, Tm, and Dy and the trichloride of Pr. Their cooperative Jahn-Teller transition temperatures are given in Table I.

TABLE I

| Material | $TbVO_4$ | $TbAsO_4$ | $DyVO_4$ | $DyAsO_4$ | $TmAsO_4$ | $TmVO_4$ | $PrCl_3$ |
|---|---|---|---|---|---|---|---|
| $T_D$(K) | 33 | 25 | 14.0 | 11.2 | 6.0 | 2.1 | 0.43 |

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## Appendix 9
## Nuclear Magnetic Resonance Thermometry
D. B. Utton

### A. Ordered Systems

A technique which can be used to extend the useful range of nuclear resonances to low temperature thermometry is to observe nuclear magnetic resonances in ordered magnetic materials. The resonance frequency is determined by the internal hyperfine magnetic field which is in general proportional to the lattice magnetization (ferromagnets) or sublattice magnetization (antiferromagnets). The resonance frequency is an intrinsic property of the material with the attendant advantage of a unique frequency-temperature relationship. A suitable thermometric material will have its cooperative transition temperature just above the temperature range over which it is to be used as a thermometer.

Two materials have been shown to be useful low temperature thermometers. Senturia [1] has made a careful study of the $^{53}Cr$, $^{79}Br$ and $^{81}Br$ nuclear resonances in ferromagnetic chromium tribromide ($T_c$ = 32.56 K) and their applicability to thermometry. He concluded that the $^{81}Br$ and $^{79}Br$ resonances, whose frequencies increase from 106 and 119 MHz respectively at 25 K to 121 and 130 MHz at 2 K, could be used with millikelvin precision over this temperature range. The samples from two independent sources were compared and found to agree to within the experimental uncertainties of several millikelvins. No hysteresis was observed. Further work on inter-sample reproducibility and also precise measurements using a servo-locked spectrometer will be necessary to establish this material as a secondary standard with millikelvin precision in the range 2 - 25 K.

Manganese fluoride is an antiferromagnet with a Neel temperature of 67.3 K. Gill et al [2] have investigated the suitability of the $^{19}F$ NMR frequency, which increases from 135 MHz at 40 K to 160 MHz at 4.2 K, with the view of using it as a secondary thermometric standard. They have demonstrated that the resonance frequency at 20 K can be determined with a self locked spectrometer to an accuracy equivalent to $\pm$ 0.11 mK. Their measurements showed that with a sample of at least 0.1 $cm^3$ this resonance can be used to determine temperatures with a precision of one millikelvin or better over the range 10 - 40 K. The resonance frequencies of four different specimens, including one with $\sim$ 0.2% $Zn^{2+}$, were shown to be in close agreement. These measurements were made using an electronic circuit containing field effect transistors which were immersed directly in the cryostat. This arrangement has the advantages of both simplicity and a small size.

It should be noted that the nuclear resonances in the two materials under discussion fall in the same frequency range. With a moderate engineering effort, a detector could be constructed which would use both materials. This device would then be capable of maintaining a temperature scale over the

range 2 - 40 K with a precision of 1 mK or better. It has already been shown that the NQR frequency of $^{35}Cl$ in $KClO_3$ can be used to maintain a temperature scale with a precision of 1 mK over the range 50 - 300 K.

B. Paramagnetic Systems

Nuclear resonance techniques can also be applied to thermometry in the temperature region below 1 K [3-6]. It is well known that in suitable materials (for example, several metals including aluminum, copper and platinum), the nuclear magnetic susceptibility is inversely proportional to temperature down into the microkelvin region, i.e., it rigorously follows the Curie law. Resonance techniques have the further advantage of discriminating against stray magnetic impurities. The precision of these measurements is typically in the range 0.1 - 1.0% and hence is useful for temperatures below 1 K. Within this limitation, however, the technique can be used with confidence as a means of interpolating between fixed points and, furthermore, can be used as a thermometer at the lowest temperatures attainable. Experiments are in progress at NBS using the pulse technique, in which the free induction decay of the $^{63}Cu$ and $^{65}Cu$ resonances in fine copper wires, is observed in a steady magnetic field of approximately 0.01 tesla. The amplitude of the free induction decay, which is proportional to the magnetic susceptibility, is used to check the thermodynamic consistency of temperatures assigned to fixed points by other techniques such as noise and nuclear orientation thermometry [7]. The nuclear resonance technique can also be used to determine temperatures directly to within 3% by using the known temperature dependence of the spin lattice relaxation time [4].

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16. ABSTRACT (*A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.*)

This article outlines a comprehensive and long-term program being carried out by NBS scientists of the Cryogenic Physics and Temperature Sections. The goals of the program are the extension of IPTS 68 below 13.81 K and the development of devices which make practical realizations of that scale convenient and reliable. We propose to contribute to international adoption of a thermodynamically accurate scale below 13.81K by analyzing the results of three thermometers: the NBS Acoustical Thermometer (already in operation for several years), noise thermometry using the Josephson effect (recently developed), and γ-ray anisotropy thermometry (recently studied in detail at NBS). Such a temperature scale will most likely be disseminated by the use of certain superconductors as thermometric reference points. Practical interpolation devices will be based on the principles of nuclear and electronic paramagnetism, nuclear quadrupole resonance, and nuclear magnetic resonance. Details of operation, measurement schemes and experimental progress made to date are included in nine appendices.

17. KEY WORDS (*six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons*) Acoustical thermometry; γ-ray anisotropy thermometry; noise thermometry; nuclear magnetic resonance; nuclear quadrupole resonance; paramagnetism; superconductivity; temperature.

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