The Provisional Low Temperature Scale from 0.9 mK to 1 K, PLTS-2000

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An internationally-accepted ultra-low temperature scale is needed to provide the basis for reliable thermometry in the temperature range in which commercial dilution refrigerators operate, and in experiments investigating the properties of ^{3}*He and other condensed matter. Several laboratories have developed* ^{3}*He melting-pressure scales, but there are substantial differences even between the most recent of them. These amount to about* 0.3 % *of* T *near* 500 mK, *rising to about* 6 % *of* T *at* 0.9 mK. *In 1996 a collaboration was initiated between low temperature physicists in national laboratories and elsewhere to derive an equation for the melting pressure of* ^{3}*He which could be accepted for international use from* 1 K *to* 0.9 mK, *the Néel temperature of solid* ^{3}*He. After an open workshop in Leiden in 1998, discussions took place to see if thermodynamic calculation of* ^{3}*He melting pressures could resolve the differences. In January 2000 the authors (apart from ALR and GS) met at NIST and were able to reach a compromise on the Provisional Low Temperature Scale, PLTS-2000. Its 1-sigma uncertainty is estimated to be* 0.3 % *of* T *(up to a maximum of* 0.5 mK*), but this rises to about* 2 % *of* T *at* 0.9 mK. *The provisional status recognizes that the PLTS-2000 is a compromise, rather than a true consensus, but it is likely to be some years before it can be replaced by a more accurate scale. The scale was announced at the Quantum Fluids and Solids conference in Minnesota, USA, in June 2000, and was formally adopted by the Comité International des Poids et Mesures in October 2000.*

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1. INTRODUCTION

The Provisional Low Temperature Scale from 0.9 mK to 1 K, PLTS-2000, is defined by the following equation relating the melting pressure p of ^{3}He to temperature T_{2000}:

$$p/\text{MPa} = \sum_{i=-3}^{+9} a_i (T_{2000}/\text{K})^i$$

$$\begin{aligned}
a_{-3} &= -1.385\,544\,2 \cdot 10^{-12} \\
a_{-2} &= 4.555\,702\,6 \cdot 10^{-9} \\
a_{-1} &= -6.443\,086\,9 \cdot 10^{-6} \\
a_{0} &= 3.446\,743\,4 \cdot 10^{0} \\
a_{1} &= -4.417\,643\,8 \cdot 10^{0} \\
a_{2} &= 1.541\,743\,7 \cdot 10^{1} \\
a_{3} &= -3.578\,985\,3 \cdot 10^{1} \\
a_{4} &= 7.149\,912\,5 \cdot 10^{1} \\
a_{5} &= -1.041\,437\,9 \cdot 10^{2} \\
a_{6} &= 1.051\,853\,8 \cdot 10^{2} \\
a_{7} &= -6.944\,376\,7 \cdot 10^{1} \\
a_{8} &= 2.683\,308\,7 \cdot 10^{1} \\
a_{9} &= -4.587\,570\,9 \cdot 10^{0}
\end{aligned}$$

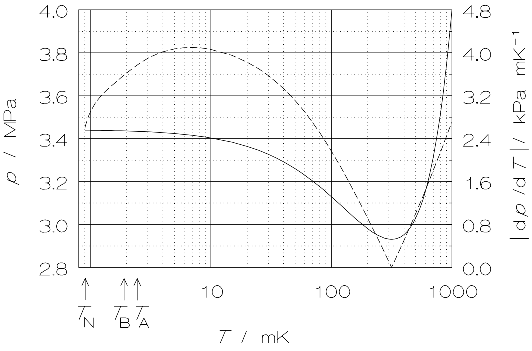

Fig. 1. The ^{3}He melting pressure, p, (full line) and the absolute value of the derivative dp/dT (dashed line) versus temperature. T_N, T_B and T_A indicate the temperatures of three phase transitions in solid or liquid ^{3}He.

The melting pressure of ^{3}He, see Figure 1, was chosen as the property on which the scale should be based because of the sensitivity and reliability with which it may be measured over a wide range, covering more than three decades of temperature, apart from a narrow region around the pressure minimum at 315.24 mK. It is a thermodynamic property with few complications arising from purity, etc, it provides an unambiguous universal definition, and is capable of being realised in laboratories around the world without the exchange of artefacts.

The pressure minimum is one of four natural features which may be located and used as fixed points of pressure and temperature, for the *in situ* calibration of the pressure transducer. The others are the transition to the superfluid 'A' phase, the 'A to B' transition in the superfluid and the Néel transition in the solid, see Figure 1. The pressure and temperature values of these four points on the PLTS-2000 are:

Point	p/MPa	T_{2000}/mK
minimum	2.93113	315.24
A	3.43407	2.444
A-B	3.43609	1.896
Néel	3.43934	0.902

2. BACKGROUND TO THE PLTS-2000

The ^{3}He melting pressure was first proposed for use in thermometry by Scribner and Adams[1]. The first melting pressure scale in general use was that of Halperin et al.[2], who used the properties of ^{3}He and the Clausius-Clapeyron equation to obtain a thermodynamic melting pressure equation.

During the 1980s seminal work by Greywall[3] led to a more accurate empirical equation, based on the NBS (NIST) Cryogenic Temperature Scale, using a SRM768$^{\mathrm{TM}}$ superconductive fixed point device down to 15 mK. He used this to calibrate a lanthanum-diluted CMN thermometer, ensuring also that on the resulting scale the specific heat capacity of liquid ^{3}He (which he had measured), would follow the linear law predicted by the Landau theory. He correlated the temperatures on his scale with measured values of the melting pressure, and so generated a p-T relation extending from 1 mK to 250 mK. The scale was widely adopted, although the differences between it and some previous laboratory scales were up to 10 % or more[3].

Subsequent work at NIST, PTB and at the University of Florida, reviewed below, led to new melting pressure equations, here denoted NIST-92 (from 6.3 mK to 250 mK, later extended to 700 mK as NIST-98), UF-95 (from T_{N} to 250 mK and, like Greywall's scale, linked to the NIST work above 15 mK), and PTB-96, (from T_{N} to 1 K)[4–6]. As Soulen and Fogle[7] pointed out, however, the differences between the PTB equation and those of NIST and UF were still significant. To try to resolve these differences, additional information was sought from thermodynamic analysis of the melting pressure[8]. Before continuing the discussion, we review these scales.

NIST-92/NIST-98 - The NIST scale is based primarily on R-SQUID Johnson noise thermometry from 6.3 mK to 738 mK, as described in Ref. 9. *No* adjustable parameters are used and thus the R-SQUID is a primary instrument. The paper describes the theory of the R-SQUID and how well the experimental results conform to that theory, and the numerous simulation experiments which confirm that the room temperature electronics processes noise signals in the way that theory predicts.

The noise thermometry scale was checked against an *independent* magnetic temperature scale employing CMN which had been calibrated against the ITS-90 from 1.25 K to 3.0 K [10]. The difference between the noise and magnetic scales varied from ~0.3 % at 100 mK to less than 0.2 % at 700 mK. In January 2000, an error (see below) in the noise temperature calculations was discovered, the result of which was to lower all temperature values by 0.15 %. Thus, the differences between the noise and magnetic temperatures is ~0.15 % at 100 mK and 0.05 % at 700 mK, which is within the experimental uncertainties in this region.

To test the accuracy of the noise thermometer scale at much lower temperatures, the R-SQUID noise thermometer was compared *directly* with another independent primary standard, a ^{60}Co nuclear orientation thermometer in the region 7 mK to 22 mK. After taking into account the 0.15 % adjustment of the noise temperatures, the nuclear orientation temperatures were about 0.5 % higher than the noise temperatures.

The linearity of the noise thermometer scale was tested by examining the temperature dependence of the susceptibility of both CMN and LCMN. Assuming the susceptibility follows a Curie-Weiss law, the magnetic temperatures obtained from the fits to the data differed from the measured noise temperatures to a degree (< 0.1 %) that was explained wholly on the basis of the statistical uncertainty in the noise temperatures. Thus, any non-linearity in the noise scale cannot exceed ~0.1 %.

The ^{3}He pressure scale is based on a piston gauge that was calibrated at NIST using helium gas with the gauge operating in the absolute mode. The stated uncertainty in the effective area of the gauge is 15 parts in 10^6 ($3\ \sigma$) and this is the dominant uncertainty in the NIST pressure scale. A melting pressure equation was published for the range from 6.3 mK to 250 mK [4], and later over the complete range 6.3 mK to 700 mK [11].

UF-95 - The University of Florida scale uses a ^{60}Co nuclear orientation (NO) primary thermometer and Pt NMR, calibrated against the NO thermometer, as a secondary thermometer[5]. Self-heating of 3 nW by radioactivity of the 5 μCi NO thermometer caused an offset between it and the platform which held the melting pressure cell and Pt thermometer. Using the functional relationship between the NO temperature T_γ and that of the platform T, and the relationship between the Pt NMR signal and T, explicit account was taken of the self heating to give $T(T_\gamma)$.

Gamma rays from the NO thermometer were counted using a liquid nitrogen cooled germanium detector at the $\theta = 0$ position. Pressures and temperatures between T_{N} and 25 mK were taken using both the NO and Pt thermometers. For each temperature measurement, γ-ray counts of the two well-resolved peaks lasted for eight hours, during which the temperature was held constant to 5 μK by monitoring the melting pressure. The temperature was determined independently from each peak, with the average used for T. This procedure was repeated for 63 pressure and temperature data points between T_{N} and 25 mK. Then the system was warmed to 210 mK, where the anisotropy was zero, for the γ-ray warm-count lasting for 20 hours.

The Pt thermometer, which was used below 7 mK, consisted of a brush of 200 wires of 5N-purity 25 μm in diameter welded to a Pt tab. A pulsed spectrometer, with a long-term stability of 0.1 %, operated at 3.427 MHz with tipping pulses of a few degrees. To avoid the problem of offset in diode detectors, the Free Induction Decay was digitized directly at 50 ns, with the time integral calculated by a computer. The background signal, determined at high T, was negligible. Scatter in individual signals was about 0.5 %, with the precision increased to 0.2 % by time averaging many signals taken during the eight-hour γ-ray counts. Since temperatures in the $p(T)$ relation were determined by a fit involving many points, the accuracy of temperatures $\Delta T/T$ is estimated to be 1 % or better.

Melting pressures were measured with a Straty-Adams capacitive pressure transducer constructed of coin silver, with a pure silver sintered powder heat exchanger[12]. A quartz pressure transducer with a stated accuracy of 0.04 % was used to calibrate the capacitive pressure gauge. The accuracy in pressure scale was improved by making a small adjustment to the value of $p_{\rm min}$ so as to match that of Fogle et al.[4]. The NIST and UF data were then in good agreement in the region of overlap from 15 mK to 25 mK.

PTB-96 - The PTB-96 scale is based on both magnetic and noise thermometry. The work began with the extrapolation of the $T_{\rm X1}$ scale[13] from above 1.2 K using a single-crystal CMN thermometer[14]. The extrapolated temperatures showed a deviation from the $T_{\rm X1}$ scale below 1.2 K that reached the order of 1 mK around 0.5 K. The CMN measurements were made down to about 40 mK, with an additional measurement at 23 mK, taken after a 16 hour equilibration period.

In the temperature range from 50 mK down to the magnetic phase transition of solid ^{3}He at about 0.9 mK pulsed Pt-NMR measurements[14] were performed. The permanent magnetic field of 28.5 mT was of high homogeneity resulting in measured spin-spin relaxation times of roughly 1 ms. Care was taken to optimise the NMR measuring technique by evaluating and correcting the free induction decay signal for background noise contributions and temperature dependent influence of electronic magnetic impurities present in the Pt sample.

A Josephson junction noise thermometer[15] was used to determine noise temperatures in the range from 1 K down to below 1 mK, although heating effects gave rise to errors in temperatures below 2.5 mK. Integration times were chosen such that statistical uncertainties (1 σ) remained below 30 μK at 50 mK, 150 μK at 315 mK and 200 μK at 736 mK. The different temperature dependence of the magnetic thermometry (signal proportional to $1/T$) and the noise thermometry (signal proportional to T) combined with the wide overlap range of temperature measurements allowed a check to be made of the value of the Weiss constant (taken to be 0.23 mK) for the CMN single crystal, and to determine the parasitic noise offset in the noise thermometer. This was found to be approximately 0.1 mK in each run, independent of temperature.

The ^{3}He melting pressure was determined using a sensor calibrated with a pressure balance traceable to the national pressure standard. At the melting pressure minimum, near 2.93 MPa, the standard uncertainty was estimated to be 40 Pa. The relative standard uncertainty of the PTB-96 scale is estimated to be 0.04 % at 1 K, and to increase to 1 % at 1 mK. Further details of the experiments are given in Ref. 6 (and to be published).

Thermodynamic Analysis - The thermodynamic analysis involved the calculation of melting pressures using the Clausius-Clapeyron equation with values for the entropies (derived from measured heat capacities) and molar volumes of the liquid and solid phases along the melting curve. The calculations were used, in particular, for obtaining more information on the shape of the melting curve at the lowest temperatures, where the largest relative differences between the laboratory scales occur.

Specifically, calculations were made for the range 25 mK to 1 mK. Values of $(\mathrm{d}p/\mathrm{d}T)_T - (\mathrm{d}p/\mathrm{d}T)_{25\ \mathrm{mK}}$ were calculated using liquid and solid heat capacities measured by Greywall[16,3], and Greywall and Busch[17] in this temperature range, and a constant value for the difference in molar volumes between the two phases.

After choosing values for p and $(\mathrm{d}p/\mathrm{d}T)$ at 25 mK, using the PTB or NIST melting pressure equation, 'thermodynamic extrapolations' of these equations for the range 25 mK to 1 mK were obtained. Results[8,18] show that above 6.3 mK, the lower limit of the NIST equation, calculated extrapolations are in good agreement with either the NIST or the PTB equations. Below 6 mK, however, the shape of the calculated curve differs from that of the PTB equation.

Similar calculations for the range above 25 mK, using the temperature at the minimum as a reference, also show good thermodynamic consistency for both the PTB and NIST equations.

3. DERIVATION OF THE PLTS-2000

As Soulen and Fogle[7] emphasise in their 1997 review, the comparison of ^{3}He melting pressure scales and equations is complicated because they contain uncertainties and possible errors in both temperature and pressure. However, at the feature temperatures, p and T values can both be compared independently, knowing that they relate to the same thermodynamic state, and the pressure scales can be normalised at these points. Soulen and Fogle used the pressure minimum although, as Greywall pointed out[19], at the lowest temperatures (below the range of the NIST scale) the pressures are best normalised using p_{A} (or, by extension, p_{N}). Soulen and Fogle concluded that, while at temperatures above the minimum the NIST and PTB scales were in reasonable agreement (to better than 0.3 % of T), at the low temperature features the differences between PTB-96 and UF-95 diverged to reach 5 % at T_{N}.

In 1998 a workshop was held at the University of Leiden, attended by 32 representatives of national laboratories, research institutes and industry[11]. The meeting did not succeed in resolving the differences, but it did affirm the need to introduce a scale, even if only on a provisional basis, and it led to renewed efforts to investigate the sources of the discrepancies, notably by the application of thermodynamics to the melting pressure-temperature relation, as discussed above.

In January 2000 the authors (except ALR and GS) met at NIST to derive a compromise scale which could be proposed for adoption by the Comité International des Poids et Mesures (CIPM) later that year. Among topics discussed were the effect of self-heating in nuclear orientation thermometry, the accuracy and modelling of the free-induction decay in platinum NMR thermometry, the magnitudes of possible offsets in noise thermometry, and the accuracy of extrapolated magnetic thermometry scales, all in the context of understanding and reducing the differences between the laboratory melting pressure scales. These were analysed and compared with each other and with thermodynamic calculation, to see how the differences were affected by making reasonable adjustments to the pressure and temperature values.

Four factors enabled agreement on the PLTS-2000 to be reached following the NIST meeting. The first was that in preparation for the meeting, Soulen and Fogle (to be published) revised the model for their R-SQUID noise thermometer[9], including terms which had been considered small. They concluded that a multiplicative correction $(1 + \alpha)$ should be applied to their measured temperatures to obtain T, where α is $-$ 0.0015. Thus the agreement with PTB-96 over the range of overlap became better than 0.15 % of T. Secondly, PTB agreed to accept a scale which departed from their magnetic thermometry, and more closely followed the noise thermometry. Thus the temperatures are about 0.08 mK higher than PTB-96 in the range from 25 mK down to 15 mK, with smaller differences at lower temperatures. Thirdly, UF accepted that the PLTS would, through a procedure of averaging between recent data, give temperature values for the ^{3}He features below those of UF-95. Finally, on this basis, and following their thermodynamic calculations, Durieux and Reesink were able to propose a solution which was acceptable to all parties.

The derivation of the scale is summarised as follows:-

1. The pressure scales of NIST and PTB were normalised so that they agree at the pressure minimum, p_{min}, taken to be 2.93113 MPa. There being

no reason to prefer one over the other, they were averaged, an amount $(p/p_{\mathrm{min}})\Delta p$ being added to all PTB pressure values and subtracted from all NIST pressure values, where $\Delta p = 55$ Pa.

2. In addition to the adjustment of the pressures, the NIST-98 temperatures were reduced by 0.15 % (see above).
3. In the range 1 K $> T_{2000} >$ 315.24 mK (the temperature at the pressure minimum), the PTB-96 equation is used (pressures adjusted as in 1).
4. In the range 315.24 mK $> T_{2000} >$ 25 mK, the PTB-96 equation is used (pressures adjusted as in 1), and with the first derivative decreased (absolute value increased) by a small amount (< 2 Pa/mK), so that the PLTS-2000 coincides with the revised NIST-98 temperatures at 25 mK.
5. In the range 25 mK $> T_{2000} >$ 0.902 mK the PLTS-2000 is based on an interpolation between adopted (p, T_{2000}) values at 25 mK, 6 mK and at the A, A-B and Néel transitions in ^{3}He.

   At 25 mK and at 6 mK the revised NIST-98 values are adopted. To obtain values at the A, A-B and Néel transitions, the PTB and UF scales were normalised to the revised NIST scale by adding the amount $(T/6\,\mathrm{mK})\Delta T$, where ΔT is 0.066 mK for PTB-96 and − 0.136 mK for UF-95, being the differences from the revised NIST-98 scale at 6 mK. Physically this corresponds to changing the Curie constant in the Pt-NMR thermometry below 6 mK by + 1.1 % for PTB-96 and − 2.3 % for UF-95. These changes are close to the estimated uncertainties in the scales. The remaining differences were then sufficiently small that the average temperatures could be adopted in the PLTS-2000.

   The pressure values for the A, A-B and Néel transitions are those of PTB (after adjustment as in 1 above), as these carry lower uncertainties.
6. Pairs of (p, T_{2000}) values were generated and fitted over the complete range by an equation expressing p as a power series in T_{2000}, as was used in PTB-96. The coefficients and the values of pressure and temperature at the features are as given in the Introduction.

In conclusion, at the lowest temperatures the PLTS-2000 is close to a thermodynamic extrapolation of the revised NIST-98 scale, passing between the UF and PTB feature values with differences of about ± 3 % at T_{N}. The differences between the various laboratory scales and the PLTS-2000 are shown in Figures 2a and 2b. Figure 2b includes a thermodynamically-calculated scale chosen to be an overall best fit which deviates from the PLTS-2000 by no more than 0.02 mK. Similar calculations have been used to investigate the self-consistency of some other scales shown in the figure[8,18]. At 1 K the PLTS-2000 is expected to be in agreement with the International Temperature Scale of 1990, ITS-90, within 0.3 mK.

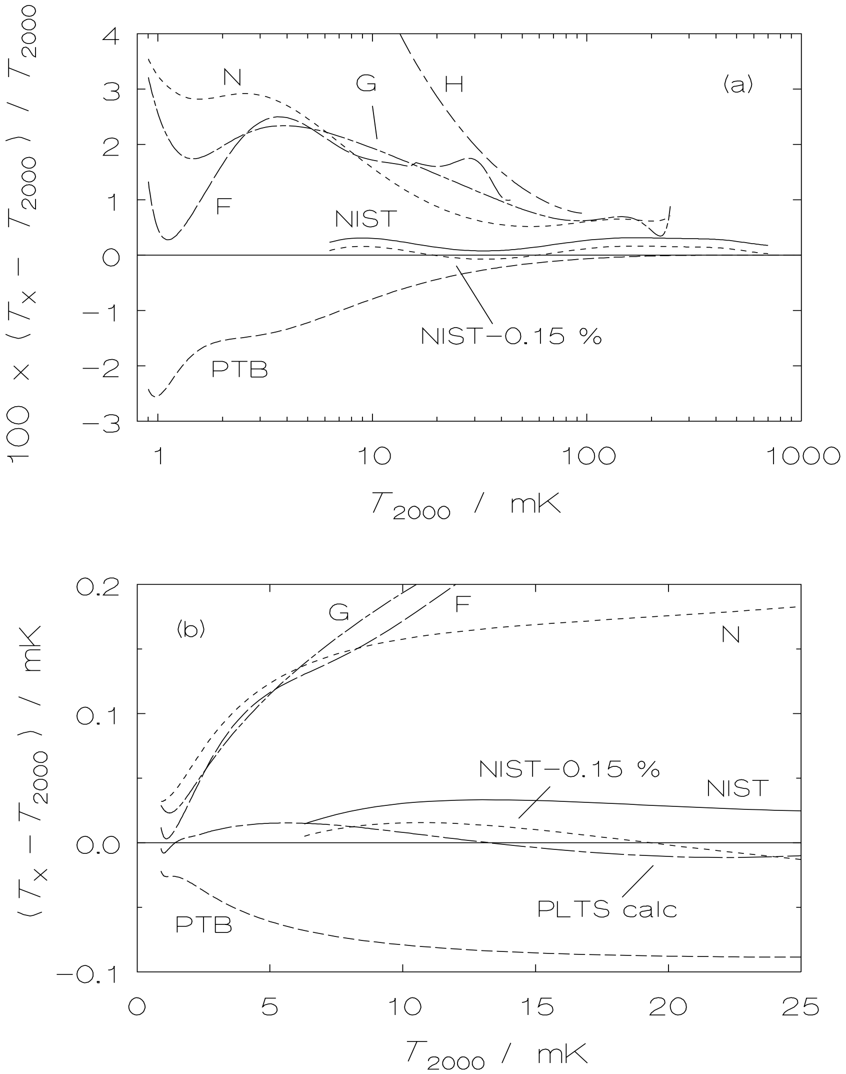

Fig. 2. a) Relative differences between laboratory scales and the PLTS-2000 as a function of temperature. H: Halperin[2], G: Greywall[3], F: Fukuyama et al.[20], NIST: NIST-98 [4] before and after reduction by 0.15 % of T (see text), N: UF-95 [5], PTB: PTB-96 [6], all scales being defined by melting pressure equations. b) Absolute differences below 25 mK, including a comparison with a thermodynamic calculation, ‘PLTS calc’ (see text).

The standard uncertainty of the PLTS-2000 in thermodynamic terms is estimated from an assumed rectangular probability distribution overlapping the scale differences and uncertainties, to be 0.5 mK down to 500 mK, decreasing linearly to 0.2 mK at 100 mK. It decreases further with falling temperature, but in percentage terms it increases to about 0.3 % of T at 25 mK and about 2 % of T at 0.9 mK. The standard uncertainty in the absolute pressures is estimated to be about 60 Pa (in about 3 MPa).

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