TRACEABILITY TO THE PLTS-2000: CONCLUSION OF THE EU PROJECT 'ULT DISSEMINATION'

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ABSTRACT

The EU-sponsored project 'ULT Dissemination' started in January 2000 with the objective of developing and evaluating methods and devices which could enable measurements of temperature below 1 K to be reliable and traceable to an extension of the ITS-90 based on the melting pressure of ^{3}He. During the first year of the project the extension was adopted by the Comité International des Poids et Mesures as the Provisional Low Temperature Scale from 0.9 mK to 1 K, PLTS-2000.

The project involved 11 partners, from National Measurement Institutes, universities and industries in 5 European countries. The devices range from the primary or near-primary methods, such as a Coulomb blockade thermometer, a current-sensing noise thermometer, NMR thermometers for Pt and ^{3}He, and a cerium magnesium nitrate magnetic thermometer, for which only limited calibration is necessary, to second sound thermometers where a more complicated model is needed, and to secondary resistive thermometers where detailed calibration data must be obtained. In addition, a superconductive reference device, SRD1000, has been produced to generate a series of 10 reference points between 15 mK and 1180 mK, and a self-contained melting pressure thermometer (MPT) has been developed. The project was concluded in December 2003, and this paper summarises the main results.

1. INTRODUCTION

The Provisional Low Temperature Scale from 0.9 mK to 1 K, PLTS-2000, is defined by a specified equation for the melting pressure of ^{3}He [1]. This is sensitive, except near the minimum, and the specified substance, ^{3}He, is well-defined and widely available. A realisation of the PLTS-2000 requires the provision of a cell of ^{3}He at high pressure (2.9 MPa to 4 MPa for the complete range) in which the liquid-solid interface is established at equilibrium, and the measurement of the (absolute) pressure. The requirements are demanding but the calibration can be simplified by using the in-built fixed points of ^{3}He, and practical guidance has been published [2, 3].

For most ULT experimentation, however, the direct realisation of the PLTS-2000 will be impractical and some other methods must be employed. The aim of the EU-sponsored project 'ULT Dissemination' has been to develop and evaluate some of the devices which can provide relatively simple but reliable and traceable thermometry. The project has now finished, and a description of the devices and outcomes are briefly reviewed.

2. DEVICES

The **Coulomb Blockade Thermometer**, CBT, is based on the electrical conductance characteristics of non-superconducting tunnel junctions. It consists of an array of small metallic islands separated by tunnel junctions from the measuring electrodes. The conductance of such a device is governed by the charging energy for individual electrons in the island, by the thermal energy $k_B T$ and by the bias eV at voltage V. The half-width of the conductance dip at low bias voltage is directly proportional to T, and the measurement provides temperature in a primary way, without calibration, using essentially a resistance measurement. Practical CBTs consist of parallel arrays of many junctions in series. The sensors can cover wide temperature ranges, depending on the fabrication parameters chosen, and since the effect originates from the energetics of virtually static charges, the thermometer is highly tolerant to even strong magnetic fields.

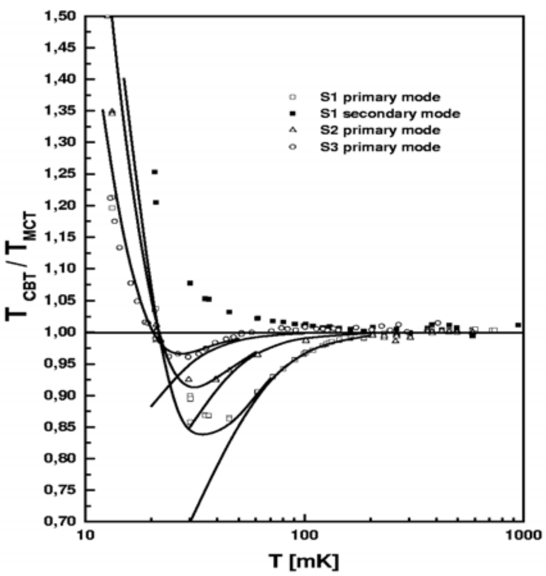

Figure 1: Calibration of three CBT sensors, S1, S2 and S3, against the melting-curve thermometer, with theoretical curves.

The device was pioneered at the University of Jyväskylä, and experimental evaluation of the CBT has been carried out at several institutes. Figure 1 shows data from CRTBT for the ratio of the CBT and MCT (Melting Curve Thermometer) temperatures for three devices, S1, S2 and S3. All of the sensors are consistent with the MCT above 200 mK within about 1 % at worst. The deviations at lower temperatures are due to lack of thermalisation which, however, is much reduced for S3 which has the largest island size. In this case the effect is about -3 % near 25 mK. Further analysis of the thermalisation has been given in [4].

Considerable progress has been made with the CBT in the project and, drawing on this experience, an improved version of the commercial CBT for ULT use is currently being tested by Nanoway Oy, Finland, using a sensor with a larger array of junctions and improved electronics.

The **current sensing noise thermometer** (CSNT) [5] uses a low T_c DC SQUID to measure the thermal noise currents in a sensor resistor R. The mean square noise current flowing in the SQUID input coil per unit bandwidth, arising from thermal noise in the resistor, is given by

$$\left\langle I_{\mathrm{N}}^{2} \right\rangle = \frac{4k_{\mathrm{B}}T}{R}\left(\frac{1}{1+\omega^{2}\tau^{2}}\right), \tag{1}$$

where k_B is Boltzmann's constant and $\omega = 2\pi f$. The time constant $\tau = L_T/R$, where $L_T = L_i + L_s$. Here L_i is the input coil inductance of the SQUID and L_s is any additional inductance in the super-conducting input connections. The frequency spectrum of the output noise is fit to equation (1) in order to extract τ and T. Recent work has involved the construction of a new sensor with much improved shielding to eliminate spurious noise. The copper foil resistor was 0.202 mΩ ± 0.2 % at 4.2 K, and the SQUID gain was measured to better than 0.15 %, hence allowing the CSNT to be used in absolute mode, without calibration. Figure 2 shows a preliminary comparison between the CSNT and a melting pressure thermometer down to 1 mK. A small change in the time constant (~ 1% over the entire temperature range) is assumed to be due to a change in sensor resistance, which has been allowed for in extracting the temperature. A more detailed comparison is in progress.

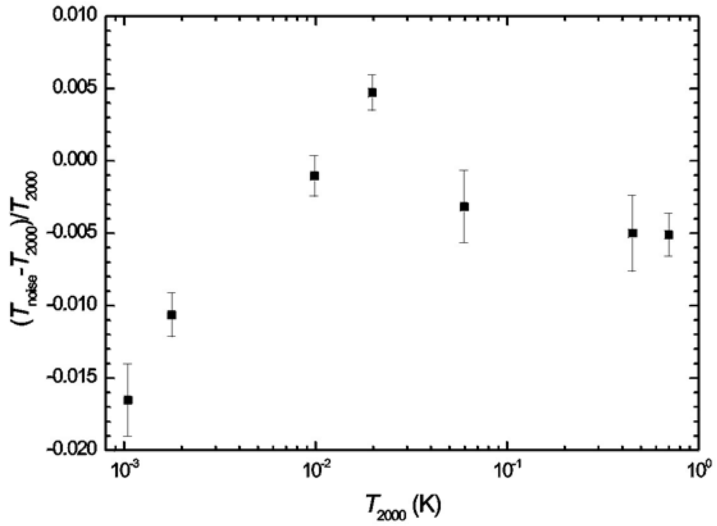

Figure 2 Comparison of the CSNT with a ^{3}He melting pressure thermometer using the PLTS-2000.

The CNRS-CRTBT has evaluated several CBTs and, in collaboration with Air Liquide, it has devised a commercialised **CMN thermometer**. It has also developed **NMR thermometers** for platinum powder and wire, and ^{3}He bulk samples and monolayers. The first NMR spectrometer used a room temperature differential preamplifier, with a radio-frequency bridge to detect the small change of impedance of a NMR tank circuit. The system was tested in measurements of the susceptibility of bulk liquid ^{3}He, a benchmark system with severe problems due to poor spin diffusion at high temperatures and Kapitza resistance at low temperatures. The measurements were made as a function of pressure (0 to 3 MPa) and temperature, down to 5 mK.

Later a second spectrometer was developed, with a low temperature (4.2 K) preamplifier designed at CNRS-CRTBT. The lower noise of this device allows the measurements to be extended up towards 1 K where the signal is small, thus providing an overlap with other thermometers. This system has been successfully tested down to 100 µK on ^{3}He monolayers. Figure 3 shows a comparison of the Pt-wire NMR thermometer with a CMN thermometer, indicating stability and sensitivity of about 1 % below 100 mK. The ^{3}He and Pt NMR thermometers have also been compared down to 4 mK with a melting pressure thermometer calibrated using a Digiquartz pressure gauge normalised at the pressure minimum. The Pt thermometer showed broad lines and was accurate to 1 % above 30 mK, but the ^{3}He thermometer showed agreement within 0.5 %.

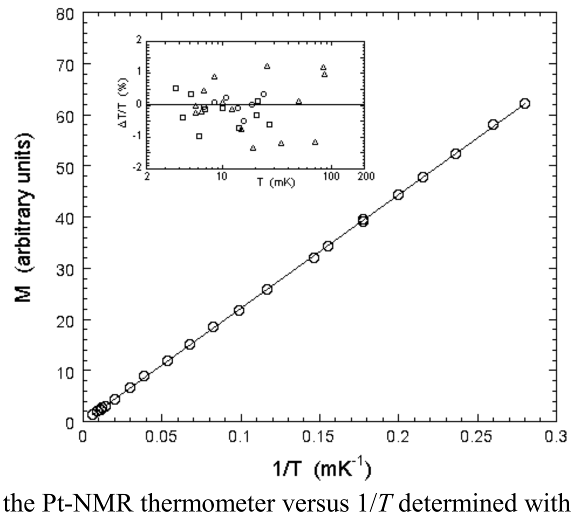

Figure 3: Magnetisation of the Pt-NMR thermometer versus $1/T$ determined with a CMN thermometer

A **second sound thermometer** has been developed at BNM-INM, using the strong temperature dependence of the velocity of the second sound (propagation of the normal density in the superfluid liquid) in ^{3}He-^{4}He mixtures, especially below 0.5 K. The velocity is obtained from the acoustic resonance frequency in a closed cavity. A simple relationship then exists between the resonance frequencies, the dimensions of the cavity and the speed of the sound propagation.

A model has been developed to deduce the temperature from the speed of sound, and the thermometer has been described fully [6]. In the experimental validation, measurements were made at 58 temperatures in the range from 20 mK to 780 mK. Agreement between the device and a melting-pressure thermometer was within ± 0.1 mK below 700 mK for 90% of the data.

A variety of **semiconductive sensors**, such as carbon, germanium, ruthenium oxide and 'Cernox' are commonly used as secondary resistance thermometers at low temperatures, including the range below 1 K. One difficulty is the rapid increase in thermal boundary resistances which leads to overheating of the sensor and ultimately complete loss of contact with the rest of the experiment. Moreover, the heat transfer is often strongly influenced by conduction through the connecting leads rather than directly to the capsule or substrate. Therefore the leads must also be thermally anchored to the experiment.

The thermal conditions for dissipative sensors has been thoroughly investigated theoretically and experimentally at PTB, using two different types of ruthenium oxide sensor, one in a helium-filled copper capsule, the other on a substrate mounted on a copper holder. In the experiments, the temperature of the thermal anchor point could be varied, and the effect of the resulting heat influx on the sensor studied. The thermal resistances could be identified, modelled and estimated. For sensors in a helium-filled capsule in particular, the internal thermal resistances should be carefully checked. Figure 4 shows the temperature dependence of a ruthenium oxide sensor on a substrate, with and without power applied to the anchor. The decoupling effect is clearly seen as the temperature is reduced.

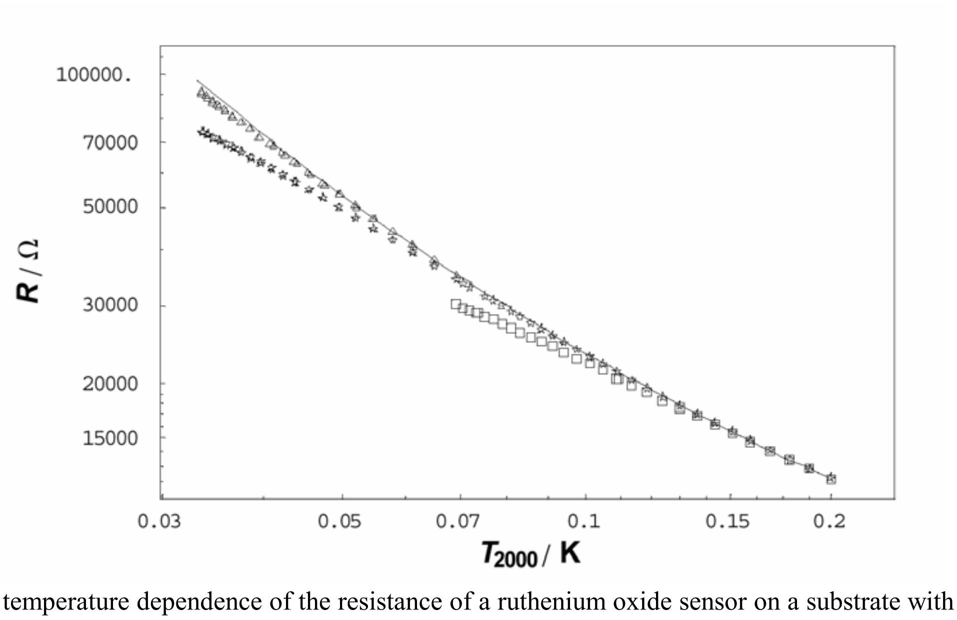

Figure 4: temperature dependence of the resistance of a ruthenium oxide sensor on a substrate with different heating power applied to the anchoring point, showing progressively larger overheating of the sensor. The thin line, triangles, stars and squares are for powers of 0 nW, 5 nW, 45 nW and 500 nW, respectively.

The **self-contained melting pressure thermometer,** developed in collaboration with Oxford Instruments Superconductivity, is based on a cylindrical capacitive sensor which contains the ^{3}He sample in an annular space [7]. The capacitance forms part of a cold tunnel diode oscillator circuit, the frequency of which is directly related to the ^{3}He pressure. The cell is connected to a relatively simple gas-handling system, with a cryo-sorption pump to generate the high pressures required, and to a pressure gauge. The pressure is approximately linear with inverse capacitance and the deviation from linearity is shown in Figure 5. One of the ^{3}He fixed points is used to check the pressure gauge and offset the effects of the pressure head of liquid in the sensing line. In contrast to the calibration curve, the non-linearity is reproducible within about 500 Pa (typically 0.1 to 0.3 mK), therefore only a few calibration points are required after cycling to room temperature.

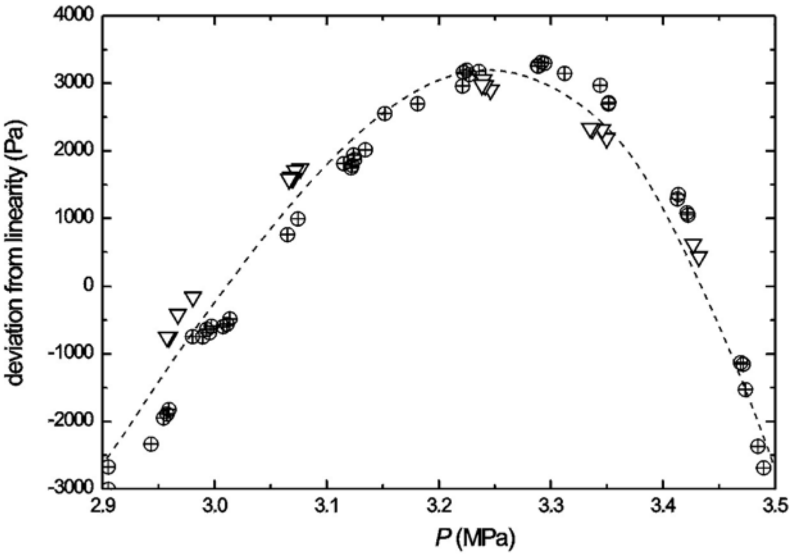

Figure 5: Non-linearity and reproducibility of the pressure transducer. Circles and triangles are from different cool-downs.

Four Dutch partners led by HDL have undertaken the development of a **superconductive reference device**, SRD1000 [8]. This is designed to replace the SRM 768 and (partly) the SRM 767, formerly made by NBS. The device contains 10 small samples, ranging from tungsten (Tc ~ 15 mK) to aluminium (Tc ~ 1180 mK), mounted on a gold-plated copper support structure.

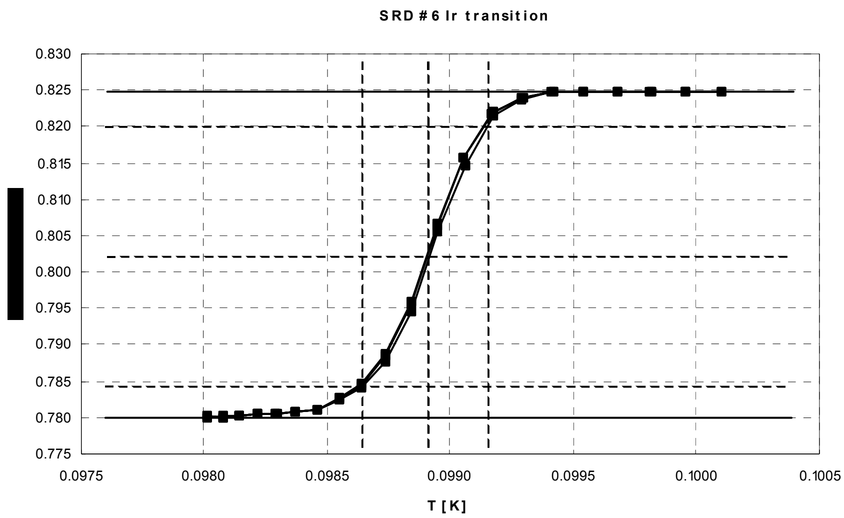

Figure 6: Superconductive transition for the Ir sample in SRD006, obtained at NPL. The mid-point is at 98.91 mK and the width is 0.52 mK.

The samples are protected from local magnetic fields by an outer Cryoperm shield and an inner niobium shield. A field compensation coil is included to permit tests of the sensitivity of the samples to small fields and of the effectiveness of the screening. Detection is achieved using a novel planar micro-coil mutual inductance system and dedicated measurement electronics are provided. The mid-point of the transition is specified to be the point half way between the SRD outputs in the fully superconductive and fully normal states, and the width is the temperature interval between 10% and 90% of the change in output (*ie* in mutual inductance).

Prototype devices have been evaluated, first at NMi VSL and later by five other partners [9]. Figure 6 shows an example of a transition, for the Ir sample in SRD006, obtained at NPL.

3. CONCLUSION

A variety of devices for establishing traceable thermometry at ultra-low temperatures have been developed and evaluated. Further work is needed in some cases to enhance the performance or practicality, but there are good prospects for improved temperature measurement in this region.

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