# European Dissemination of the Ultra-low Temperature Scale, PLTS-2000

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**Abstract.** The first phase of the EU collaborative project on sub-kelvin thermometry, 'ULT Dissemination', is nearing completion, leading to the development of several thermometers and devices, and the instrumentation needed to disseminate the new Provisional Low Temperature Scale, PLTS-2000, to users. Principal among these are a current-sensing noise thermometer (CSNT), a CMN thermometer adapted for industrial use, a Coulomb blockade thermometer, a second-sound acoustic thermometer and a superconductive reference device SRD-1000. Several partners have set up $^3$He melting-pressure thermometers to realise the PLTS-2000, and will check it using Pt-NMR, CMN and other thermometers. The scale, which was formally adopted by the Comité International des Poids et Mesures in October 2000, covers the range of temperature from 1 K down to 0.9 mK, and is defined by an equation for the melting pressure of $^3$He. The SRD employs novel fabrication and detection techniques with up to 10 samples, and is expected to meet the requirement for fixed points below 1 K, formerly filled by the NIST SRM 767 and 768. Other devices included in the project are ruthenium oxide sensors and a self-contained $^3$He melting pressure thermometer. This paper reviews the project progress to date and indicates the potential for research, metrological and industrial application of the devices developed.

## INTRODUCTION

The Provisional Low Temperature Scale from 0.9 mK to 1 K, PLTS-2000, was adopted by the Comité International des Poids et Mesures in October 2000, in order to provide the basis for reliable temperature measurement over the wide range in which commercial dilution refrigerators operate. It is defined in terms of an equation for the melting pressure of $^3$He and extends down to the superfluid and Néel feature temperatures. A brief description of the PLTS-2000 is given elsewhere in these proceedings [1], and a fuller account has been published [2]. Following its introduction there is a need for primary and secondary thermometers and fixed points for investigating the scale and disseminating it to users. The European project 'ULT Dissemination' was started in January 2000 to develop several devices with their associated instrumentation. These include noise, acoustic, Coulomb blockade, CMN and NMR thermometers, a superconductive reference device, and a self-contained $^3He$ melting pressure thermometer. Some ruthenium oxide resistive sensors are also being investigated. The paper is an up-date of a report published earlier [3].

# THERMOMETERS AND DEVICES

A brief account of the thermometers and devices under development now follows.

## Current Sensing Noise Thermometer

A Current Sensing Noise Thermometer (CSNT) is under development at Royal Holloway University of London (RHUL), in which a DC SQUID is used to measure the thermal noise current in a resistor and the temperature is obtained using the Nyquist formula. The thermometer is fast and can be used over the temperature range from 4.2 K to below 1 mK. If careful measurements of the value of the resistor and the system gain are made then the thermometer is, in principle, absolute. Alternatively, if the gain is measured at one point the thermometer can be used as a secondary thermometer over the entire temperature range without further calibration. A commercial DC SQUID from Quantum Design is being used as the preamplifier, with a coupled energy sensitivity of 500 *h*, where *h* is Planck's constant. The sensor resistors are cut from copper foil 25 μm thick and of 99.9 % purity. A detailed report of RHUL work on the CSNT to date has been published [4].

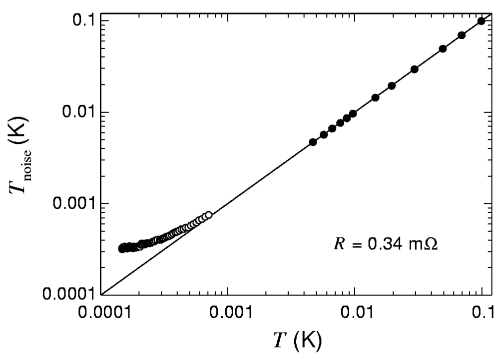


**FIGURE 1.** Temperature obtained from the CNST, $T_{noise}$, versus that obtained from a $^3He$ melting pressure thermometer (full circles) and a platinum NMR thermometer (open circles). The thermometer is usable over more than four orders of magnitude in temperature.

Measurements were made with the copper resistor mounted on a base made of the machinable glass ceramic MACOR. The sensor resistor, of value 0.34 mΩ, was cooled on a nuclear demagnetization cryostat to below 1 mK. The noise thermometer was used as a secondary thermometer, its gain being calibrated at 100 mK against a $^3He$ melting pressure thermometer using the PLTS-2000. A preliminary comparison was made with the melting pressure thermometer down to 4.7 mK. In addition, the CSNT was compared with a platinum NMR thermometer down to below 1 mK, achieving a minimum electron temperature in the noise thermometer of 300 μK.

The data above 4.5 mK were taken with the heat switch to the stage closed and mixing chamber held at constant temperature with a temperature controller. They agree with the PLTS-2000 to within 1 %. The data below 1 mK were taken whilst slowly warming in a field of 45.5 mT following a demagnetization. At temperatures above 1 mK the warming rate was too fast to ensure thermal equilibrium. It is clear that at the lowest temperatures the CSNT reads hotter than the platinum. The data follow the functional form $T_{noise}^2 - T^2$ = const, as expected for a temperature independent heat leak to the sensor, which is being cooled via a pressed metallic contact. If this expression remains valid to higher temperatures then the present CSNT would read 40 μK above the true temperature at 1 mK and 8 μK hotter at 5 mK. More work is required in the region between 1 mK and 5 mK, demagnetizing to higher final fields in order to increase the heat capacity of the nuclear stage.

Using this thermometer a measuring time of 200 s was necessary to obtain 1.5 % precision in temperature (independent of the temperature), and the measured amplifier noise temperature $T_N$ (the temperature at which the resistor and SQUID contribute equally to the zero frequency noise power) was 8 μK. The speed of the thermometer can be increased, at the expense of an increased $T_N$, by choosing a larger value sensor resistor. For example, for use with a dilution refrigerator of base temperature 5 mK, a sensor resistance of 5 mΩ would have $T_N$ ~30 μK and a precision of 1 % should be obtainable in around 10 s. This is quite reasonable for a practical general-purpose thermometer.

## Coulomb Blockade Thermometer

A Coulomb blockade thermometer, CBT, which is under development at the University of Jyväskylä, uses the non-linear current-voltage characteristics of an array of tunnel junctions [5]. For *N* junctions the halfwidth of the dip in conductance is proportional to $Nk_{\mathrm{B}}T/e$. A number of Coulomb blockade sensors have been constructed with optimum parameters, and laboratory tests down to 80 mK have shown satisfactory results. Coulomb blockade thermometers (CBTs) are being developed for use down to 10 mK.

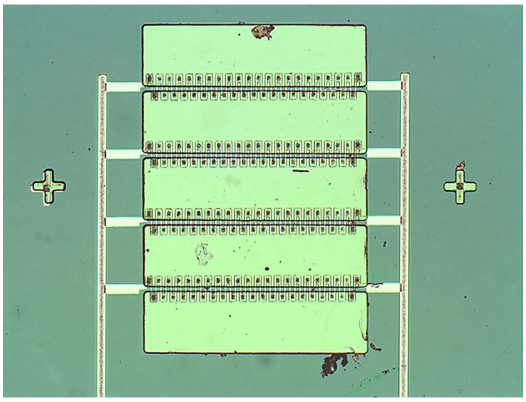

**FIGURE 2.** A prototype CBT sensor with improved properties. Four junction arrays with aluminium electrodes run horizontally between the busbars about 500 µm apart. This sensor was fabricated using thermostable PES resist. On top of the sensor array there is a 200 nm thick silicon monoxide layer to insulate the magnet film which was deposited at the end. The ferromagnetic film is the structure in the figure with five rectangles around the junction arrays. There is a narrow gap between each section to create a strong magnetic field on the aluminium to suppress superconductivity. The thickness of the magnetised cobalt film is 150 nm.

The thermalisation and the stability of the sensors have been improved by better lithographical methods of fabrication. The two alternative paths to this end have been (a) to use silicon nitride mechanical masks for resist-free and more contamination-free patterning. Partial success was achieved, but there was a problem with the mechanical stability of a silicon nitride mask, when complicated patterns are exposed. A better solution (b) turned out to be the use of heat resistant resist (PES, polyphenylen-ether-sulphone), which allows contamination-free patterning combined with the large structures needed in this project. All the prototype sensors are now fabricated using PES-resist.

Thermalisation of the thermometer, in particular electrons in the thermometer, is still a subject of development. Recent tests at CNRS/CRTBT show that the area of the metallic islands in the CBT sensor improve thermalisation, but to reach absolute accuracy of a few percent at $T < 50$ mK requires one more step of redesign of the island geometry and film thicknesses. The projected accuracies are < 0.5 % between 4.2 K and 0.1 K, about 2 % down to 30 mK and 5 % below this. The devices have high immunity to magnetic fields, although to date this has only been demonstrated down to 50 mK [6].

Devices of this specification will be valuable and convenient sensors for thermometry associated with dilution refrigerators.

## Pt and $^3$He NMR thermometers

Platinum powder and $^3$He NMR thermometers have been manufactured and tested at CNRS-CRTBT. The electronics (including low temperature pre-amplifiers) have been built and tested, and measurements using this cwNMR system have been successfully performed with both Pt and $^3$He samples. The high sensitivity, very low power, spectrometer consists of a room temperature differential preamplifier, and a radio-frequency bridge detection of the small change of impedance of a NMR tank circuit (Q-meter configuration). A computer is used to control the magnetic field and the data acquisition.

The system has been tested by conducting a detailed series of measurements of the susceptibility of bulk liquid $^3$He, a benchmark system where severe measuring problems are encountered 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). This thermodynamic property is in principle very well known, following the work of Ramm *et al* [7], whose tabulated data are widely used. However, a substantial discrepancy (about 7 %, depending on pressure and temperature) was found, which therefore opens an interesting debate on the Fermi liquid parameters of liquid $^3$He, with consequences well beyond their initial goal, which was testing the quality of the cw-NMR spectrometer in a very demanding situation. The results will be presented at the International Conference on Low Temperature Physics (Hiroshima, August 2002).

Recently a second spectrometer has been developed, which incorporates a low temperature (4.2 K) preamplifier designed at CNRS-CRTBT. The lower noise of this device allows the measurements to be extended towards the high temperature end (i.e. towards 1 K) where the signal is small, thus providing an overlap with other thermometric methods with adequate sensitivity. The system has first been tested at 4.2 K (noise and gain measurements). After some modifications of the circuit, a low noise figure has been achieved. This system has been successfully tested down to 100 µK on $^3$He monolayers.

A comparison of the $^3$He and Pt cw-NMR thermometers has been made with a melting pressure thermometer in terms of the PLTS-2000, see Figure 3. The preliminary measurements show that the system can be used to perform an adequate comparison with the ultra-low temperature scale once the Superconductive Reference Device is available for the calibration.

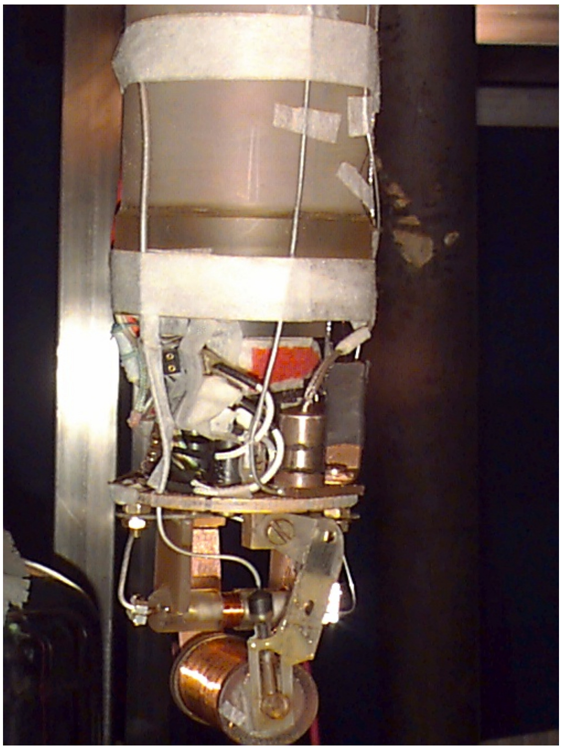

**FIGURE 3**. Comparison of the $^3$He and Pt cw-NMR thermometers (below the copper plate) with the melting pressure thermometer (on the plate). Above is the mixing chamber of the CNRS-CRTBT dilution refrigerator, base-temperature < 4 mK.

## CMN Thermometer

CMN magnetic thermometry constitutes a simple and reliable method in the temperature range down to a few millikelvins. It provides high sensitivity, an excellent stability, it is highly reproducible and the thermal time constant is relatively short, on the order of minutes. For these reasons, the CMN thermometer is presently an essential tool for the test and operation of dilution refrigerators, in particular in the experimental environment of large industrial and research facilities, where electromagnetic interference is significant. A rugged device has been designed and built at CNRS-CRTBT, with the aim of providing good transferability for interpolating a temperature scale. It is currently undergoing trials in the laboratories of Air Liquide.

The main difficulty encountered in the construction of CMN thermometers is the choice of the materials: the salts, the coil formers and wires, supports and shields must be carefully selected and tested. The CMN thermometers described here have been shielded against stray magnetic fields using a superconducting niobium shield. External fields up to 50 mT were applied to the devices without any observable change of the measured values.

The CMN thermometer calibration was performed using a powerful dilution refrigerator equipped with calibrated carbon resistors and a $^3$He melting pressure thermometer (PLTS 2000), see Figure 4. The temperature is determined with a resolution of 10 µK at 50 mK, with an absolute deviation in relation to the reference temperature smaller than 1 %.

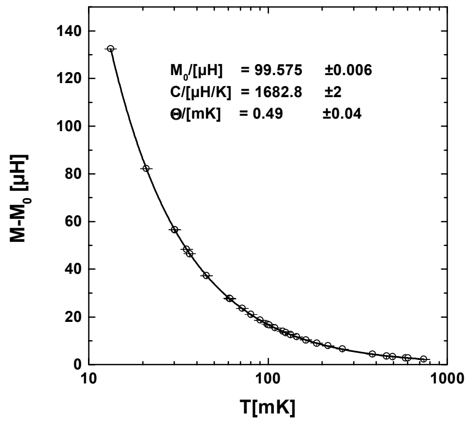


**FIGURE 4**. Calibration of the CMN thermometer.

## Second-Sound Thermometer

This thermometer uses the strong temperature dependence of the velocity of the second sound in $^3$He-$^4$He mixtures (propagation of the normal density in the superfluid liquid), especially below 0.5 K. The velocity is obtained from the acoustic resonance spectra 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 of the speed of sound [8].

The thermometer is described fully elsewhere [9]. The cell is sealed at room temperature with a 1 % mixture of $^3$He in $^4$He under pressure, so that near

4.2 K liquid condenses and fills a cylindrical acoustic resonator chamber. At each end of the chamber, two identical electrostatic transducers are used to mechanically excite and detect the second sound.

The second sound thermometer has been compared with a melting pressure thermometer at 58 temperatures in the range from 20 mK up to 780 mK The results demonstrated the self-consistency of the thermometric system, and more particularly the validity of the model describing $T$ as a function of the speed of the second sound, although divergences arose above 700 mK, see Figure 5 [9].

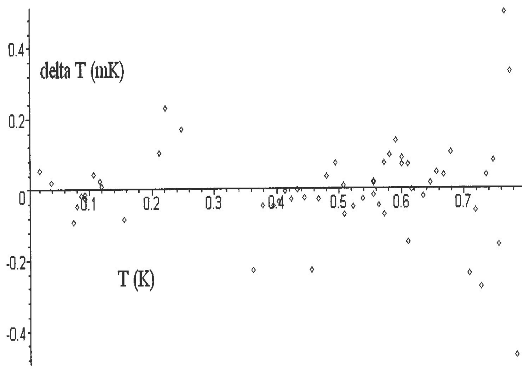


**FIGURE 5.** Validation of the self-consistency of the thermometric system consisting of the second sound thermometer and the melting pressure thermometer. The differences increase above 0.7 K where the acoustic thermometer is less sensitive.

The thermometer is self-contained and is now in use as a laboratory standard at BNM-INM. It will be used to study and calibrate other thermometers and the superconductive reference device, SRD 1000.

## Self-Contained $^3$He Melting Pressure Thermometer

A self-contained $^3$He melting pressure thermometer is being developed at RHUL in collaboration with Oxford Instruments Superconductivity (Professor V Mihkeev). The thermometer is designed to be both easy to construct and simple to operate. It is based on a cylindrical pressure gauge [10], with good linearity of pressure versus inverse capacitance. The readout electronics is based on a tunnel diode oscillator circuit since one of the capacitance plates is necessarily grounded. A low power back diode (BD7) is used as the active element of the oscillator circuit. Evaluation of the thermometer down to 20 mK, by comparison with a current sensing noise thermometer is in progress. The self-contained melting pressure thermometer will allow convenient and direct dissemination of the PLTS-2000.

## Superconductive Reference Device

In addition to using thermometers for disseminating temperature scales and standards, it is useful to have access to fixed points against which the thermometers can be checked or calibrated. There are a number of phase transitions at low temperatures which can in principle be used in the range below 1 K, including those provided by liquid and solid $^3$He, superconductivity and magnetism, although in general they are not first order transitions and there is no heat of transition which can stabilize the temperature. For many years superconductive samples were available in Standard Reference Materials SRM767 and SRM768 from the National Bureau of Standards, now NIST, but these have been discontinued. An important part of the present project has been to develop a replacement device, the Superconductive Reference Device, SRD-1000, using samples with transitions between 15 mK (tungsten) and 1.2 K (aluminium).

The development is described in another paper in these proceedings [11], but an example of a transition in an $Ir_{92}Rh_{08}$ alloy (a material not used in the SRM768 device) is given in Figure 6.

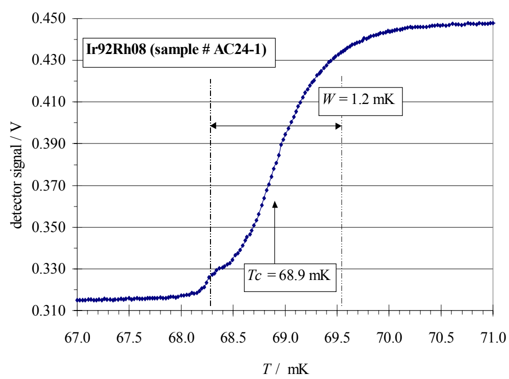


**FIGURE 6**. Superconductive transition in a sample of $Ir_{92}Rh_{08}$ , # AC24-1.

# EVALUATION

In the second phase of the project several partners will evaluate these various thermometers, with a view to establishing their potential for application in ULT experiments. In addition, some ruthenium oxide resistive sensors are under investigation at PTB.

Figure 7 shows a view of the dilution refrigerator at NPL as an example of an experiment in preparation.

The project will run until the end of 2003, and the results will be presented in further publications.

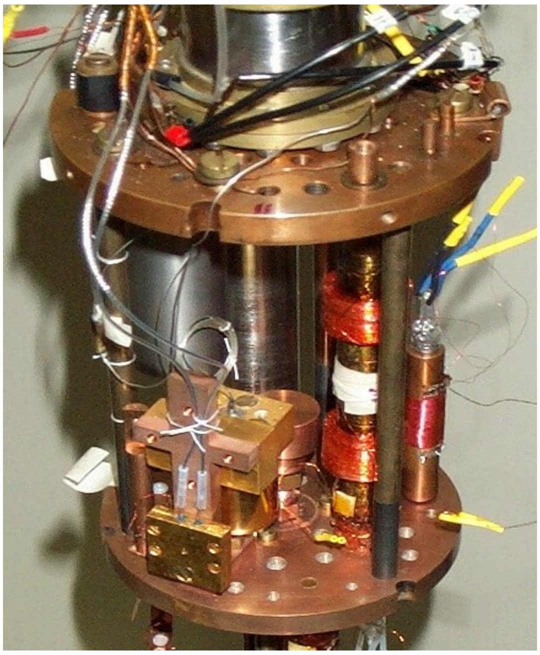

**FIGURE 7**. The thermometer stage of the NPL dilution refrigerator below (and isolated through a heat switch from) the mixing chamber, loaded (from left to right) with a PTB melting pressure thermometer, a SRM768 device (behind), a $^{60}$Co nuclear orientation thermometer (at the centre), a CMN thermometer and a rhodium-iron resistance thermometer. The $PrNi_5$ nuclear coolant is suspended beneath the plate and is not shown.

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