Evaluation of Uncertainty in the Realization of the Provisional Low Temperature Scale from 10 mK to 1 K at NMi

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1 Abstract

The Provisional Low Temperature Scale (PLTS-2000) is realized at NMi between 10 mK and 1 K by operating a ³He melting curve thermometer (MCT). The calibration of the pressure transducer of the MCT, through the use of pressure reference points, is presented. The sources of uncertainties, arising in the process of calibration of the transducer, are described in details and evaluated. The accuracy of the realization of the PLTS-2000 is evaluated. With the exclusion of a narrow range centred around the minimum of the ³He melting curve (T = 315.24 mK), where the sensitivity of the MCT reduces to zero (and the uncertainty diverges), the accuracy obtained is suitable for high-precision sub-Kelvin calibration of secondary thermometers and superconductive reference devices.

2 Introduction

In 2000 a provisional extension of the ITS-90 [1] from 0.9 mK to 1 K (Provisional Low temperature Scale, PLTS-2000 [2]) was adopted by the Comite' International des Poids et Mesures. The melting pressure of ³He was selected as the carrier and the scale was defined by an internationally accepted polynomial $p_m = p_m(T_{2000})$ relating the ³He melting pressure p_m to temperature T_{2000} (see Figure 1).



Figure 1 ³He melting curve and coefficients of the polynomial $p_m = p_m(T_{2000})$ which defines the PLTS-2000.

The practical realization of the PLTS-2000 requires the so-called ³He melting pressure (or curve) thermometer (MCT) to be operated [3]. The MCT is essentially a closed chamber, containing both liquid and solid ³He at equilibrium; an in situ capacitive pressure transducer measures the pressure p_m inside the chamber and the melting pressure is transformed into temperature *T*.

The accuracy of the PLTS-2000 realization depends on the accuracy of the calibration of the pressure transducer. Different options are possible for the calibration of the pressure sensor:

- 1. Use of primary pressure standard (pressure balance).
- 2. Use of calibrated secondary pressure standard (Digiquartz etc).
- 3. Use of intrinsic reference points of the ³He melting curve (the minimum at 315.24 mK, the superfluid phase transitions at 2.444 mK and 1.896 mK and the solid phase transition at 0.902 mK).
- 4. Use of superconductive reference points (calibrated SRM768 [4] and SRD1000 [5] devices, for example).

The highest accuracy can be achieved by using option 1 at cost of considerable workload. Option 3 is very attractive because no external pressure standards or reference points are required but the ³He phase transitions are not accessible with ordinary dilution refrigeration and a demagnetisation cooling stage is needed.

At NMi we adopted a mixed approach: the minimum of the melting curve and a set of superconductive transitions (provided by a calibrated SRM768 device) are used as pressure reference points to calibrate the capacitive pressure transducer of a MCT produced by PTB. In this paper the uncertainty of the calibration procedure is calculated and the final uncertainty in the realization of the PLTS-2000 is evaluated.

3 Pressure reference points

The capacitive pressure transducer is calibrated by using pressure reference points. By pressure reference points we mean physical states occurring at a known melting pressure (or at the known equivalent temperature). At NMi we use as pressure reference points the minimum of ³He melting pressure curve and a set of superconductive transitions provided by the SRM768 device.

The minimum of the ³He melting curve is an intrinsic reference point and its pressure (and temperature) value is fixed by the definition of the PLTS-2000. The superconductive reference points of the SRM768 are regarded as external standards: the superconductive transitions of the reference materials included in the device were calibrated at PTB [5] but, in order to use them as reference pressure points at NMi, they have to be realized again at NMi (in thermal equilibrium with our MCT) so that the sensor capacitance value corresponding to the midpoint of the transitions can be measured. As a consequence, the uncertainty associated with the calibration procedure at PTB must be combined with the uncertainty associated with the use of the device at NMi.

The sources of uncertainties associated with the calibration of the SRM768 device at PTB are (see Table 1):

- Realization of the transition: the reproducibility of the midpoint of the superconductive transition for several passages through the transition.
- Residual magnetic field: the shift in the midpoint of the transition produced by the residual magnetic field within the shield where the superconductive samples are enclosed.
- Realization of the melting pressure: uncertainty related to the use of the pressure balance.

The sources of uncertainties associated with the use of the SRM768 device at NMi are the same encountered by PTB in the calibration phase (but we leave out the uncertainty related to the measurement of the melting pressure). For the first two components of Table 1 there is correlation between the calibration at PTB and the use at NMi: to take into account this correlation we considered only the contributions from PTB.

		Use at NMi		
	Realization of transition /Pa	Residual magnetic field /Pa	Melting pressure measurement /Pa	Pressure control stability /Pa
W transition	158	39	118	47
Be transition	150	37	112	22
Ir transition	86	43	108	45
AuAl ₂ transit.	27	27	106	16
AuIn ₂ transit.	25	17	93	35

Table 1 Component standard uncertainties involved in the calibration at PTB and in the use at NMi of the superconductive reference points of SRM768 device.

The output of the observation of the minimum of the melting curve and of the superconductive transitions of the SRM768 at NMi is the corresponding capacitance values measured by the pressure transducer. The uncertainty components for the measured capacitance values are reported in Table 2: the stability, the non-linearity and the temperature sensitivity of the capacitance bridge used are provided by the manufacturer, the control stability

(which is the effectiveness of maintaining a given level of pressure (or temperature equivalently) within the MCT), is dictated by the temperature control system adopted.

	Stability Capacitance Bridge /aF	Non-linearity Capacitance Bridge /aF	Temperature Sensitivity Capacitance Bridge /aF	Temperature Control Stability /aF
W transition	100	6	12	260
Be transition	100	6	12	250
Ir transition	110	6	12	260
AuAl ₂ transition	100	6	12	60
AuIn ₂ transition	100	6	12	190
Minimum	100	6	12	100

Table 2 Standard uncertainties components for the measurement of the capacitance at the pressure reference points. The capacitance bridge used is Andeen Hagerling type 2500A. The degrees of freedom are infinite for the first three components and 9 for the last one.

The pressure reference points and corresponding measured capacitance values with associated combined standard uncertainties are reported in Table 3 for our last measurement run (October 2002).

	Capacitance at pressure reference point /pF	Capacitance standard uncertainty u(C) /pF	Pressure reference point /MPa	Pressure reference point standard uncertainty u(p) /MPa
W transition	36.4040	0.0003	3.38168	0.00021
Be transition	36.2074	0.0003	335165	0.00019
Ir transition	34.7913	0.0003	3.12780	0.00015
AuAl ₂ transition	34.1724	0.0001	3.02323	0.00011
AuIn ₂ transition	33.8897	0.0002	2.97370	0.00010
Minimum melting curve	33.6471	0.0001	2.93113	0

 Table 3 Pressure reference points and corresponding measured capacitance value with associated combined standard uncertainties.

4 Calibration of the pressure transducer

A linear relation between the inverse of the capacitance C measured by the pressure sensor and pressure p is adopted with constant coefficients a and b:

$$p(C) = a + b \cdot (1/C)$$

The constant coefficients *a* and *b* and their experimental variance u(a) and u(b) are determined by weighted linear regression of the reference points. Different sets of reference points were selected in different measurement runs. The history of our pressure sensor over the past two years is reported in Figure 2(a). In our last measurement run (October 2002 in Figure 2(a)) Be, Ir, AuAl₂, AuIn₂ and the minimum of the melting curve were used as reference points. The numerical results were the following:

$$a = 8.87MPa$$
 $b = -199.9pF \cdot MPa$ $s(p) = 0.0006MPa$
 $u(a) = 0.01MPa$ $u(b) = 0.3pF \cdot MPa$ $r(a,b) = -0.99966$

where u(a) and u(b) are the standard uncertainties of *a* and *b*, s(p) is the overall uncertainty of the fit and r(a,b) is the correlation coefficient between *a* and *b*. The combined standard uncertainty of any pressure measurement can be derived from the expression:

$$u^{2}(p) = u^{2}(a) + u^{2}(b)/C^{2} + (b^{2}/C^{4}) \cdot u^{2}(C) + (2/C) \cdot u(a) \cdot u(b) \cdot r(a,b) + u^{2}_{non-linearity}(p)$$

where the first three terms represents the usual propagation of uncertainties, the forth term accounts for the strong correlation between a and b and the last term is added to include the non-linearity of the pressure sensor (the linear functional relationship does not describe perfectly the response of the pressure transducer).

The standard temperature uncertainty $u(T_{2000})$ can be calculated for all the range by simply dividing the standard pressure uncertainty by the derivative of the melting pressure polynomial with respect to temperature (see Figure

2(b)):
$$u(T_{2000}) = \frac{u(p)}{dp(T)/dT}$$



Figure 2 (a) History of NMi pressure transducer. (b) Uncertainty of the realization of the PLTS-2000 at NMi.

The divergence of uncertainty in correspondence with the minimum of the melting curve does not allow practical measurements between 290 and 340 mK.

Conclusions

The uncertainty for the realization of the PLTS-2000 at NMi was calculated in accordance with the "Guide to the Expression of Uncertainty in Measurement". The calibration of the MCT, using the minimum of the ³He melting curve and a set of superconductive points as pressure reference points, proved to provide accurate temperature measurements in the range 10 mK – 1 K, with the exclusion of a narrow range centred around the minimum of the ³He melting curve. The realization of the PLTS-2000 described in the present paper will be used for the calibration of newly developed commercial superconductive reference devices (SRD1000).

5 References

1. Preston-Thomas H. Metrologia, v.27, p. 3 (1990).

2. Bureau International des Poids et Mesures, Comite' Consultatif de Thermometrie (CCT), Working group 4 Report to CCT, Document CCT/2000-26, April 2000.

3. Schuster G., Hoffmann A. and Hechtfischer D., Realization of the temperature scale PLTS-2000 at PTB, Physikalisch-Technische Bundesanstalt Braunschweig und Berlin Presse und Offentlichkeitsarbeit, Braunschweig, 2001, 29 p.

4. Soulen J.F., Dove R.B., SRM 768:Temperature Reference Standard for use below 0.5 K, NBS Special Publication n. 260-62, 1979, 37 p.

5. Bosch W.A. et al., 'Proceedings of the 8th International Symposium on Temperature and Thermal Measurements in Industry and Science (TEMPMEKO 2001)', edited by Fellmuth B. et al., VDE Verlag, Berlin, 2001, pp. 397-401.

6. Hechtfischer D. and G Schuster, Calibration of SRM 768 superconductive fixed point devices, Technical Report of EU project "European Ultra-Low Temperature Scale and Traceability", Contract n. SMT4-CT96-2052, 1999