EVALUATION OF THE UNCERTAINTY IN THE REALIZATION OF THE PLTS-2000 AT NMi

A. Peruzzi and M.J. de Groot

Nmi van Swinden Laboratorium, Delft, The Netherlands

1 Abstract

The Provisional Low Temperature Scale (PLTS-2000) is realized at NMi from 10 mK to 1 K by operating a ^{3}He melting curve thermometer (MCT) produced by PTB. The calibration of the pressure transducer through the use of reference points is described. The sources of uncertainties, arising from the process of calibration of the transducer, are identified and evaluated. The final accuracy of the realization of the PLTS-2000 is calculated for the whole range in which the MCT is operated.

2 Introduction

The PLTS-2000 [1] is defined by an internationally accepted polynomial $p_m = p_m(T_{2000})$ relating the ^{3}He melting pressure p_m to temperature T_{2000}.

The uncertainty of the PLTS-2000 realization is directly related to the uncertainty of the calibration of the pressure transducer for which different options are possible:

1. Use of primary pressure standard (pressure balance).
2. Use of calibrated secondary pressure standard (Digiquarts etc).
3. Use of intrinsic properties of ^{3}He (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 [2] and SRD1000 [3] devices, for example).

Benefits and drawbacks of the four options are extensively described in a recent comprehensive publication of PTB [4]. The highest accuracy can be achieved by using option 1 at cost of large workload. Option 3 is attractive for the self-consistency of the pressure transducer calibration although the ^{3}He phase transitions are not accessible to ordinary dilution refrigeration and an additional demagnetization 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 SRM 768 device previously calibrated at PTB) are used as pressure reference points (see Table 1) to calibrate the capacitive pressure transducer of a MCT produced by PTB. In this document the uncertainty arising from the calibration procedure is evaluated and the final uncertainty in the realization of the PLTS-2000 is calculated.

3 Reference points and associated uncertainty

Reference points for the calibration of the pressure transducer are physical states of known melting pressure to be realized in thermal equilibrium with the MCT in order to measure the corresponding capacitance value of the pressure transducer.

Physical State	Temperature /mK	^{3}He Melting pressure /MPa	Measured capacitance /pF
W transition	15.44	3.38168	36.4040
Be transition	23.25	3.35165	36.2074
Ir transition	100.79	3.12780	34.7913
$AuAl_2$ transition	161.70	3.02323	34.1724
$AuIn_2$ transition	207.78	2.97370	33.8897
Minimum	315.24	2.93113	33.6471

Table 1. Reference points used at NMi, corresponding temperatures, ^{3}He melting pressures, and capacitances values of the pressure transducer.

To use the reference points in the calibration of the pressure transducer, the sources of uncertainty in the melting pressures and in the corresponding measured capacitances must be preliminary identified and evaluated.

3.1 Pressure uncertainties at reference points

As regards the minimum of the ^{3}He melting curve, its pressure value is fixed by the definition of the PLTS-2000 and no uncertainty is associated with it.

The superconductive transitions of the SRM 768 are regarded as external standards and the uncertainties arising in their calibration at PTB have to be combined with the uncertainties associated with their use at NMi.

The sources of uncertainties associated with the calibration of the SRM768 device at PTB [5] are (see Table 2):

- Realization of the transition: the reproducibility of the midpoint of the superconductive transition in at least four independent measurements, each of them including several passages through the transition.
- Residual magnetic field: the shift in the midpoint of the transition, produced by the residual magnetic field acting on the reference samples, was calculated and taken as uncertainty of the uncorrected value.
- Realization of the melting pressure: uncertainty related to the measurement of the melting pressure.

The sources of uncertainties associated with the use of the SRM 768 at NMi are the same encountered at PTB but some additional specifications are needed:

- For the first two components, the strong correlation between the calibration at PTB and the use at NMi is taken into account by dropping the NMi components.

- The melting pressure measurement component must be replaced at NMi by simply the pressure control stability because we just realize the required reference level of melting pressure without measuring it.

	Calibration at 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
$AuAl_2$ transition	27	27	106	16
$AuIn_2$ transition	25	17	93	35

Table 2 Component standard uncertainties involved in the calibration at PTB and in the use at NMi of the superconductive reference points of SRM768 device.

3.2 Capacitances uncertainties at reference points

The uncertainty components related to the measurement of the capacitance values at the reference points are reported in Table 3. The calculation of the components associated with the use of the capacitance bridge is straightforward from the measured capacitances and losses, and the specifications provided by the manufacturer of the bridge.

The uncertainty related to the calibration of the bridge is not included because we are not interested in the absolute value of the measured capacitances. A contribution from the experimental control stability is included instead.

	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 3 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.

	Pressure reference point /MPa	Pressure reference point standard uncertainty u(p) /MPa	Capacitance at pressure reference point /pF	Capacitance standard uncertainty u(C) /pF
W transition	3.38168	0.00021	36.4040	0.0003
Be transition	335165	0.00019	36.2074	0.0003
Ir transition	3.12780	0.00015	34.7913	0.0003
$AuAl_2$ transition	3.02323	0.00011	34.1724	0.0001
$AuIn_2$ transition	2.97370	0.00010	33.8897	0.0002
Minimum	2.93113	0	33.6471	0.0001

Table 4 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 transducer and pressure *p* is adopted as calibration curve with constant parameters *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 a weighted least-squares fit of the reference points. To include in the fit both the uncertainties in *C* and in *p*, the "effective variances" σ_i^{eff} are used as weights [6]:

$$\left(\sigma_i^{eff}\right)^2 = \sigma_i^2(p) + \left(\left.\frac{dp(C)}{dC}\right|_{C_i}\right)^2 \sigma_i^2(C)$$

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, $AuAl_2$, $AuIn_2$ and the minimum of the melting curve were used as reference points.

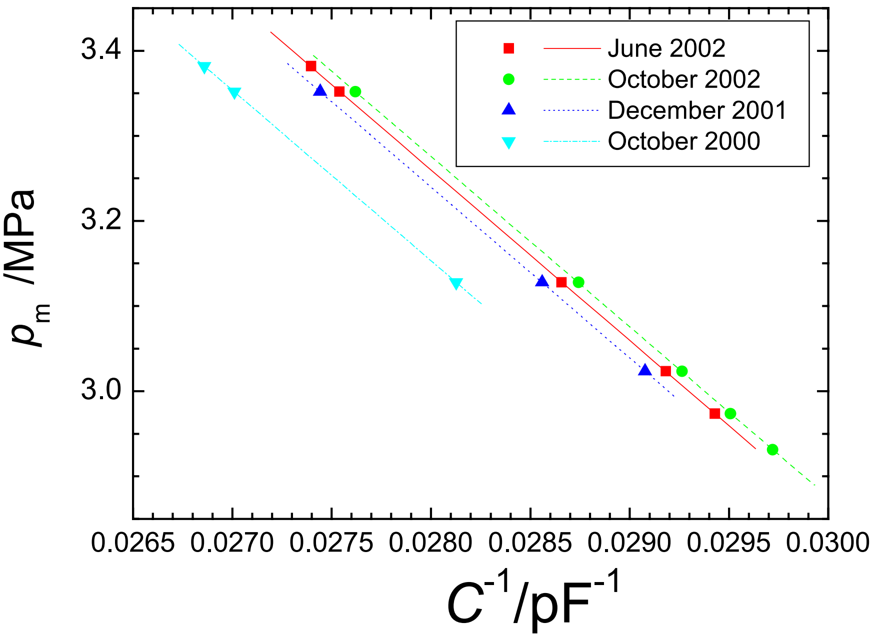

Figure 1 History of NMi pressure transducer over the past two years.

The numerical results of our last measurement run were the following:

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

in which *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*.

5 Uncertainty in the realization of the PLTS-2000

The combined standard uncertainty of any pressure measurement can be derived from the expression:

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

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 standard temperature uncertainty $u(T_{2000})$ can be calculated for the whole range by simply dividing the standard pressure uncertainty by the derivative of the melting pressure polynomial with respect to temperature (see Figure 2):

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

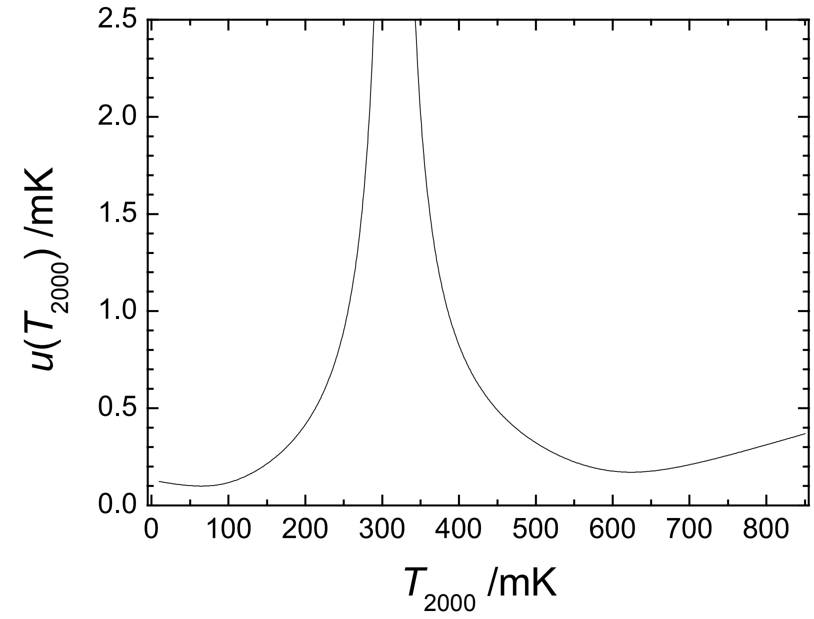

Figure 2 Uncertainty of the realization of the PLTS-2000 at NMi.

The divergence of the uncertainty around the minimum of the melting curve is produced by the null sensitivity of the MCT at the minimum itself. Excluding the range around the minimum, the uncertainty in the PLTS realization is between 0.2 and 0.5 mK.

5 Conclusions

The uncertainty in 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, 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 ^{3}He melting curve. The realized PLTS-2000 described will be used for the calibration of newly developed commercial superconductive reference devices (SRD1000).

6 References

1. Working group 4 Report to CCT, Document CCT/2000-26, April 2000.

2. 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.

3. 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.

4. 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.

5. 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).

6. Orear J., 'Least squares when both variables have uncertainties', Am. J. Phys. 50, (1982), p. 912-916.