Traceable Low and Ultra-Low Temperatures in the Netherlands

A. Peruzzi\textsuperscript{1}, W.A. Bosch\textsuperscript{2}
\textsuperscript{1}NMi-VSL, Nederlands Meetinstituut van Swinden Laboratorium, Delft, The Netherlands
\textsuperscript{2}HDL, Hightech Development Leiden, Leiden, The Netherlands

E-mail: aperuzzi@nmi.nl

Abstract. The basis for worldwide uniformity of low and ultra-low temperature measurements is provided by two international temperature scales, the International Temperature Scale of 1990 (ITS-90) for temperatures above 0.65 K and the Provisional Low Temperature Scale of 2000 (PLTS-2000) for temperatures in the range 0.9 mK to 1 K. Over the past 10 years, the thermometry research in the Netherlands provided substantial contributions to the definition, realization and dissemination of these scales. We first give an overview of the Dutch contributions to the ITS-90 realization: a) $^3$He and $^4$He vapour pressure thermometer range of the ITS-90, 0.65 K to 4 K (1997), b) $^4$He interpolating constant volume gas thermometry for the ITS-90 range 3 K to 24.5 K (2007) and c) cryogenic fixed points for the ITS-90 range 13.8 K to 273.16 K (2005). Then we highlight our work on $^3$He melting pressure thermometry from 10 mK to 1 K (2003) to support the dissemination of the PLTS-2000. Finally we present the current status of the Dutch calibration facilities and dissemination devices providing for traceable low and ultra-low temperatures for use in science and industry: a) the NMi-VSL cryogenic calibration facility for the range 0.65 K to 273.16 K and b) the SRD1000 superconductive reference devices for the range 10 mK to 1 K.

1. Introduction

The thermodynamic temperature scale is the fundamental scale of temperature to which all temperature measurements should be related. Direct measurements of the thermodynamic temperature are very complex, time consuming and poorly reproducible. For all practical purposes and for international use, practical international scales \cite{1} were introduced. The current International Temperature Scale (ITS-90, \cite{2}) extends upwards from 0.65 K to the highest temperature practicably measurable. In year 2000 the ITS-90 was supplemented by the Provisional Low Temperature Scale (PLTS-2000, \cite{3}), which covers the temperature range from 0.9 mK to 1 K.

The goal of this paper is twofold:

1. Give account of the substantial contributions that the Dutch thermometry community has provided in the past 10 years to the definition and realization of the internationally agreed temperature scales.

2. Present the calibration facilities that are currently available in the Netherlands to allow academic, industrial and accredited laboratories working in the field of low temperature to access SI-traceable temperatures.
2. $^3$He and $^4$He vapour pressure thermometer for the ITS-90 range from 0.65 K to 5 K (1997)

From 0.65 K to 5.0 K, the ITS-90 temperature $T_{90}$ is defined in terms of the vapour pressure $p$ of liquid and vapour $^3$He or $^4$He at equilibrium, using specified interpolation equations $T_{90} = f(p)$. This range of the ITS-90 was realized at NMi-VSL in 1997. The pressure of $^3$He and $^4$He, contained in a copper reservoir within a $^3$He evaporation cryostat, was measured by using a combination of a gas-operated pressure balance and a capacitive diaphragm gauge at room temperature (see [4]).

3. $^4$He interpolating constant volume gas thermometer for the ITS-90 range 3 K to 24.5 K (2007)

Between 3.0 K and 24.5561 K, the ITS-90 is defined by means of a helium interpolating constant-volume gas thermometer (ICVGT) calibrated, using specified interpolation equations, at three defining fixed points: 1) the triple point of neon (24.5561 K), 2) the triple point of equilibrium hydrogen (13.8033 K), 3) a temperature between 3.0 K and 5.0 K to be determined using a $^3$He or a $^4$He vapour pressure thermometer.

The ICVGT range of the ITS-90 was realized at NMi-VSL with $^4$He as thermometric gas in 2007. A one liter gold-plated cylindrical copper bulb (see Fig. 1) is temperature-controlled at the desired nominal temperature, within a vacuum chamber submersed in liquid helium. The bulb is filled with high-purity (6N) $^4$He, at a density of approximately 184 mol/m$^3$, from the external room-temperature thermometric gas supply through a 1 mm capillary stainless steel tube. The pressure of the $^4$He in the gas bulb is then measured at any nominal temperature between 3.0 K and 24.5561 K at the highest accuracy level (20 ppm) by using a combination of a gas-operated pressure balance and a capacitive diaphragm gauge.

4. Cryogenic fixed points for the ITS-90 range from 13.8 K to 273.16 K

For the subrange from 13.8033 K to 273.16 K, the interpolation instrument specified by the ITS-90 is the capsule standard platinum resistance thermometer (CSPRT), calibrated at 8 defining fixed points: the triple points of equilibrium hydrogen (13.8033 K), neon (24.5561 K), oxygen (54.3584 K), argon (83.8058 K), mercury (234.3156 K), and water (273.016 K) and at two additional temperatures close to 17.0 K and 20.3 K, to be determined with the ICVGT. The cryogenic fixed points (hydrogen, neon, oxygen and argon) are realized at NMi-VSL by using state-of-the-art sealed triple-point cells (STPCs) of the corresponding high-purity gases (99.999% to 99.9995%), accomodated in an adiabatic calorimeter (see Figure 2 and reference [5]).

5. Realization of the PLTS-2000 in the range 10 mK to 1 K

From 2000 to 2003, access to the PLTS-2000 was made available at NMi-VSL with the purpose of spinning up the development of a superconductive reference device (SRD1000, see later in this paper). The practical realization of the PLTS-2000 requires a $^3$He melting pressure thermometer (MPT) to be operated. The MPT is essentially a closed compression chamber, containing both liquid and solid $^3$He.
at equilibrium. An in situ capacitive pressure sensor measures the melting pressure $p_m$ inside the chamber, and the measure melting pressure is transformed into temperature $T_{2000}$ by applying the inverse of the agreed equation $p_m = p_m(T_{2000})$ (see Figure 3).

**Figure 3** $^3$He equilibrium melting pressure as a function of temperature.

A picture of the experimental set-up at NMi-VSL is shown in Figure 4. The MPT cell is mounted on a gold-plated comparator block attached to the mixing chamber stage of a dilution refrigerator. On the bottom of the comparator block a number of superconductive devices are attached: 2 NIST superconductive devices (SRM767 and SRM768) [6,7], and up to 5 of the SRD1000 devices under development.

6. NMi-VSL cryogenic calibration facility for the range 0.65 K to 273.16 K

The result of the experiments described in the previous chapters (the so-called “materialization of the scale”) is a set of reference thermometers carrying the ITS-90 (the so-called “wire scale”). These reference thermometers are then used in the calibration facility (see Figure 5) to calibrate users’ thermometers by direct comparison. A calibration certificate is released to the costumer, reporting the measurement data, the interpolation function and the expanded uncertainty at coverage factor $k = 2$ (corresponding to approximately 95% confidence level).

7. Superconductive reference device SRD1000 for the range 10 mK to 1 K

In the period from 2000 to 2003 a Dutch consortium comprising NMi-VSL, the company HDL, Leiden University and the University of Twente developed technology for a superconductive reference device SRD1000 and related detection electronics [8]. The device includes 10 stable reference points that are provided by the superconductive transitions of samples of several materials in the range from 10 mK to 1 K. The output voltage of the electronics versus sensor temperature shows distinct transitions at the reference temperatures $T_C$ (see Figure 6). An evaluation of several sets of devices and electronics by various metrological institutes in Europe proved that the concept was reliable for transferring the PLTS-2000 scale [9]. In the period from 2003 to 2006, several systems were built by HDL and calibrated at PTB (Physikalisch-Technische Bundesanstalt, Berlin). Measurements have shown that the typical uncertainty levels for determining the $T_C$’s range from about 0.04 mK at 15 mK to about 0.8 mK at 1.2 K. The systems are now in use by several ultra-low temperature research laboratories in Europe and Japan.
In 2008 a new system was introduced that integrates the concept of realizing calibrated reference points and that of a continuous temperature reading between 10 mK and 1 K with a CMN (Cerium Magnesium Nitrate) paramagnetic susceptibility thermometer. The resolution of the continuous thermometer is better than 0.5 mK below 0.5 K. A new type of detection electronics allows simultaneous measurement of the SRD and CMN sensor elements, thus providing in-situ temperature calibration of the CMN signal. Figure 7 shows the combined sensor block and Figure 8 the MIDS-202 detection electronics. For more information, see the web site: http://www.xs4all.nl/~hdleiden/srd1000.

8. Conclusions
The achievements of primary thermometry in the Netherlands in the past 10 years were reviewed. The facilities and dissemination devices made recently available will improve traceability of science and industry in the low and ultra-low temperature fields.

9. References
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