Dissemination of the European Ultra-low Temperature Scale

'ULT Dissemination'

Contract No: G6RD-CT-1999-00119

Deliverable 2.1

Report on

Development of SRD Technology

Authors:

W.A. Bosch
HDL Hightech Development Leiden
P.O. Box 691, 2300 AR Leiden, The Netherlands

R. Jochemsen
Universiteit Leiden, Leids Instituut voor Onderzoek in de Natuurkunde
P.O. Box 9504, 2300 RA Leiden, The Netherlands

M.J. de Groot, A. Peruzzi
Nederlands Meetinstituut, Van Swinden Laboratorium
P.O. Box 654, 2600 AR Delft, The Netherlands

J. Flokstra, D. Veldhuis
Universiteit Twente
P.O. Box 217, 7500 AE Enschede, The Netherlands

Date: May 2003

This report and other information on the SRD development can be found at the website:
http://www.xs4all.nl/~hdleiden/srd1000

This work was partially funded by the European Commission under the Measurement and Testing activity of the Programme for Competitive and Sustainable Growth, contract no. G6RD-CT-1999-00119.

1. Executive Summary

This report describes the results of a project to develop SRD technology. The aim of the project is to acquire knowledge on how to produce Superconductive Reference Devices, SRDs. The devices are intended to provide users with the means to generate 10 calibrated reference temperatures between 10 mK and 1200 mK on the PLTS-2000, the Provisional Low Temperature Scale from 0.9 mK to 1 K [1]. In the SRDs, the sharp transition from superconducting to normal state of a set of metal samples is detected and used to establish reference temperatures for the scale. The transitions are measured using a mutual inductance technique, which detects the change of magnetic flux through the samples.

Experience with the former SRM 767 and SRM 768 devices produced by NIST [2, 3] followed by further metrological research [4, 5], has shown that the transitions of bulk metal samples are a very useful and accurate direct means of providing traceable thermometry below 1 K. These SRMs are no longer available. The new device, referred to as SRD1000, is intended as a follow-up to the SRMs and will support the dissemination of the PLTS-2000. Compared with the previous devices the SRD1000 will contain many novel design features for a highly accurate realisation of the reference temperatures. Five prototypes of the SRD1000 were to be built and calibrated during the project to acquire sufficient experience with such devices.

The development project was carried out by a Dutch consortium, of which each member had its specific tasks:

HDL	overall development of the devices, detectors, materials and electronics
LION	development of preparation procedures for bulk reference materials
NMi-VSL	development of a PLTS-2000 calibration facility
UT	development of a planar flux detector, study on thin-film materials

The project was broken down into several main tasks, each with its specific goal, set of activities and outputs. Details of the results of these tasks are described in the Sections 2 to 6 of this report.

- **Design review (Section 2):** the technical goals of the development and specifications of the various activities were defined;

- **Research on the reference materials (Section 3):** ten types of reference materials were selected and various batches of samples were prepared, tested and evaluated; this task resulted in knowledge on the sample production processes; batches of samples qualified for the prototype SRDs were produced;

- **Development of the SRD1000 sensor (Section 4):** this task resulted in a proven design of a novel planar detector system, sensor housing and shields for mutual inductance measurement on samples of the reference materials to detect the transitions; components for five sets of SRD sensors were produced;

- **Development of the SRD1000 electronics (Section 5):** this task resulted in a proven design of a novel mutual inductance measurement system and related

electronics tools dedicated to highly accurate realisation of the transition temperatures of the samples; five sets of electronics were produced;

- **Assembly, calibration and dissemination of SRDs (Section 6):** a low temperature calibration facility was constructed and tested; five prototype SRDs were assembled, each supporting ten reference temperatures; they were calibrated against the PLTS-2000, together with their related measurement electronics.

With respect to the planning, a considerable overrun in time and cost occurred while carrying out the project. This was mainly caused by having to solve unexpected problems in preparing the reference materials. Many trials were needed to produce batches, which showed transitions of sufficient quality to suffice the requirement on the reference temperatures. In the end, sufficient good quality material was available for the first prototypes. However, the sample preparation processes need some additional development in order to be able to manufacture a next series of SRDs of equal or better quality. Other delays were caused by the modifications needed to the calibrated set-up to reach sufficiently low temperatures and to finalize the design parameters of the electronics.

The development resulted in five sets of calibrated prototype SRD1000 sensors and measurement electronics. Each prototype sensor contained ten reference materials with clear superconductive transitions along the PLTS-2000.

Analysis of the calibration data of these prototypes shows that the relative uncertainty in the realization of the reference temperatures resulting from the combined uncertainties of the SRD1000 instrumentation and the calibration set-up, ranges between 0.9 % at 15 mK to less than 0.1 % at 1 K. This meets the requirements on the reference temperatures to support and disseminate the PLTS-2000, given the uncertainty of the scale.

In a subsequent development phase the prototypes will be sent to other partners of the project for a metrological evaluation of their performance.

At various relevant conferences and symposia, the consortium reported on the status and results of the development in several papers, posters and oral presentations, see references [9] to [14]. This resulted in a positive response from the market. Already some 20 institutes and companies (15 in Europe, 3 in the USA and 2 in Japan) expressed their interest and the intention of buying the SRD1000.

After the evaluation of the prototypes, HDL will start commercial exploitation by developing a small pilot series of devices and electronics in cooperation with the present project partners NMi-VSL, LION and others. Meanwhile production and sales strategies will be explored and agreements for cooperation will be made. Based on a first estimate of the market, sales are expected of at least 15 sets of SRD1000 and electronics each year for the coming 10 to 15 years.

2. Design review

The goal of the SRD technology development is to acquire knowledge on how to produce a device that enables users to establish reference points for the PLTS-2000. During a design review it was decided to build a set of five prototype devices, based on the proven concept of the former SRM 767 and SRM 768 devices [2, 3]. In such devices the superconducting to normal transition of a set of metal samples is used to establish reference temperatures for the temperature scale.

The transitions are measured using a mutual inductance technique, which detects the change of magnetic flux through the samples. Figure 1 shows the output signal of the proposed SRD1000 sensor as a function of temperature. The superconductive transition is recorded while the temperature rises in small steps. The output signal V increases from V_S in the superconducting state to V_N in the normal state and $V_N - V_S = \Delta V$. The reference temperature T_C of the transition is defined as the temperature at which 50% of the transition is completed, so at a signal voltage $V = Vs + 0.5\ \Delta V$. The width W_C of the transition is defined as the temperature interval for 80% of the transition: $W_C = T(V_N - 0.1\ \Delta V) – T(V_S + 0.1\ \Delta V)$.

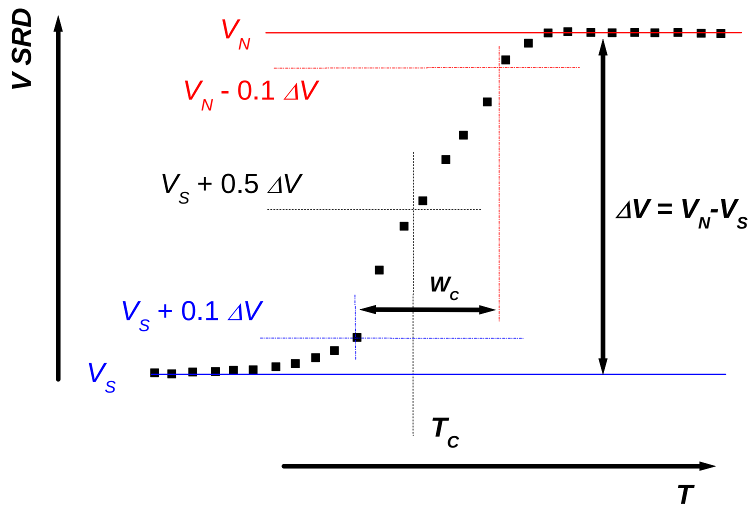


FIGURE 1. Definition of the T_C and W_C of a superconductive transition

It was decided that the SRD1000 prototypes should have at least 10 reference points to support the PLTS-2000 with determination uncertainty at least smaller than the uncertainty of the scale. Figure 2 shows the arrangement of the reference points along the scale. The new device combines the arrangement of points in the SRM 767 (3 of the 5 samples) and SRM 768 devices, and provides two additional points between 20 mK and 100 mK.

To detect the transitions, a mutual inductance coil system had to be developed which a) enables a good signal-to-noise ratio despite the use of small samples, b) allows a good thermal contact with the samples and c) minimizes the detection of spurious signals from the vicinity of the samples. It was decided to develop a novel planar micro-coil system together with dedicated measurement electronics to detect the transitions, assuming that this would ensure a high accuracy when the reference points are reproduced.

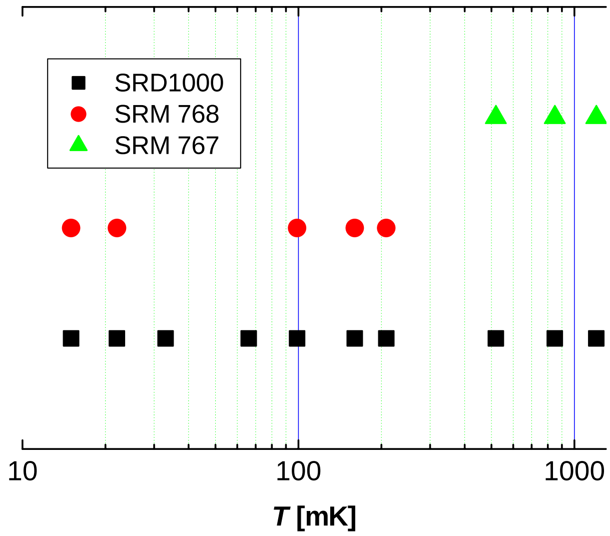


FIGURE 2. Arrangement of the reference temperatures for the SRD1000 and for the former NIST SRM 768 and SRM 767 devices.

3. Research on reference materials

3.1. Bulk materials

Several batches of the reference materials were prepared, shaped and tested. Like in the SRM 767 and SRM 768, samples of W, Ir, $AuAl_2$, $AuIn_2$, Cd, Zn and Al were tried for the reference points with T_C = 15, 100, 160, 208, 520 1200 mK respectively. Furthermore alloys of $Ir_{73}Rh_{27}$, $Ir_{80}Rh_{20}$, and $Ir_{92}Rh_{08}$ were tested for their suitability to provide reference points in the temperature region between 15 mK and 100 mK, thus omitting the poisonous Be (T_C = 20 mK) which was used in the SRM 768.

Many trials with various preparation processes were needed to produce samples, which showed transitions of sufficient quality to meet the requirements on the reference temperatures. This resulted in a considerable overrun in time and costs in comparison with the original planning of the project.

A selection was made of samples of ten materials to be included in the prototype devices. It was decided to use Be instead of the $Ir_{73}Rh_{27}$ to establish a reference point near 20 mK in the prototypes. The Be samples require Al spot welds to reduce supercooling, and precautions are necessary while handling due to its toxicity. But when careful preparations are made, the transitions of the Be samples are far superior compared with those of the $Ir_{73}Rh_{27}$ samples. The transition widths observed for Zn and Cd are much larger than what might be expected from samples of single crystals. Despite many efforts, the quality of Zn and Cd transitions could not be improved.

During a subsequent development of a pilot series, more efforts will be made to find a solution.

TABLE 1. Materials selected for the prototypes, their specifications, the minimum and maximum values found for the transition temperature T_C, the transition width W_C and the related uncertainty $\Delta T_C/T_C$ of the determination of T_C. The transition width in typical SRM 767 and SRM 768 devices is listed as well.

Selected Material	Specification and sample preparation	T_C [mK]	W_C [mK]	$\Delta T_C/T_C$ [%]	Typical W_C in SRM 767 / 768 [mK]
W	4N single crystal [1] spark cut to 3×3×0.2 mm sample, all sides polished and rounded off, Al spot weld on top of sample to reduce supercooling, ultrasonically cleaned in acetone and alcohol	15.0 – 15.2	0.1 – 0.6	0.05 - 0.3	0.7
Be	2N8 foil [2] sheet cut to 3×1.5 mm sample, ultrasonically cleaned in acetone and alcohol, Al spot weld on top of sample to reduce supercooling	20.8 – 22.0	0.08 - 0.8	0.01 - 0.1	0.2
$Ir_{80}Rh_{20}$	4N5 Ir powder [2] and 4N Rh powder [2] mixed to proper ratio, arc melted to form droplet-shaped sample, annealed for one week in arc melt furnace close to melting point, spark cut to 3×1.5×0.5 mm sample, all sides polished and rounded off, ultrasonically cleaned in acetone and alcohol	32.1 - 32.3	0.6 - 1	0.02 - 0.03	-
$Ir_{92}Rh_{08}$		65.5 - 66.1	0.5 – 0.9	0.02 - 0.03	-
Ir	melt 4N5 Ir powder [2] to form droplet shaped sample, spark cut to 3×1.5×0.5 mm sample, all sides polished and rounded off, ultrasonically cleaned in acetone and alcohol	94 – 99	0.4 - 1	0.03 - 0.08	0.8
$AuAl_2$	5N4 Au powder [2] and 5N Al powder [2] arc melted to form a seed crystal, single crystal is grown in tri-arc furnace using seed crystal, spark cut to 3×1.5×0.5 mm sample, all sides polished and rounded off, ultrasonically cleaned in acetone and alcohol	160.8 - 160.9	0.5 – 1.7	0.01 - 0.03	0.3
$AuIn_2$	6N Au powder [3] and 6N In powder [4] Au arc melted to form a seed crystal, single crystal is grown in tri-arc furnace using seed crystal, spark cut to 3×1.5×0.5 mm sample, all sides polished and rounded off, ultrasonically cleaned in acetone and alcohol	208 - 211	0.7 – 9.4	0.007 - 0.1	0.4
Cd	6N shot [2] melted and annealed to single crystal, spark cut to 3×3×0.2 mm sample, ultrasonically cleaned in acetone and alcohol	516 – 518	8.7 – 17	0.05 - 0.1	2.5
Zn	6N shot [3] melted and annealed to single crystal, spark cut to 3×3×0.2 mm sample, ultrasonically cleaned in acetone and alcohol	844 - 845	5.9 - 14	0.03 - 0.07	8.4
Al	5N5 foil [2] sample cut to 5×3 mm, ultrasonically cleaned in acetone and alcohol	1165 - 1166	3.5 - 8	0.01 - 0.03	2.6

[1] Goodfellow, [2] Alfa Aesar, [3] Cominco, [4] Kawecki Biliton; 4N, 2N8, 4N5, etc. refer to nominal purities of 99.99 %, 99.8 % and 99.995 % respectively

Table 1 gives an overview of the finally selected materials and their specifications. The characteristics of the preparation of the samples are indicated. The table also gives the minimum and maximum values observed for a) the transition temperature T_C, b) the transition width W_C, and c) the related uncertainty $\Delta T_C/T_C$ of the determination of T_C within a test run. This uncertainty is derived from the width and the step height of a transition in relation to the noise of the detection electronics (see also Section 6.4). The W_C in typical SRM 767 and SRM 768 devices is listed as well.

Typical transitions of samples of the selected materials are shown in the Appendix of this report. A summary of the results of the measurements on all samples produced during the project is presented at the website: *http://www.xs4all.nl/~hdleiden/srd1000*

For most prototype samples, the estimated uncertainty in the determination obtained is better than the standard uncertainty of the PLTS-2000 in thermodynamic terms, which is 0.5 mK down to 500 mK, 0.2 mK at 100 mK and 0.3 % of T at 25 mK.

At present, the quantity of good quality samples of some materials is rather limited. In order to be able to produce a next series of SRDs of equal or better quality, a further development is required of the processes for sample preparation.

3.2. Film materials

A survey of multi-layers as reference materials was made to find stable reference points between 200 mK and 500 mK. Ti/Au and Mo/Au bi-layers were selected for further research.

A set of thin film samples of these materials was prepared using a laser deposition technique. No superconductivity could be detected using the inductive measurement technique of the SRDs.

A further study on the relation between the thickness of thin superconducting layers and the transition temperature revealed that layers much thinner than the penetration depth effectively screen the flux in a mutual inductance experiment and that superconductivity should have been detected. This means that most likely during laser deposition the background pressure influences dramatically the forming of superconducting layers from Ti and Mo due to reaction processes most probably with oxygen.

Other deposition techniques like sputter deposition should therefore be used to fabricate appropriate bi-layers.

4. Development of the SRD1000 sensor

Figure 3 gives an example of a SRD1000 test sensor, showing the main features of the developed device with detection circuitry for the transitions. The sensor is thermally connected to the experimental region of a cryogenic cooling system for thermometry measurements.

The SRD1000 uses a mutual inductance system consisting of planar niobium micro-coils (a1) to detect the superconducting to normal transitions of the reference samples. The samples are directly attached to this detecting system (a2).

The detector and samples are thermally connected to a gold-plated copper holder (b1 is without and b2 is with detector).

A magnetic shield (c) surrounds the copper holder, consisting of a Cryoperm outer shield and a niobium inner shield. It reduces ambient magnetic fields by a factor of 500 or more.

The geometry of the planar coil system allows miniaturization of the total SRD1000 sensor. The sensor may hold up to 12 reference samples and an optional CMN thermometer in a volume of less than ∅ 10 mm and 50 mm length (d).

Furthermore, the planar detector behaves like an ideal mutual inductance with minimal coupling to the copper holder, resulting in a sensor output that is almost independent of temperature at temperatures between the reference points. The geometry also allows a short thermal path between the samples and copper holder.

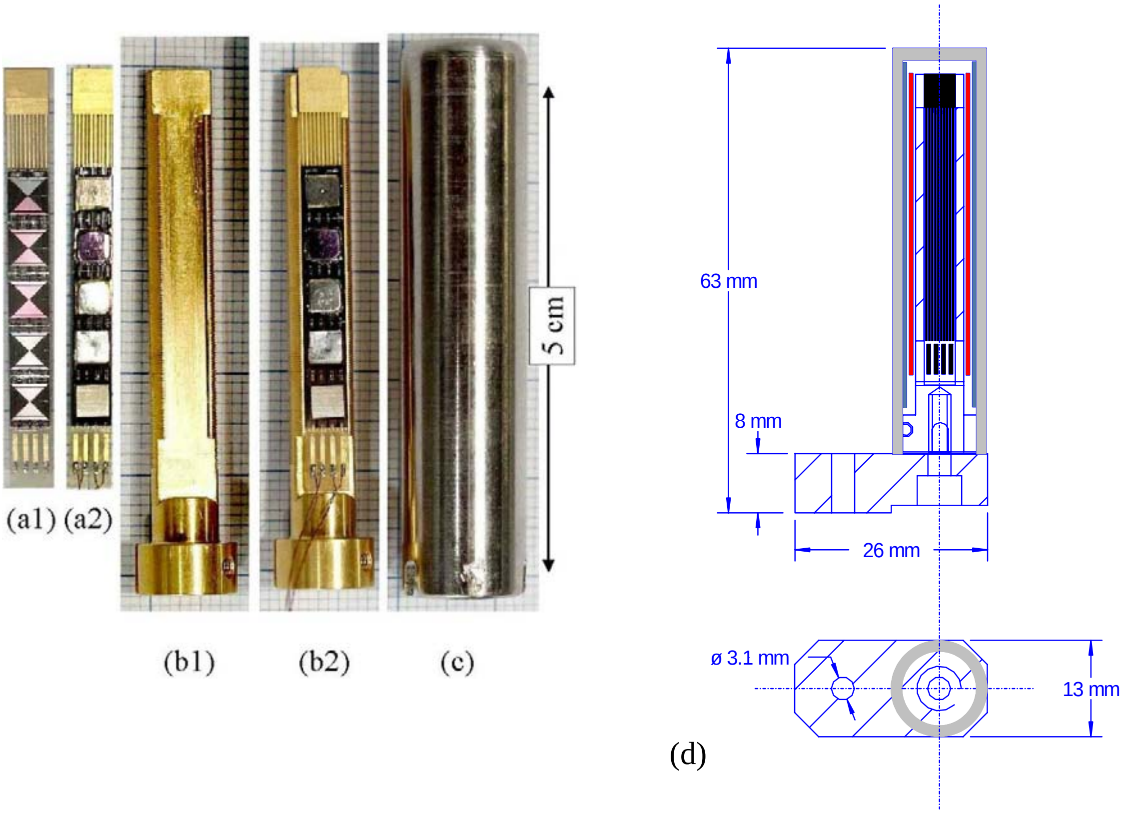


FIGURE 3. SRD1000 test sensor, which consists of two planar detector systems (a1); on each detector five samples are attached (a2); a gold plated copper holder (b1) carries the detector systems (b2); and magnetic shielding (Cryoperm and niobium) surrounds the copper holder (c); the sensor is small enough to fit in most cryogenic applications (d)

5. Development of the SRD1000 electronics

5.1. Mutual inductance measurement system

Figure 4 shows the main unit of the room-temperature electronics *MIDS-10* that was developed to measure the mutual inductance of the SRD1000. A primary AC current of 50 μA_{EFF} and 976.5 Hz is applied to the sensor, resulting in an AC measurement field of about 0.3 μT_{EFF} at the samples. This selection of frequency and current results in a good signal-to-noise ratio for all transitions, while at the same time a very low self-heating of the sensor and a minimal shift of the T_C values is accomplished.

FIGURE 4. The *MIDS-10* main unit of the SRD1000 electronics

The electronics provides a DC output signal proportional to the mutual inductance of the sensor; see Figure 5. Once set, the system does not need any adjustments from the operator to measure all ten transitions. The steps in the staircase function shown in the figure are located at the transition temperatures. For intermediate temperatures, the output voltage level indicates the temperature zone of the sensor.

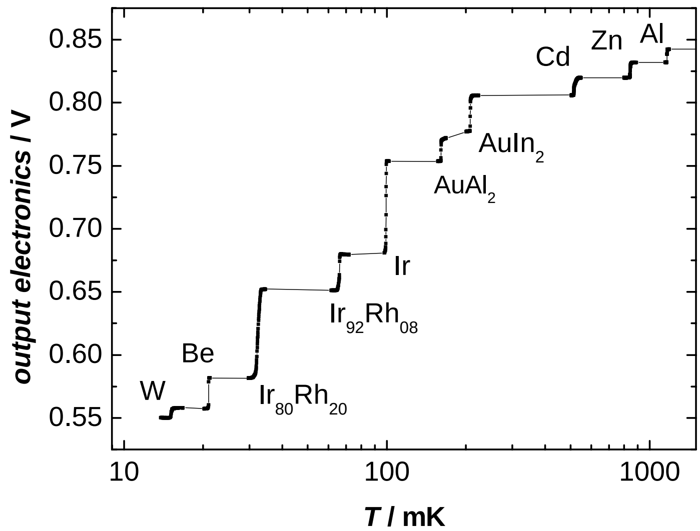


FIGURE 5. *MIDS-10* output signal as a function of the temperature (example for SRD1000 prototype no. 004); the transitions of the ten reference materials are indicated

The circuitries of the current source, amplifiers and AC detector of the electronics are balanced and designed for ultra-low noise operation, enabling a high stability of the output signal (better than 50 ppm/°C) and an excellent immunity from external interferences. The signal-to-noise ratio ranges from about 20 for the W transition to about 200 for the Ir transition.

Five prototypes of the mutual inductance measurement system were constructed, calibrated and tested. Analysis showed that the prototypes enable a high accuracy determination of the SRD1000 transitions. The uncertainty in the determination of *Tc* due to instrumentation uncertainty (transition width, detector signal-to-noise ratio etc.) is expected to be smaller than 0.1% of the temperature reading for most samples (see also Table 4)

5.2. Electronic tools for minimizing residual fields near the samples

Magnetic fields shift the T_C values of the reference samples downwards. Tabel 2 shows the estimated values for the shift dT_C / dB.

Table 2. Estimated field dependence of the reference temperatures

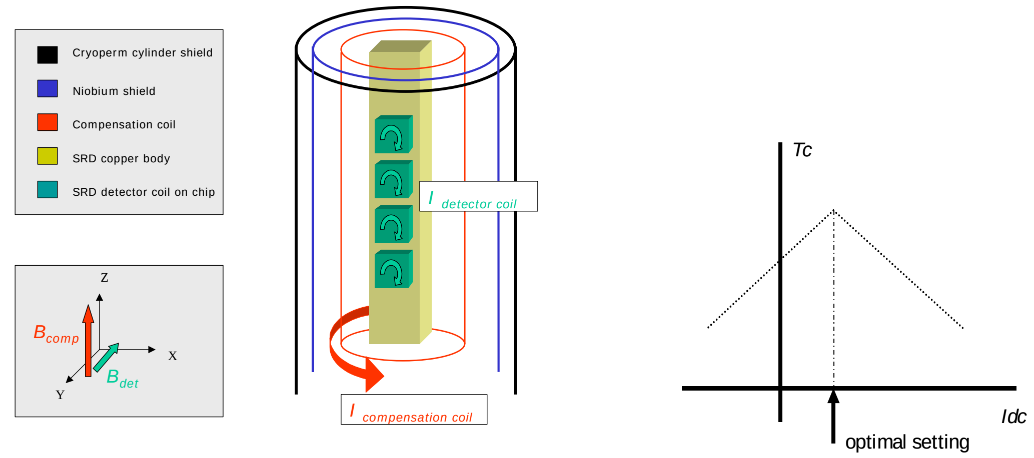
#	material	T_C	dT_C / dB
		[mK]	[mK/uT]
1	W	15	-0.09
2	Be	22	-0.09
3	$Ir_{80}Rh_{20}$	35	-0.01
4	$Ir_{92}Rh_{08}$	62	-0.01
5	Ir	100	-0.05
6	$AuAl_2$	160	-0.13
7	$AuIn_2$	208	-0.08
8	Cd	520	-0.1
9	Zn	850	-0.09
10	Al	1200	-0.06

Two types of electronic tools were developed to minimise and check for residual fields inside the Cryoperm / niobium shielding near the samples:

1) *DCS-10*: an AC current source with a coil to degauss the Cryoperm shielding at room temperature before the start of the measurements;
2) *ACS-10*: a high-precision adjustable DC current source to generate magnetic fields inside the shielding at low temperatures.

Figure 6a gives the shielding and field configuration of the SRD1000. The DC current of the *ACS-10* can be directed through a compensation coil surrounding the samples to check or compensate for field components in the Z-direction. When the current is superimposed on the AC primary current of the SRD detectors, one can also check for field components in the X, Y plane. Because of its very low capacitive impedance and stability the DC current source does not influence the SRD signal quality.

Figure 6b illustrates the analysis of the residual field using the DC current source. For a specific transition, one determines T_C as a function of an additional DC current I_{DC} through the compensation coil or the primary coil. At the maximum of $T_C(I_{DC})$, one will find the optimal setting for the compensation current. Using the coil constant of the compensation coil $B/I_{DC} = 0.012\ T/A$ for the compensation coil or $B/I_{DC} = 0.006\ T/A$ for the detector coil, one can also estimate the residual field value at the sample.



a) b)

FIGURE 6. a) shielding and field configurations of the SRD1000 , b) T_C of a sample versus a DC current I_{DC} in the detector or compensation coil

6. Assembly, calibration and dissemination of SRDs

6.1. Calibration set-up

A set-up was developed which enables an ultra-low temperature calibration of four SRDs at a time. The SRDs are attached to a copper comparator block containing the necessary thermometers to support thermometry see Figure 7: a ^{3}He melting-pressure thermometer (MCT), SRM 768 device #58, a SRM 767 device, a RhFe standard thermometer, Speer carbon and RuO_2 resistance thermometers. The comparator block is connected to the mixing chamber of the dilution refrigerator. All sensors are connected to dedicated measuring equipment. Temperature control and data collection is achieved by a LabView control program.

The residual fields inside the degaussed magnetic shielding of the devices were checked using the $AuAl_2$ transition and the procedure as described in Section 5.2. The residual fields appeared to be less than about 0.2 µT and thus the shifts of T_C were considered small enough to be neglected.

The PLTS-2000 is realized from 10 mK to 1 K by operating the MCT, which was developed by PTB [6, 7]. At the start of each calibration run, the capacitive pressure transducer in the MCT is calibrated by using six reference points: the minimum of ^{3}He melting pressure curve at 315.24 mK and the superconductive transitions of W, Be, Ir, $AuAl_2$ and $AuIn_2$ provided by the SRM 768 device #58, which was previously calibrated by PTB [8].

To record a transition the temperature is increased in discrete steps. Each transition is recorded in at least ten temperature steps. After each step and before a measurement, an interval of at least 5 minutes is taken to ensure thermal equilibrium between the SRDs, the MCT and other thermometers. Later, the transitions are also recorded while the temperature is decreased in order to check for hysteresis.

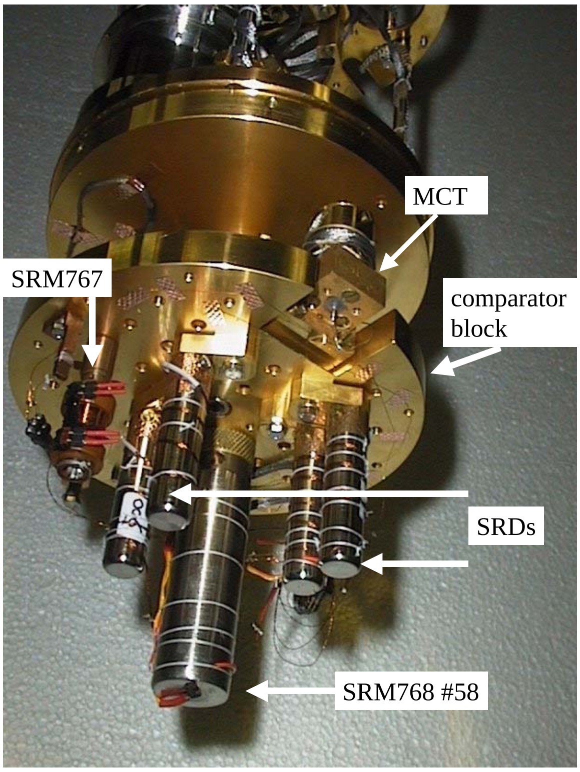


FIGURE 7. The calibration set-up

6.2. Calibration results

Five prototype SRDs (# 003, 004, 005, 006, 007) were assembled and calibrated during ULT test runs 10 and 11. Table 3 gives the T_C and W_C values of the transitions. None of the transitions showed hysteresis. In some prototypes, the $AuAl_2$ and $AuIn_2$ samples have been replaced by other samples of the same materials before the evaluation of the prototypes by other partners.

TABLE 3. Summary of the calibration results of calibration runs 10 and 11

#	material	SRD003 (run 11)		SRD004 (run 11)		SRD005 (run 10)		SRD006 (run 11)		SRD007 (run 11)	
		Tc [mK]	*Wc [mK]*	*Tc [mK]*	*Wc [mK]*	*Tc [mK]*	*Wc [mK]*	*Tc [mK]*	*Wc [mK]*	*Tc [mK]*	*Wc [mK]*
1	W	1)	1)	1)	1)	16.03	0.58	1)	1)	1)	1)
2	Be	21.12	0.11	21.98	0.08	20.98	0.84	20.98	0.51	20.77	0.09
3	$Ir_{80}Rh_{20}$	32.09	0.97	32.32	0.99	31.85	0.5	32.22	0.62	32.18	1.02
4	$Ir_{92}Rh_{08}$	65.52	0.66	66.10	0.94	65.70	0.93	65.93	0.45	65.87	0.89
5	Ir	94.38	1.07	99.34	0.55	99.53	0.38	99.39	0.41	95.59	1.07
6	$AuAl_2$	2)	2)	160.89	0.73	160.93	0.82	160.89	1.7 4)	160.84	1.26 4)
7	$AuIn_2$	207.94	0.94	207.80	1.58	206.24	0.67	207.89	7.04 4)	210.51	9.38 4)
8	Cd	515.9	9.1	516.9	14.0	525.3 3)	17.4	517.7	8.7	518.3	15.2
9	Zn	844.6	8.6	843.7	5.9	819.1 3)	13.7	844.1	9.9	845.2	13.2
10	Al	1165	3.5	1166	3.5	1055 3)	8	1165	5	1200	5

1) these W transitions were not observed in run 11, because the lowest temperature was about 16 mK;
2) the $AuAl_2$ sample shows a transition step too small to be detected and will be replaced before the evaluation of this SRD1000;
3) in run 10 the T_C of SRD005 for Cd, Zn, and Al were determined using a Speer resistor. Later it was found out that the Speer calibration differed considerably from the PLTS-2000 at these transition temperatures;
4) some $AuAl_2$ and $AuIn_2$ samples show (next to a sharp transition) a long tail towards higher temperatures and will be replaced by better quality samples before the evaluation of the SRDs.

6.3. Uncertainty in the realization of the PLTS-2000

A calculation was made to determine the uncertainty $\mu(T_{2000})$ in realizing the PLTS-2000 by using the pressure minimum and five SRM 768 reference points for the calibration of the capacitive pressure transducer in the MCT [14].

Figure 8 shows the results of this calculation. Between 10 mK and 1 K the uncertainty ranges from 0.2 mK to 1 mK, with the exclusion of a range from 250 to 380 mK, centered around the minimum of the melting curve.

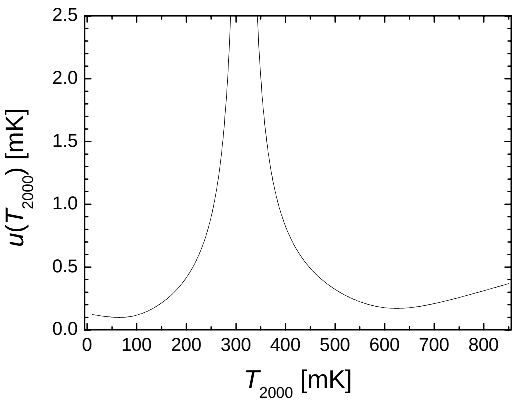


FIGURE 8. Uncertainty in the realization of the PLTS-2000 during the calibration

6.4. Uncertainty in the determination of T_C

All SRD transitions are characterised by a width W_C, height ΔV and signal noise ΔN. These parameters result in an uncertainty ΔT_C when a T_C is determined from a specific transition.

The width W_C of the transition is defined as the temperature interval in which 80% of the total transition step ΔV occurs, see Figure 1. For a symmetrical and smooth transition, one can estimate the slope dT/dV around T_C :

$$dT / dV \cong W_C / 0.8\ \Delta V \qquad (1)$$

The maximum noise at the system output ΔN is about 0.4 $mV_{peak\text{-}peak}$ when the detection electronics is set to the 'slow'-mode. The signal-to-noise (S/N) ratio of a transition is defined as the ratio between the step size ΔV and the noise 0.4 $mV_{peak\text{-}peak}$:

$$S/N = \Delta V / \Delta N = \Delta V / 0.4 \qquad (2)$$

From (1), one can approximate the ratio between the noise related temperature uncertainty ΔT_C around T_C and the system noise ΔN:

$$\Delta T_C / \Delta N = W_C / 0.8\ \Delta V \qquad (3)$$

Using (2), (3) one can derive for ΔT_C , when signal levels are measured in mV:

$$\Delta T_C = W_C \,.\, \Delta N / (0.8\, \Delta V) = W_C / (0.8\, S/N) = Wc / 2\, \Delta V \qquad (4)$$

The range of values for $\Delta T_C / T_C$ observed for the prototypes are listed in Table 1.

6.5. Uncertainty in the reference points

Table 4 gives the total uncertainty in the reference points for SRD1000 prototype no. 004 resulting from the uncertainties ΔT_C and $\mu(T_{2000})$. These results are typical for all SRD1000 prototypes. The uncertainty ΔT_C is relatively small compared to $\mu(T_{2000})$, except for the Zn and Cd transitions. The uncertainty in the reference points is comparable with the standard uncertainty of the PLTS-2000 in thermodynamic terms, which is specified as 0.5 mK down to 500 mK, 0.2 mK at 100 mK and 0.3 % of *T* at 25 mK.

TABLE 4. Estimated total uncertainty in the T_C resulting from ΔT_C and $\mu(T_{2000})$ for SRD004

#	material	T_C	W_C	ΔV	S/N	ΔT_C		$\mu(T_{2000})$		*total uncertainty*
		[mK]	[mK]	[mV]	[-]	[mK]	[%]	[mK]	[%]	[%]
1	W	15.1	0.2	8	20	0.013	0.083	0.120	0.80	0.88
2	Be	21.98	0.08	24	60	0.002	0.008	0.115	0.52	0.53
3	$Ir_{80}Rh_{20}$	32.32	0.99	70	175	0.007	0.022	0.109	0.34	0.36
4	$Ir_{92}Rh_{08}$	66.10	0.94	29	73	0.016	0.025	0.099	0.15	0.18
5	Ir	99.34	0.55	73	183	0.004	0.004	0.116	0.12	0.12
6	$AuAl_2$	160.89	0.73	17	43	0.021	0.013	0.248	0.15	0.16
7	$AuIn_2$	207.80	1.58	28	70	0.028	0.014	0.465	0.22	0.23
8	Cd	516.9	14	14	35	0.500	0.097	0.285	0.06	0.16
9	Zn	843.7	5.9	12	30	0.246	0.029	0.362	0.04	0.07
10	Al	1166	4	10	25	0.200	0.017	-	-	$\cong$ 0.02

7. Conclusion

During the project, five SRD1000 prototypes with dedicated electronics were designed, built, tested and calibrated to develop knowledge on SRD technology. Each prototype contains ten reference materials with clear superconductive transitions.

Analysis shows, that the relative uncertainty in the realization of a reference temperature resulting from the combined uncertainties of the SRD1000 instrumentation and the calibration set-up, ranges between 0.9 % at 15 mK to less than 0.1 % at 1 K. This meets the requirements on the reference points to support and disseminate the PLTS-2000, given the uncertainty of the scale.

In a subsequent development phase, the SRD1000 prototypes will be metrologically evaluated by the other partners of the project. The transition temperatures on the PLTS-2000 will be found, the width and repeatability of the superconductive transitions will be determined and the effects of thermal and measuring parameters will be finalized.

The sample preparation processes still need some additional development in order to be able to manufacture a next series of SRDs of equal or better quality.

8. References

1. Rusby, R. L., Durieux, M., Reesink, A. L., Hudson, R. P., Schuster, G., Kühne, M., Fogle, W. E., Soulen, R. J. and Adams, E. D., *Journal of Low Temperature Physics*, 2002, **126**, 633-642.

2. Schooley, J. F., Soulen, R. J. Jr, and Evans, G. A. Jr, *Standard Reference Materials: Preparation and Use of Superconductive Fixed-Point Devices, SRM 767,* NBS Special Publication 260-44, 1972, pp. 1-35.

3. Soulen, R. J. Jr, and Dove, R. B., *SRM 768: Temperature Reference Standard for Use below 0.5 K*, NBS Special Publication 260-62, U. S. Govt. Printing Office, Washington, D. C., 1979, pp. 1-37.

4. El Samahy, A. E., Durieux, M., Rusby, R. L., Kemp, R. C., and Kemp, R. G., "Realizations of the Superconductive Transition Points of Lead, Indium, Aluminium, Zinc and Cadmium with SRM 767 Devices," in *Temperature: Its Measurement and Control in Science and Industry*, edited by J. F. Schooley, AIP, New York, 1982, **5**, pp. 261-265.

5. Fellmuth, B., in *Temperature: Its Measurement and Control in Science and Industry*, Instrument Society of America, Pittsburgh, 1992, **6**, pp. 233-238.

6. Hoffmann, A., and Schuster, G., "Design aids for the operating system of a ^{3}He melting curve thermometer," in *Progress Report European Ultra-Low Temperature Scale and Traceability*, contract no. SMT4-CT96-2052, November 1996.

7. 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.

8. Hechtfischer D., and Schuster G., "Calibration of SRM 768 superconductive fixed point devices", Technical Report of EU project "European Ultra-Low Temperature Scale and Traceability", Contract no. SMT4-CT96-2052, 1999.

9. Storm, A. J., Bosch, W. A., de Groot, M. J., Jochemsen, R., Mathu, F., and Nieuwenhuis, G. J., "SRD1000: A New Superconductive Reference Device for thermometry below 1000 mK" in *Proceedings of the 7th International Symposium on Temperature and Thermal Measurements (TEMPMEKO 1999)*, edited by J. Dubbeldam et al., NMi van Swinden Laboratorium, Delft, 1999, pp. 142-146.

10. Storm, A. J., Bosch, W. A., de Groot, M. J., Jochemsen, R., Mathu, F., and Nieuwenhuis, G. J., "A New Superconducting Reference Device for Thermometry below 1000 mK", Physica B 284-288 (2000), pp. 2008-2009.

11. Bosch, W. A., Chinchure, A., Flokstra, J., de Groot, M. J., van Heumen, E., Jochemsen, R., Mathu, F., Peruzzi, A., and Veldhuis, D., "Status Report on the Development of a Superconductive Reference Device for Precision Thermometry below 1 K", in *Proceedings of the 8th International Symposium on Temperature*

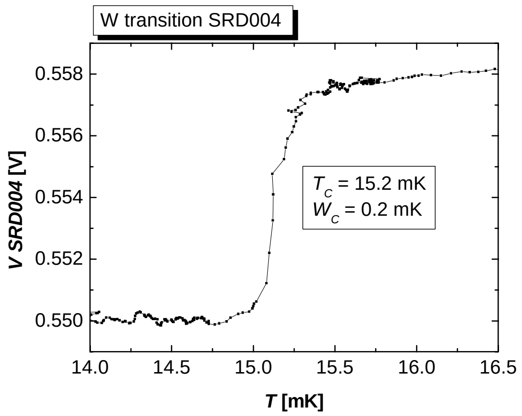
and Thermal Measurements in Industry and Science (TEMPMEKO 2001), edited by B. Fellmuth et al., VDE Verlag, Berlin, 2001, pp. 397-401.

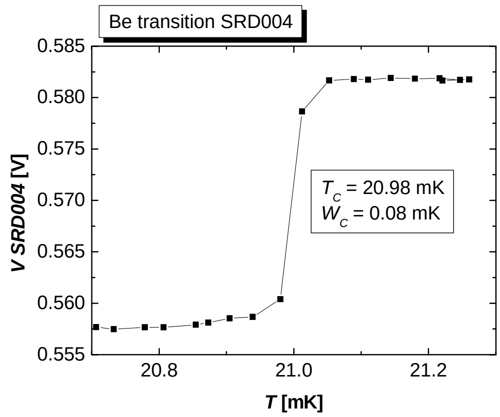
12. Bosch, W. A., Chinchure, A., Flokstra, de Groot G.E., J., de Groot, M. J., van Heumen, E., Jochemsen, R., Mathu, F., Peruzzi, A., and Veldhuis, D., “A Superconductive Reference Device for thermometry below 1K” , poster presented at the LT23 conference Japan.

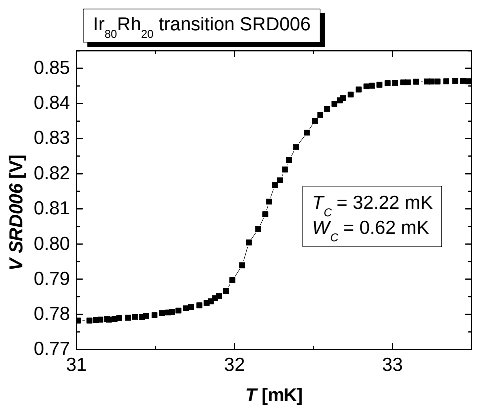
13. Bosch, W.A., Flokstra, J., de Groot, G. E., de Groot, M.J., Jochemsen, R., Mathu, F., Peruzzi, A., and Veldhuis, D., “First Prototypes of the Superconductive Reference Device SRD1000”, in *Proceedings of the 8th Symposium on Temperature: Its Measurement and Control in Science and Industry*, Chicago, 2002.

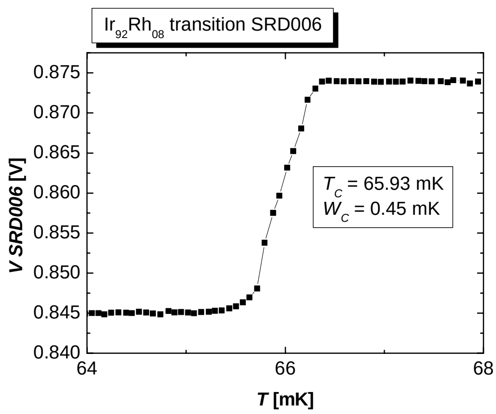
14. Peruzzi, A., de Groot, M.J., “Evaluation of Uncertainty in the Realization of the Provisional Low Temperature Scale from 10 mK to 1 K at NMi”, in *Proceedings of the International Conference on the Uncertainty of Measurement UNCERT 2003*, St. Catherine’s College, Oxford, UK: 9-10 April 2003; this paper was also submitted to the CCT in April 2003.

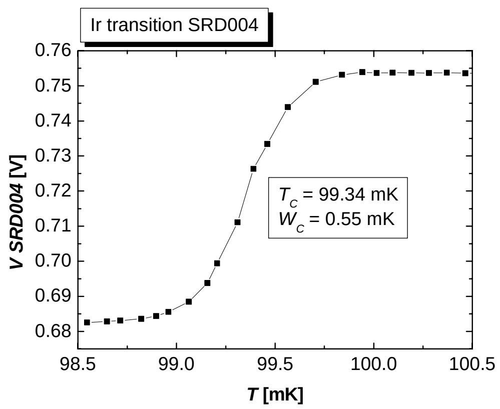
Appendix. Transitions of samples of the selected materials

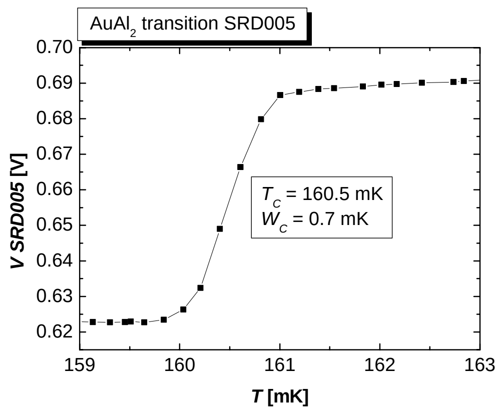


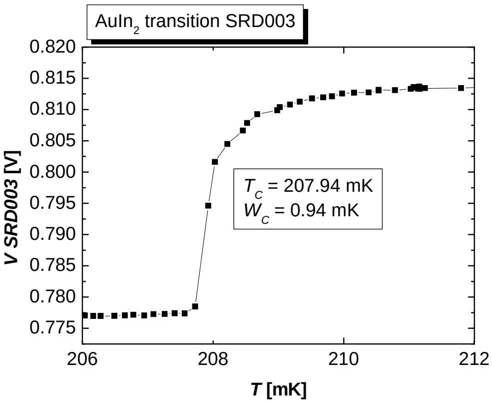


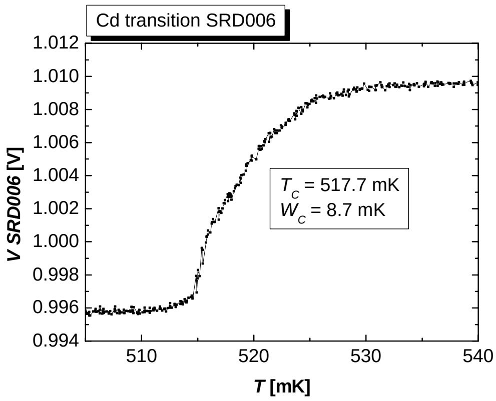


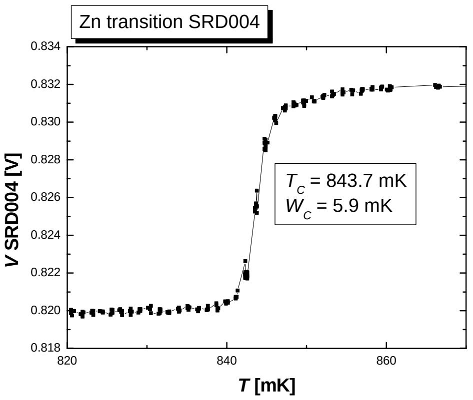


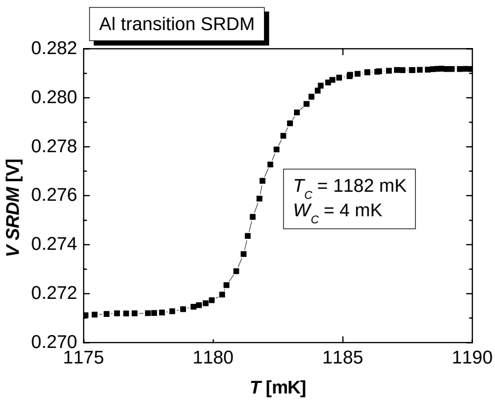





AuIn2 transition SRD003
T_C = 207.94 mK
W_C = 0.94 mK
V SRD003 [V]
T [mK]
0.820
0.815
0.810
0.805
0.800
0.795
0.790
0.785
0.780
0.775
206
208
210
212


Cd transition SRD006
T_C = 517.7 mK
W_C = 8.7 mK
V SRD006 [V]
T [mK]
1.012
1.010
1.008
1.006
1.004
1.002
1.000
0.998
0.996
0.994
510
520
530
540


Zn transition SRD004
T_C = 843.7 mK
W_C = 5.9 mK
V SRD004 [V]
T [mK]
0.834
0.832
0.830
0.828
0.826
0.824
0.822
0.820
0.818
820
840
860


Al transition SRDM
T_C = 1182 mK
W_C = 4 mK
V SRDM [V]
T [mK]
0.282
0.280
0.278
0.276
0.274
0.272
0.270
1175
1180
1185
1190