# Dissemination of the European Ultra Low Temperature Scale
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# REPORT

WP 5
Evaluation of superconductive reference devices

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"Promoting Competitive and Sustainable Growth"

"Measurement and Testing"

RTD Project

# 1. Experimental Set-up

## *1.1. Cryostat*

The evaluation of the SRD1000 superconductive reference device was carried out in a dilution refrigerator located inside a shielded room. All thermometers, reference-point devices, and the SRD1000 device N° 004 were tightly connected to a comparison block made of oxygen free copper. The block was fastened to the mixing chamber of the dilution refrigerator. Its temperature was stabilised by a temperature controller.

The measurement equipment consisted of a number of self-made conductance bridges, mutual-inductance bridges and temperature controllers, a commercial resistance / mutual inductance bridge LR-700, a self-made capacitance bridge, a commercial ASL-F18 LC (low current) ac resistance bridge, the mutual inductance detection system MIDS-10, and the adjustable current source ACS-10 from HDL. The data acquisition from as well as the control of the bridges and the temperature controllers was realised using programmable μ-controllers. The data was transferred via optical lines to outside the shielded room where it was collected and displayed by a PC. The data acquisition program on the PC also allowed operating the temperature controller from outside the shielded room. The output voltage of the MIDS-10 was measured by a DVM and the data was transferred to the PC outside the shielded room also via optical lines.

The SRD1000 N° 004 device was not only fastened to the comparison copper block, but in addition, its leads were wound several times around a copper rod and glued to it by GE-varnish for heat sinking at the temperature of the copper block. For the electrical connection from the copper block up to the top flange of the cryostat, three shielded twisted pairs of leads (for the primary, secondary, and compensation coils) were used which were heat sunk at several temperature levels. Between the top flange feed-through of the cryostat and the MIDS-10, shielded cables supplied by HDL were used.

## *1.2. Melting-Pressure Thermometer, PLTS-2000*

The SRD1000 device was tested against our realisation of the PLTS-2000 and a copy of the PTB-96 [1]. The PLTS-2000 was realised by means of a Melting-Pressure Thermometer (MPT) as described in detail in [2]. The expanded relative uncertainty (coverage factor $k = 2$) of the realisation rises from about 0.01% to 0.3% in the temperature range from 1 K to 0.015 K [3,4]. One Rhodium-Iron Resistance Thermometer (RIRT) carried a copy of the PTB-96 and was used for checking purposes. Another RIRT carrying a PTB realisation of the ITS-90 was used for recording the Al transition at 1.17 K. Before the evaluation experiments, it was carefully checked that the realisation of the PLTS-2000 and the copy of the PTB-96 coincided on a level better than 100 μK in the temperature range above the $^3$He melting-pressure minimum. A Carbon Resistance Thermometer (CRT) and several superconductive reference points were also applied for performing additional checks.

The realisation of the PLTS-2000, i.e. the measurement of temperatures $T_{2000}$, at the highest level of uncertainty is time consuming. It was done in the following way. Before the experiments at temperatures below 1 K, i.e. in the working range of the dilution refrigerator and definition range of the PLTS-2000, the MPT sensor was trained at a temperature near 1.4 K between 2.9 MPa and

4.1 MPa. After that, the MPT sensor was calibrated using a quartz-oscillator pressure transducer (QPT) calibrated against a pressure balance. The uncertainty components caused by the hydrostatic head correction and the determination of the effective area of the pressure balance were eliminated by adjusting the QPT calibration to the pressure value at the minimum of the $^3$He melting curve and to pressure values corresponding to the transitions of calibrated superconductive reference-point samples. The pressure values were assigned to the reference temperatures according to the PLTS-2000. As reference points we used the superconductive-to-normal-conductive transition of tungsten (0.015 K, ~3.38 MPa) and molybdenum (0.918 K, ~3.8 MPa). In connection with the minimum of the melting curve, these points cover nearly the whole pressure range of the PLTS-2000 if the MPT is used from 0.015 K to 1 K. The non-linearity and the hysteresis of the MPT sensor used are very well known and a three-point pressure adjustment of the QPT calibration was sufficient. Because of the given ratio of free volume to sinter volume, the operation of this special MPT sensor requires different filling pressures for different working ranges in temperature. Thus, the MPT calibration was checked several times at the minimum and at superconductive reference points using different fillings of the MPT sensor. In addition, the temperature values $T_{2000}$ were permanently compared with temperature readings $T_{90}$ and $T_{\mathrm{PTB\text{-}96}}$ of the RIRTs carrying copies of the scales PTB-96 and ITS-90 and / or temperature readings of a very stable CRT. This was done to ensure that the MPT is on the melting curve, i.e. not overfilled, and to detect any possible shift of the pressure calibration.

### *1.3. Experimental procedure*

Before describing our experimental procedure, we want to mention that only after the evaluation experiments at PTB were finished we have received the “Procedure for Evaluation of SRD1000 Prototypes” prepared by A. Peruzzi and W. Bosch [5]. Nevertheless, the experiments were carried in a way that is in complete agreement with this guideline.

The SRD1000 device has been evaluated in the following way. First, after the cool-down of the cryostat to liquid helium temperatures, it was checked that the equipment operated correctly. Then, the cryostat was further cooled down to temperatures below 15 mK. During the cool-down, a complete sweep through all of the superconductive transitions was taken in order to have a rough estimate about the transition temperatures and heights of the signal changes at the transitions for each of the samples. Next, the temperature of the comparison block was stabilised at a value slightly below that of a selected superconductive transition where the output signal of the MIDS-10 showed no temperature dependence, i.e. the selected sample was completely in the superconductive state. After that, the temperature of the comparison block was changed using staircase pattern sweeps up and down in temperature. The range for the temperature sweep was chosen in a way to cover the whole transition from the superconductive state to the normal-conductive state and to record a sufficient number of points along the sensitive part of the transition. Each temperature plateau of the staircase pattern sweep lasted at least 30 minutes. This ensured that for all temperature data points measured at the end of the plateaus, thermal equilibrium was reached between the SRD1000 device and the thermometers used. During all the experiments, the averaging time of the data acquisition system was set to 1 min. For the superconductive transitions of $Ir_{80}Rh_{20}$, $Ir_{92}Rh_{08}$, Ir, $AuAl_2$, $AuIn_2$, Cd, and Zn the temperature stabilisation at the plateaus of the staircase pattern was realised using a CRT and a PID temperature controller. The transitions of W and Be were recorded in another way. Here, we have stabilised the temperature at the midpoint of the superconductive transition of our own W and Be reference points, the transition temperatures of which are slightly higher than those of the corresponding transition temperatures of the SRD1000 device. A constant current was sent through the primary coil of our reference-point devices in order to shift down the transition temperature. By stepwise rising up and lowering down the constant current we were able to go through the W and Be transitions of the SRD1000 N°004 device. Because the temperature of the Al transition is outside the working range of the dilution refrigerator, the Al transition was recorded in slowly drifting up in temperature the whole insert of the cryostat. During the warm-up, the data points were taken every 30 μK. Except for the Al transition, each of the superconductive transitions was recorded up and down in temperature at least twice.

### *1.4. Measurements with the SRD1000 and MIDS-10*

The MIDS-10 was only used in the filter mode “slow”. It was checked that the operation of the device did not affect our other measurement equipment. Even at the lowest temperature around and below 10 mK we have found no indication for any interference between the MIDS-10 and other devices.

We have checked the magnetic shielding of the SRD1000 device and tested it for residual magnetic fields possibly present at the position of the reference-point samples. This was done by setting the temperature stabilisation at the midpoint of the superconductive transition of a selected sample. Then, a constant current was sent through the compensation coil of the SRD1000 device that was stepwise changed in the range from approximately -3 mA to 3 mA.. The complete compensation of residual fields in direction of the compensation coil corresponds to the current value for which the maximum of the temperature reading $T_{2000}$ is observed. As an example, Figure 1 shows the shift of the uncorrected transition temperature of the $AuAl_2$ sample in dependence on the current $I_{comp}$ through the compensation coil. The maximum transition temperature is found for nearly zero compensation current, i.e. the suppression of the external magnetic fields in the direction of the compensation coil (z-direction) by the shielding of the SRD1000 device is quite good. An analogous result was found for the $Ir_{80}Rh_{20}$, $Ir_{92}Rh_{08}$, Ir, $AuAl_2$, $AuIn_2$, and Cd transitions. The shielding in the x-y-direction was also checked for the $AuIn_2$ transition by connecting the ACS-10 to the corresponding input of the MIDS-10 preamplifier and watching the temperature readings in dependence on the current. The result was the same as for the field compensation in the z-direction. Therefore, during all experiments no additional field compensation was applied.

## 2. Results

The determination of the transition temperatures of the reference-point samples is illustrated in Figure 2 for the reference data supplied by HDL for the superconductive transition of Zn. From the record of the voltage output signal of the MIDS-10 electronics in dependence on temperature the voltage values in the completly normal- and superconductive state, $V_N$ and $V_{SC}$ respectively, were determined. The midpoint of the transition was taken equal to $(V_N + V_{SC})/2$ and is represented by the red horizontal line in Figure 2. Then, all voltage versus $T_{2000}$ data points were approximated by an interpolating function using *Mathematica*. This corresponds to the construction of a fitting polynomial for the input data. The uncorrected superconductive transition temperature $T_c^*$ was taken as the intersection of the interpolating function with the midpoint value $(V_N + V_{SC})/2$ and is shown by the red vertical line in Figure 2. The width of the superconductive transition $\Delta T_c$ was taken from the intersection of the interpolating function with the ends of the 80% interval of the voltage change $(V_N - V_{SC})$ centred at the value of $(V_N + V_{SC})/2$. The corresponding blue horizontal and vertical lines are also shown in Figure 2. The use of interpolating functions of different "Interpolation Order" in *Mathematica* yielded $T_c^*$ values differing by no more than 15 µK. The absolute values for $T_c^*$ and $\Delta T_c$ derived in this way differ somewhat from the values supplied by HDL (HDL values: $T_c^*$ = 0.8437 K , $\Delta T_c$ = 5.9 mK; PTB values: $T_c^*$ = 0.8439 K, $\Delta T_c$ = 5.6 mK). This may be caused by the procedure how $T_c^*$ was determined and / or because the voltage values for the normal- and superconductive state were chosen slightly different.

During the measurements, the magnetic field of the excitation current of the MIDS-10 causes a depression of the transition temperature values. In Figure 1, this effect is illustrated by the rounding of the $T_c^*$ versus $I_{comp}$ dependence near $I_{comp} = 0$. In order to take into account this $T_c^*$ depression by the excitation current, a correction $\delta T_c$ has to be applied. The value of $\delta T_c$ can be found by taking the intersection of the linear extrapolations of the dependence $T_c^* = f(I_{comp})$ from both the negative and positive current values as the true value for $T_c$. In our experiments, this correction was determined for the $Ir_{80}Rh_{20}$, $Ir_{92}Rh_{08}$, Ir, $AuAl_2$, $AuIn_2$, and Cd transitions by investigating explicitly the dependence of $T_c^*$ on $I_{comp}$. The $\delta T_c$ corrections for the investigated samples were found to be well below the uncertainty of the scale realisation. Therefore, for the W, Be, Zn, and Al samples, for which we did not measure the dependence of the transition temperature on $I_{comp}$, the maximum $T_c^*$ values were taken as $T_c$. In Table 1, the transition temperature $T_c = T_c^* + \delta T_c$ and the transition width $\Delta T_c$ are presented for all reference-point samples of the SRD1000 device N° 004 measured at PTB.

For the investigated SRD1000 device N°004, we have had problems to record repeatedly the transition of the Be reference point. This was caused either by our temperature stabilisation technique in crossing the transition or, possibly, by the relatively poor thermal contact of the Be sample. In one case we even observed a super-cooling of the transition below that of the W sample.

In Figure 3, the output voltage of the MIDS-10 electronics is given in dependence on the temperature $T_{2000}$ for the superconductive transitions of all reference-point samples contained in the SRD1000 device N° 004. For each of the superconductive transitions, the corresponding lines used for the determination of $T_c^*$ and $\Delta T_c$ are also shown.

**Table 1.** Values for the transition temperature $T_c$ and transition widths $\Delta T_c$ for all reference point samples of the SRD1000 device N° 004 measured at PTB. $\delta T_c$ is the correction to uncorrected $T_c^*$ due to the depression by the measuring current of the MIDS10 device (see text).

| ref. point | material | $T_c^*$ K | $\delta T_c$ mK | $T_c$ K | $\Delta T_c$ mK |
|---|---|---|---|---|---|
| 1 | W | 0.01515 | - | **0.0152** | **0.17** |
| 2 | Be | 0.02005 | - | **0.0201** | **0.03** |
| 3 | Ir80Rh20 | 0.03170 | 0.012 | **0.0317** | **1.13** |
| 4 | Ir92Rh08 | 0.06571 | 0.022 | **0.0657** | **0.94** |
| 5 | Ir | 0.09923 | 0.015 | **0.0992** | **0.57** |
| 6 | AuAl2 | 0.16060 | 0.013 | **0.1606** | **0.44** |
| 7 | AuIn2 | 0.2078 | 0.029 | **0.2079** | **0.65** |
| 8 | Cd | 0.5205 | 0.006 | **0.5205** | **14.5** |
| 9 | Zn | 0.8517 | - | **0.8517** | **5.42** |
| 10 | Al | 1.1782 | - | **1.1782** | **1.66** |

## 4. References


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2. .Schuster, G., Hoffmann, A., and Hechtfischer, D., "*Realisation of the temperature scale PLTS-2000 at PTB*", PTB, Braunschweig, PTB-ThEx-21, ISBN 3-89701-742-3, 2001

3. Engert J., Fellmuth B., Hoffmann A., BIPM Com. Cons. Thermométrie 22, Document CCT/03-09 (2003).

4. Engert J., Fellmuth B., Hoffmann A., "Uncertainty Budget for the Realisation and Dissemination of the Provisional Low Temperature Scale PLTS-2000 at PTB", 2nd International Seminar on Low Temperature Thermometry, Wrocław, ISBN: **???**, 2003

5. Peruzzi A., Bosch W.A., "*Procedure for the Evaluation of SRD1000 Prototypes*", Http://www.xs4all.nl.~hdleiden/srd1000

**5. Figures**

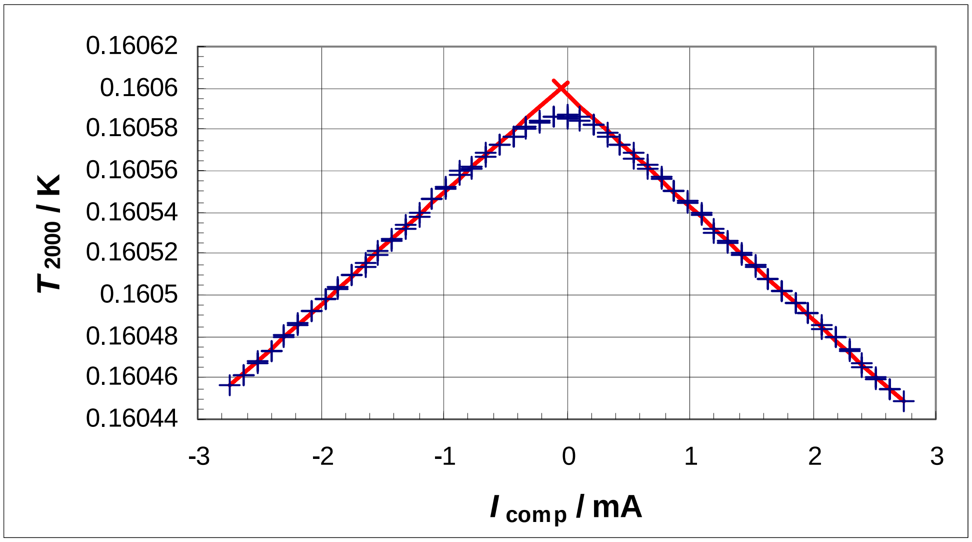


**Figure 1.** Shift of the uncorrected transition temperature of the $AuAl_2$ sample caused by a stepwise change of the current through the compensation coil of the SRD1000 device. The red lines are linear fits for the evaluation of the transition-temperature depression by the measuring current of the MIDS-10 device (see text).

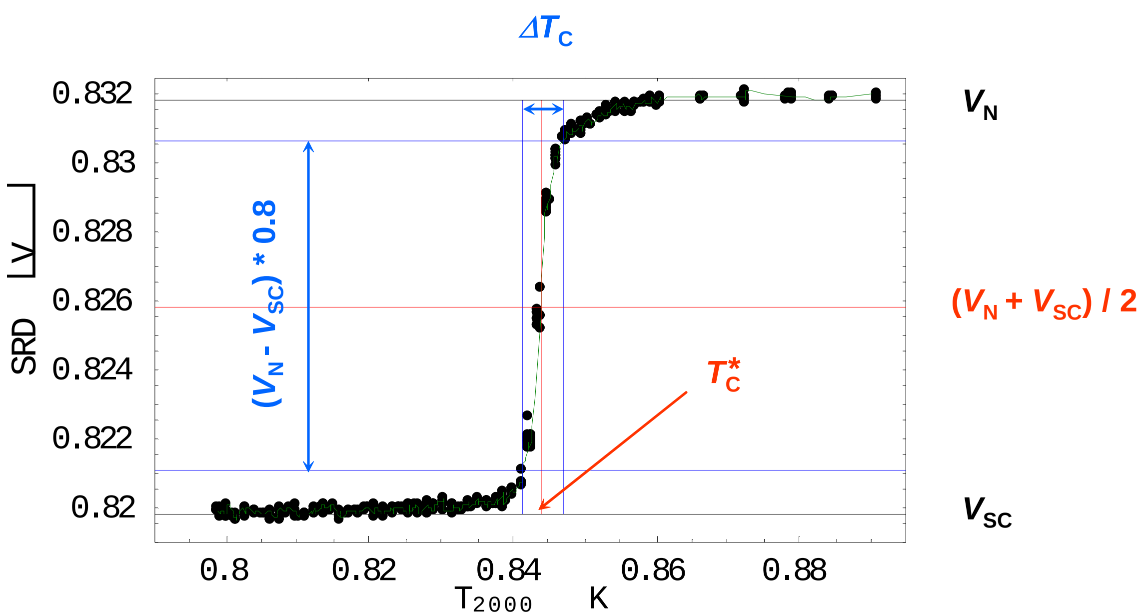


**Figure 2.** Determination of $T_c^*$ and $\Delta T_c$ from the output voltage SRD of the MIDS-10 electronics in dependence on temperature $T_{2000}$ for the superconductive transition of the Zn sample of the SRD1000 device N° 004 measured at NMi. For details see text.

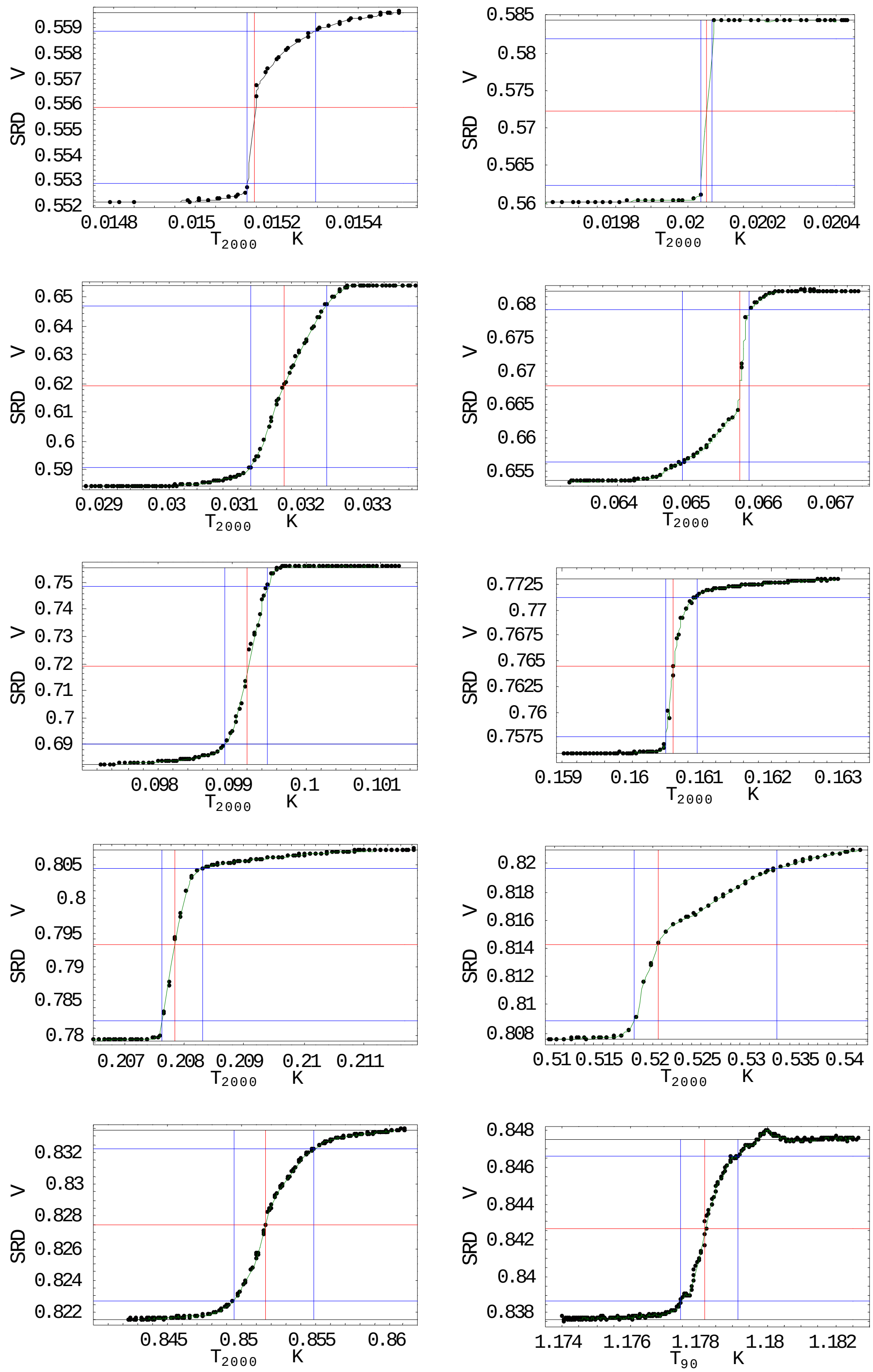


**Figure 3.** The output voltage of the MIDS-10 electronics in dependence on temperature $T_{2000}$ for the superconductive transitions of the W, Be, $Ir_{80}Rh_{20}$, $Ir_{92}Rh_{08}$, Ir, $AuAl_2$, $AuIn_2$, Cd, Zn, and Al samples contained in SRD1000 device N° 004. For details see text.