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Report on evaluation of SRD technology

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“Promoting Competitive and Sustainable Growth”

“Measurement and Testing”

RTD Project

1 Executive summary

This report describes the results of the metrological evaluations performed on four SRD1000 prototype devices. The SRD1000 is a Superconductive Reference Device that generates 10 reference temperatures between 10 mK and 1200 mK. The reference temperatures are provided by the superconductive-to-normal transitions of 10 different reference materials included in the SRD1000 sensor. For the details of the SRD1000 technology, see deliverable 2.1 [1].

The four SRD prototype devices were produced in the previous development phase, preliminary tested and calibrated at NMi VSL, further improved (when needed) and finally transferred to evaluator partners as follows (see Figure 1):

SRD003 to CNRS-CRTBT
SRD004 to PTB
SRD005 to BNM-INM/CNAM
SRD006 to NPL

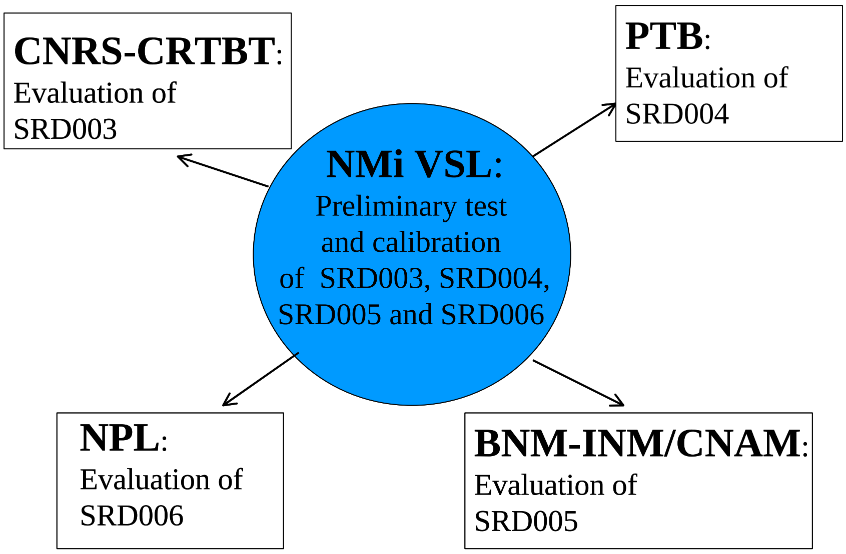


Figure 1: Scheme of the evaluation of the SRD1000 prototypes.

Each partner evaluated the assigned SRD1000 prototype following (when compatible with their experimental constraints) the procedure specified in the protocol [2]. The evaluation consisted mainly of:

- Magnetic field tests to estimate the influence of the residual and measuring fields on the transition temperatures.
- Repeated observations of the transitions under specified controlled thermal conditions.
- Determination of the transition temperature and transition width of each transition.

Details of the evaluation are described in Sections 2 to 6 as follows:

- **Evaluation procedure and measurement conditions (Section 2):** the procedures adopted and the experimental conditions realized by the partners during their evaluations.

- **Thermometry and Uncertainty (Section 3):** the origin of the local temperature scales used and the uncertainty claimed by each partner in the determination of the transition temperature values.
- **Magnetic field tests (Section 4):** investigation of the effects of the residual external magnetic fields and measuring field on the superconductive transitions.
- **Results (Section 5):** Observation of the superconductive transitions, determination of the transition temperatures and transition widths.

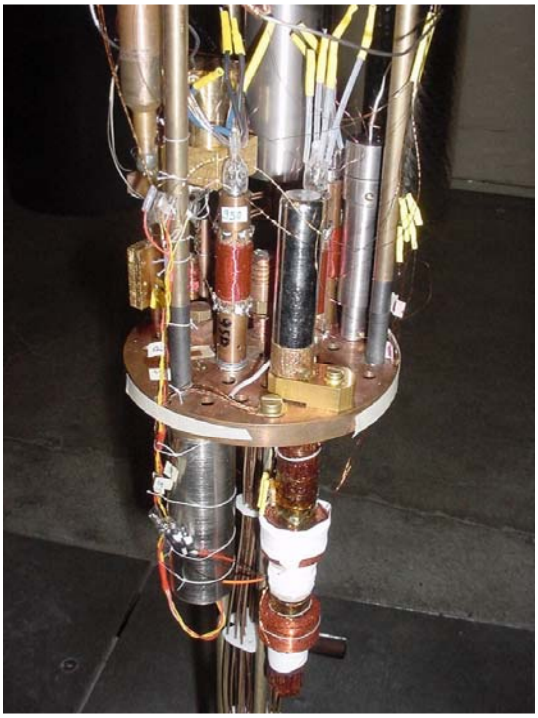
2 Evaluation procedure and measurement conditions

The superconductive transitions of the reference samples were observed by monitoring the DC output voltage of the SRD1000 prototypes as a function of temperature. The details of the procedure followed and the measurement conditions realized by each evaluator are described in the following paragraphs and summarized in Table 1. For comparison, the procedure and the measurement conditions at NMi VSL during the preliminary tests and calibration of the prototypes are also reported in the same table.

	CNRS-CRTBT	**PTB**	**BNM-INM/CNAM**	**NPL**	**NMi VSL**
Minimum temperature	5 mK	7 mK	30 mK	32 mK	12 mK
Temperature control	Weak link + two TRMC2 controllers	Carbon Resistor + PID controller + autotuning	Second sound thermometer + Labview PID control via PC	Weak link + Carbon Resistor + PID	Carbon Resistor + PID controller
Temperature sweep pattern	Staircase (10 min plateaus) + continuous sweep	Staircase, 30 min. plateaus at least	Staircase, 30 min plateaus	Staircase, 8 – 20 min. plateaus, continuous for $Ir_{80}Rh_{20}$	Staircase, 5 – 10 min. plateaus
Temperature stability	Better than 0.01 mK	Better than 0.01 mK	0.2 mK at Al 0.05 mK at Cd 0.02 mK at $Ir_{92}Rh_{08}$ 0.05 mK at $Ir_{80}Rh_{20}$	0.15 mK at Al 0.02 mK at Zn 0.15 mK at Cd 0.03 mK at $AuIn_2$ 0.01 mK at $AuAl_2$ 0.01 mK at Ir 0.03 mK at $Ir_{92}Rh_{08}$	1.0 mK at Al 0.2 mK at Zn 0.09 mK at Cd 0.02 mK at $AuIn_2$ 0.02 mK at $AuAl_2$ 0.04 mK at Ir 0.01 mK at $Ir_{92}Rh_{08}$ 0.01 mK at $Ir_{80}Rh_{20}$ 0.01 at Be 0.01 at W
Passages across SC transition	1 Warming-cooling (staircase) + 1 warming-cooling (continuous)	Warming-cooling	Cooling-warming	Warming-cooling-warming	Warming-cooling
Repeatability tests	Two cool-downs	Two warming-cooling cycles at least for all transitions	Two cooling-warming cycles for all transitions	Two warming-cooling-warming cycles for Al	Many warming-cooling cycles

Table 1: Procedure followed and measurement conditions realized by each evaluator. For comparison also the measurement procedure and conditions at NMi VSL during the preliminary tests and calibration are reported.

Additional details can be found in the reports prepared by each evaluator partner [3,4,5,6]. Pictures of the experimental set-up at NPL and CNRS-CRTBT are shown in Figure 2.



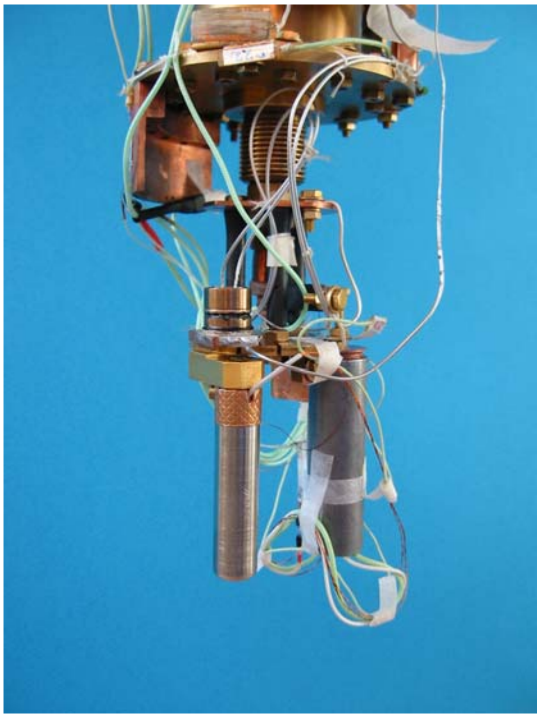


Figure 2: Experimental set-up at NPL (left) and at CNRS-CRTBT (right).

2.1 Minimum temperature

The minimum temperature of CNRS-CRTBT and PTB was respectively 5 mK and 7 mK, which allowed to investigate all the superconductive transitions of the assigned prototypes.

Owing to not completely understood parasitic heat leaks, the minimum temperature that INM-BNM/CNAM could reach was 32 mK, which did not allow them to observe the W, Be and $Ir_{80}Rh_{20}$ transitions.

A similar problem was experienced by NPL, whose lowest temperature was about 30 mK (just below the $Ir_{80}Rh_{20}$ transition). Consequently NPL could not observe the W and Be transitions, while $Ir_{80}Rh_{20}$ transition could be observed only under not perfectly controlled thermal conditions.

2.2 Temperature sweep pattern

For the temperature sweep across the transitions, all the partners adopted a staircase pattern in which small temperature steps (0.1 mK or less) were alternated to stabilization plateaus of several minutes (8-30 minutes each) to allow thermal equilibrium.

A preliminary fast continuous sweeping through the transitions was often performed to localize the range of the transitions to be investigated.

2.3 Passages across the transitions

To investigate the presence of hysteretical behaviours, the passage across the transitions was performed both by warming and cooling.

CNRS-CRTBT and PTB adopted a warming-cooling sequence, BNM-INM/CNAM a cooling-warming sequence and NPL a warming-cooling-warming sequence.

2.4 Repeatability tests

To check for short-term repeatability of the transitions, the selected sequences were repeated on the same cool-down (twice at BNM-INM/CNAM, twice at least at PTB) or after warming up the system to room temperature (CNRS-CRTBT and NPL).

3 Thermometry and Uncertainty

For temperatures below 1 K, the thermometry adopted by each partner in the evaluation of the assigned prototypes originated from the respective local PLTS-2000 [7] realizations.

Differences in the practical realizations of the scale mainly concern the calibration of the capacitive pressure sensor (CPS) for the ^{3}He melting pressure:

- CNRS-CRTBT calibrated the CPS against a quartz-oscillator pressure transducer (QPT) and then adjusted the calibration using the observation of the minimum of the ^{3}He melting pressure curve.
- PTB calibrated the CPS against a QPT that was previously calibrated against a pressure balance. The calibration was than adjusted using the minimum of the ^{3}He melting pressure curve and the superconductive transitions of W and Mo as pressure reference points.
- INM-BNM/CNAM calibrated the CPS against a QPT that was previously calibrated against a pressure balance.
- NPL calibrated the CPS directly against a pressure balance and then adjusted the calibration using the observation of the minimum of the ^{3}He melting pressure curve.
- NMi VSL calibrated the CPS with five superconductive transitions of a SRM768 previously calibrated by PTB on PTB-96 scale and then re-calculated on PLTS-2000.

Above 1 K, PTB, NPL and BNM-INM/CNAM used Rhodium Iron Resistance Thermometers (RIRT) carrying their local realizations of ITS-90. CNRS-CRTBT used a CMN (Ce and Mg Nitrate) thermometer calibrated with a NBS 767a superconductive device.

Additional thermometers and devices were used for cross checks:

- CNRS-CRTBT extrapolated the CMN below 1 K and checked its agreement with their PLTS-2000 realization.
- PTB used RIRT and Carbon Resistance Thermometers (CRT) carrying PTB-96 and PTB ITS-90 scales.
- INM-BNM/CNAM used a Second Sound Thermometer (SST).
- NPL used RIRTs carrying NPL ITS-90 scales and a CMN and a Glass Capacitance thermometer (GCT).
- NMi used a SRM767a to compare the NIST transitions to SRD1000 transitions.

The thermometry adopted by the partners in the evaluation is summarized in Table 2.

Each partner involved in the evaluation of the SRD prototypes was asked to estimate the uncertainty in the determination of the transition temperature values.

The uncertainty in the determination of the transition temperature values is due to:

1. The uncertainty in the local realization of T_{2000}.
2. The uncertainty arising from temperature uniformity and stability of comparator block.
3. The uncertainty due to the residual and measuring magnetic fields.
4. The uncertainty in the identification of the midpoint of the transition.

The resulting expanded uncertainty claimed by the evaluators at each reference temperature is reported in Table 3.

	CNRS-CRTBT	**PTB**	**BNM-INM/CNAM**	**NPL**	**NMi VSL**
T < 1 K	MPT + Digiquartz + minimum melting curve	MPT + Digiquartz + pressure balance + minimum melting curve + W + Mo	MPT + Digiquartz + pressure balance	MPT + pressure balance + minimum melting curve	MPT + minimum melting curve + calibrated SRM768
T > 1 K	CMN	RIRT carrying PTB ITS-90	RIRT carrying NPL ITS-90	RIRT carrying NPL ITS-90	RIRT carrying NPL ITS-90
Cross checks	CMN and NBS 767a	RIRT carrying PTB-96, CRT and superconductive reference points	SST	RIRT carrying ITS-90, CMN and GCT	SRM767a

Table 2: Thermometry adopted by the partners in the evaluation of the SRD1000 prototypes. MPT stands for Melting Pressure Thermometer, RIRT for Rhodium Iron Resistance Thermometer, CRT for Carbon Resistor Thermometer, SST for Second Sound Thermometer, GCT for Glass Capacitance Thermometer. Last column refers to preliminary tests and calibration of the prototypes at NMi VSL.

	CNRS-CRTBT /mK	PTB /mK	BNM-INM/CNAM /mK	NPL /mK	NMi VSL /mK
W	0.04 (0.03)	0.06 (0.04)	-	-	0.2
Be	0.04 (0.03)	0.06 (0.04)	-	-	0.2
$Ir_{80}Rh_{20}$	0.04 (0.03)	0.24 (0.06)	-	0.12 (0.06)	0.2
$Ir_{92}Rh_{08}$	0.06 (0.04)	0.20 (0.06)	0.6 (0.2)	0.10 (0.08)	0.2
Ir	0.06 (0.05)	0.14 (0.06)	0.4 (0.1)	0.12 (0.10)	0.2
$AuAl_2$	0.08 (0.07)	0.14 (0.10)	0.4 (0.1)	0.16 (0.12)	0.5
$AuIn_2$	0.16 (0.14)	0.20 (0.14)	0.4 (0.1)	0.32 (0.24)	0.9
Cd	0.18 (0.12)	2.90 (0.12)	4.0 (0.1)	0.74 (0.20)	0.6
Zn	0.1 (0.06)	1.08 (0.06)	6.0 (0.2)	0.58 (0.10)	0.8
Al	4 (4*)	0.48 (0.36*)	1.4 (0.2*)	1.1 (1.0*)	5.0

* Uncertainty in terms of local ITS-90 realization.

Table 3: Expanded uncertainty (k = 2) reported by the evaluators in the determination of the transition temperature of each reference point. In parenthesis the uncertainty component from the PLTS-2000 realization only is reported. Last column refers to preliminary tests and calibration of the prototypes at NMi VSL.

The evaluation of the uncertainty component arising from the identification of the transition midpoint is under discussion among the partners.

This component results from many contributions:

- The signal to noise ratio of the output voltage of the SRD1000 sensor.
- The localization of the output voltages corresponding to the full superconductive (V_S) and full normal (V_N) states.
- The number of experimental points available along the sensitive part of the transition (which is related to the temperature control stability achieved).
- The mathematical function adopted to interpolate between the experimental points.

To take into account all these contributions PTB added an uncertainty component equal to 1/10 of the transition width. Consequently the overall uncertainty resulted often much higher for PTB than for other partners (see table 3), although the uncertainty in the PTB scale realization was comparable to (or better than) that claimed by the other partners.

4 Magnetic field tests

The transition temperature T_C of the reference samples is depressed by magnetic fields. For the reference materials of the SRD1000 the field dependence of T_C is estimated to be about – 0.1 mK/μT.

The partners performed magnetic field tests on the assigned SRD1000 prototypes in order to:

- Estimate the residual external field present on the reference samples and verify the effectiveness of the SRD1000 shielding in screening external magnetic fields.
- Estimate the T_C depression produced by the AC measuring field.

The tests were performed in the two following ways (see [2]):

1) By observing the T_C depression when DC currents were superimposed to the AC primary current in the detector coil, to check for field components in the Y direction (see Figure 3).
2) By observing T_C depression when DC currents were directed through the compensation coil, to check for field components in the Z direction.

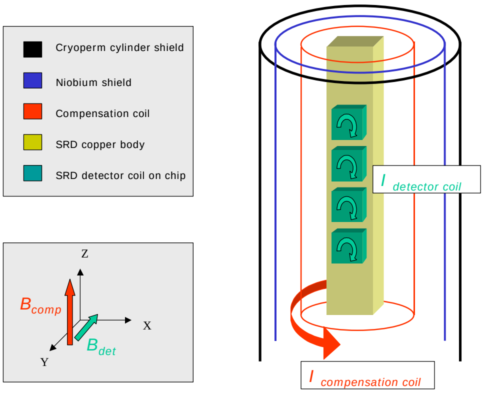


Figure 3: Shielding and field configurations of the SRD1000.

Using the coil constants of the compensation coil and detector primary coil, the residual field components could be estimated and the corrections for the depression of the transition temperature values due to the measuring field could be applied.

		CNRS-CRTBT	PTB	NPL
Y direction	B^{meas}	0.3 μT	0.3 μT	0.3 μT
	ΔT_c^{meas}	0.018 mK	0.013 mK	0.022 mK
	B^{res}	< 0.1 μT	< 0.1 μT	0.2 – 0.3 μT
	ΔT_c^{res}	< 0.006 mK	≤ 0.005 mK	0.020 mK
Z direction	B^{res}	< 0.1 μT	0.4 μT	0.2 – 0.5 μT
	ΔT_c^{res}	< 0.006 mK	≤ 0.005 mK	< 0.010 mK

Table 4: Magnetic field tests performed by the evaluator partners on the $AuAl_2$ reference sample. Y direction test refers to DC currents superimposed to the AC primary current of the SRD detector, Z direction test refers to DC currents directed through the compensation coil. B^{meas} is the excitation field, ΔT_c^{meas} is the depression of the transition temperature due to the excitation field, B^{res} is the residual external field and ΔT_c^{res} is the depression of the transition temperature due to the residual external field.

In Table 4 the results of the magnetic field tests are summarized. We conclude that the correction for the depression of the transition temperature, due to the residual external field and to the excitation field, is well below the uncertainty in the scale realization and can be neglected.

5 Results

The superconductive transitions observed by the partners during the evaluation are shown in Figure 4. To compare the transitions observed in different devices, the voltage output from the SRD1000 was reported as percentage of the total voltage change along the transition (0% corresponds to full superconducting state and 100% corresponds to full normal state).

The differences between transition temperatures of reference samples of the same material reflect differences in the preparation techniques and non-homogeneity of samples prepared from the same batch. For this reason each device needs to be individually calibrated.

Most of the reference samples exhibited smooth and sharp transitions with the exceptions of cadmium and zinc. Additional research in the preparation of reference samples of these materials is on-going.

PTB could not record properly the Be transition (no experimental point along the sensitive part of the transition) and in one case they also observed a supercooling of the Be transition. Possible explanations are a poor thermal contact between the sample and the copper holder of the SRD1000 sensor and an insufficient suppression of supercooling caused by poor Al spot welding.

CNRS/CRTBT observed hysteresis between warming and cooling on the tungsten transition. NPL observed a non-reproducibility of the warming passage of iridium transition of the level of 0.01 mK.

The transition temperatures and widths determined by the partners are reported in Table 5. In the same table, the transition temperature and width values as determined by NMi VSL during the preliminary tests and calibration are reported in parenthesis.

The differences between the transition values determined by NMi VSL and those determined by the other labs for the same samples in some cases significantly exceeded the uncertainties claimed. An overview of these differences is shown in Figure 5 for the PLTS-2000 range.

The discrepancies were attributed to a not optimised thermometry at NMi VSL during the preliminary tests and calibration of the prototypes. For this reason no conclusion was drawn about the long-term reproducibility of the observed superconductive transitions.

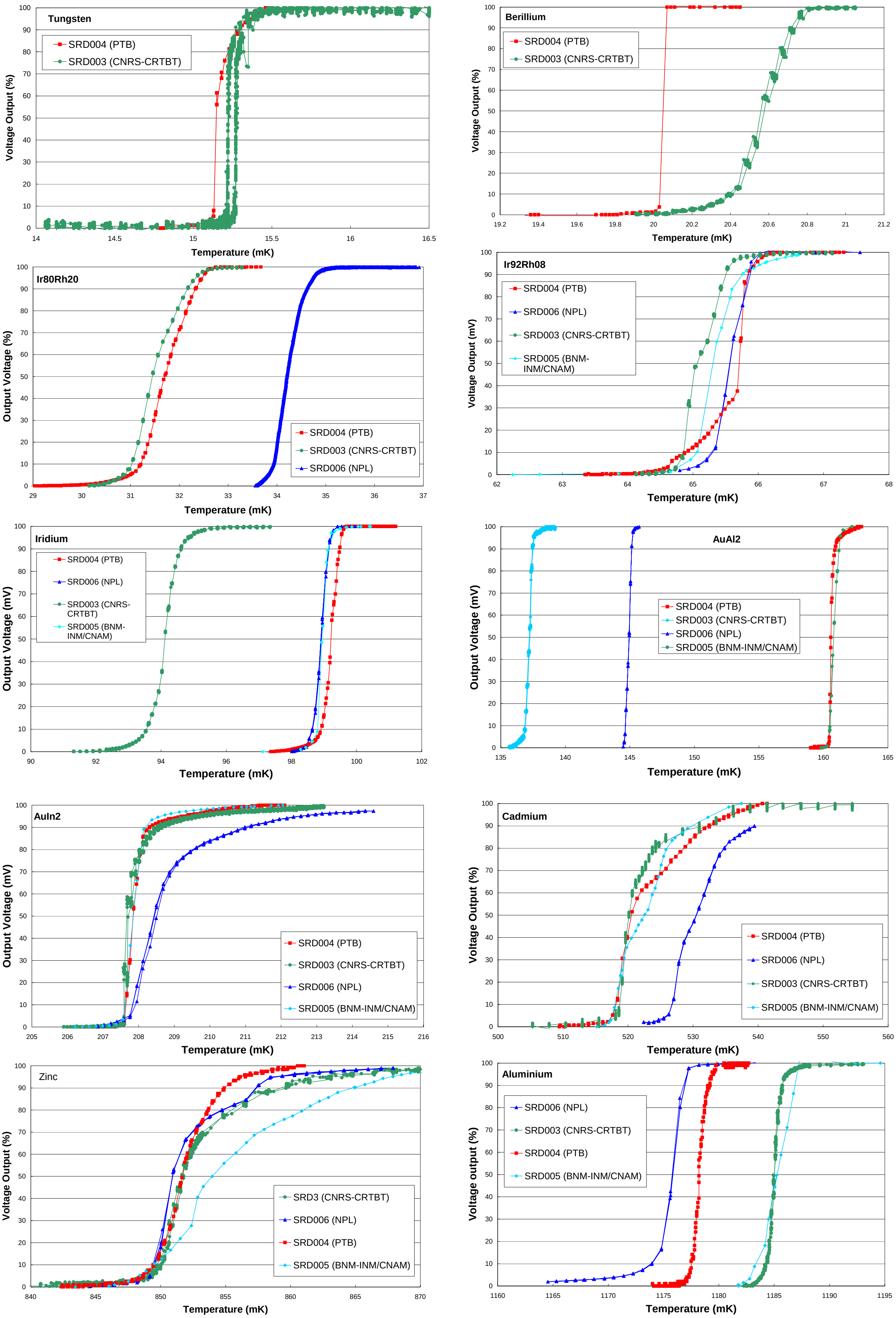


Figure 4: Superconductive transitions observed by the partners during the evaluation.

	SRD003 (CNRS-CRTBT)		SRD004 (PTB)		SRD005 (BNM-INM/CNAM)		SRD006 (NPL)	
	T_C /mK	**W_C /mK**	**T_C /mK**	**W_C /mK**	**T_C /mK**	**W_C /mK**	**T_C /mK**	**W_C /mK**
W	**15.25** (-)	**0.07** (-)	**15.2** (-)	**0.17** (-)	**-** (16.03)	**-** (0.58)	**-** (-)	**-** (-)
Be	**20.56** (21.12)	**0.33** (0.11)	**20.1** (21.98)	**0.03** (0.08)	**-** (20.98)	**-** (0.84)	**-** (20.98)	**-** (0.51)
$Ir_{80}Rh_{20}$	**31.45** (32.09)	**1.17** (0.97)	**31.7** (32.32)	**1.13** (0.99)	**-** (31.85)	**-** (0.50)	**34.21** (32.22)	**0.68** (0.62)
$Ir_{92}Rh_{08}$	**65.05** (65.52)	**0.65** (0.66)	**65.7** (66.10)	**0.94** (0.94)	**65.3** (65.70)	**0.7** (0.93)	**65.57** (65.93)	**0.57** (0.45)
Ir	**94.13** (94.38)	**1.07** (1.07)	**99.2** (99.34)	**0.57** (0.55)	**98.9** (99.53)	**0.3** (0.38)	**98.91** (99.39)	**0.52** (0.41)
$AuAl_2$	**137.23** (n. a.)	**0.56** (n. a.)	**160.6** (160.89)	**0.44** (0.73)	**160.8** (160.93)	**0.7** (0.82)	**144.95** (n. a.)	**0.48** (n. a.)
$AuIn_2$	**207.72** (207.94)	**1.0** (0.94)	**207.9** (207.80)	**0.65** (1.58)	**207.9** (206.24)	**0.5** (0.67)	**208.4** (n. a.)	**3.20** (n. a.)
Cd	**520.18** (515.9)	**12.5** 9.1	**520.5** (516.9)	**14.5** (14.0)	**522.6** (525.3)	**12.1** (17.4)	**530.4** (517.7)	**12.6** (8.7)
Zn	**851.71** (844.6)	**9.08** (8.6)	**851.7** (843.7)	**5.42** (5.9)	**853.3** (n. a.)	**15.6** (13.7)	**850.9** (844.1)	**7.8** (9.9)
Al	**1185** (n. a.)	**3.4** (n. a.)	**1178.2** (1166)	**1.66** (3.5)	**1185** (n. a.)	**3.1** (n. a.)	**1178.6** (n. a.)	**3.8** (n. a.)

Table 5: Transition temperatures T_C and transition width W_C observed during the evaluation of the SRD prototypes. In parenthesis the values determined at NMi during the preliminary tests and calibration are reported; n. a. in parenthesis means comparison not applicable either because the sample was replaced or the thermometry at NMi was not optimised.

Among all the W samples that were transferred to the partners for the evaluation, only the one from SRD005 was measured at NMi VSL. This is due to the fact that SRD005 was calibrated in run 10 at NMi (June 2002), when the minimum temperature reached was 12.8 mK, while SRD003, SRD004 and SRD006 were calibrated in run 11 (October 2002), when the minimum temperature reached was 16.1 mK.

6 Conclusions

The evaluation performed on the four prototypes proved that the SRD1000 is a convenient and reliable means of transferring the Provisional Low Temperature Scale 2000 (PLTS-2000).

The transition temperature values of the SRD1000 can be identified with an uncertainty that ranges from a few hundredths of mK (at the lowest temperatures) up to few mK (at the highest temperatures).

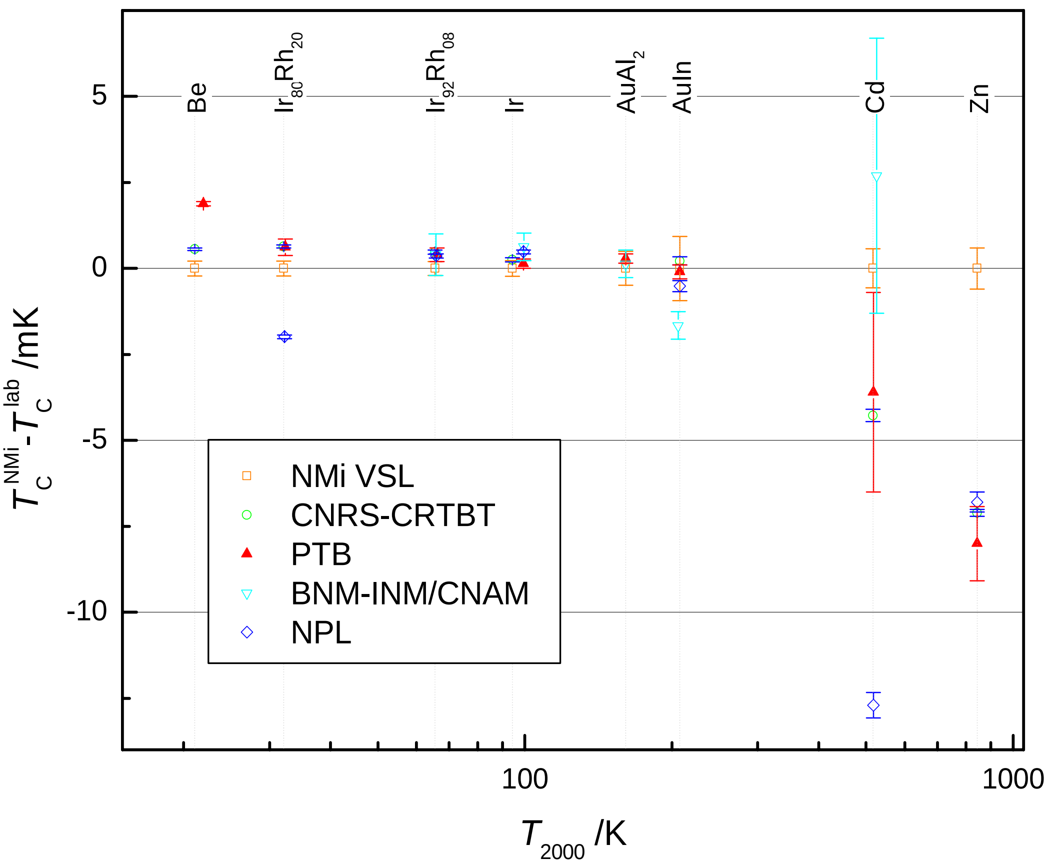


Figure 5: Differences between the transition temperatures measured at NMi VSL and those measured at the evaluator labs on the same reference samples. Aluminium point is not included because outside the PLTS-2000 range.

The residual magnetic field tests demonstrated that the magnetic shielding of the SRD sensor is very effective (attenuation factor higher than 500). As consequence, the depression of the transition temperature, due to residual and measuring fields is well below the uncertainty in the scale realization and can be neglected.

Although each sample must be individually calibrated, and the long-term reproducibility of the SRD1000 reference points still needs to be proved, all the evaluator partners agree in considering the SRD1000 a highly valuable device for high-accuracy thermometry below 1 K.

6 References

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