EVALUATION OF SUPERCONDUCTIVE REFERENCE DEVICE SRD1000 PROTOTYPES

Andrea Peruzzi[1], Martin J. de Groot[1], Valerie Goudon[2], Matthias Meschke[2], Henry Godfrin[2], Jost Engert[3], Bernd Fellmuth[3], Yves Hermier[4], Laurent Pitre[4], Celine Rives[4], George Bonnier[4], Stephan Schöttl[5] and Richard Rusby[5]

[1]NMi VSL, Delft, The Netherlands
[2]CNRS-CRTBT, Grenoble, France
[3]PTB, Berlin, Germany
[4]BNM-INM/CNAM, Paris, France
[5]CBTLM, NPL, Teddington, UK

ABSTRACT

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 SRD sensor. In the frame of EU project "Ultra-Low Temperature Dissemination," four SRD1000 prototypes were transferred to as many labs for evaluation. Each lab independently performed extensive measurements on the assigned SRD1000 prototypes: residual magnetic field tests, repeated observations of the transitions under specified controlled thermal conditions and determination of the transition temperature and transition width of each transition. The results of the evaluation are reported in this paper.

1. INTRODUCTION

Temperatures down to few milliKelvin are nowadays steadily available to many academic and industrial research laboratories. Nevertheless, reliable and accurate thermometry below 1 K is still limited to a few national metrology institutes and not accessible to the large majority of the users.

To provide the ultra-low temperature research community with the basis for accurate and reliable temperature measurement below 1 K, in October 2000 the Comite' International des Poids et Mesures adopted a provisional extension of the International Temperature Scale of 1990 (ITS-90) from 0.9 mK to 1 K (Provisional Low Temperature Scale, PLTS-2000) [1]. The effort required for the practical realization of the PLTS-2000 is such (expensive, time-consuming and complicated experimental techniques to be mastered by experienced personnel) [2] that more direct and practical means of disseminating it to final users are considered essential.

Superconductive temperature reference points proved to be a useful means of disseminating low temperature scales. In the seventies the NIST developed and commercialized two different superconductive reference devices: the SRM 767 [3] for the range 0.5 K – 7.2 K and the SRM 768 [4] for the range below 0.5 K. In the nineties the production of these devices was discontinued by NIST, and a Dutch consortium of research institutes and industries initiated the development of a new superconductive reference device, named SRD1000 [5-7], for the range 15 mK – 1200 mK. The adoption of the PLTS-2000, further enlarged the interest towards the SRD1000 as it is regarded as a very effective means of dissemination and comparison of independent realizations of the new scale.

In the frame of EU project "Ultra-Low Temperature Dissemination," four SRD1000 prototypes were produced by HDL [8], tested and calibrated at NMi VSL and finally transferred to as many independent labs (CNRS-CRTBT, PTB, BNM-INM/CNAM and NPL) for evaluation. The results of the evaluation are summarized in this paper.

2. EVALUATION PROCEDURE AND MEASUREMENT CONDITIONS

The superconductive transitions of the reference samples were observed by monitoring the DC output of the mutual inductance system in the SRD1000 as a function of temperature. The minimum temperatures of CNRS-CRTBT and PTB were 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 and NPL could reach were respectively 32 mK and 30 mK, which did not allow them to observe the W, Be and, in the case of BNM-INM, the $Ir_{80}Rh_{20}$ transitions.

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 with stabilization plateaus of several minutes (8-30 minutes each) to allow thermal equilibrium. 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.

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 lab in the evaluation of the assigned prototype originated from the respective local PLTS-2000 realizations. For the details of the local realization at PTB, BNM-INM/CNAM and NMi VSL see respectively [9,10,11]. 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.

Each lab independently estimated the uncertainty in the determination of the transition temperature values. The principal uncertainty components arose from the PLTS-2000 realization, the residual magnetic fields and the localization of the transition mid-point. The expanded uncertainty claimed by the labs at each reference temperature is reported in Table 1. Different approaches in estimating the uncertainties resulted in different claimed uncertainties even when the same equipment was used by the labs.

Table 1: Expanded uncertainty (k = 2) claimed by the labs in the determination of the transition temperature of each reference point. In parenthesis the expanded uncertainty (k = 2) in their realization of the PLTS-2000 at the transition temperature of each reference point of the SRD1000 is reported. Last column refers to preliminary tests at NMi VSL.

	CNRS-CRTBT /mK	**PTB /mK**	**BNM-INM /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)	-	0.20 (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)	1.20 (0.20)	0.6
Zn	0.10 (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)	1.0

4. MAGNETIC FIELD TESTS

The transition temperature T_C of the reference superconductive 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.

Magnetic field tests were performed on the SRD1000 prototypes in order to a) estimate the residual external field experienced by the reference samples within the SRD1000 shielding and b) estimate the T_C depression produced by the AC measuring field.

The tests were performed in the two following ways (see [2] for the details of the magnetic field tests):

1) By observing the T_C depression when DC currents were superimposed to the AC primary current in the detector planar coils, 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.

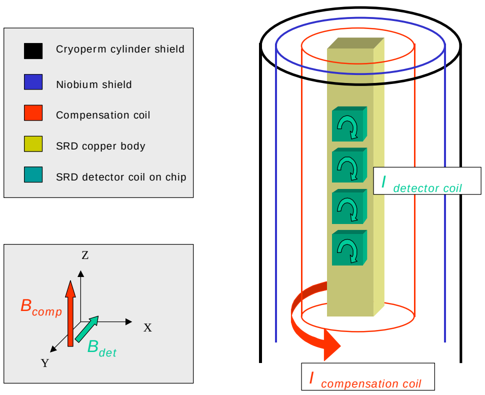

Figure 1: Magnetic shielding and field configurations of the SRD1000: the magnetic shield surrounding the SRD copper body consists of a Cryoperm outer shield and a niobium inner shield. The measuring field (Y direction) is produced by the detector planar coils and the additional field (Z direction) is produced by the compensation coil.

Using the coil constants of the compensation coil and detector primary coil, the residual field components could be estimated (see Table 2) and the corrections for the depression of the transition temperature values due to the measuring field could be applied.

Table 2: Magnetic field tests performed 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.

		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

5. RESULTS

All the superconductive transitions observed are shown in Figure 2. 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 going on.

The transition temperatures and widths as determined by the partners are reported in Table 3. 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.

Table 3: 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.

	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.)

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. These 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.

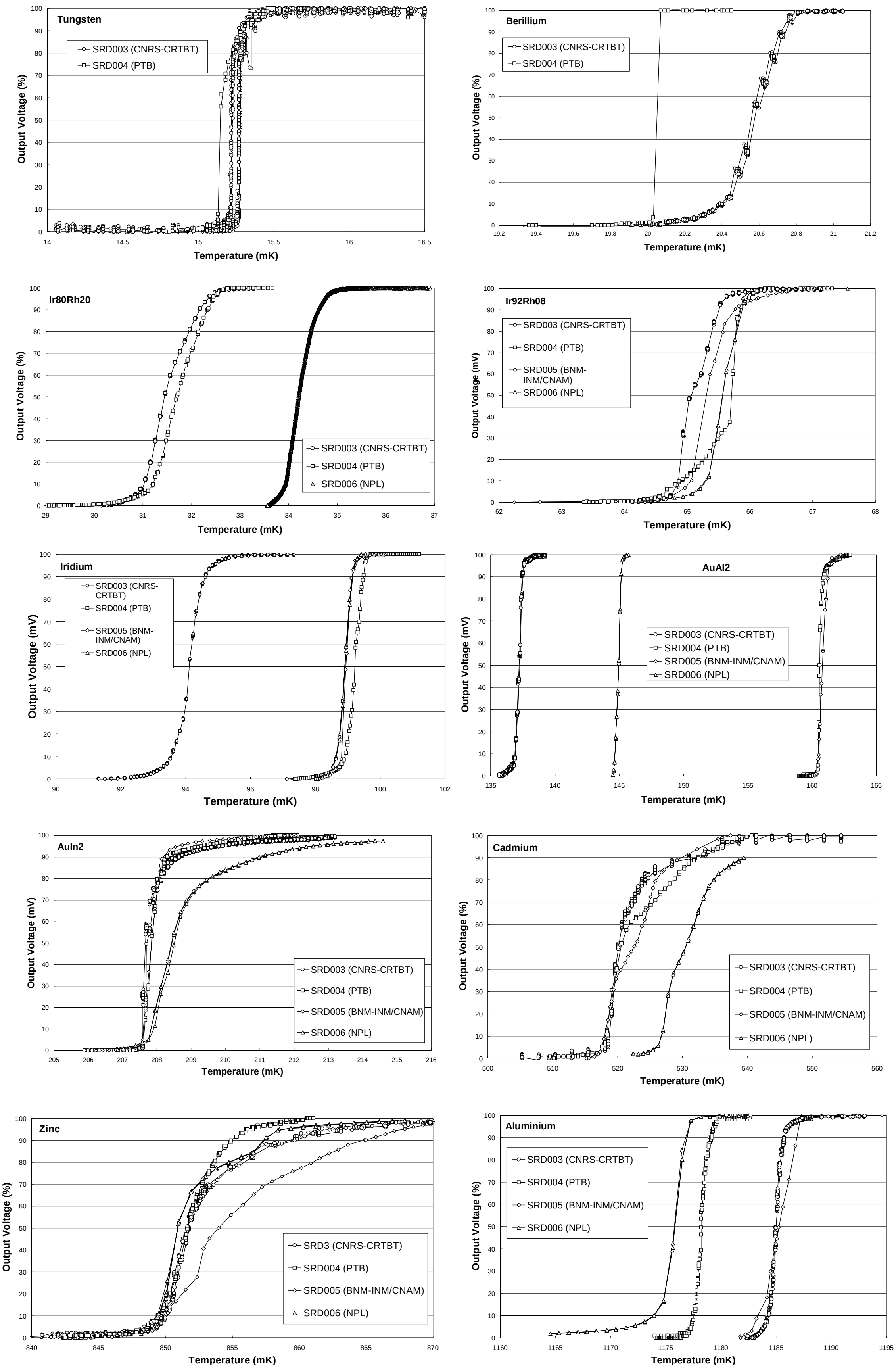

Figure 2: Superconductive transitions observed by each lab on the assigned SRD1000 prototype.

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). 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, the SRD1000 proved to be a highly valuable device for high-accuracy thermometry below 1 K.

AKNOWLEDGEMENTS

This work is part-funded by the European commission under the Measurement and Testing activity of the Competitive and Sustainable Growth programme, Contract No G6RD-CT-1999-00119.

REFERENCES

[1] Proces-verbaux du Comite' International des Poids et Mesure, 68 (2001) p.129.

[2] Schuster G.,Hoffmann A., Hechtfischer D., "Realization of the temperature scale PLTS-2000 at PTB", PTB-Bericht PTB-ThEx-21, 2001, ISBN 3-89701-742-3..

[3] Schooley J. F., Soulen R. J. Jr., Evans G. A. Jr., "Standard Reference Materials: Preparation and Use of Superconductive Fixed-Point Devices, SRM 767", *NBS Special Publication 260-44*, U.S. Govt. Printing Office, Washington D.C., 1972, pp. 1-35.

[4] Soulen R. J. Jr., Evans G. A. Jr., "SRM 768: Temperature Reference Standard for Use below 0.5 K", *NBS Special Publication 260-62*, U.S. Govt. Printing, Washington D.C., 1979, pp. 1-37.

[5] Storm A. J., Bosch W. A., de Groot M. J., Jochemsen R., Mathu F., Nieuwenhuis G J., *Proceedings of the 7th International Symposium of Temperature and ThermalMeasurements (TEMPMEKO99)*, edited by Dubbeldam J. et al., Delft, NMi van Swinden Laboratorium, 1999, pp. 142-146.

[6] Bosch W. A., Chinchure A., Flokstra J., de Groot M. J., van Heumen M. J., Jochemsen R., Mathu F., Peruzzi A., Veldhuis D., *Proceedings of the 8^{h} International Symposium of Temperature and Thermal Measurements (TEMPMEKO2001),* edited by Fellmuth B. et al., Berlin, VDE Verlag, 2001, pp. 397-401.

[7] Bosch W. A., Flokstra J., de Groot G. E., de Groot M. J., Jochemsen R., Mathu F., Peruzzi A., Veldhuis D., *Temperature: Its Measurement and Control in Science and Industry, Volume 7*, edited by Ripple D. C., American Institute of Physics, Melville, New York, 2003, pp. 155-160.

[8] Hightech Development Leiden, Leiden, The Netherlands, e-mail: HDLinfo@xs4all.nl.

[9] Engert J., Fellmuth B.,Hoffmann A., "Realization, Dissemination and Comparison of the ITS-90 and the PLTS-2000 below 1 K at PTB", *Journal of Low Temp. Phys.*, **134**, n. 1-2 (2004), pp. 425-430.

[10] Pitre L., Hermier Y., Bonnier G., *Temperature: Its Measurement and Control in Science and Industry, Volume 7*, edited by Ripple D. C., American Institute of Physics, Melville, New York, 2003, pp. 95-100.

[11] Peruzzi A., de Groot, M. J., *2nd International Seminar on Low Temperature Thermometry*, Wroclaw, Poland, Ed. by Szmyrka-Grzebyk et al., 2003, pp. 35-40.

Addresses of the Authors:

Andrea Peruzzi, Nederlands Meetinsituut, P.O. Box 654, 2600 AR Delft, The Netherlands, tel. + 31 15 269 1519, fax +31 15 261 2971, e-mail: aperuzzi@nmi.nl, website: www.nmi.nl.