Advanced Cryogenics Course

CRYOCOURSE 2011

Thermometry at low temperatures

NO, NMR, CBT, VWR, Fix Points

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Thermometry: what is « a good thermometer »?

The thermometrical physical property $x(T)$ should vary rapidly as a function of temperature, compared with the accuracy of the measurement: **good sensitivity $(\Delta x/x)/(\Delta T/T)$.**

$x(T)$ should be **easy to determine experimentally**.

The temperature dependence $x(T)$ should be **adjustable by a simple law**.

The **operation temperature range** should be as large as possible.

The internal and external **equilibrium times** should be short: small heat capacity, good thermal conductivity and good thermal contact to the surroundings.

The measurement should introduce a **minimum of heat**

Low Temperature Thermometry

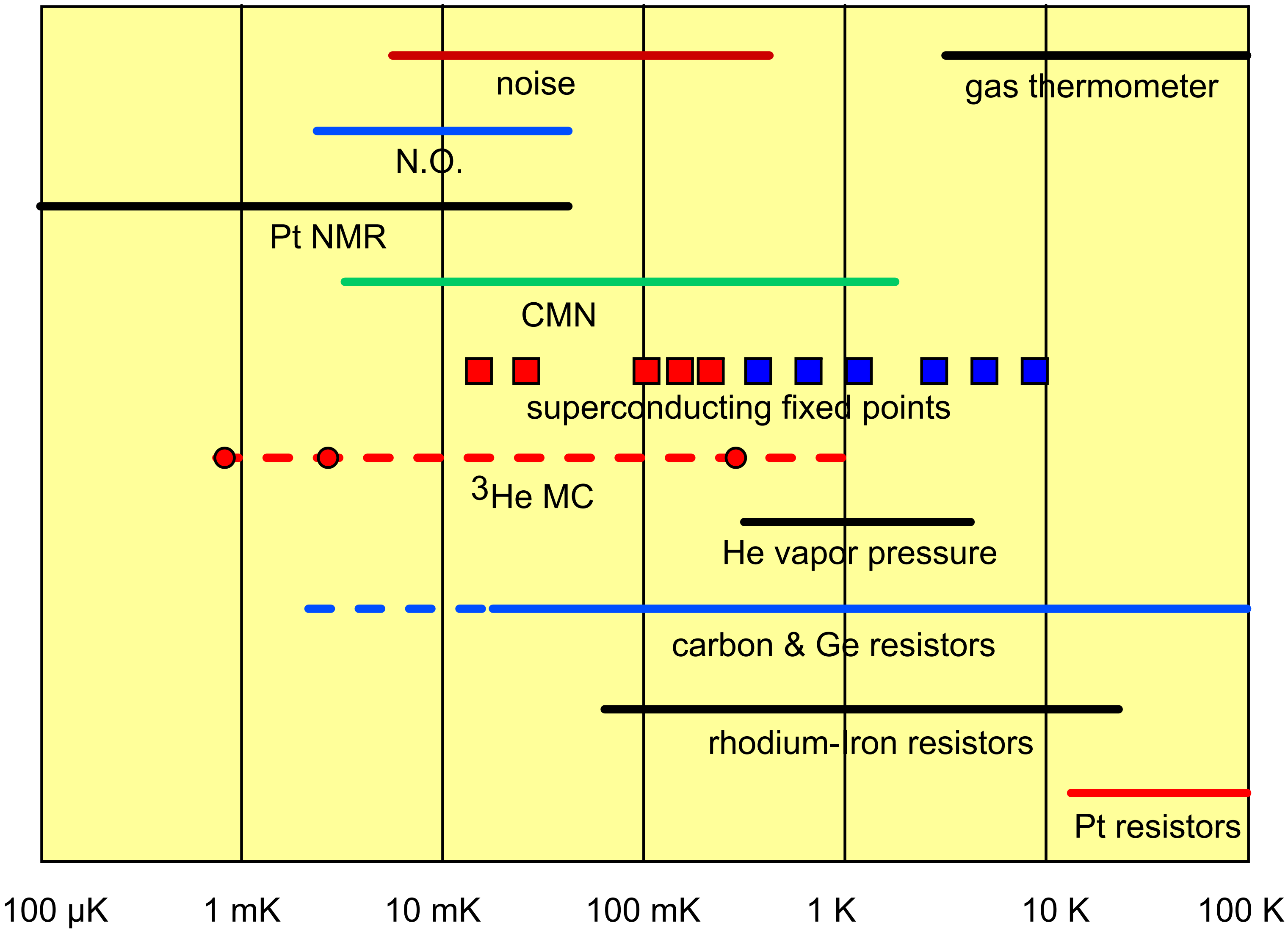
The development of an accurate thermometry is a delicate task. Furthermore, there exists presently no simple device able to provide temperature values with a reasonable accuracy in a large temperature range under different experimental conditions (magnetic field, vibrations, electromagnetic perturbations).
As a result, one has to work with many different types of devices:

- primary thermometers.
- temperature fixed points.
- temperature interpolation devices.
- secondary thermometers

in order to reproduce in the laboratory the accepted temperature scale.

Sophisticated cryogenic electronic instrumentation, as well as a low temperature dilution refrigerator and a nuclear demagnetization stage, are also needed for this purpose.

Low Temperature Thermometers



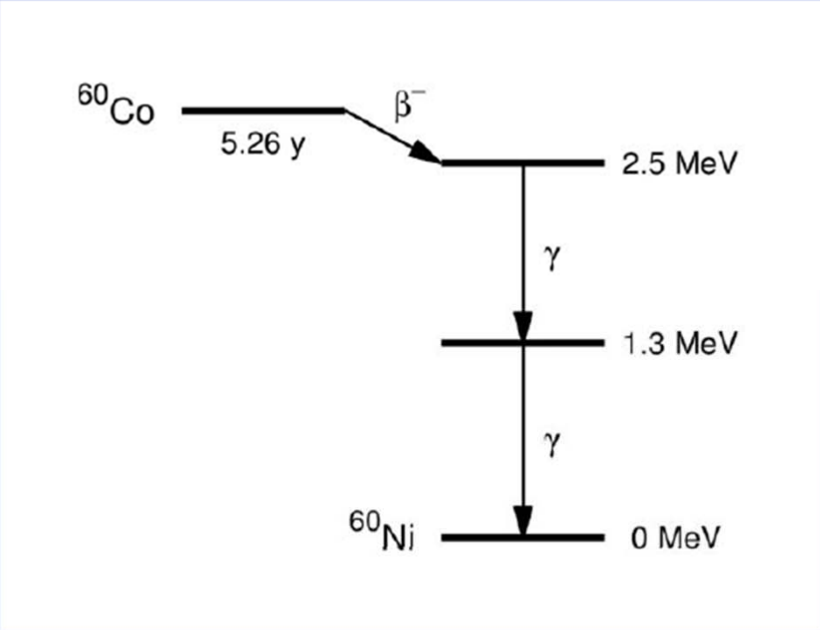
Co-60 thermometer

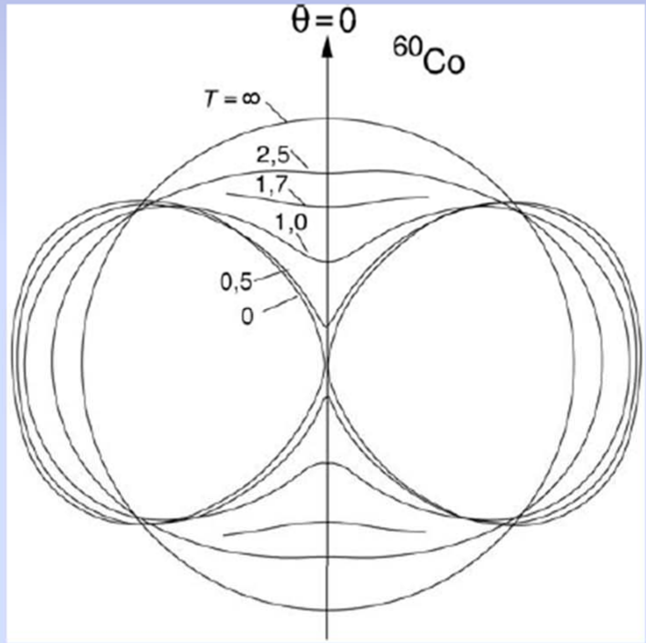
- Important as primary thermometer
  - We use it to calibrate more sensitive thermometers
- Thermometer is based on measuring the anisotropy of γ-rays emitted from polarized nuclei
  - Nuclear levels are split by magnetic field into ($2I+1$) hyperfine levels (I is the spin quantum number of nucleus)
  - The population numbers of the sublevels follow Boltzmann statistics and so depend on temperature
  - On the other hand orientation of nuclei vary from level to level -> temperature depended angular radiation pattern $W(\phi,T)$
- Sensitivity is reasonable between 3 and 30 mK, at higher temperatures all energy levels are equally populated and at lower temperatures only the lowest level is populated.
- Measuring one spectrum takes roughly one hour

Nuclear Orientation Thermometer

example ^{60}Co

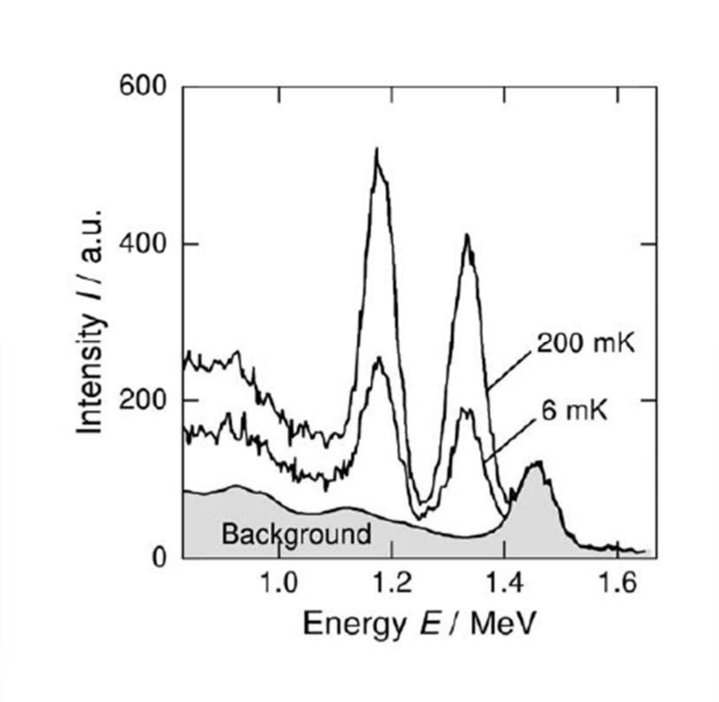
nuclear spin I
magnetic field: splitting into (2I+1) sublevels

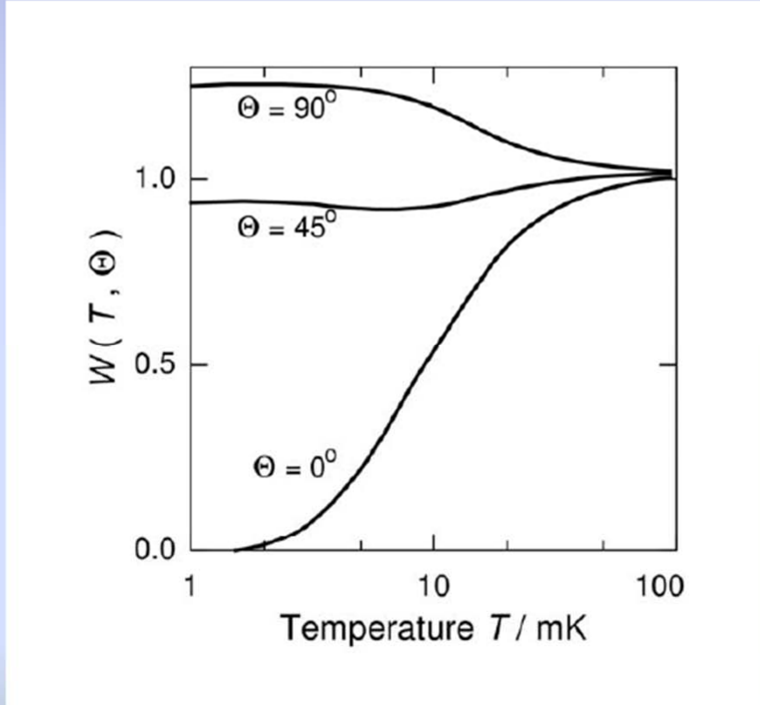




Nuclear Orientation Thermometer

$$W(T,\Theta) = 1 + \sum_{k=2,4,\ldots}^{k_{\max}} G_k\, U_k\, F_k\, B_k(T)\, P_k(\cos\Theta)$$





Platinum thermometer

The nuclear paramagnetism of platinum constitutes the basis of a very convenient thermometer for the temperature range 0.2 - 100 mK.

Given the **Korringa constant** on the order of 30 sec.mK, pulsed NMR is particularly suited for the measurement of the nuclear susceptibility.

At high temperatures, the signal becomes too small, and the amplifier noise is a strong limiting factor.

Thermal contact and heat leaks constitute the main limitation at ultra-low temperatures.

Pulsed NMR spectrometers working at low frequency (250 kHz and lower) are commercially available.

Recently, cold amplifiers have been developed which allow using cw NMR spectrometers for high accuracy thermometry purposes.

Platinum NMR is an excellent solution when magnetic fields are present in the experimental set-up.

However, since spectrometers work at fixed frequencies, the field cannot be changed continuously.

Pt-NMR thermometer

- Sample is placed in a static magnetic field B_0
- Susceptibility of nuclear spins follows the Curie law

$$\chi_n = \Lambda_n / T$$

- We measure the magnetization M_n by applying an excitation pulse at the Larmor frequency to tip the magnetization by a small angle θ
- After excitation pulse there is a component

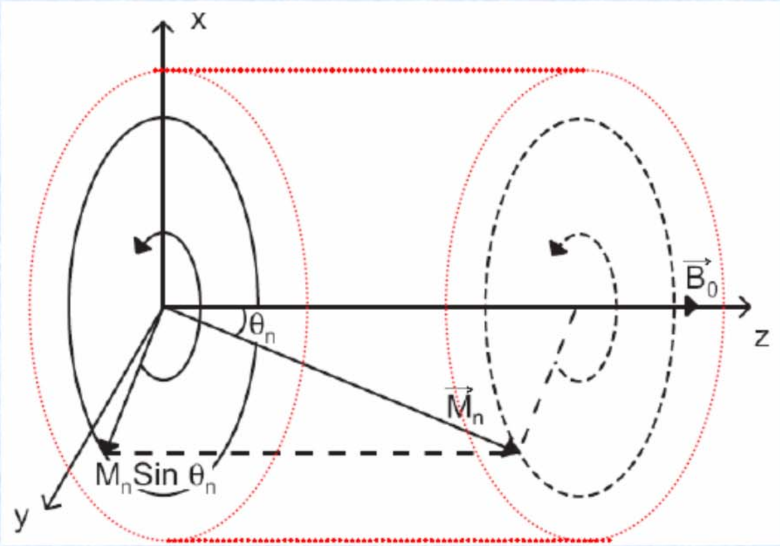
$$B_y = B_1 sin(\omega_n t)$$

precessing in the XY-plane

- This component induces a voltage

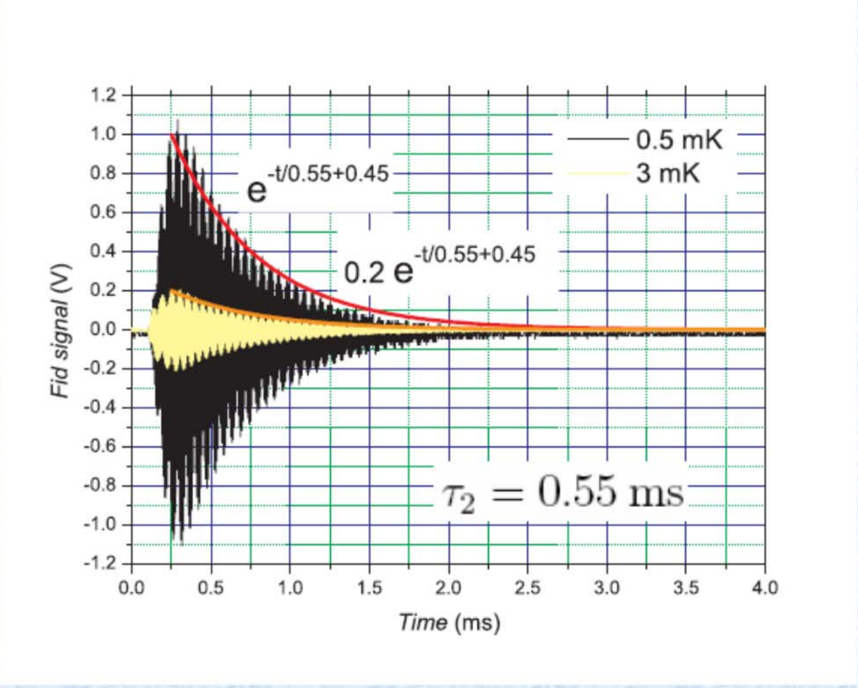
$$U = \alpha \omega_n M_n sin\theta_n,$$

to pick-up coil.



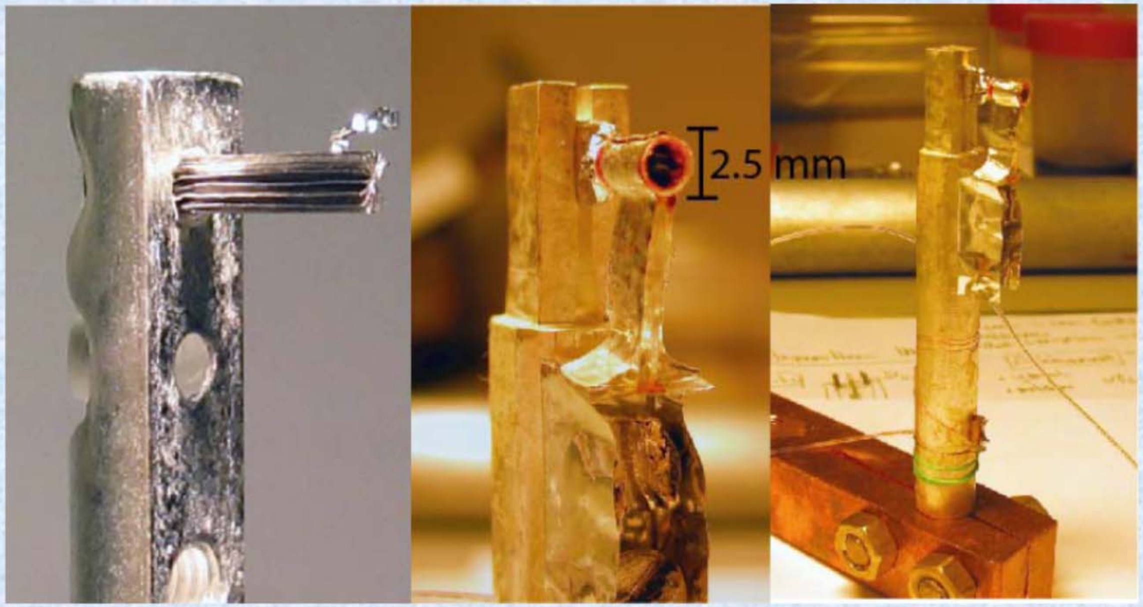
•This signal is called the free induction decay and it's amplitude is directly proportional to magnetization and thus inversely proportional to temperature

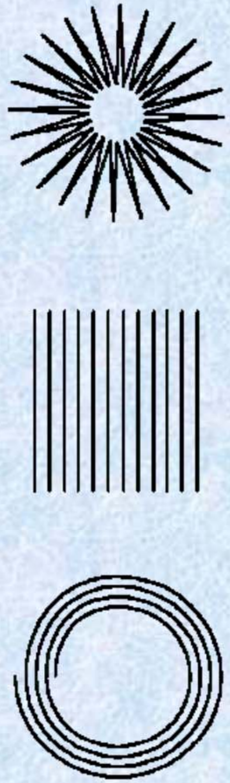
Fid signal



- Two important timescales
  - τ_1 determines nuclear spin systems relaxation time to electron system's temperature $\tau_1 = \kappa / T_e$, where κ is Korringa constant (0.030 sK for Pt, $\tau_1 \sim 5$ min at 100 μK)
  - τ_2 determines spin-spin relaxation time. Sufficient time is needed for measurement to be made. For Pt $\tau_2 \sim 1$ ms
- In addition Pt has only one magnetic isotope ^{195}Pt with nuclear spin ½ -> simplifies signal
- Pt is a good sample material

Pt sample





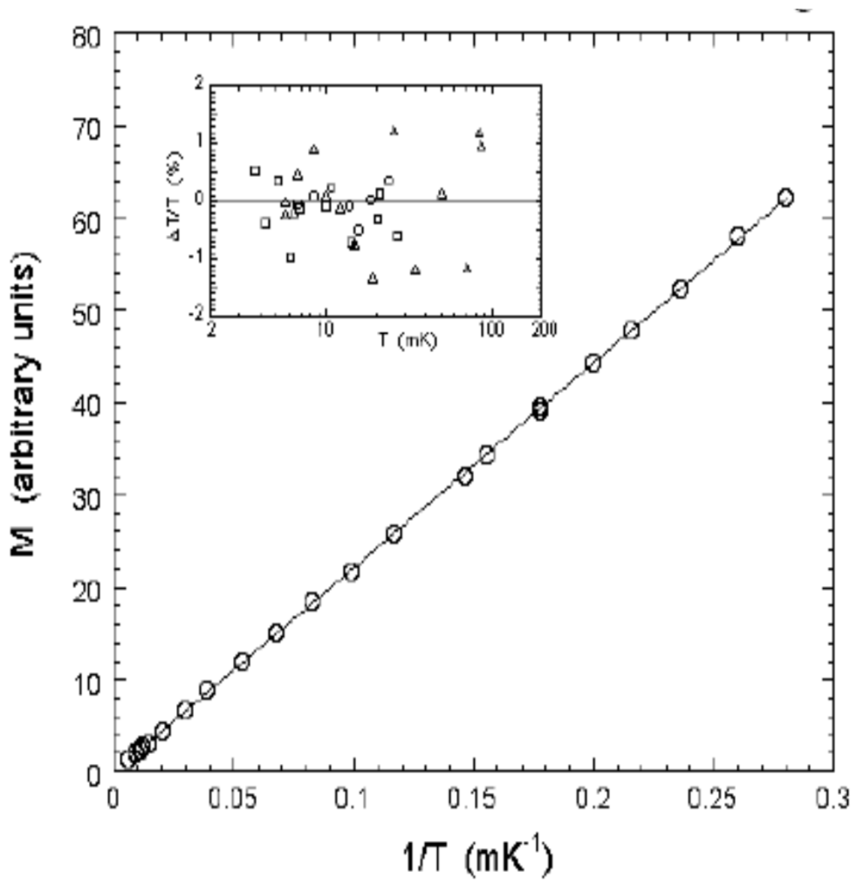
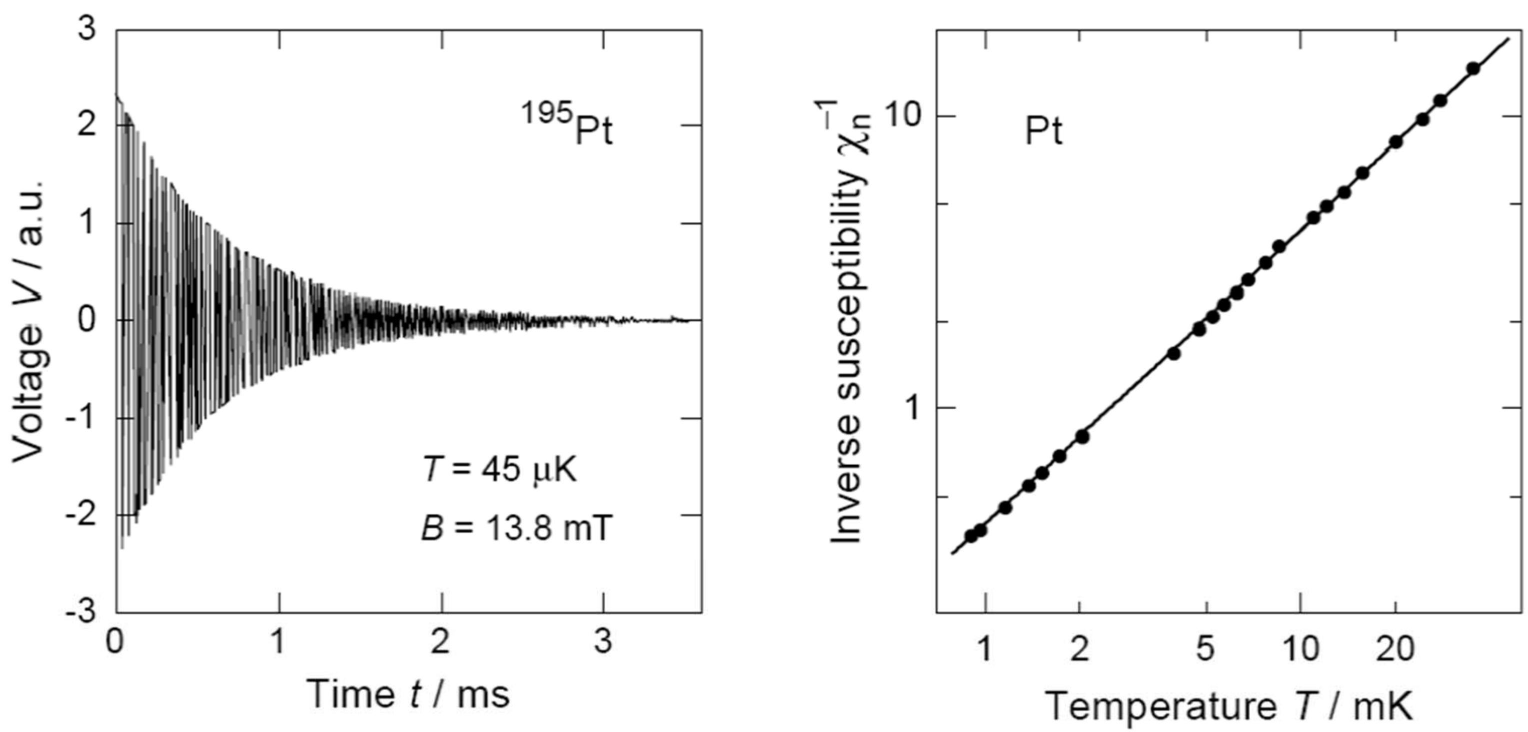


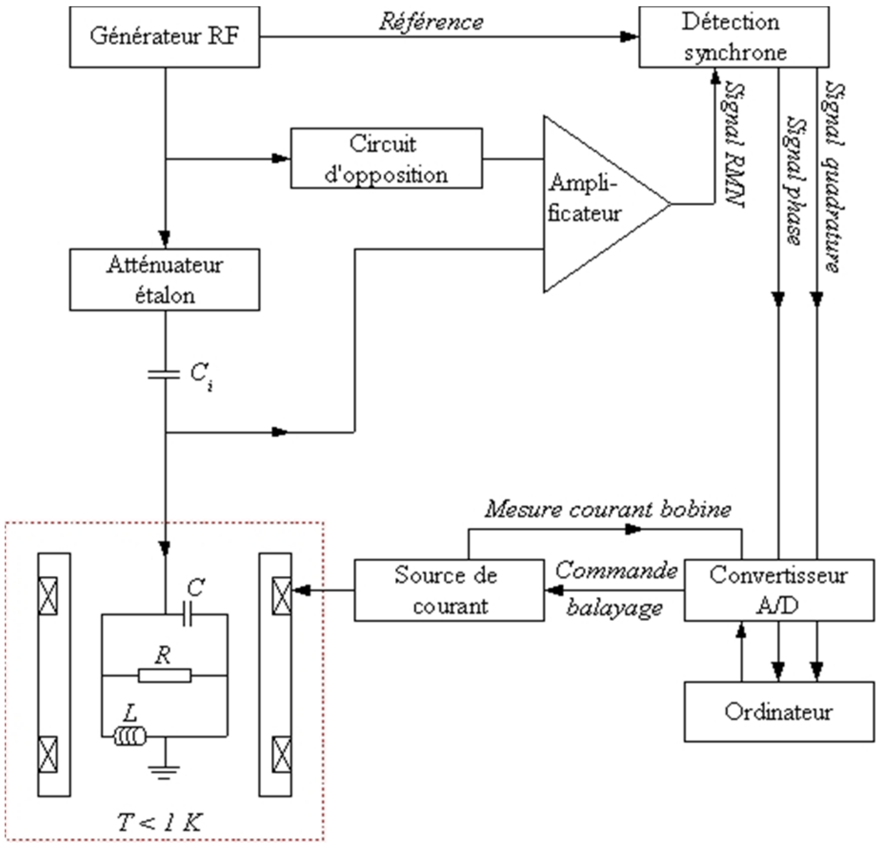
Figure 3: Magnetisation of the Pt-NMR thermometer versus $1/T$ determined with a CMN thermometer

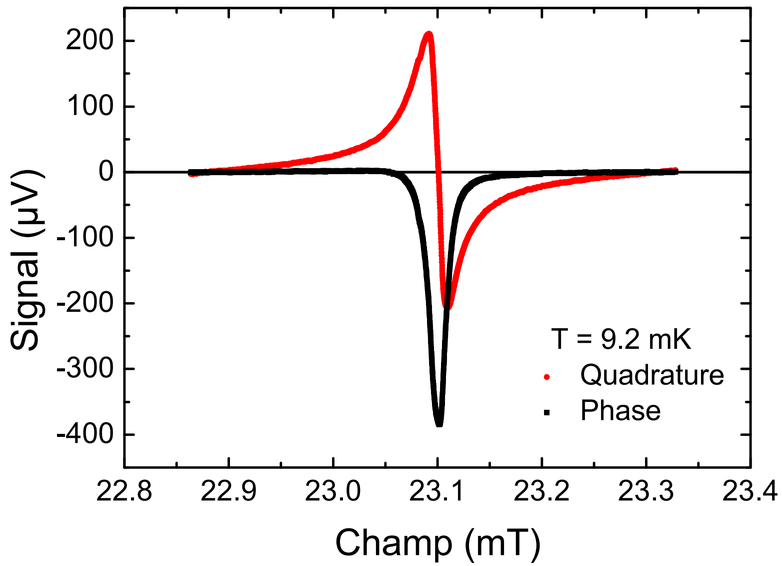
Pt-NMR-Thermometry



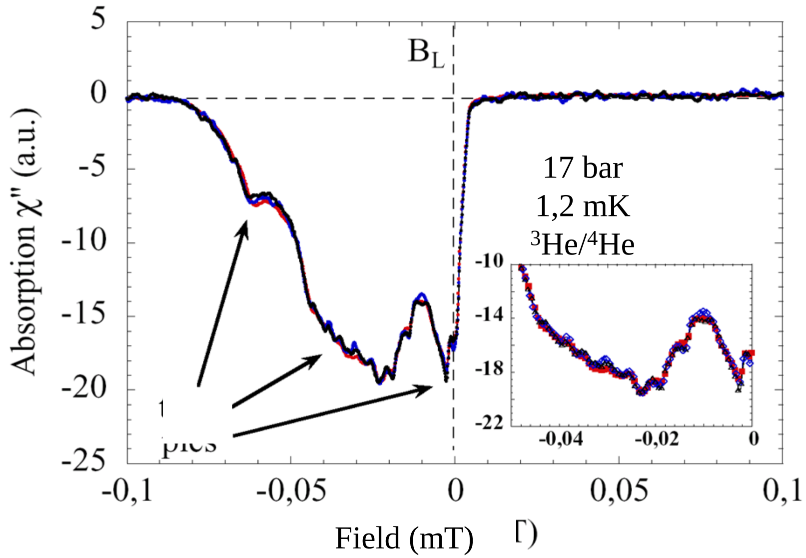
$$M_y(t) = \frac{1}{2} M_0 \cos(\Omega t)\, e^{-t/\tau_2^*}$$

cw NMR Thermometry





Susceptibility of bulk ^{3}He



Texture in superfluid ^{3}He

2D-3He NMR

- A new type of thermometer **(2D-3He NMR)** has been developed by the CNRS-CRTBT (Grenoble).
- It is based on the NMR measurement of the nuclear susceptibility of a 3He monolayer adsorbed on a graphite substrate.
- The saturation magnetization of this system is determined at temperatures on the order of 100 µK and fields on the order of 1 Tesla, thus allowing to use the Brillouin law characteristic of the device to determine absolute temperatures above 1 mK.
- This type of thermometer should allow us to establish a temperature scale starting from the absolute zero, and compare this scale to the International Scale, which has been constructed from high temperature extrapolations.

2D ^{3}He NMR

A monolayer of 3He adsorbed on graphite can be used as a very sensitive thermometer in the range 80 µK –1K.

Cw-NMR techniques are used for this purpose.

As in the case of platinum NMR, a small magnetic field is necessary.

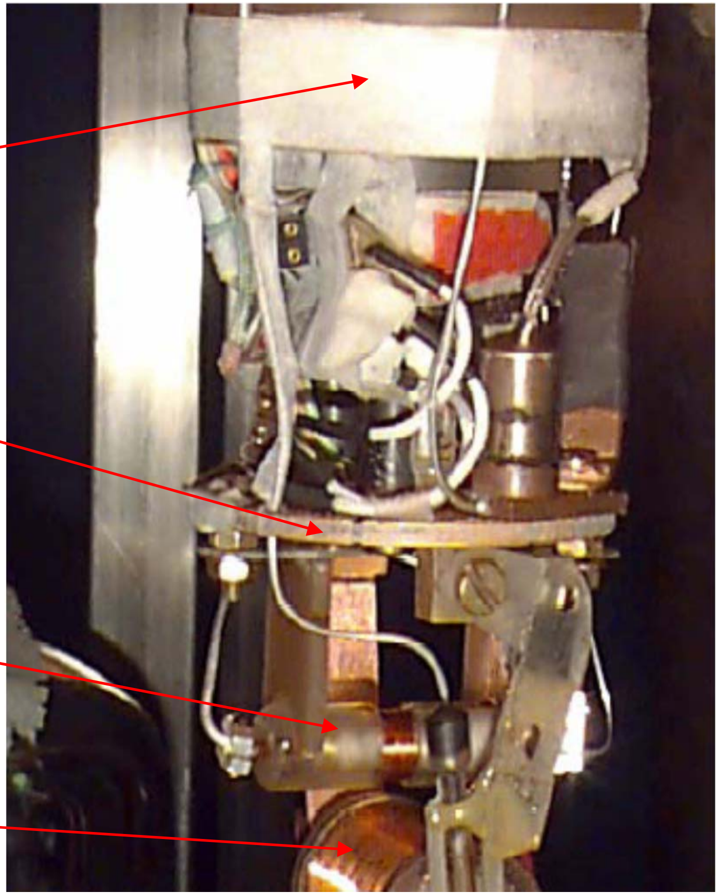
The advantage of 2D (two-dimensional) 3He NMR is that much lower temperatures can be measured.

mixing chamber of the dilution refrigerator

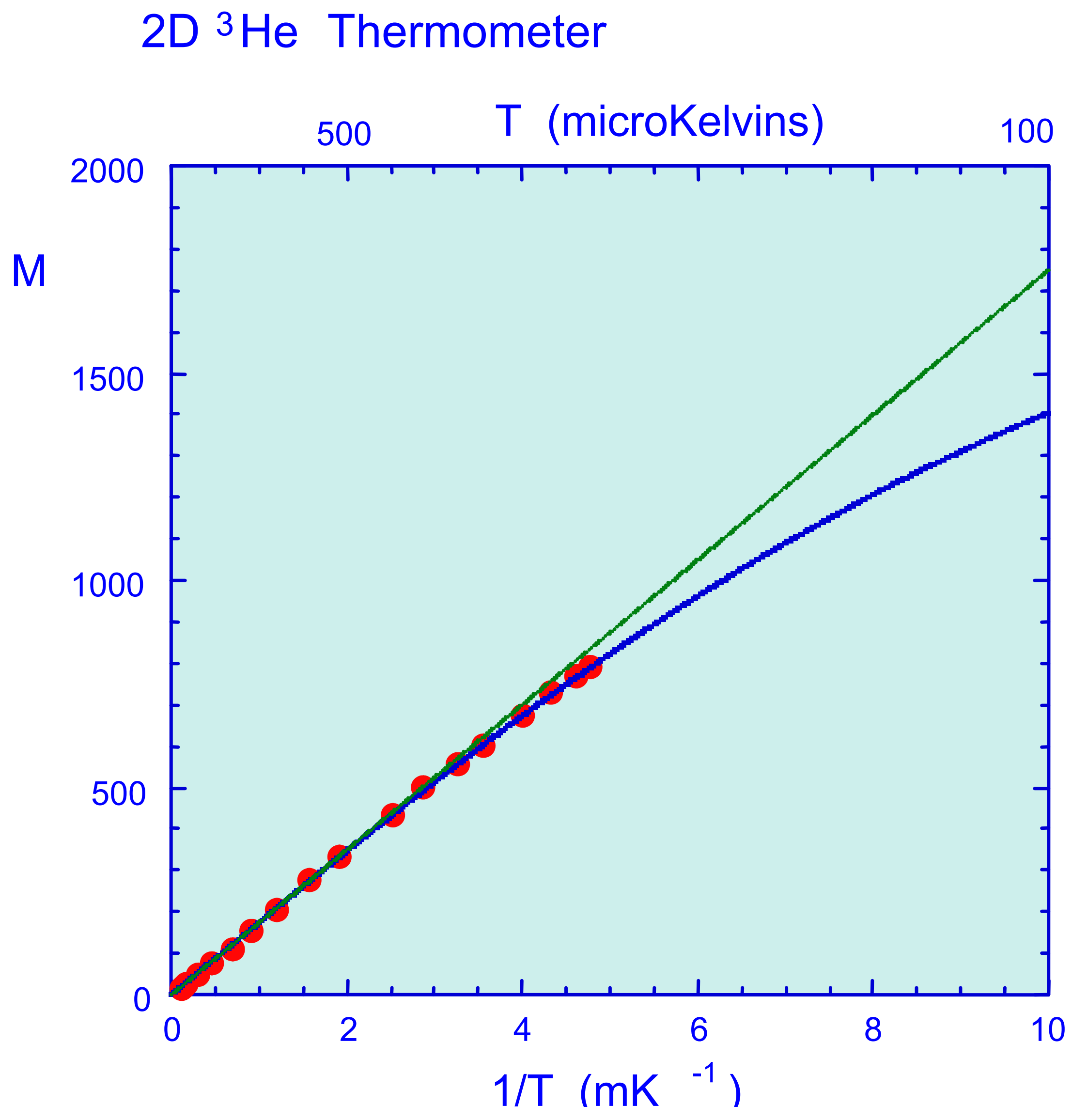
copper plate and melting curve thermometer

experimental cell (NMR of liquid 3He)

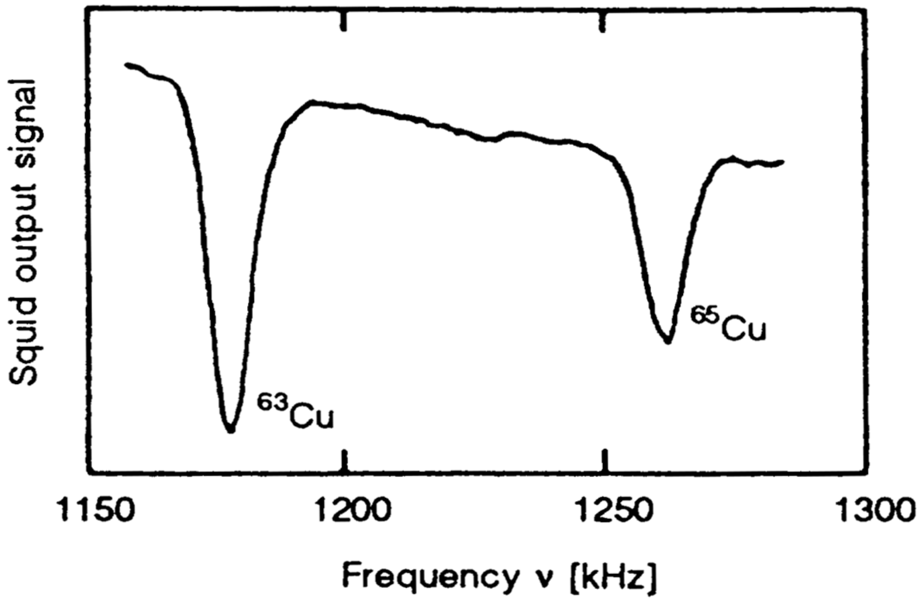
2D-3He NMR thermometer



Adsorbed 3He
NMR at ULT



SQUID NMR : Copper lines

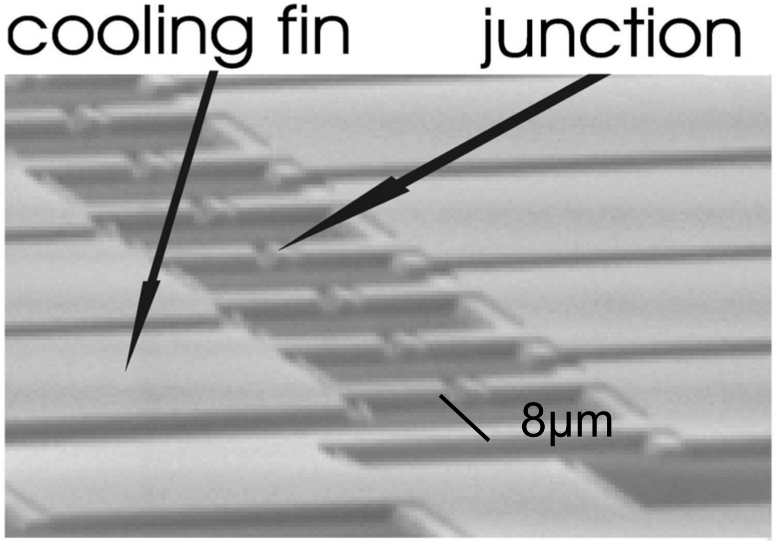


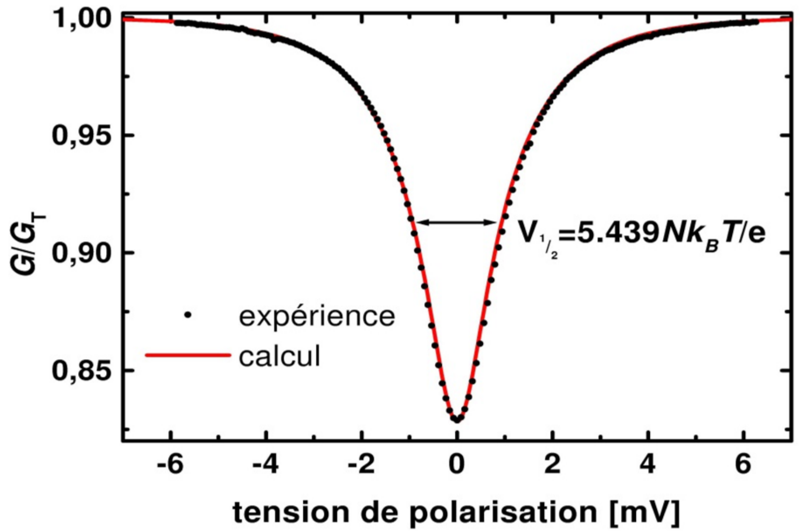
Coulomb Blockade thermometers

- A new type of device has been developed at the University of Jyväskylä: the **Coulomb Blockade Thermometer (CBT)** which could constitute a very attractive alternative given its simplicity of operation.
- It consists of an array of tunnel junctions of sub-micron size.
- The conductance of the system depends on the dc bias voltage and on the temperature in a universal way.
- In addition to this “primary mode”, the CBT can also be used in the “secondary mode” for fastest operation.
- The response is practically linear in the absolute temperature, and independent on the magnetic field (up to about 30 Teslas!).
- Its accuracy is believed to be on the order of 0.5%, which is satisfactory even for rather demanding applications.
- The first commercial CBTs are available with the finnish company Nanoway.
- Present devices are able to cover the temperature range 30 mK-50K.

Coulomb blockade thermometer

Primary thermometer - nanofabricated device





Nanoway (Finland) – Univ. of Jyväskylä
HUT-Helsinki and CNRS/CRTBT

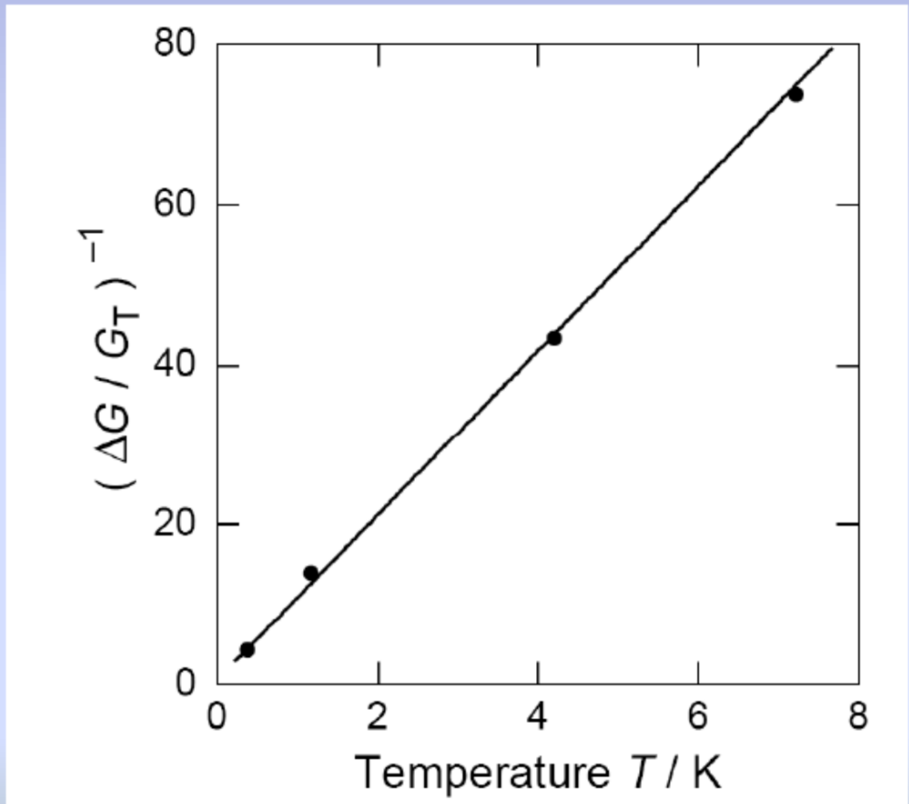
$$V_{1/2} \approx 5.439 \times \frac{N \cdot k_B \cdot T}{e}$$

T= 45.7± 0.3 mK

Electron thermalization at millikelvin temperatures in metallic islands probed by Coulomb blockade thermometry
M. Meschke, J. P. Pekola, F. Gay, R. Rapp and H. Godfrin. J. of Low Temp. Phys. 134, 1119 (2004).

Primary Coulomb Blockade Thermometry by a pure AC measurement.
M. Meschke, J.P. Pekola, H. Godfrin; IEEE Transactions (submitted, 2006)

Coulomb Blockade Thermometer



can be used between 50 mK and 30 K

Heating effects in a CBT

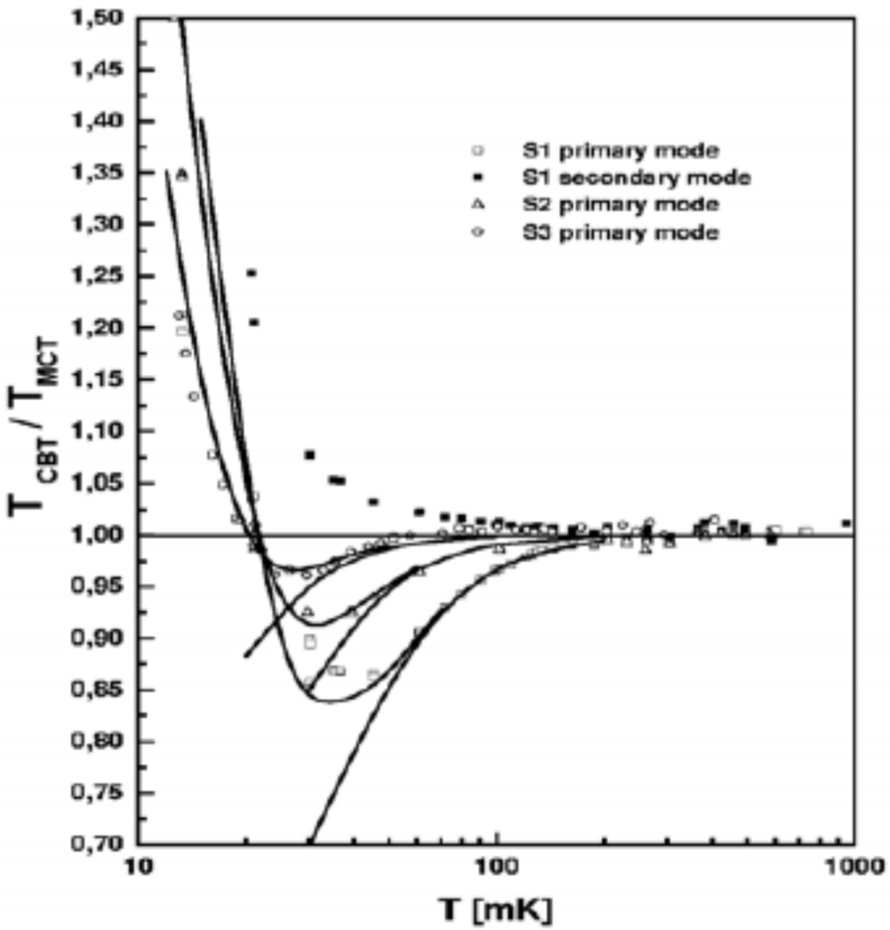


Figure 1: Calibration of three CBT sensors, S1, S2 and S3, against the melting-curve thermometer, with theoretical curves.

Thermometry in normal and superfluid ^3He

- Superconducting vibrating wires

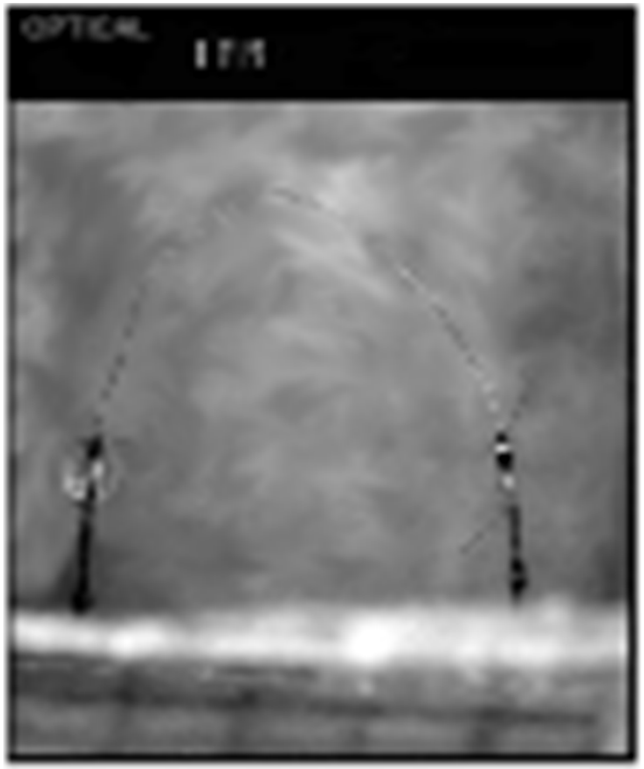
- Quartz tuning forks

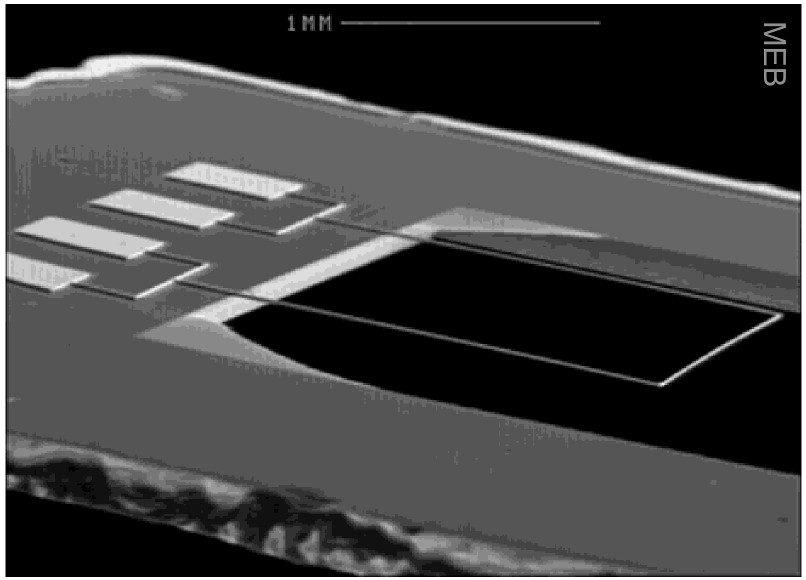
Superconducting vibrating wires

- By hand : Ni-Ti 4.5µm
  difficult to make

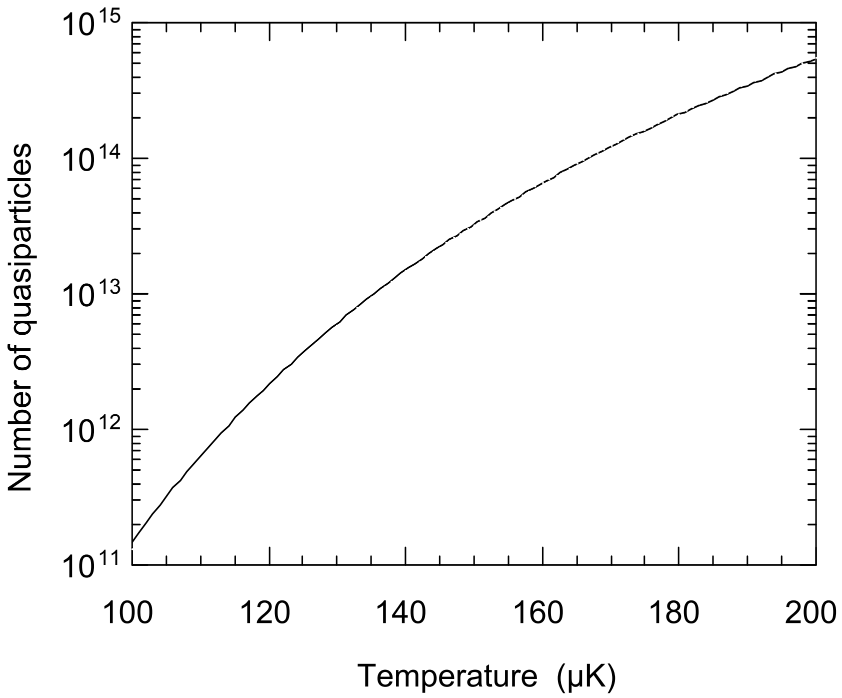
- Nanofabricated

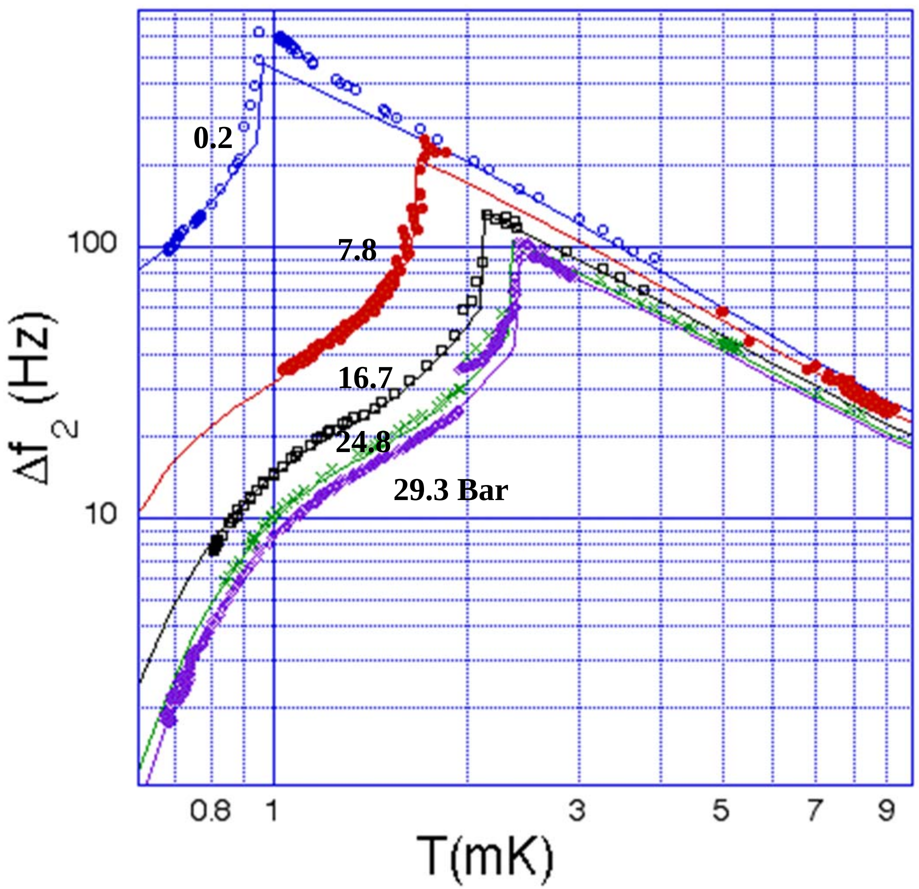
- Known technique
- Fabrication in series
- High Q (> 40.000)





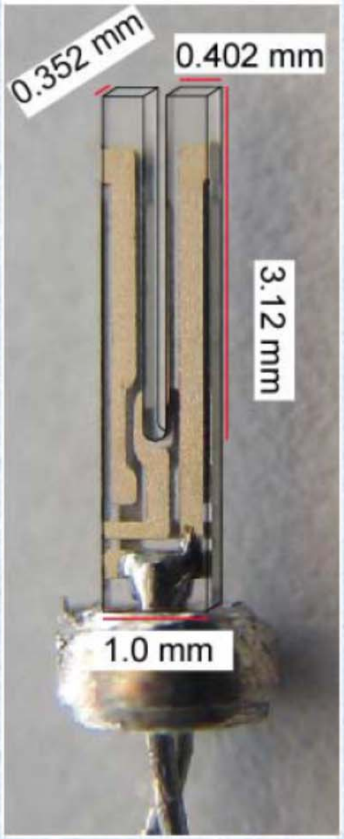
New temperature scale based on vibrating wires immersed in superfluid ^{3}He





C. Winkelmann, E. Collin, Yu.M. Bunkov and H. Godfrin,

Quartz tuning forks



- We consider replacing VWR's by quartz tuning forks
- Several advantages
  - No need for magnetic field, independent of external magnetic fields
  - Simpler measurement configuration
  - Very high Q-values (10^9), temperature independent intrinsic damping
  - High signal quality (one point measurement)
  - Cheap mass products
- Some questions/disadvantages
  - More complicated geometry
  - Higher frequency (acoustic losses)
  - Heating?
- We are still testing these

Temperature Fixed Points and Transfer Devices

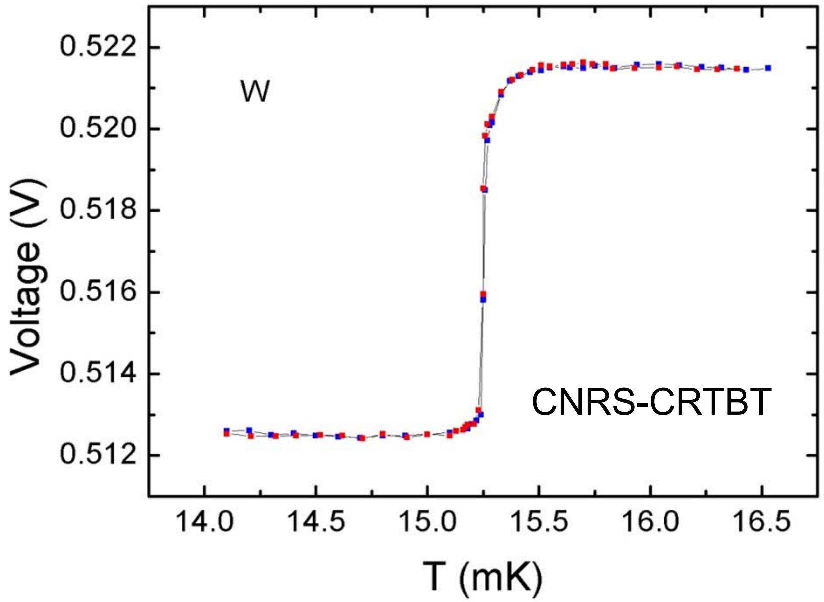
- The superconducting transition of several metallic elements or alloys provides convenient “markers” or “fixed points” for checking the accuracy of a local temperature scale.

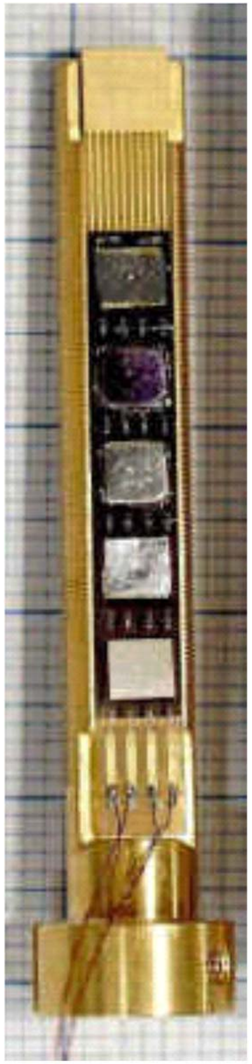
- The **SRM 767 and SRM 768 devices made by the NIST (formerly NBS)** are currently used in many cryogenic laboratories for this purpose. They consist of a bunch of metallic wires in thermal contact with a copper holder. A set of coils (mutual inductor) allows to determine the jump in susceptibility associated with the superconducting transition of the different wires.
- The corresponding temperatures (in the range 10 mK – 10 K) are given by a calibration performed at NIST.

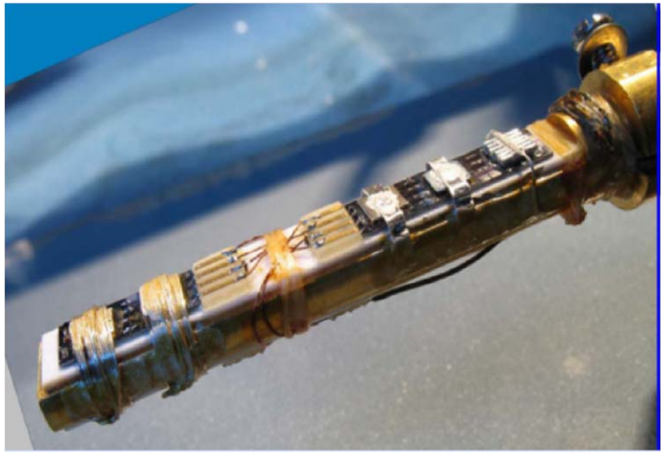
- Unfortunately, the system must be carefully shielded against magnetic fields, thus restricting severely its applicability in low temperature measurements.

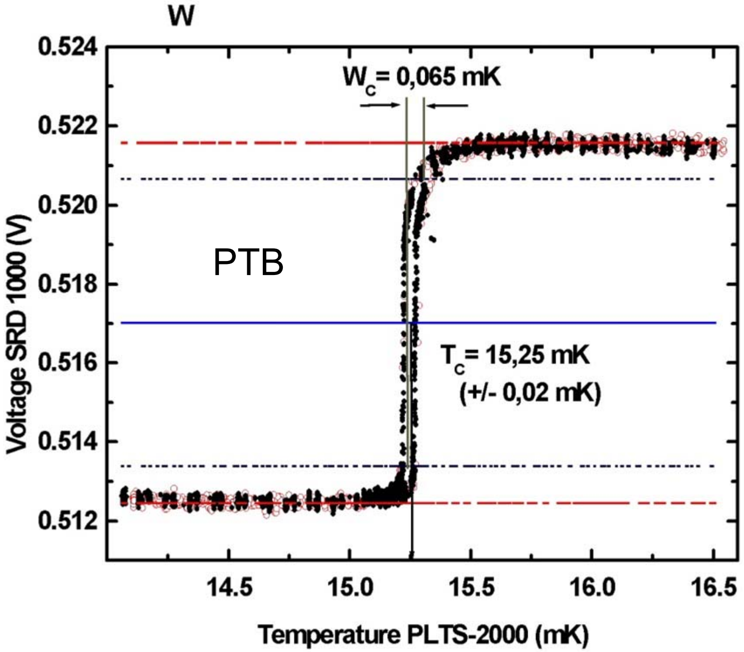
- These devices are not manufactured any longer, but **new superconducting fixed point devices (SRD1000)** have been developed in the framework of the European program on low temperature thermometry.

Fixed points device
Prototype SRD-1000





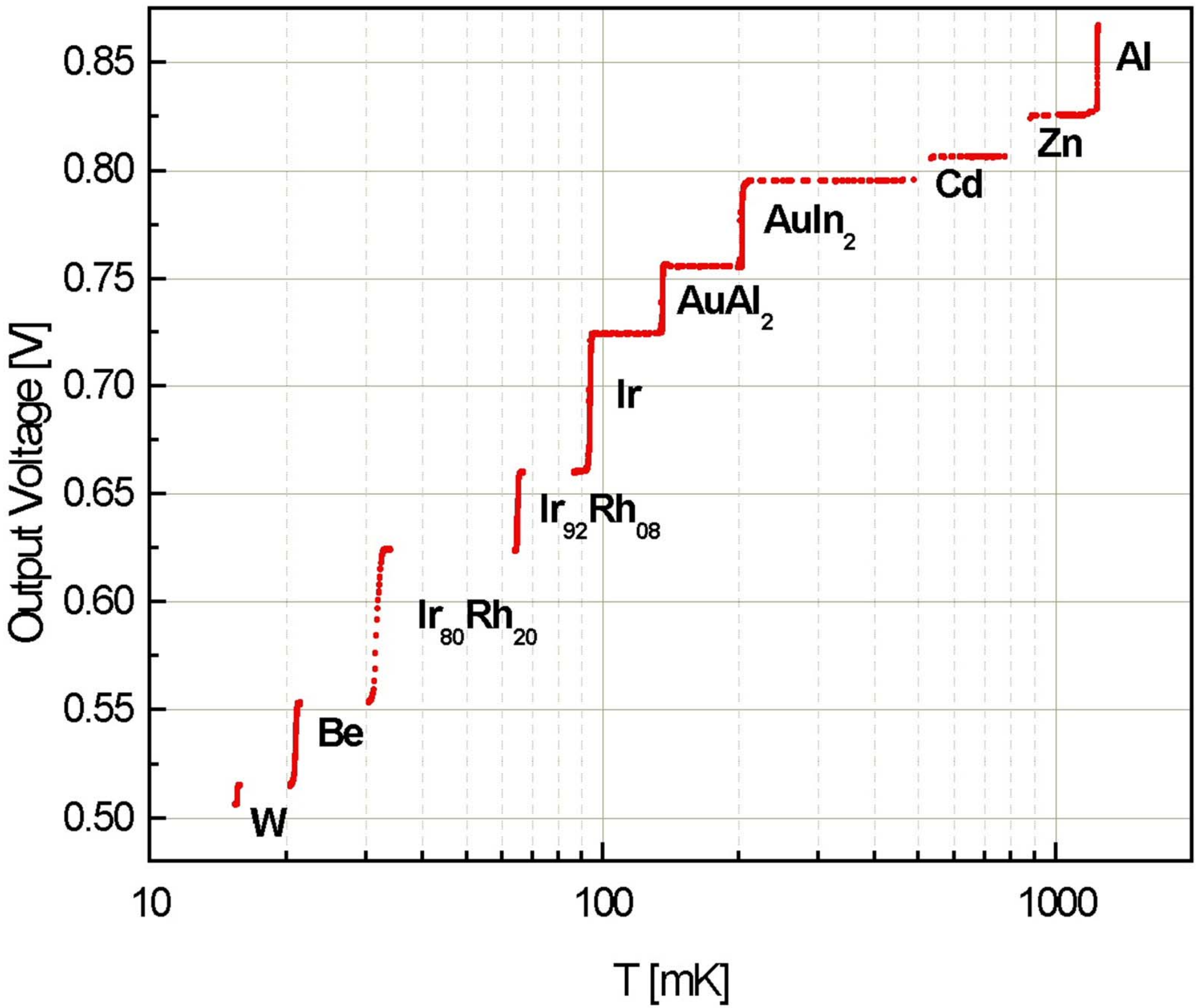




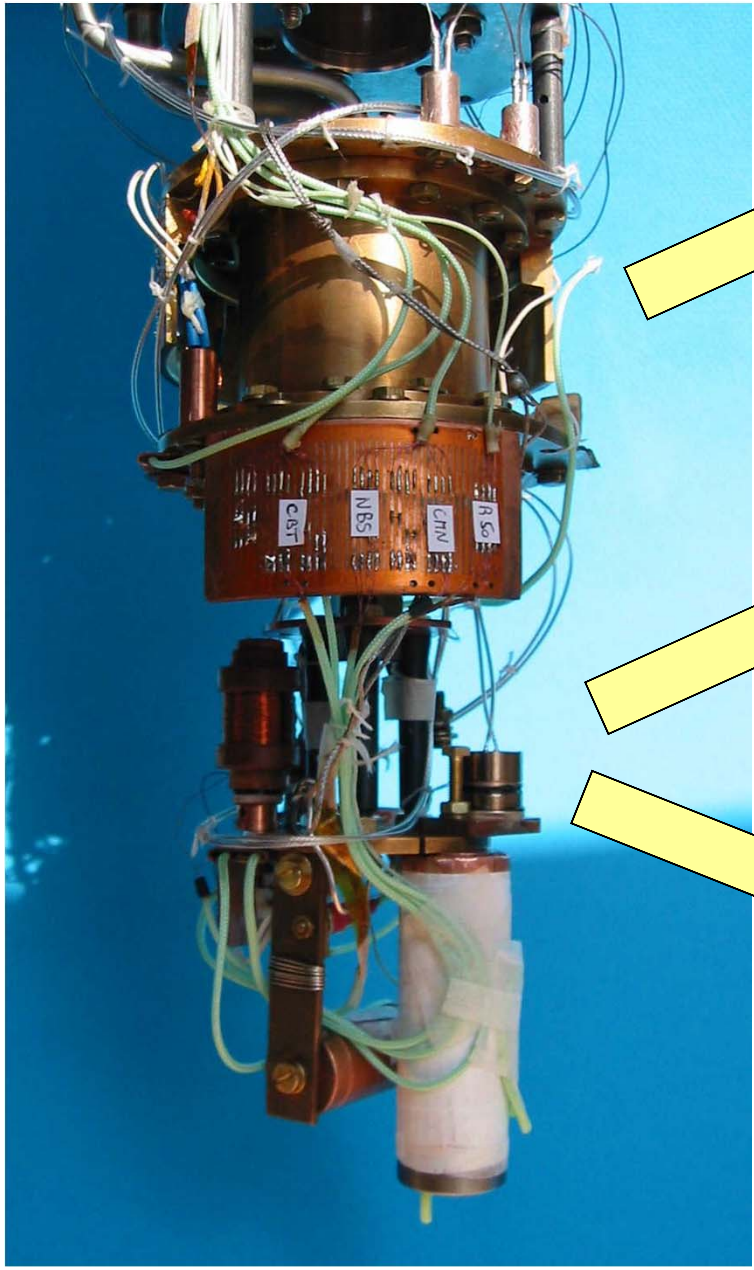
* Developed in Leiden, evaluated together with European metrology Laboratories
* 10 superconducting materials with Tc in the range 15 mK (W) to 1,2 K (Al)

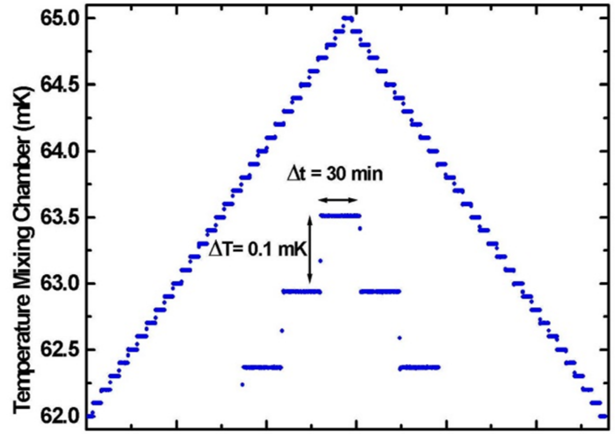
A. Peruzzi et al., TEMPMEKO 2004, Croatia (2004)
S. Schöttl et al., J. Low Temp. Phys. 138, 941 (2005)

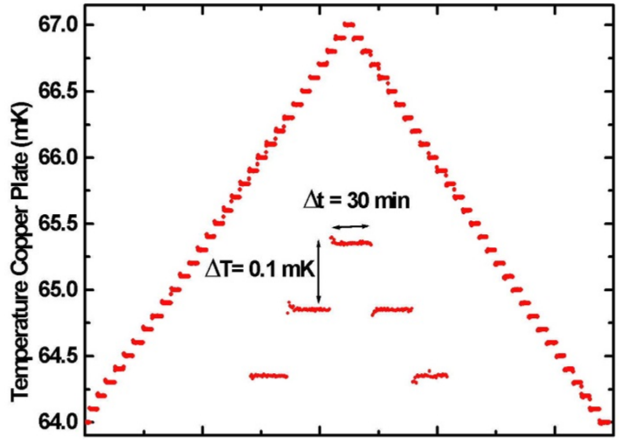
Evaluation of superconductive reference device

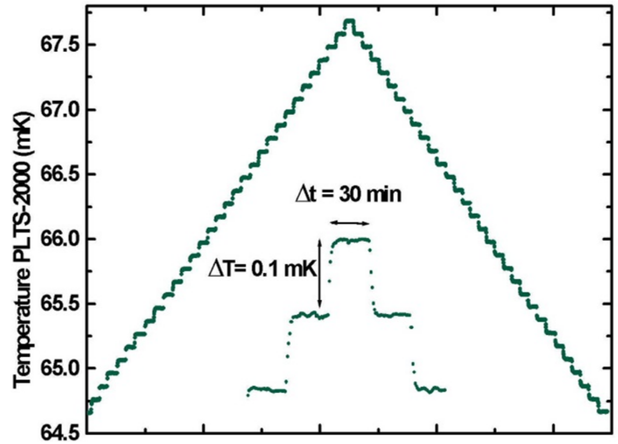


Calibration of the Fixed-points device

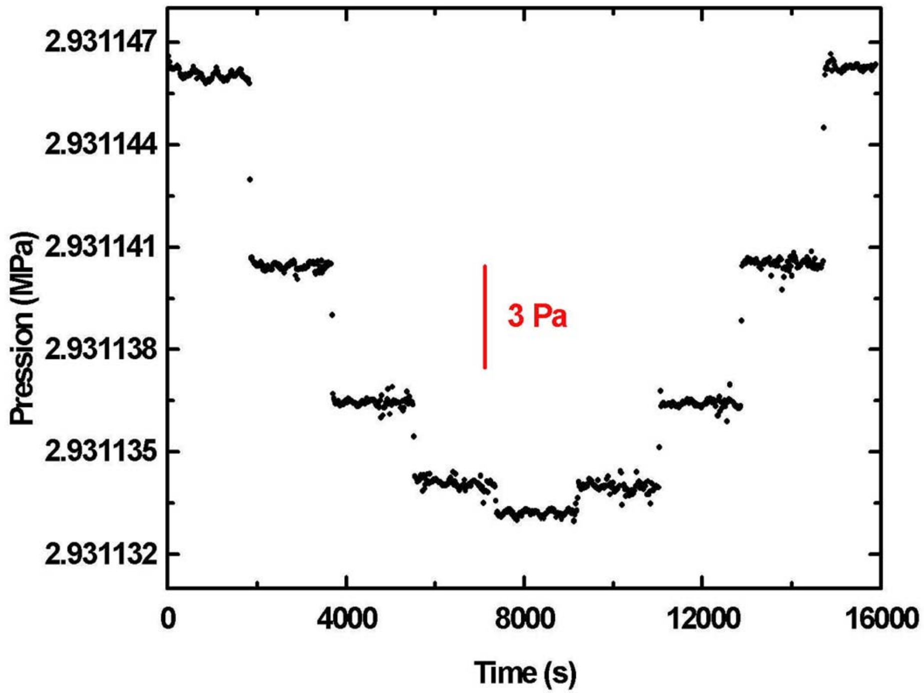


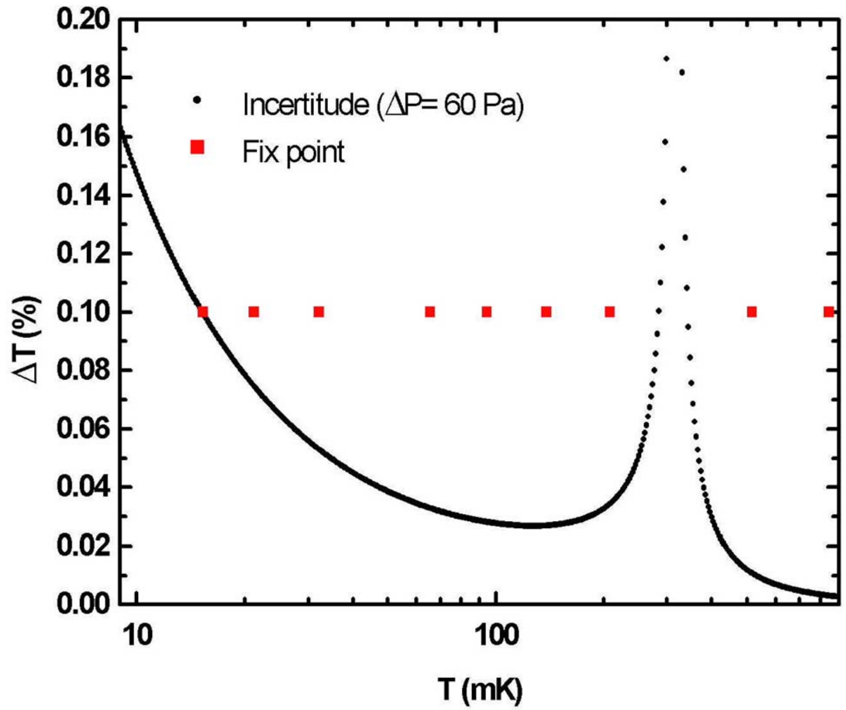


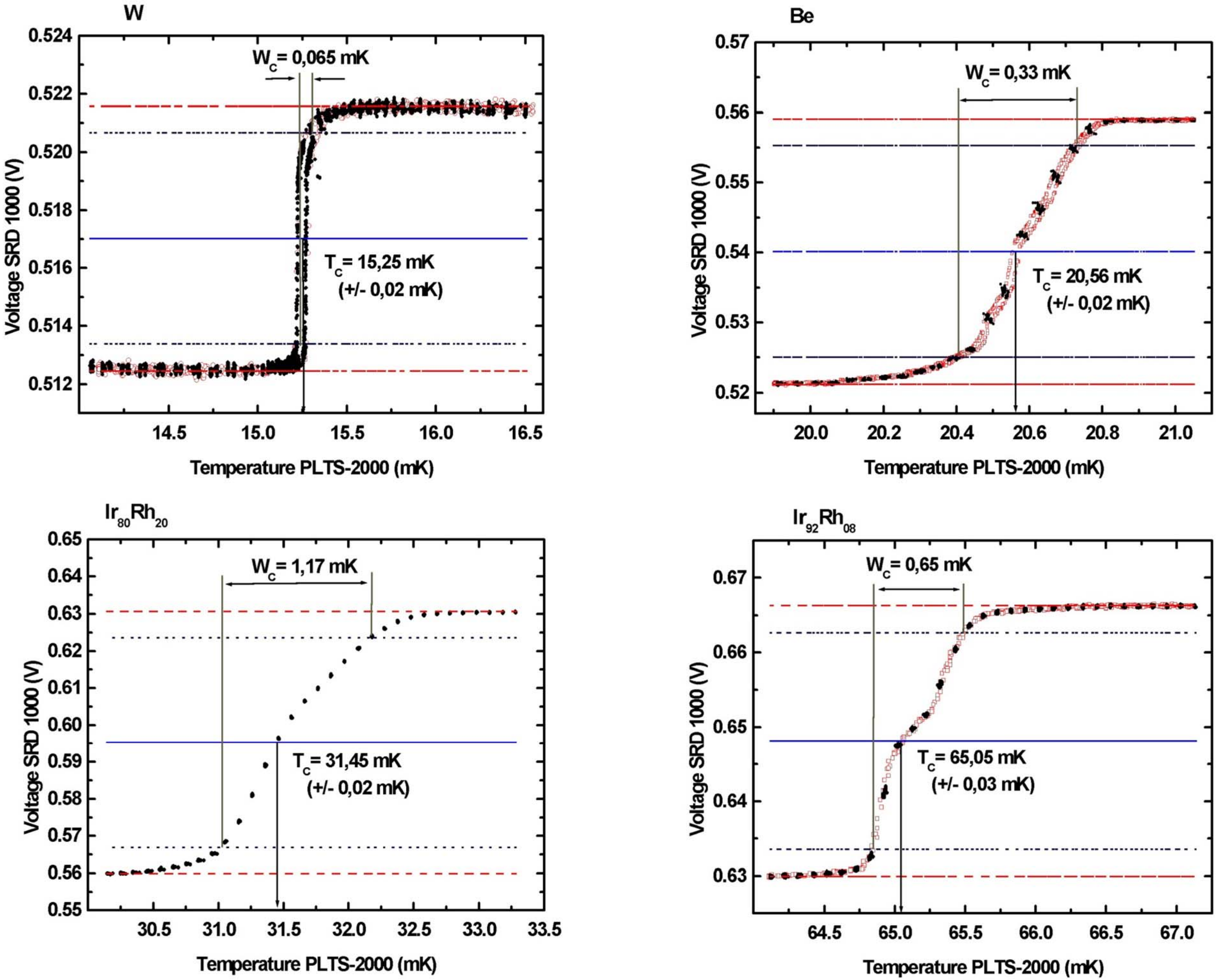


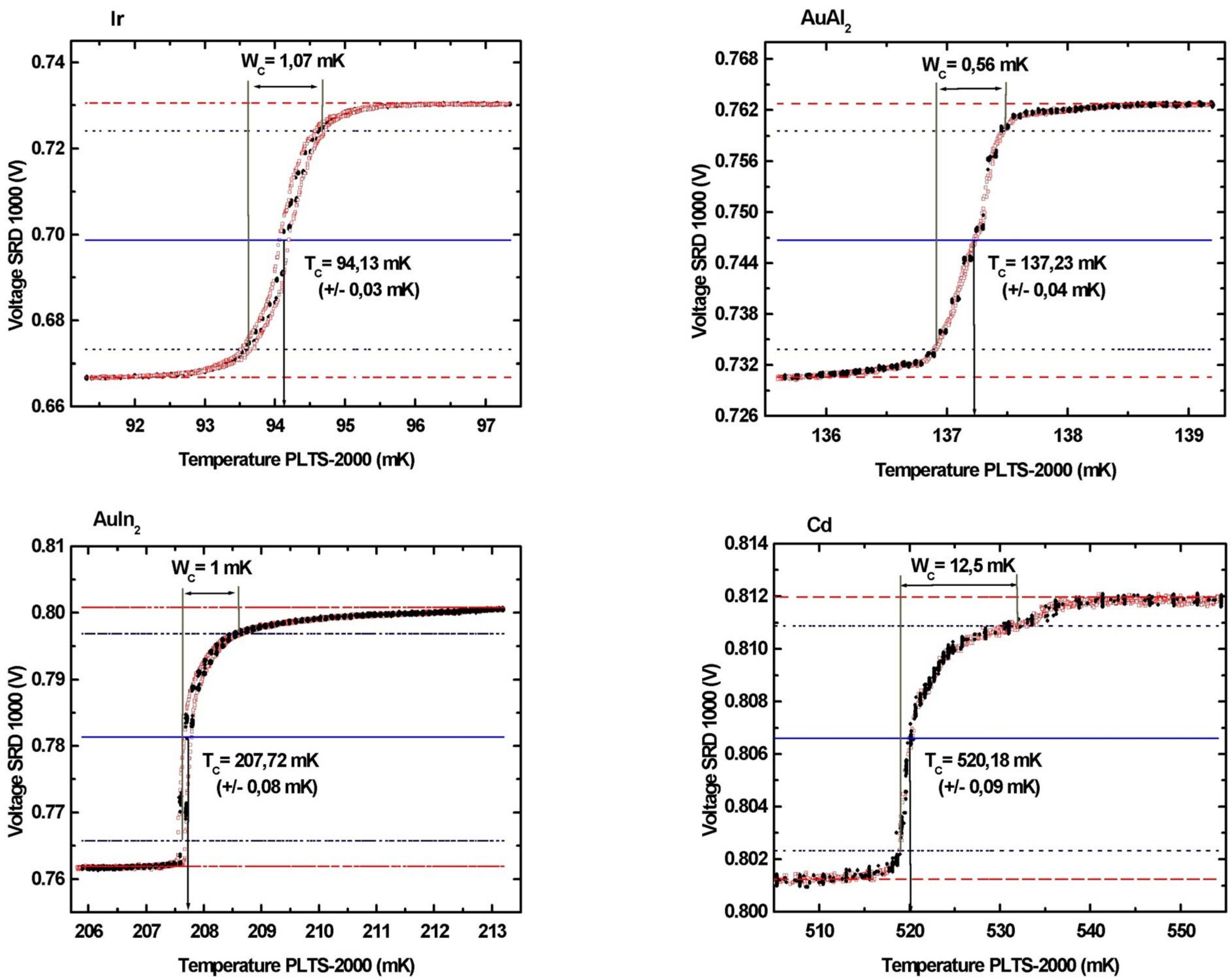


Sensitivity and uncertainty of MCT






W
Wc= 0,065 mK
Tc= 15,25 mK
(+/- 0,02 mK)
Voltage SRD 1000 (V)
Temperature PLTS-2000 (mK)
Be
Wc= 0,33 mK
Tc= 20,56 mK
(+/- 0,02 mK)
Voltage SRD 1000 (V)
Temperature PLTS-2000 (mK)
Ir80Rh20
Wc= 1,17 mK
Tc= 31,45 mK
(+/- 0,02 mK)
Voltage SRD 1000 (V)
Temperature PLTS-2000 (mK)
Ir92Rh08
Wc= 0,65 mK
Tc= 65,05 mK
(+/- 0,03 mK)
Voltage SRD 1000 (V)
Temperature PLTS-2000 (mK)



Ir
W_C = 1,07 mK
T_C = 94,13 mK
(+/- 0,03 mK)
Voltage SRD 1000 (V)
0.74
0.72
0.70
0.68
0.66
92
93
94
95
96
97
Temperature PLTS-2000 (mK)
AuAl2
W_C = 0,56 mK
T_C = 137,23 mK
(+/- 0,04 mK)
Voltage SRD 1000 (V)
0.768
0.762
0.756
0.750
0.744
0.738
0.732
0.726
136
137
138
139
Temperature PLTS-2000 (mK)
AuIn2
W_C = 1 mK
T_C = 207,72 mK
(+/- 0,08 mK)
Voltage SRD 1000 (V)
0.81
0.80
0.79
0.78
0.77
0.76
206
207
208
209
210
211
212
213
Temperature PLTS-2000 (mK)
Cd
W_C = 12,5 mK
T_C = 520,18 mK
(+/- 0,09 mK)
Voltage SRD 1000 (V)
0.814
0.812
0.810
0.808
0.806
0.804
0.802
0.800
510
520
530
540
550
Temperature PLTS-2000 (mK)

Material	**T (MCT)** [mK]	**ΔT** [%]	[mK]	**Wc** [mK]	**S/N**
W	15.25	0.1	0.02	0.065	23
Be	20.56	0.08	0.02	0.33	95
$Ir_{80}Rh_{20}$	31.45	0.054	0.02	1.17	177
$Ir_{92}Rh_{08}$	65.05	0.033	0.03	0.65	91
Ir	94.13	0.029	0.03	1.07	159
$AuAl_2$	137.23	0.027	0.04	0.56	80
$AuIn_2$	207.72	0.035	0.08	1.0	97
Cd	520.18	0.011	0.09	12.5	27

Transferring the scale using SRD1000 (preliminary work)

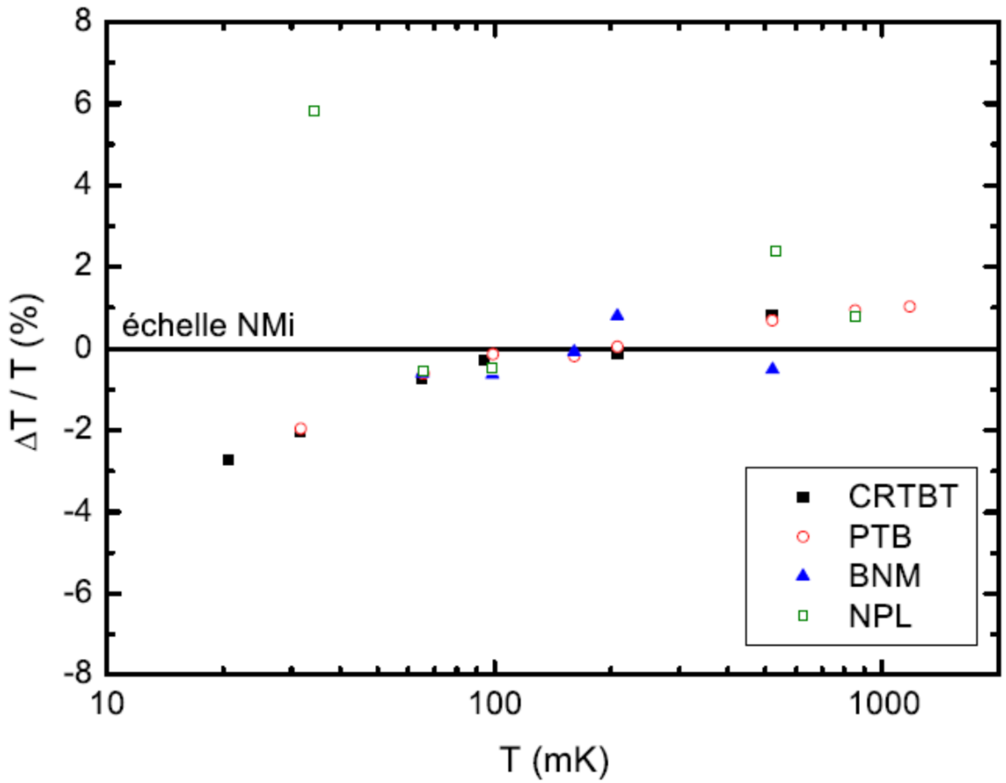


FIG. 2.13 – Comparaison des échelles de températures s'appuyant sur le thermomètre à courbe de fusion entre les différents laboratoires ayant testé les prototypes SRD1000.