

DRTBT2024

LABORATOIRE
NATIONAL
DE MÉTROLOGIE
ET D'ESSAIS



le cnam



MÉTROLOGIE DES BASSES TEMPÉRATURES

Laurent Pitre

LNE-Cnam

Cnam



University established in 1794
Staff: 2,500; Students: 70,000
Store the metric standard since 1848

LNE



Providing calibration services since 1901
Staff: 1,500

Note that Bureau International des Poids et Mesures is a metrology international organism but not link to Cnam or LNE

LNE-Cnam



LNE (78 Trappes)

**The other part of
Photometry and
Radiometry**

**Thermometry
One part of
Photometry and
Radiometry**



Cnam (93 Saint-Denis)



LNE (75 Paris)

Length

Thermometry

We never measure the temperature directly; we measure an effect that depends on the temperature.

During this talk, I will present measurements on pressure, capacitance, resistance, magnetic susceptibility, acoustic resonance, microwave resonance, phase transition of pure substances, superconductor transition, noise and liquid dilation.

The very first thermometer

The first scale made by Torricelli, Ferdinand II de Medici and Galileo in 1650

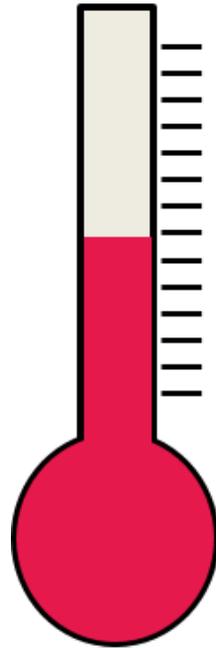


from <https://www.museogalileo.it/en>

The very first thermometer

In 1650 -50 years before Fahrenheit and 100 years before Celsius

- A thermometer repeatable after **370 years!!!**
- More than 300 thermometers were produced.
- Exchangeable thermometer.
- **Use of freezing point of water as temperature reference in 1650 !!!!**



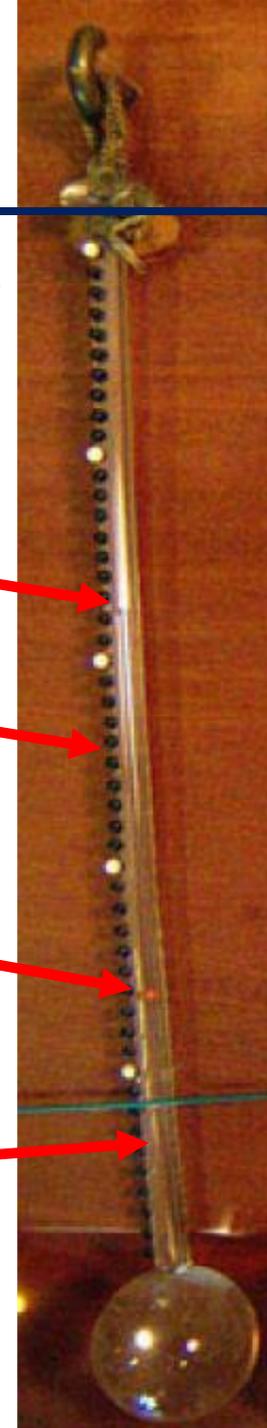
Camuffo D. and Bertolin C. "The earliest temperature observations in the world: the Medici Network (1654–1670)" Climatic Change (2012)

Alcohol 80 %

Spacing for the graduation 1,8 mm

freezing point of water!!!
Used to seal thermometer

Internal diameter from 1,1 to 1,2 mm.

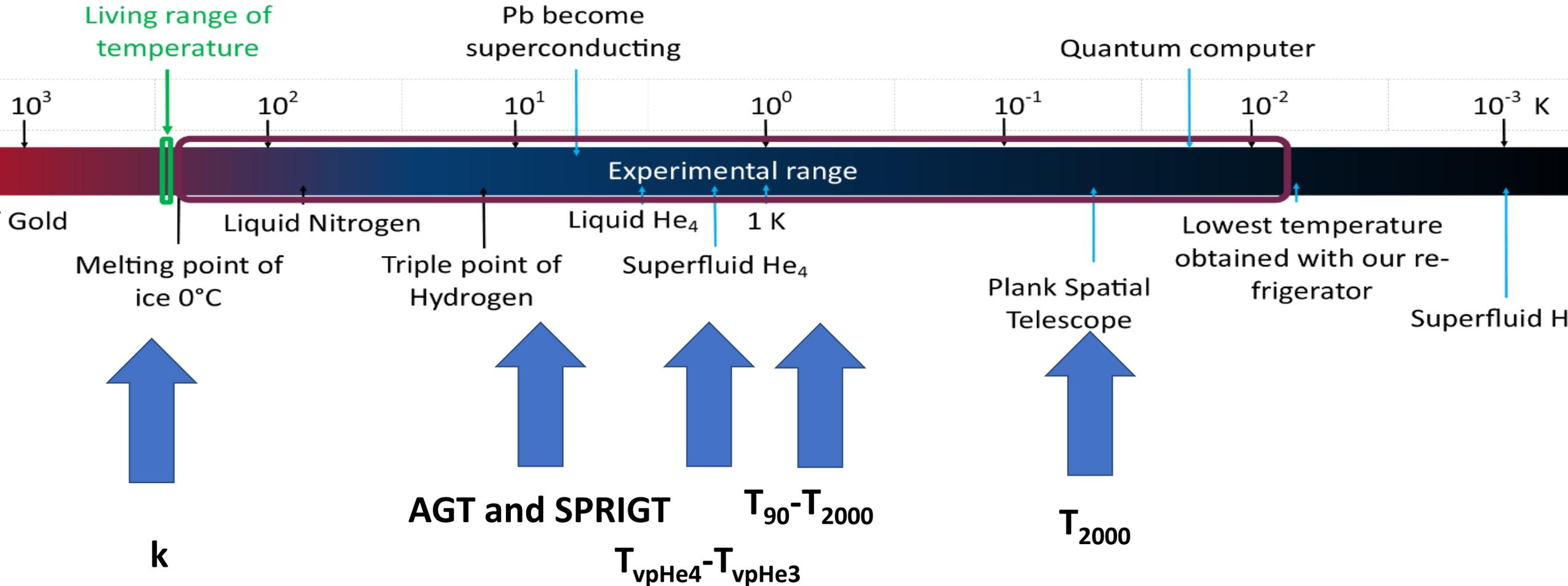


A good thermometer

- The relationship between what we measure and the temperature must be established by the laws of physics.
- The thermometer should be able to be used across a very wide range of temperatures.
- It should be precise and stable over time.
- It should be robust, easy to use, affordable and with very high resolution.
- It should be able to be used in environments inaccessible to humans: chemical, magnetic, nuclear, vacuum, etc

This thermometer don't exist

The Low temperature at LNE-Cnam



Outline

- International System of Units
- Boltzmann constant determination
- Thermometry below 1K
 - PLTS2000
 - SDR1000
 - MFFT
 - Resistance measurement
- ITS90
 - 0.65K-5K vapor pressure of He₃ and He₄
 - Primary thermometer, SPRIGT and AGT
 - 24.5K-273K Fix point and CSPRT25
 - RdFe
- Conclusion and the search of metrological sensor

Why an International System of Units ?

In the past different country = different units
This harmed commerce and the spread of idea

Over time many systems of units were used:

Sumerian Nippur cubit 4500 years old

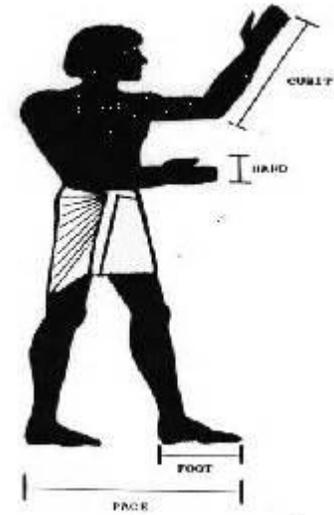
Egypt cubit 3500 years old

China 尺 (chi) 3000 years old

France Lieue de Paris 1700

English British Imperial Unit 1825

France *mètre* and the metric system 1790

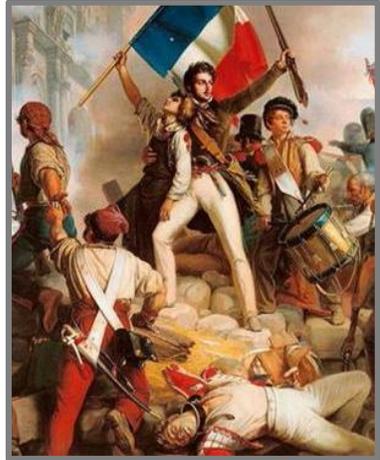


France 1789: register of grievances

Sagesse.
13.
que la diversité de poids et de mesures de Province
à Province et de Ville à Ville, étant un
obstacle à la prospérité du Commerce dont
elle ralentit et gêne toutes les opérations,
il soit établi un même poids et une
septième page / Le Marquis de Marquisville, premier
conseil au sieur

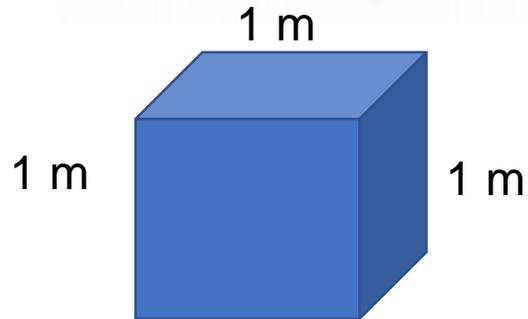
Extrait du cahier de doléances de Nîmes (1789). Arch. dép. du Gard : C 1196.

The historical metric system (1793)



Decimal unit

Divide by 10 000 000 any $\frac{1}{4}$ of meridian of Earth and you have 1 metre



A cube of 1 m \times 1 m \times 1 m filled with water is 1000 kg



With a metre long pendulum you get 2 second

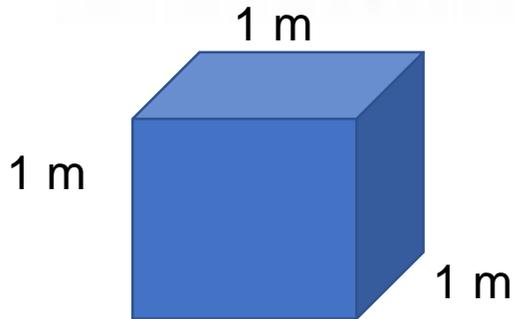
Can be realized anytime, anywhere

The historical metric system (1793)



Concept not easy to realize, not precise enough

7 years for two surveyors to measure 1000 km along a meridian and get 1 metre



Depending of the temperature of the water and its quality



Gravity is not constant on Earth. To get 2 s the pendulum length is actually 99.7 cm

Artefact is more stable and easy to use

Units defined by artefacts before 2019

Mass

Length

Thermodynamic Temperature



Credit: NIST

Kilogram

Platinum-iridium kilogram

Credit: NIST

Meter

Platinum-iridium meter bar

Credit: NIST

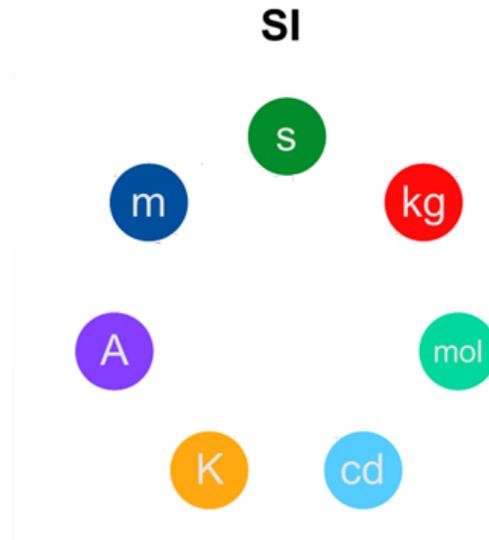
Kelvin

Triple-point of water cell

Artefacts are convenient to use and standardize but not stable for long term

The historical metric system (1793)

- 1799 metric system adopted by law of the french republic
- 1800-1810 Napoleon impose the metric system in all Europe except UK
- 1860 Maxwell and Lord Kelvin add electric units and make it coherent (CGS)
- 1870 Metre convention (17 countries) only for m and kg
- 1971 The *Système International* (SI) with 7 base units



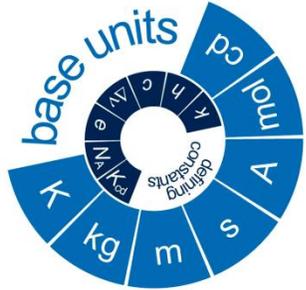
BUT all those units were linked to artefacts, and

- the artefacts are not stable over a long time period (100 years)
- the artefacts reduce the possibility of innovation

The new SI opens the way to new possibilities

Units redefined by universal constants

The 7 commandments



- **Time** : the caesium hyperfine frequency, $\Delta\nu_{\text{Cs}}=9\,192\,631\,770\text{ Hz}$
- **Length** : the speed of light in vacuum, $c=299\,792\,458\text{ m/s}$
- **Mass**: the Planck constant, $h=6.626\,070\,15\times 10^{-34}\text{ Js}$
- **Electric Current**: the elementary charge, $e=1.602\,176\,634\times 10^{-19}\text{ C}$
- **Thermodynamic Temperature**: the Boltzmann constant, $k=1.380\,649\times 10^{-23}\text{ J/K}$
- **Amount of Substance**: the Avogadro constant, $N_{\text{A}}=6.022\,140\,76\times 10^{23}\text{ mol}^{-1}$
- **Luminous Intensity**: the luminous efficacy of a defined visible radiation,
 $K_{\text{cd}}= 683\text{ lm/W}$

Consequence in temperature: the triple point of water loses its central role in temperature measurements

Boltzmann constant determination

LNE 2017 Helium

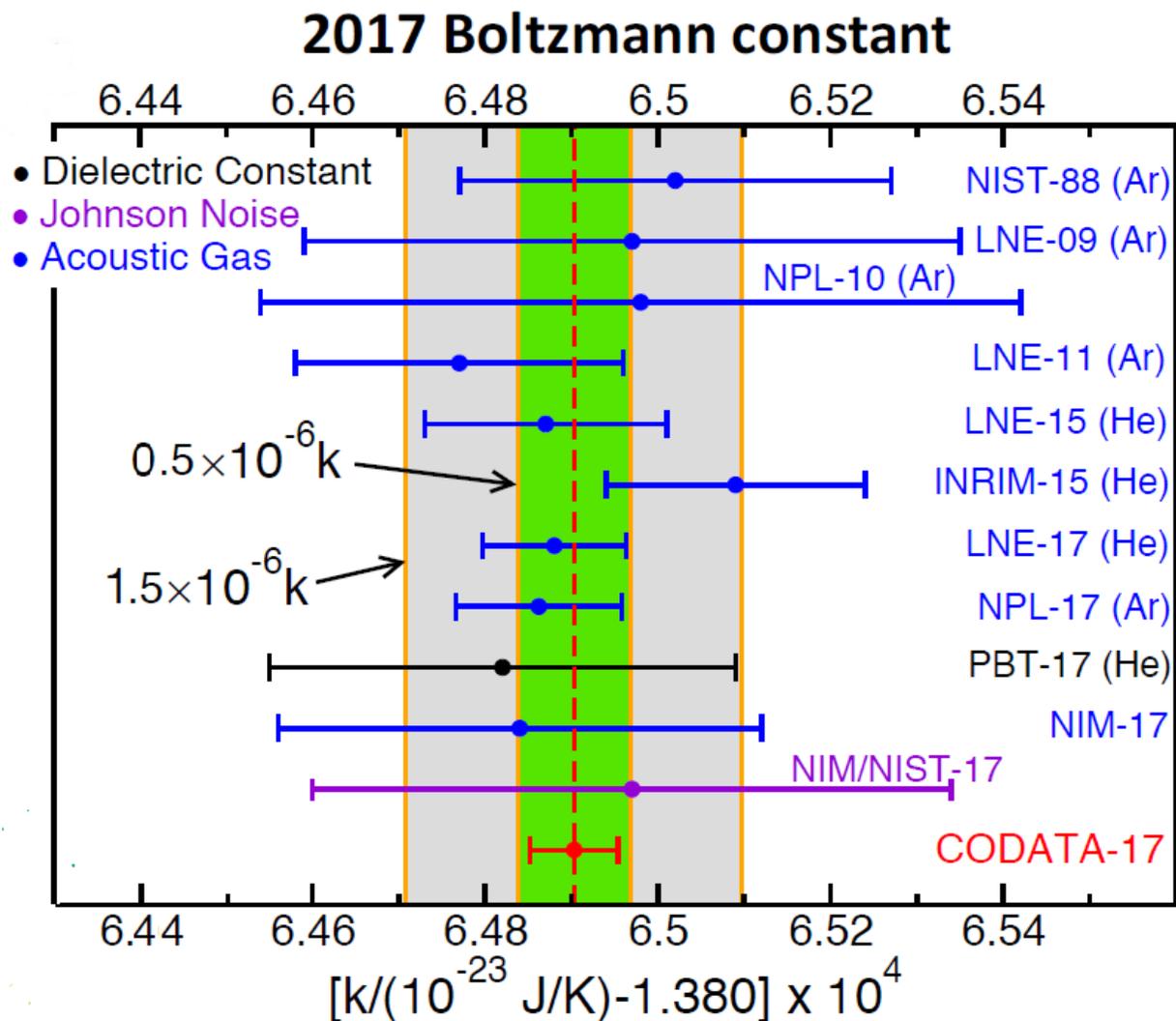
$$k = 1.380\,648\,37(81) \times 10^{-23} \text{ J/K}$$

relative uncertainty

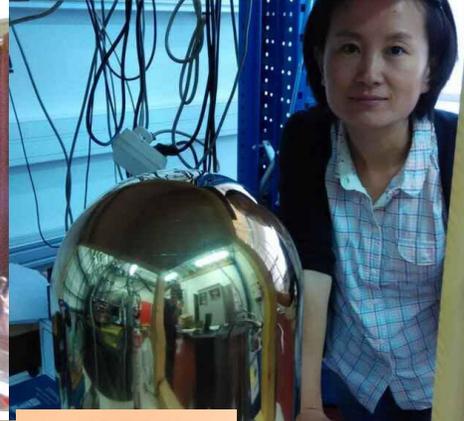
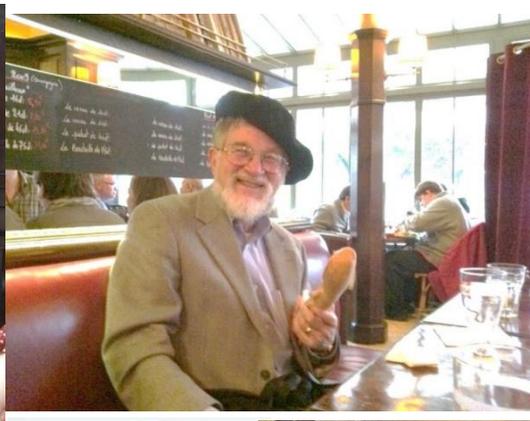
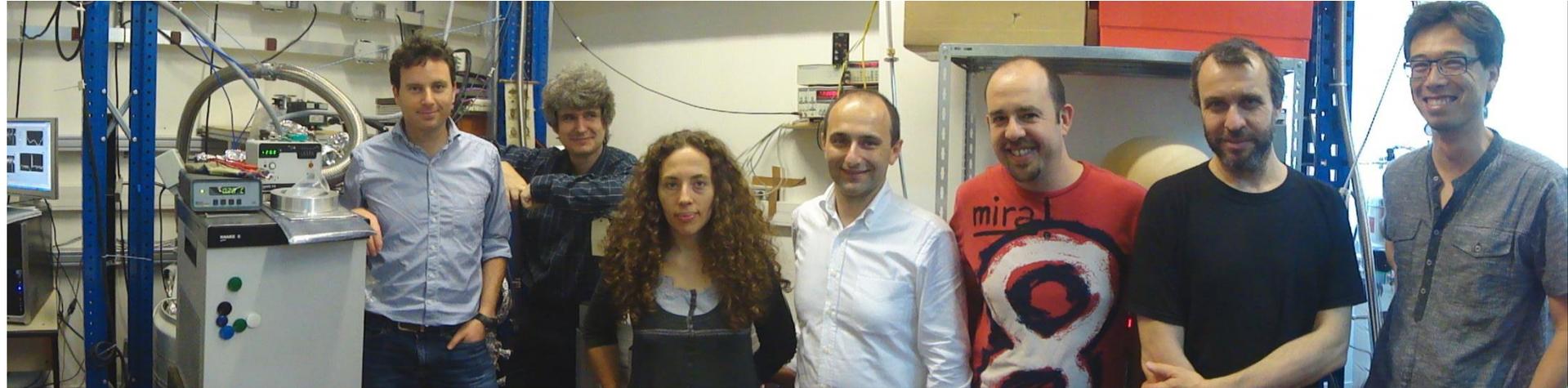
$$u(k) / k = 0.60 \times 10^{-6}$$

$$k = 1.380649 \cdot 10^{-23} \text{ J} \cdot \text{K}^{-1}$$

Depuis 2019



A Team

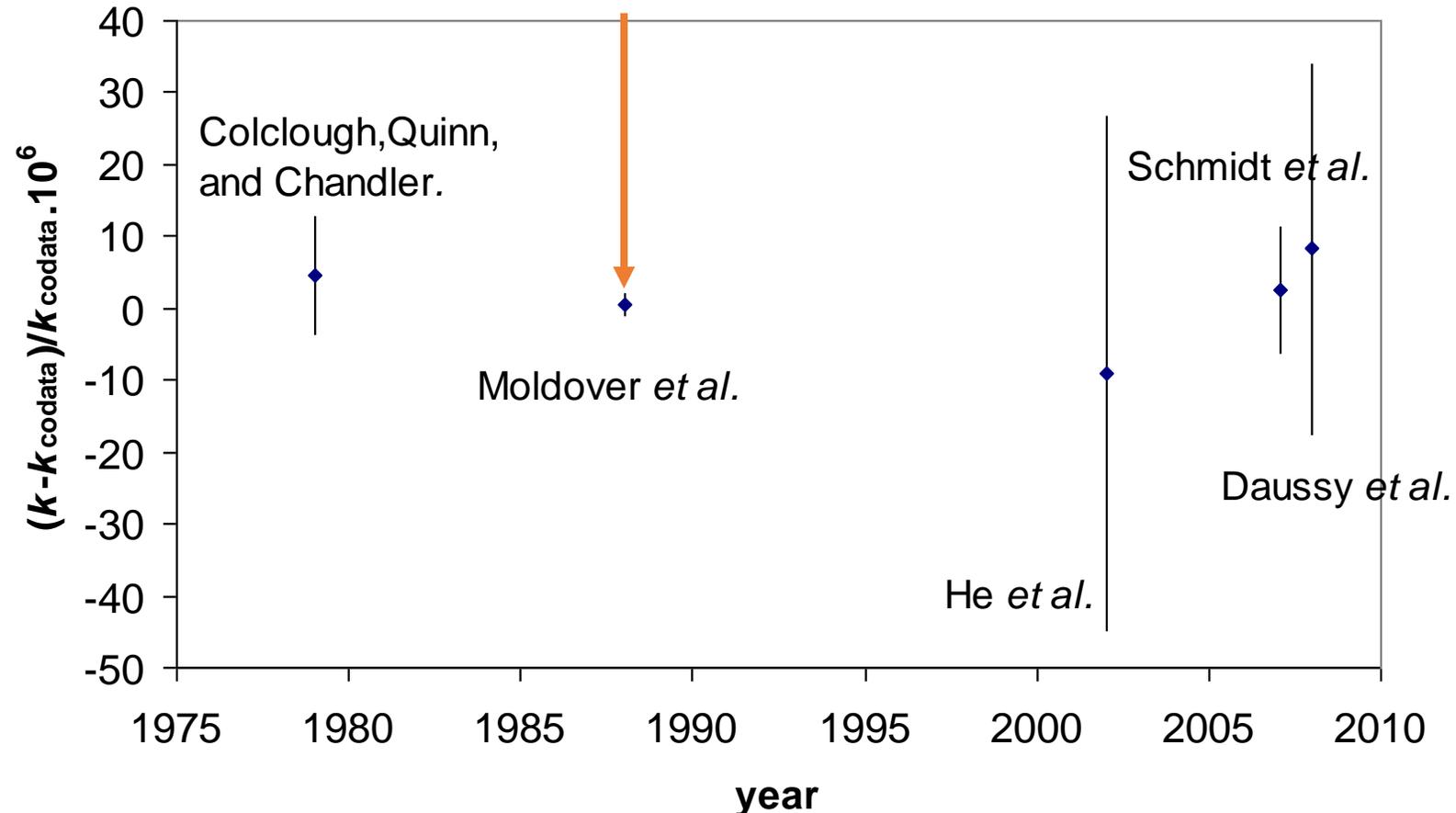


L.Pitre, F. Sparasci, L. Risegari, C. Guianvarc'h, C. Martin, M. E. Himbert, M. Plimmer, A. Allard, B. Marty, P. G. Albo, B. Gao, M. R. Moldover and J. B. Mehl

Boltzmann constant determination

The situation in 2008

Uncertainty is 7 times lower compared to the other results

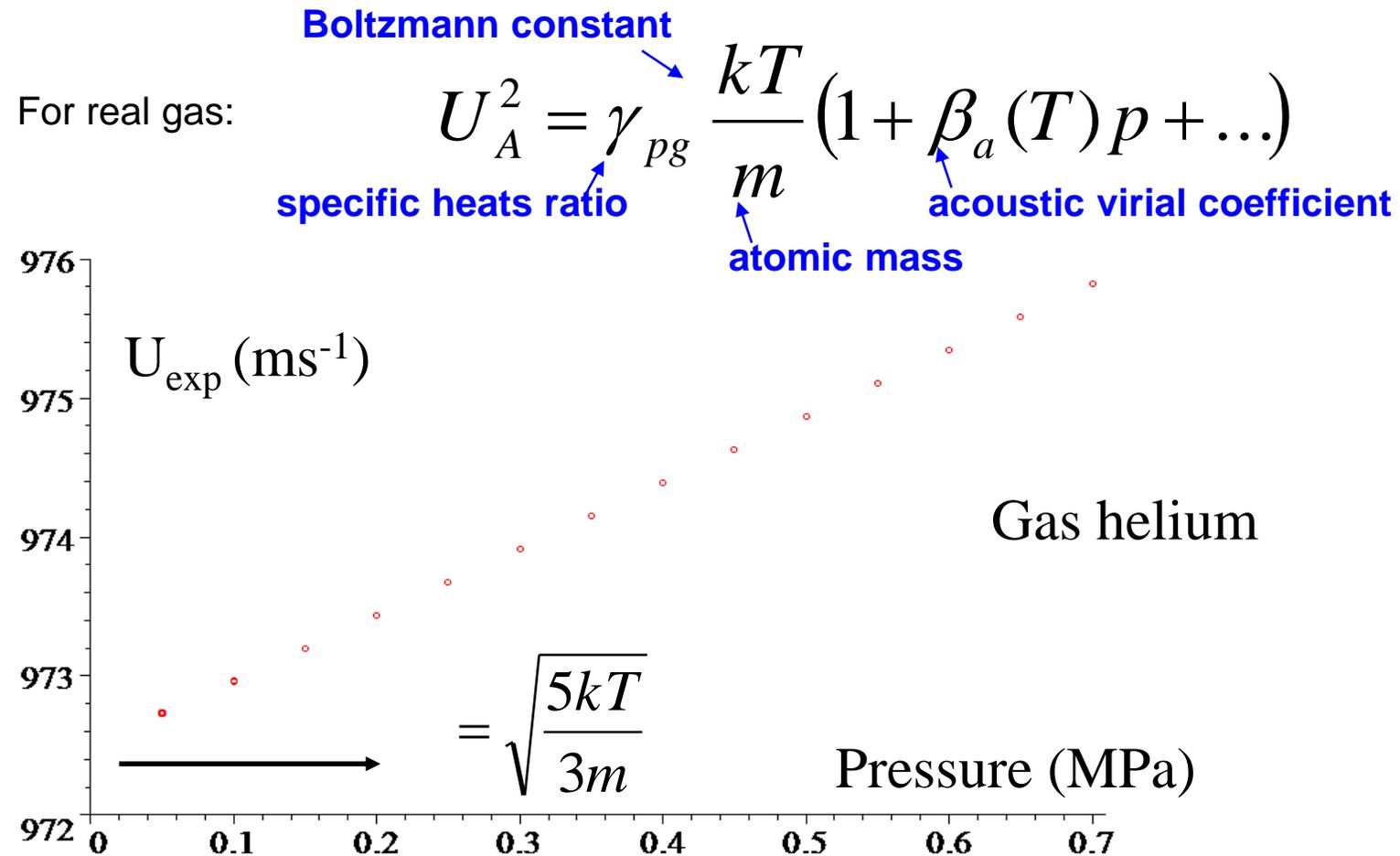


if $k=1$, body temperature = $4.2822209384000000000224 \cdot 10^{-21} \text{K}$

If the numerical value of k is correct, no experiment should detect any change due to the redefinition of the unit

Acoustic Determination of the Boltzmann Constant

Principle of the experiment



Standing wave in a spherical cavity

Speed of sound

$$U_A = \frac{\text{Radius}}{Z_{nl}^A} 2\pi \langle f_{nl}^A + \Delta f_{nl}^A \rangle$$

Eigenvalue
Resonance frequency

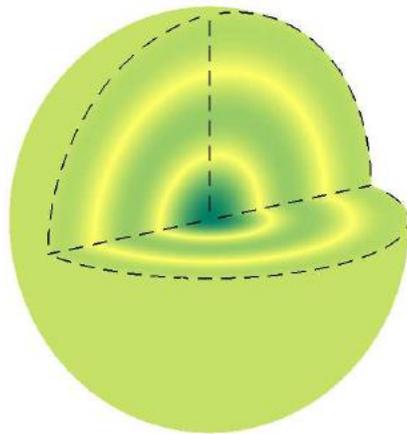
Boundary layer
tube and
microphone effect

Eigenvalue Speed of light

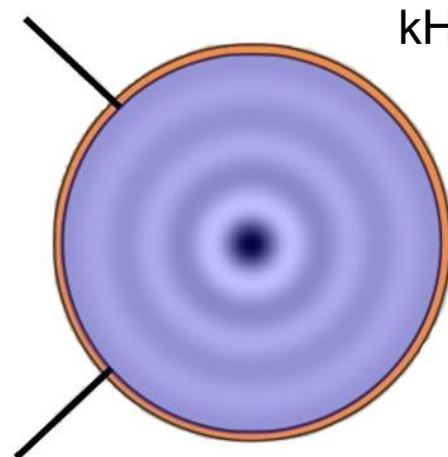
$$\text{Radius} = \frac{Z_{nl}^{EM} c}{2\pi \langle f_{nl}^{EM} + \Delta f_{nl}^{EM} \rangle}$$

Resonance frequency

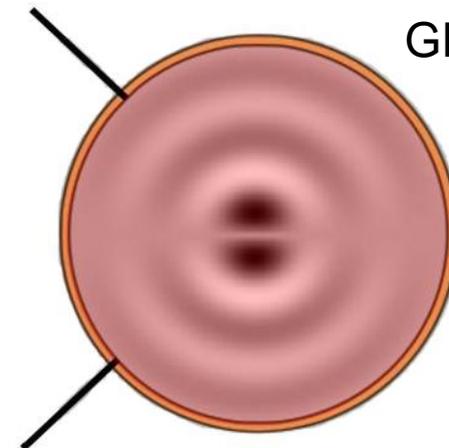
Skin depth
Holes and
Antennas effect



$l = 0, n = 3$



kHz



GHz

k determination with speed of sound measurement

Principle of the experiment

Simultaneous acoustic (u) and microwave (c) resonances in a cavity

Acoustic resonances measurement

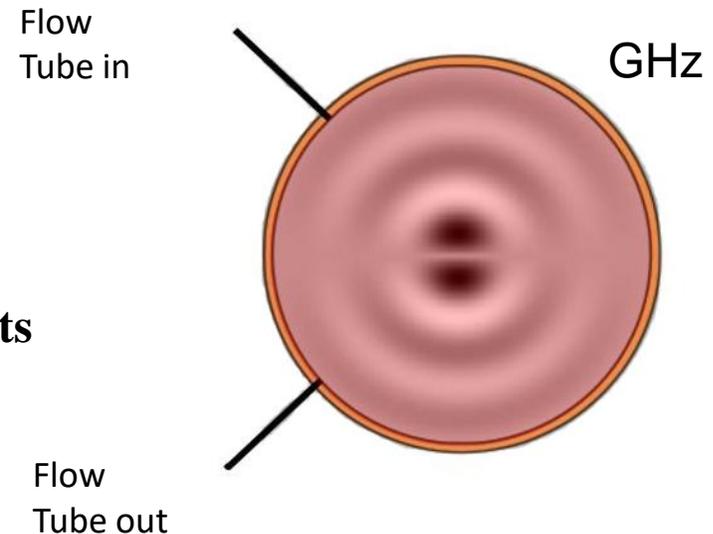
⇒ Boltzmann constant but linked to a volume

Microwave resonances measurement

⇒ Volume measurement

Gas flow minimizes the effects of impurities

Non-quite spherical simplify the microwave measurements



Acoustic Determination of the Boltzmann Constant

Relationship between the Boltzmann constant and acoustic/microwave measurements

With $U_A^2 = \gamma_{pg} \frac{kT}{m} (1 + \beta_a(T)p + \dots) U_A = \frac{Radius}{Z_{nl}^A} 2\pi \langle f_{nl}^A + \Delta f_{nl}^A \rangle Radius = \frac{Z_{nl}^{EM} c}{2\pi \langle f_{nl}^{EM} + \Delta f_{nl}^{EM} \rangle}$

Gas atomic mass Speed of light in vacuum (exact)

$$k = \left\langle \frac{3}{5} \frac{mc_0^2}{T_{tp,water}} \left(\frac{Z_{nl}^{EM}}{Z_{nl}^A} \right)^2 \lim_{p \rightarrow 0} \left(\frac{\langle f_{nl}^A + \Delta f_{nl}^A \rangle}{\langle f_{nl}^{EM} + \Delta f_{nl}^{EM} \rangle} \right)^2 \right\rangle$$

Measured resonance frequency Correction (theory)

Quasi-sphere's eigenvalues

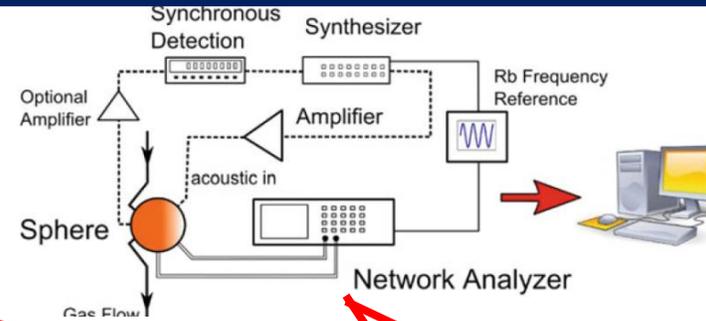
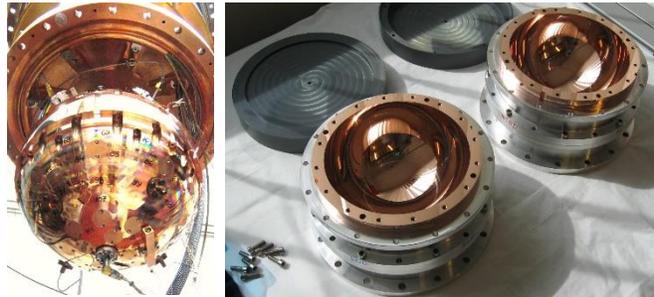
Average over measured acoustic and electromagnetic modes

Polynomial extrapolation to Zero pressure limit

Ratio: removes artefact effects at the first order

k determination with speed of sound measurement

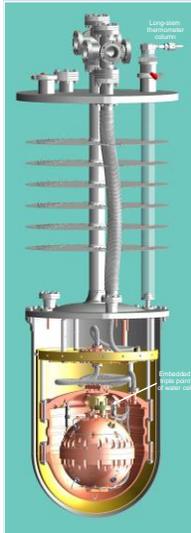
A 3.1 liter sphere



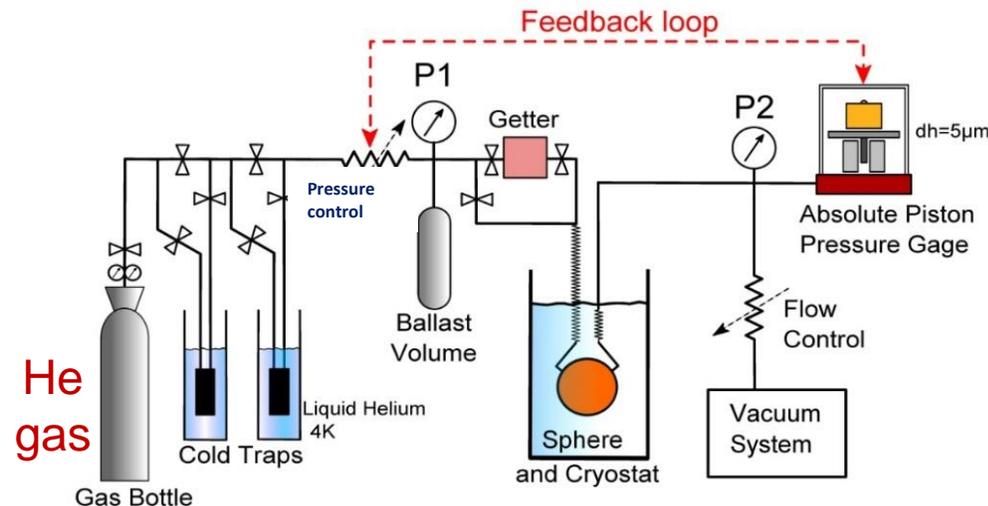
20 Instruments
used at the
state of the art

$$k = \left\langle \frac{3}{5} \frac{mc_0^2}{T_{tp,water}} \left(\frac{Z_{nl}^{EM}}{Z_{nl}^A} \right)^2 \lim_{p \rightarrow 0} \left(\frac{\langle f_{nl}^A + \Delta f_{nl}^A \rangle}{\langle f_{nl}^{EM} + \Delta f_{nl}^{EM} \rangle} \right)^2 \right\rangle$$

Adiabatic Cryostat
(weak link to the thermal bath)

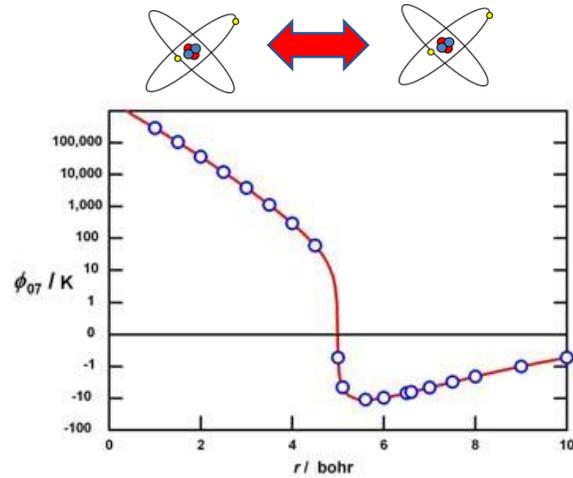


Ultra clean gas handling systems
With a piston gage as pressure measurement

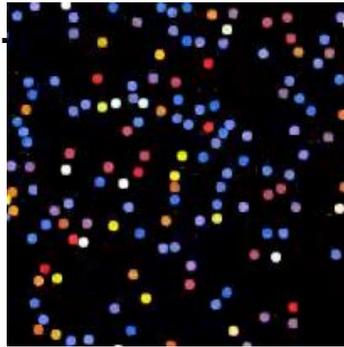


ab initio calculation for the thermo-physical propriety of Helium gas

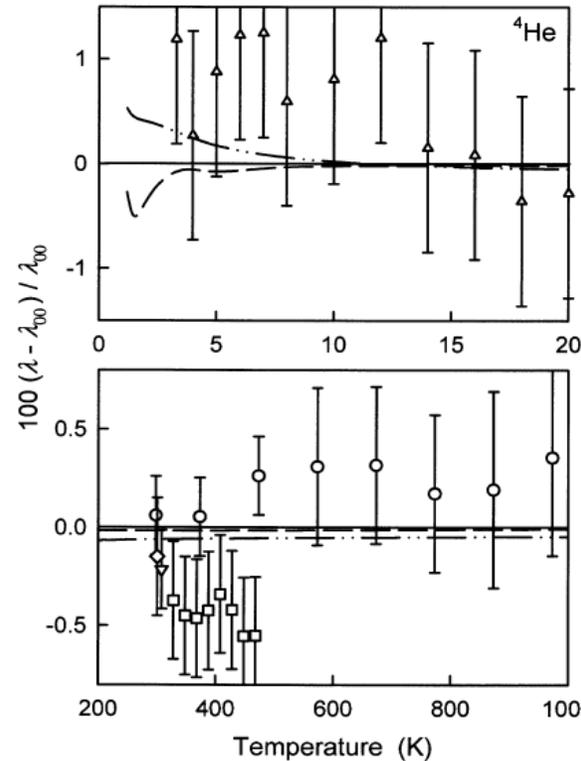
Microscopic propriety



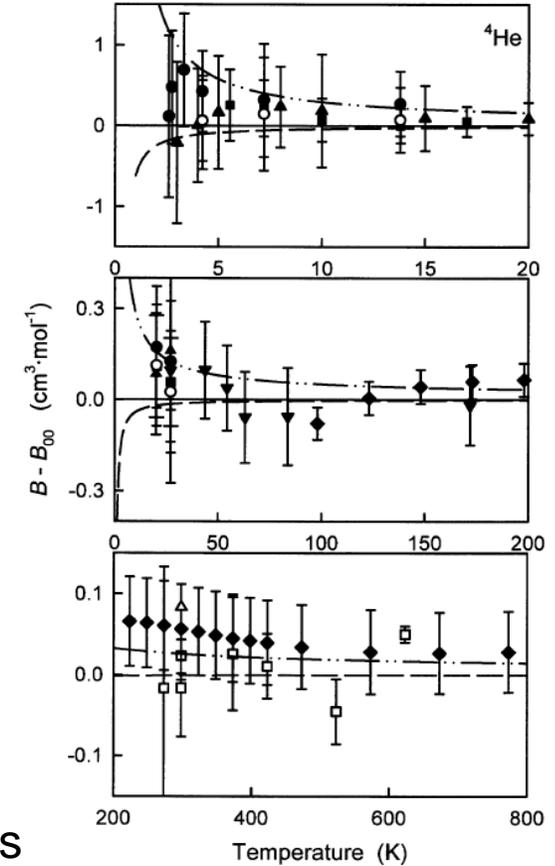
Simulation of many collisions with the helium interaction potential.



Macroscopic propriety



Conductivity of helium-4 gas



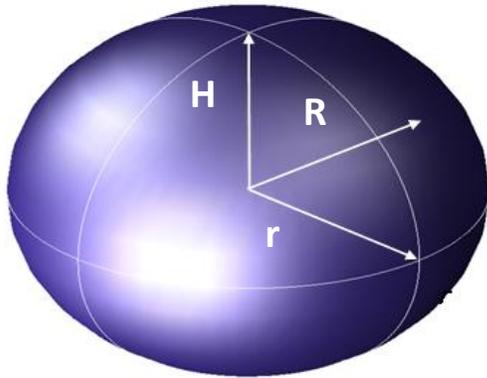
viscosity of helium-4 gas

Ab initio calculation of fluid properties for precision metrology

Giovanni Garberoglio, Christof Gaiser, Roberto M Gavioso, Allan H Harvey, Robert Hellmann, Bogumił Jeziorski, Karsten Meier, Michael R Moldover, Laurent Pitre, Krzysztof Szalewicz, Robin Underwood J. Phys. Chem. Ref. Data 52, 031502 (2023)

A Non-Quite Spherical Cavity

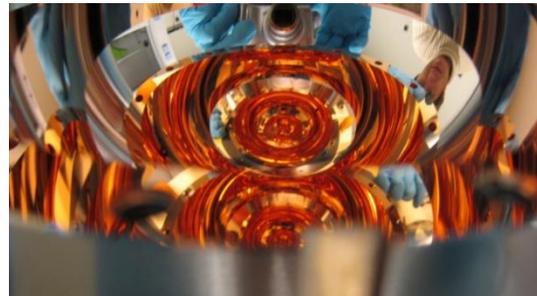
- The use of a slightly deformed spherical geometry, a triaxial ellipsoid, removes the degeneracy of resonator modes



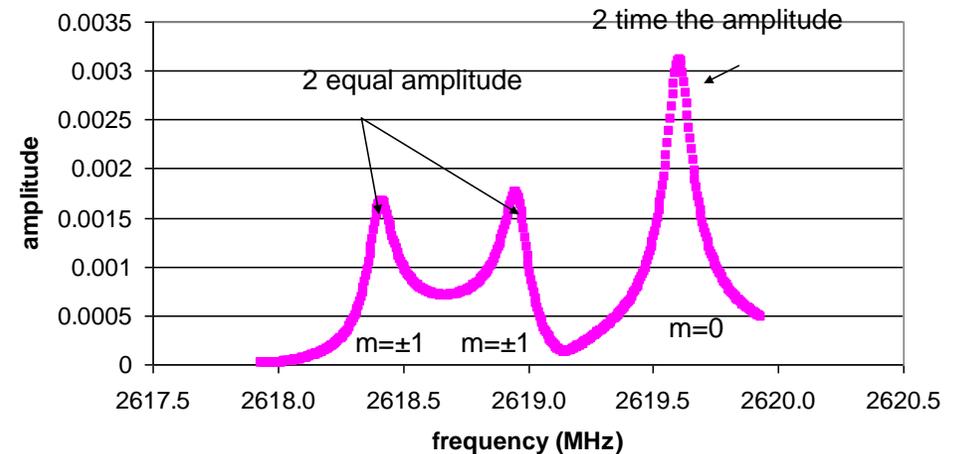
$$\frac{x^2}{(89.950)^2} + \frac{y^2}{(89.975)^2} + \frac{z^2}{(90.000)^2} = 1$$

Inner shape: the difference between r, R and H is 0.025 mm:

H = 90.000 mm
R = 89.975 mm
r = 89.950 mm

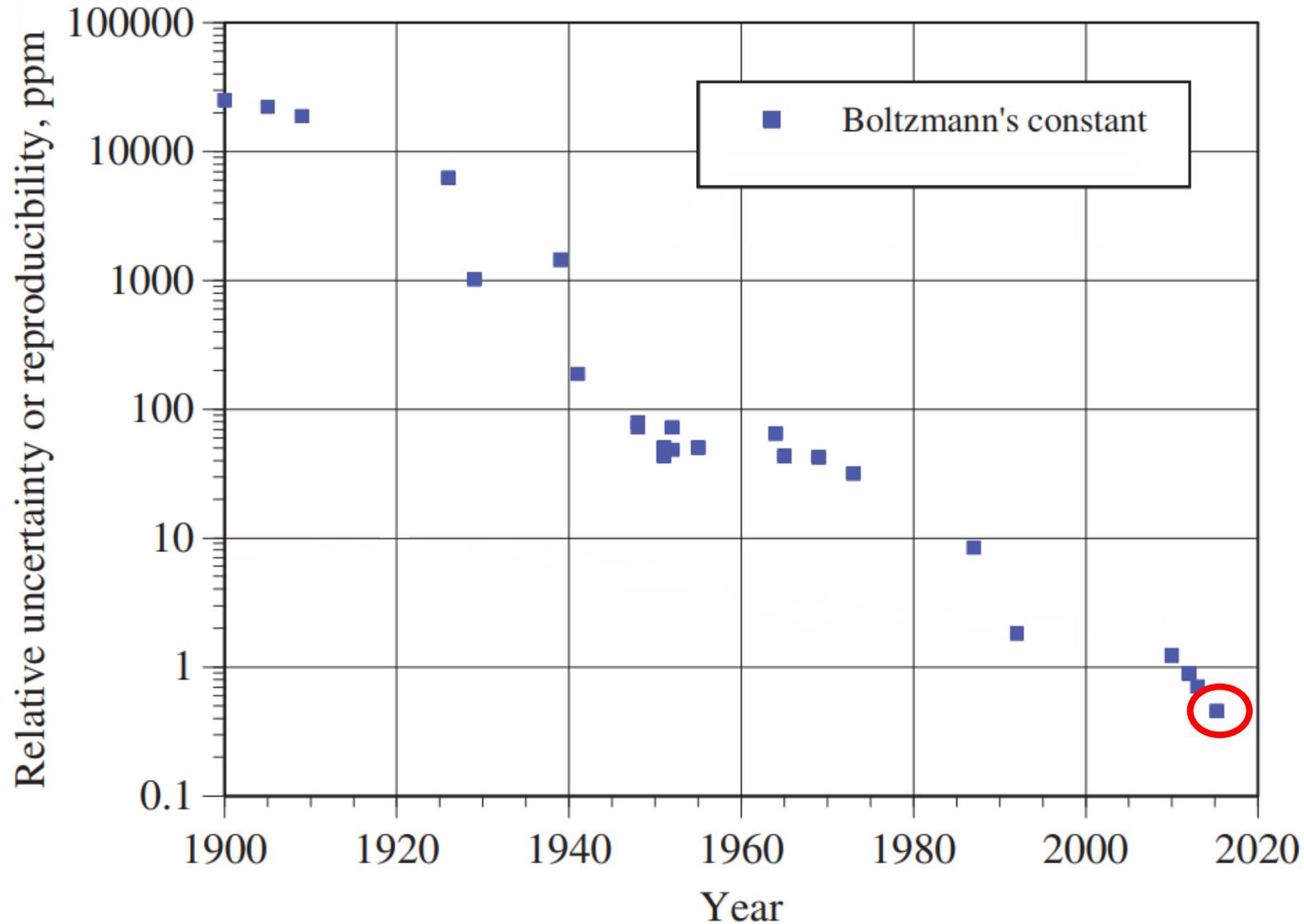


Electromagnetic measurements in very good agreement with the theoretical model
TM11 BCU3



Uncertainty of the Boltzmann constant determination

Uncertainty of the Boltzmann constant determination in function of the time



After 2017, the Boltzmann constant will no longer be measured because its value has been established by definition.

LNE-Cnam

If you want more information about the changes to the SI (International System of Units)

Search on Google: 'YouTube LNE unité'

Find 7 talks about the 7 units (in French, one hour each, with hardly any equations)

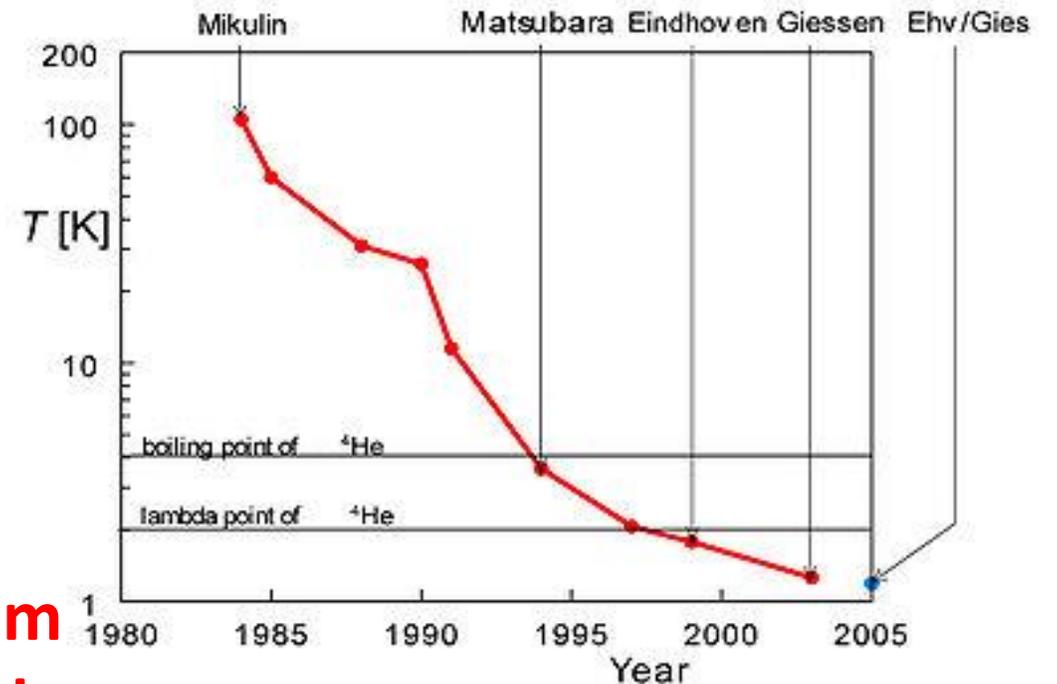
Temperature measurement at low temperatures

The cryocooler revolution

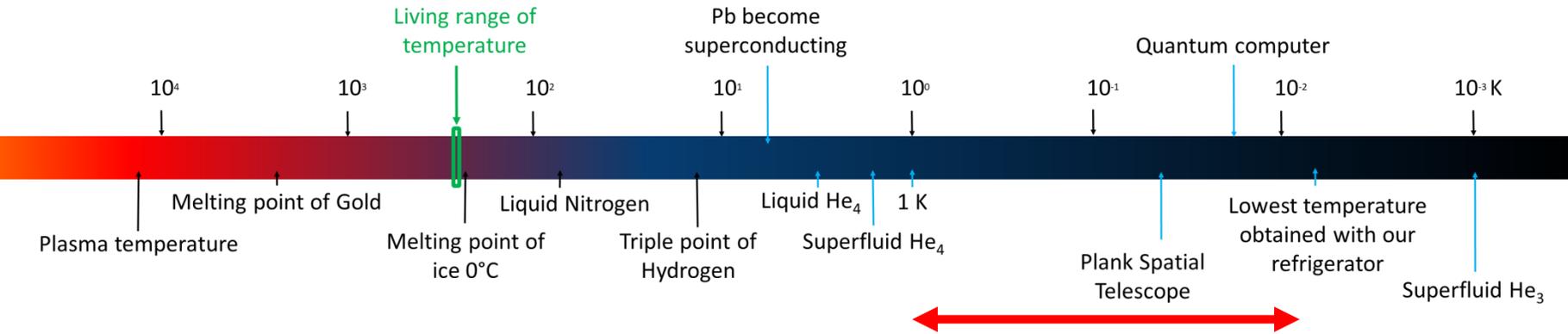


4 K

No need for liquid helium
Time is not more a problem
But also no more thermometry check at 4.2K



Thermometry below 1K at LNE-Cnam



- constructed a reference scale, PLTS-2000.
- calibrated this against a stable instrument, the SRD1000
- compared our PLTS2000 to a primary thermometer, the MFFT
- compared our scale to three resistively calibrated thermometers from three different companies.

PhD student Clément Tauzin



Provisional Low Temperature Scale 2000 or PLTS2000 or T₂₀₀₀

Base on the thermophysical melting curve of Helium-3

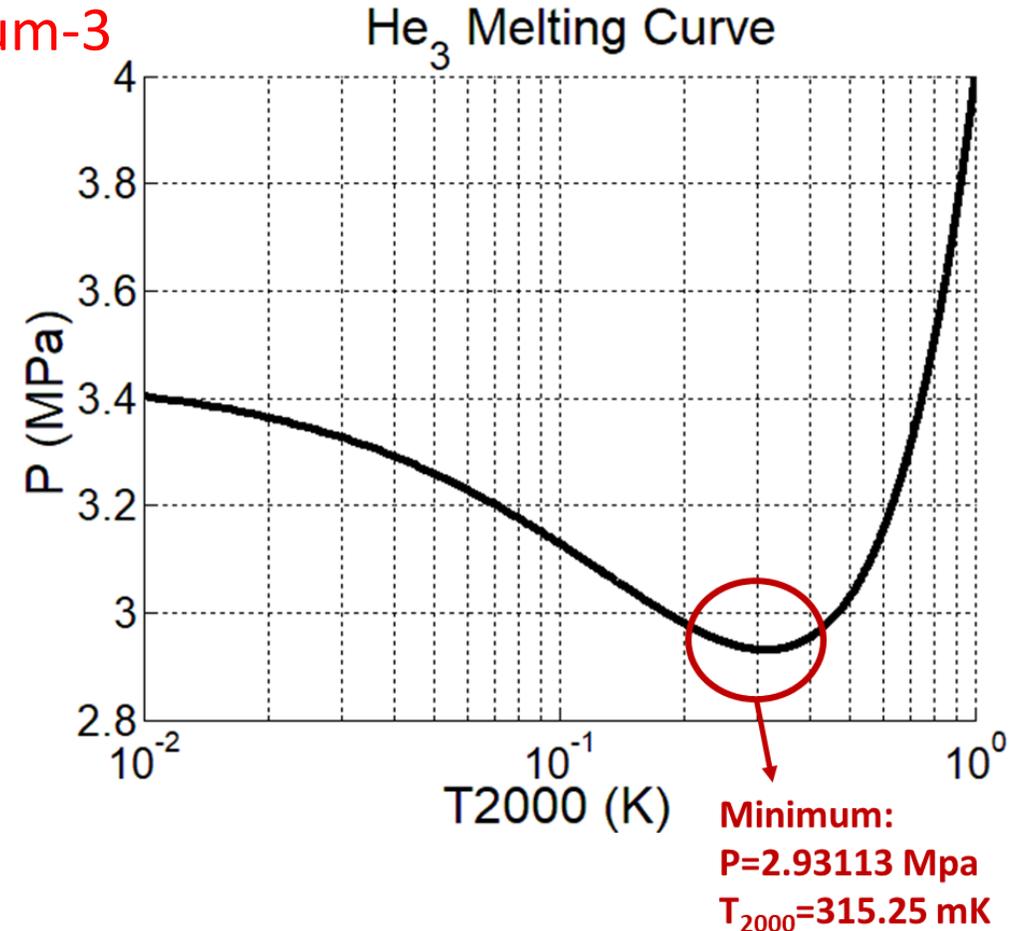
$$p_m/\text{MPa} = \sum_{i=-3}^{+9} a_i (T_{2000}/\text{K})^i$$

with the following coefficients:

$$\begin{aligned} a_{-3} &= -1.3855442 \cdot 10^{-12}, & a_{-2} &= 4.5557026 \cdot 10^{-9}, \\ a_{-1} &= -6.4430869 \cdot 10^{-6}, & a_0 &= 3.4467434 \cdot 10^0, \\ a_1 &= -4.4176438 \cdot 10^0, & a_2 &= 1.5417437 \cdot 10^1, \\ a_3 &= -3.5789853 \cdot 10^1, & a_4 &= 7.1499125 \cdot 10^1, \\ a_5 &= -1.0414379 \cdot 10^2, & a_6 &= 1.0518538 \cdot 10^2, \\ a_7 &= -6.9443767 \cdot 10^1, & a_8 &= 2.6833087 \cdot 10^1, \\ a_9 &= -4.5875709 \cdot 10^0. \end{aligned}$$

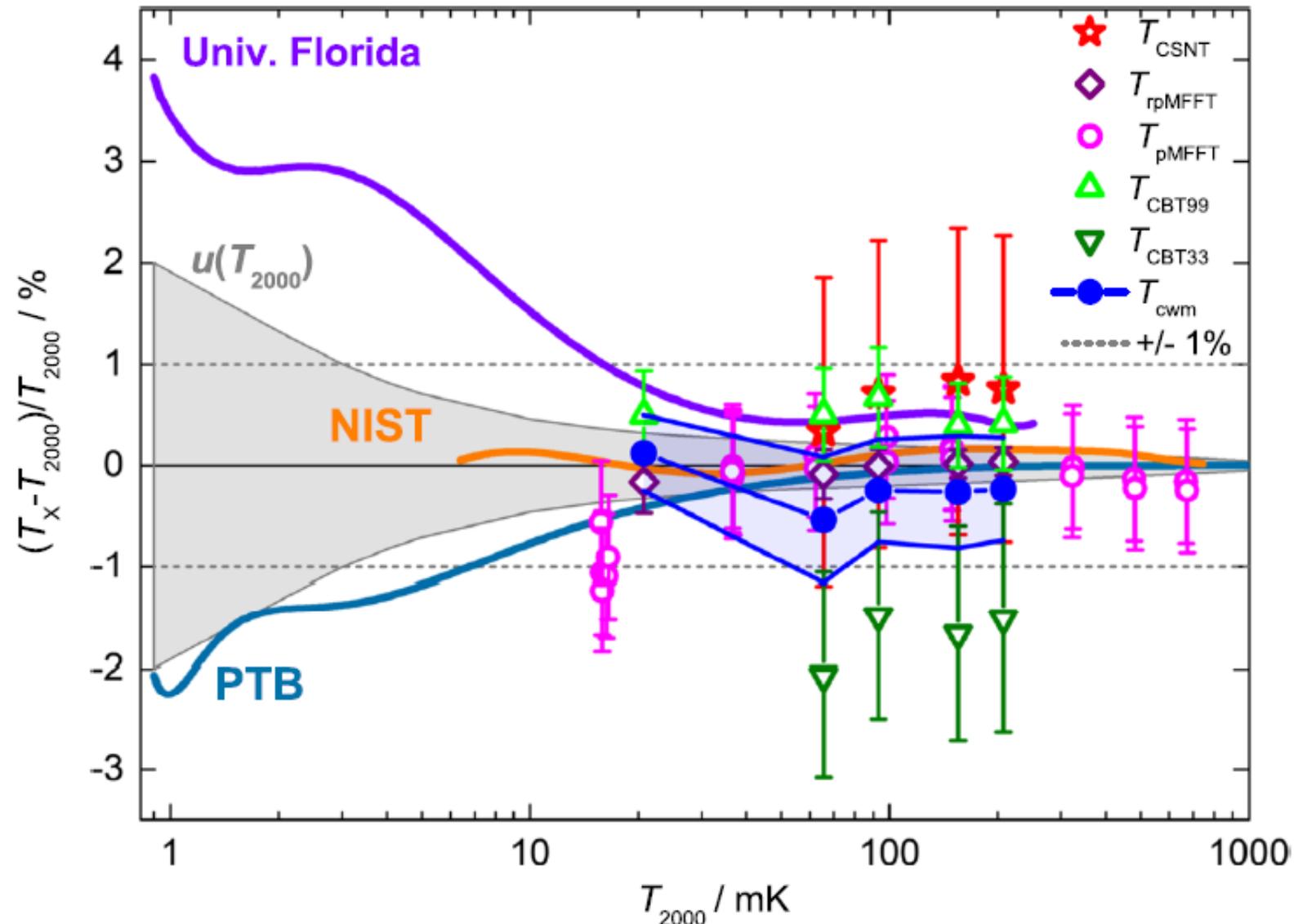
We measure P we deduce T₂₀₀₀

The equation is **valid from 0.9 mK to 1 K**, covering more than three decades.



Why Provisional in PLTS2000 ?

Below 7 mK, T_{2000} is the average of the scales from PTB and the University of Florida. It is why the world provisional in PLTS2000



Capacitance strain gauge

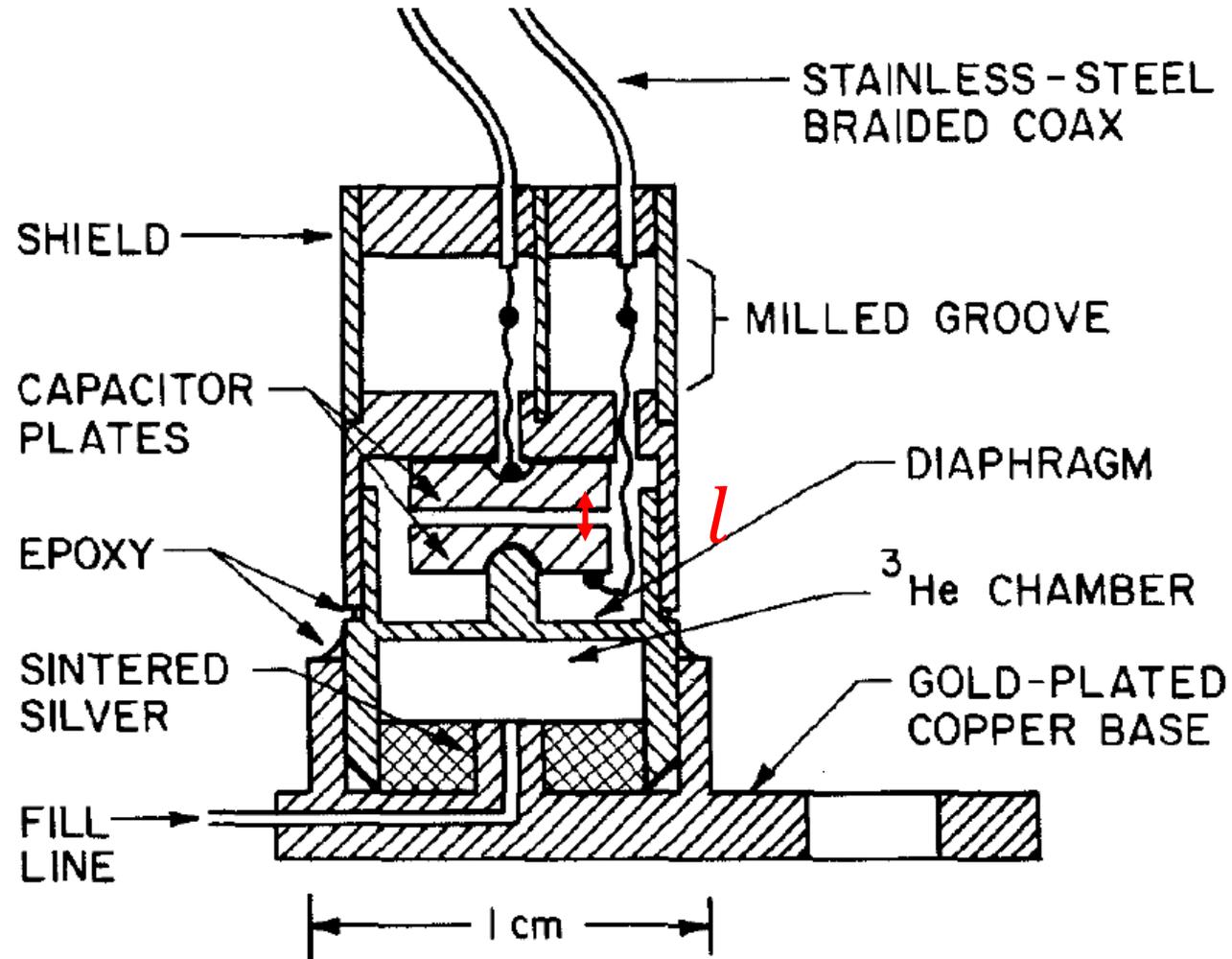


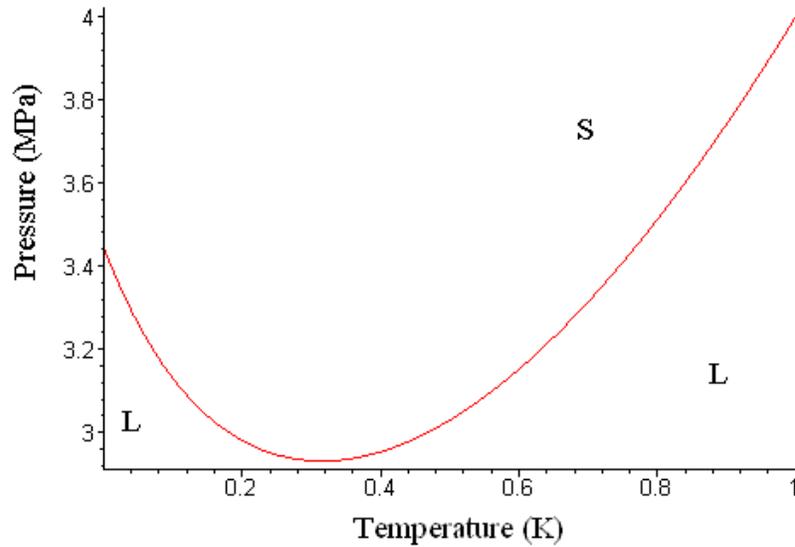
Fig. 1. Melting curve thermometer.

$$C = \frac{\epsilon A}{l}$$
$$\frac{dC}{C} = - \frac{dl}{l}$$



8 Digits instrument

Understand the Melting Curve of Helium-3?



Clausius–Clapeyron equation

$$\left(\frac{dP}{dT}\right)_m = \frac{S_L - S_S}{V_L - V_S}$$

$$V_L = V_S + 1,314 \text{ cm}^3/\text{mol}$$

$$V_L > V_S$$

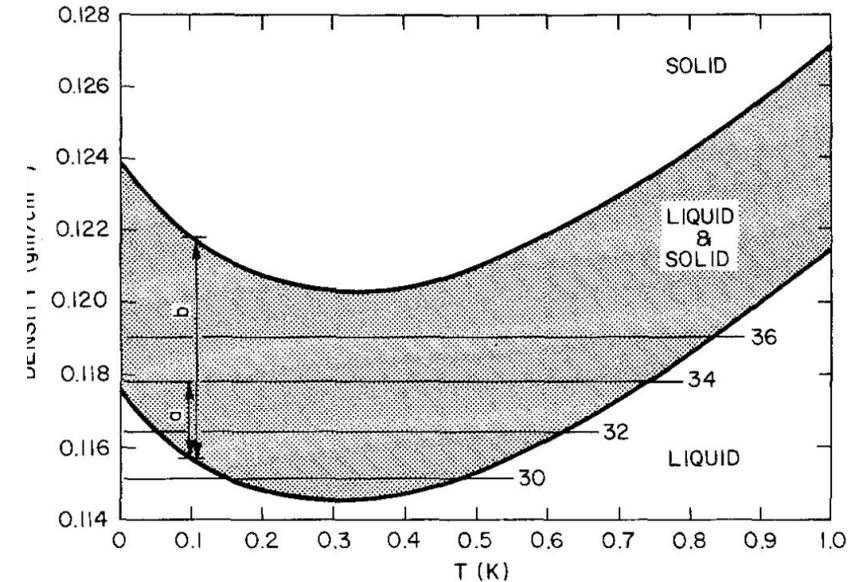
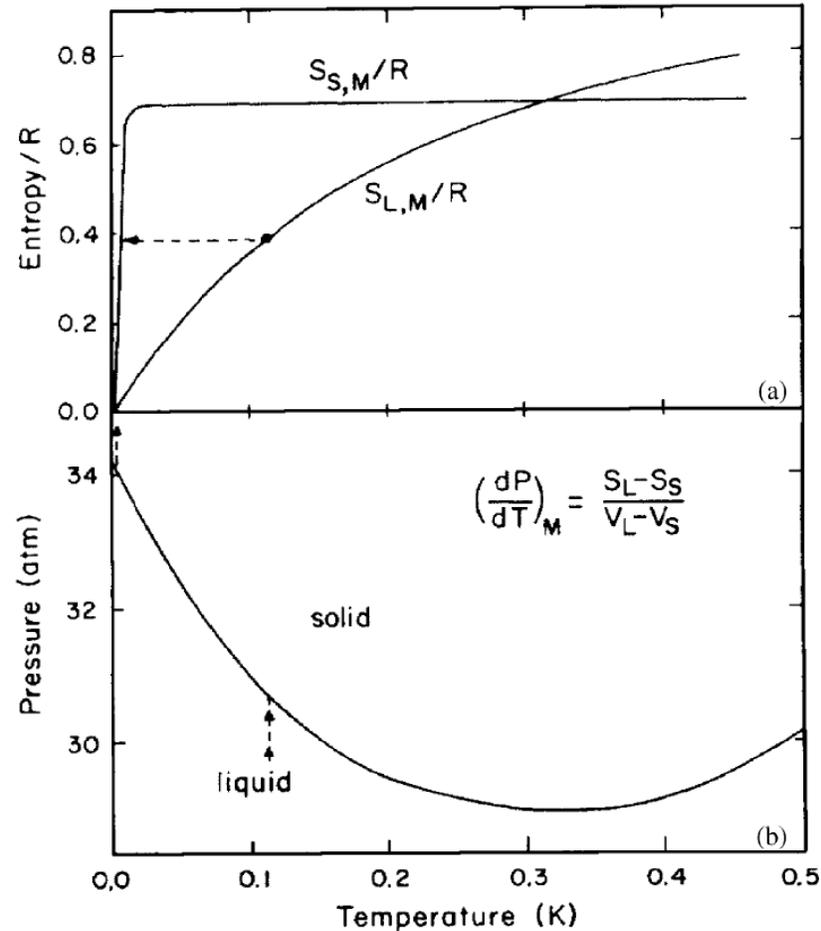
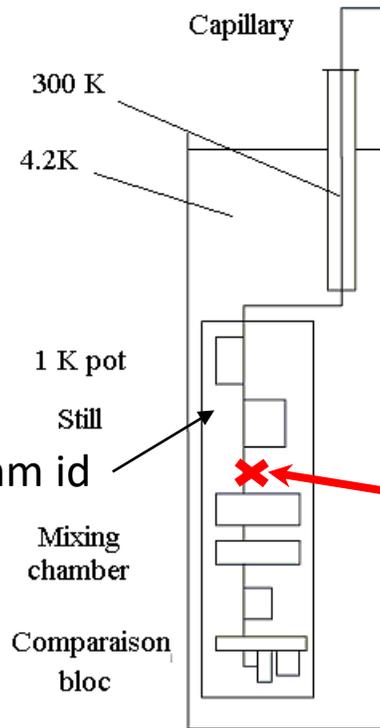


Fig. 2. Density of liquid and solid ^3He along the melting curve plotted using data from Refs. 3 and 4. Other details are given in the text.

How to isolate the Melting Curve Thermometer?

Normal use of a DR

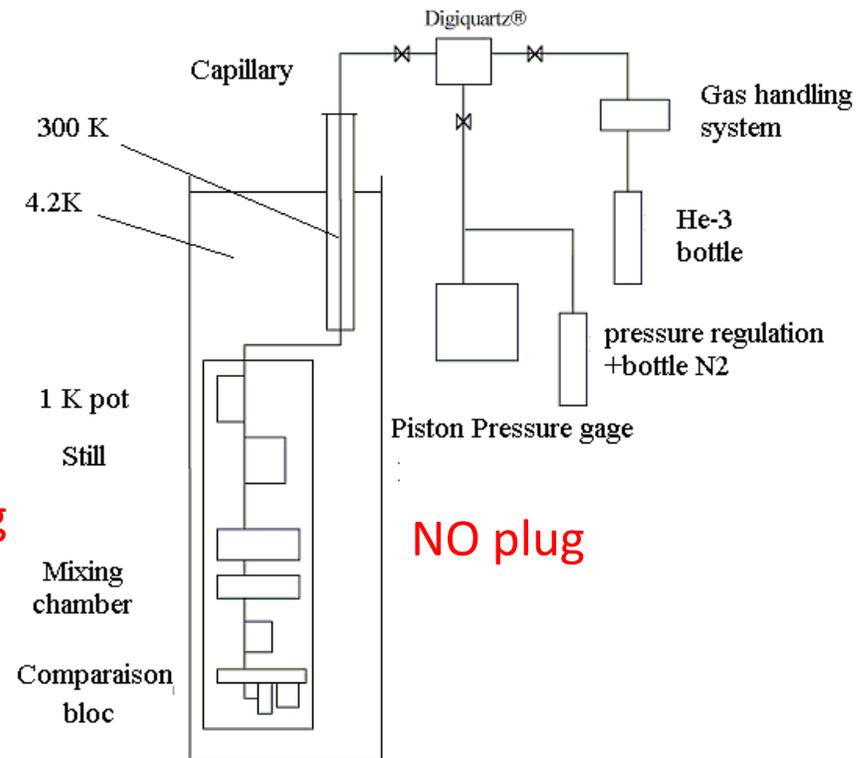


Small capillary
Less than 0,5mm id

He3 solid plug

MCT isolate and can be use as a thermometer

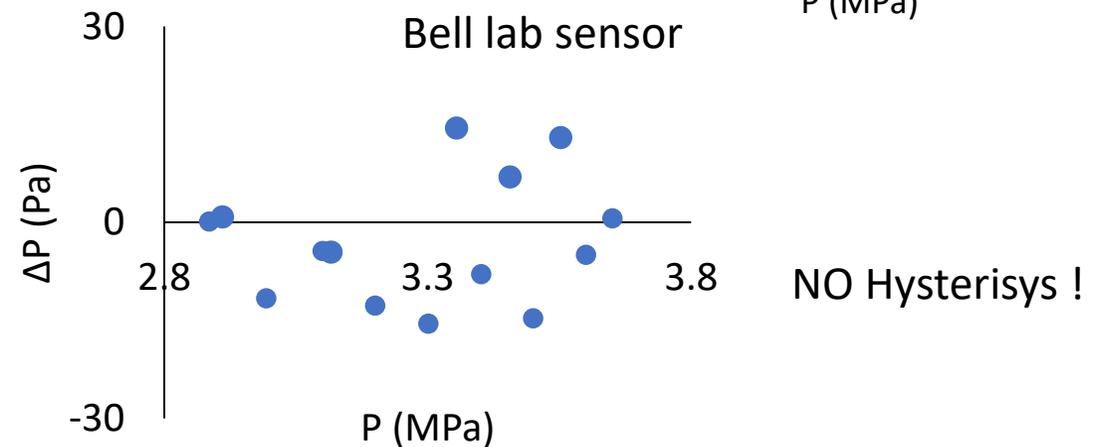
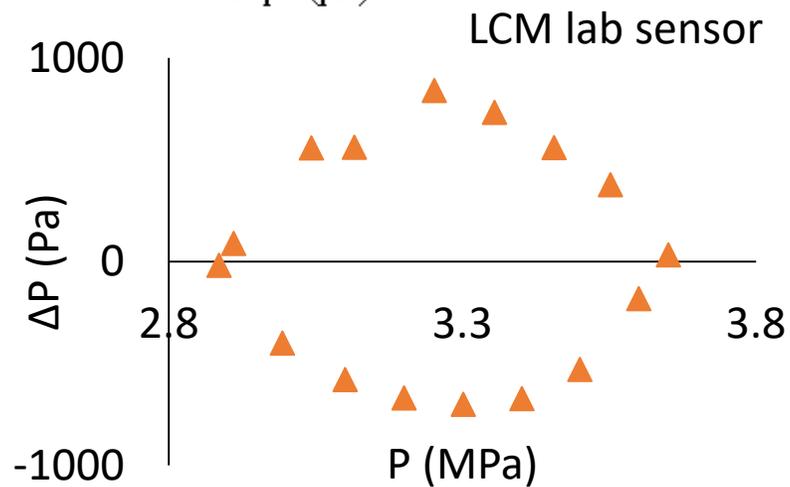
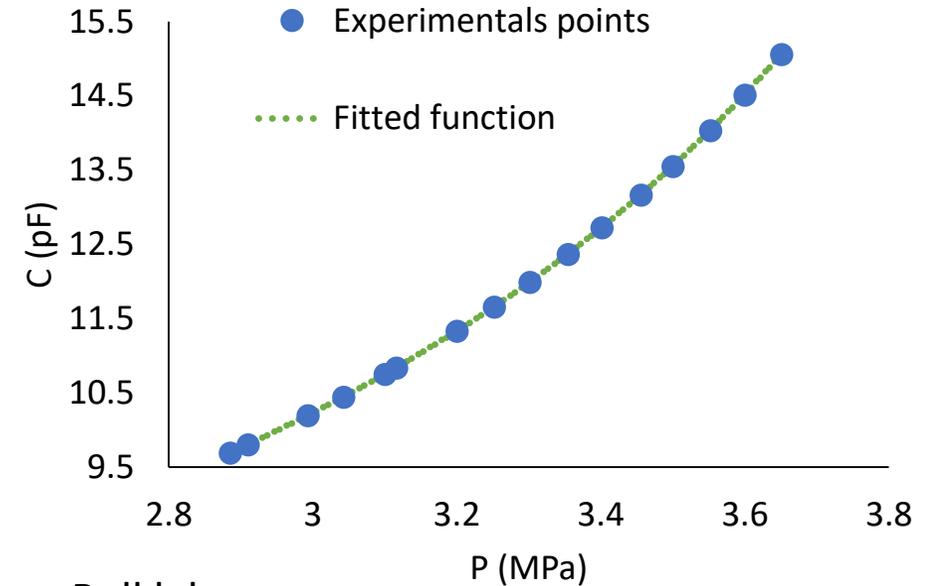
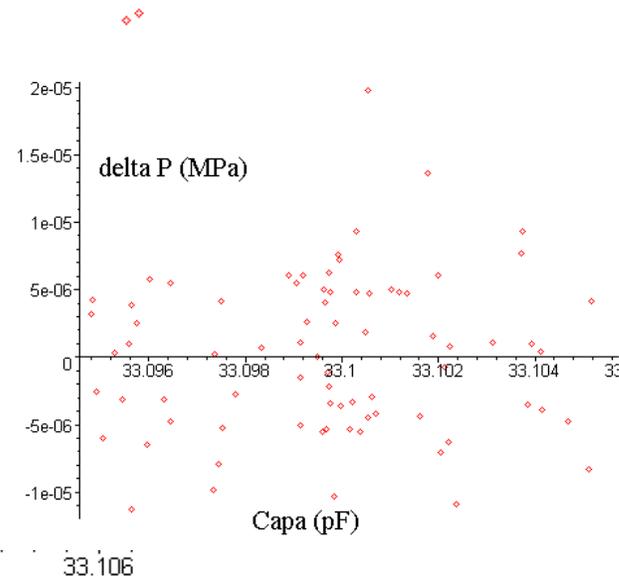
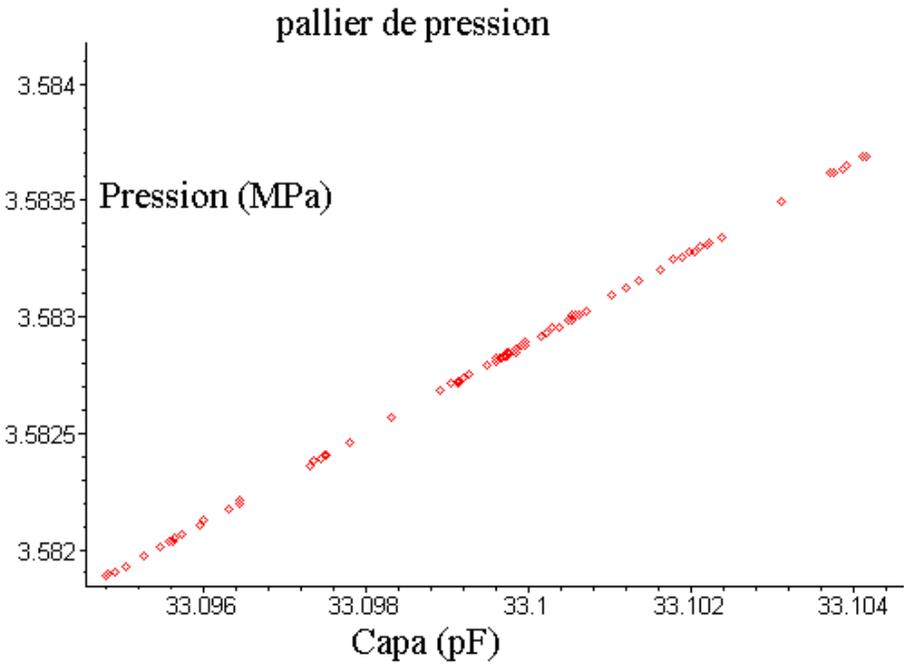
Every stage at 1.1K



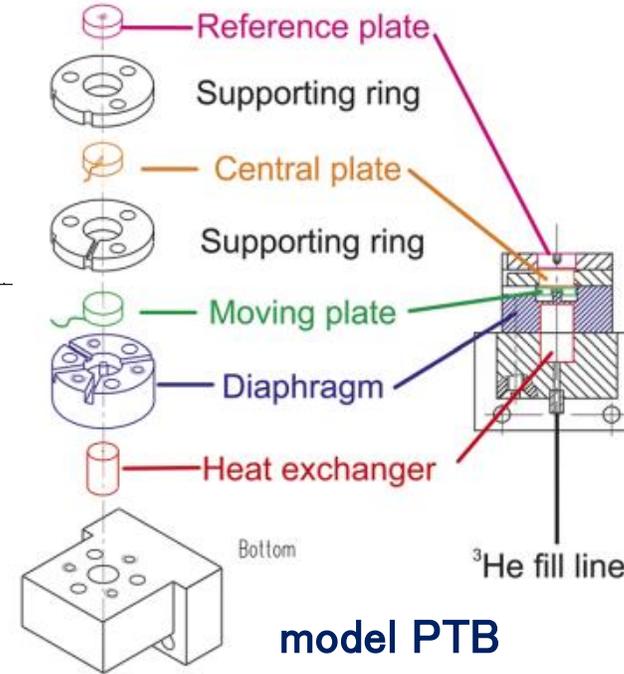
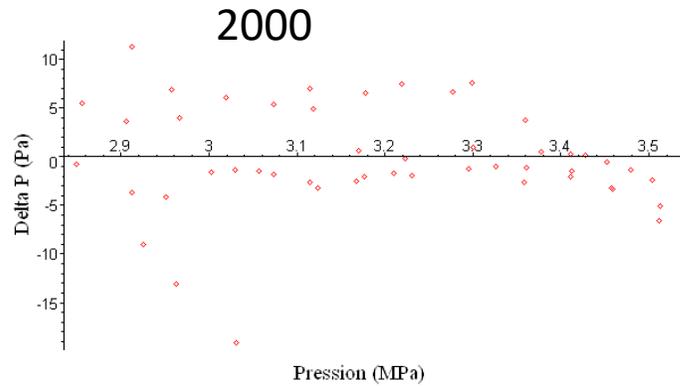
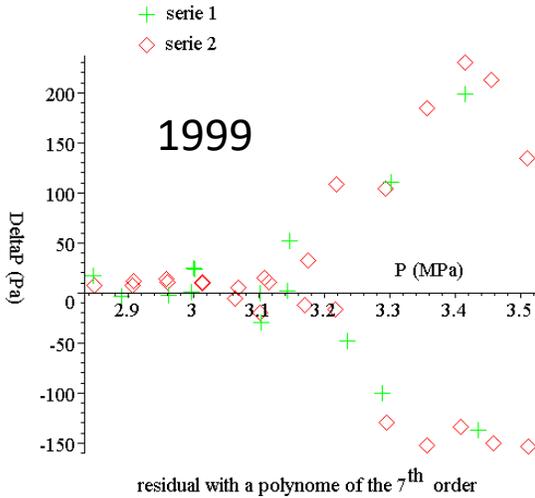
NO plug

MCT can be calibrate with a secondary pressure sensor as Digitquartz

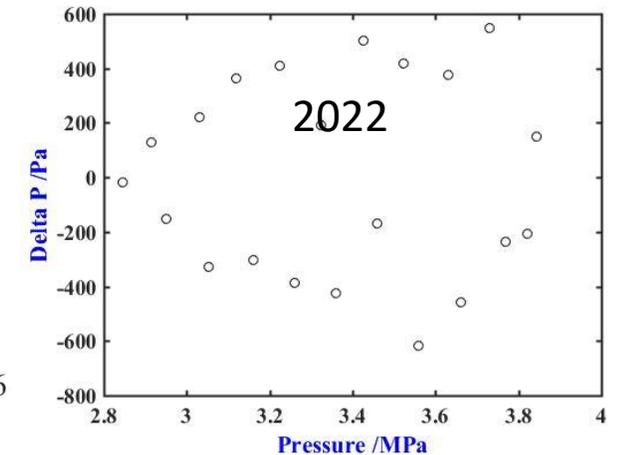
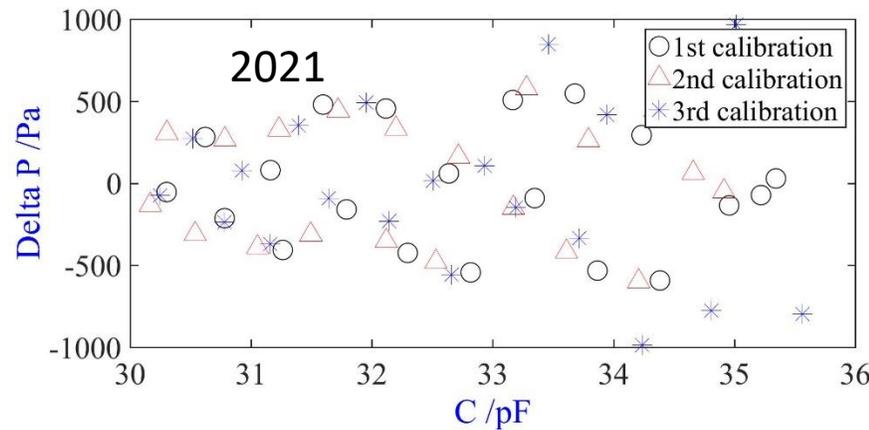
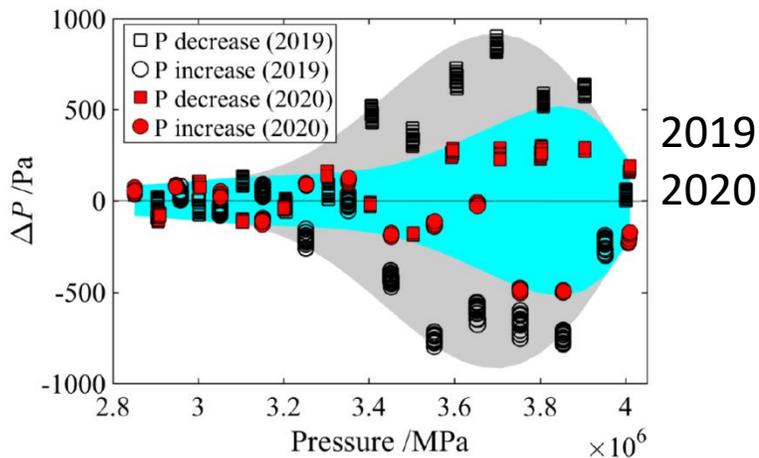
Calibration of the capacitance strain gauge



Hysterisys of the same capacitance strain gauge over time

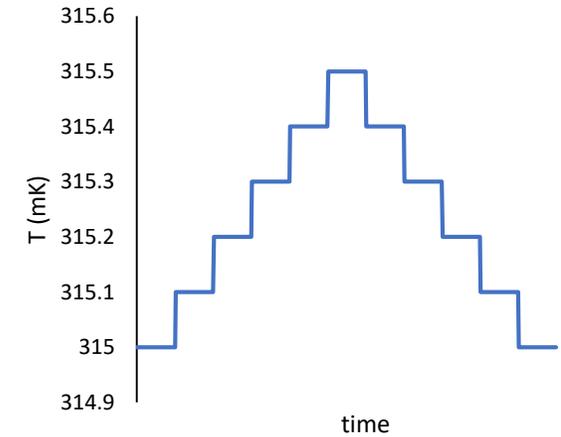
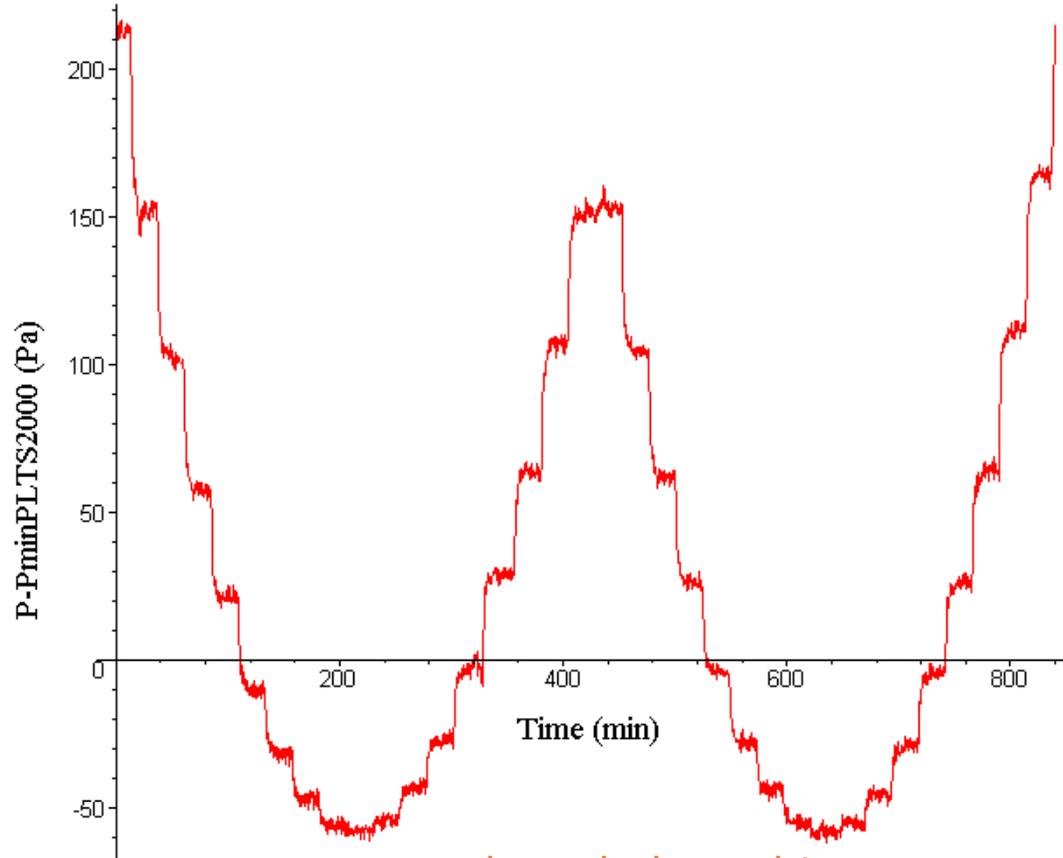


The bolt and the indium joint play a critical role in the mechanical behavior of the capacitance strain gauge.



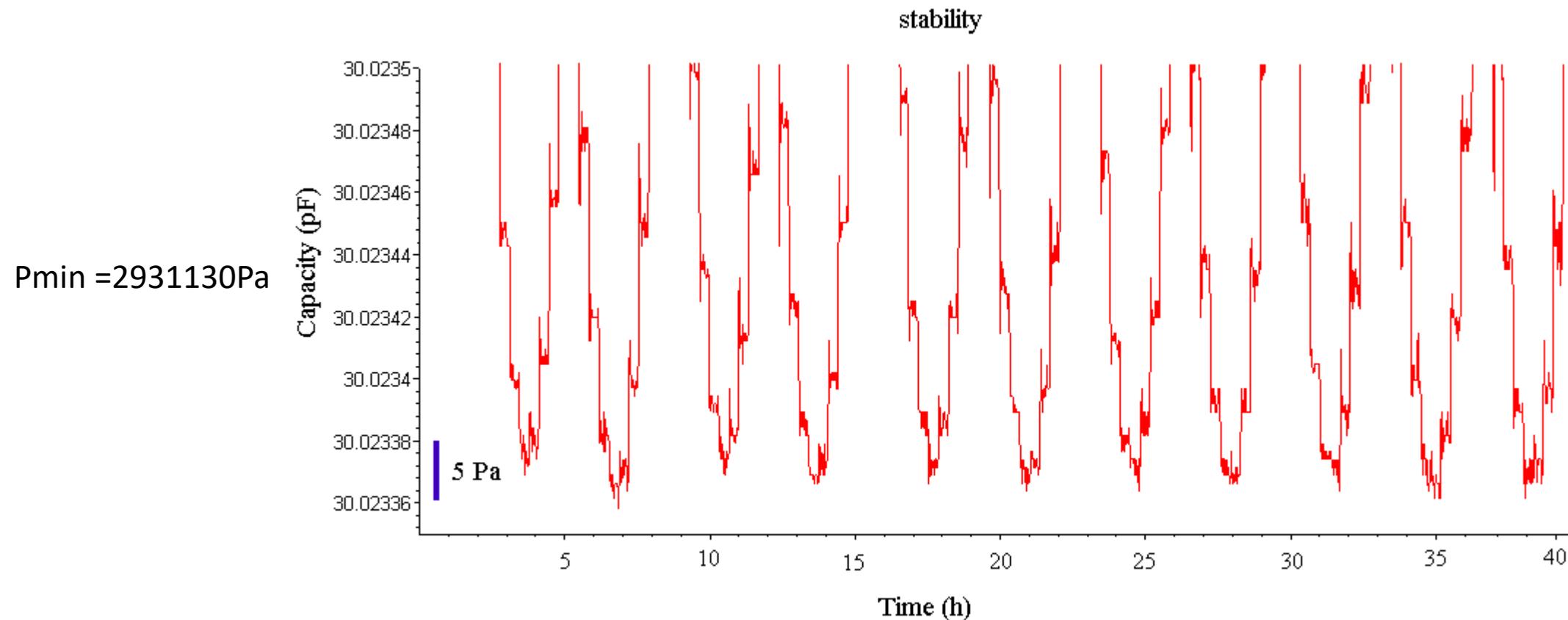
Very precise realization of the melting pressure minimum

$P_{\min} = 2931130 \text{ Pa}$



Passage through the melting pressure minimum in step of
1 mK with rising and falling temperature

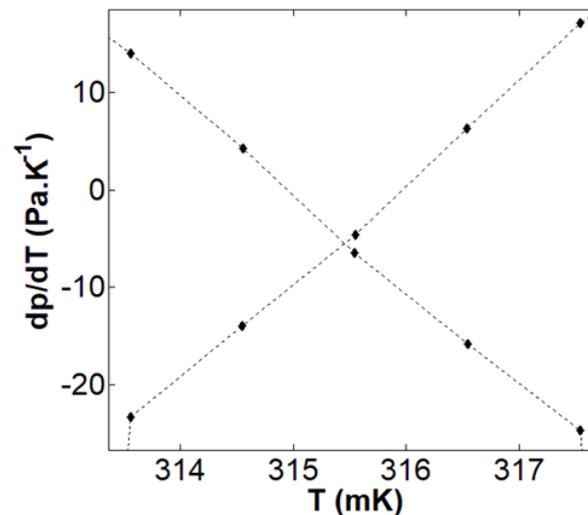
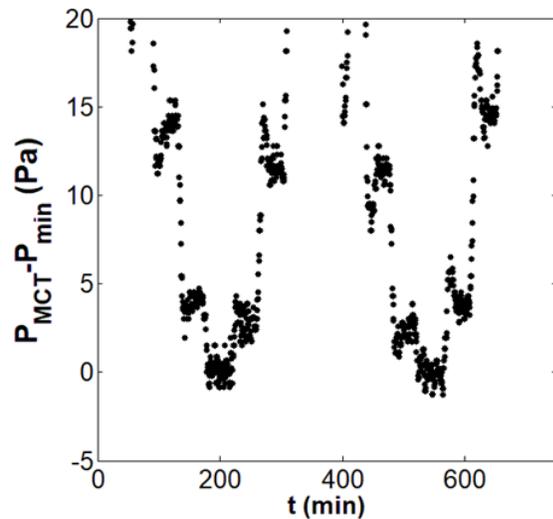
Stability of the melting pressure minimum



Because PLTS2000 give the pressure of the minimum
We can use the minimum to estimate the hydrostatic correction

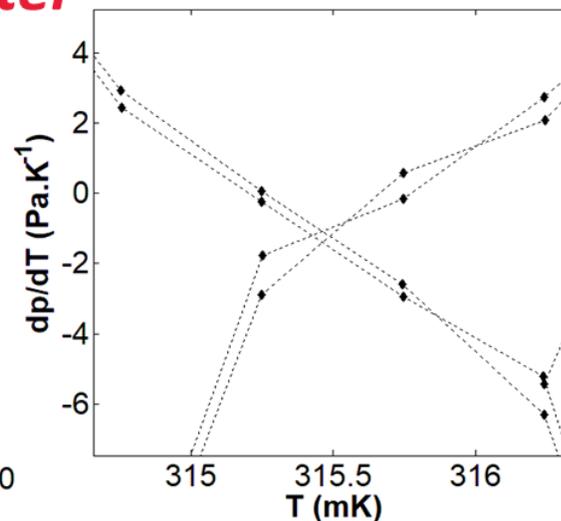
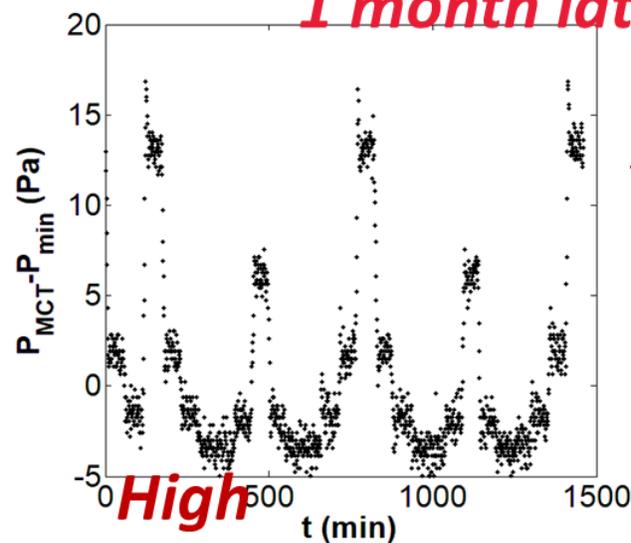
Temperature of the melting pressure minimum

$P_{\min} = 2.931\ 130\ \text{MPa}$



$T_{\min} = 315.24$

1 month later

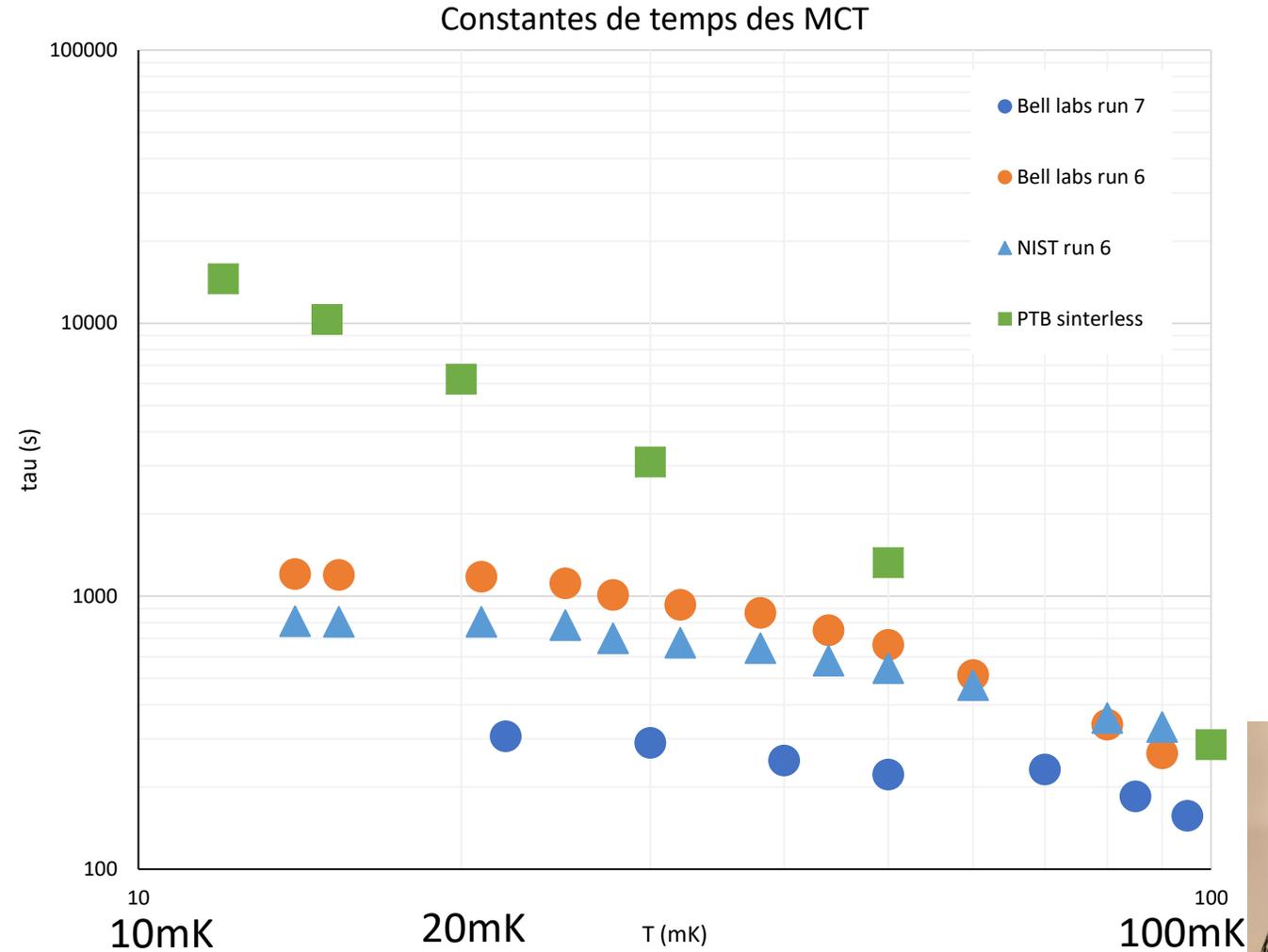
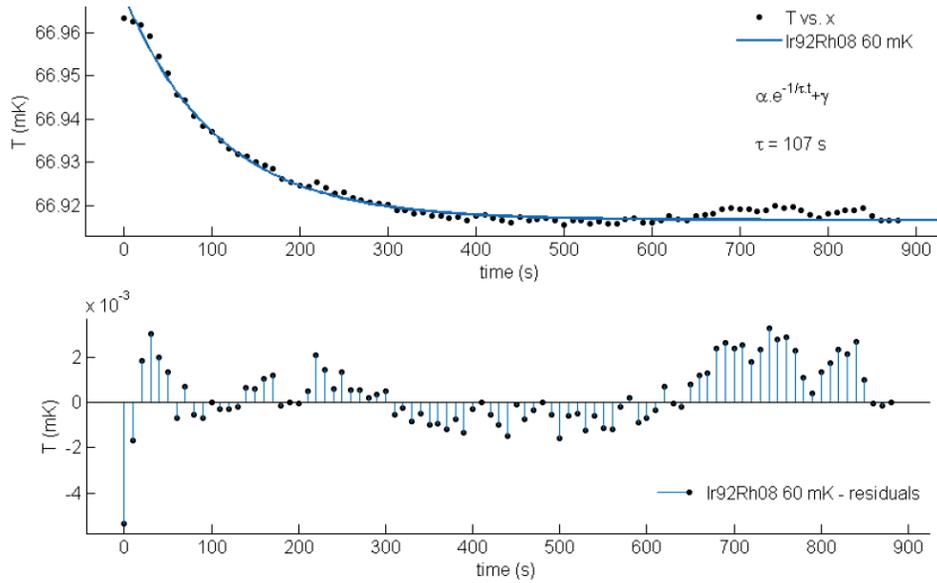


High stability



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Time constant of the melting curve thermometer

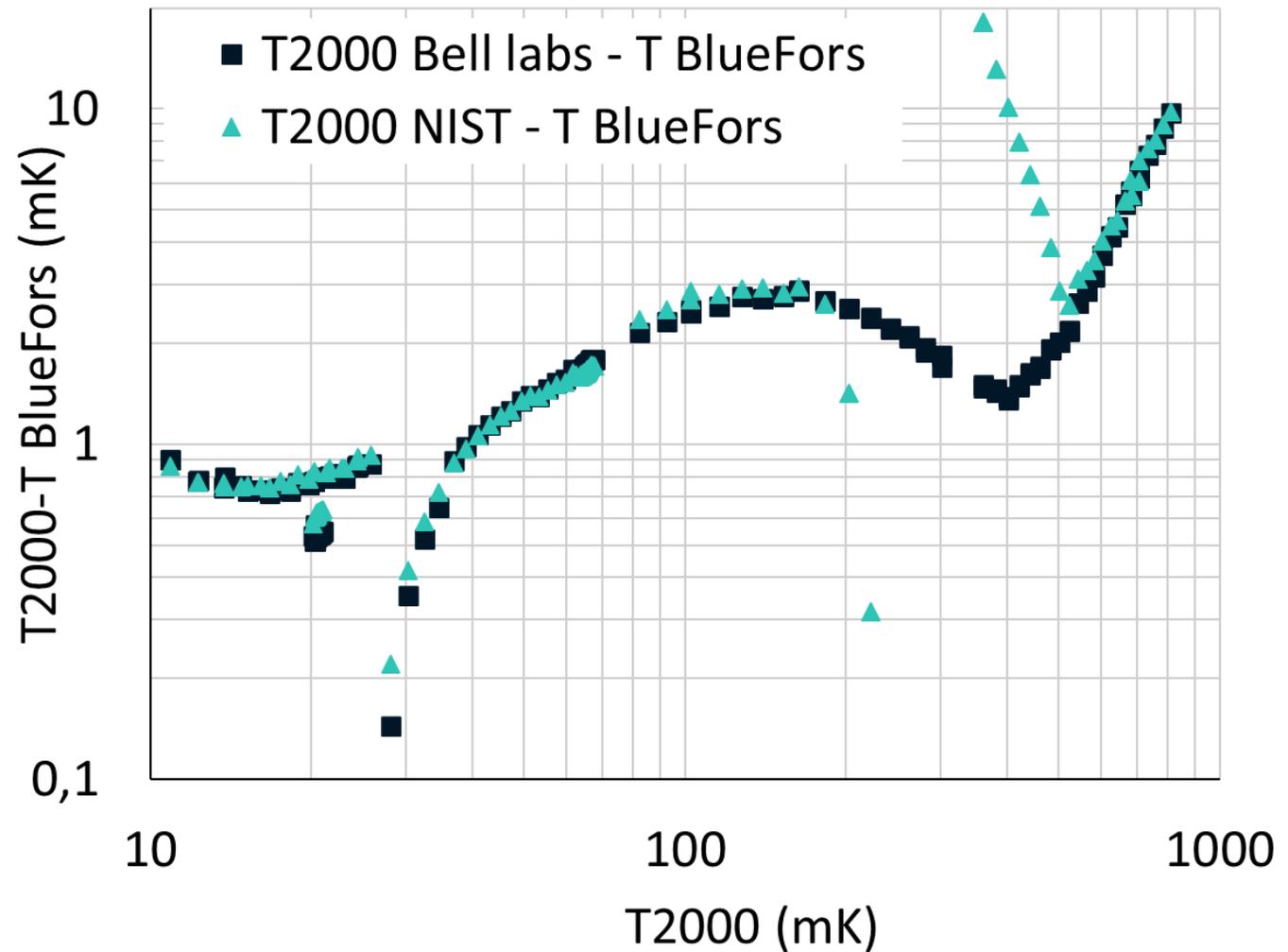


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Comparison of Two Melting Curve Thermometers



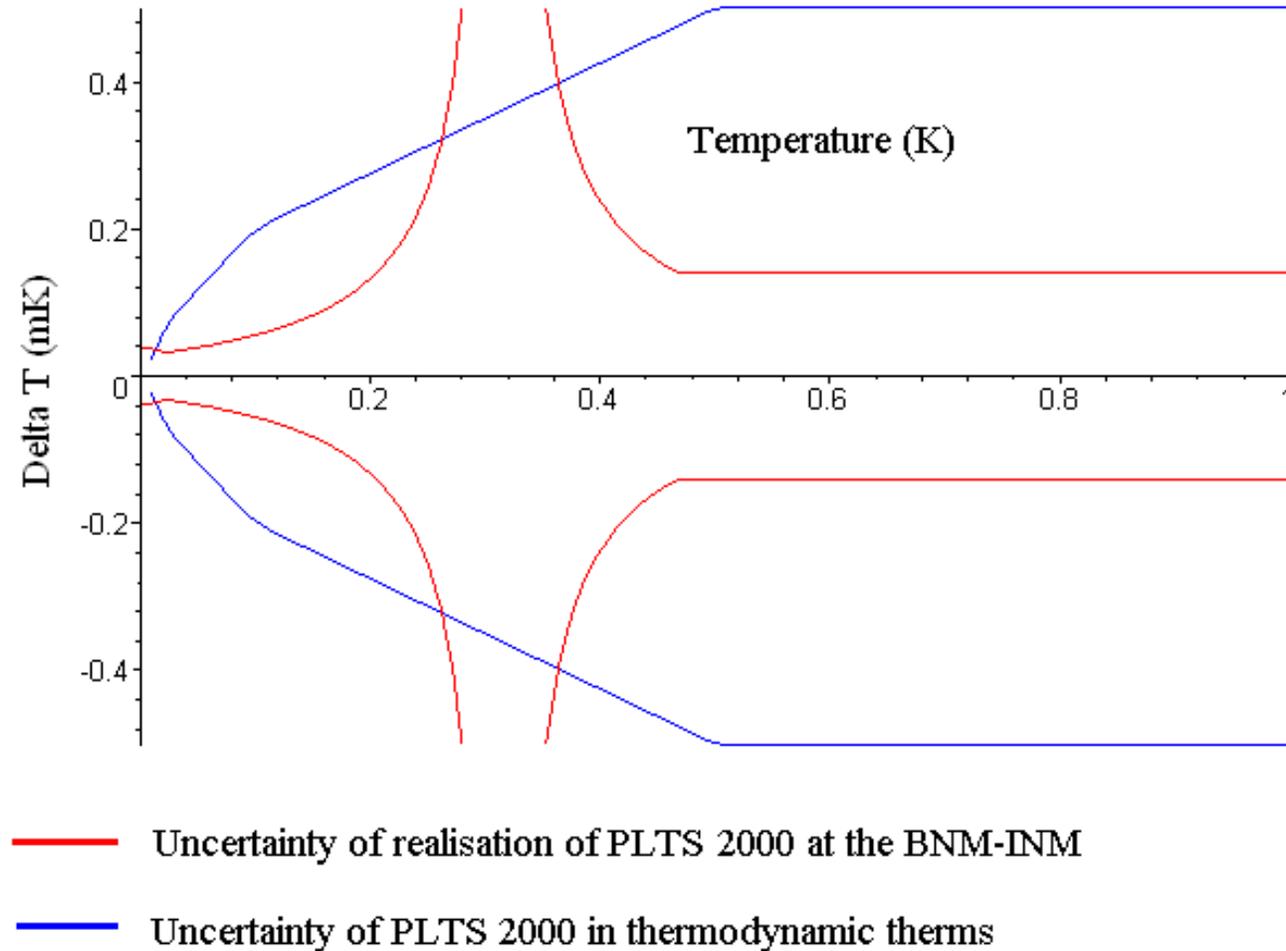
Direct comparison of 2 MCTs and BlueFors thermometry



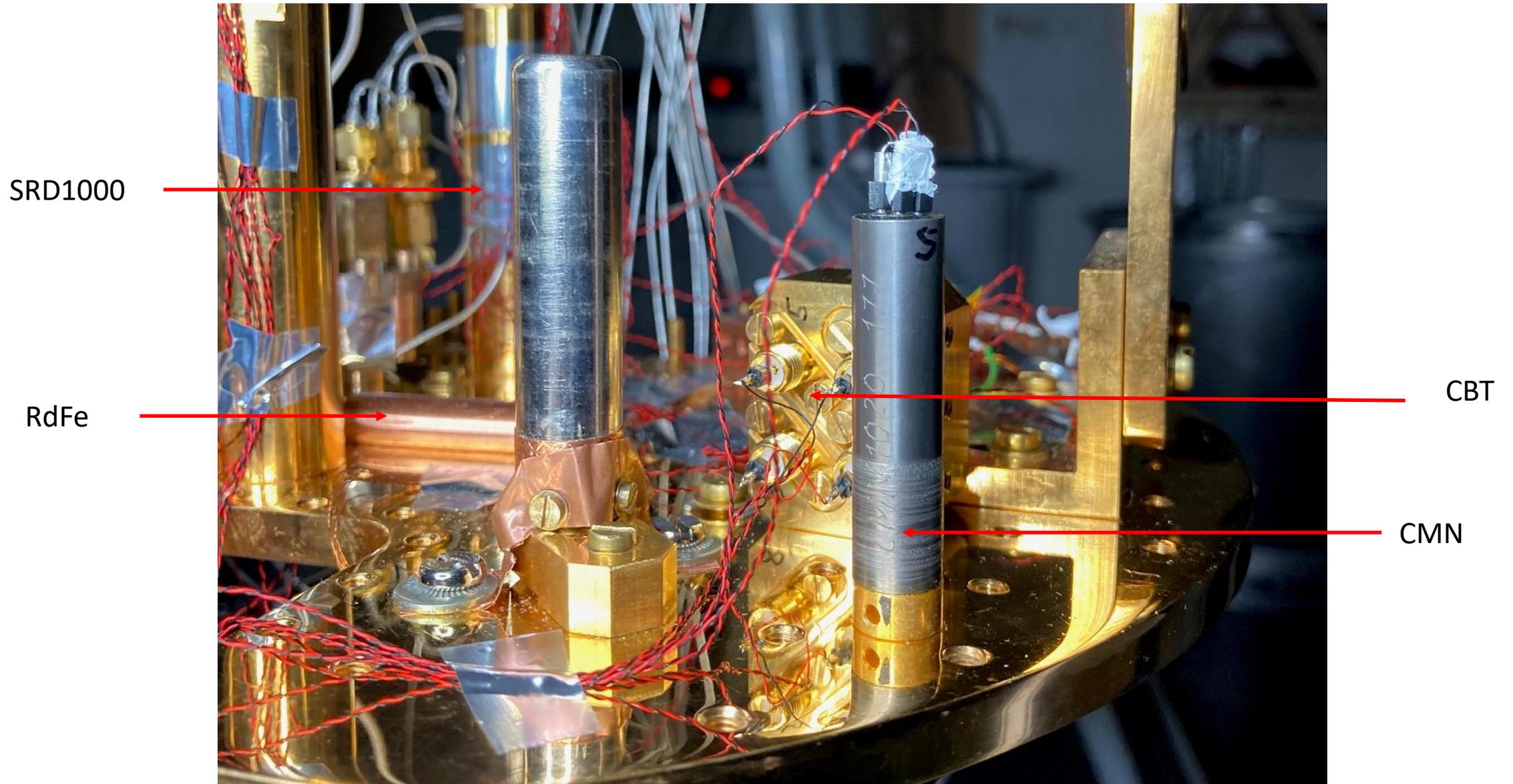
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Uncertainty of PLTS2000 at LNE

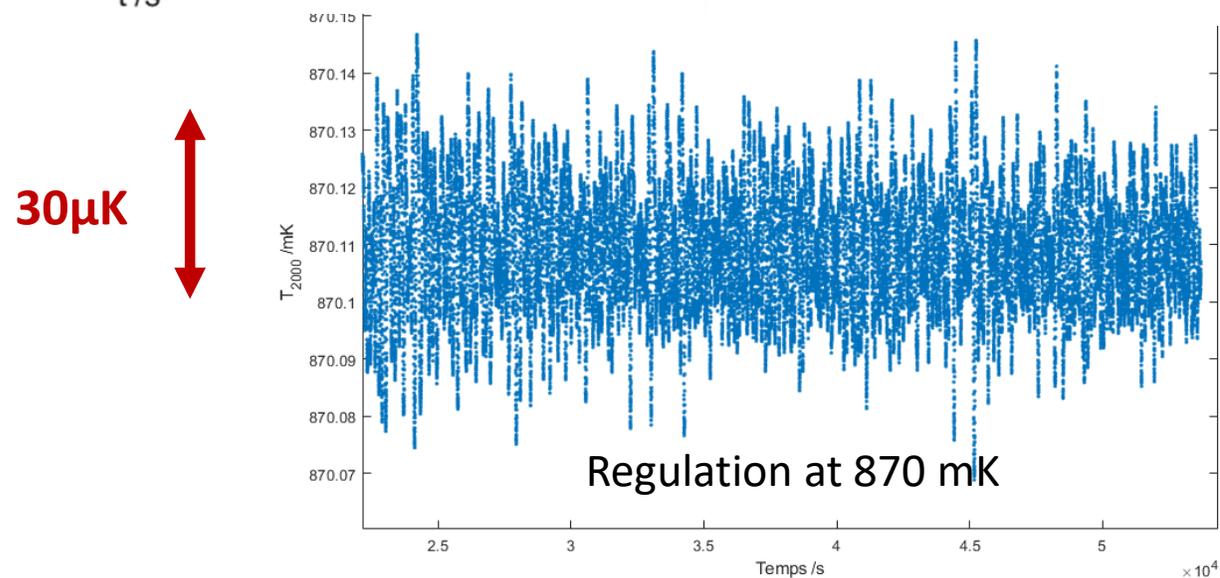
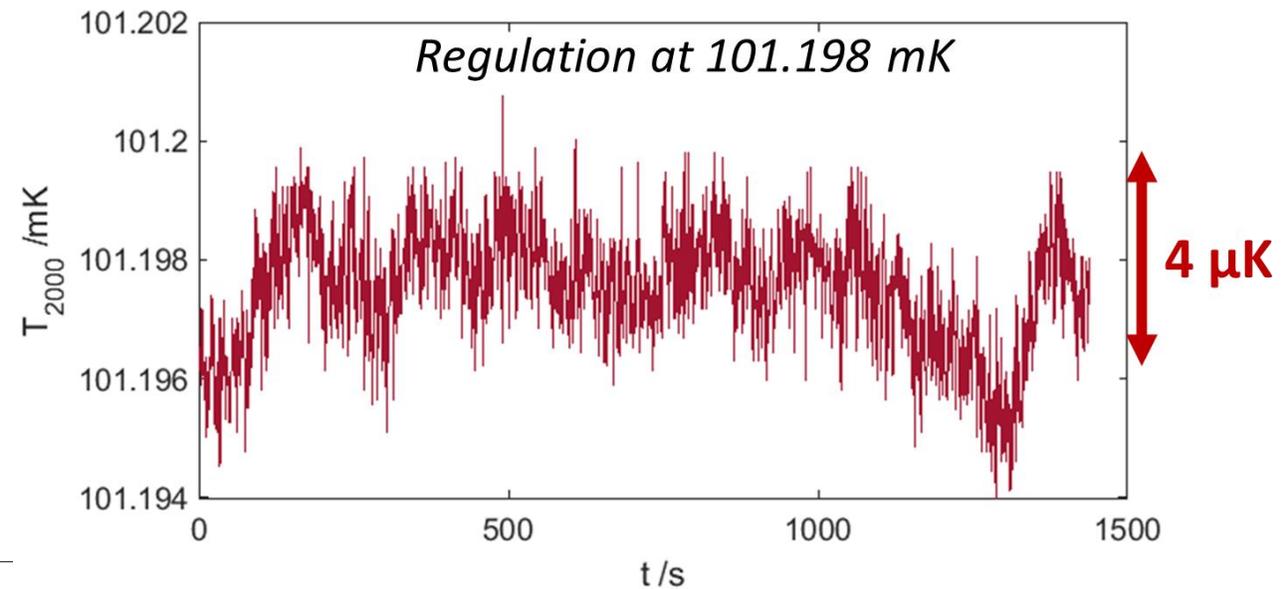
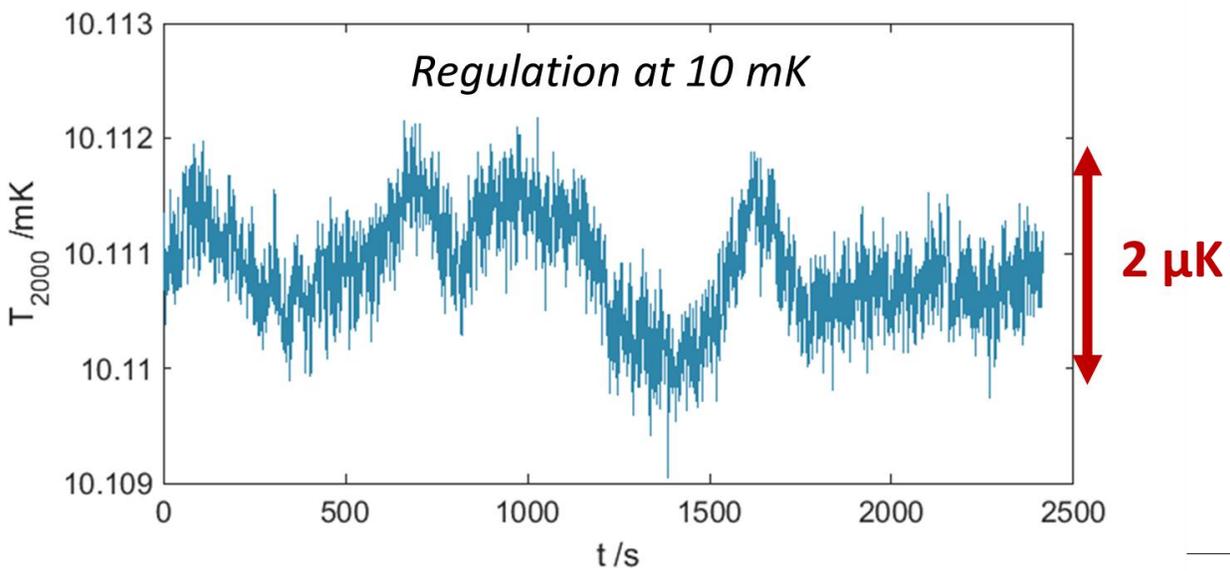
The complete uncertainty budget 2024 is not finished, but it is expected to be better than, the one from 2002.



Low temperature thermometry compared to PLTS2000



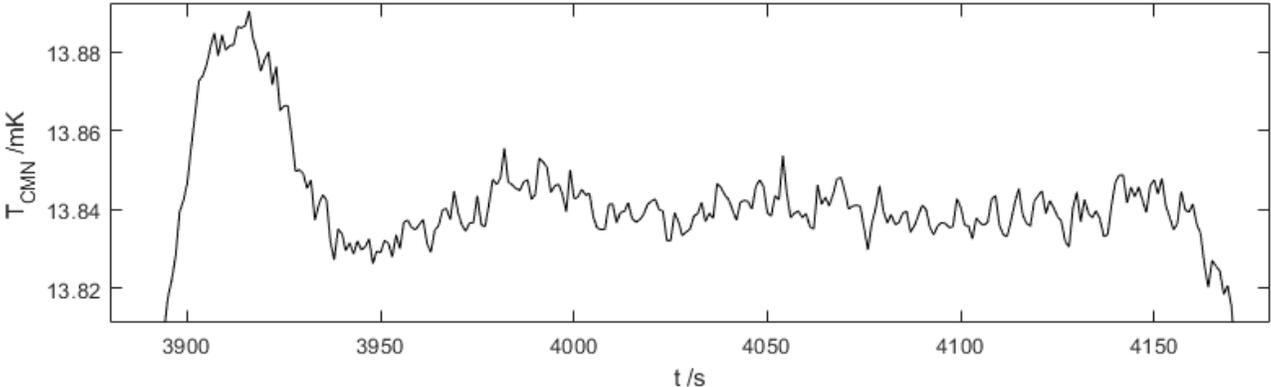
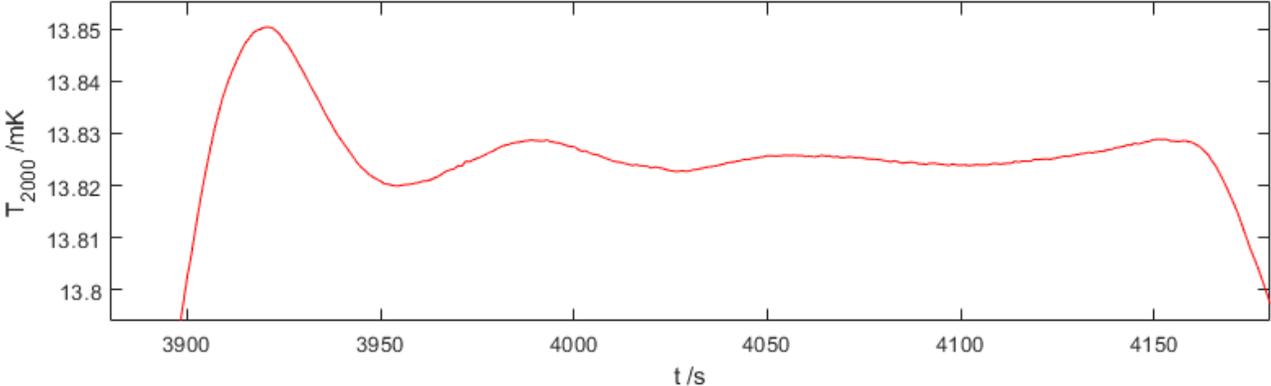
Low temperature thermometry regulation



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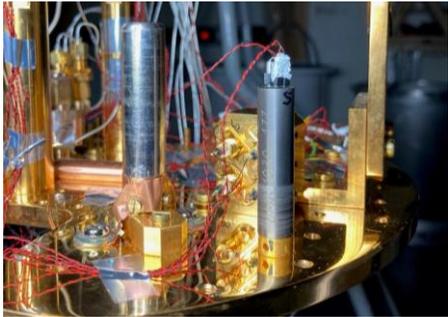
Low temperature thermometry regulation

@13,8mK

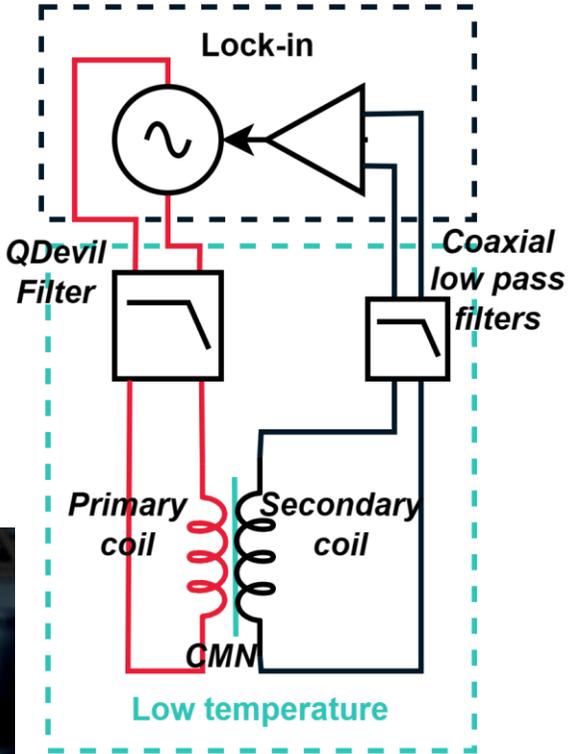


Curie weiss law

$$\chi = b + \frac{a}{T + \Delta}$$



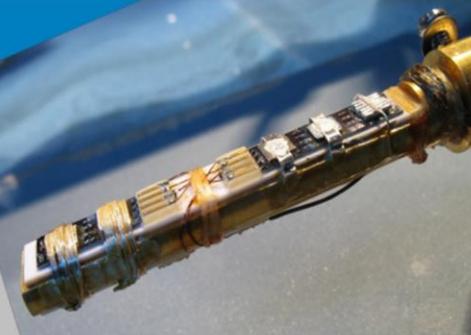
CMN



SRD1000 from HDL

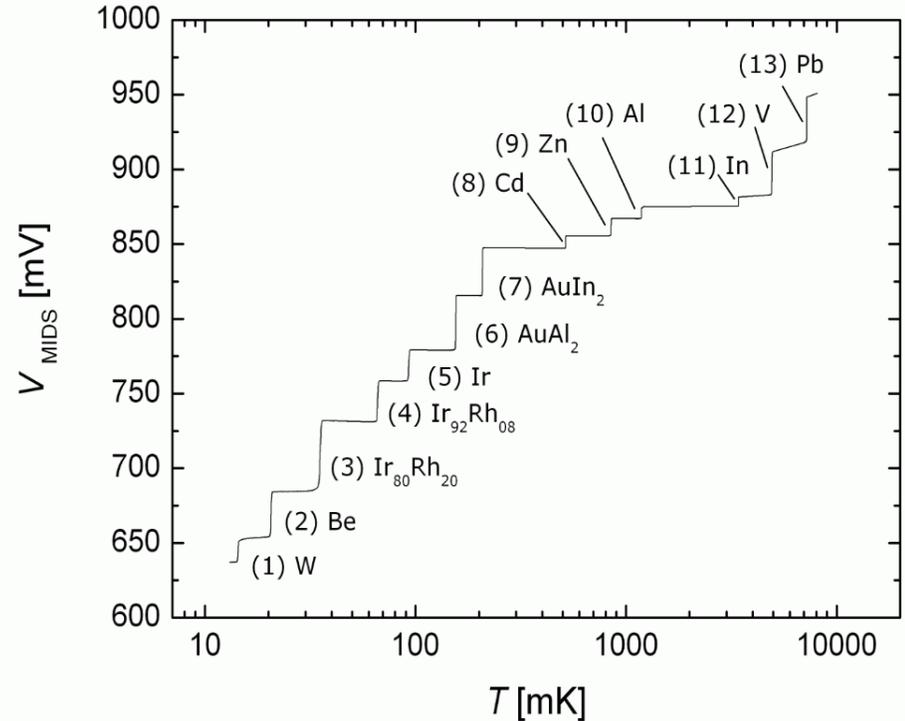
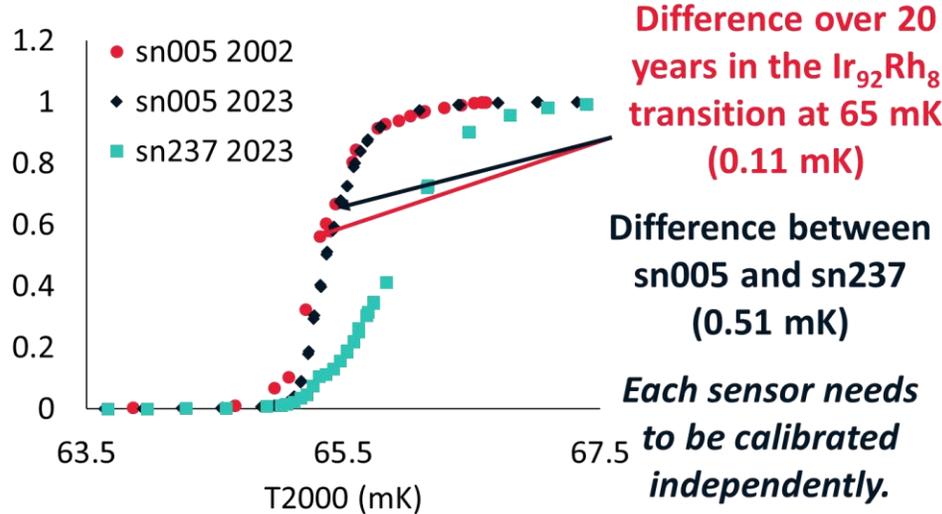
SRD1000 Superconducting transition

We obtain the transition temperatures of 13 superconducting elements by measuring their magnetic susceptibility.



Inside of an SRD1000

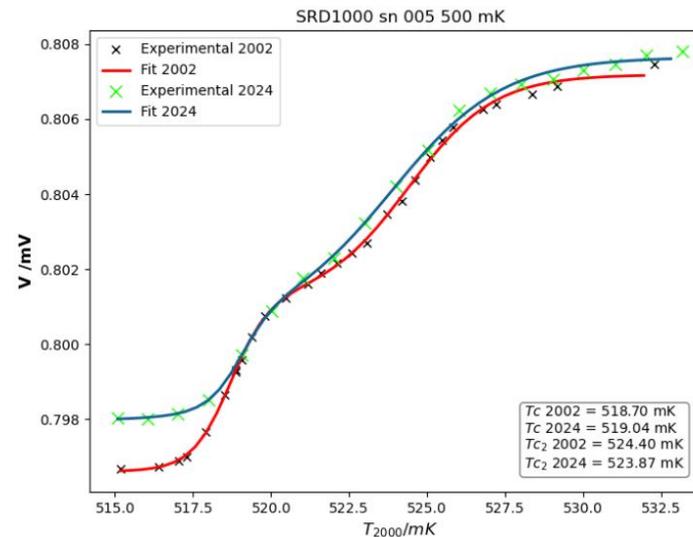
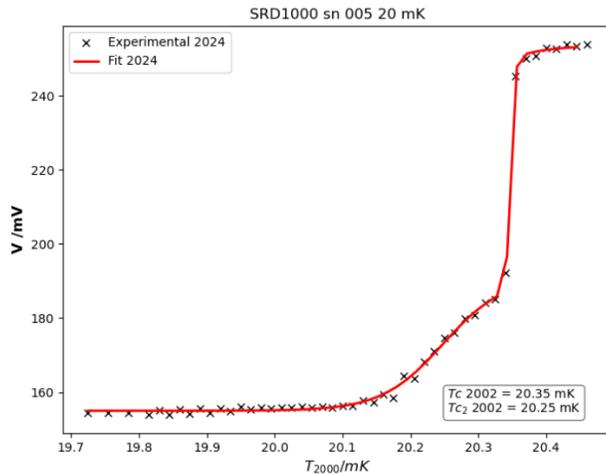
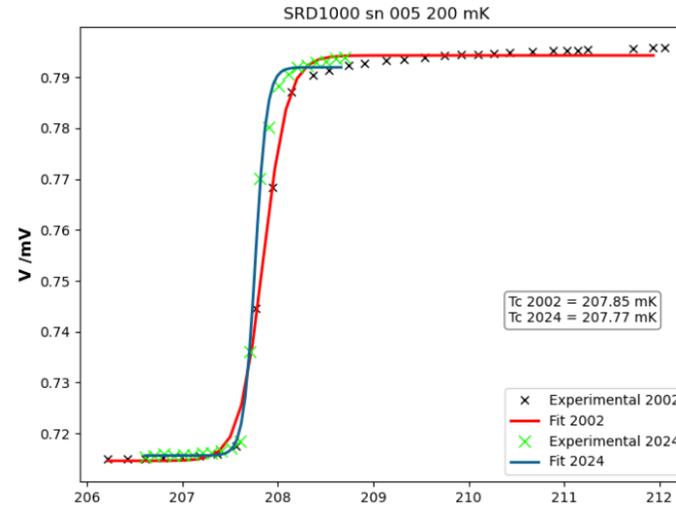
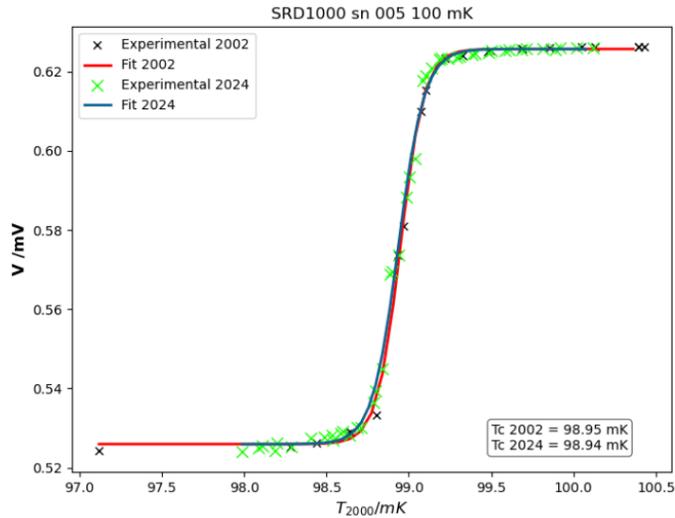
We compared 2 SRD1000, one (sn005) which shows the aging of the sensor over more than 20 years and the second, (sn237) bought in 2021 is tested in order to compare 2 same sensors.



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SRD1000 from HDL

Use of **sigmoid function** models on the transitions (conducted in collaboration with Rod White (NZ)).



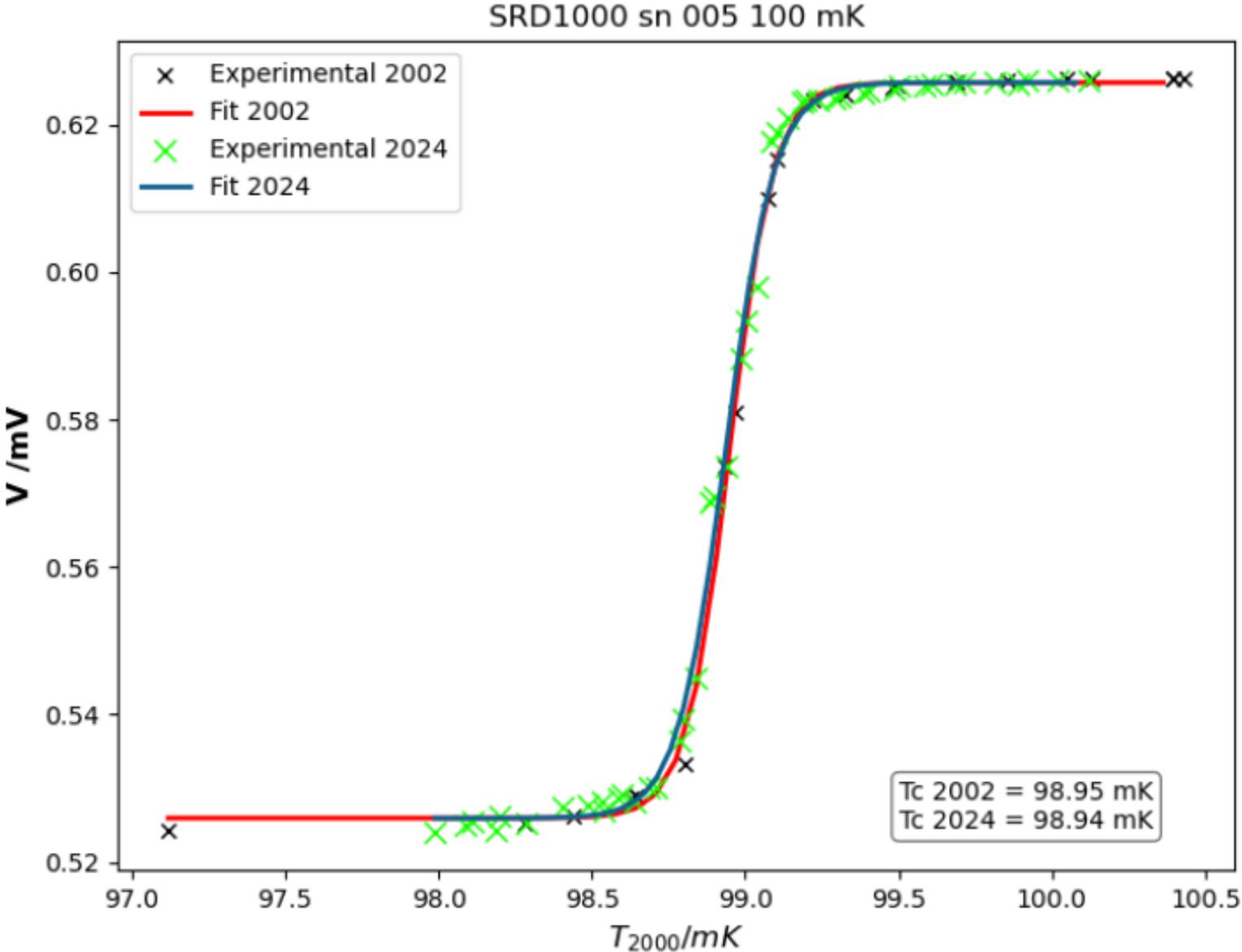
The data points represent the average of at least 15 minutes with very stable regulation, where half the points are taken during a rise in temperature and the other half during a decrease... showing no hysteresis.

Two sigmoid functions can accurately fit even a more complex transition.



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SRD1000 from HDL over 22 years

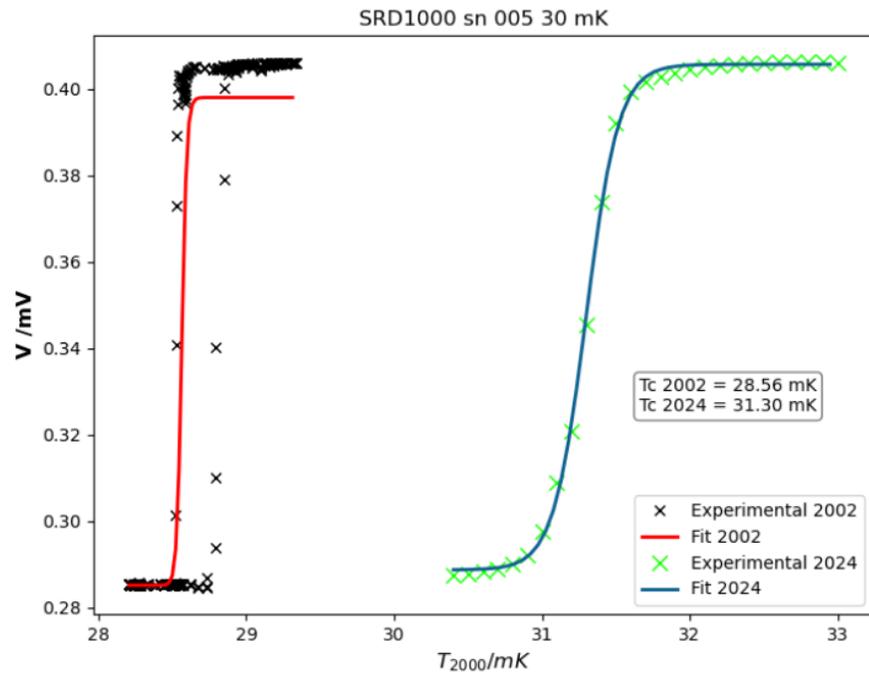


The SRD1000 from HDL is very stable; however, the shape of some transitions changes over the years. Uncertainty budget will come...

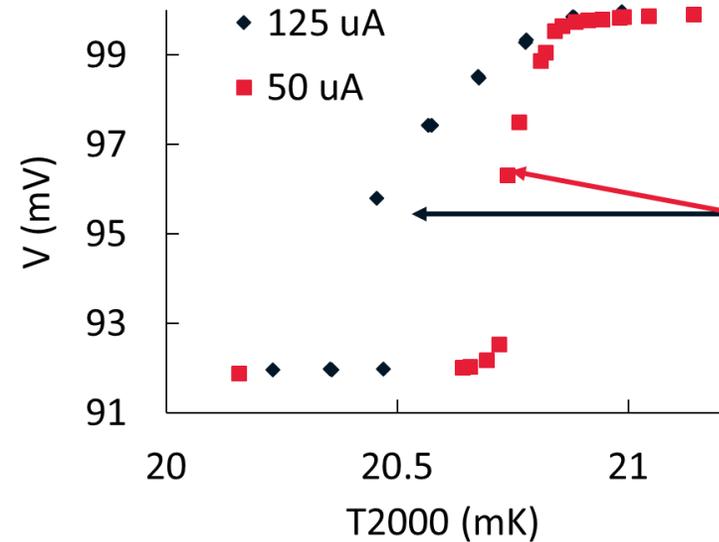


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SRD1000 from HDL



Without microwave filter With a filter



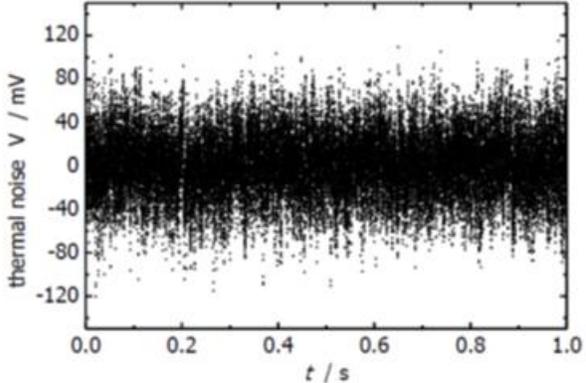
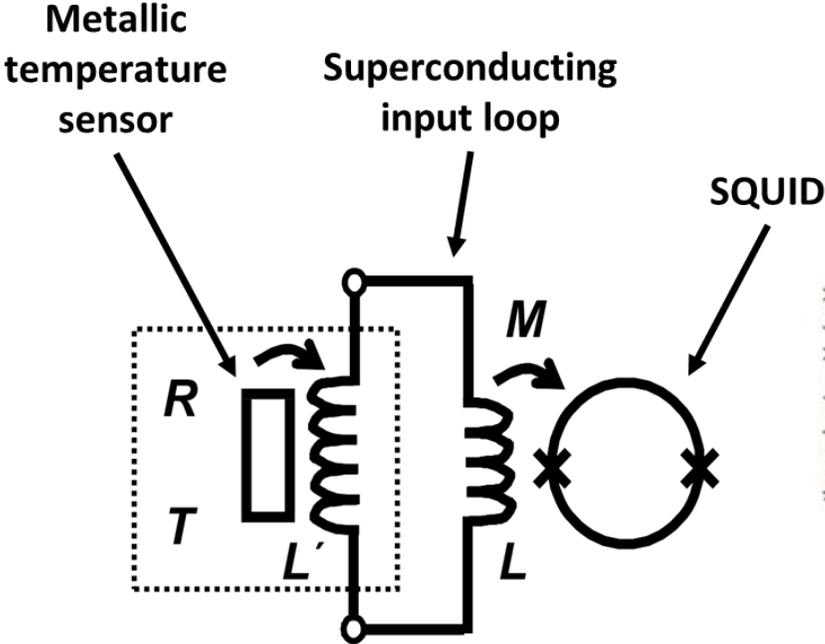
Influence of the excitation current in the Be transition at 20 mK with the sn237 (0.3 mK)

Even the shape of the transition has changed by reducing the current.

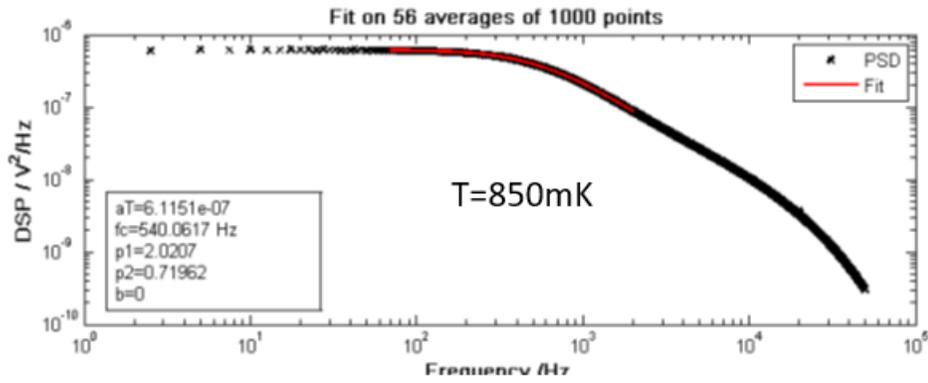


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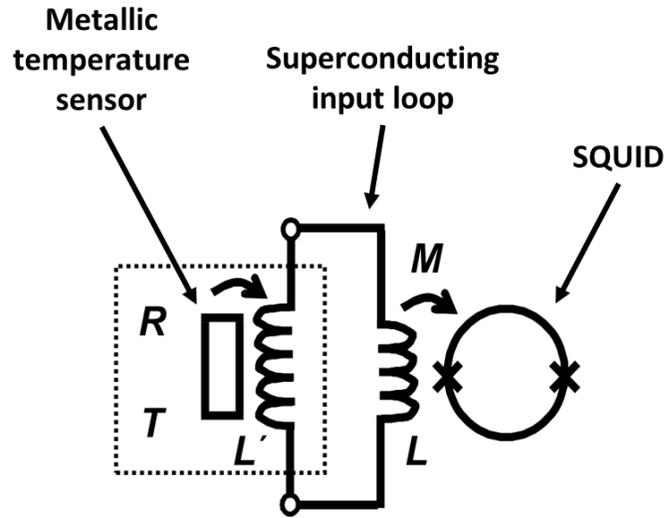
TMFFT from Magnicon



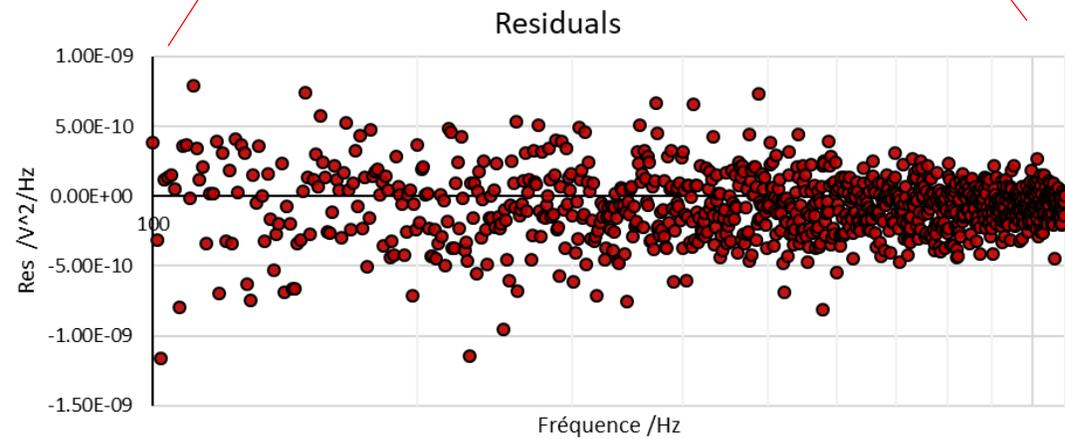
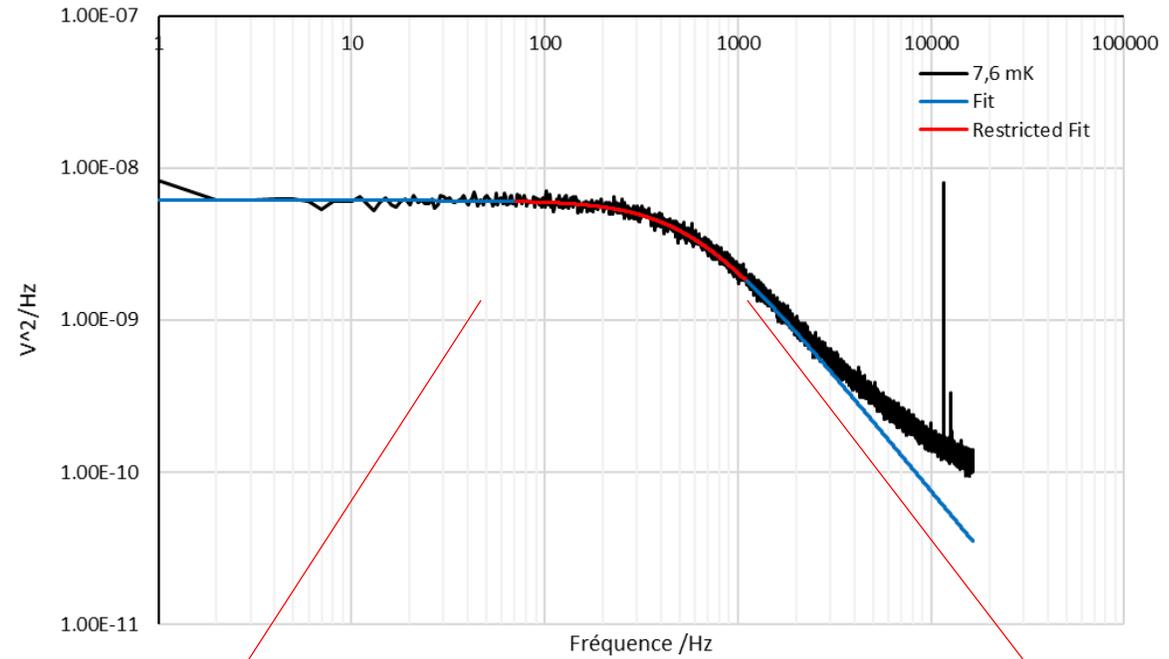
fft



TMFFT from Magnicon

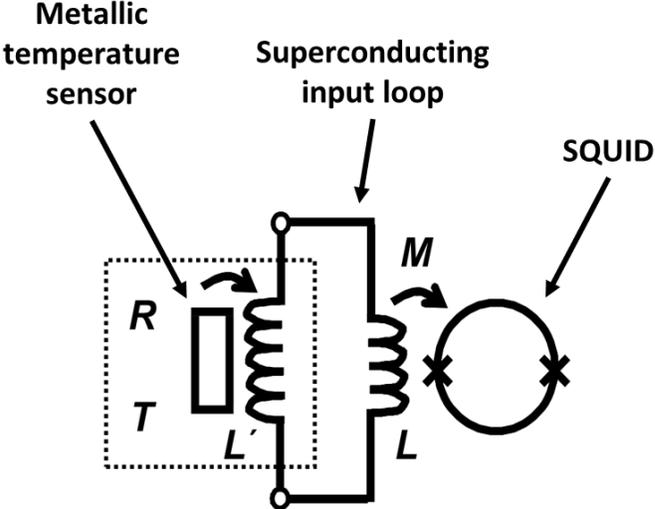


$$S_{\Phi}(f, T) = \frac{aT}{\left(1 + \left(\frac{f}{f_c}\right)^{2p_1}\right)^{p_2}} + b$$

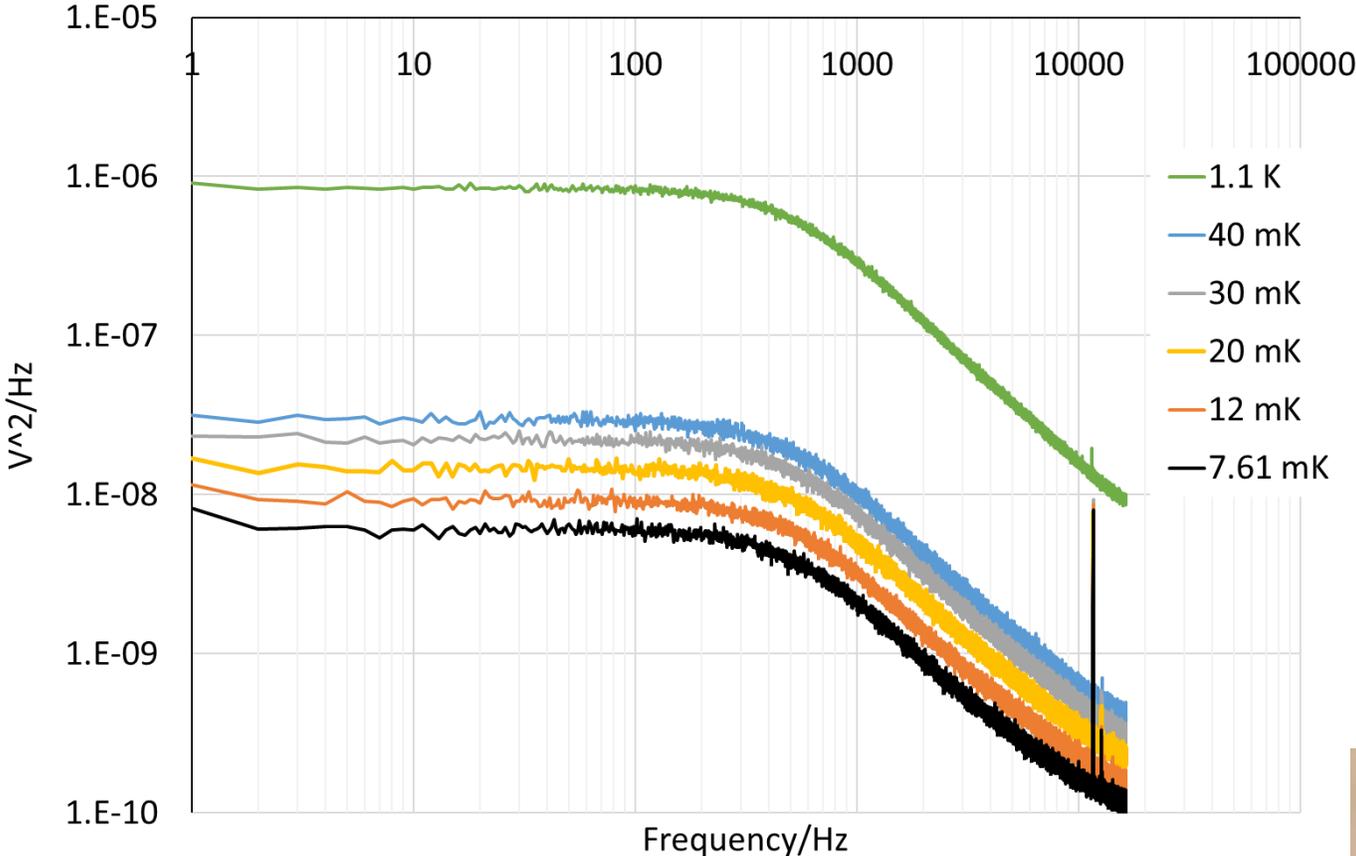


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TMFFT from Magnicon



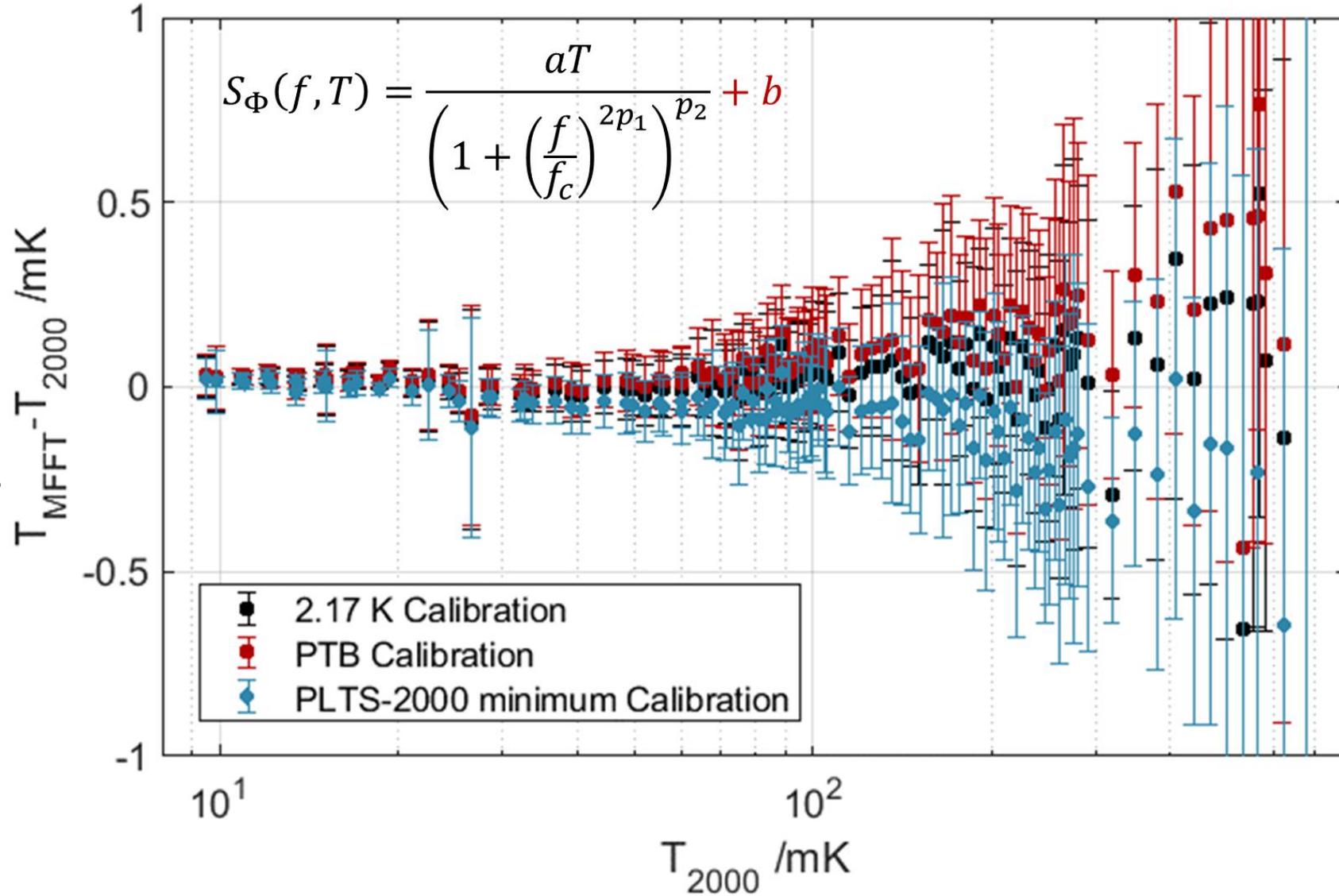
$$T = T_{ref} \frac{S_0(T)}{S_0(T_{ref})}$$



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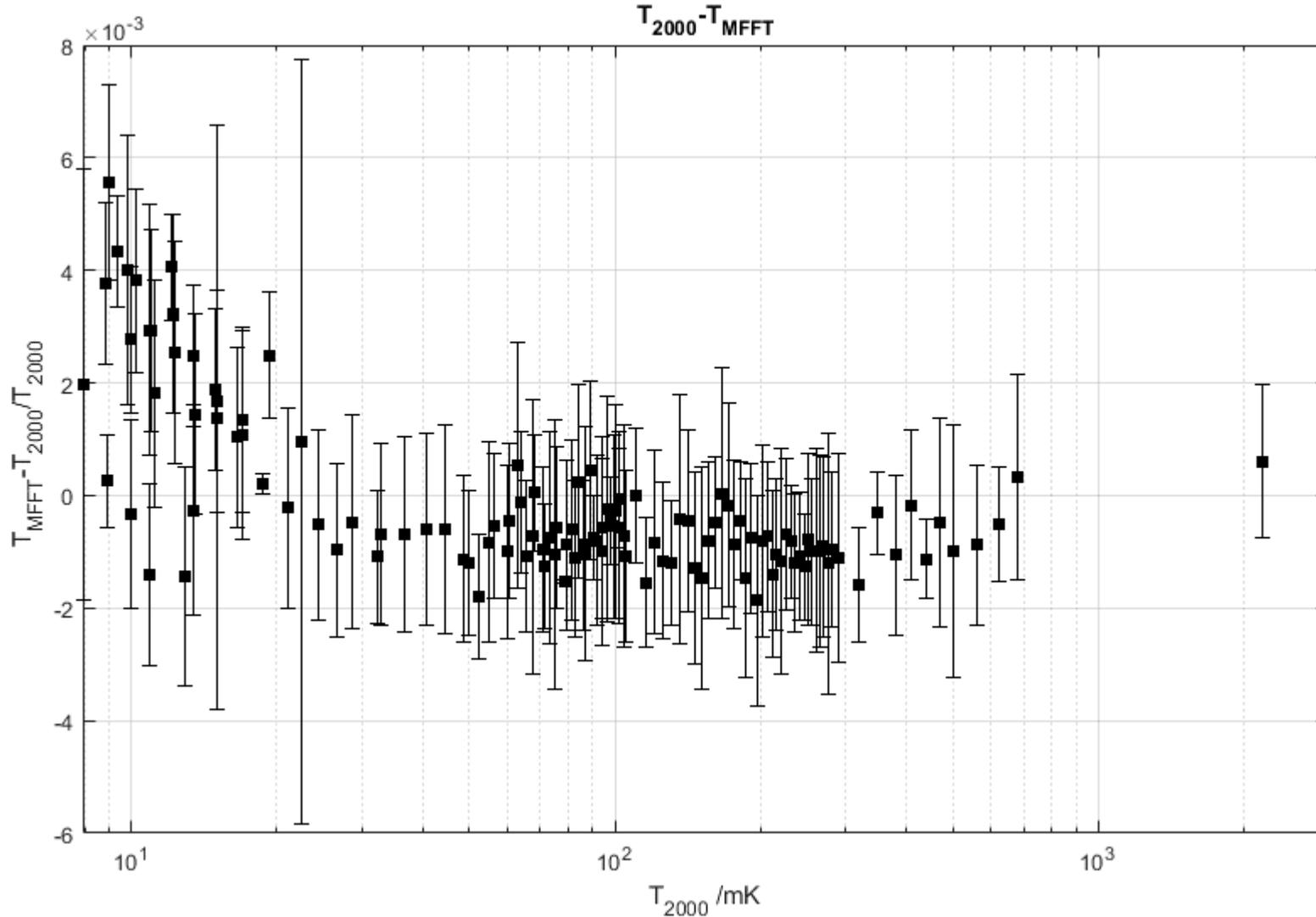
$T_{\text{MFFT}} - T_{2000}$ at LNE

120 temperature
measurements
Preliminary results



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$T_{\text{MFFT}} - T_{2000}$ at LNE-Cnam



0.5%

Preliminary results:
The uncertainty budget is not finished.
Soon, Clément Tauzin will defend his PhD.



Clément Tauzin
Doctorant

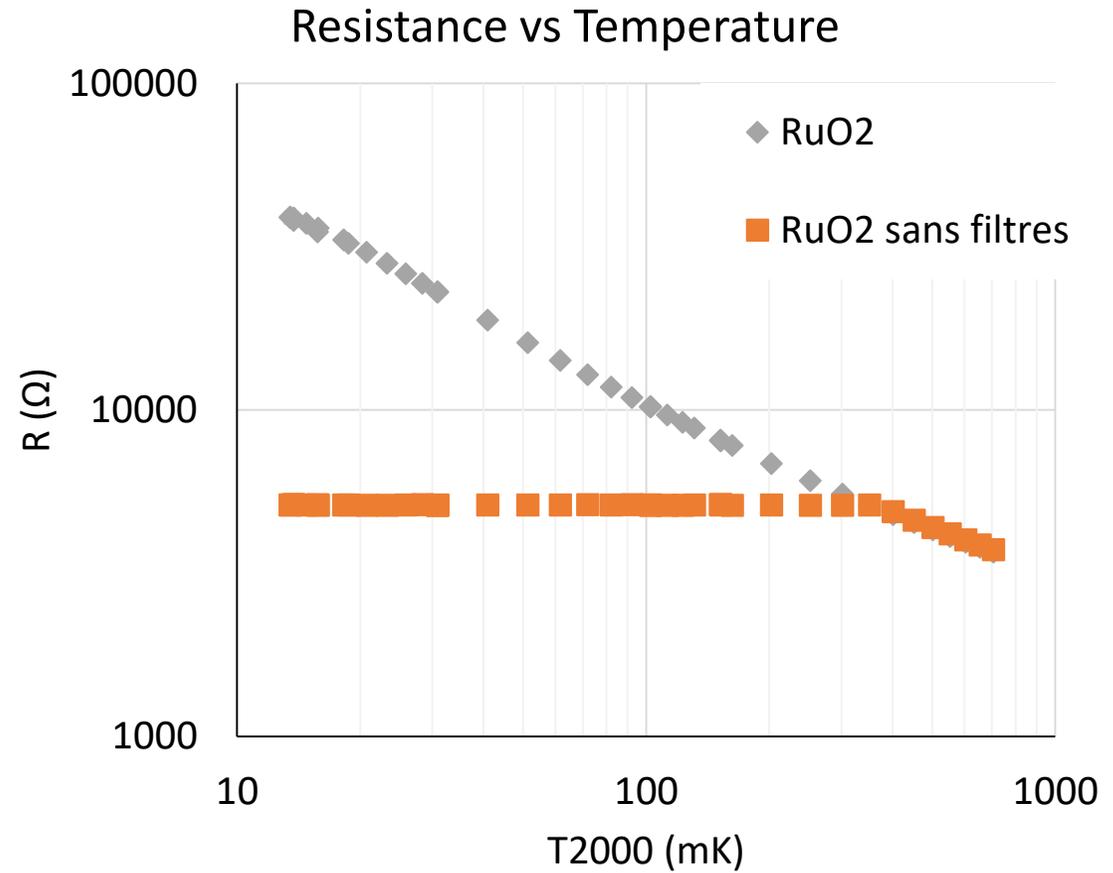
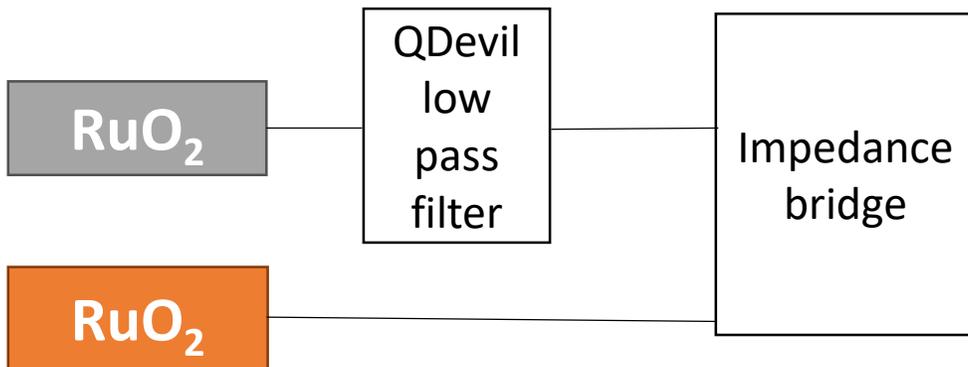
$T_{\text{MFFT}} - T_{2000}$ at LNE

- Over three months of noise integration
- 120 temperature measurements
- Work in progress (uncertainty budget not yet complete), but results are very promising.

- Excellent linearity of PLTS-2000 as a function of temperature from 8 mK to 800 mK, even with a 12-parameter polynomial for PLTS-2000!
- Primary relative temperature measurements at 2.1768 K and 315.24 mK are equivalent.

Resistive Thermometer and T2000 at LNE-Cnam

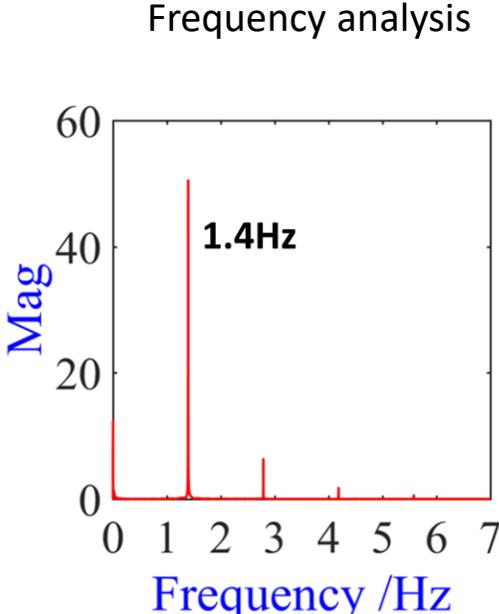
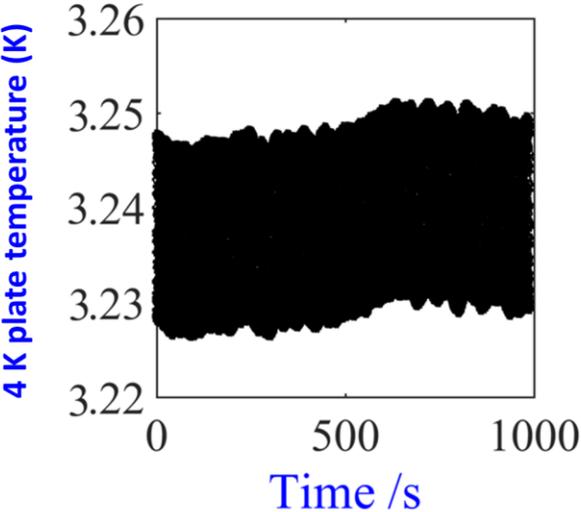
Same bridge
Same position
Same excitation
Same sensor



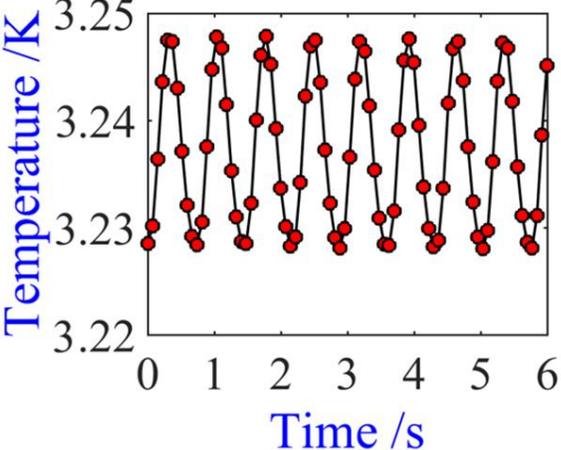
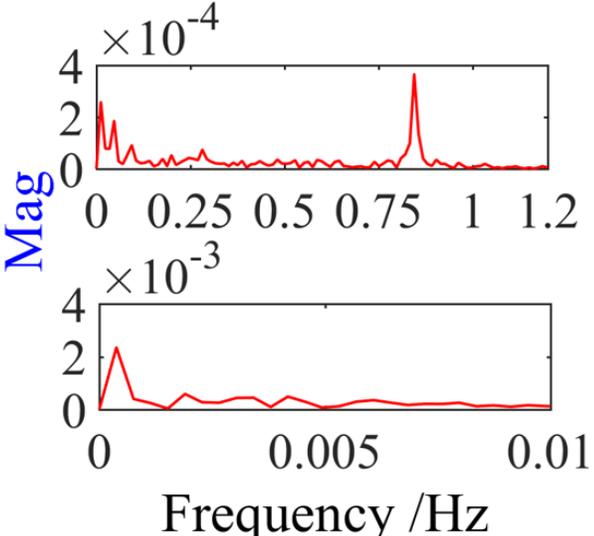
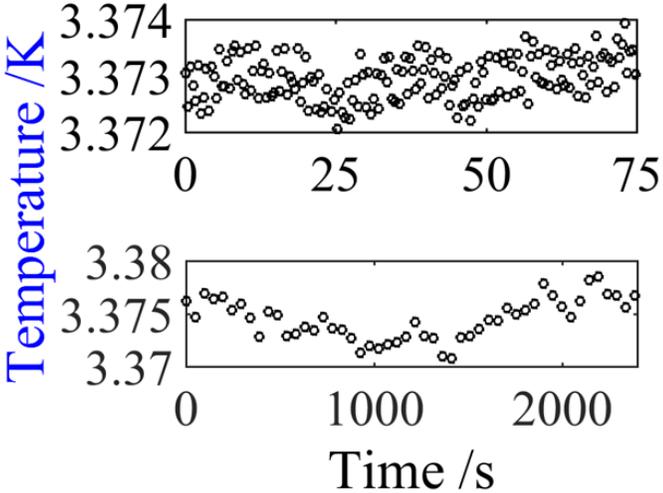
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Resistive Thermometer and sampling

Temperature measurement performed at LNE-Cnam on the 4 K plate @ 10 Hz



But if we use only instruments and settings provided by Bluefors, we cannot observe any thermal oscillations because the sampling rate is too low

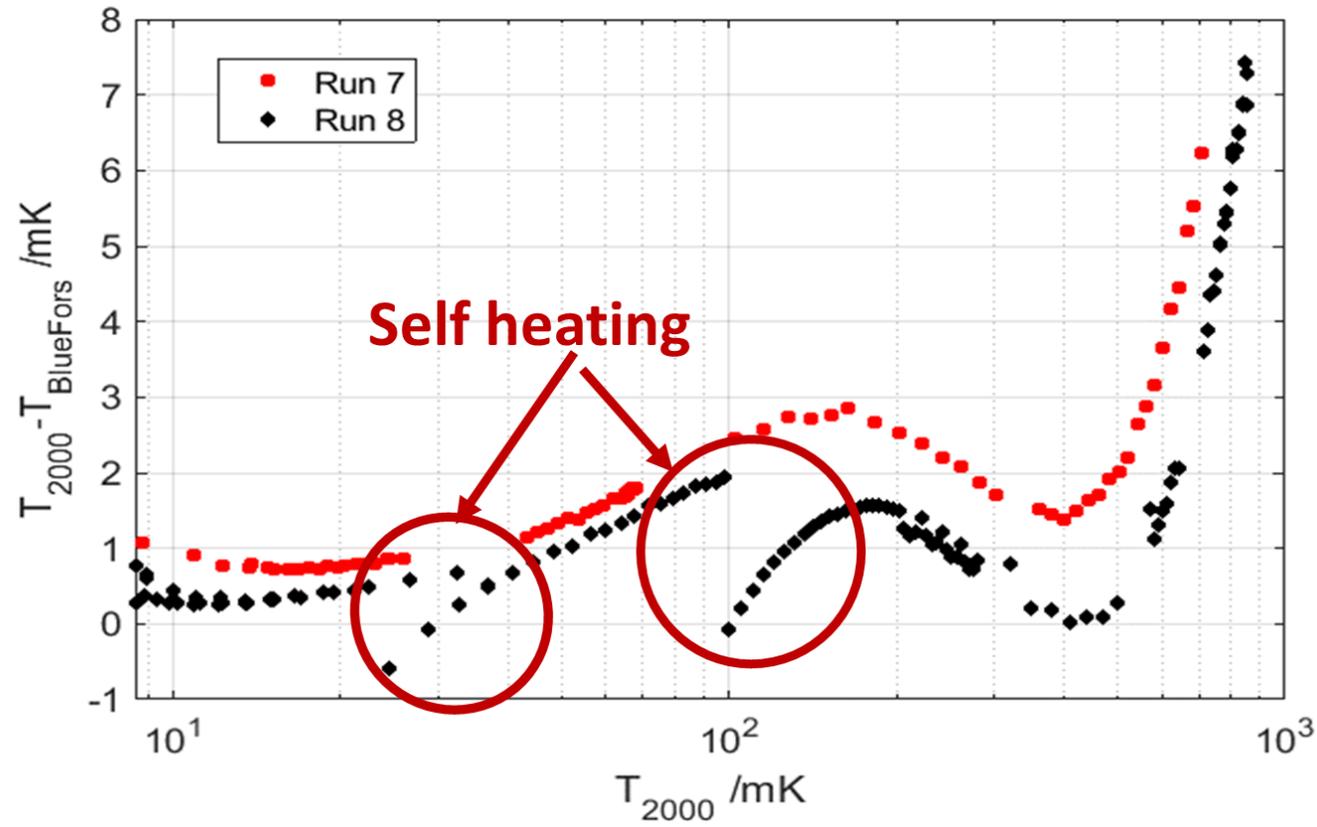


Temperature measurements were conducted by LNE-Cnam on the 4 K plate of a DR from BlueForth using a 10 Hz digital multimeter to measure a Cernox thermometer through a 4-wire resistance measurement method. sinusoidal temperature oscillations are well visible

Resistive Thermometer and T2000 at LNE

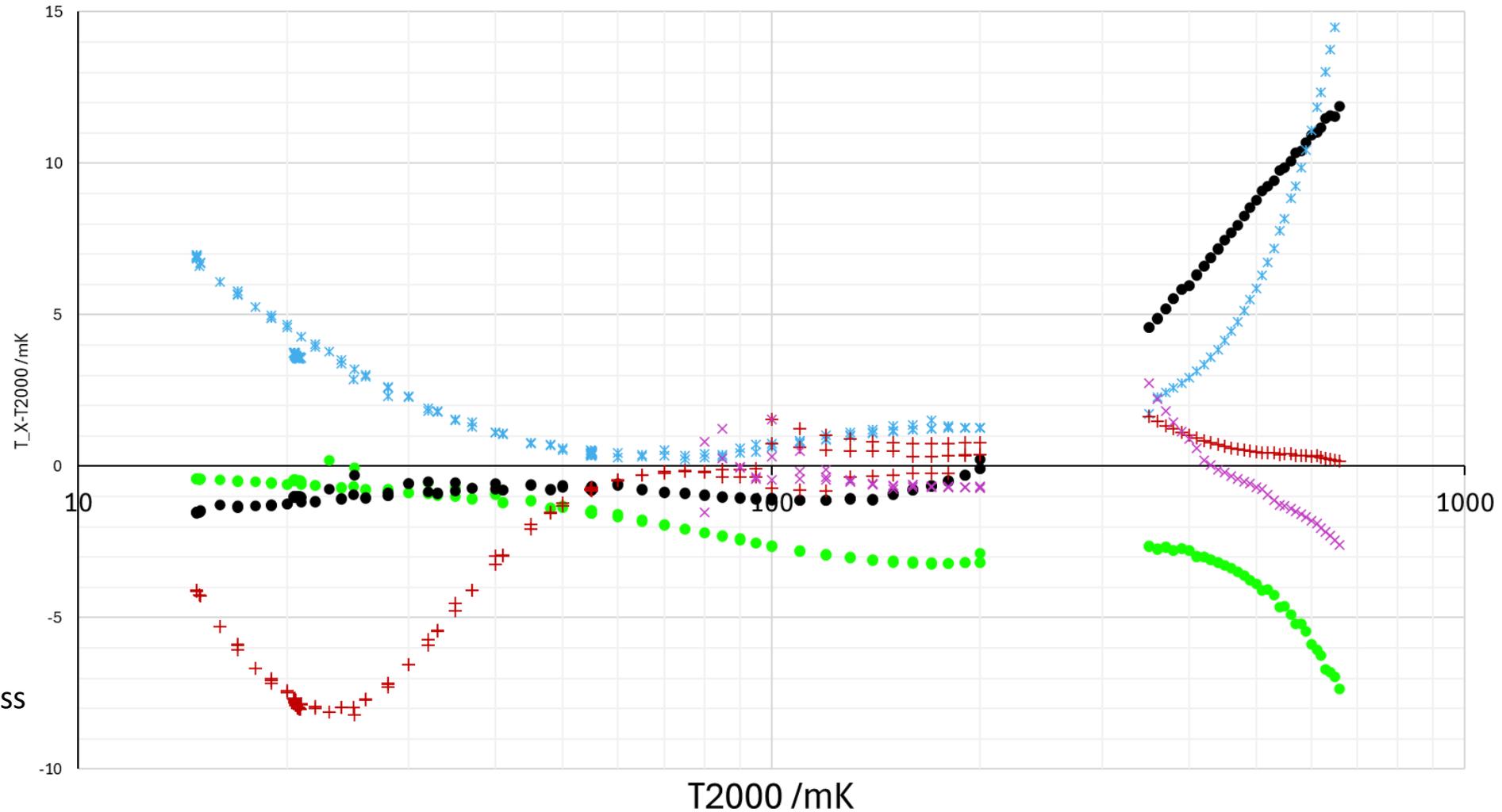
Self heating and repeatability over 2 cooldowns!!!

*Direct comparison between BlueFors and PLTS-2000
over 2 runs*



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Resistive Thermometer and T2000 at LNE



Work in progress

● T2000-T7 ● T2000-T6 × T2000-TT2151 + T2000-RuO2_H × T2000-X151184

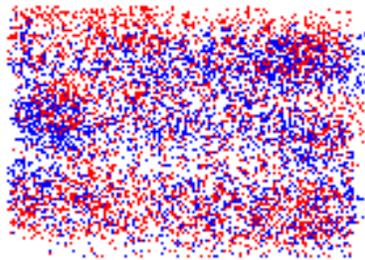


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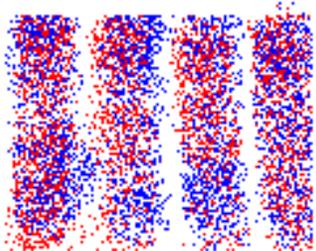
Second Sound in the mixture of He³-He⁴

In superfluid helium, the normal component of the Helium-3/Helium-4 mixture can move frictionlessly through the superfluid Helium-4 component.

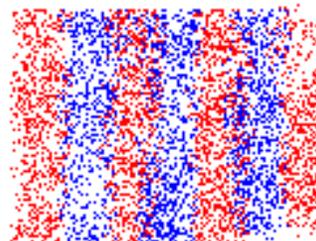
In **first** (normal) sound, acoustic waves propagate as compressions and rarefactions i.e. density varies spatially



fluid with two elements



first sound



second sound

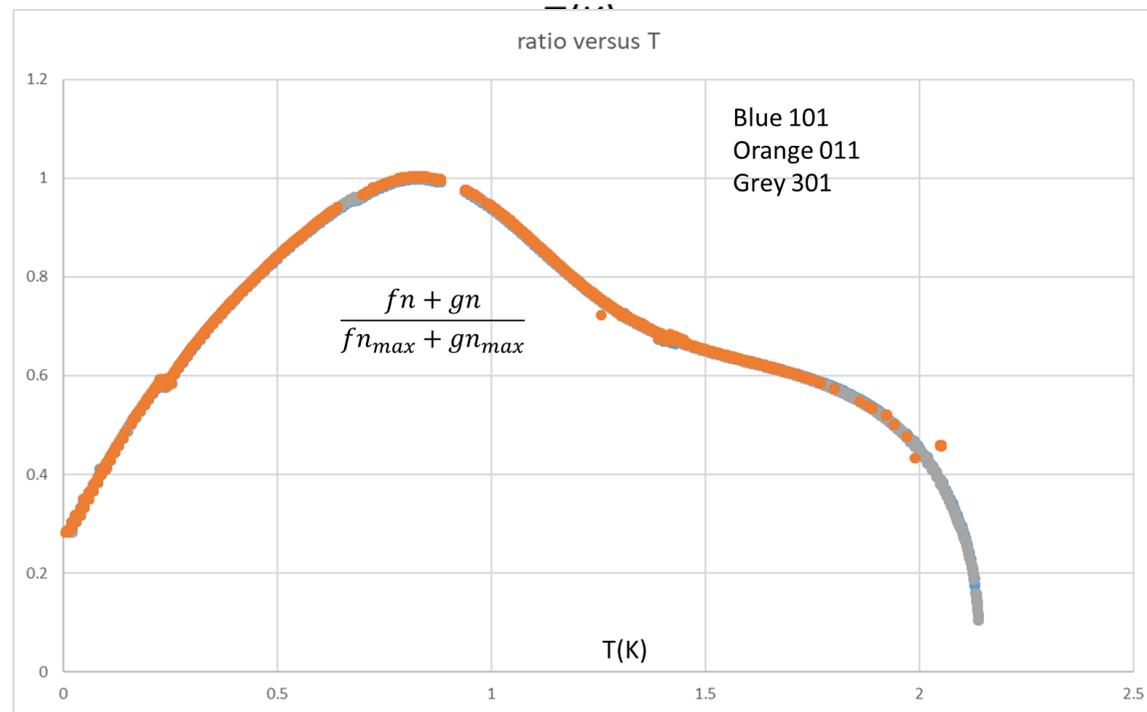
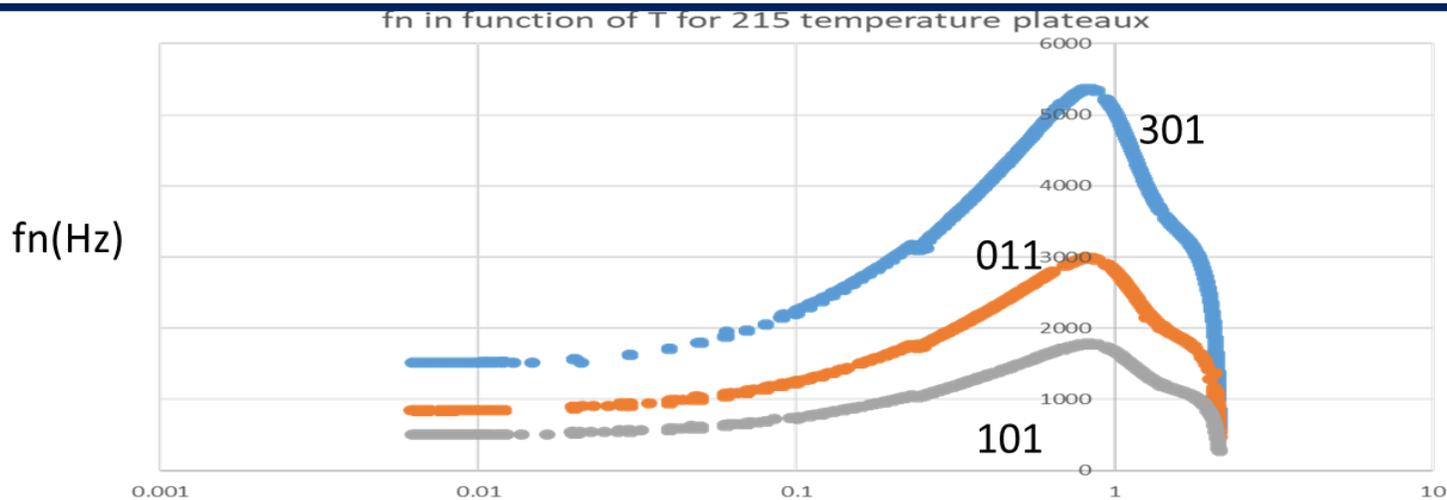
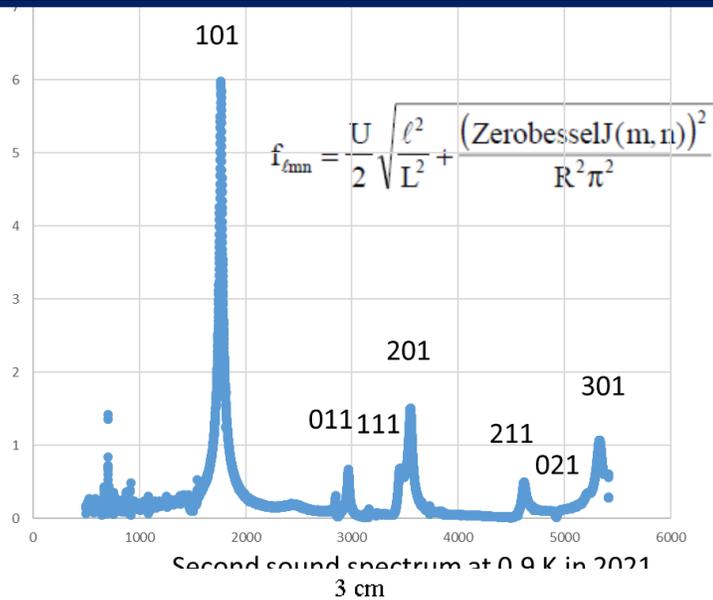
In **second** sound the total density of the mixture remains constant

$$\rho_m = \rho_s + \rho_{4n} + \rho_3$$

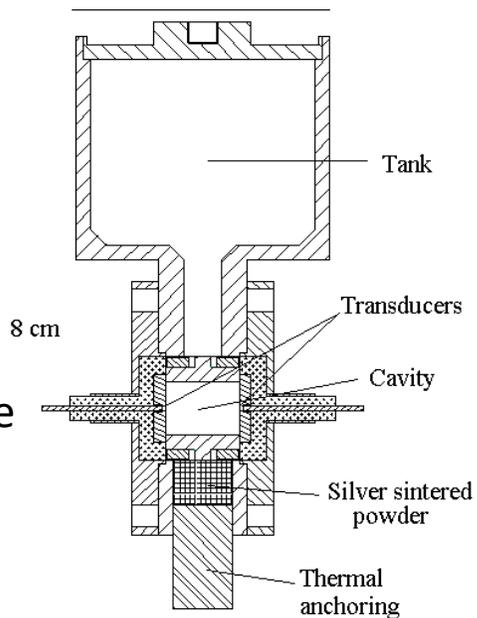
Superfluid Normal
He⁴ He⁴

With a He³ concentration around 1%, the liquid in a He³-He⁴ mixture exhibits the properties similar to a gas consisting of particles heavier than He³ (approximately 2.3 times). It resembles a gas where 'pressure' is substituted by the He³ concentration (determined by X), while the speed of sound varies with the square of temperature (T²).⁶¹

Second Sound Thermometer in the mixture of He³-He⁴

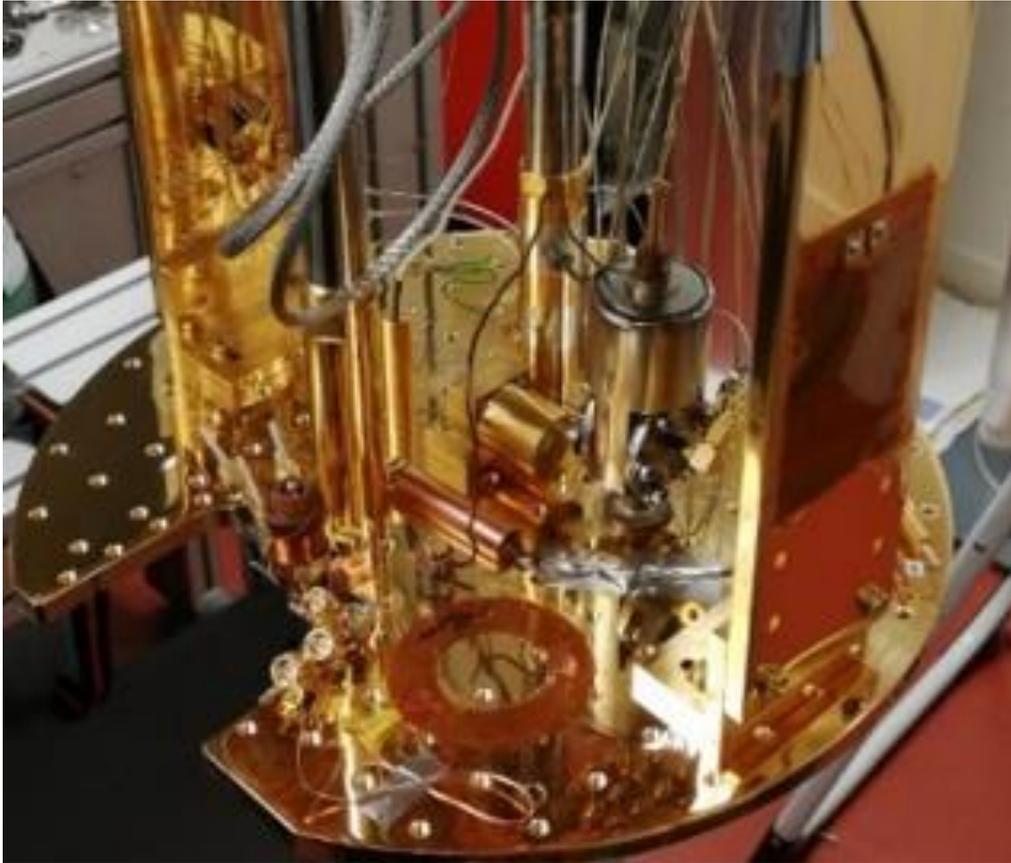


Sealed
at room
temperature



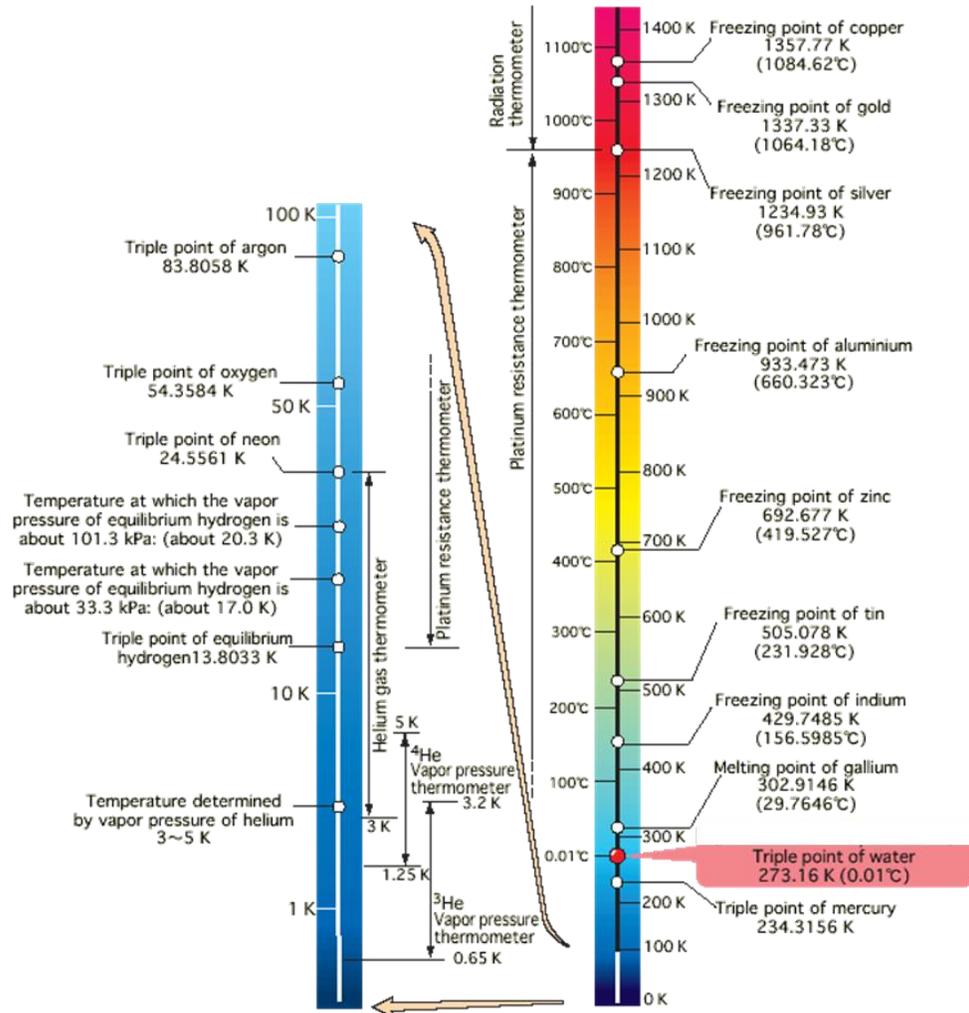
Second Sound Thermometer in the mixture of He^3 - He^4

Measurement of the second sound in a ^3He - ^4He mixture with 3 acoustic modes from 8.3 mK to 2.15 K (factor 262 in T)



- Connected with thermophysical properties.
- Easy to measure and not dependent on cable impedance.
- Stable cell (over 20 years).
- Easy to install (no filling tube required).
- Redundancy in measurement (multiple modes).
- Potential for use as a primary thermometer.
- Wide range of applications.

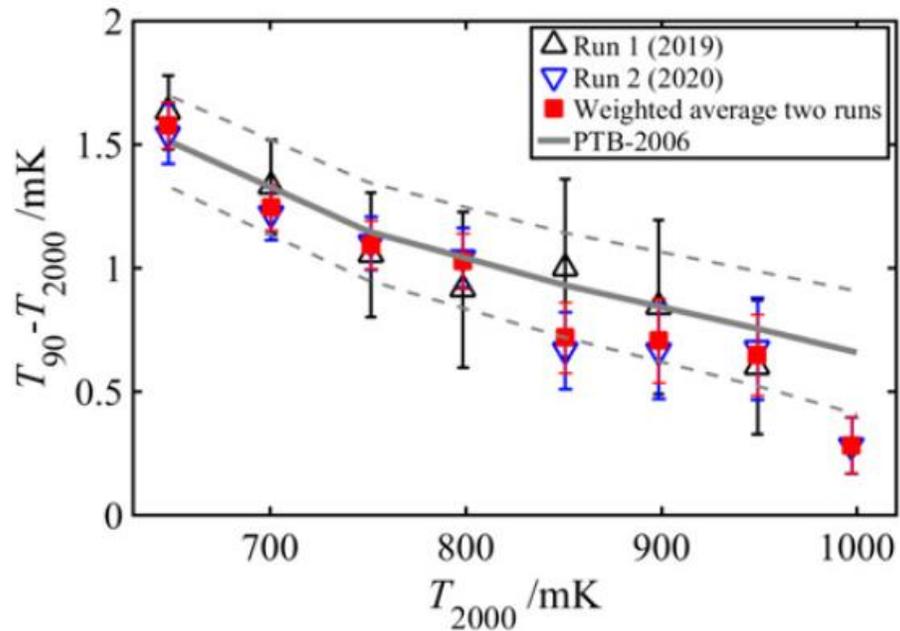
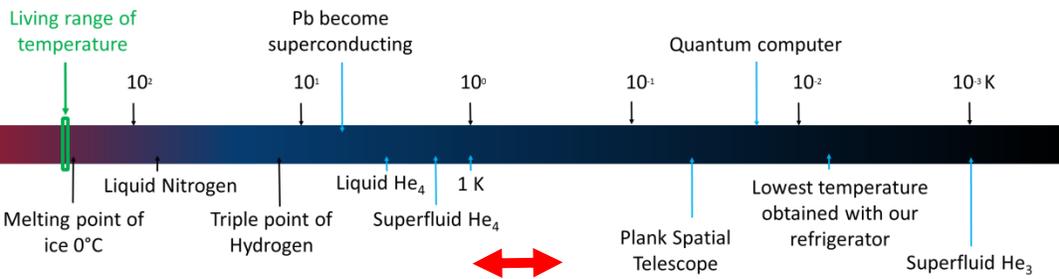
International Temperature Scale 1990



ITS90 is an approximation of the thermodynamic temperature
From **0,65K to 5K** ITS 90 give:

- A thermometer
- Interpolating equation

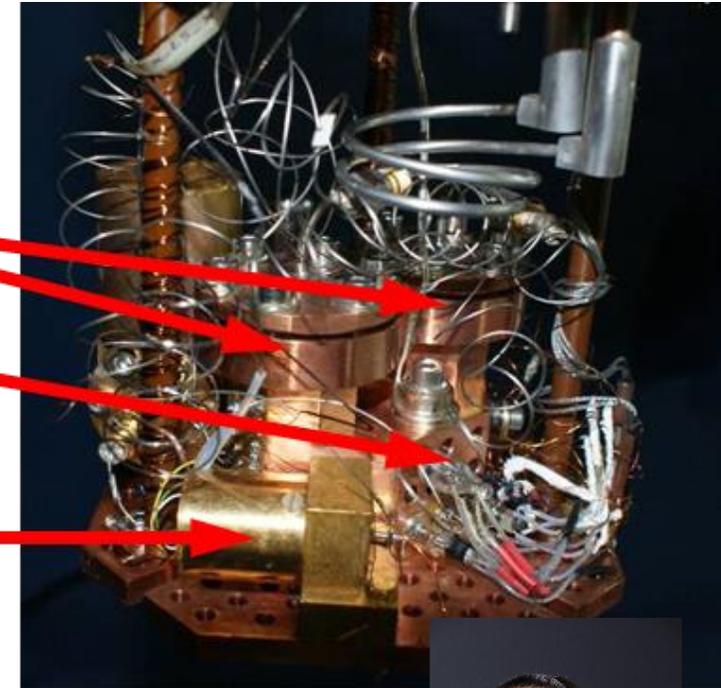
Measurement with two scales between 0,65K and 1K $T_{90}-T_{2000}$



Vapor Pressure Chambers

RhFe Thermometers

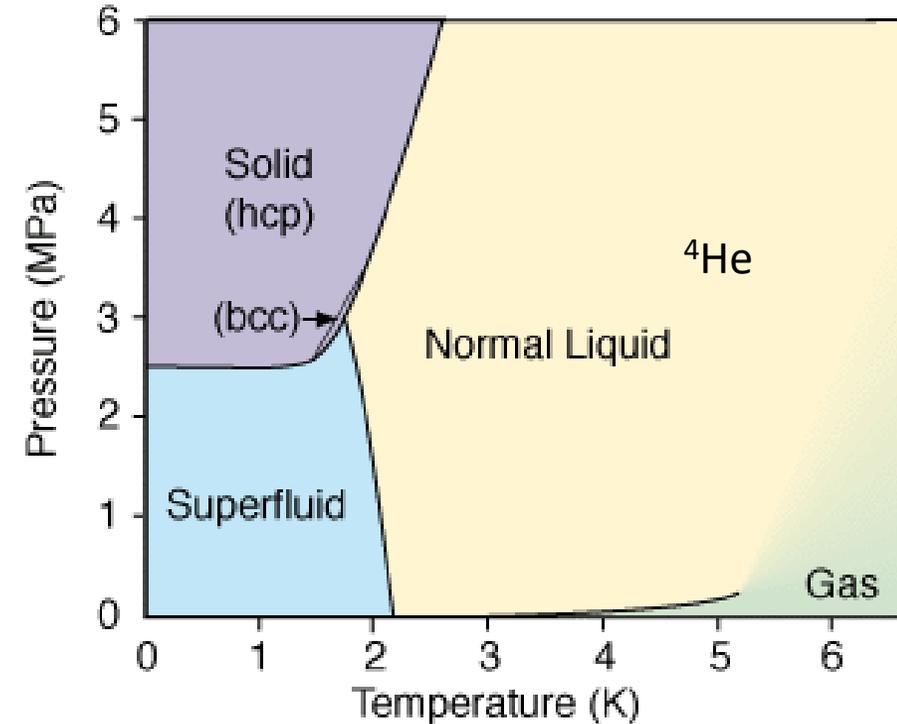
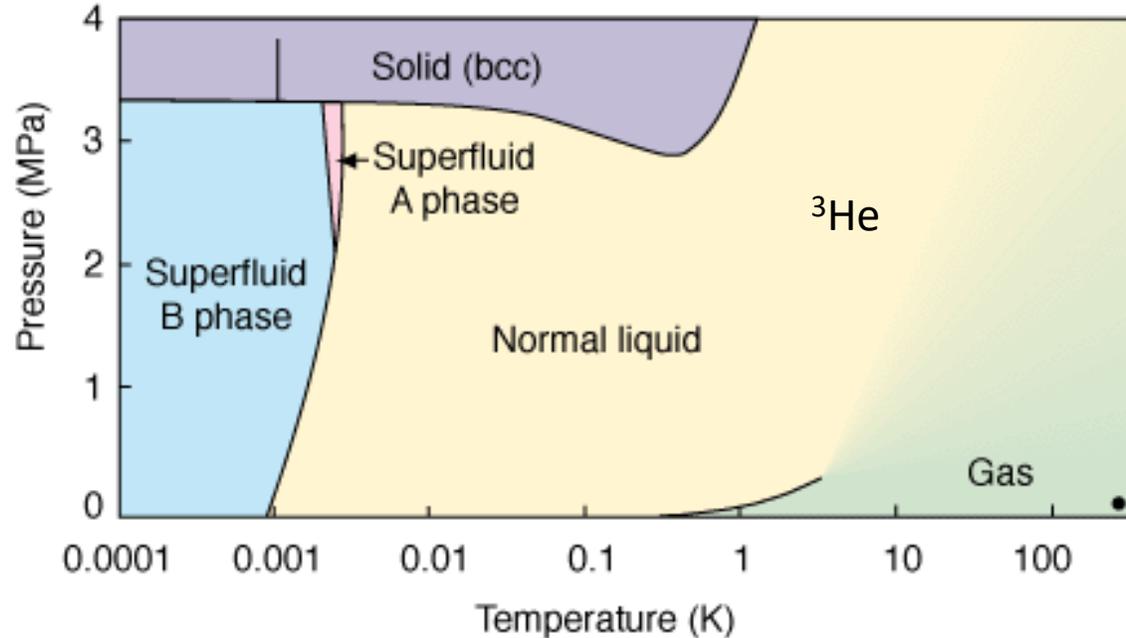
Melting-curve thermometer



Changzhao Pan

C Pan, FSparasci, M Plimmer, L Risegari, J-M Daugas, G Rouille, B Gao, L Pitre Direct comparison of ITS-90 and PLTS-2000 from 0.65 K to 1 K at LNE-CNAM Metrologia 58 (2), 025005 <https://doi.org/10.1088/1681-7575/abd845>

Helium-3 and Helium-4



<http://l.tl.tkk.fi/research/theory/helium.html>

C Pan, H Zhang, G Rouillé, B Gao, L Pitre *Journal of Physical and Chemical Reference Data* 50 (4), **Helmholtz Free Energy Equation of State for ^3He – ^4He Mixtures at Temperatures Above 2.17 K** 043102

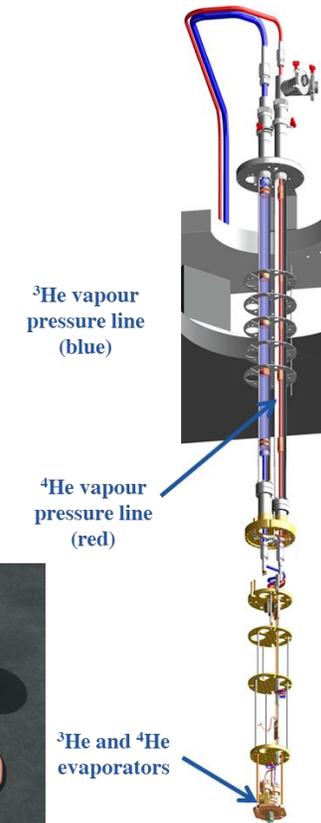
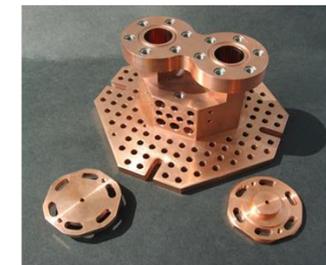
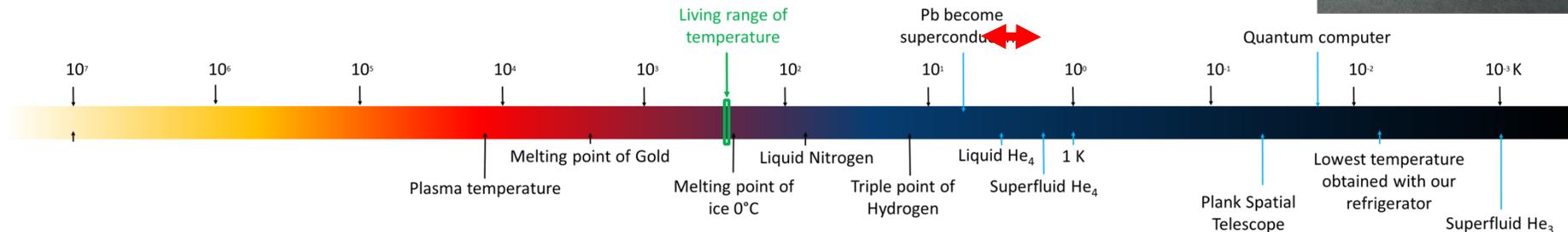
<https://doi.org/10.1063/5.0056087>

TVPHe4–TVPHe3 2.2K–3.2K

- ^3He – ^4He vapour-pressure thermometer was installed in a commercial dilution refrigerator
- The system was made of one copper block:
 - two separate cylindrical vapour-pressure chambers, one for ^3He and the other for ^4He
 - Comparison block for 9 resistance thermometers

Collaboration with
INTiB (Poland)
TIPC-CAS (China)

Article at ITS10



Fernando Sparasci



Aleksandra Kowal

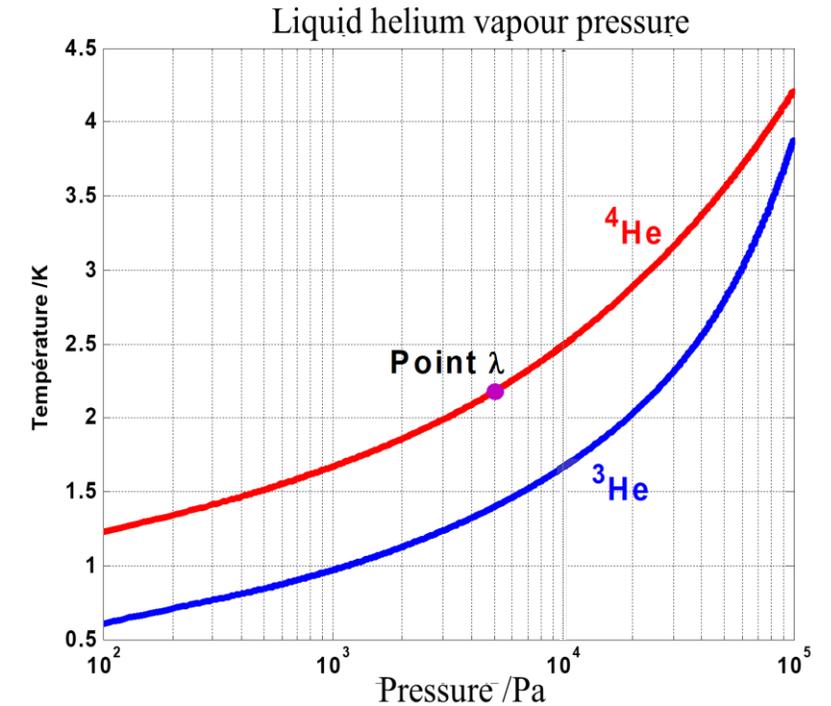
From 0.65 K to 5.0 K helium vapour–pressure temperature equations

From ITS90

$$T_{90}, \text{K} = A_0 + \sum_{i=1}^9 A_i [(\ln(p, \text{Pa}) - B)/C]^i$$

The values of the constants A_0 , A_i , B and C are given:

- for ^3He in the range 0.65 K to 3.2 K
- for ^4He in the range 1.25 K to 2.1768 K (the λ point) and 2.1768 K to 5.0 K.

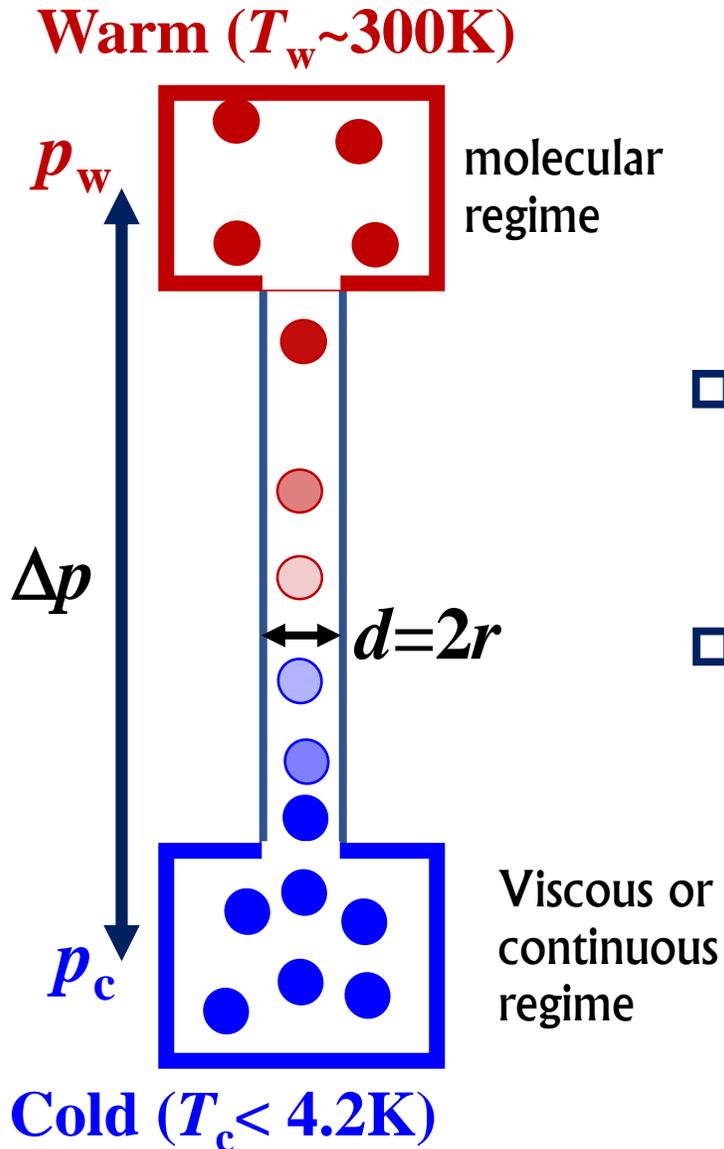


Fernando Sparasci



Aleksandra Kowal

Thermomolecular pressure difference



A thermomolecular pressure difference appears at lower pressures when the gas mean free path is comparable to the tube diameter, and it plays an important role in **vapor pressure thermometry**.

□ Ideal case

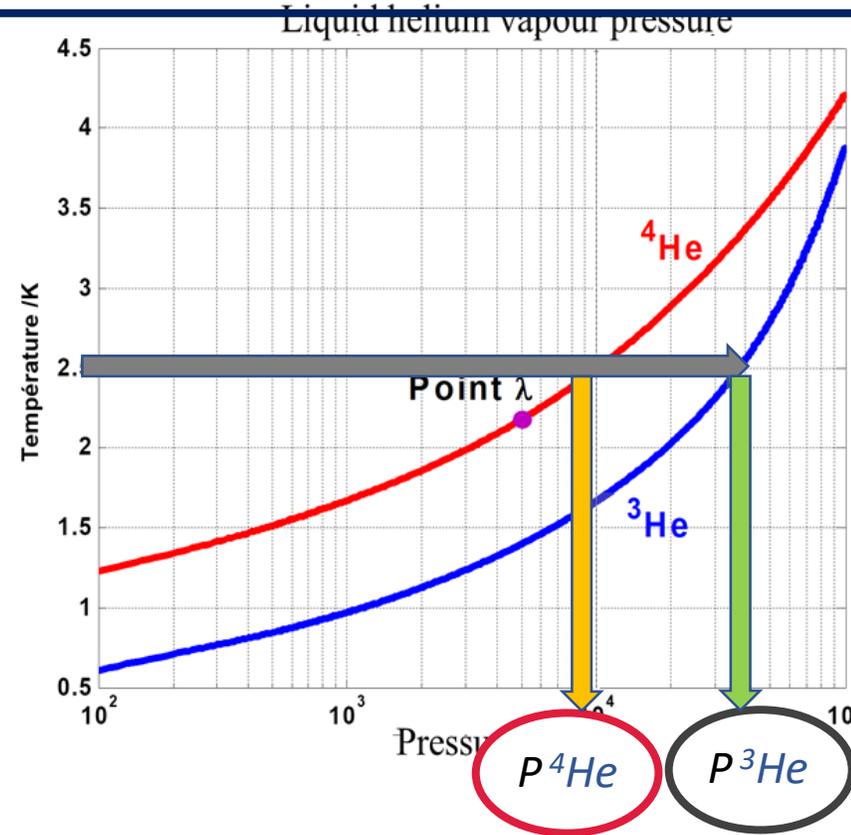
$$\frac{p_w}{p_c} = \left(\frac{T_w}{T_c} \right)^{1/2}$$

□ In practice
complicated

$$\frac{\Delta p}{p_c} = \frac{p_w - p_c}{p_c} = \frac{2 \times 10^{-9}}{\left(\frac{rp_c}{\text{m} \cdot \text{Pa}} \right)^{1.99}} \left[\left(\frac{T_w}{\text{K}} \right)^{2.27} - \left(\frac{T_c}{\text{K}} \right)^{2.27} \right]$$

No general theory is applicable in all possible experimental cases. More understanding is needed

^3He – ^4He vapour pressure thermometer at LNE–CNAM



$$P^{4\text{He}} = P_{\text{measured}} + \Delta P_{\text{calibration}} + \Delta P_{\text{hydrostatic}} + \Delta P_{\text{thermomolecular}}$$

$$P^{3\text{He}} = P_{\text{measured}} + \Delta P_{\text{calibration}} + \Delta P_{\text{hydrostatic}} + \Delta P_{\text{thermomolecular}}$$

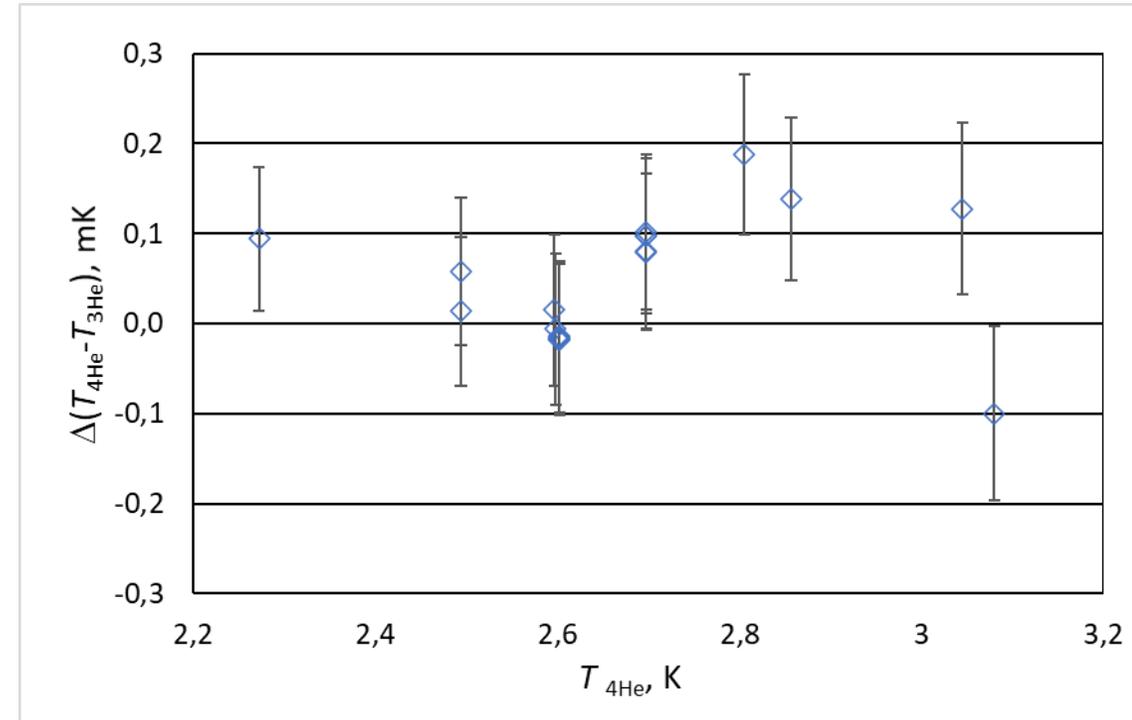
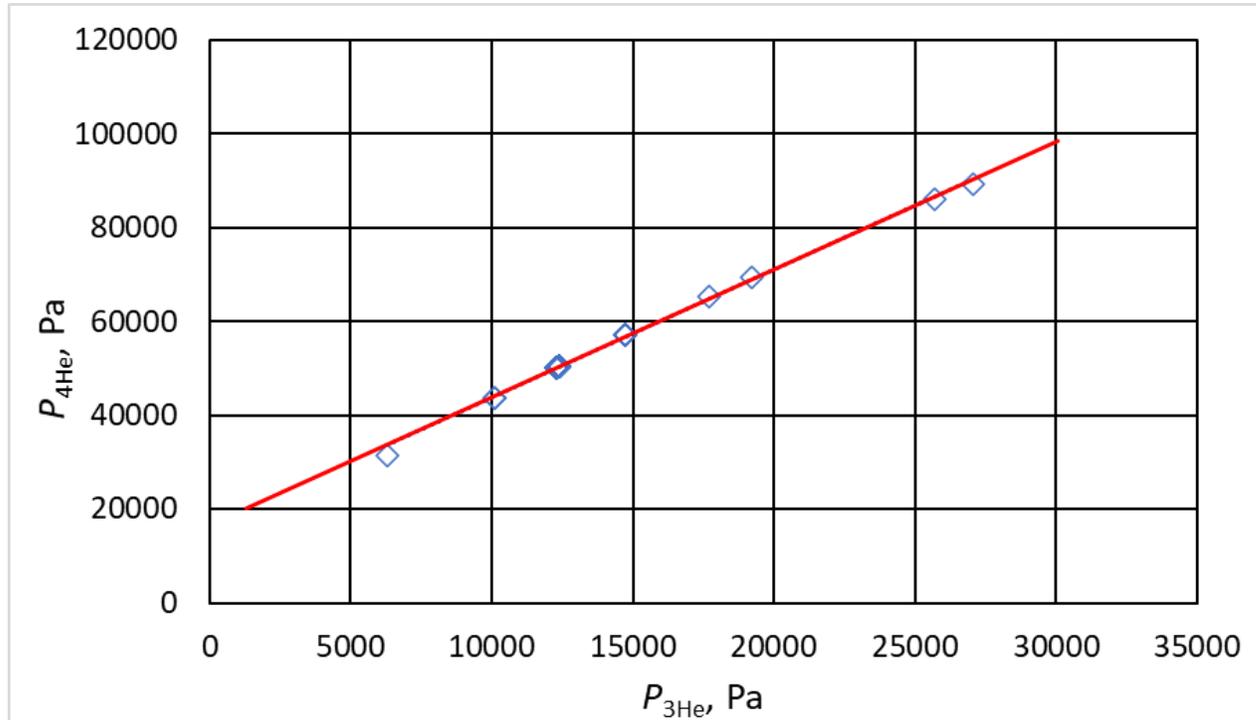


Fernando Sparasci



Aleksandra Kowal

TVPHe4–TVPHe3 2.2K–3.2K



The Direct Comparison of ^3He and ^4He Vapor-Pressure Thermometers at LNE-Cnam within their Overlapping Temperature Range. A. Kowal, F. Sparasci *et al.* Accepted for publication on the ITS10 symposium

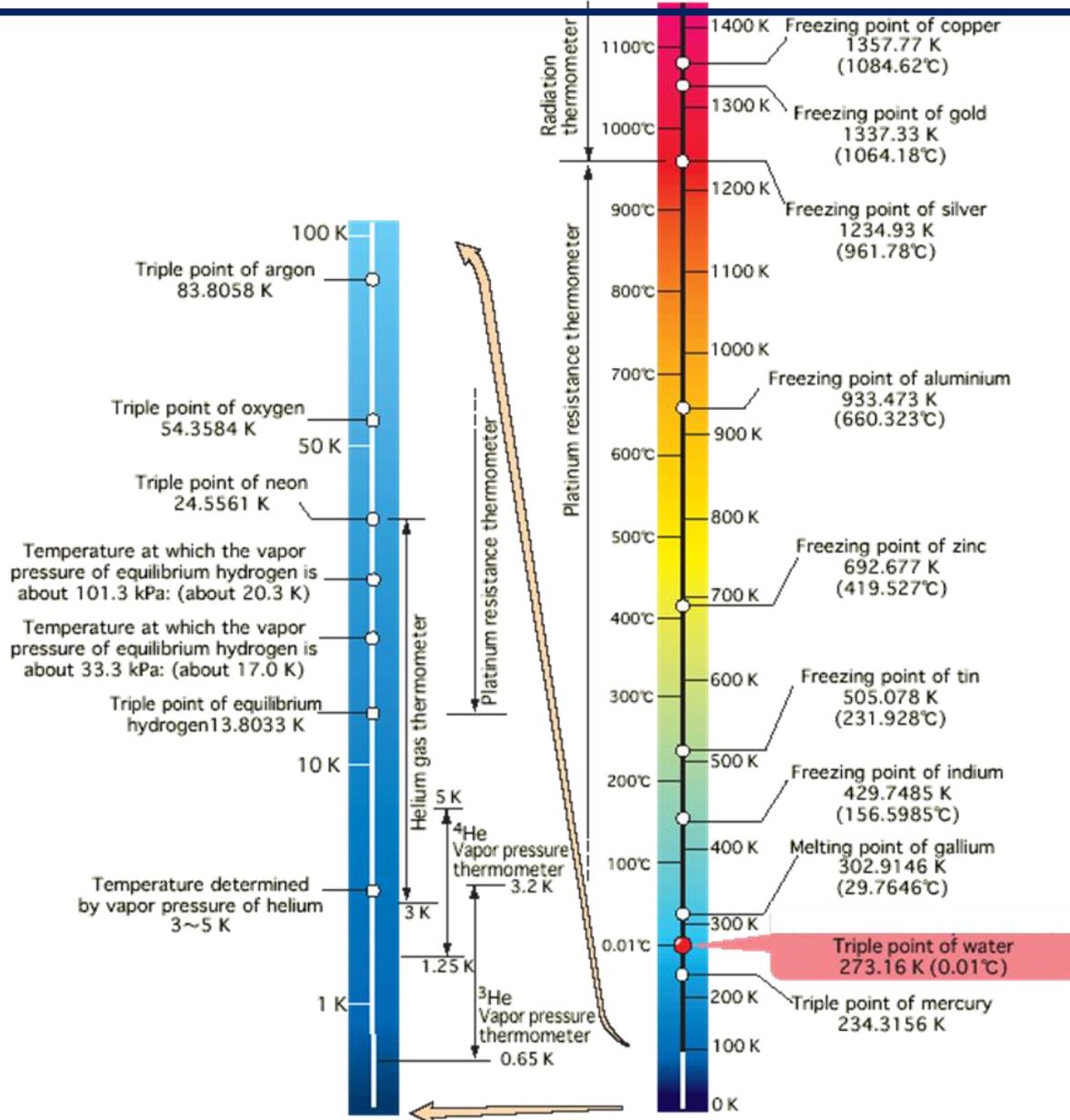


Fernando Sparasci



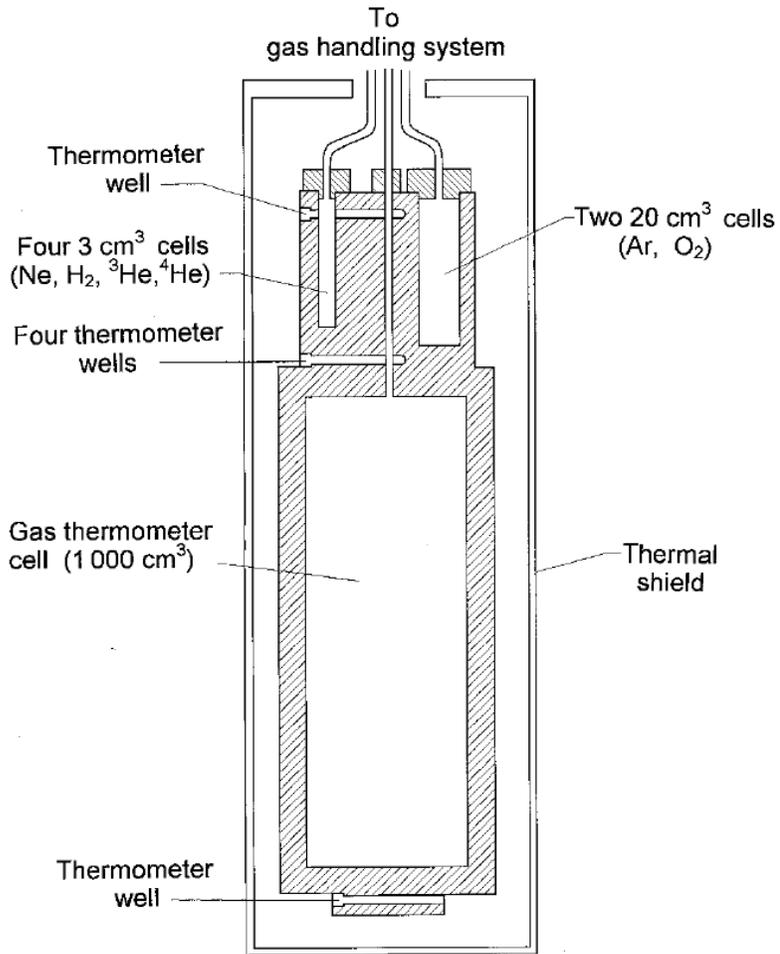
Aleksandra Kowal

International Temperature Scale 1990



ITS90 is an approximation of the thermodynamic temperature
From 3K to 24,5 K IST 90 give:
 A thermometer
 Fix point
 And interpolating equation

But a very complex ITS90 scale below 25K



As far as I know, the most recent implementation of ITS90 for temperatures below 25K occurred in 1996 at NIST. Since then, the only method used worldwide has been the wire scale.

However, there is an issue with the wire scale in that it relies on a resistive thermometer that is no longer being manufactured.

Does the primary thermometry is the solution?

Pro

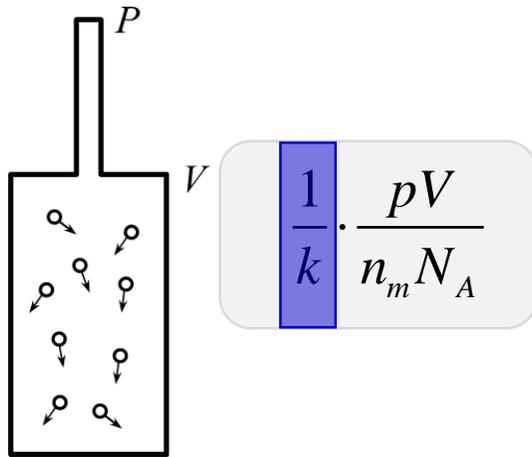
- Pollution has minimal effect at temperatures below 25K.
- Gases with higher density yield stronger signals.
- Progress has been made in ab initio calculations.
- There is a well-established model in place.
- Perturbations from the shell have been eliminated.

Con

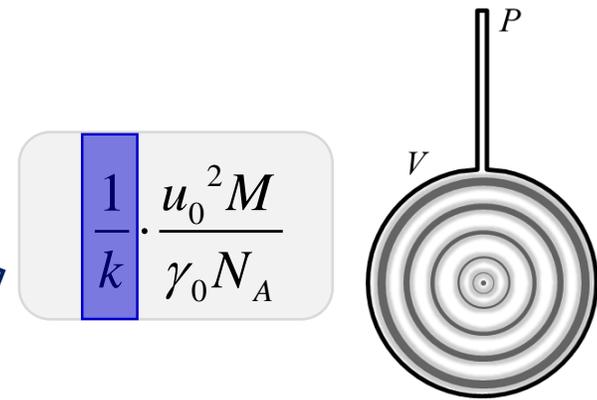
- Hydrostatic correction must be determined.
- Multiple measurements are necessary at identical temperatures.
- Analysis is complex.
- Need to know the accommodation coefficient

Typical primary thermometry in low temperature

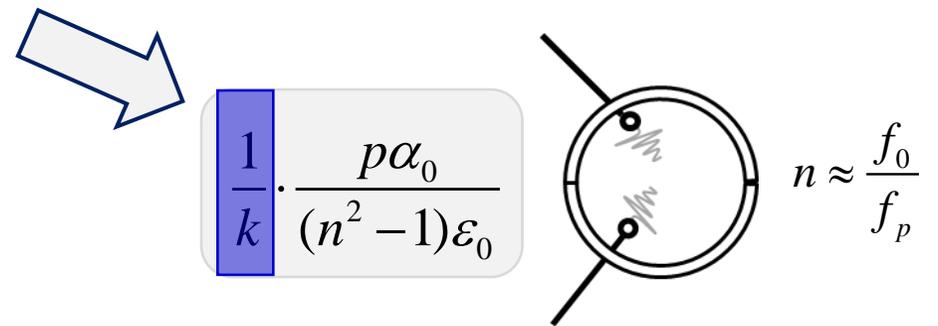
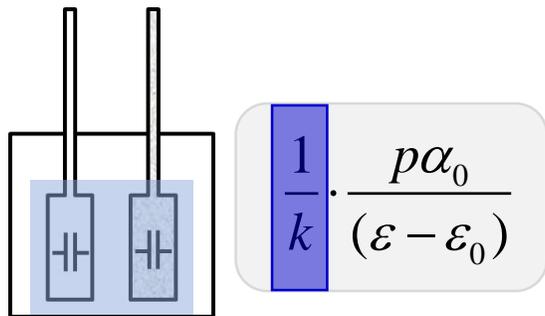
Constant Volume Gas Thermometry (**CVGT**)



Acoustic Gas Thermometry (**AGT**)



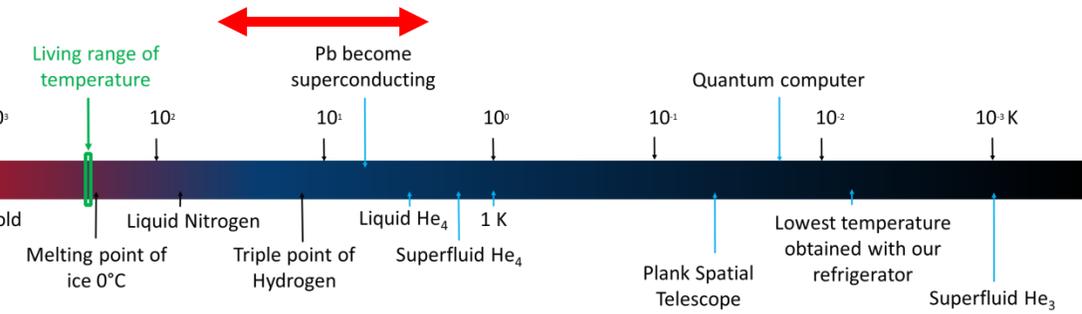
T



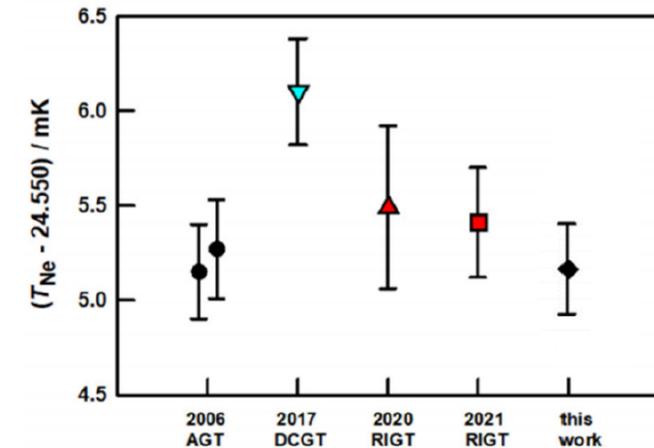
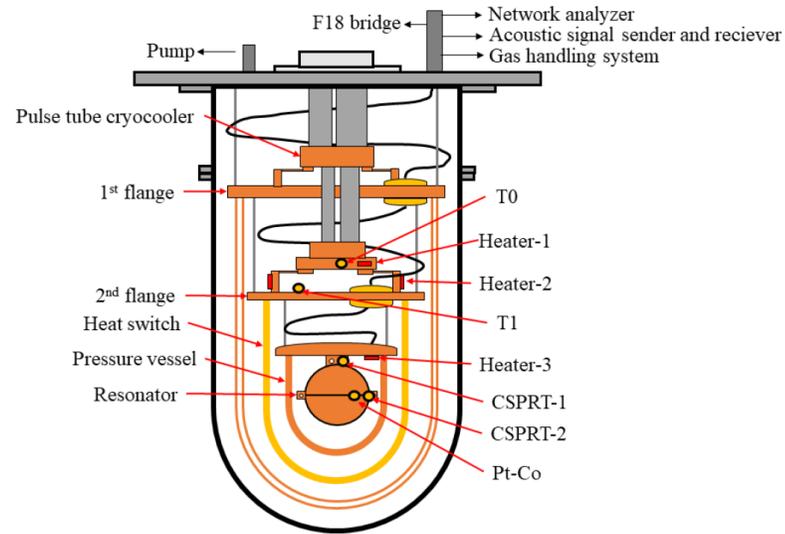
Dielectric-constant gas thermometry (**DCGT**)

Refractive-index gas thermometry (**RIGT**)

AGT and the world's first primary thermometry calibration at Low temperature



We used a quasi-spherical AGT to calibrate the Pt-Co RS144.08 resistance thermometer with the result:
 $(9.607\ 2558 \pm 0.000\ 006\ 3) \ \Omega$ at the thermodynamic temperature $T = (24.55499 \pm 0.000\ 16) \ \text{K}$



Changzhao Pan, Fernando Sparasci, Haiyang Zhang, Pascal Gambette, Mark Plimmer, Dario Imbraguglio, Roberto Maria Maria Gavioso, Michael R Moldover, Bo Gao, Laurent Pitre “Acoustic measurement of the triple point of neon TNe and thermodynamic calibration of a transfer standard for accurate cryogenic thermometer” 2021 Metrologia 58 045006

Does the relative primary thermometry is the solution?

Pro

- Pollution has minimal effect at temperatures below 25K.
- Gases with higher density yield stronger signals.
- Progress has been made in ab initio calculations.
- There is a well-established model in place.
- Analysis is straightforward.
- Measurements can be taken quickly.
- Uncertainty is equivalent to that of the reference temperature.

Con

- Hydrostatic correction must be determined.
- Perturbations from the shell are possible.
- Need to know the accommodation coefficient

Theory of SPRIGT

- Lorentz–Lorenz equation:

$$T \approx \frac{3A_\varepsilon p}{R[n^2(T, p) - 1]}$$

- If p is fixed at 2 temperatures:

$$\frac{T}{T_{\text{ref}}} \approx \frac{n^2(T_{\text{ref}}, p) - 1}{n^2(T, p) - 1}$$

- n^2 can be estimated by the ratio of resonance frequencies:

$$n^2(T, p) \approx \left(\frac{(f_m)_{T,0}}{(f_m)_{T,p}} \right)^2$$

1. Ratio is used to reduce the absolute pressure effect
2. Advance of the “*ab initio*” calculation of gas thermal-properties
3. High accuracy of microwave resonances

We need stable p , T and good measurement of f_m



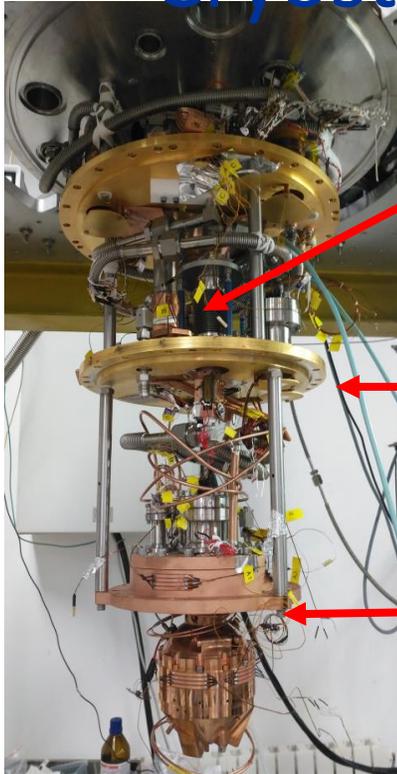
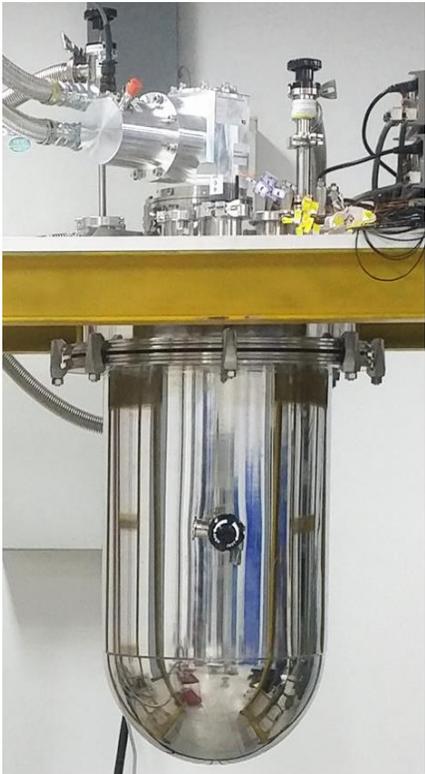
Bo Gao
(Bo)

Cryostat

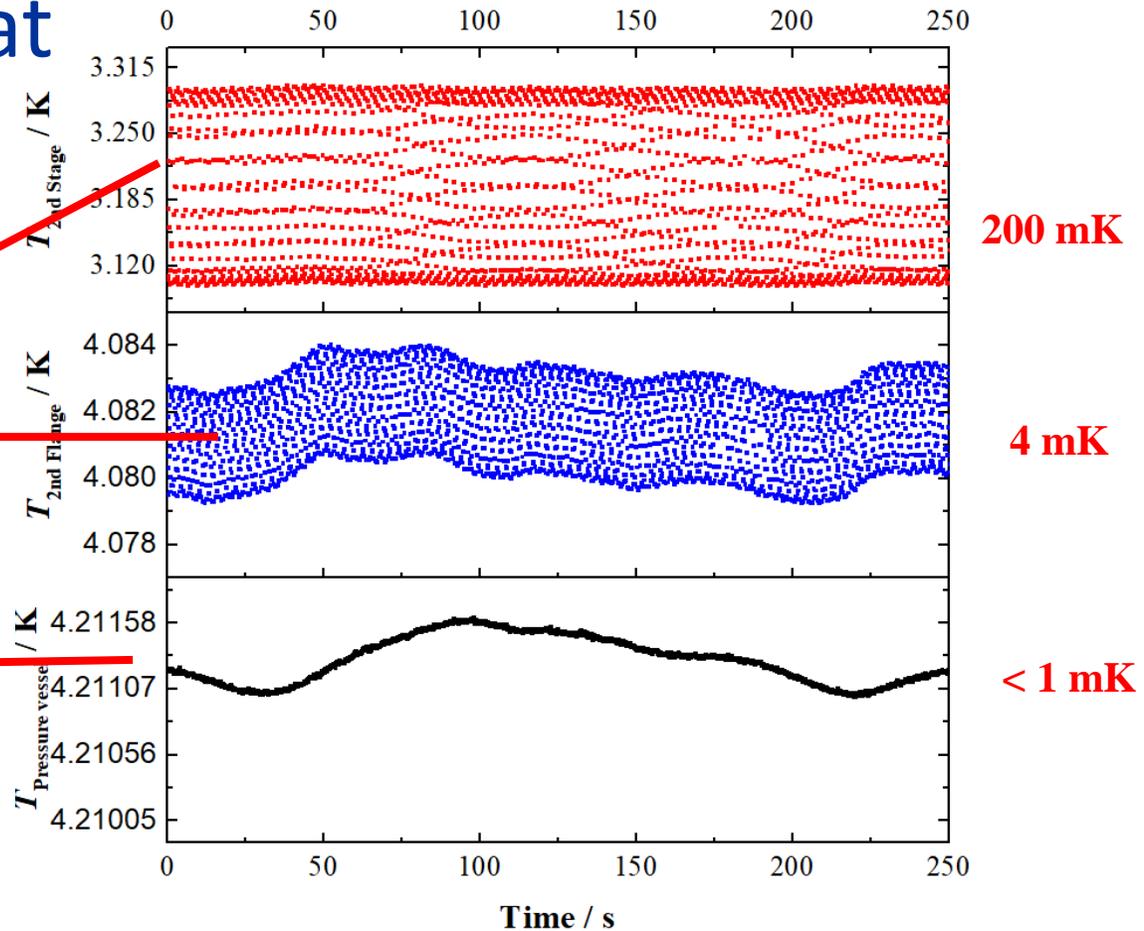
300K



5K



Cryostat

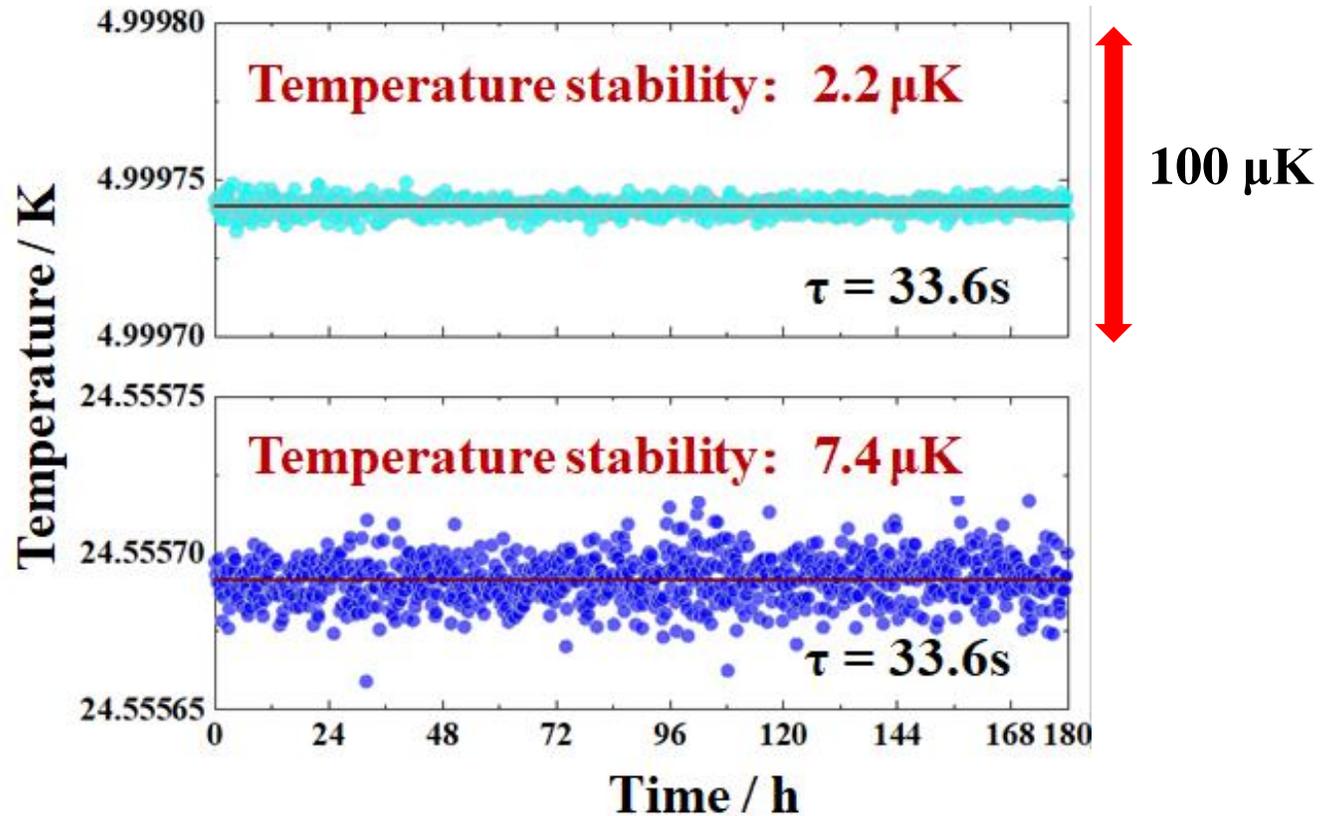


Temperature oscillation is reduced from 200 mK to < 1 mK with passive elements



Bo Gao
(Bo)

Temperature control in vacuum



1. Chinese SPRIGT realizes high temperature stability in the range of 5–25 K. *Science Bulletin*. 2018, 63(12), 733–734.
2. Realization of an ultra-high precision temperature control in a cryogen-free cryostat. *Review of Scientific Instruments*, 2018, 89, 104901
3. Thermal response characteristics of a SPRIGT primary thermometry system. *Cryogenics*. 2019, 97, 1-6.

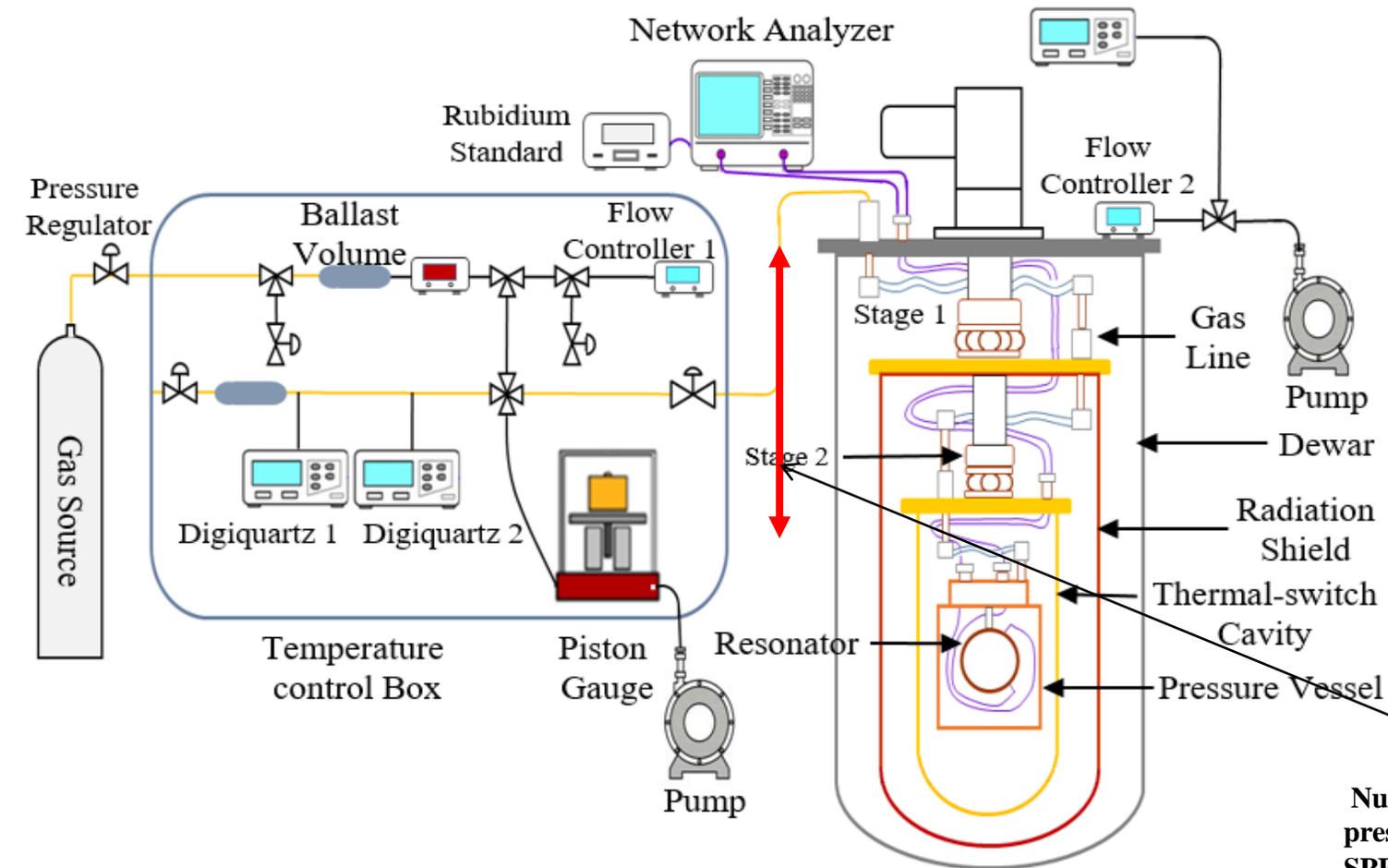
One-week stability is better than 8 μK in vacuum



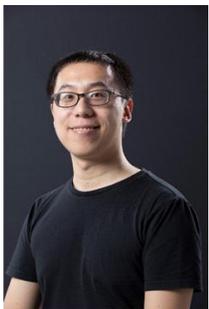
Bo Gao
(Bo)

SPRIGT system

Schematic diagram of SPRIGT Digiquartz 3



Numerical and experimental study of the hydrostatic pressure correction in 1 gas thermometry: A case in the SPRIGT. Changzhao Pan et al. *International Journal of Thermophysics* (2020) 41:108

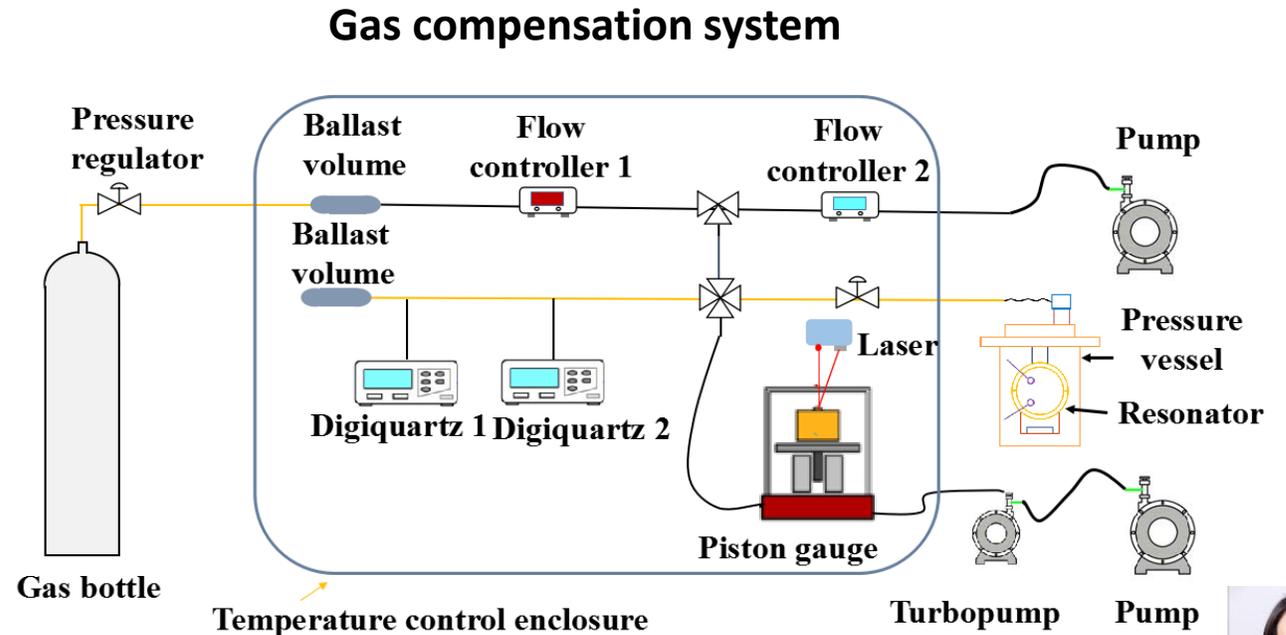
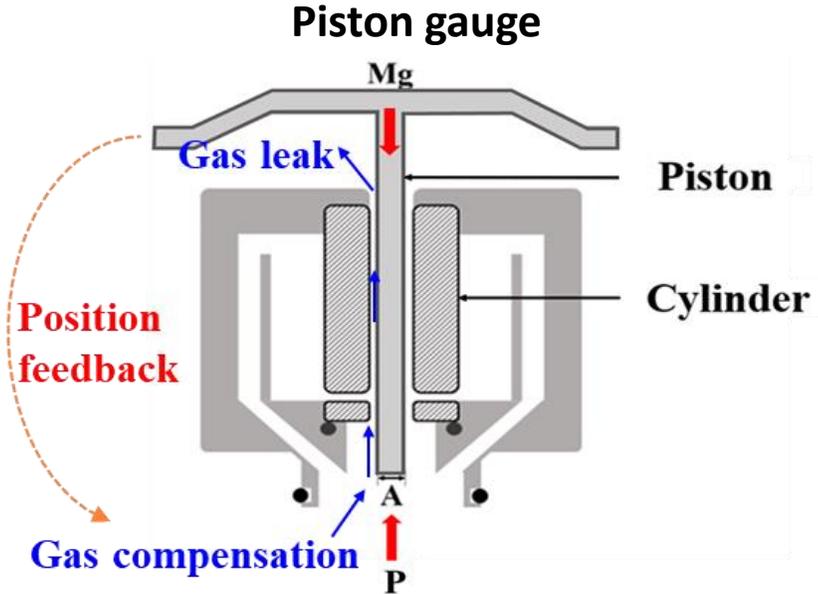


Changzhao Pan

Pressure control system

Challenge: The pressure cannot be controlled for a long time due to the piston falling caused by the **unavoidable leak**.

Solution: Gas compensation system



1. Ultra-stable pressure is realized for Chinese Single Pressure Refractive Index Gas Thermometry in the range 30 kPa to 90 kPa. *Science Bulletin*, 2018, 63:1601-1603.

This method can realize long term pressure stability

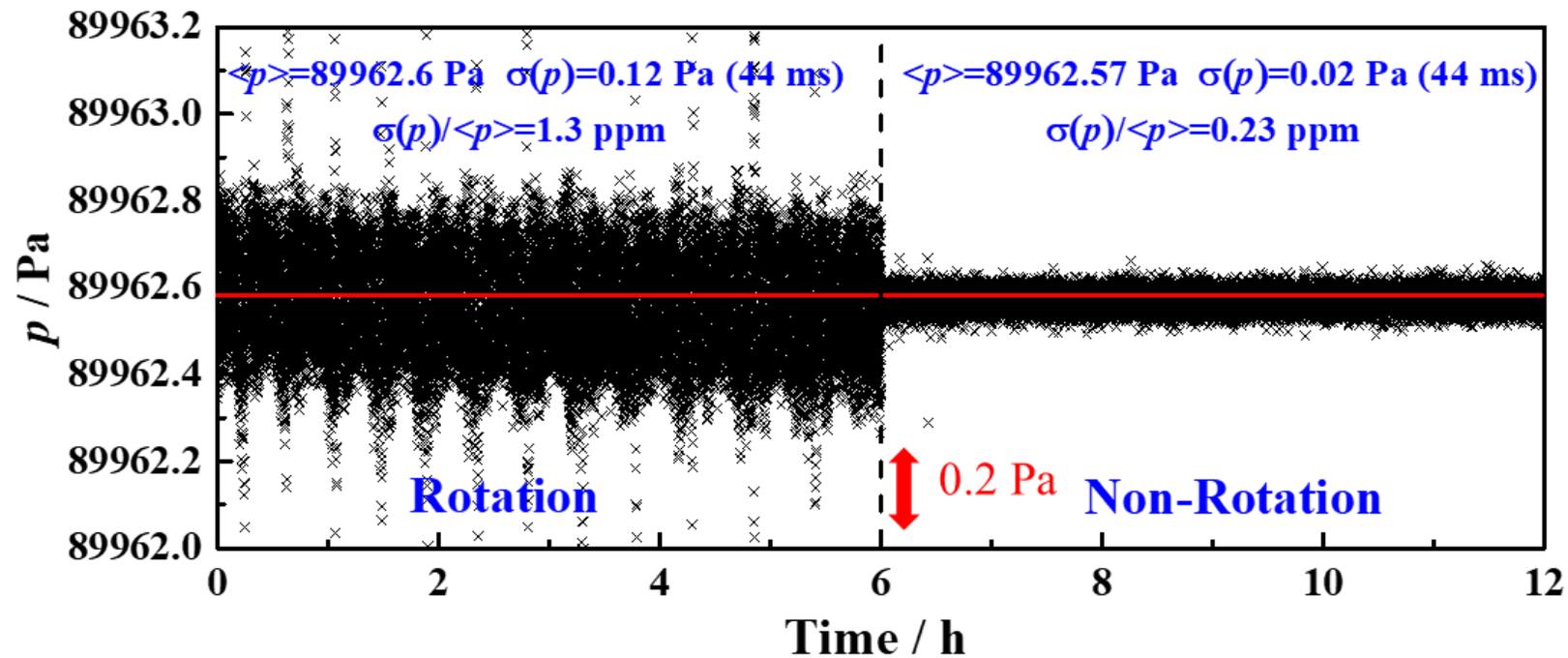


Bo Gao
(Bo)

Piston rotation

Challenge: The rotation makes the piston swing. It's hard to control the piston position.

Solution: Non-rotation

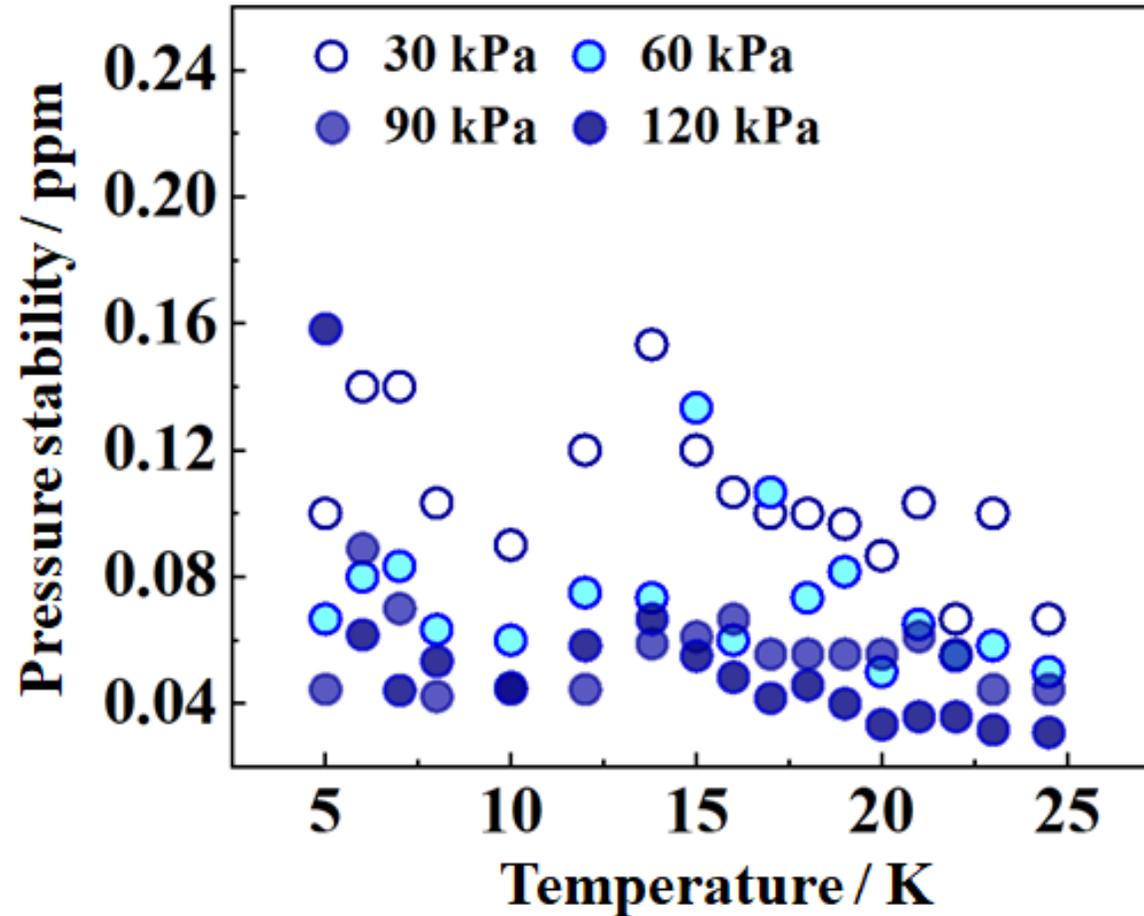


The pressure stability is improved without changing the mean value



Bo Gao
(Bo)

Connection to cryostat



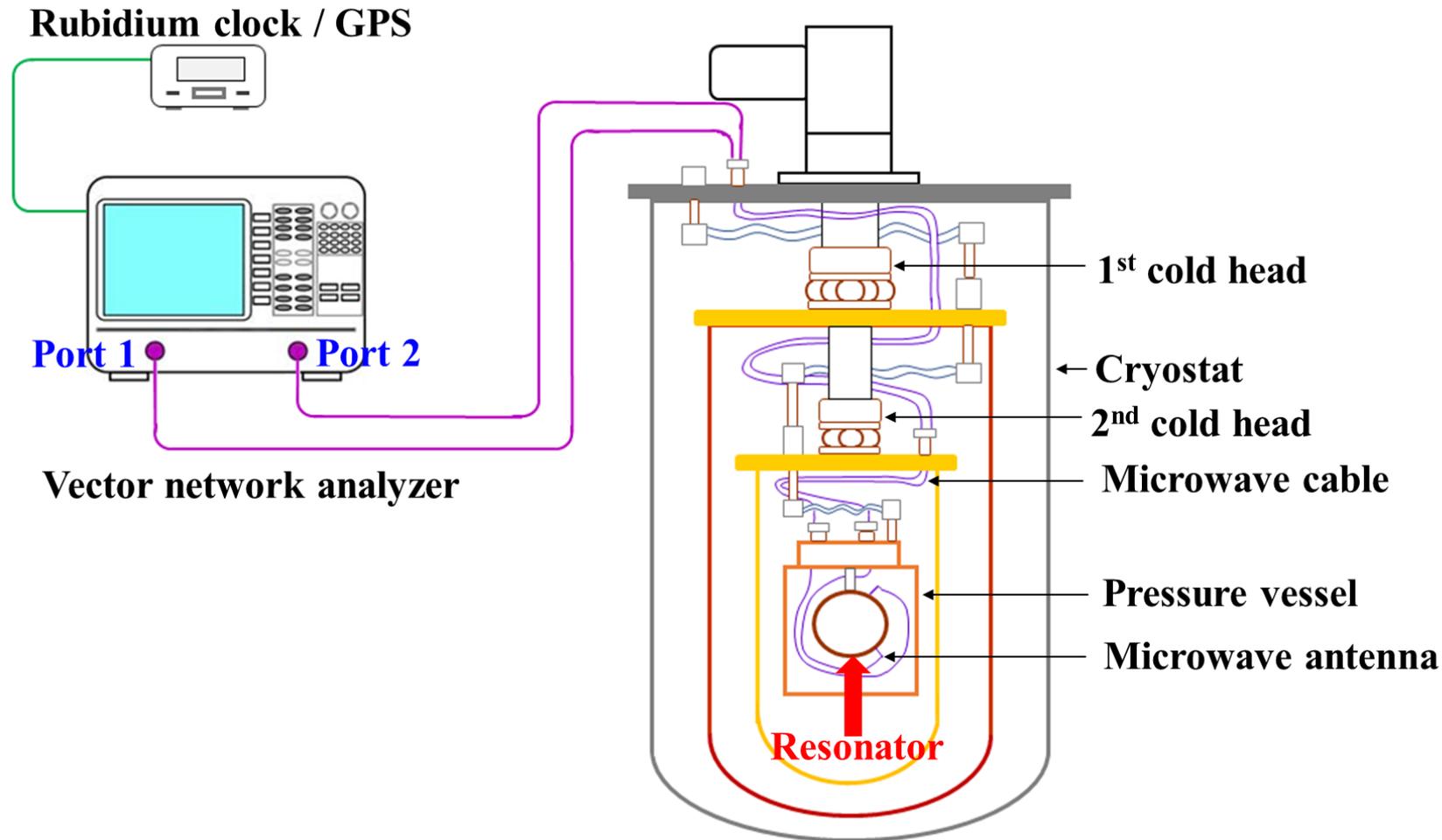
1. Realization of ppm level pressure stability for primary thermometry using a primary piston gauge, *Measurement*, 2020, 160: 107807.

High stability pressure (< 0.16 ppm) is realized from 5 K – 24.5 K



Bo Gao
(Bo)

Microwave measurement system

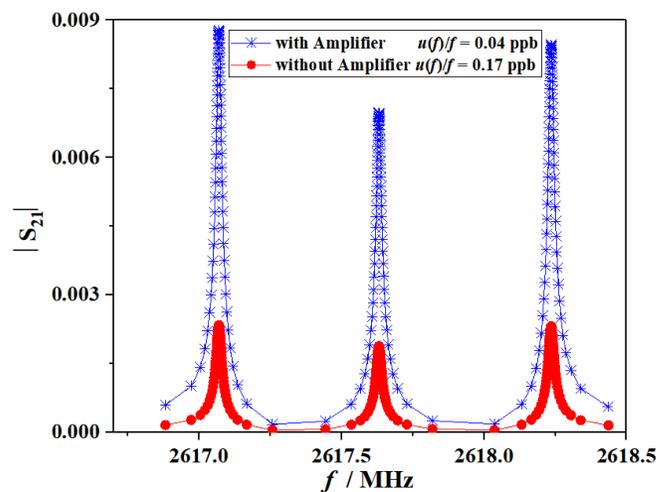


1. A high-stability quasi-spherical resonator in SPRIGT for microwave frequency measurements at low temperatures. *Science Bulletin*, 2019, 64:286-288.
2. Resonance frequency measurement with accuracy and stability at the 10^{-12} level in a copper microwave cavity below 26 K by experimental optimization, *Measurement Science and Technology*, 2020, 31: 075011.

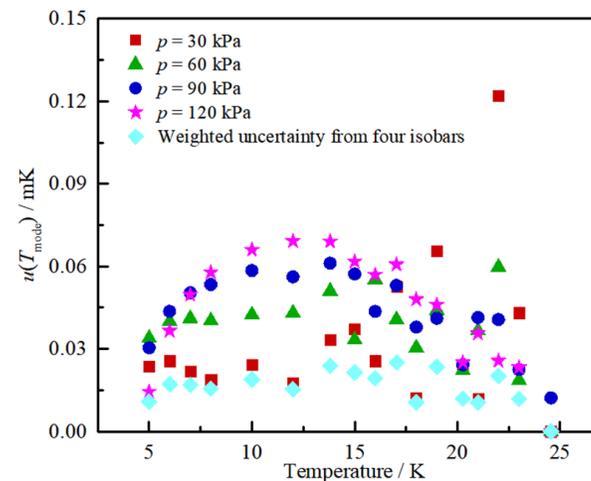


Bo Gao
(Bo)

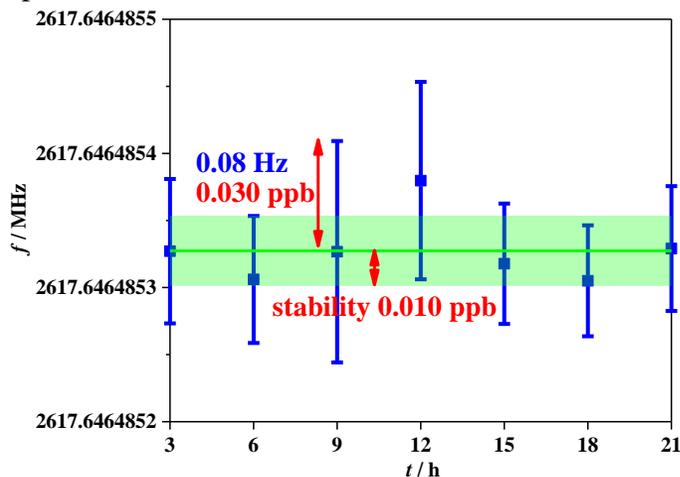
Experimental results



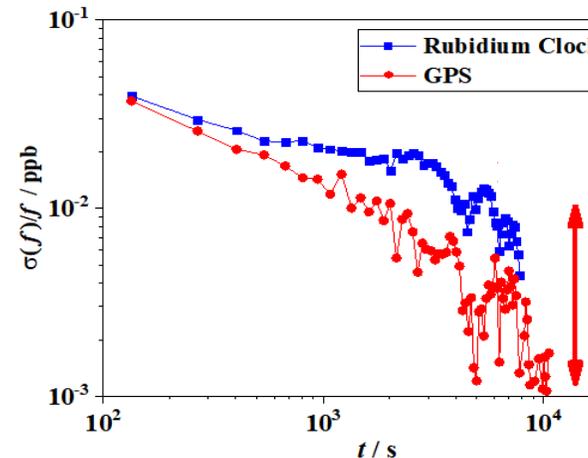
(a) S21 parameter of TM11 mode at 5 K with and without amplifier.



(b) Thermodynamic temperature difference from TM11, TE11 and TE13 modes at 30-120kPa.



(c) High-accuracy measurement on TM11 at 8 K under vacuum.



(d) Allen variance of TM11 mode at 5 K with FS725 and GPS.

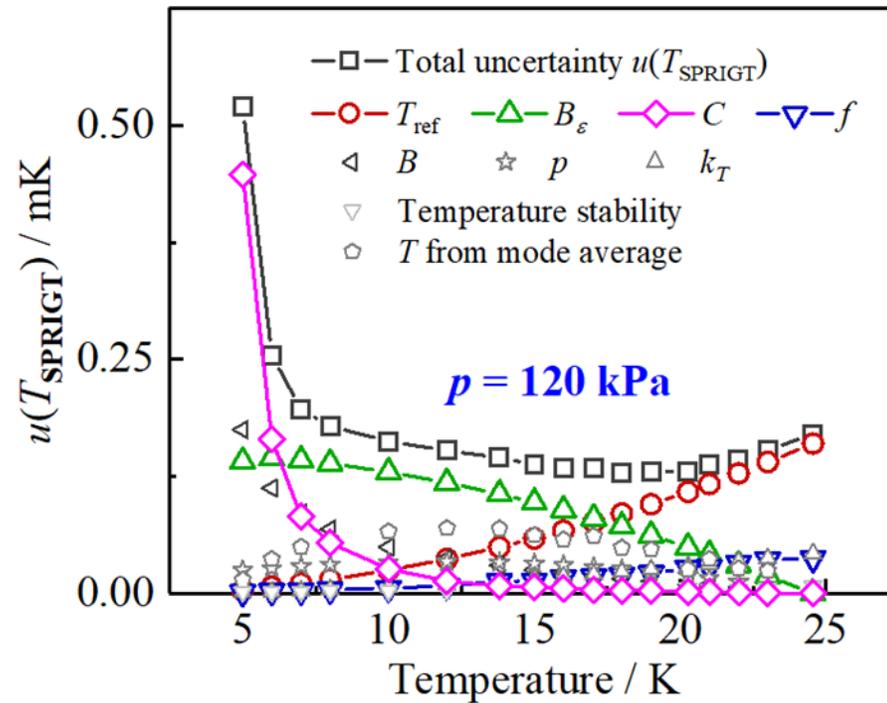
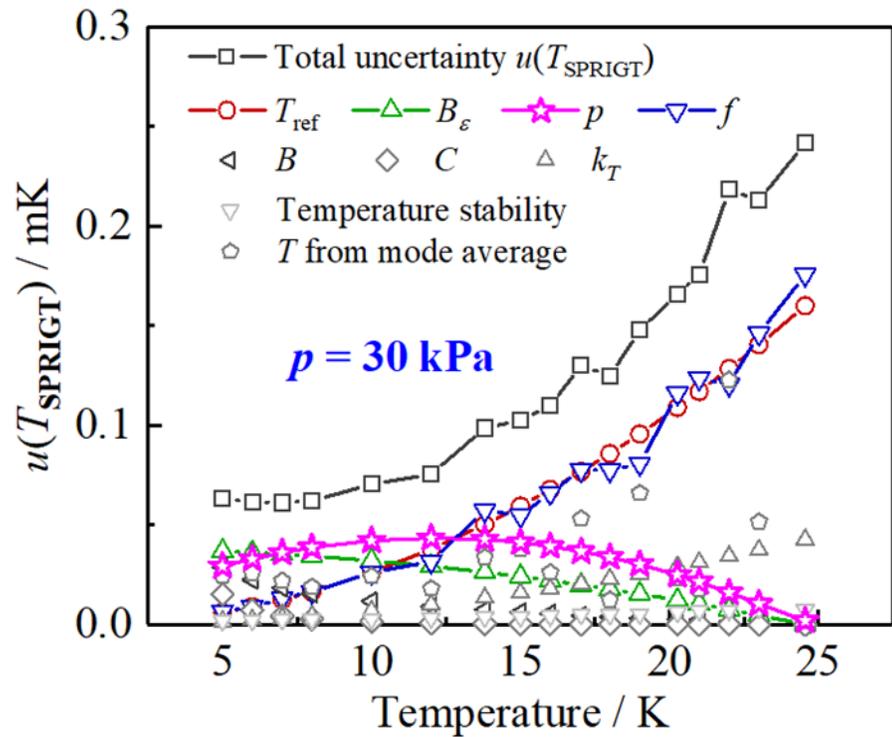
in practice < 0.01 ppb, good mode consistency on T_{SPRIGT}

10ppt!!!
11 Digits



Bo Gao
(Bo)

Uncertainty budget

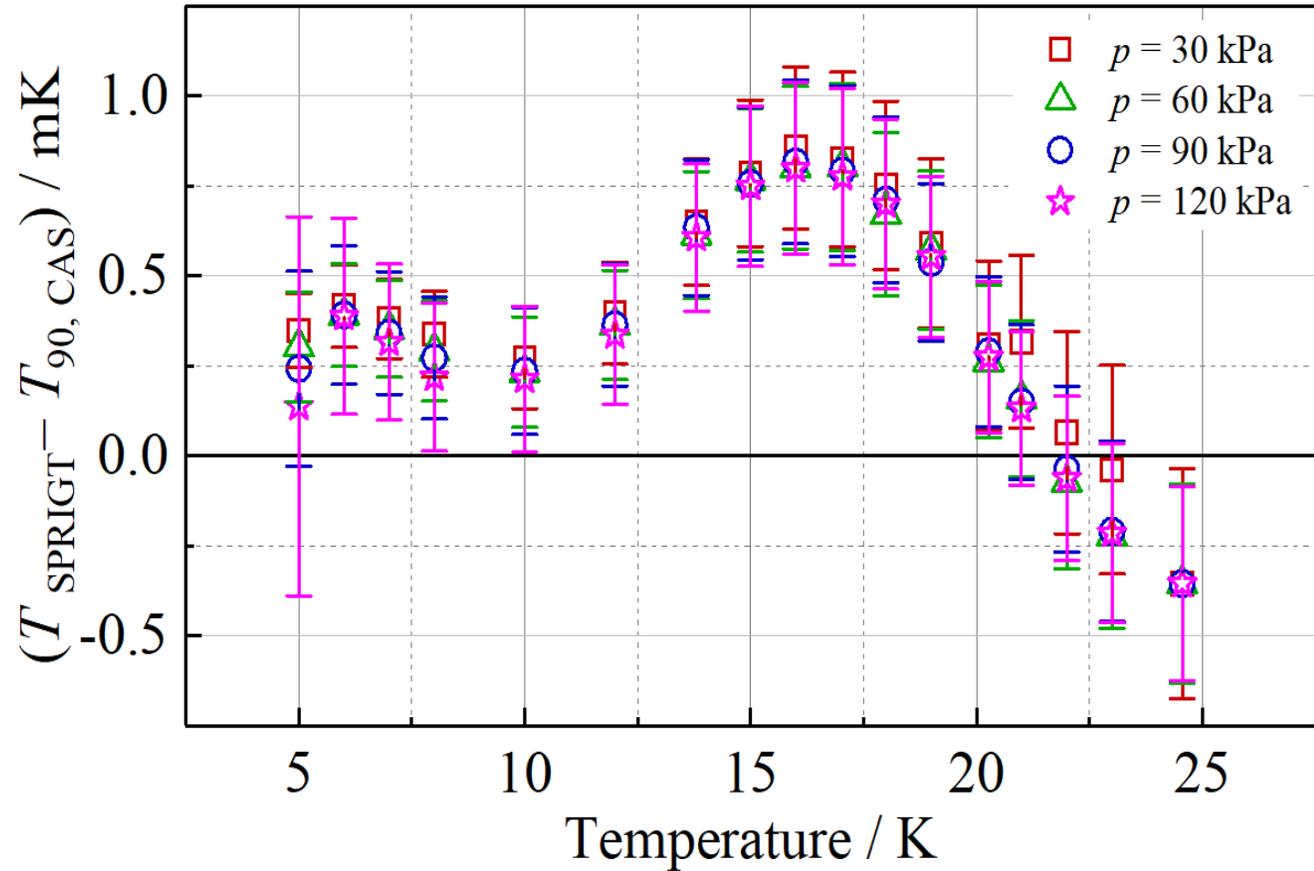


Main uncertainty components: T_{ref} and C



Bo Gao
(Bo)

T-T90 at 4 pressures

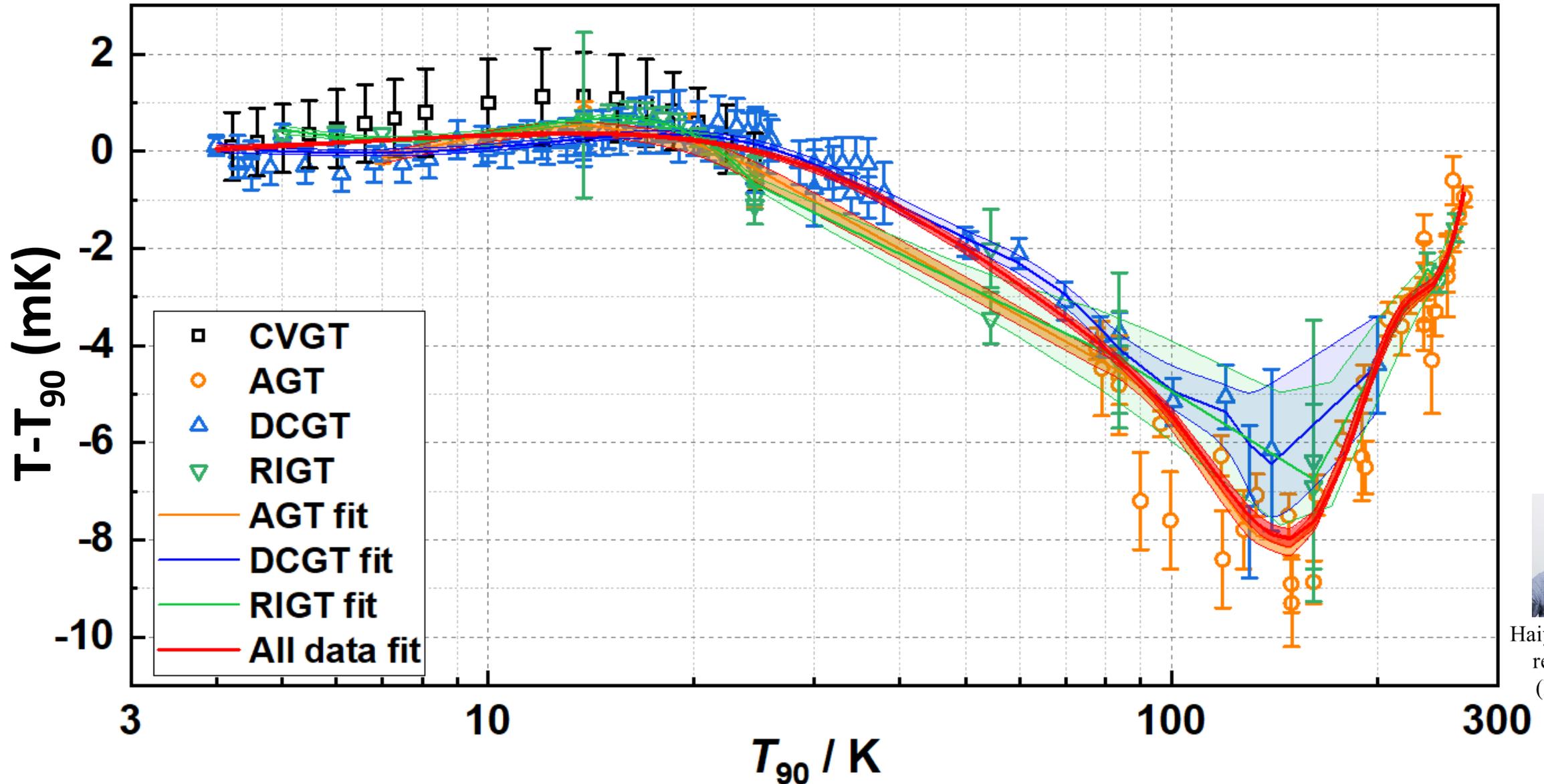


Different pressures give same $T-T_{90}$, Good repeatability and consistency



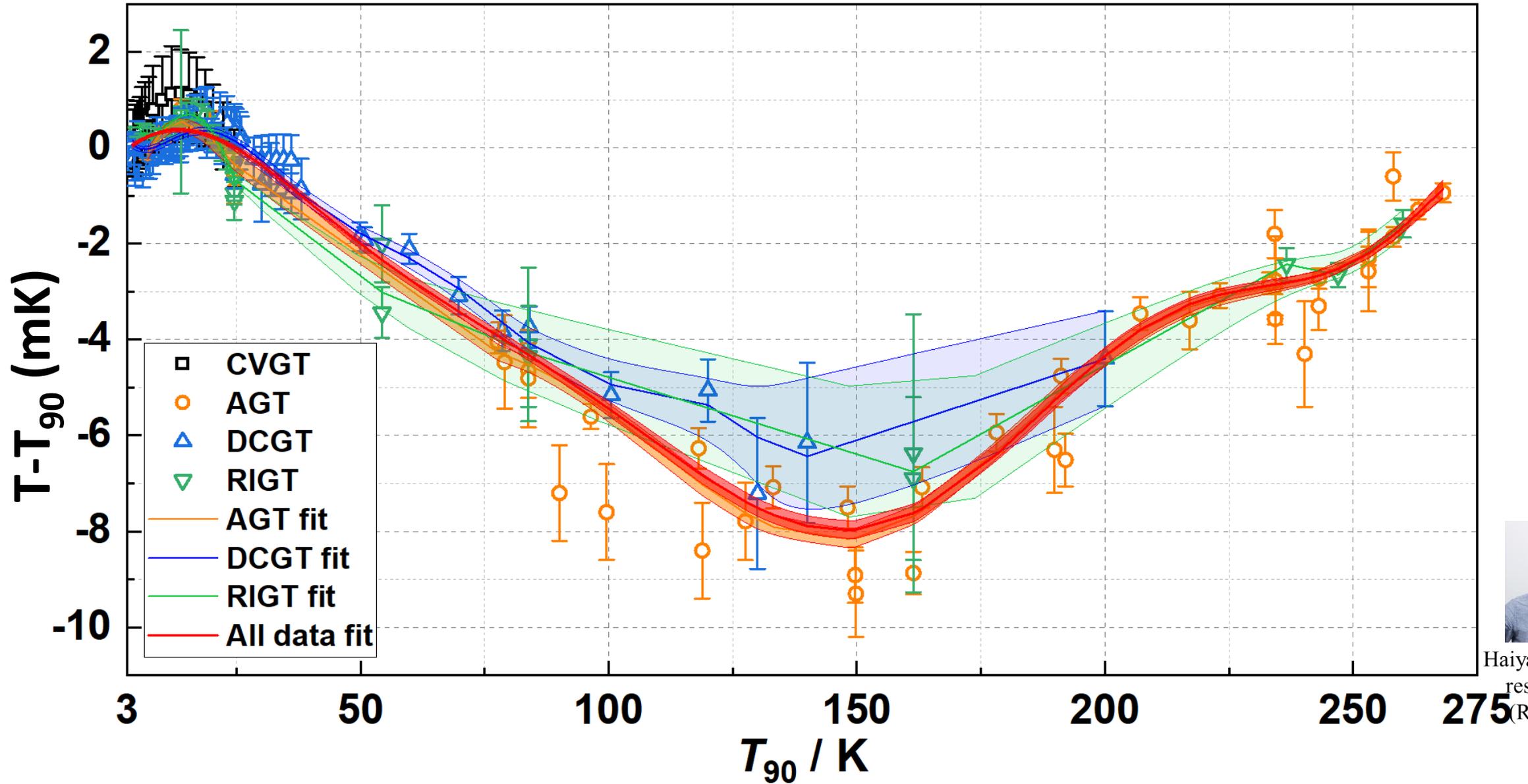
Bo Gao
(Bo)

The overall fit and separate fit of different primary thermometry



Haiyang Zhang
researcher
(Richard)

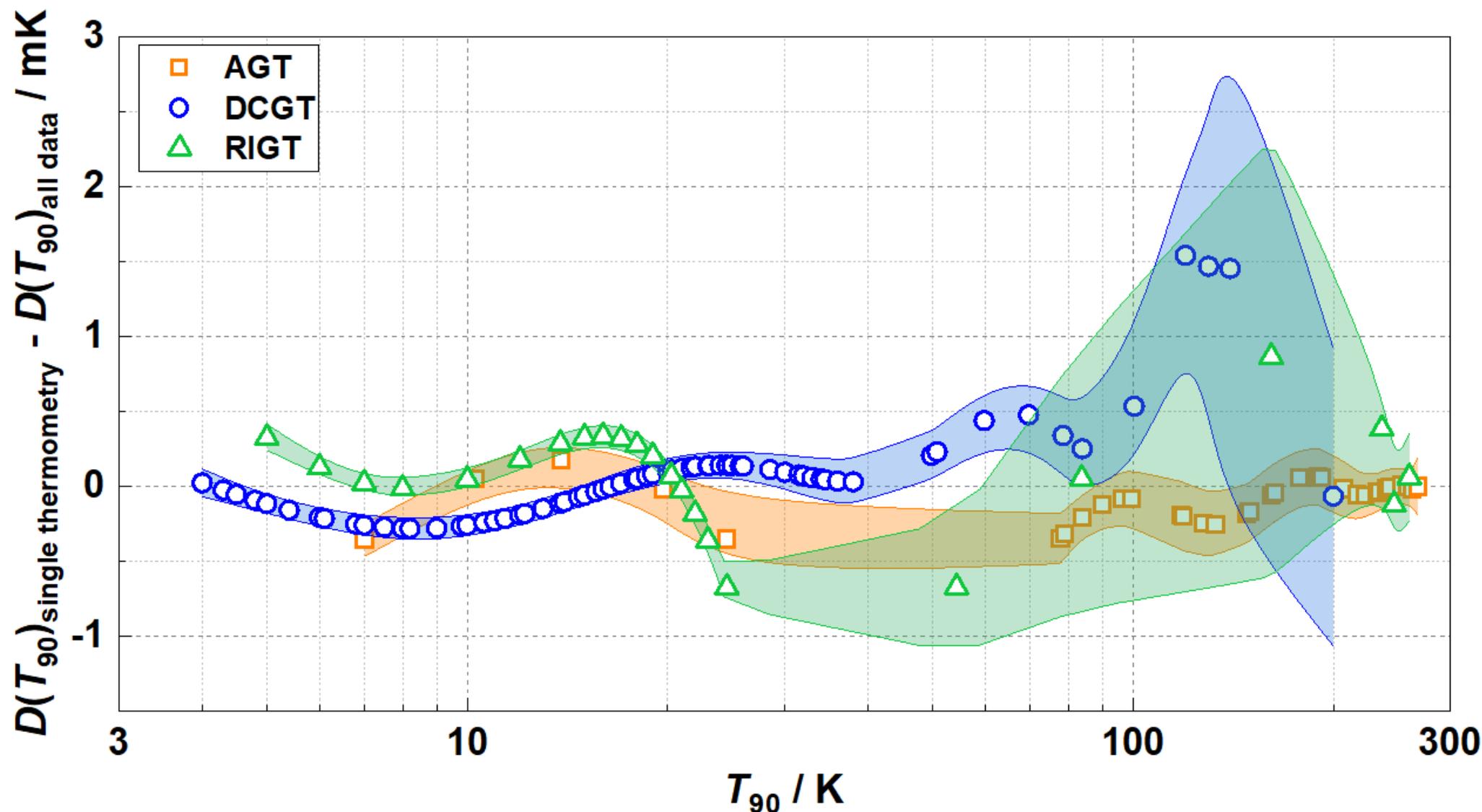
The overall fit and separate fit of different primary thermometry



Haiyang Zhang
researcher
(Richard)

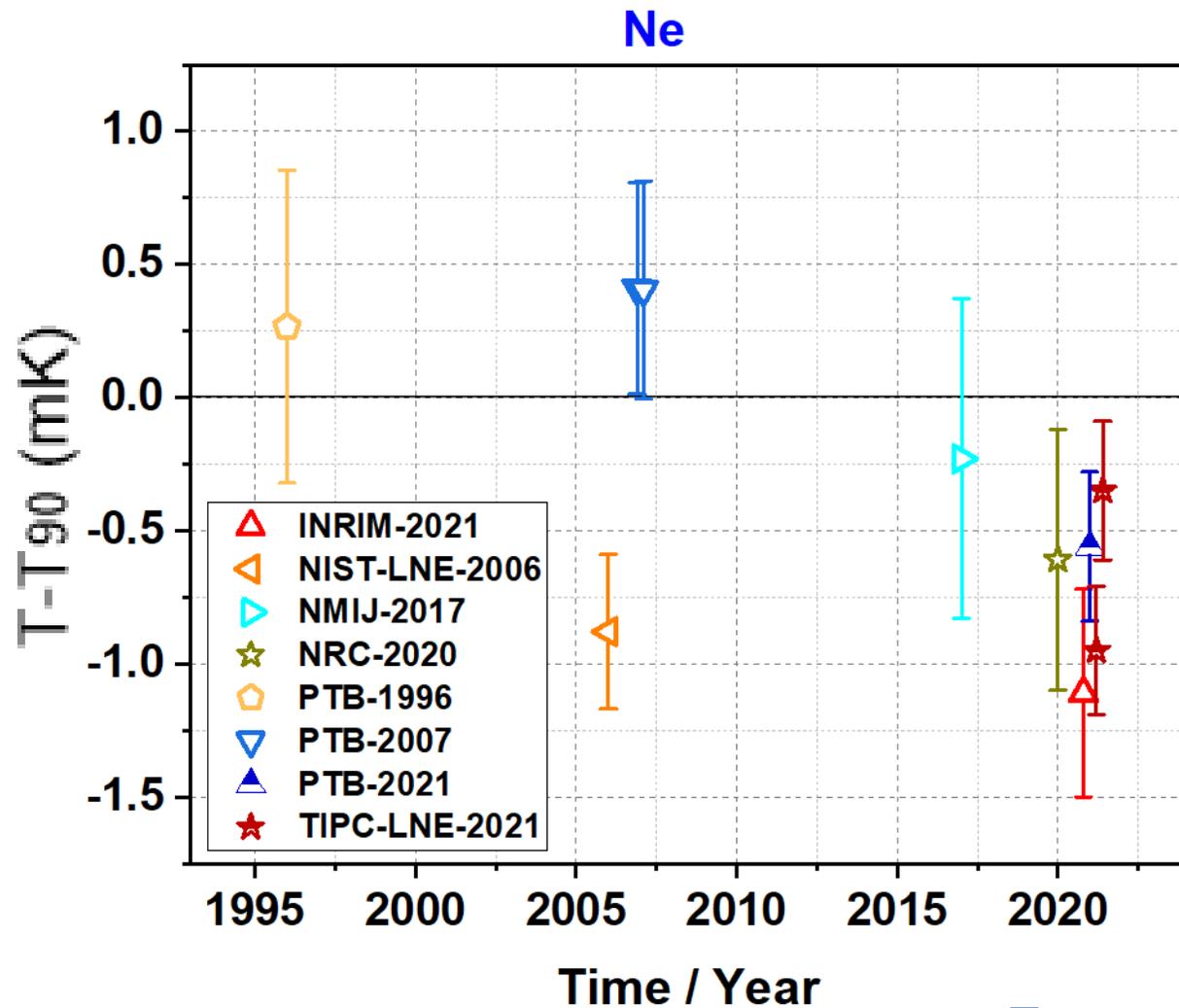
Does the primary thermometry give the same T? Yes

The overall fit and separate fit of different thermometry



Haiyang Zhang
researcher
(Richard)

Measurements at around Ne fixed point, chronologically



	<i>T</i> ₉₀ points
INRIM-2021	24.5561
NIST-LNE-2006	24.551
NMIJ-2017	24.5559
PTB-1996	24.6
PTB-2008	24, 25
PTB-2021	24.55518
TIPC-LNE-2021	24.5561, 24.55535
NRC-2020	24.5561

European project: DireK-T

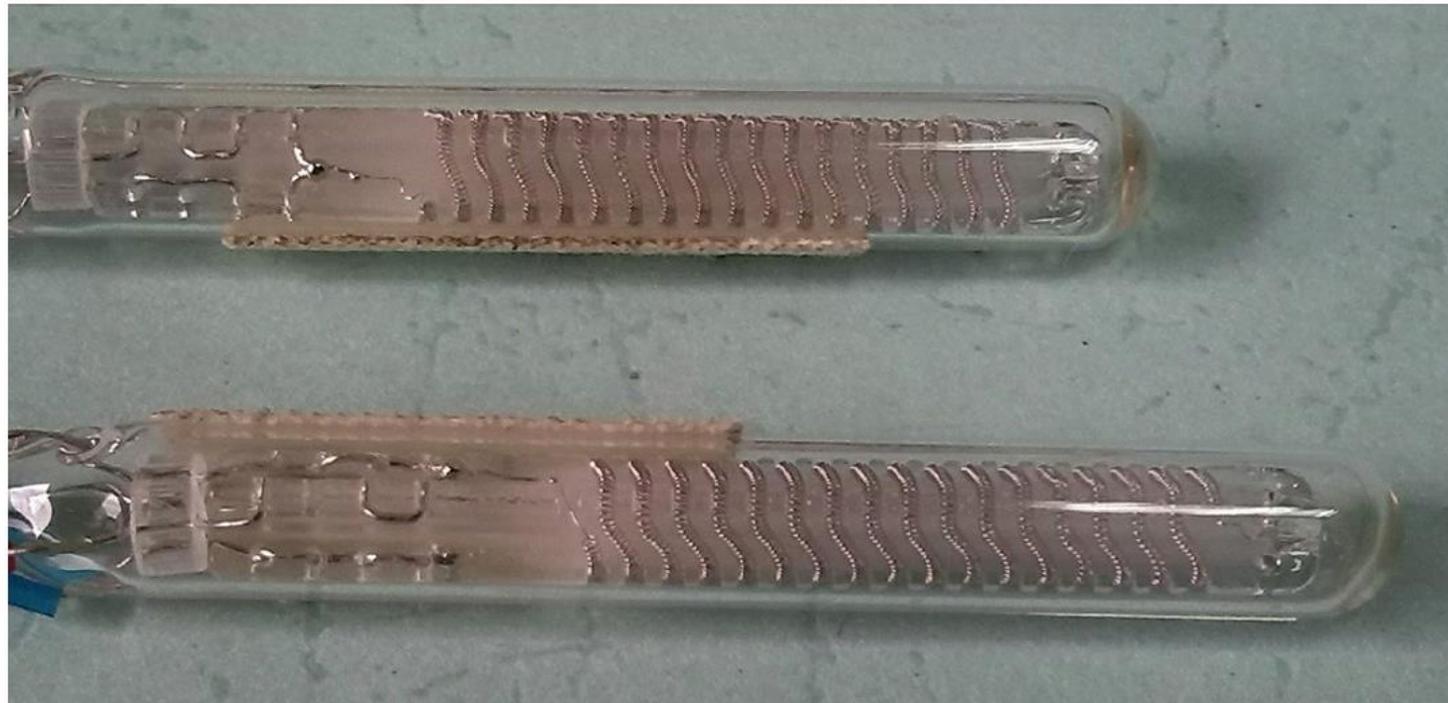
From T90, how to get T?

Visit the BIPM website and search for 'T-T90';
you'll find the last equation there.

Personally, I disagree with the 2022 equation (I believe tables one and two should not be averaged). I couldn't convince my colleagues, and I have requested that my name be removed from the paper.

RdFe thermometer from Yunnan Dafan Meter Industrial

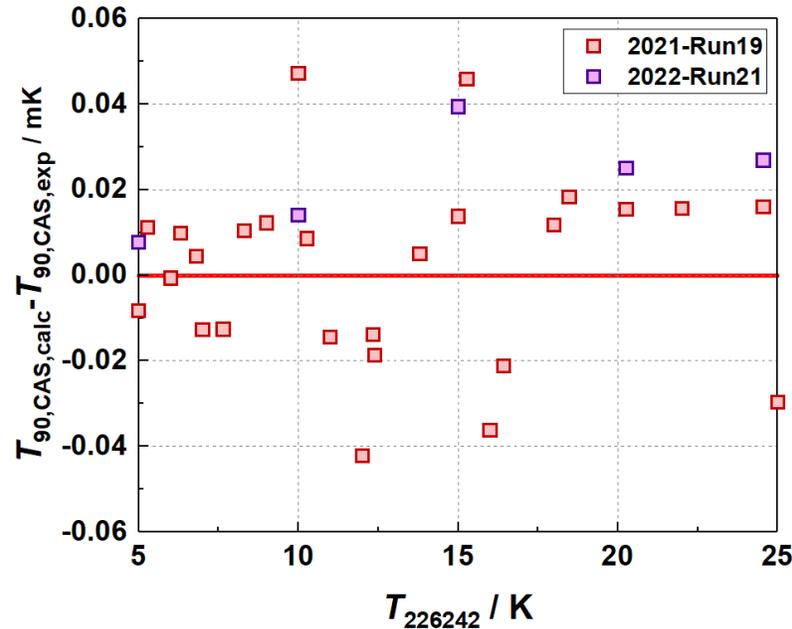
The reference standard for the range of 0.65 to 25 K. Only one company continues to produce reference standard RdFe



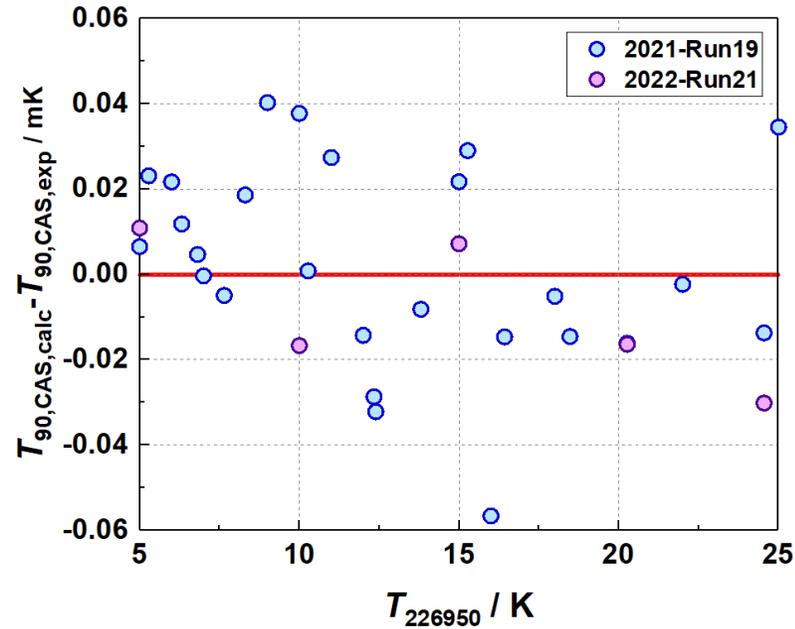
Maintaining T_{90} with RdFe

□ Comparison of experimental T_{90} over a 2,5 years

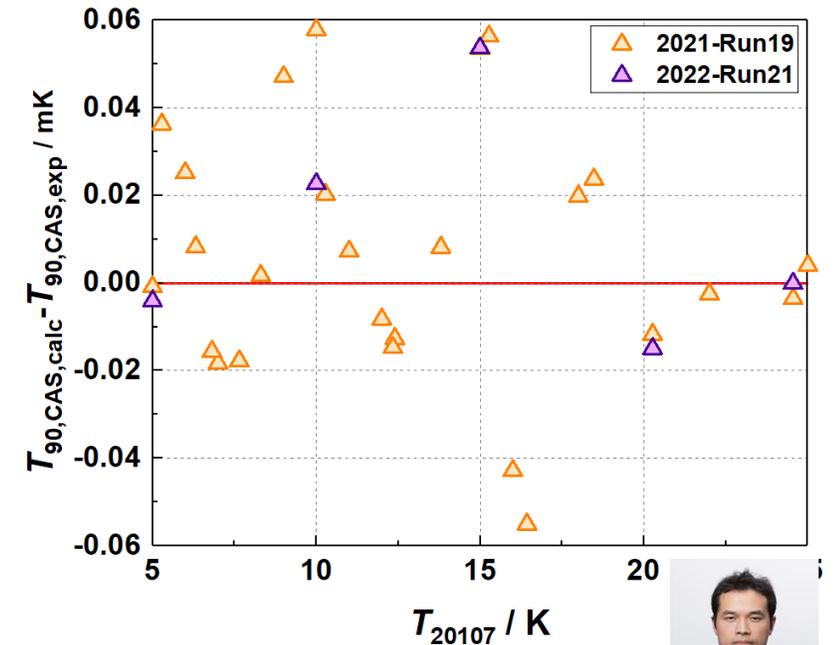
226242, $\pm 0.042\text{mK}$



226950, $\pm 0.056\text{mK}$



20107, $\pm 0.058\text{mK}$



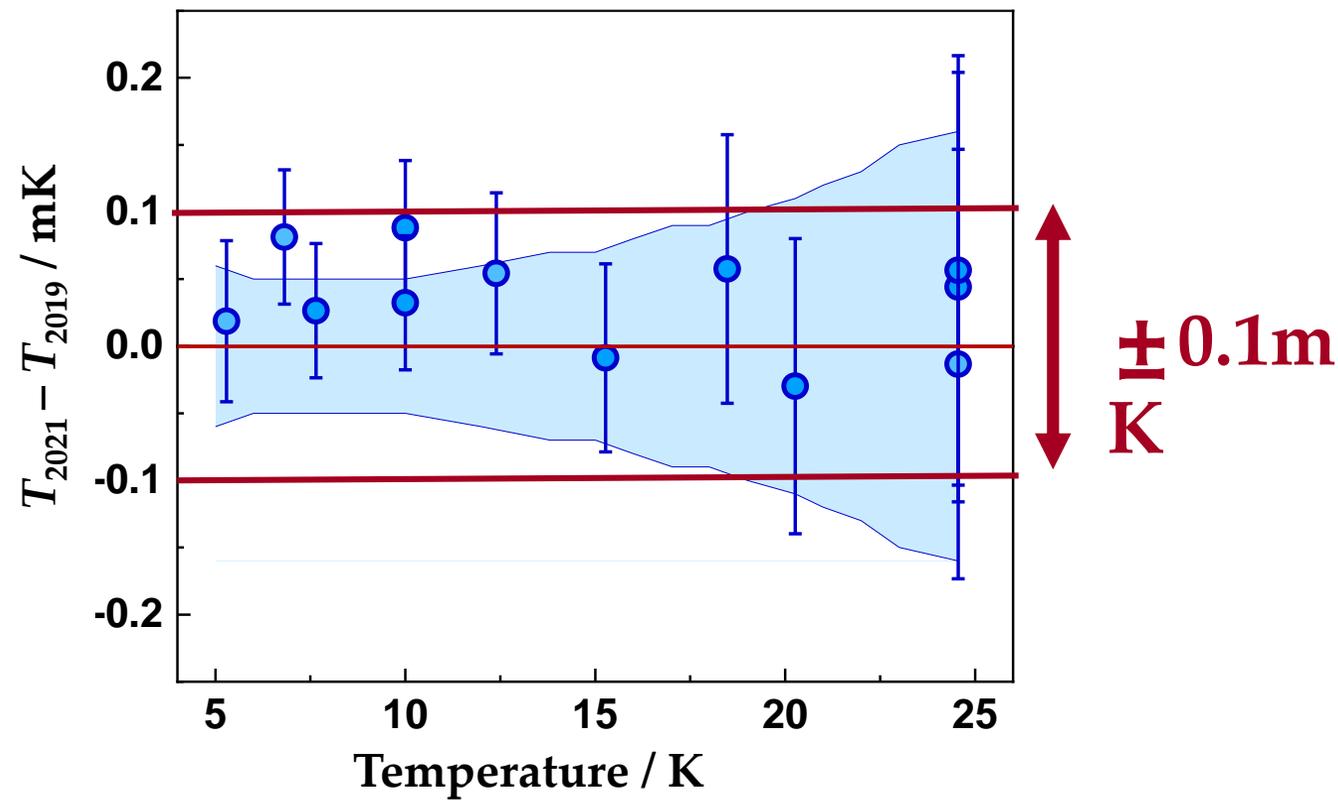
T_{90} reproducibility within $\pm 0.058\text{mK}$
Stability better than 0.028mK



Haiyang Zhang
researcher
(Richard)

Stability of SPRIGT

- After 2.5 years, the difference of T in the two rounds of experiments ($T_{2021} - T_{2019}$)

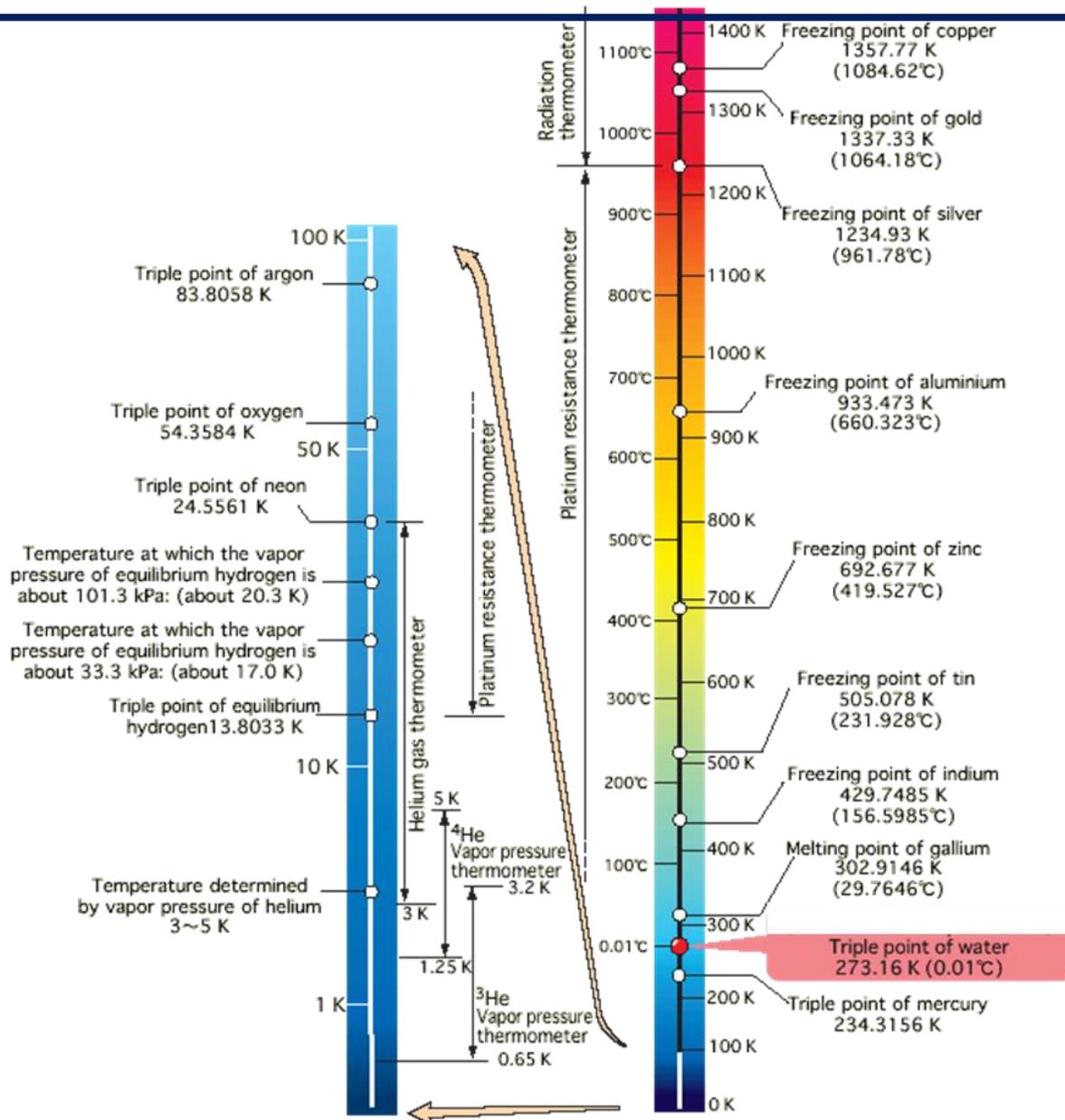


T_{SPRIGT} has a stability better than 0.1mK



Haiyang Zhang
researcher
(Richard)

International Temperature Scale 1990



ITS90 is an approximation of the thermodynamic temperature
 From **13,8K to 273,16 K** IST 90 give:
 A thermometer
 Fix point
 And interpolating equation

The Pt thermometer is at its beginning

Platinum Resistive thermometer is since 1887 and in metrology 1923

Concept by Siemens and Implementation by Callendar in 1887

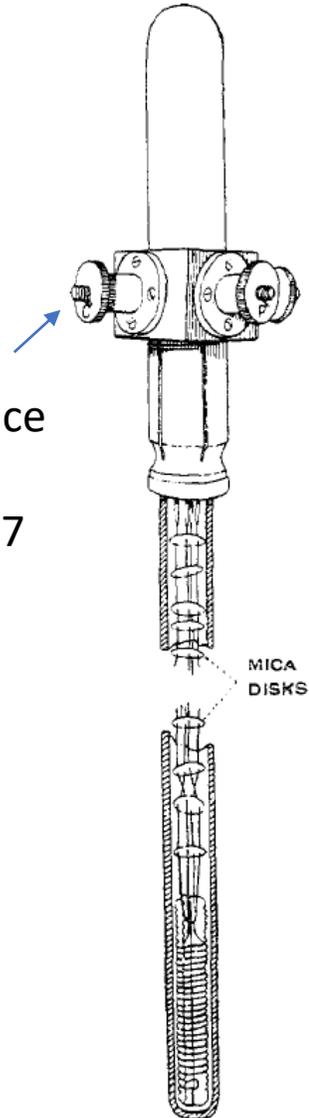
Progress in platinum resistance thermometry

C R Barber¹ and J A Hall¹

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[British Journal of Applied Physics, Volume 13, Number 4](#)

4-wire resistance measurement method in 1887

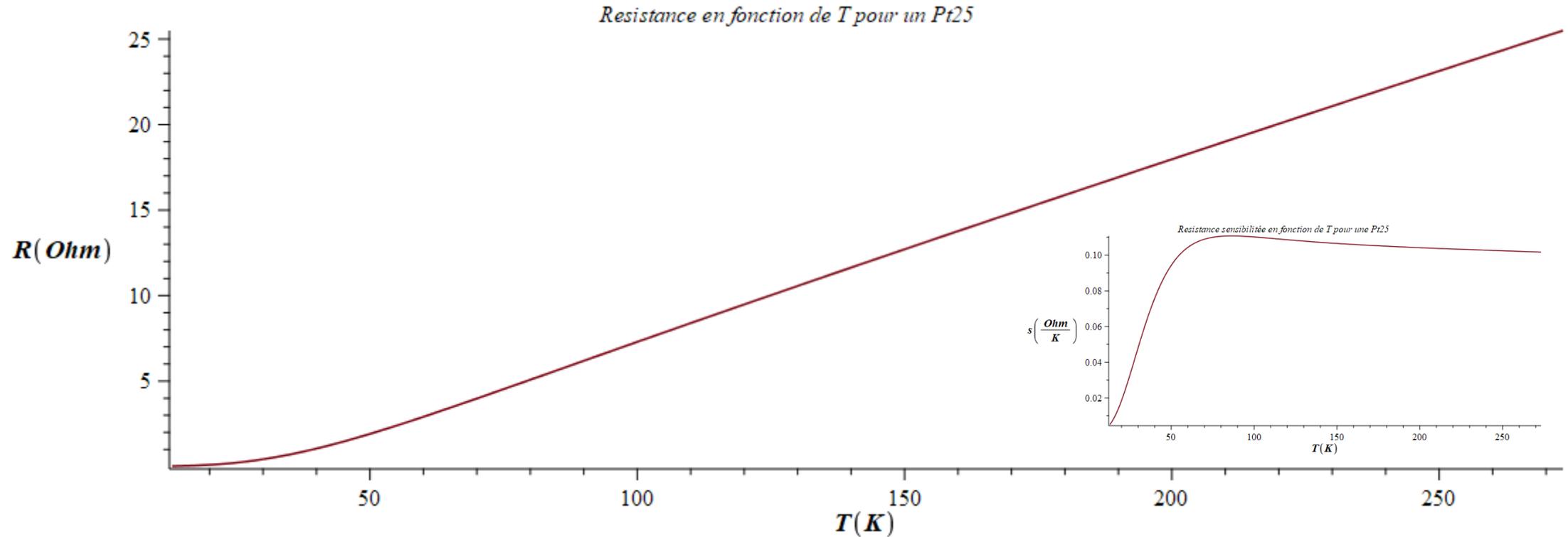


From **A short history of temperature scales**
Terry Quinn*

“During these 1923 discussions the form of the future scale was agreed. It would comprise the platinum resistance thermometer from $-38.81\text{ }^{\circ}\text{C}$ up to the freezing point of aluminium at $650\text{ }^{\circ}\text{C}$ ”

Figure 2. Callendar's design of thermometer.

Pt25 thermometer curve



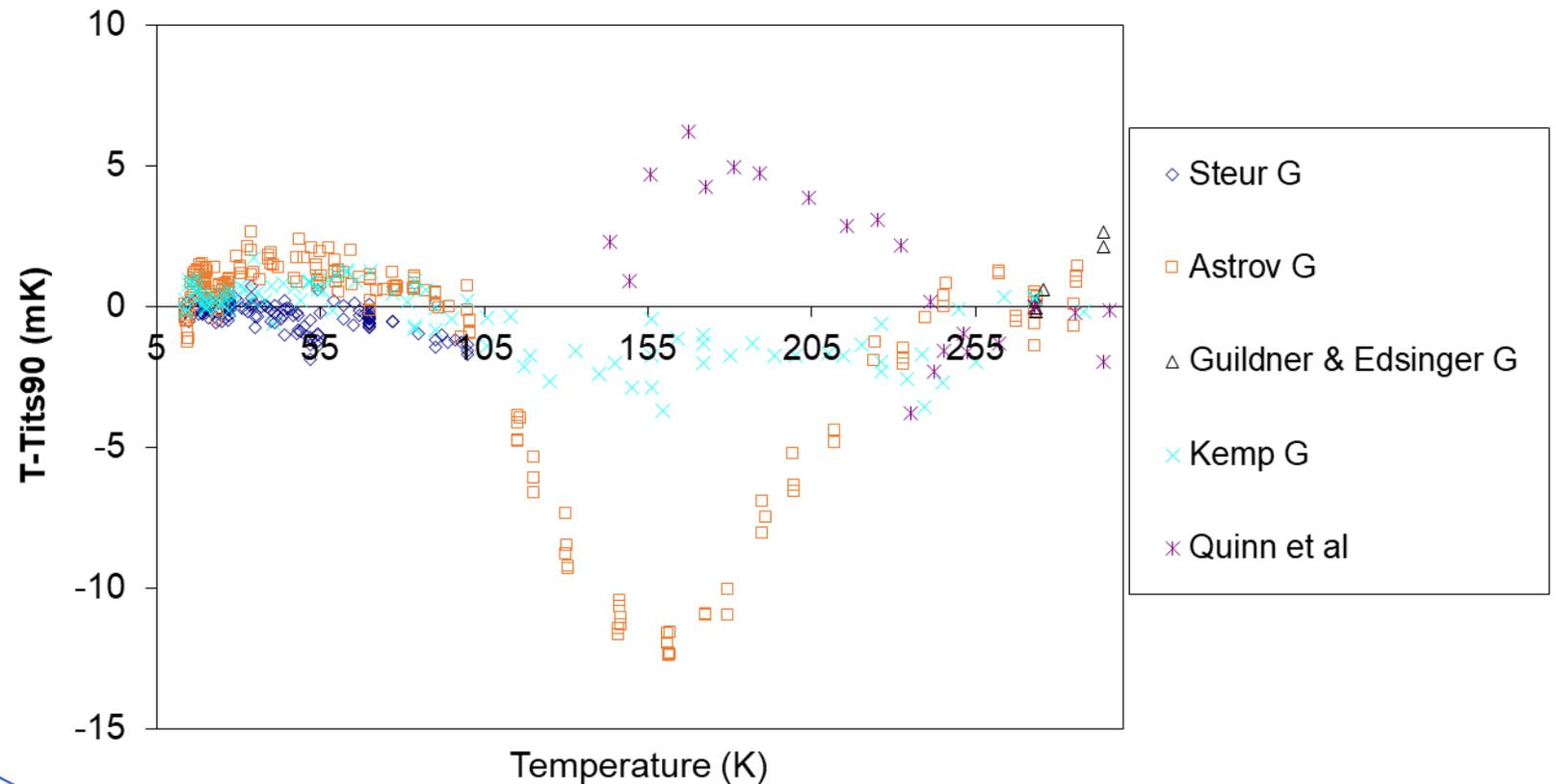
$$W = \frac{R(T)}{R(273.16)}$$

$$\ln[W_r(T_{90})] \equiv A_0 + \sum_{i=1}^{12} A_i \left[\frac{\ln(T_{90}/273.16 \text{ K}) + 1.5}{1.5} \right]^i$$

$$13.8033 \text{ K} \leq T_{90} \leq 273.16 \text{ K},$$

Origine of the ITS90 reference function

Differences between published thermodynamic temperature determinations and the ITS-90 in the range 4K-300K

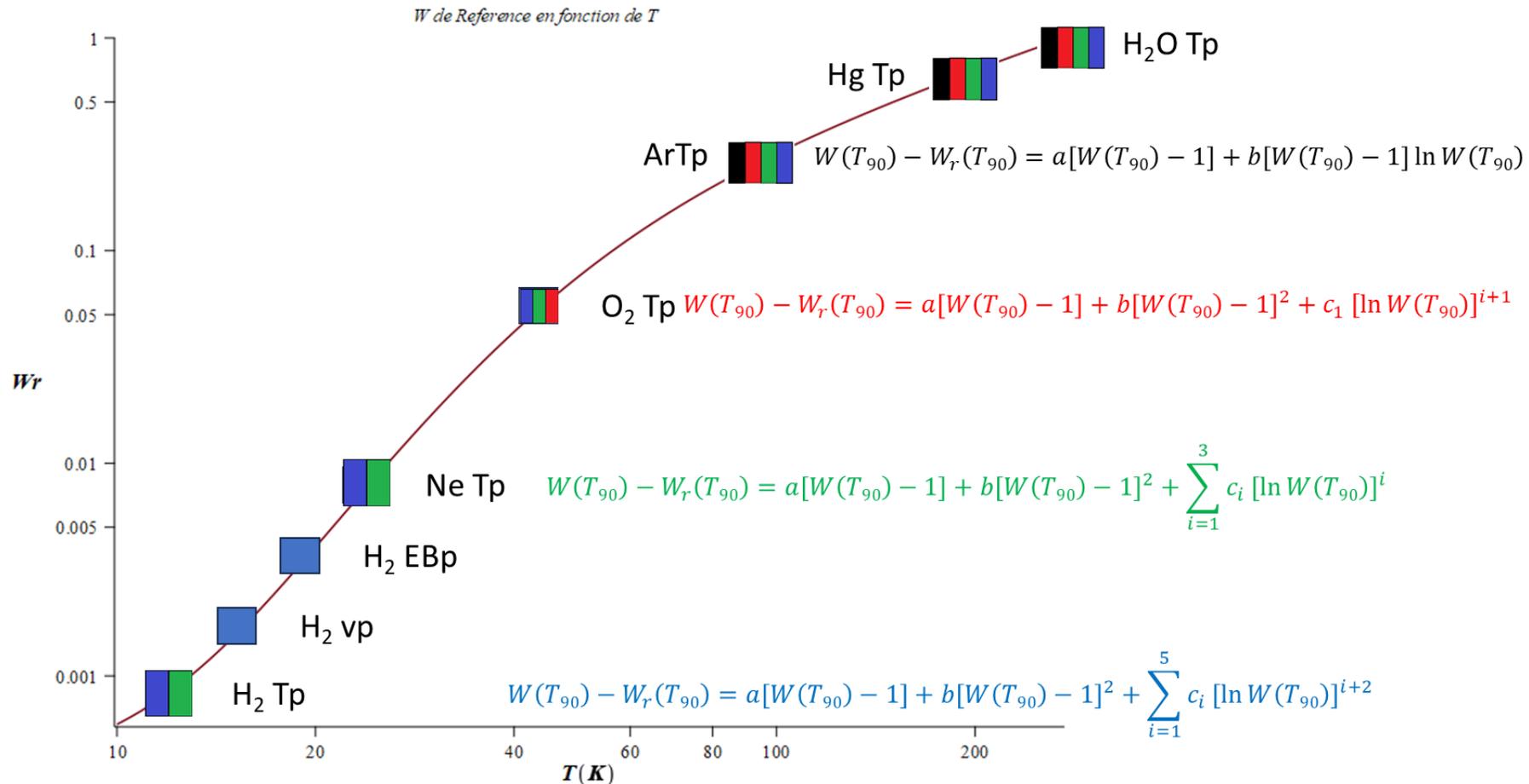


It is the result of a fit from the best known relationship at that time (before 1990) between some resistance of CSPRPT25 and the most accurate understanding of thermodynamic temperature.

$$\ln[W_r(T_{90})] \equiv A_0 + \sum_{i=1}^{12} A_i \left[\frac{\ln(T_{90} / 273.16 \text{ K}) + 1.5}{1.5} \right]^i \quad 13.8033 \text{ K} \leq T_{90} \leq 273.16 \text{ K},$$

ITS90 reference and deviation function

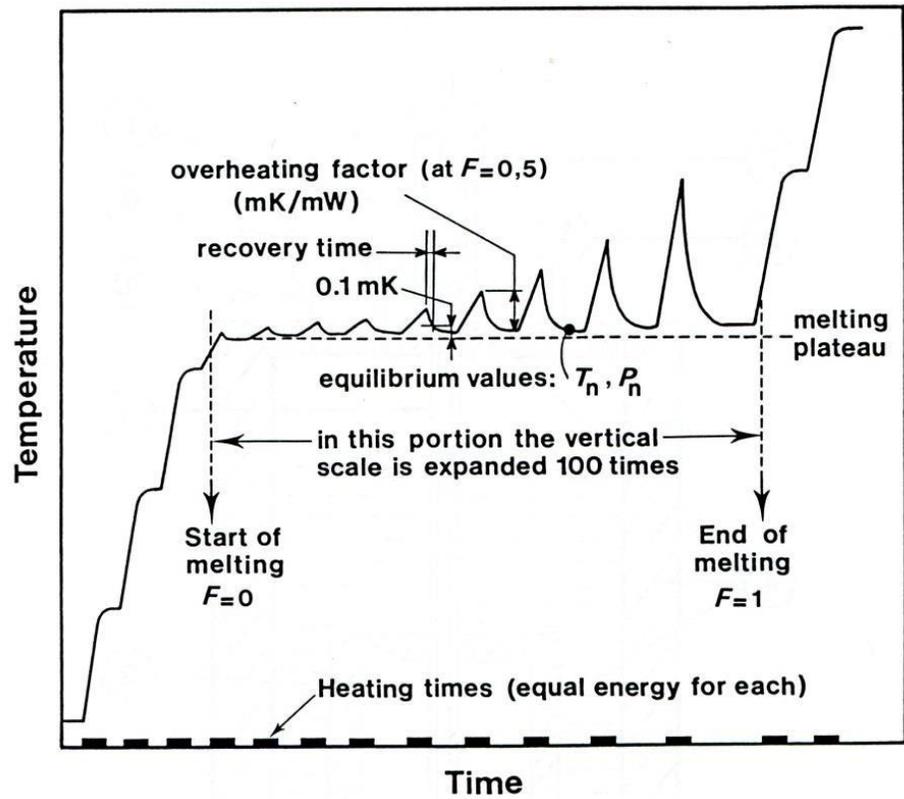
Every CSPRT25 must be calibrated using specific fixed points of the ITS-90. The number of fixed points and the equation for the deviation function depend on the temperature range in which the thermometer will be used.



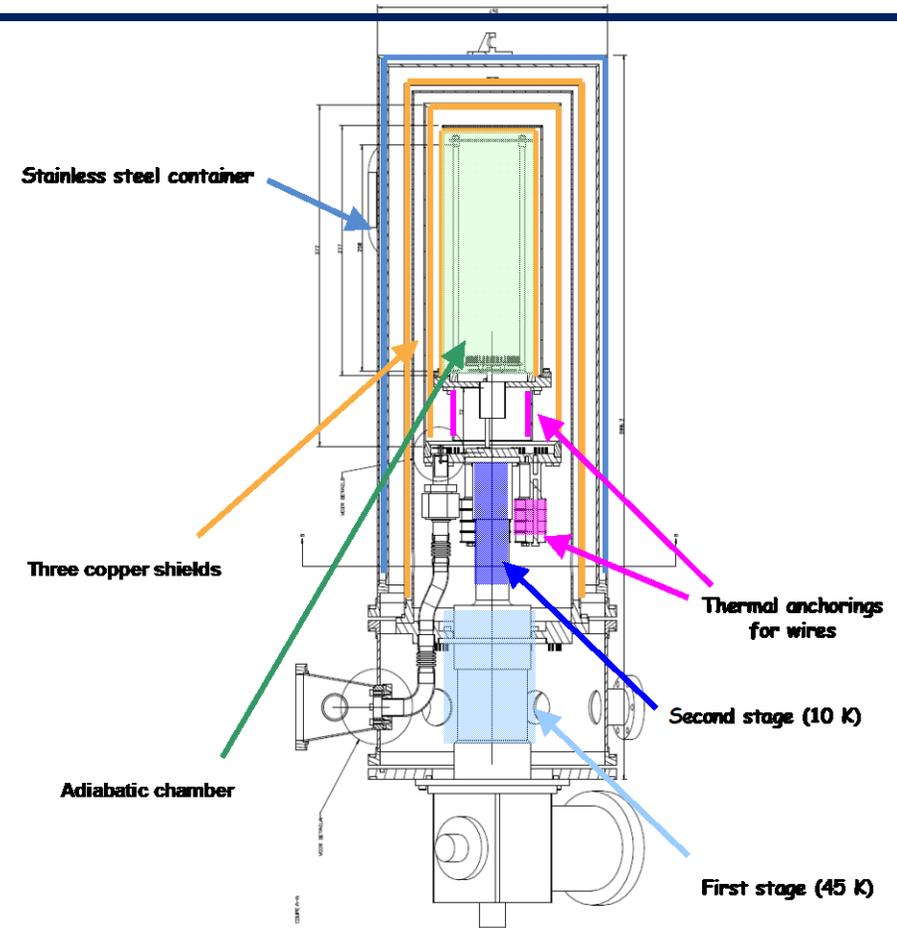
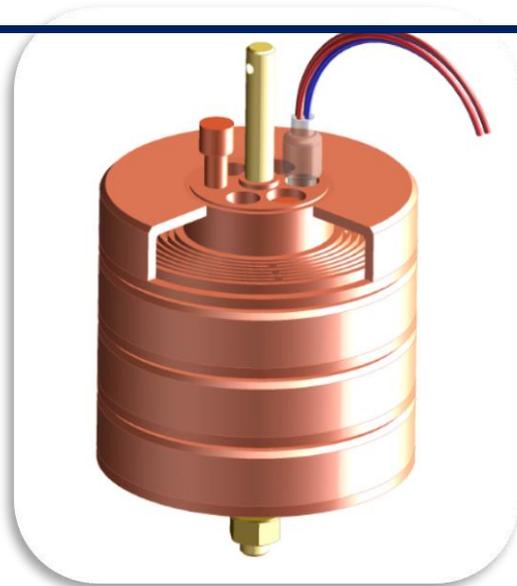
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$$13.8033 \text{ K} \leq T_{90} \leq 273.16 \text{ K},$$

Cryogenic fix point at LNE



Graph BIPM Guide to the Realization of the ITS90. Cryogenic Fixed Points

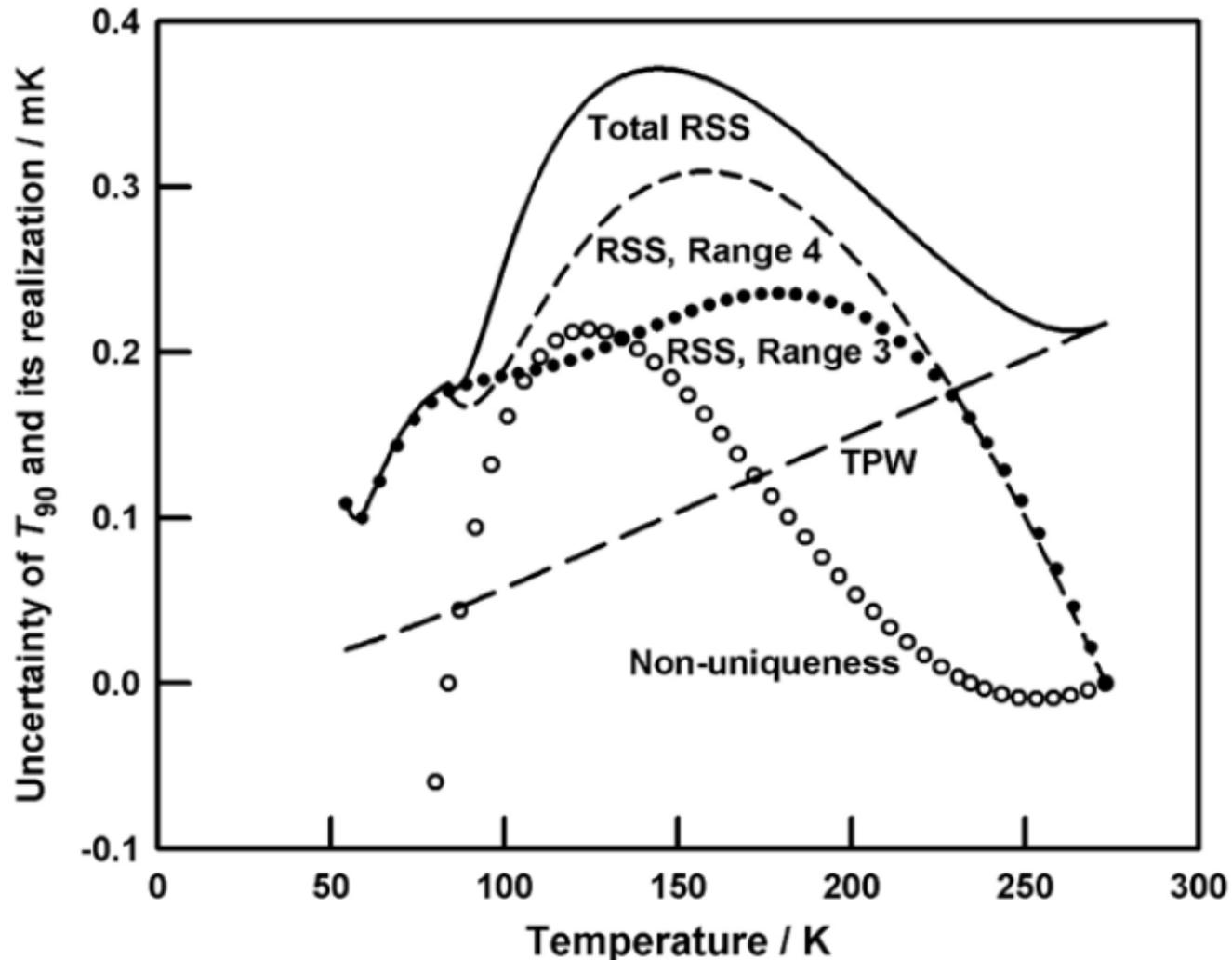


G. Bonnier



Y. Hermier

Example of the propagation of uncertainty with ITS90



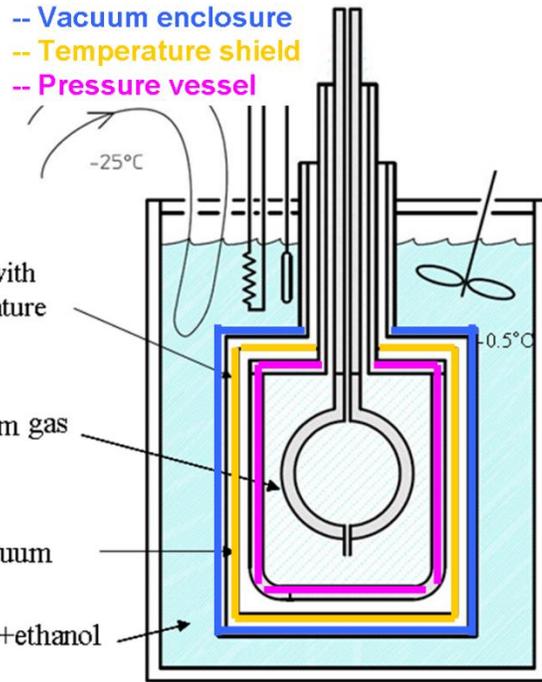
Acoustic thermometry: new results from 273K to 77K and progress towards 4K, L Pitre, M R Moldover and W L Tew Metrologia 43 (2006) 142–162

Uncertainty of the Boltzmann constant determination

The most challenging use of the CSPRT Pt25 was in the project for determining the Boltzmann constant

Uncertainty of the Boltzmann constant determination

Quasi adiabatic cryostat



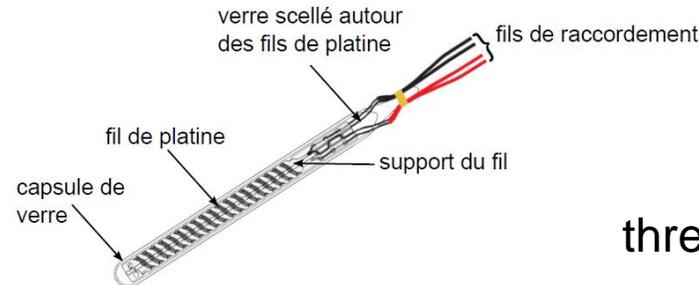
shield with temperature control

helium gas

vacuum

water+ethanol

Thermometers Calibrated at TPW



Thermal Cartography of the Resonator

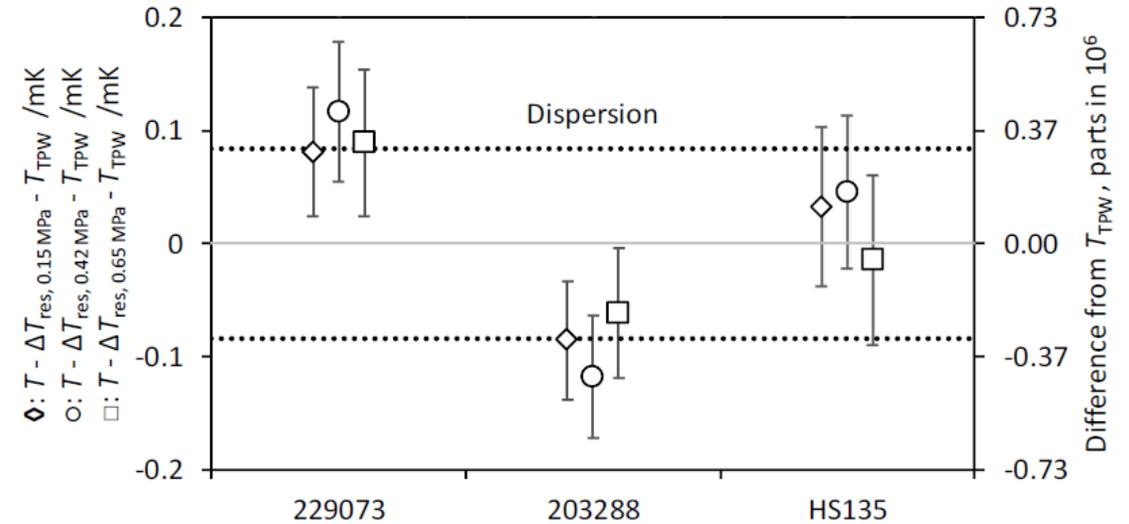


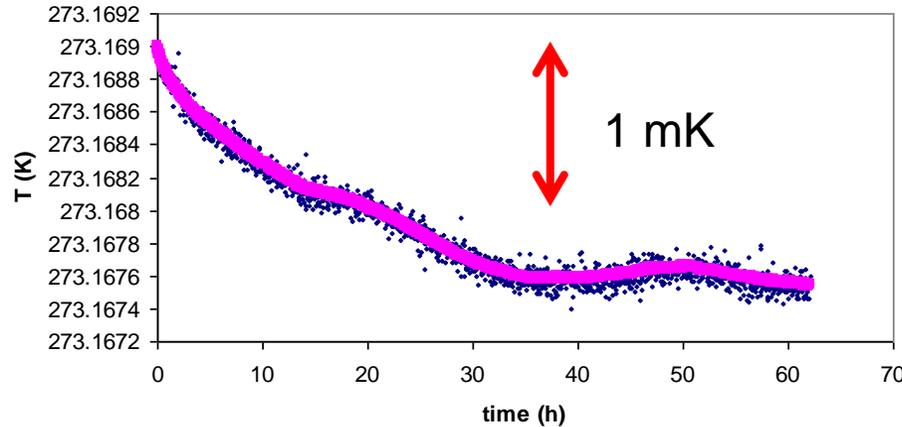
Figure 1. Temperature of the resonator measured using three different capsule-type standard platinum resistance thermometers, during three different runs at three different gas pressures. Values are expressed as differences with respect to T_{TPW} , both in millikelvin and in parts in 10^6 . The dotted lines represent the uncertainty interval related to the dispersion of thermometer readings.

All of those manufacturer have stopped their production.

three capsule thermometers

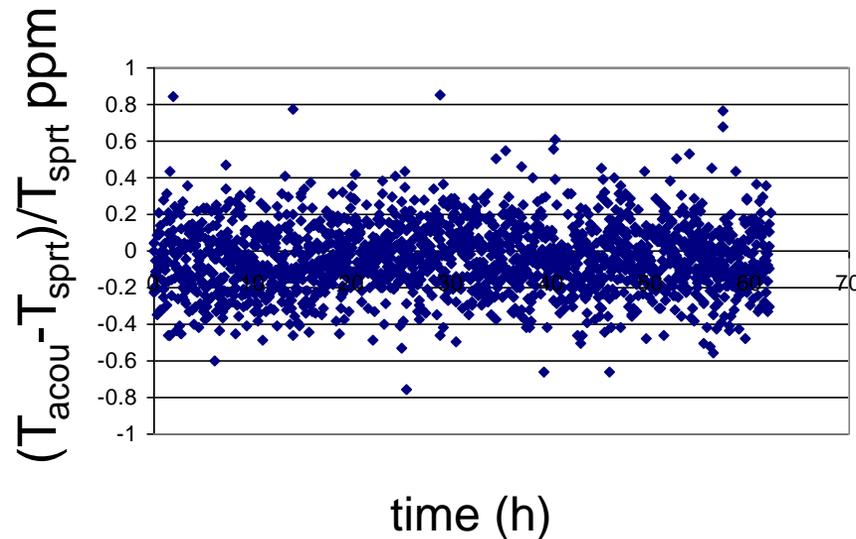
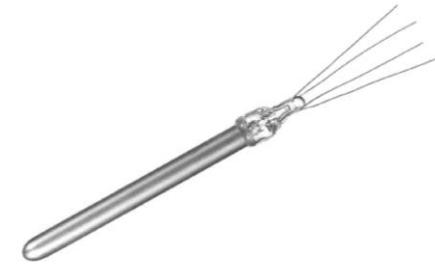
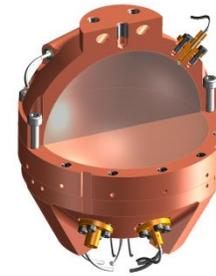
Uncertainty of the Boltzmann constant determination

Same Thermostat, Two Types of Thermometer



2 types of thermometer

• T_{acou}
 ■ T_{sprt}

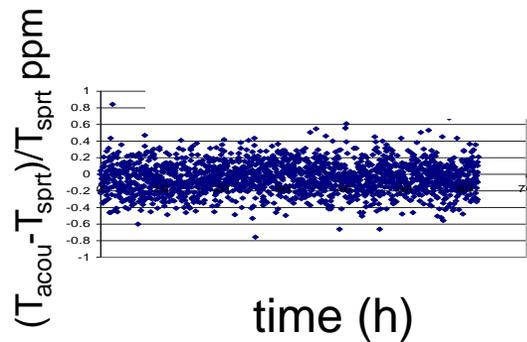
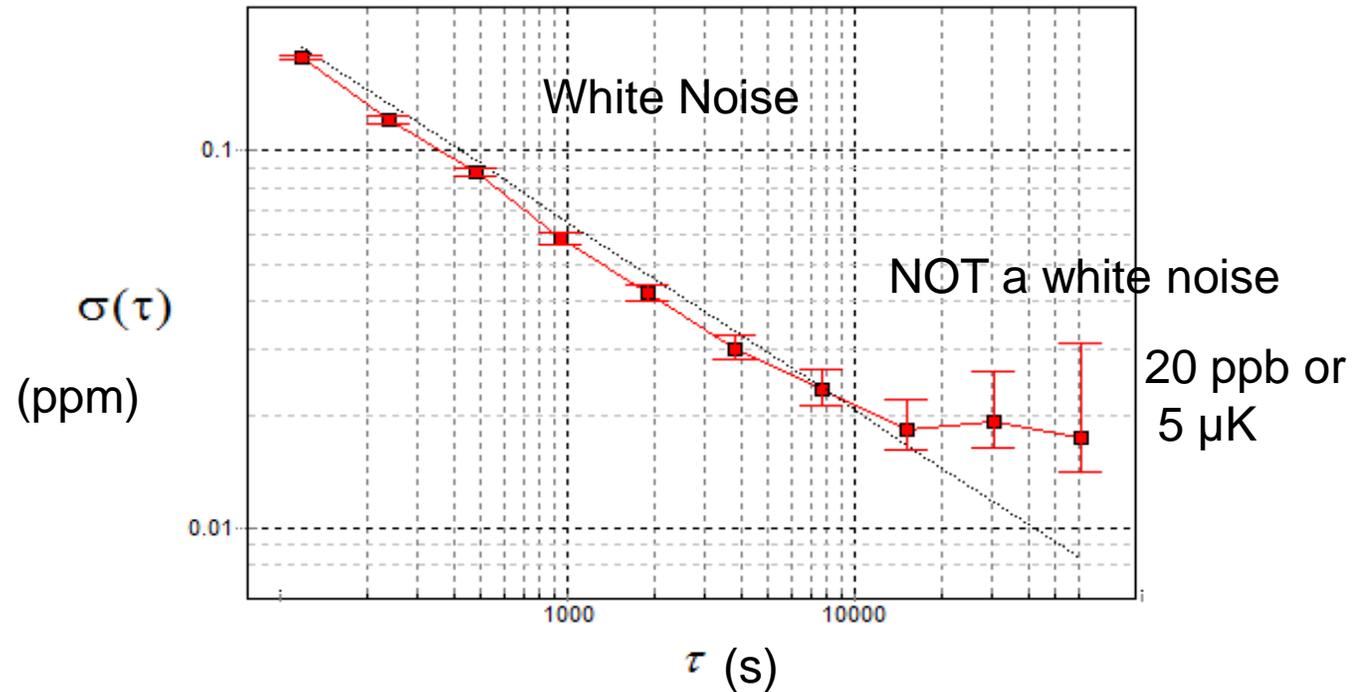


reference	10 MHz	25 Ohm
method	Sinus around the frequency f_n	Resistance bridge

To study the noise, we remove the temperature dependence

Uncertainty of the Boltzmann constant determination

Analyze of the Allan Variance at 273.16K with 2 thermometers



Uncertainty of T_{TPW} for the Boltzmann constant determination

Thermal Cartography of the Resonator BCU4

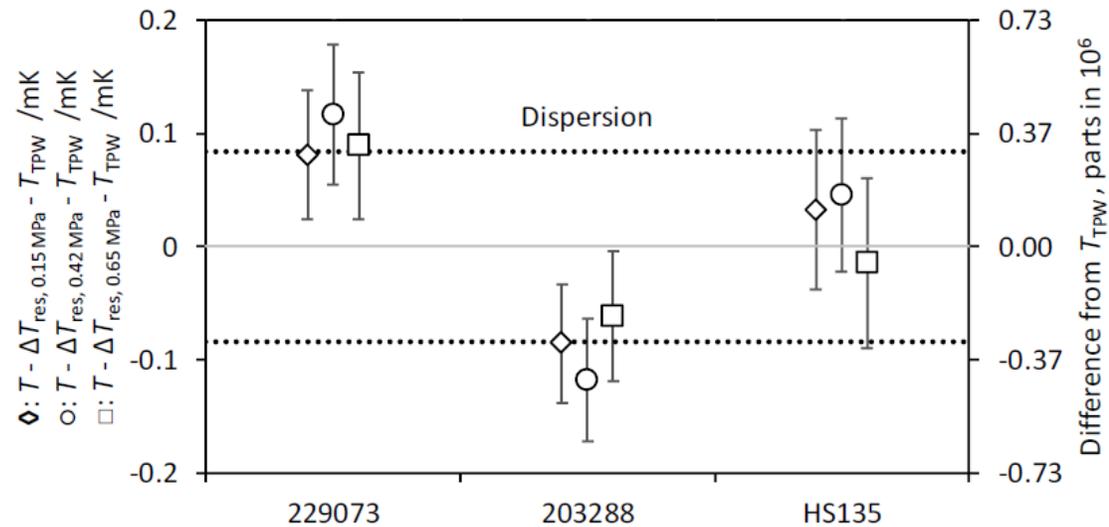


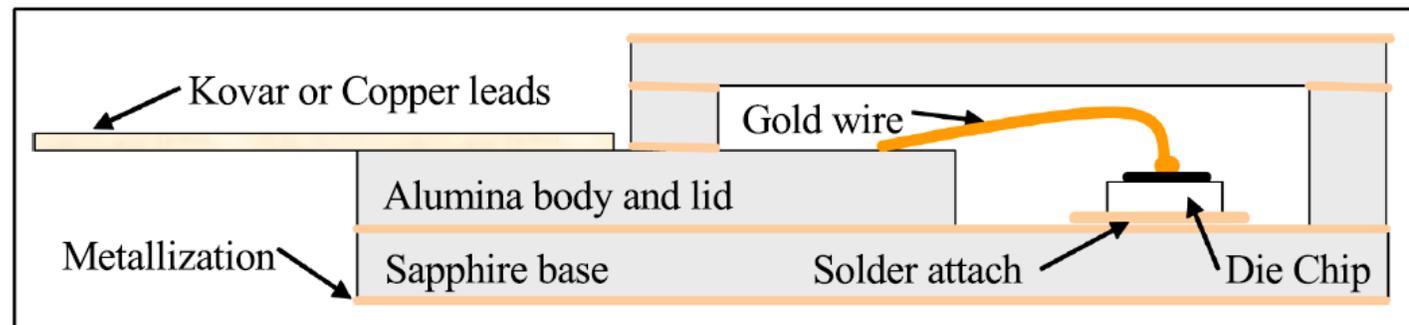
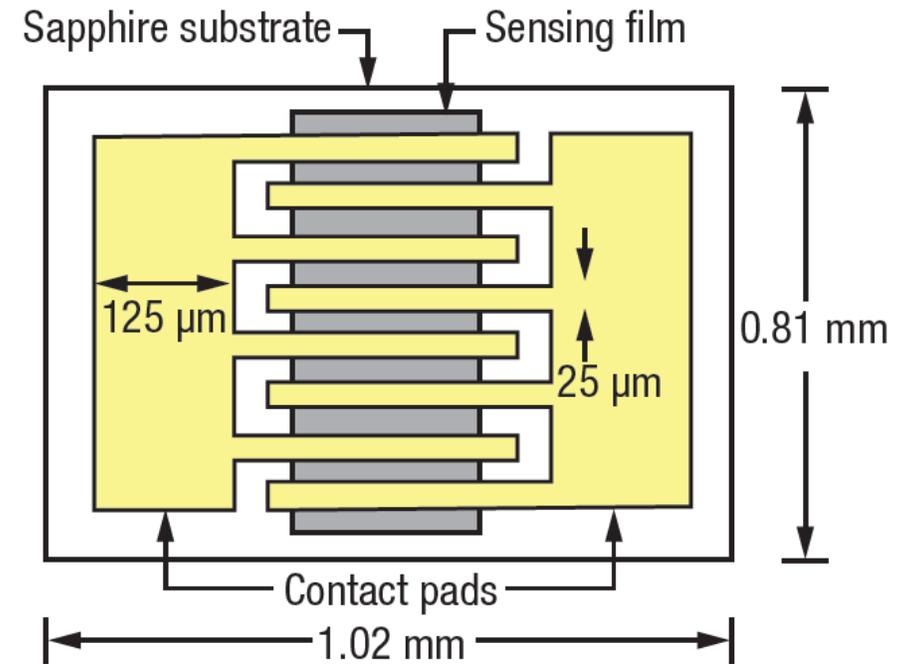
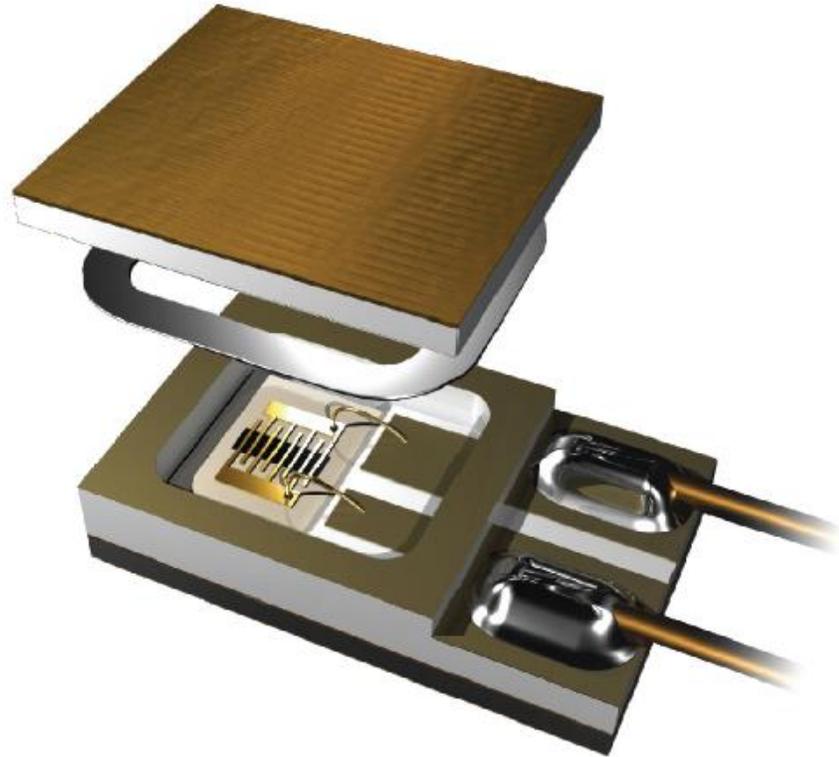
Figure 1. Temperature of the resonator measured using three different capsule-type standard platinum resistance thermometers, during three different runs at three different gas pressures. Values are expressed as differences with respect to T_{TPW} , both in millikelvin and in parts in 10^6 . The dotted lines represent the uncertainty interval related to the dispersion of thermometer readings.

Term	Uncertainty in k (Parts in 10^6)	Note
Repeatability and self-heating	0.08	Evaluated with measurements performed on the resonator
Standard resistor stability	0.05	Temperature stability and time-drift of the standard resistor
Resistance bridge	0.04	Bridge linearity and bandwidth
Dispersion of thermometer readings	0.31	Standard deviation of the temperatures measured on the resonator (scaled to T_{TPW}), includes the contribution of the correction ΔT_{res}
TPW calibration	0.21	Calibration at the TPW, including TPW repeatability, isotopic effect, purity, hydrostatic head effect and heat fluxes
TOTAL	0.39	Square root of the sum of squares

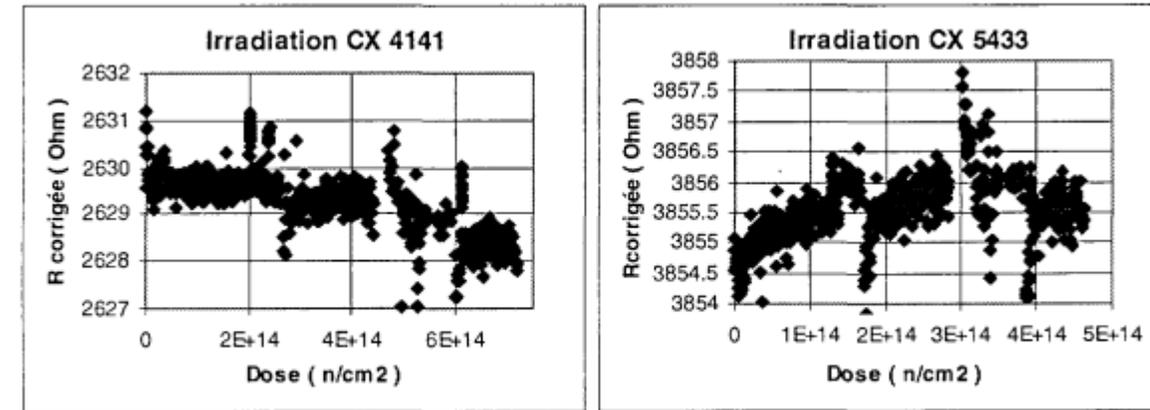
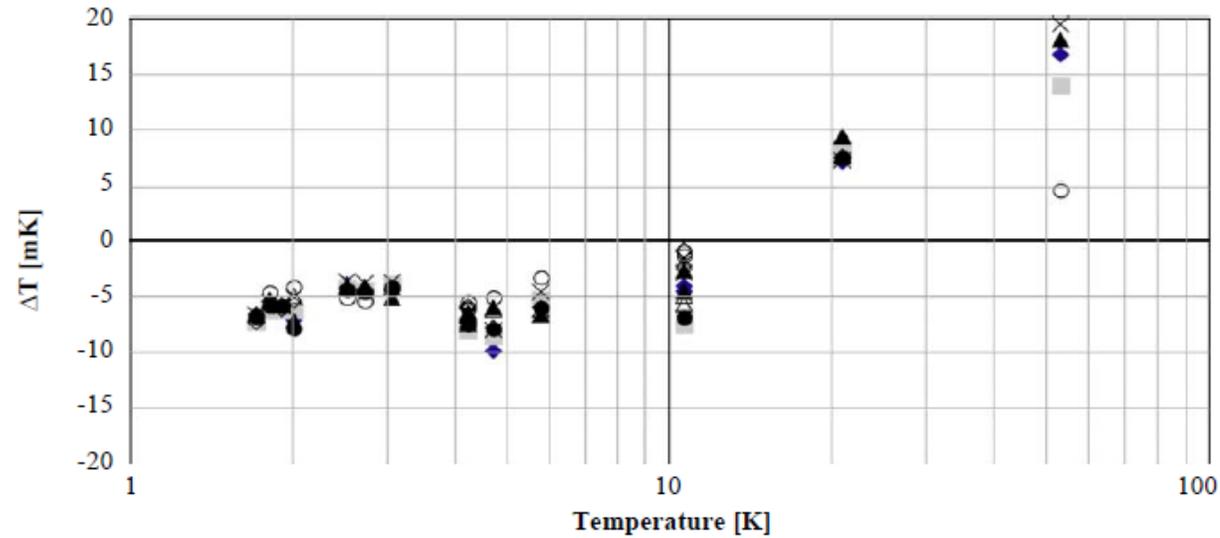
or 0.1 mK

CERNOX

Cernox from lakeshore



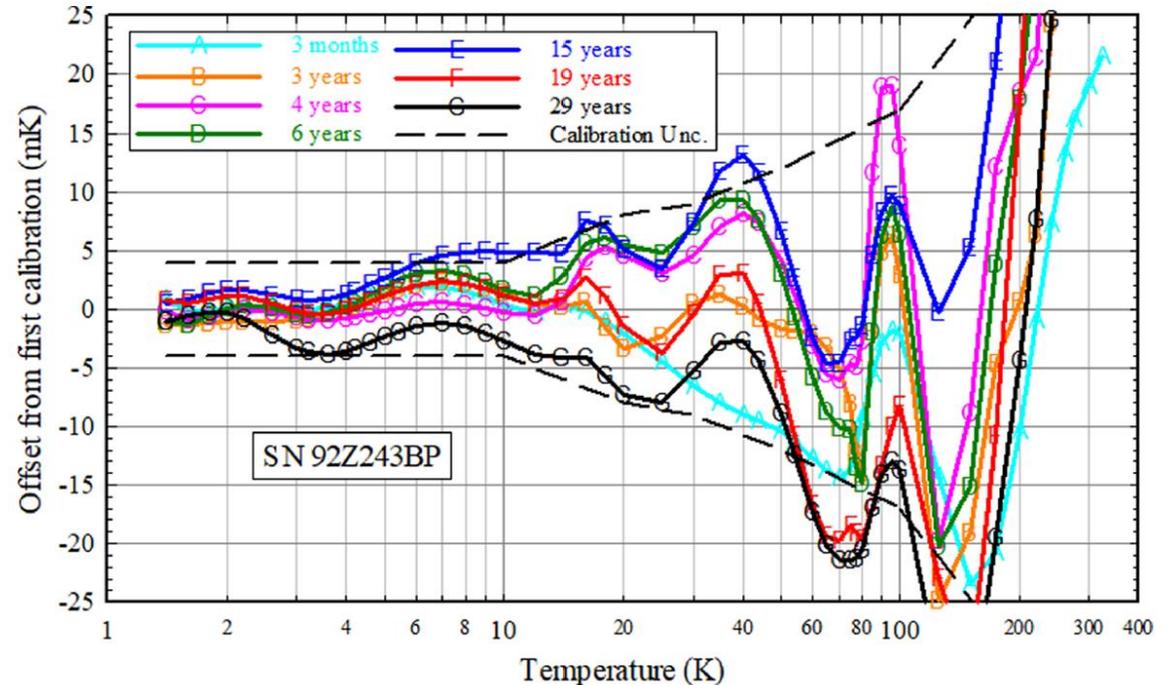
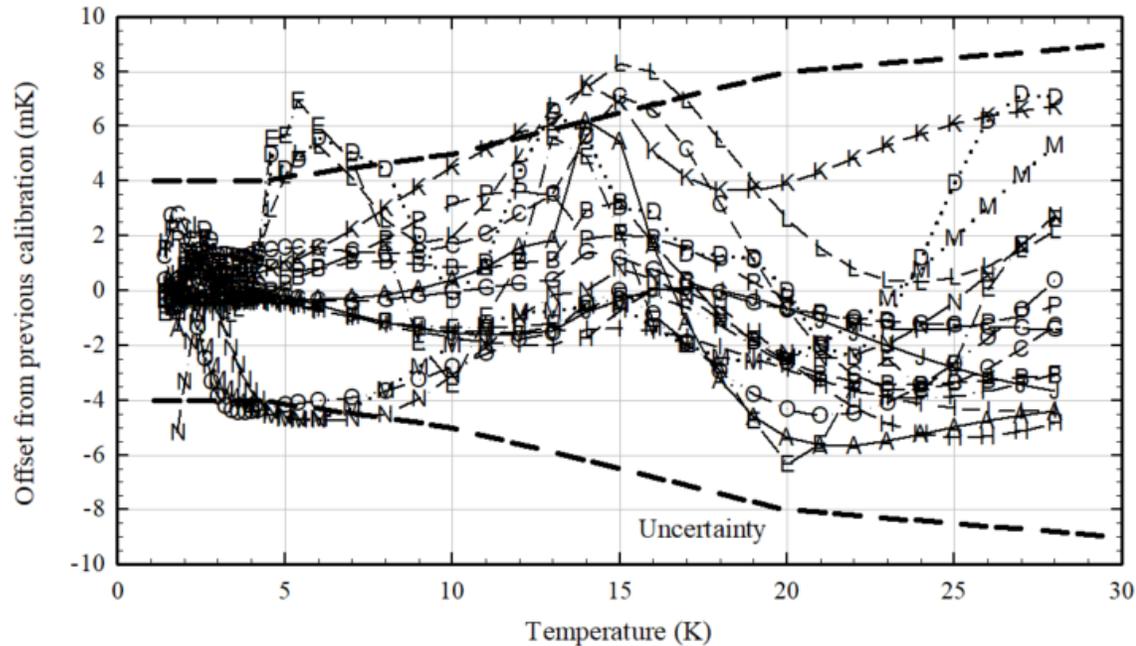
Cernox good against irradiation



Figures 30 et 31

FIGURE 5. Crosscheck between IPN and CERN calibration. ($\Delta T = T_{\text{CERN}}(R) - T_{\text{IPN}}(R)$)

Stability of Cernox over time: $\pm 5\text{mK}$ @ 4K



temperature offset from first calibration as a function of temperature cycling and elapsed storage time at room temperature

We have observed much worse than ± 5 mK

We need better stability

In Conclusion

The international temperature measurement structure

The International Bureau of Weights and Measures (BIPM), through the Consultative Committee for Temperature (CCT), coordinates efforts to establish a consistent temperature scale worldwide.

Whether you purchase a sensor from France, the USA, or Japan, there's no need to adjust your measurements based on the sensor's origin.

Periodically, the CCT conducts international comparisons, known as key comparisons. At the highest precision, the CCT employs resistance thermometers.

The need for a new metrological sensor for thermometry

Below 273K 2 type of resistive sensor have the metrological propriety

-CSPRT (Pt25)

-Rhodium Iron Capsule type thermometer

CCT K2 Key comparaison

Most of the thermometers provided were 25.5 Ω Leeds and Northrup (L&N)-style CSPRTs, some of which were manufactured by Yellow Springs International (YSI); one of the NPL thermometers was manufactured by Tinsley.

All of those manufacturer have stopped their production.

CCT K1 Key comparaison

-Rhodium Iron Capsule type thermometer from Tinsley

This manufacturer has stopped its production.

**All the metrological institutes worldwide need to characterize new sensors for thermometry or new company production
At least 10 years of study are needed.**

In Conclusion

- Need for a new metrological sensor for low-temperature thermometry... please help us.
- LNE-Cnam has developed a characterized platform for temperatures below 4K.
- Direct thermodynamic measurements will become increasingly common in the future.
- ITS-90 will remain in use for many more years due to company investments in quality systems.
- The T-T90 equation will be updated from time to time.

The need for a new metrological sensor for thermometry

If your lab has any of the following items and they are not being used:

- Rhodium Iron Capsule type thermometer from Tinsley
- Leeds and Northrup CSPRT 25
- SRD1000 from HDL

Please contact me, as my lab **could purchase** them.

Thank you

**And see you at Tempmeko Oct 2025
in France**

My Encouragement Stick



My new management method

Encouragement Stick (*Keisaku*)

Looking for a new PhD student

- 3 years
- Low temperature subject
- Location Paris with 2000 euro/month