



Advanced Cryogenics Course

CRYOCOURSE 2011

Melting Curve Thermometry at very low temperatures

Melting curve thermometry – PLTS2000 Temperature scale

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Outline of the talk

- Introduction
- Low Temperature Scales - PLTS2000
- The ${}^3\text{He}$ melting curve thermometer
- Calibration of the ${}^3\text{He}$ melting curve thermometer
- Conclusions

Introduction : Temperature Scales

1742 : Celsius Phase transitions of water at atmospheric pressure
 $T_{\text{melting}} = 0^\circ$ $T_{\text{boiling}} = 100^\circ$

1848 : Kelvin Thermodynamic scale (absolute) : T_0

1887 : degré centigrade defined by the *Comité International des Poids et Mesures*
First practical temperature scale

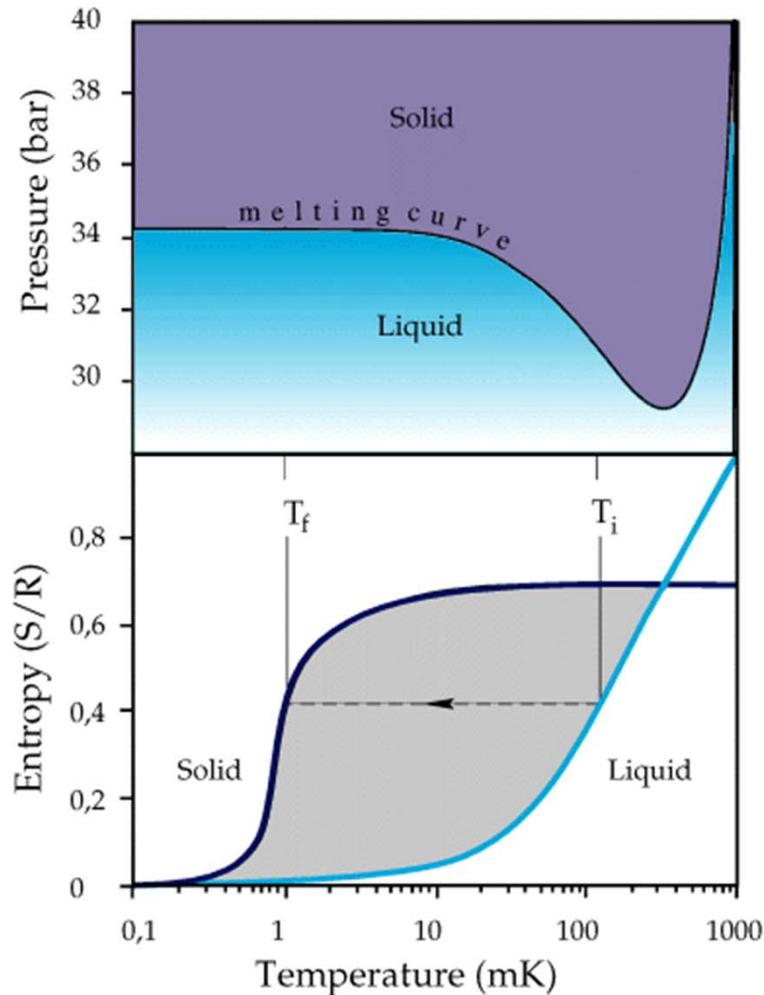
1927 : ITS-27 is adopted. Defines the fixed points and the interpolation laws.
Lower limit: LO₂

1948 : introduction of the « degré Celsius ».
International scales IPTS-48 et ITS-48

1954 : Definition of the « degré Kelvin » from the triple point of water:
 $1 \text{ degré Kelvin} = T_t / 273,16$ $T_t = 0,01 \text{ C}$
 $T_0 = 0 \text{ K} = -273,15 \text{ K}$

1968 : IPTS-68 reaches 13,81 K (triple point of hydrogen)

Temperature scales (2)



1976 : EPT-76, from 0,5 to 30 K, defined by the triple points of pure substances, transition temperatures of superconductors, and the vapor pressure la tension de vapeur of ^3He and ^4He .
Differs from IPTS-68 by a few millikelvins.

1990 : « International Temperature Scale » ITS-90.
0.65 K to 1358 K

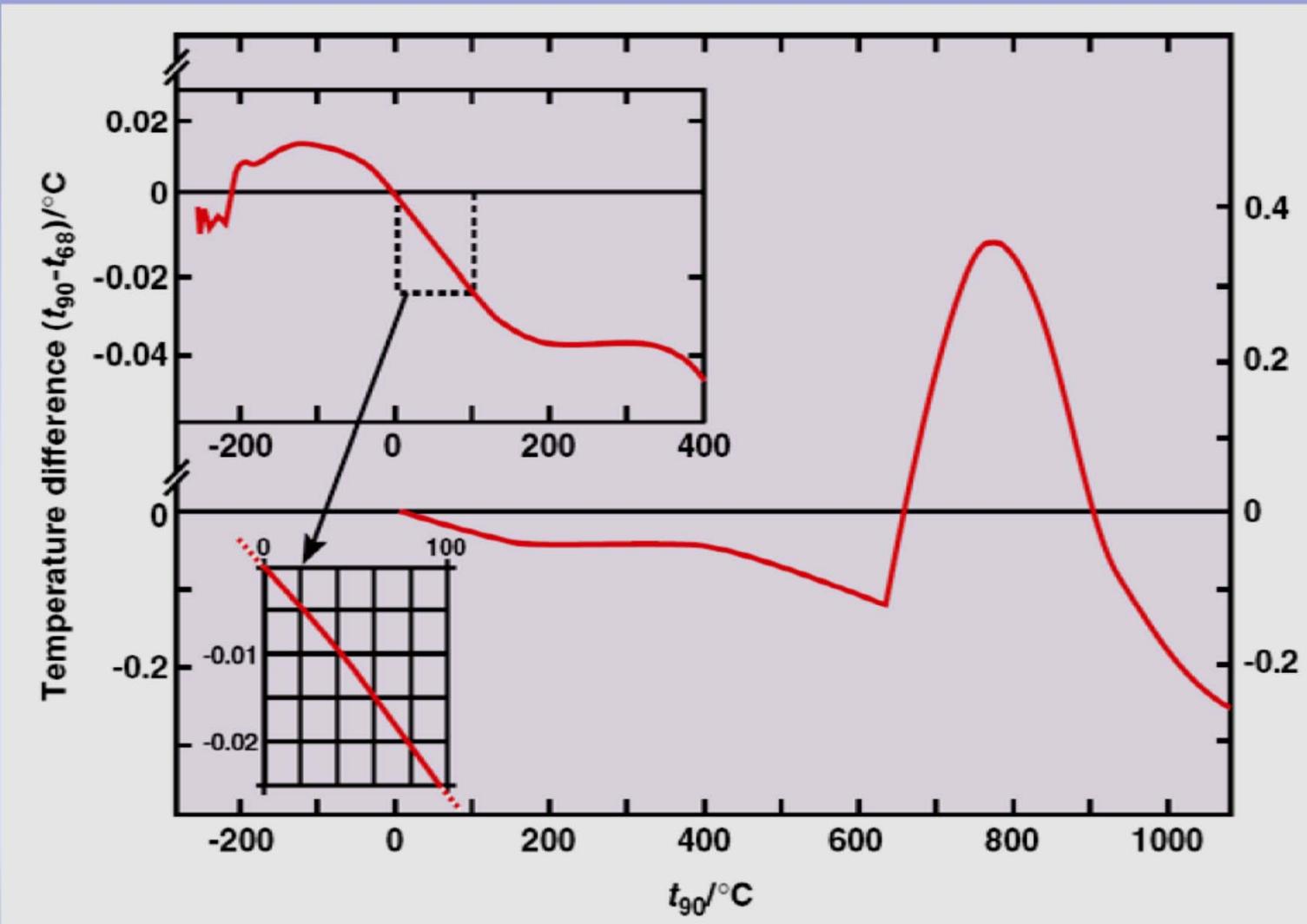
2000 : « Echelle Provisoire pour les Basses Températures EPBT-2000 » (**PLTS-2000**)
Based on the ^3He melting curve.
Defined down to 0,9 mK

Non-official Temperature Scales exist below this temperature...

ITS-90

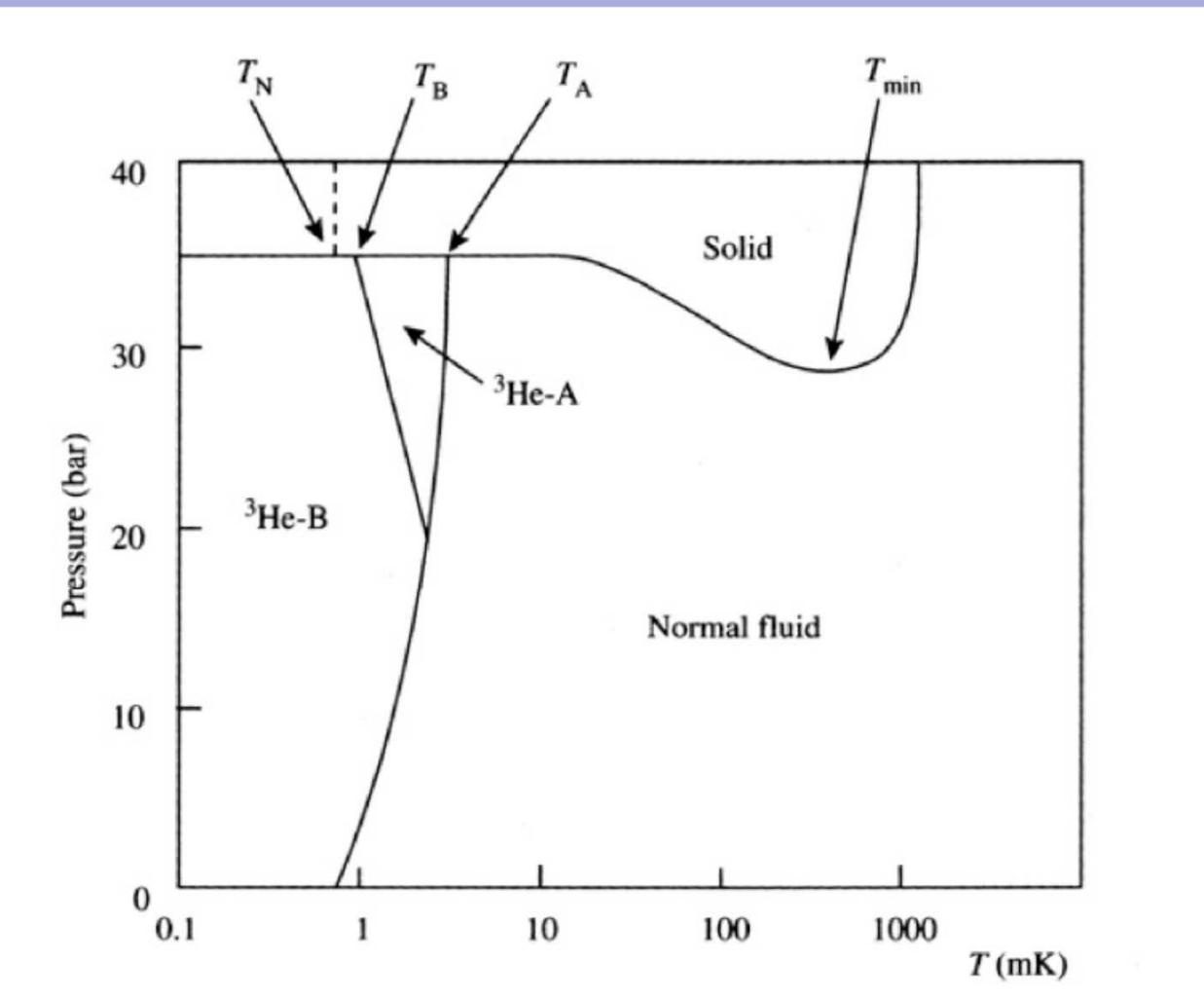
Substance and its state	Defining point in Kelvins (range)	Defining point in degrees Celsius (range)
Vapor-pressure / temperature relation of helium-3 (by equation)	(0.65 to 3.2)	(-272.50 to -269.95)
Vapor-pressure / temperature relation of helium-4 below its lambda point (by equation)	(1.25 to 2.1768)	(-271.90 to -270.9732)
Vapor-pressure / temperature relation of helium-4 above its lambda point (by equation)	(2.1768 to 5.0)	(-270.9732 to -268.15)
Vapor-pressure / temperature relation of helium (by equation)	(3 to 5)	(-270.15 to -268.15)
Triple point of hydrogen	13.8033	-259.3467
Triple point of neon	24.5561	-248.5939
Triple point of oxygen	54.3584	-218.7916
Triple point of argon	83.8058	-189.3442
Triple point of mercury	234.3156	-38.8344
Triple point of water	273.16	0.01
Melting point ¹ of gallium	302.9146	29.7646
Freezing point ¹ of Indium	429.7485	156.5985
Freezing point of tin	505.078	231.928
Freezing point of zinc	692.677	419.527
Freezing point of aluminum	933.473	660.323
Freezing point of silver	1234.93	961.78
Freezing point of gold	1337.33	1064.18
Freezing point of copper	1357.77	1084.62

Difference between ITS-90 and ITS-68



PLTS-2000

^3He - melting curve



PLTS 2000

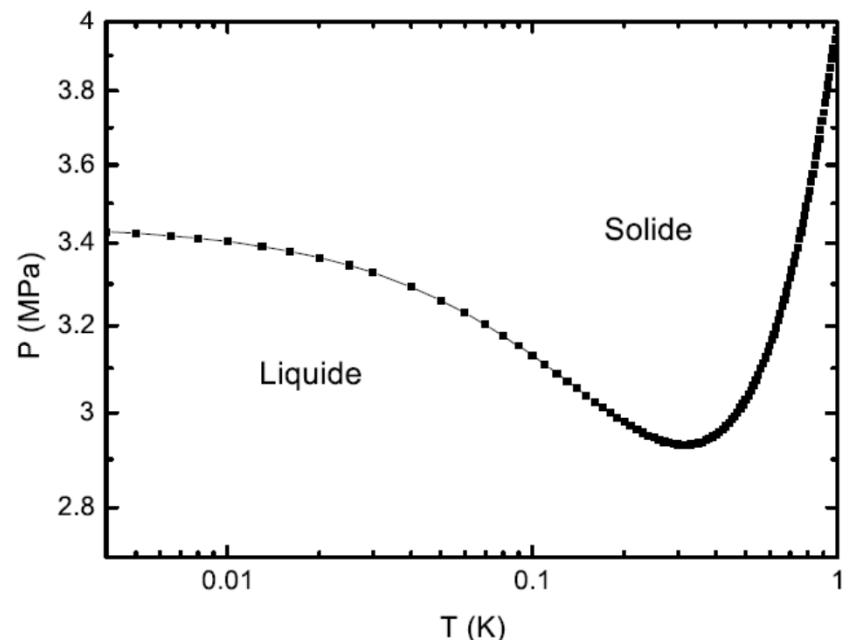
$$P(\text{MPa}) = \sum_{i=-3}^9 a_i \cdot T^i \quad \text{avec } T \text{ en Kelvin}$$

$$\begin{aligned} a_{-3} &= -1.3855442 \cdot 10^{-12} \\ a_{-2} &= 4.5557026 \cdot 10^{-9} \\ a_{-1} &= -6.4430869 \cdot 10^{-6} \\ a_0 &= 3.4467434 \cdot 10^0 \\ a_1 &= -4.4176438 \cdot 10^1 \\ a_2 &= 1.5417437 \cdot 10^1 \\ a_3 &= -3.5789853 \cdot 10^1 \end{aligned}$$

$$\begin{aligned} a_4 &= 7.1499125 \cdot 10^1 \\ a_5 &= -1.0414379 \cdot 10^2 \\ a_6 &= 1.0518538 \cdot 10^2 \\ a_7 &= -6.9443767 \cdot 10^1 \\ a_8 &= 2.6833087 \cdot 10^1 \\ a_9 &= -4.5875709 \cdot 10^0 \end{aligned}$$

Fixed points of the PLTS-2000

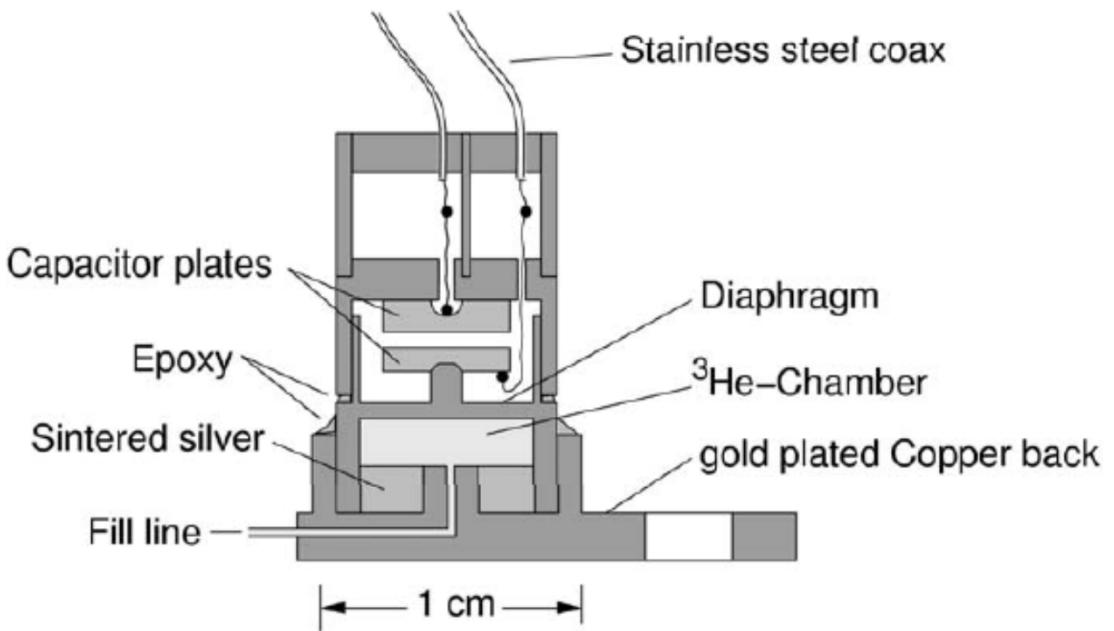
Fixed points	p/MPa	T ₂₀₀₀ /mK
Minimum	2.93113	315.24
A	3.43407	2.444
A-B	3.43609	1.896
Néel	3.43934	0.902



Greywall's scale

1986

0.9 mK – 250 mK



$$P - P_A(\text{bar}) = \sum_{i=-3}^5 a_i \cdot T^i \quad \text{avec } T \text{ en mK} \quad (7.2)$$

La pression est relative à P_A , pression du point fixe de la transition entre la phase liquide normale et superfluide-A.

Les coefficients a_i sont :

$$a_{-3} = -0.19652970 \cdot 10^{-1}$$

$$a_{-2} = 0.61880268 \cdot 10^{-1}$$

$$a_{-1} = -0.78803055 \cdot 10^{-1}$$

$$a_0 = 0.13050600$$

$$a_1 = -0.43519381 \cdot 10^{-1}$$

$$a_2 = 0.13752791 \cdot 10^{-3}$$

$$a_3 = -0.17180436 \cdot 10^{-6}$$

$$a_4 = -0.22093906 \cdot 10^{-9}$$

$$a_5 = 0.85450245 \cdot 10^{-12}$$

Differences between LT temperature scales

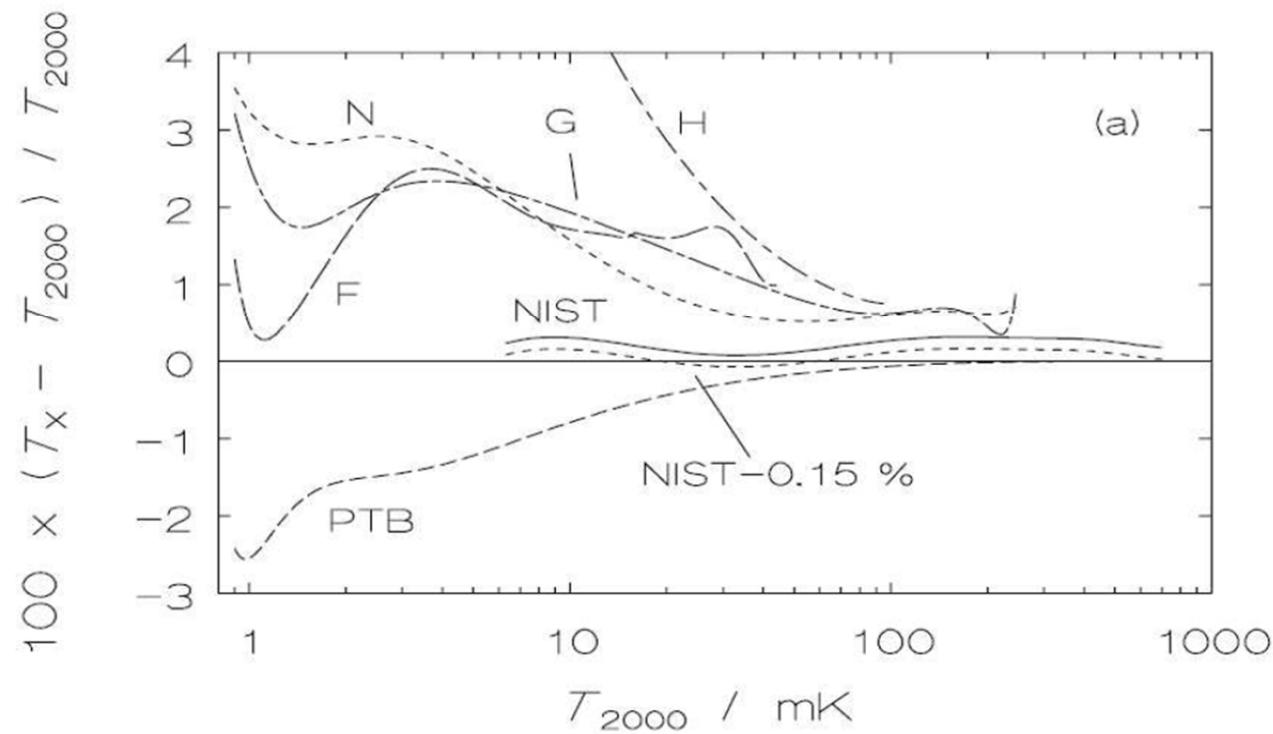


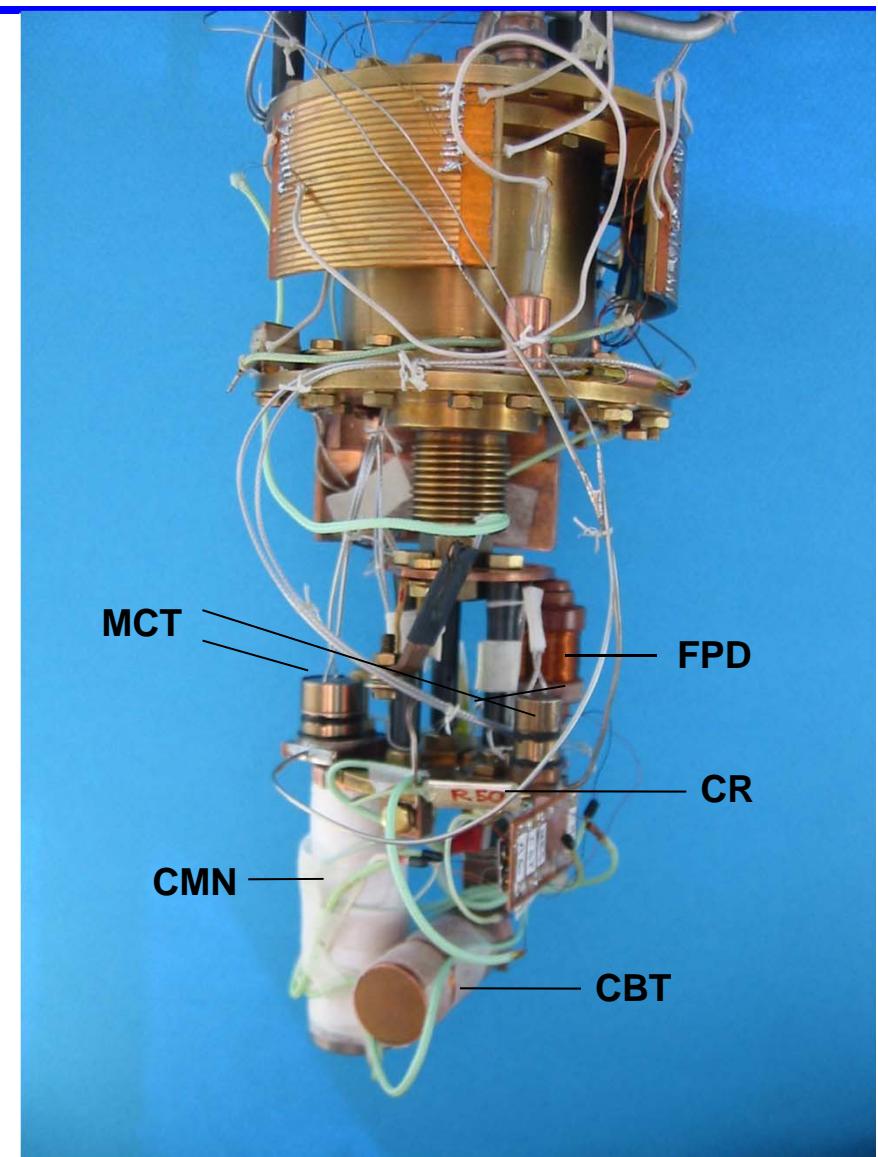
FIG. 7.2 – Différence relative entre l'échelle PLTS-2000 et les échelles de température antérieures des laboratoires. Pour reconnaître les échelles sur le graphique G : Greywall, H : Halperin, F : Fukuyama, N : UF-95 et les échelles du NIST-98 et PTB-96 [23].

Grenoble low temperature calibration facility

Collaboration with the BNM

Thermometrical set-up :

- Two melting curve thermometers (MCT)
- A Coulomb Blockade thermometer (CBT)
- Two superconducting fixed points devices (FPD): SRM768 and SRD1000
- A CMN thermometer
- Carbon resistances (CR)



Metrological set-ups

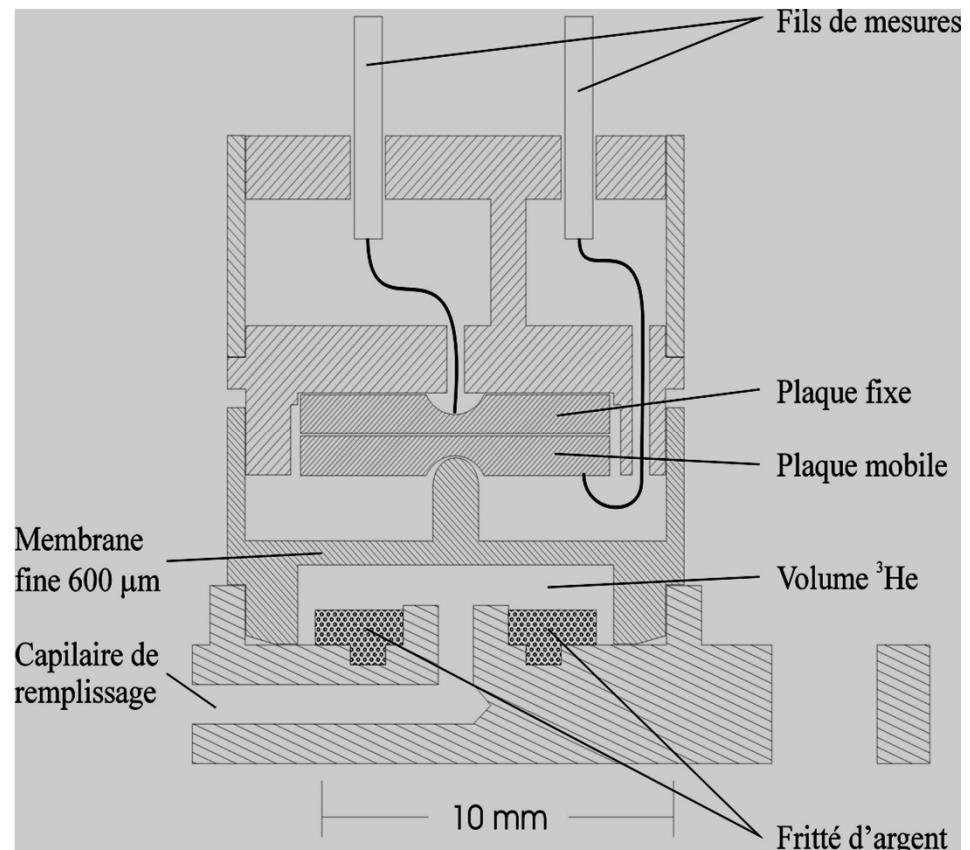
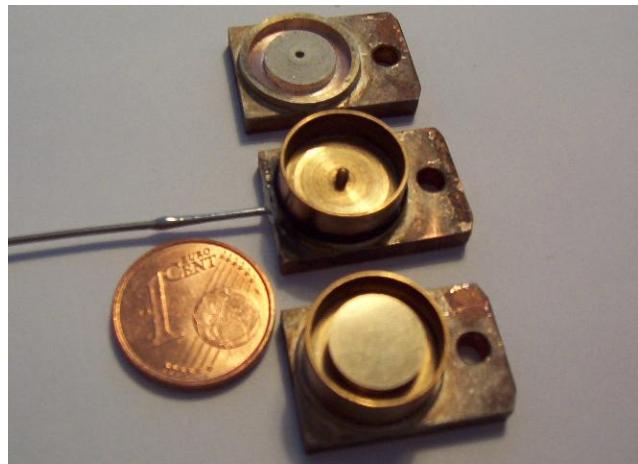


The thermometer stage of the NPL dilution refrigerator below (and isolated through a heat switch from) the mixing chamber, loaded (from left to right) with a PTB melting pressure thermometer, a SRM768 device (behind), a ^{60}Co nuclear orientation thermometer (at the centre), a CMN thermometer and a rhodium-iron resistance thermometer. The PrNi_5 nuclear coolant is suspended beneath the plate and is not shown.

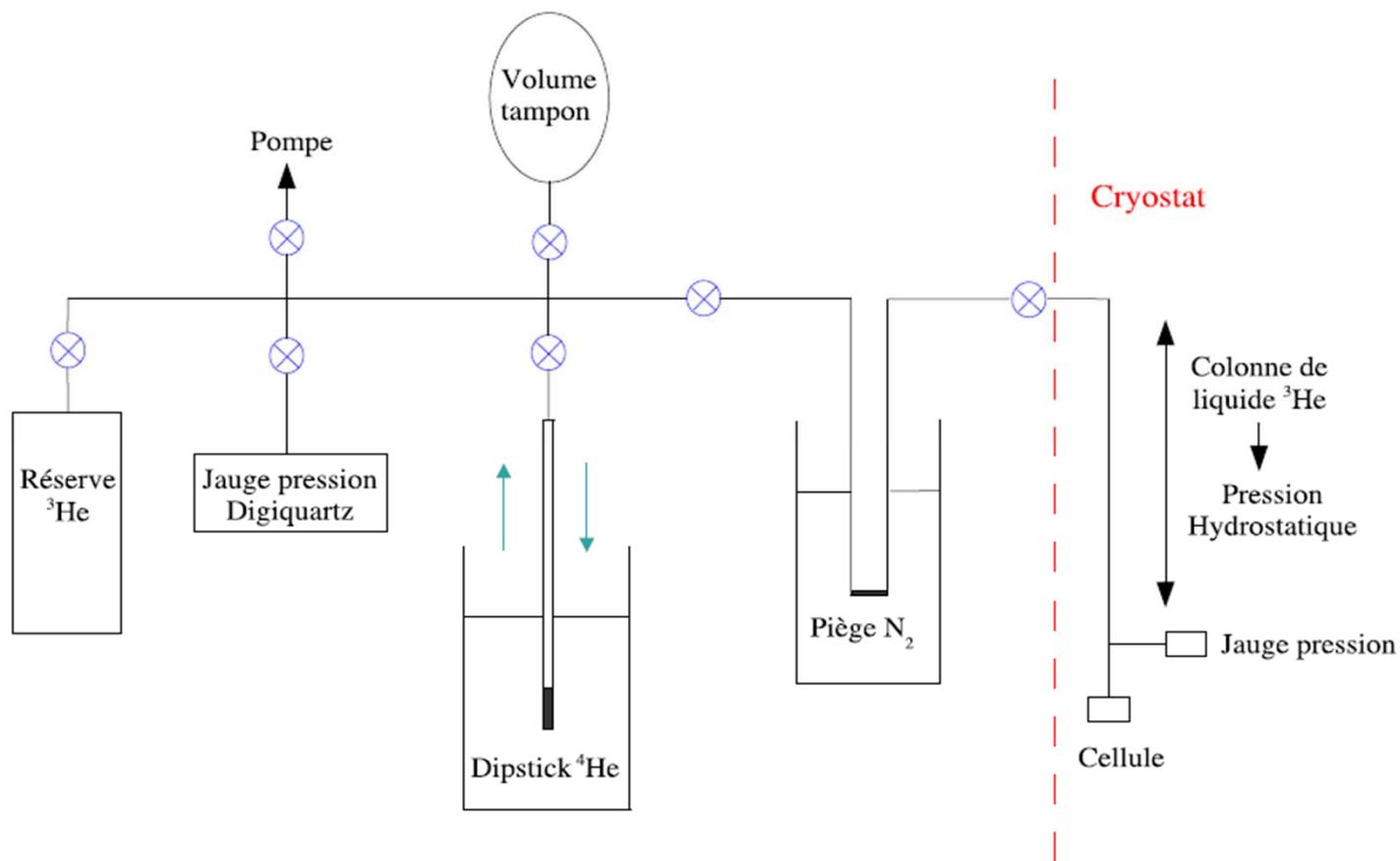
The ${}^3\text{He}$ melting curve thermometer

- Cell containing ${}^3\text{He}$
- Solid-liquid in equilibrium
- Thin metallic membrane
- Capacitive measurement of the displacement
- PLTS-2000 equation between 0,9 mK and 1 K :

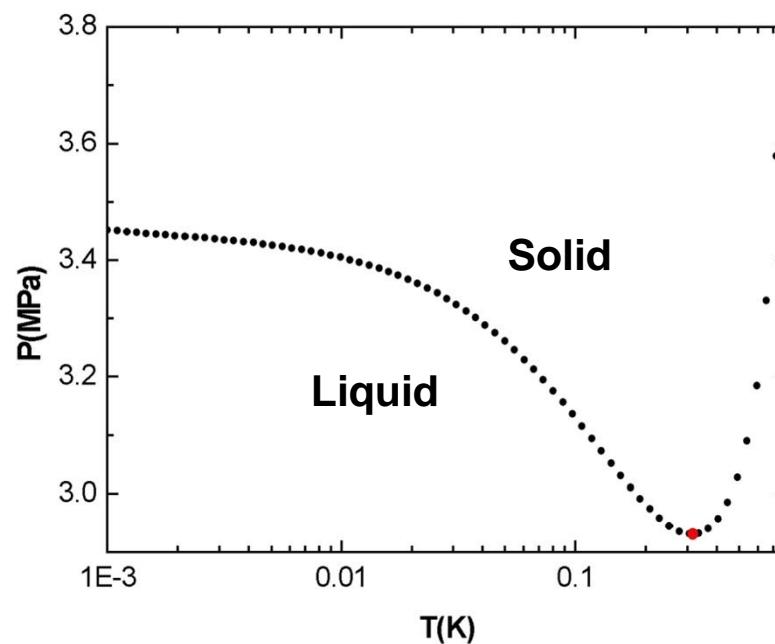
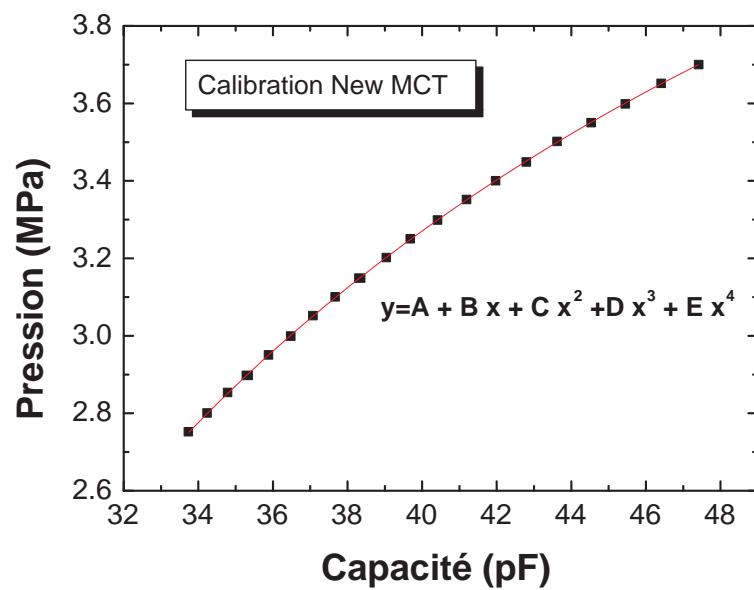
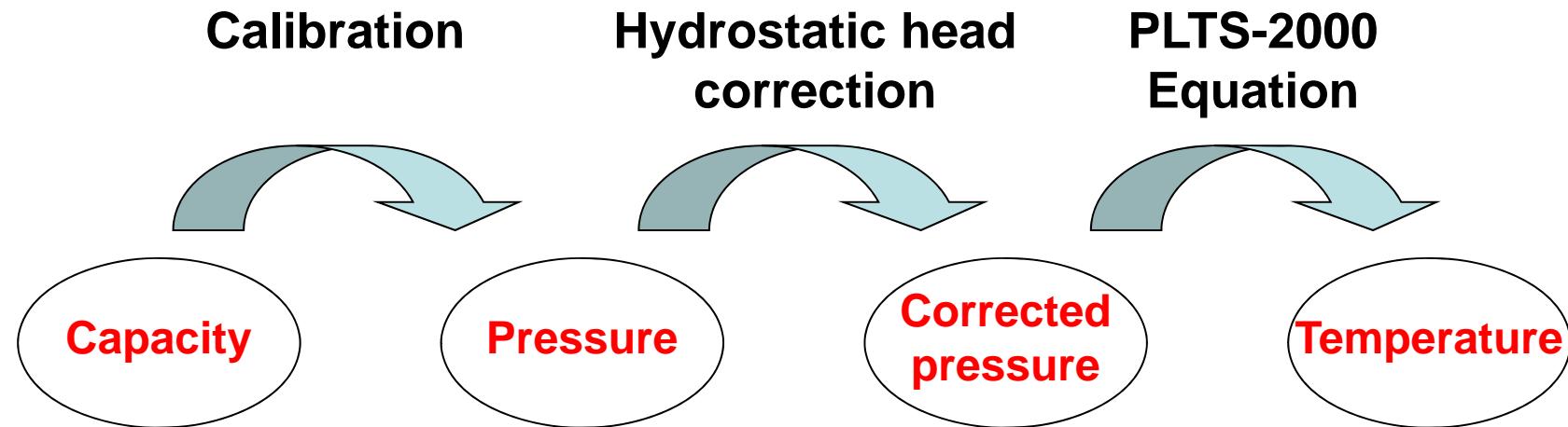
$$P(\text{MPa}) = \sum_{i=-3}^9 a_i \cdot T^i \quad \text{avec } T \text{ en } \text{mK}$$



Pressurisation system (« dipstick »)



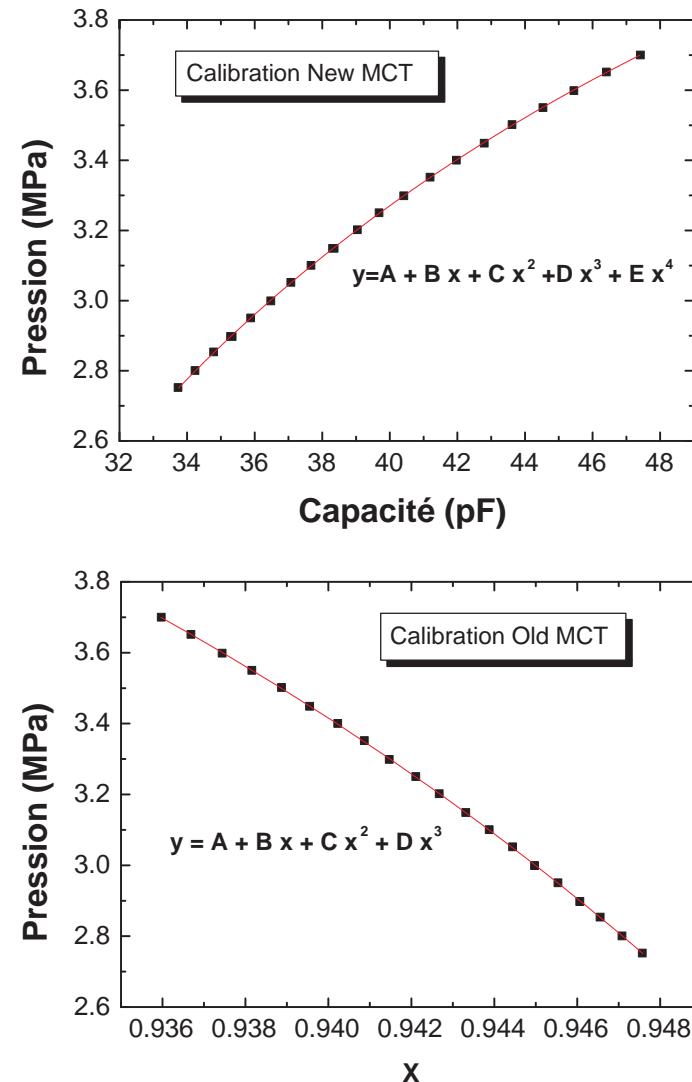
Mesuring the temperature





MCT Calibration

- Pressure is measured with dead-weight or a Digiquartz gage
- Train the membrane of the MCT by repeated pressure cycles
- Calibrate at several stable ^3He pressures
- Determine the relation $P(C)$
- Polynomial fit



Calibration sensitivity

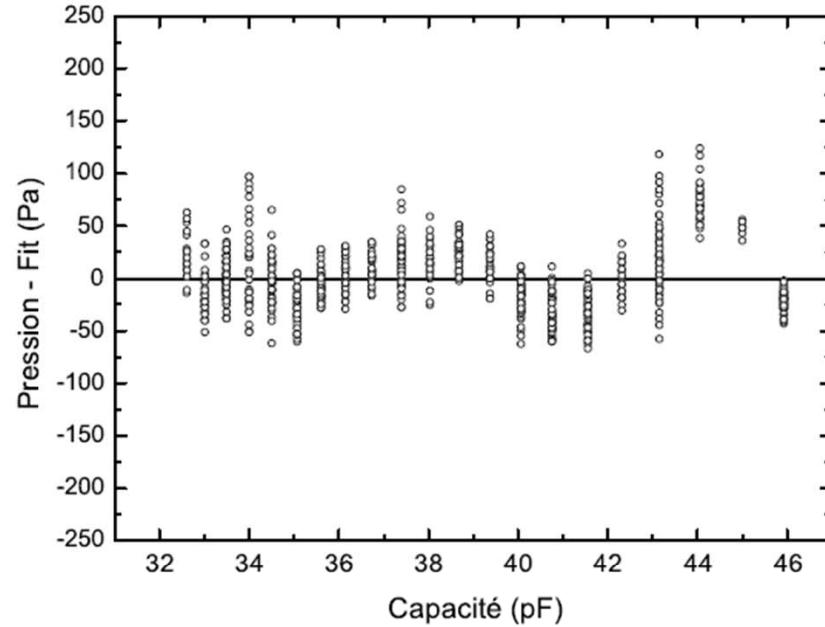
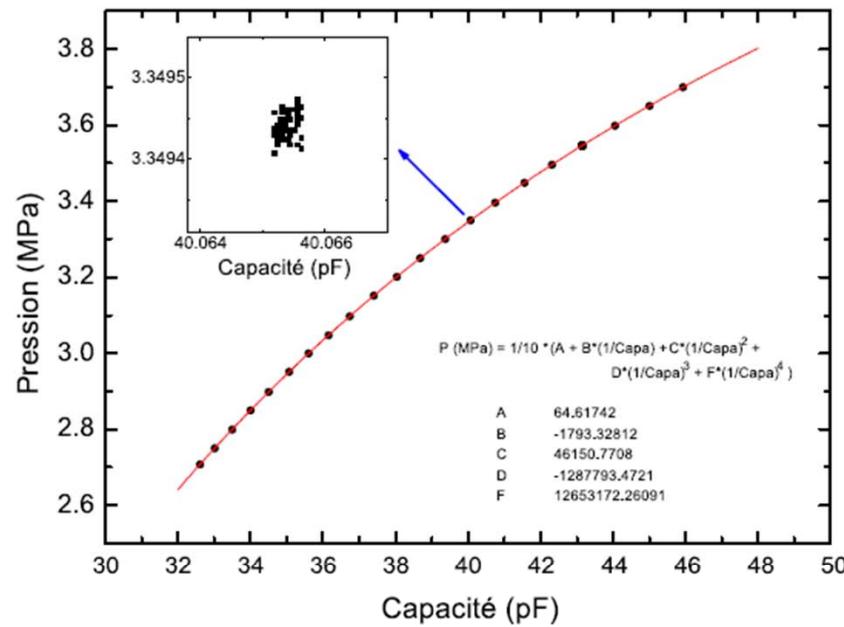
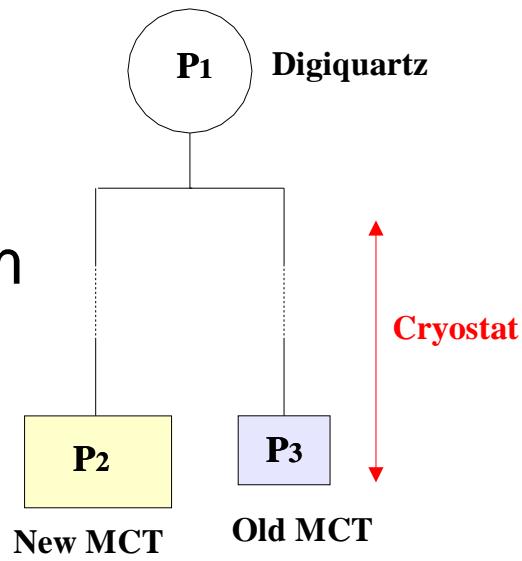


FIG. 2.5 – Etalonnage du thermomètre à courbe de fusion entre 2.7 MPa et 3.7 MPa, ainsi que l'écart entre l'ajustement et la mesure de pression. L'erreur observée est inférieure à 100 Pa.

Calibration uncertainty budget

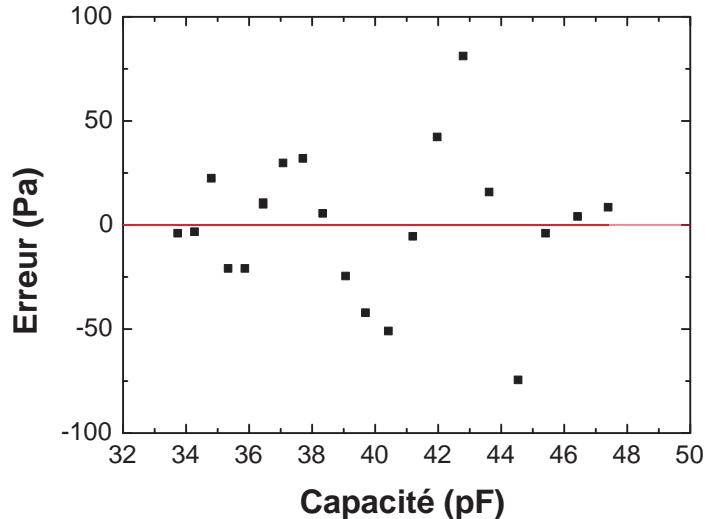
- Sensitivity and accuracy of the pressure gage

- Pressure equilibrium

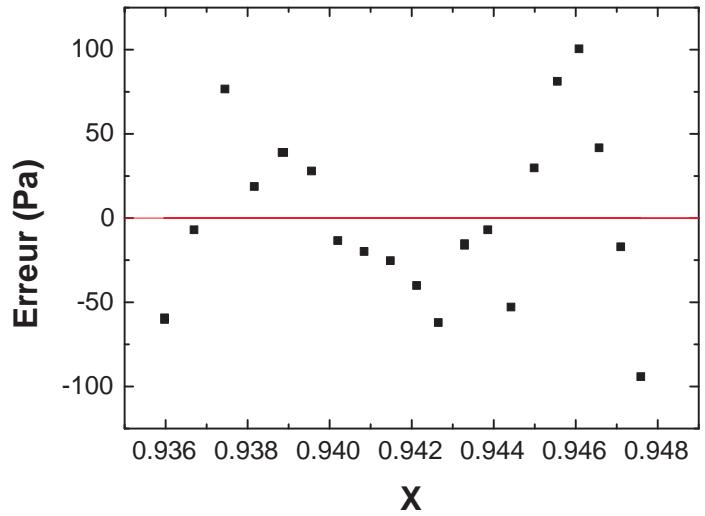


➡ *Uncertainty of 100 Pa over 3 MPa : 0,003%*

New MCT:



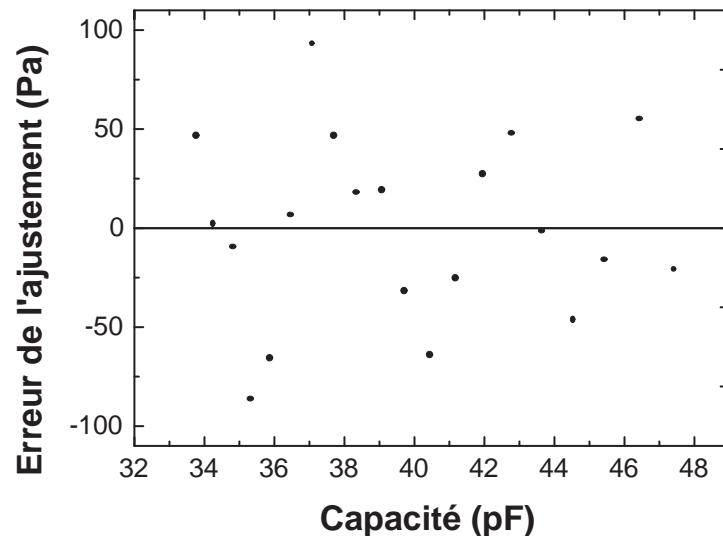
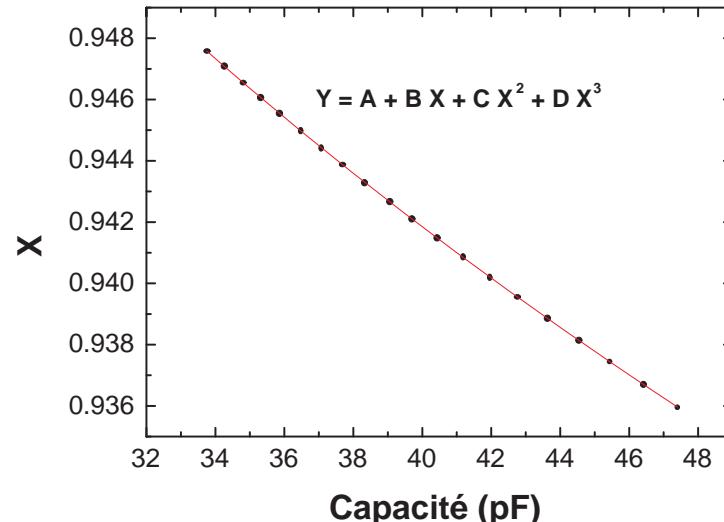
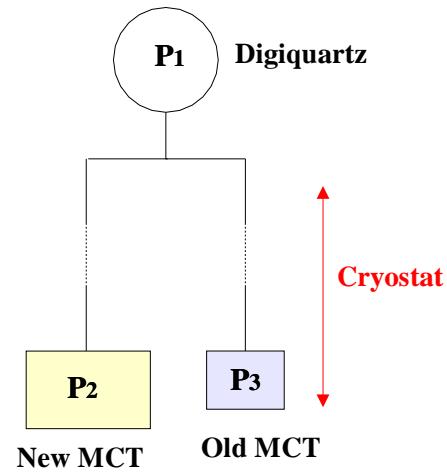
Old MCT:



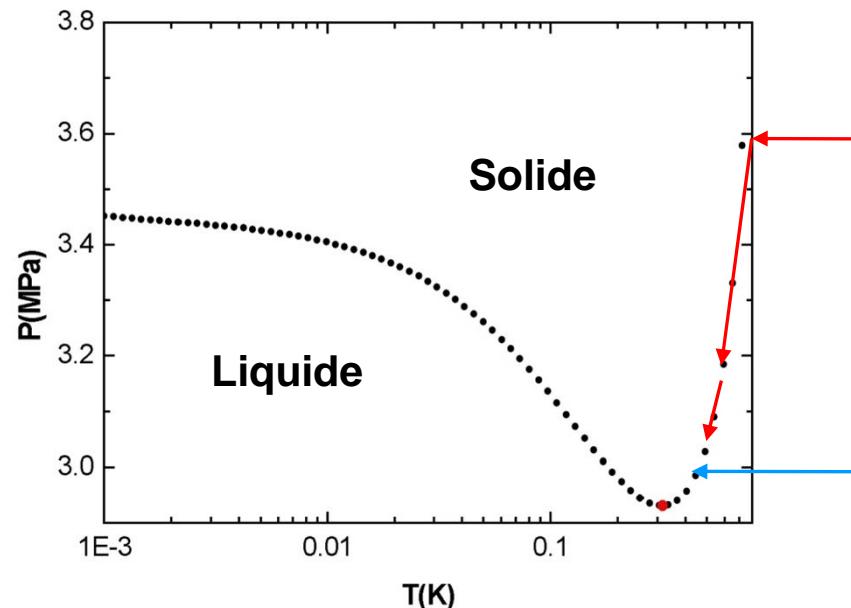
Calibration : the importance of pressure equilibrium

- Sensitivity and accuracy of the pressure gage

- Pressure equilibrium!



Cool-down of the MCT: effect of initial pressure

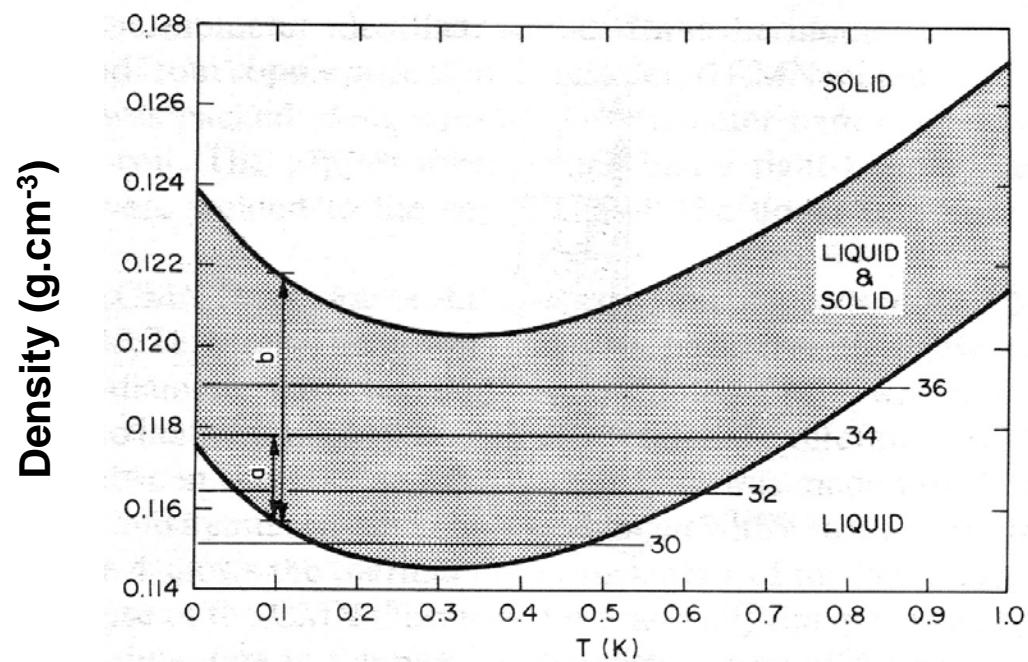


3,6 MPa

Up to 80 % of
solid ^3He in the
thermometer

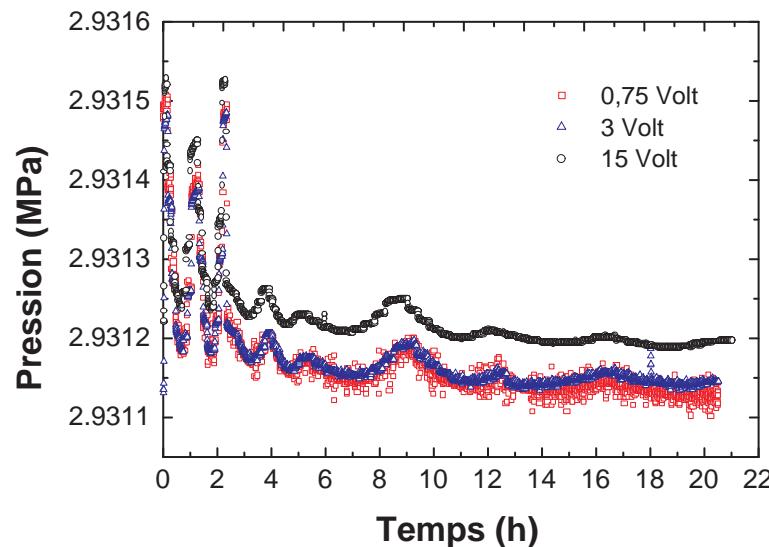
3 MPa

Up to 10 % of
solid ^3He

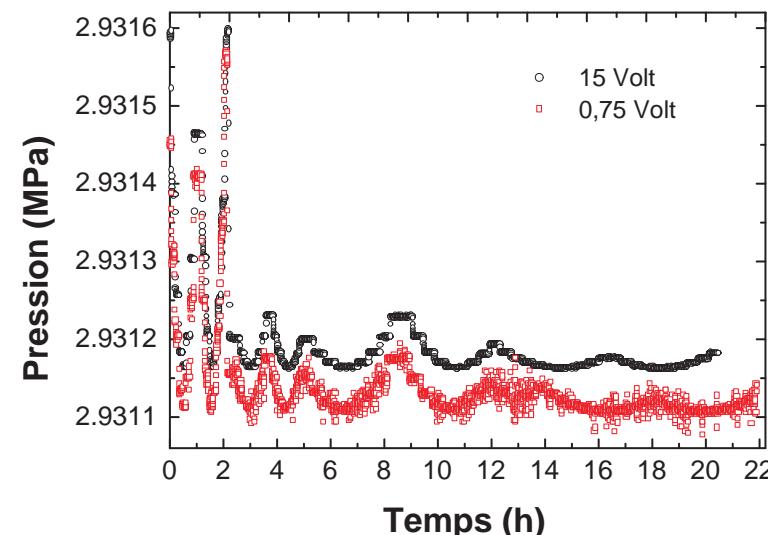
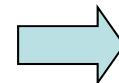


Determination of the minimum of the melting curve

Inaccuracy of the minimum
of the MC determined
with 80 % of solid ^3He



Measurements
with 10 % of solid



L → { The time constant depends on the amount of solid
Variation with voltage: electrostatic force

Hydrostatic pressure correction

- Height of the ${}^3\text{He}$ column during calibration ?

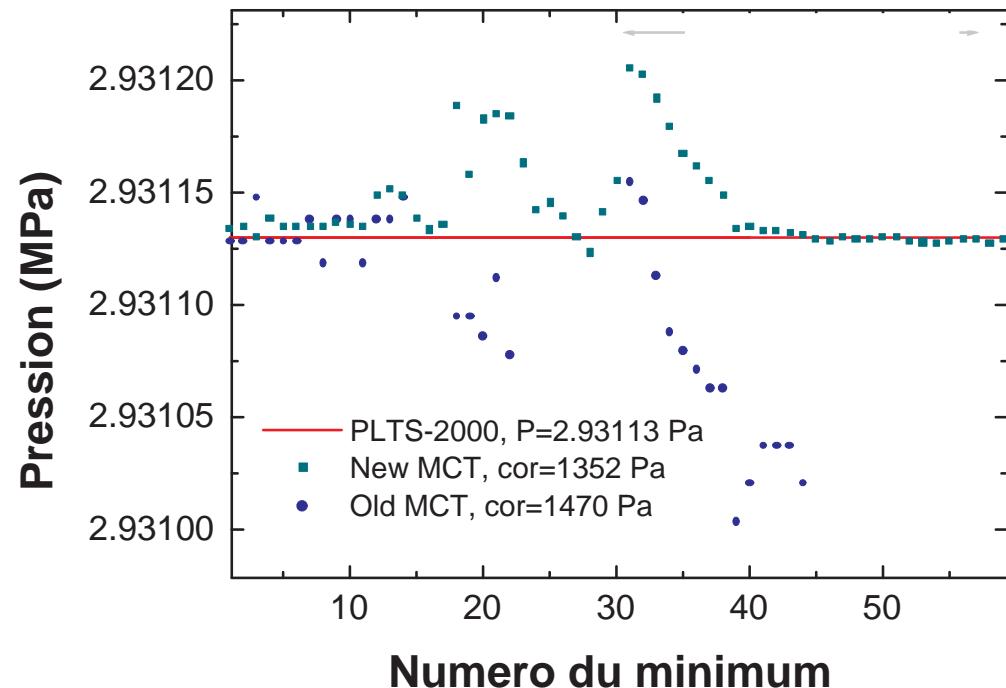


- Average several minima



- Shift the pressures to the PLTS-2000 value:

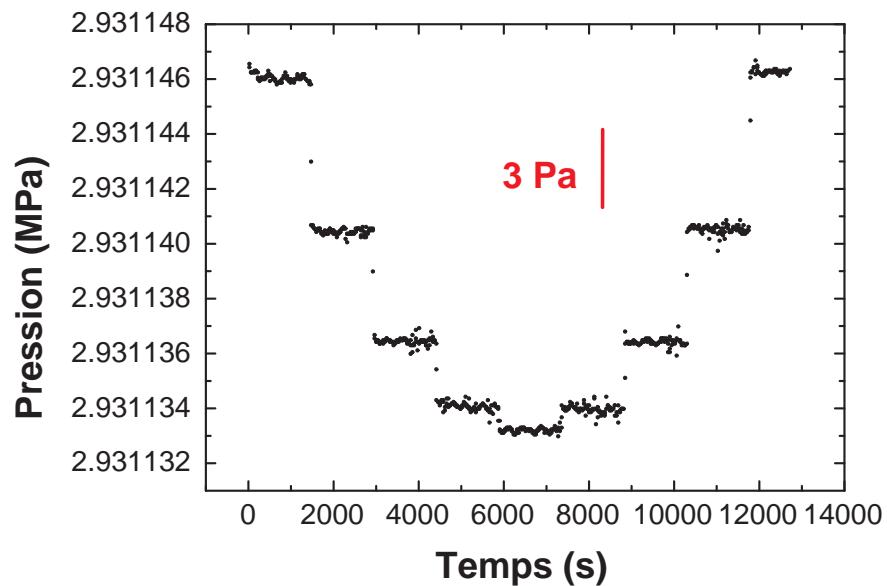
$$P_{\min} = 2,93113 \text{ MPa}$$



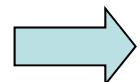
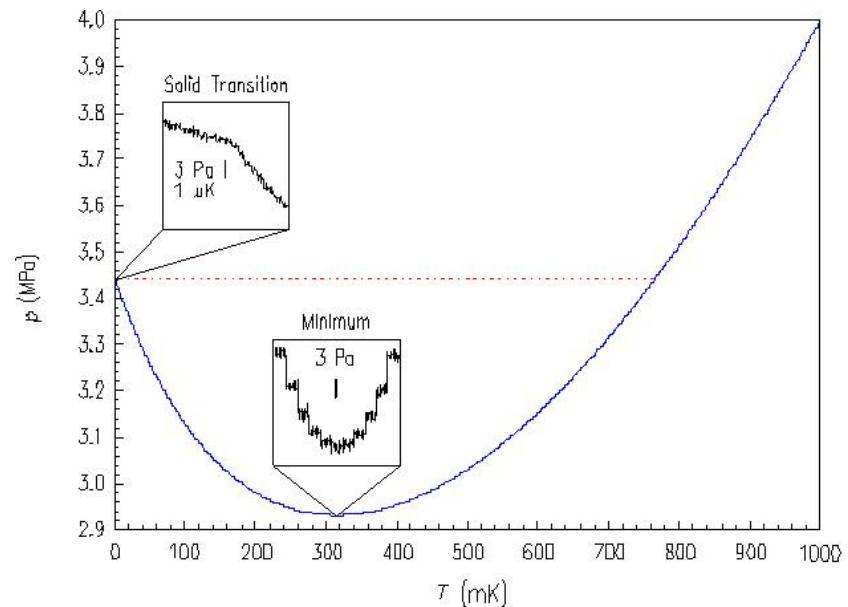
**Correction is 1352 Pa
in our case
height on about 1 m**

Accuracy of the minimum pressure

Our measurement



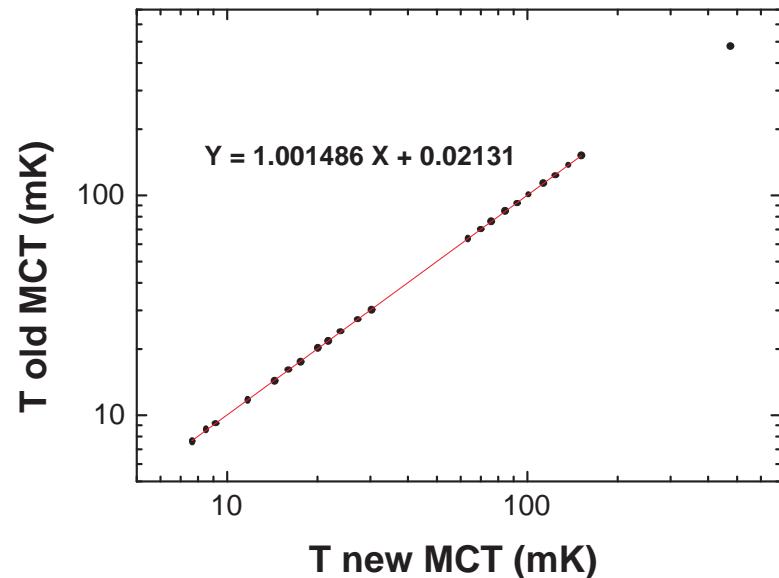
PLTS-2000 (PTB)



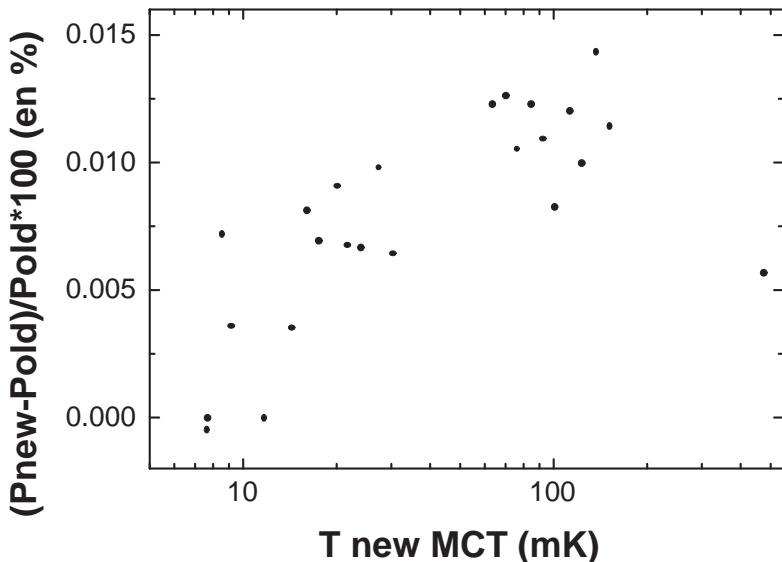
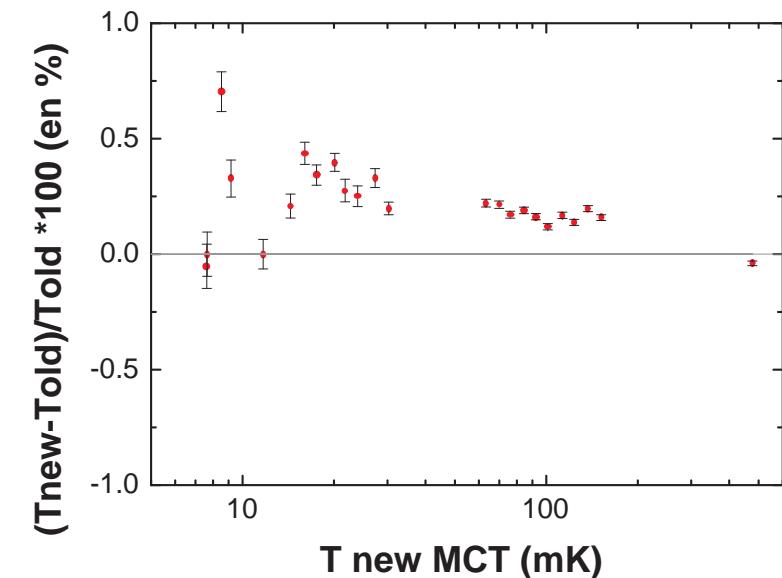
Very high relative accuracy

Absolute accuracy possible

Comparing two thermometers



Maximum difference : 0,015 %
1-sigma uncertainty in
temperature : about 0,3 %



Several cool-downs, comparison of the MCT (recalibrated each time)
with the calibrated carbon resistor

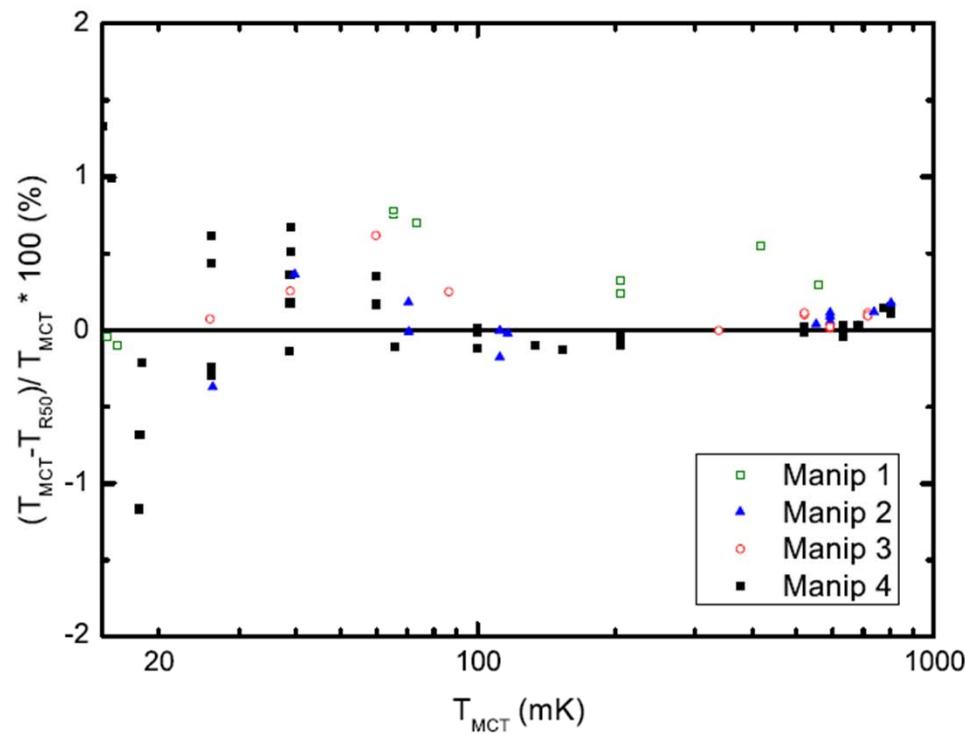


FIG. 2.17 – Ecart entre la température du MCT et de R50 pour différentes mises à froid.

Reproducibility of the pressure calibration of the MCT

Pressure calibration at each cool-down is really needed!!!

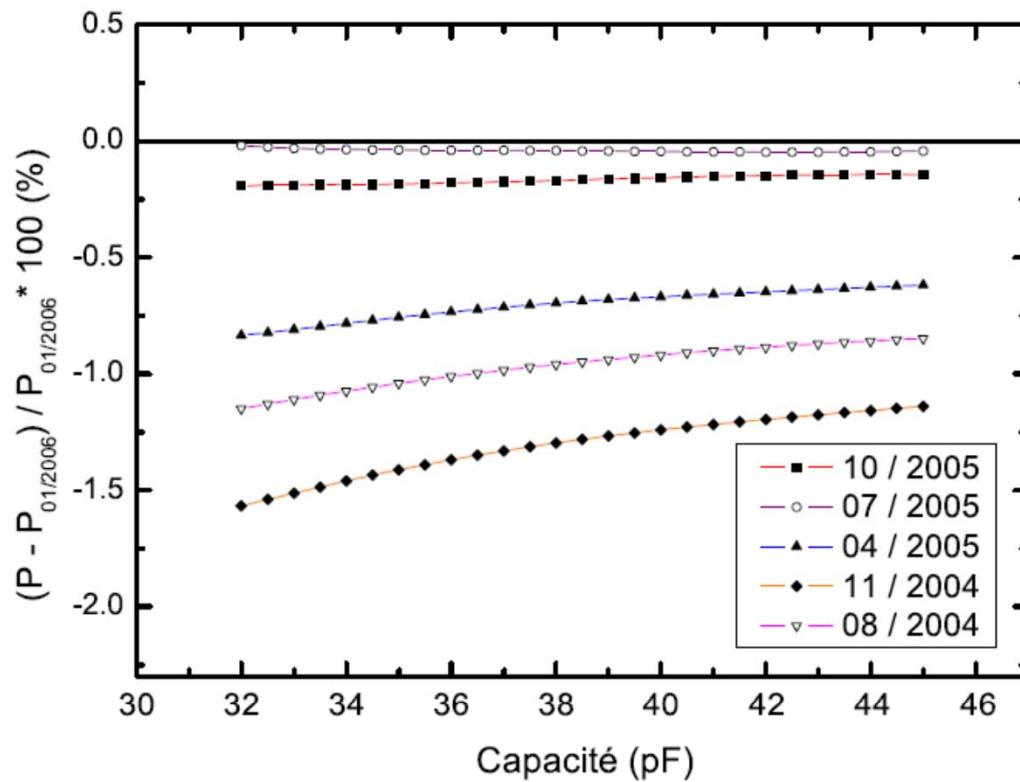


FIG. 2.6 – Ecart entre différents étalonnages réalisés depuis la fabrication du thermomètre à courbe de fusion par rapport à l'étalonnage de janvier 2006. L'erreur pourrait atteindre jusqu'à 2 % si l'on ne réalisait pas l'étalonnage à chaque refroidissement.

Conclusions

Summary

- Design and construction of a melting curve thermometer
- Pressure calibration of the MCT
- Realization of the PLTS-2000 temperature scale
- Identification of the possible instrumental errors
- Check with two thermometers, different amounts of solid, different measuring conditions

Conclusions

- The MCT requires a rather complex procedure
- It is very sensitive, accurate, and independent on the magnetic field