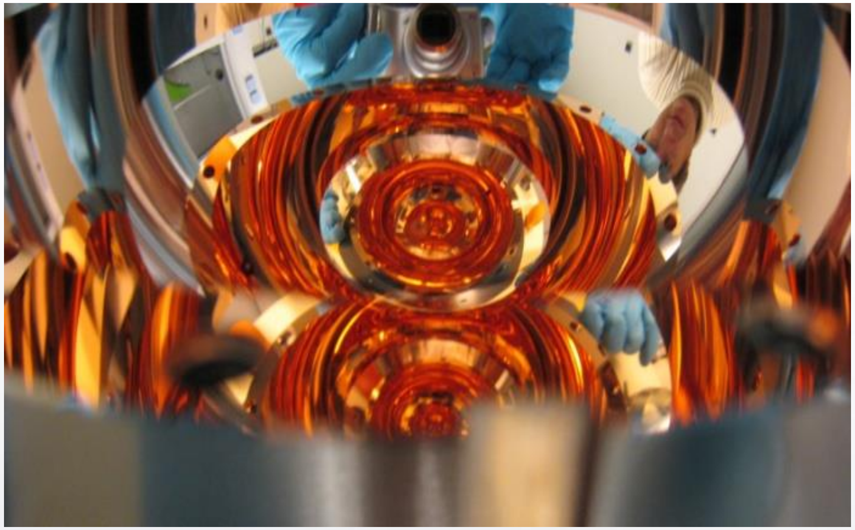


Résonateur micro-onde quasi sphérique: de la détermination de la constante de Boltzmann à la mesure primaire de la pression.

Laurent Pitre (LNE- Cnam)

Laboratoire commun de métrologie LNE-CNAM

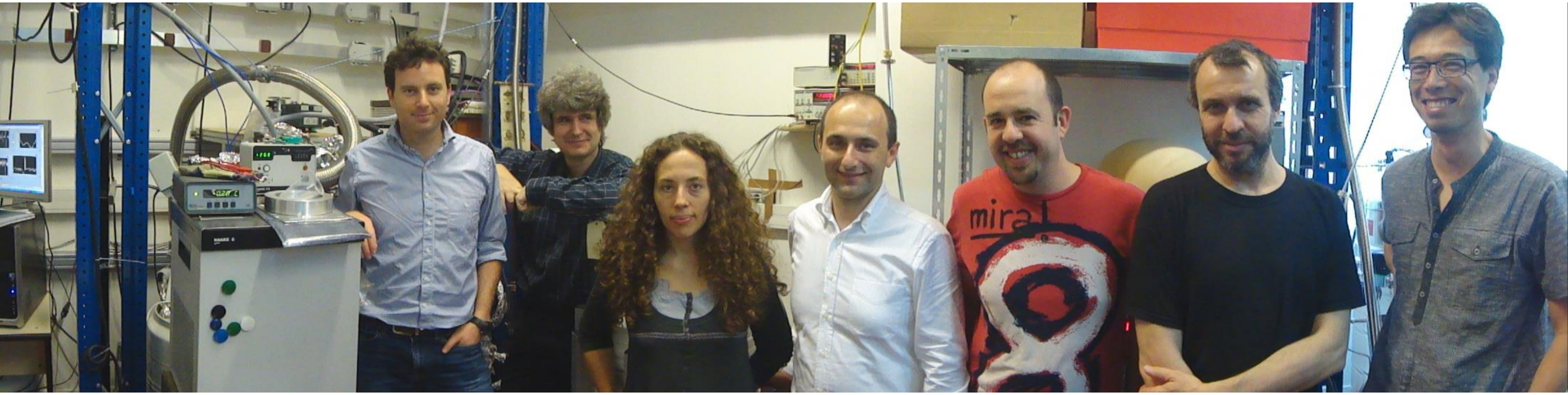
Cnam

LNE

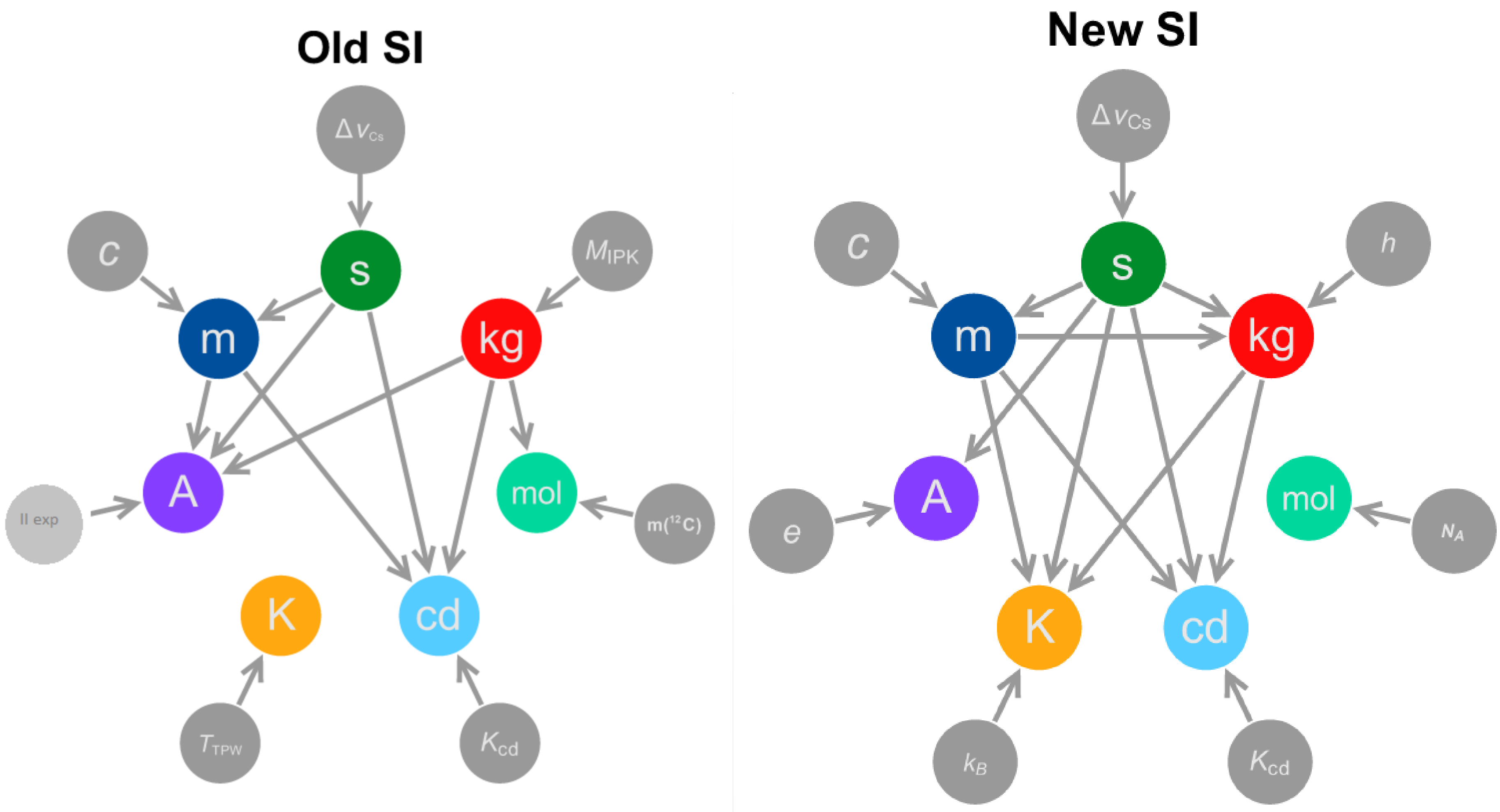
Creation du CNAM 1794
Dépôt des étalons du système metrique 1848

Creation du LNE 1901

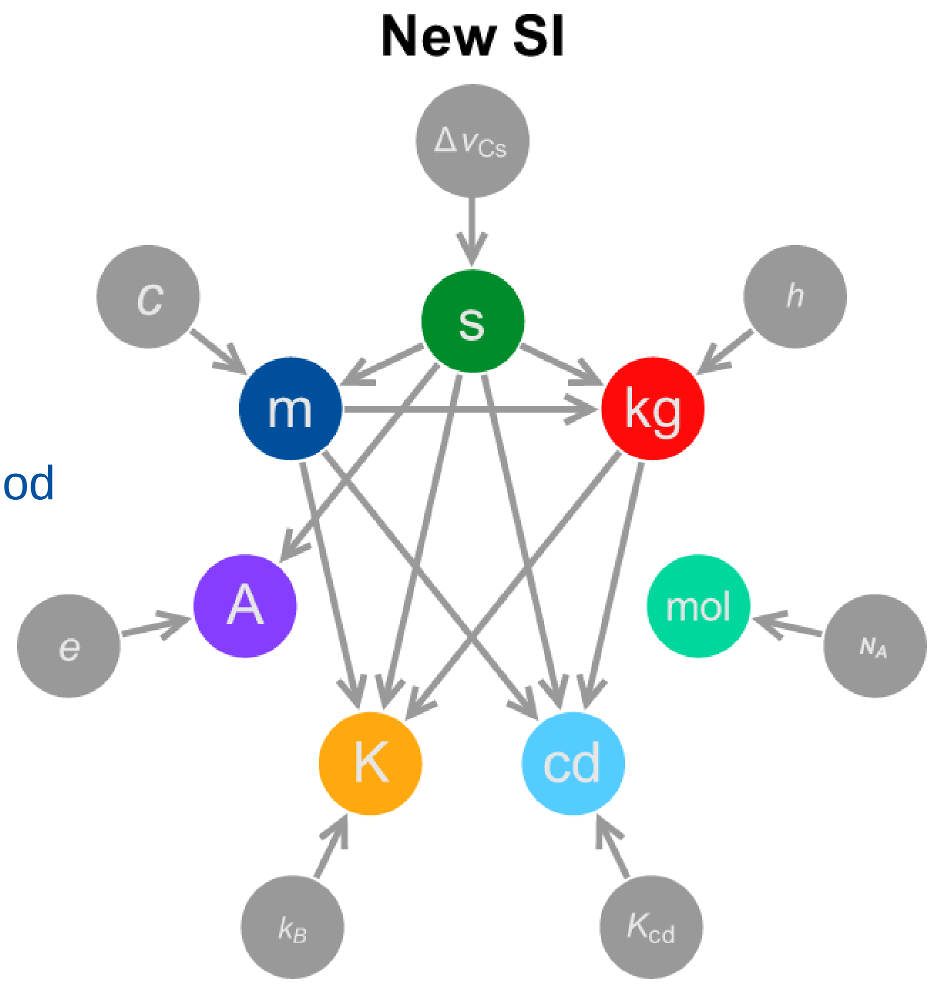
Une Equipe



Les unités de base du SI d'aujourd'hui et demain



Link to fundamental constant
Measurement traceable over very long period
Will be official the 20th may 2019



Les déterminations de la Constant de Boltzmann au CODATA 2014

LNE09 **Helium** gas, 0.5 liters resonator, hand polished inner

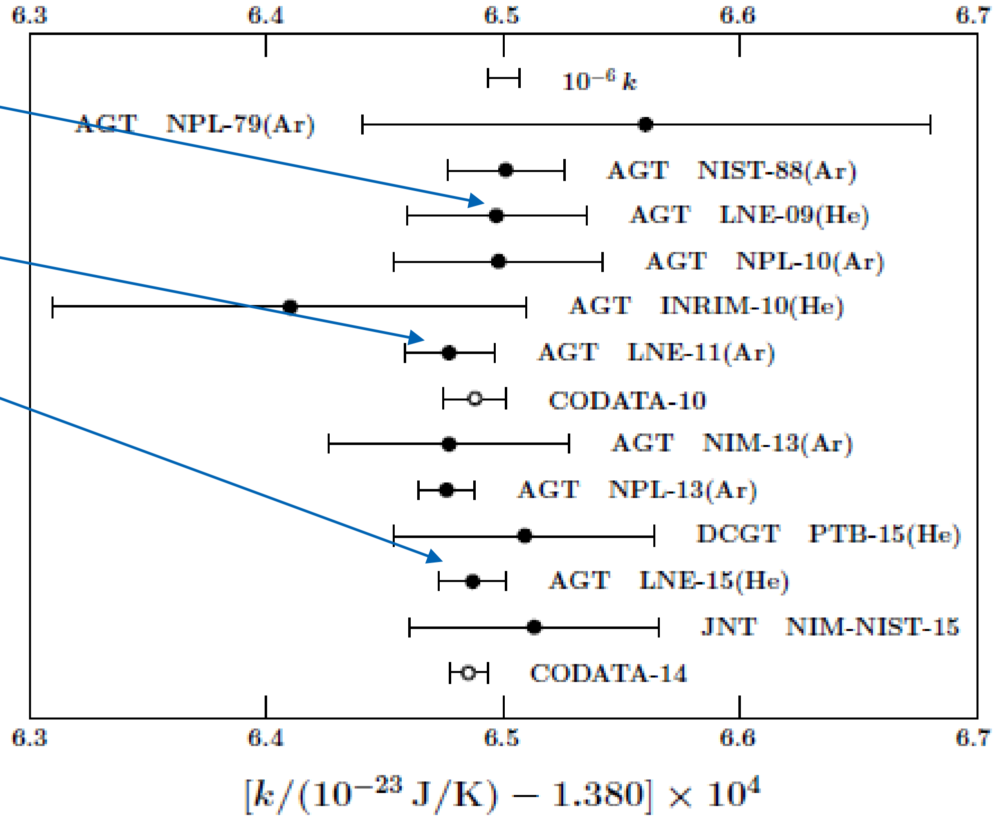
$k=(1.3806495+0.0000037)*10^{-23}$J/K

LNE13 **Argon** gas, 0.5 liters resonator, surface diamond turned

$k=(1.3806477+0.0000017)*10^{-23}$J/K

LNE15 **Helium** gas, 0.5 liters resonator, surface diamond turned

$k=(1.3806485+0.0000014)*10^{-23}$J/K



CODATA 2014

$k=1.380\ 648\ 52(79) \times 10^{-23}$J/K

From CODATA Recommended Values of the Fundamental Physical Constants:2014

page 52

Le LNE a, en moyenne pondéré, la détermination de k avec la plus petite incertitude au monde, donnée prise en compte par le CODATA 2014

En cour d'analyse

LNE17 **Helium** gas, 3 liters resonator, surface diamond turned

$k=(1.38064XX+0.0000009)*10^{-23}$J/K

PLAN

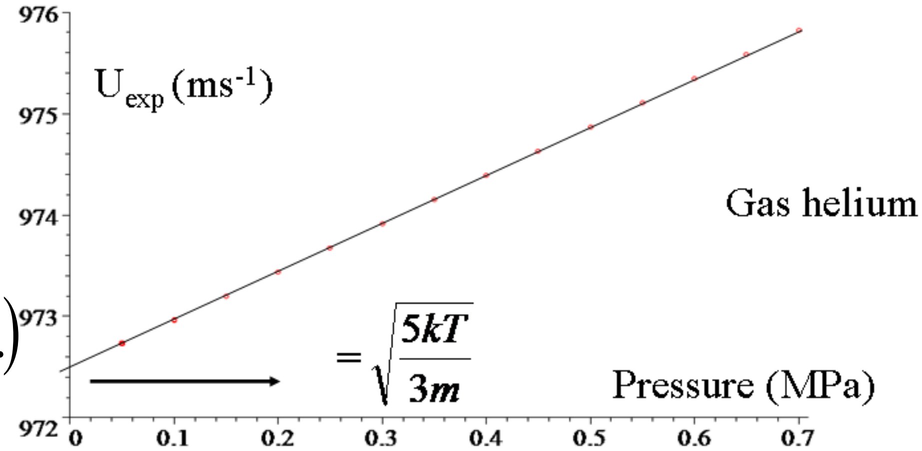
- La determination de la constant de Boltzmann au LNE-CNAM
- mesure primaire de la pression à l’aide de résonateur supraconducteur

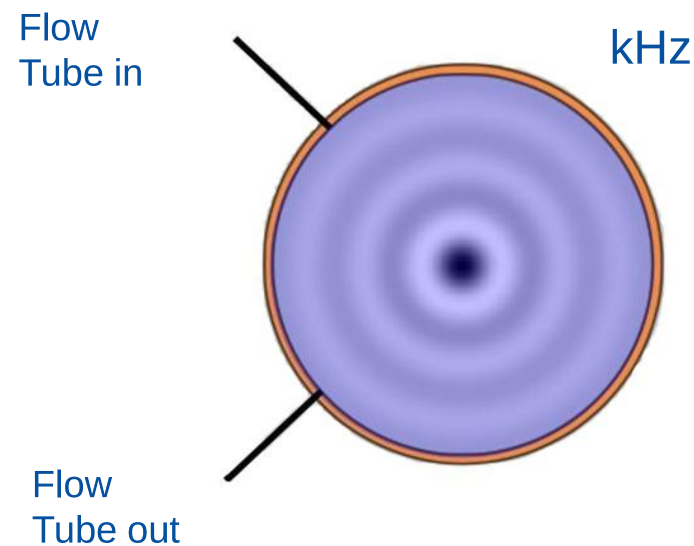
Principle of the experiment

For real gas:

$$u_A^2 = \gamma_{pg} \frac{RT}{M_{^4\mathrm{He}}} \left(1 + \beta_a(T)\rho + \gamma_a(T)\rho^2 \ldots\right)$$

- universal gas constant (R)
- zero pressure limit of specific heats ratio (γ_{pg})
- ^{4}He molar mass ($M_{^4\mathrm{He}}$)
- acoustic virial coefficients from *ab initio* calculations (β_a, γ_a)





- Acoustic resonances measurement

  Boltzmann constant but linked to a volume

- Microwave resonances measurement

  Volume measurement

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

Relationship between the Boltzmann constant and acoustic/microwave measurements

$$k = \left\langle \frac{3}{5} \frac{m c_0^2}{T_{tp,water}} \left(\frac{Z_{nl}^{EM}}{Z_{nl}^{A}} \right)^2 \lim_{p \to 0} \left(\frac{< f_{nl}^{A} + \Delta f_{nl}^{A} >}{< f_{nl}^{EM} + \Delta f_{nl}^{EM} >} \right)^2 \right\rangle$$

Gas atomic mass

Speed of light in vacuum (exact)

Measured resonance frequency

Correction (theory)

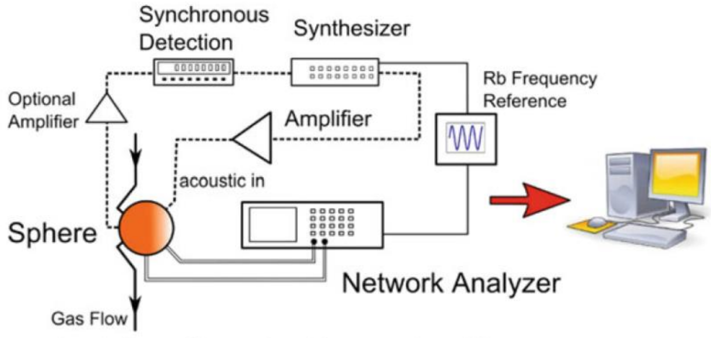
Quasi-sphere's eigenvalues

Polynomial extrapolation to Zero pressure limit

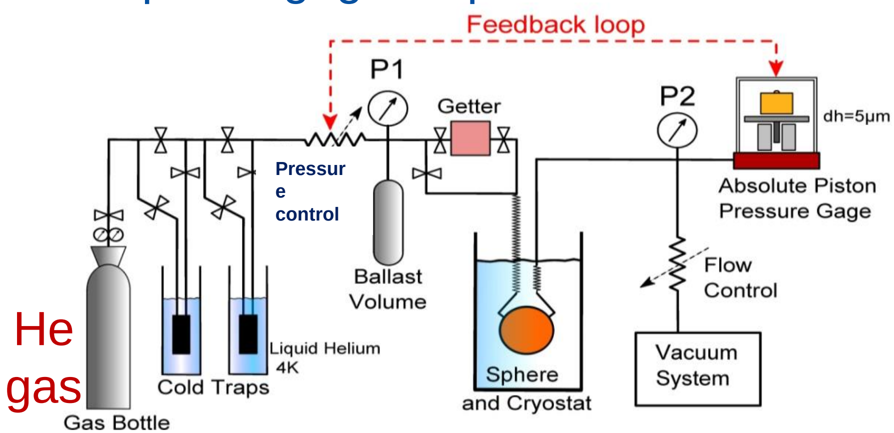
Average over measured acoustic and electromagnetic modes

Ratio: removes artefact effects at the first order

20 Instruments used at the state of the art

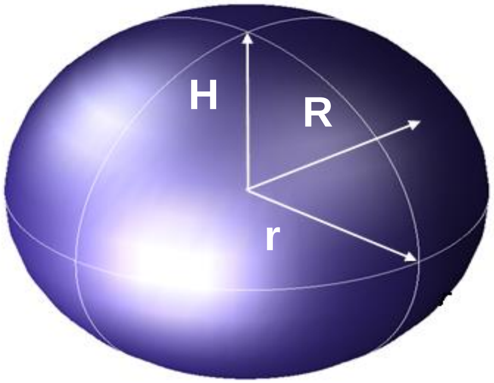


Ultra clean gas handling systems
With a piston gage as pressure measurement

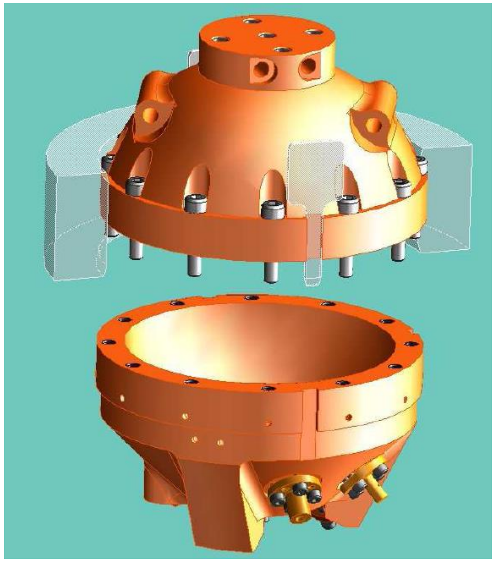


Adiabatic Cryostat
(weak link to the thermal bath)

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



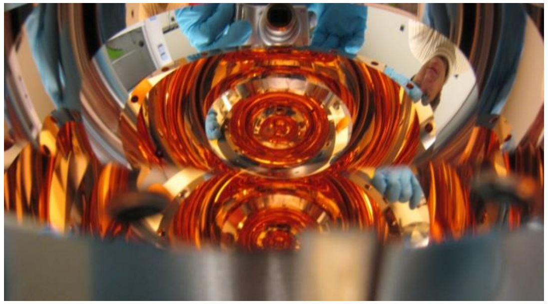
$$\frac{x^2}{(49.950)^2}+\frac{y^2}{(49.975)^2}+\frac{z^2}{(50.000)^2}=1$$



0.5 L diamond turned resonator

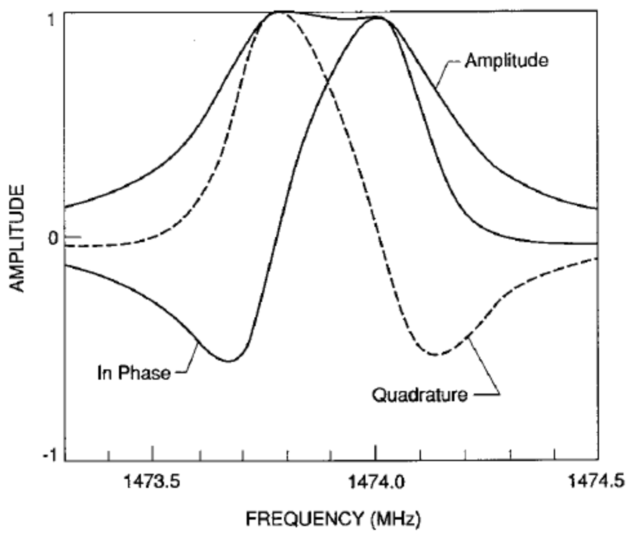
Inner shape: the difference between r, R and H is 0.025 mm:

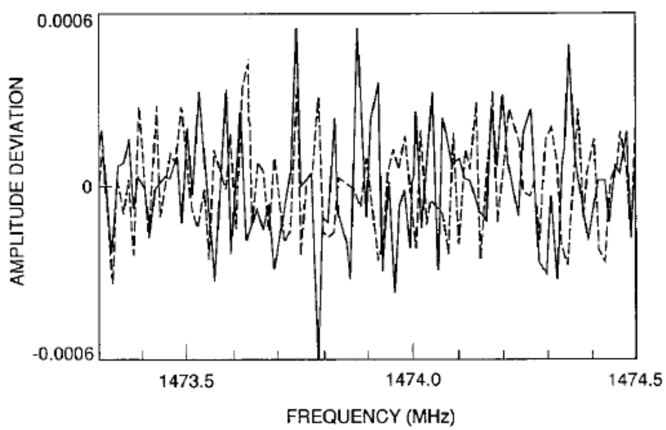
H = 50.000 mm
R = 49.975 mm
r = 49.950 mm

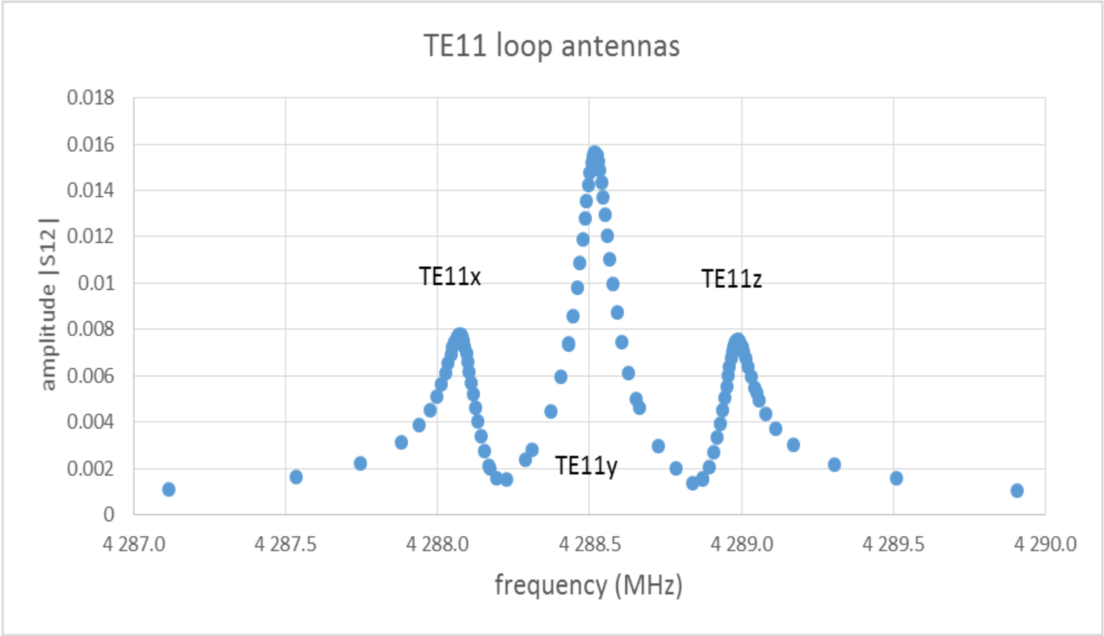


$$k=\left\langle \frac{3}{5}\frac{mc_0^2}{T_{tp,water}}\left(\frac{Z_{nl}^{EM}}{Z_{nl}^{A}}\right)^2 \lim_{p\to 0}\left(\frac{< f_{nl}^{A}+\Delta f_{nl}^{A} >}{< f_{nl}^{EM}+\Delta f_{nl}^{EM} >}\right)^2\right\rangle$$

A Non-Quite Spherical Cavity







3 over 3 resonances observed
in a quasi sphere

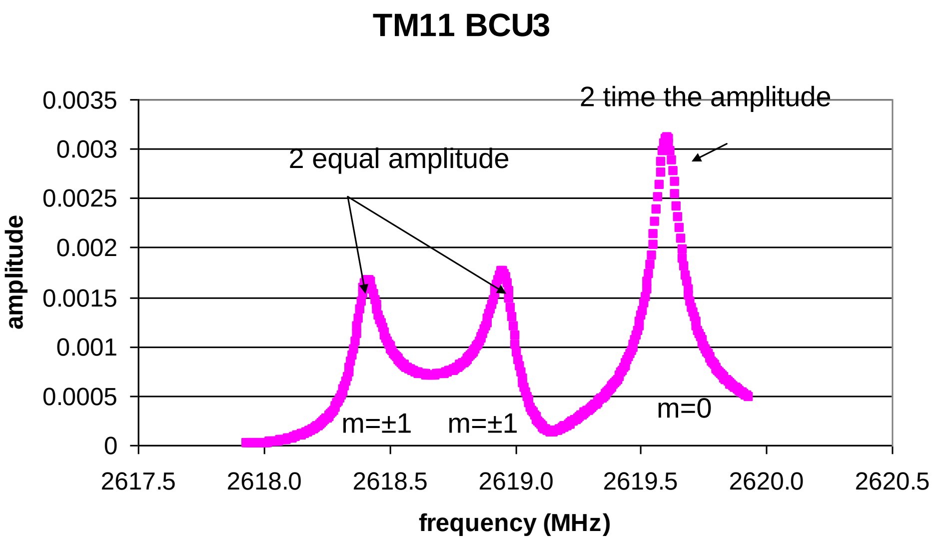
Only 2 over 3 resonances
observed in a “perfect” sphere

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

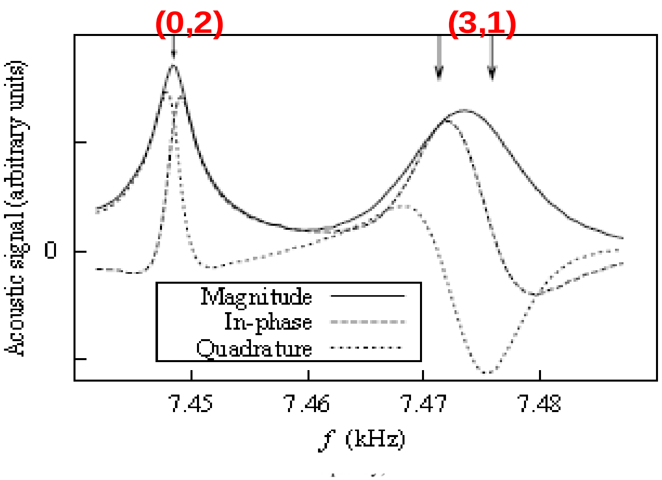
A Non-Quite Spherical Cavity

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

Electromagnetic measurements in very good agreement with the theoretical model



Acoustic measurements in a good agreement with the theoretical model



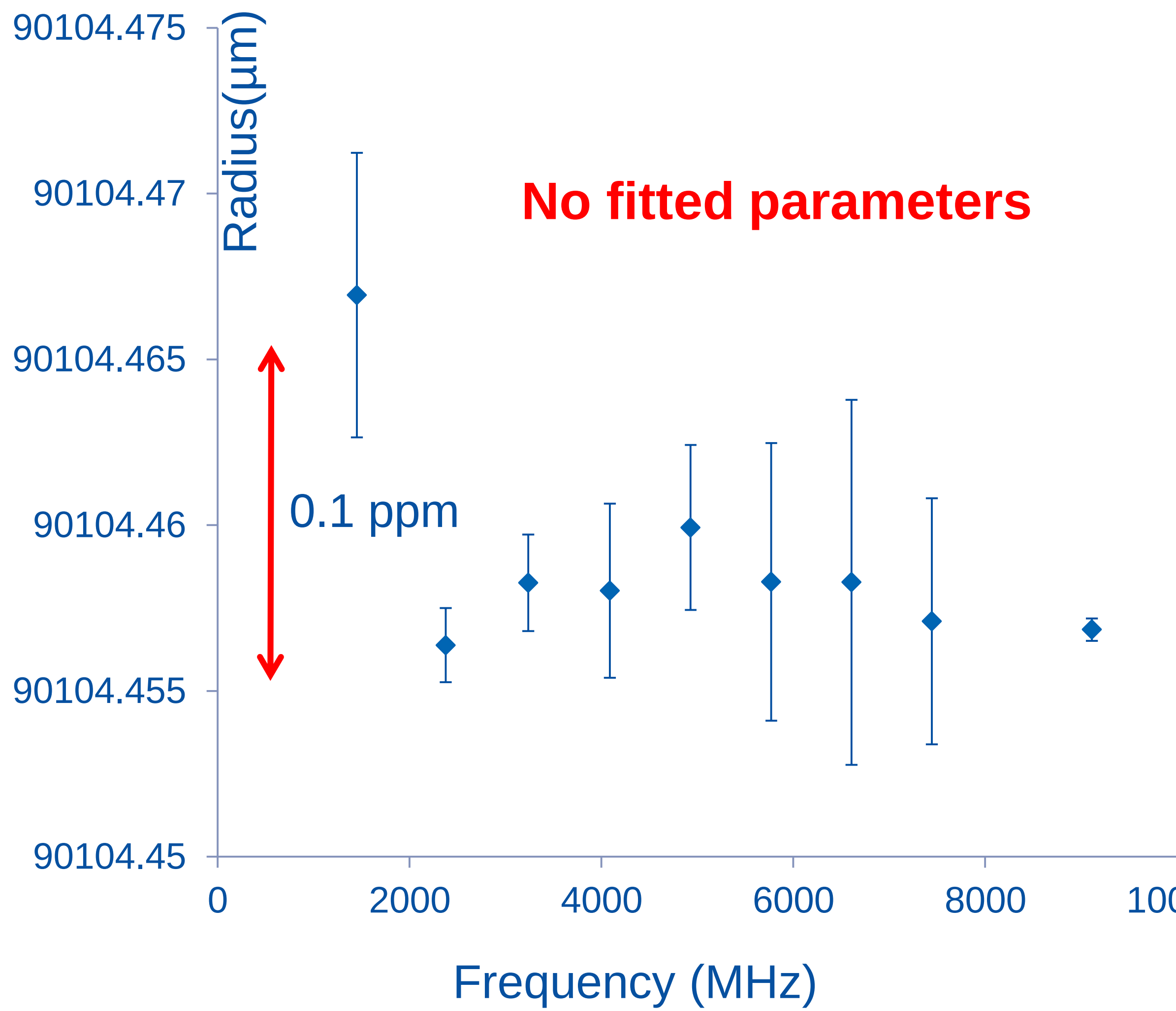
$$k = \left\langle \frac{3}{5}\frac{mc_0^2}{T_{tp,water}} \left(\frac{Z_{nl}^{EM}}{Z_{nl}^{A}}\right)^2 \lim_{p\to 0}\left(\frac{< f_{nl}^{A} + \Delta f_{nl}^{A} >}{< f_{nl}^{EM} + \Delta f_{nl}^{EM} >}\right)^2 \right\rangle$$

Eigenvalue — Speed of light

$$Radius = \frac{Z_{nl}^{EM} c}{2\pi < f_{nl}^{EM} + \Delta f_{nl}^{EM} >}$$

Resonance frequency

Skin depth
Holes and
Antennas effect



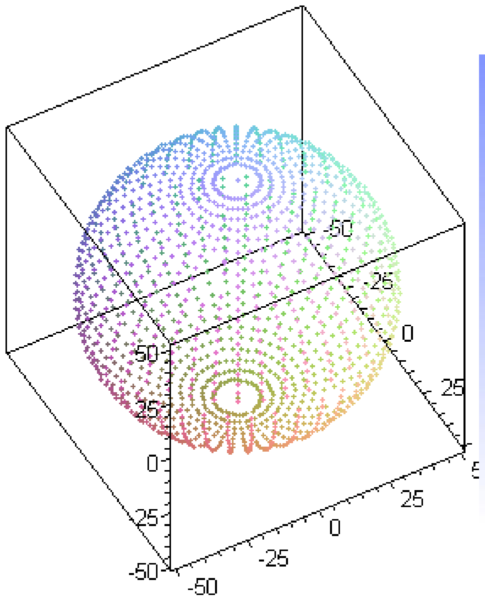
3.1 liters
Copper diamond turn quasi spherical resonator

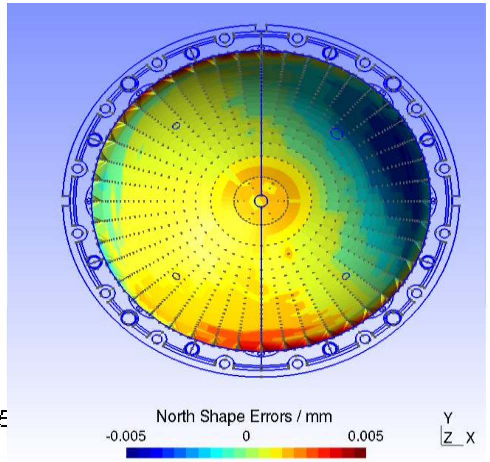
Mehl J B 2009 Second-order electromagnetic eigenfrequencies of a triaxial ellipsoid *Metrologia* **46** 554–9

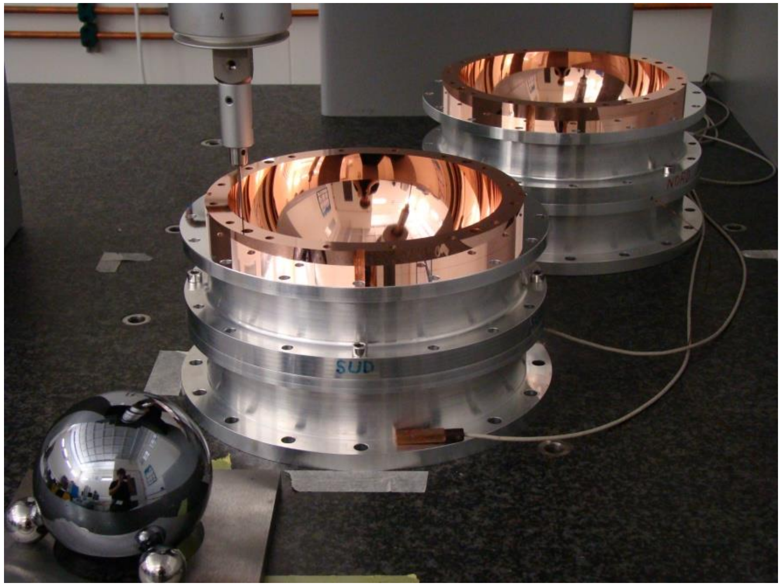
$$k_B = \left\langle \frac{3}{5}\frac{mc_0^2}{T_{tp,water}}\left(\frac{Z_{nl}^{EM}}{Z_{nl}^{A}}\right)^2 \lim_{p\to 0}\left(\frac{< f_{nl}^{A} + \Delta f_{nl}^{A} >}{< f_{nl}^{EM} + \Delta f_{nl}^{EM} >}\right)^2 \right\rangle$$

Measurement of the Volume

- Comparison of the microwave technique to CMM measurements
- Use of a CMM as a comparator
- Cooperation between LNE-CNAM, NPL, INRiM, UWA, Jim Mehl



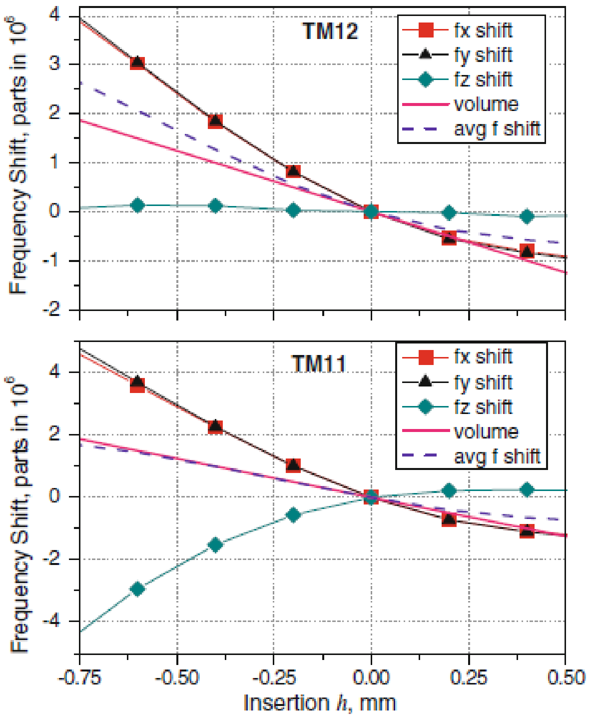


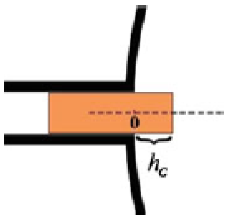


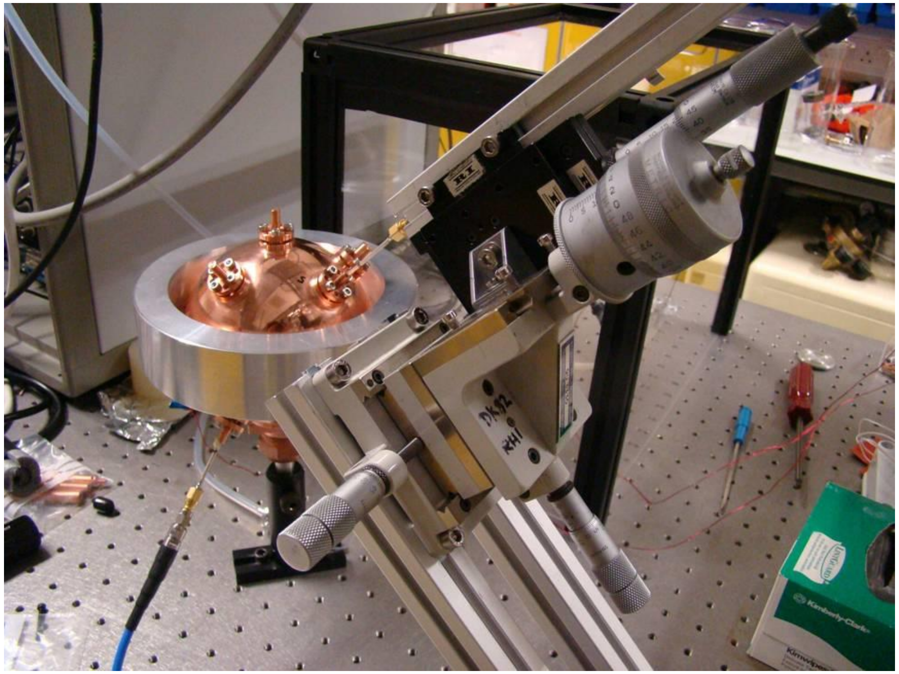
M. de Podesta, E. F. May, J. B. Mehl, L. Pitre, R. M. Gavioso, G. Benedetto, P. A. Giuliano Albo, D. Truong and D. Flack. Metrologia, 47, 588-604, (2010)

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

Holes and Antennas effect



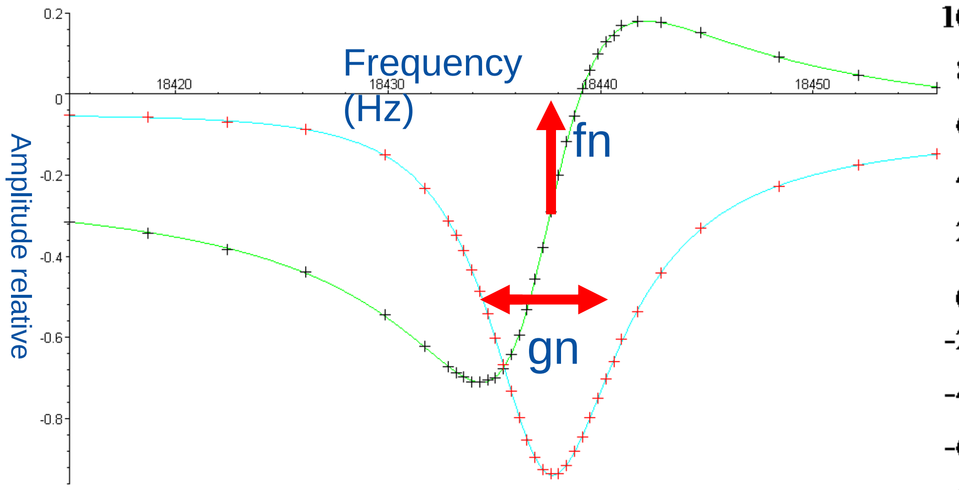


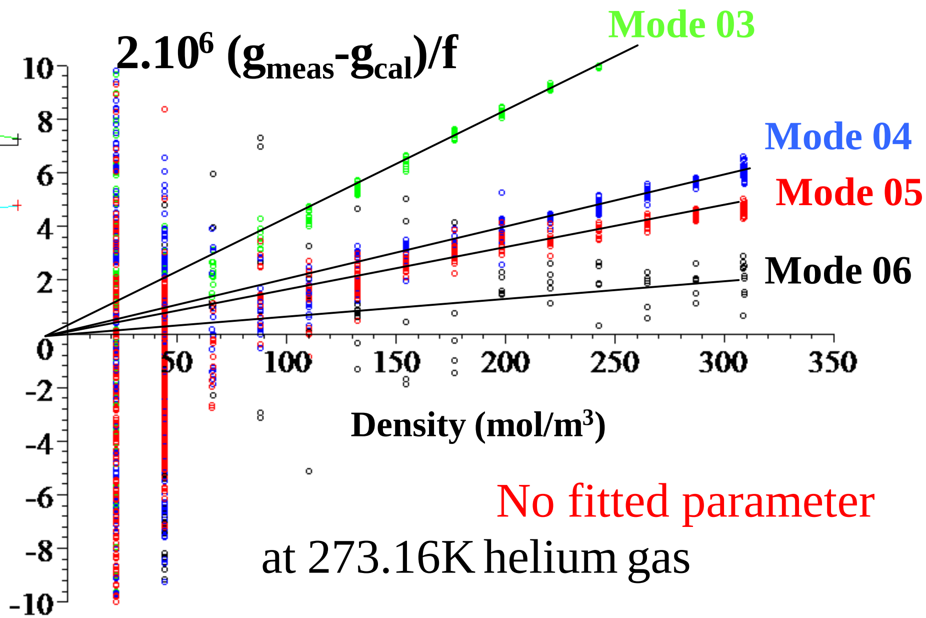


Underwood R J, Mehl J B, Pitre L, Edwards G, Sutton G and de Podesta M 2010 Waveguide effects on quasispherical microwave cavity resonators *Meas. Sci. Technol.* **21** 075103

$$k_B = \left\langle \frac{3}{5} \frac{m c_0^2}{T_{tp,water}} \left(\frac{Z_{nl}^{EM}}{Z_{nl}^{A}} \right)^2 \lim_{p \to 0} \left(\frac{< f_{nl}^{A} + \Delta f_{nl}^{A} >}{< f_{nl}^{EM} + \Delta f_{nl}^{EM} >} \right)^2 \right\rangle$$

Comparison between measured half-width and calculated from thermal physical propriety of helium gas and acoustic model





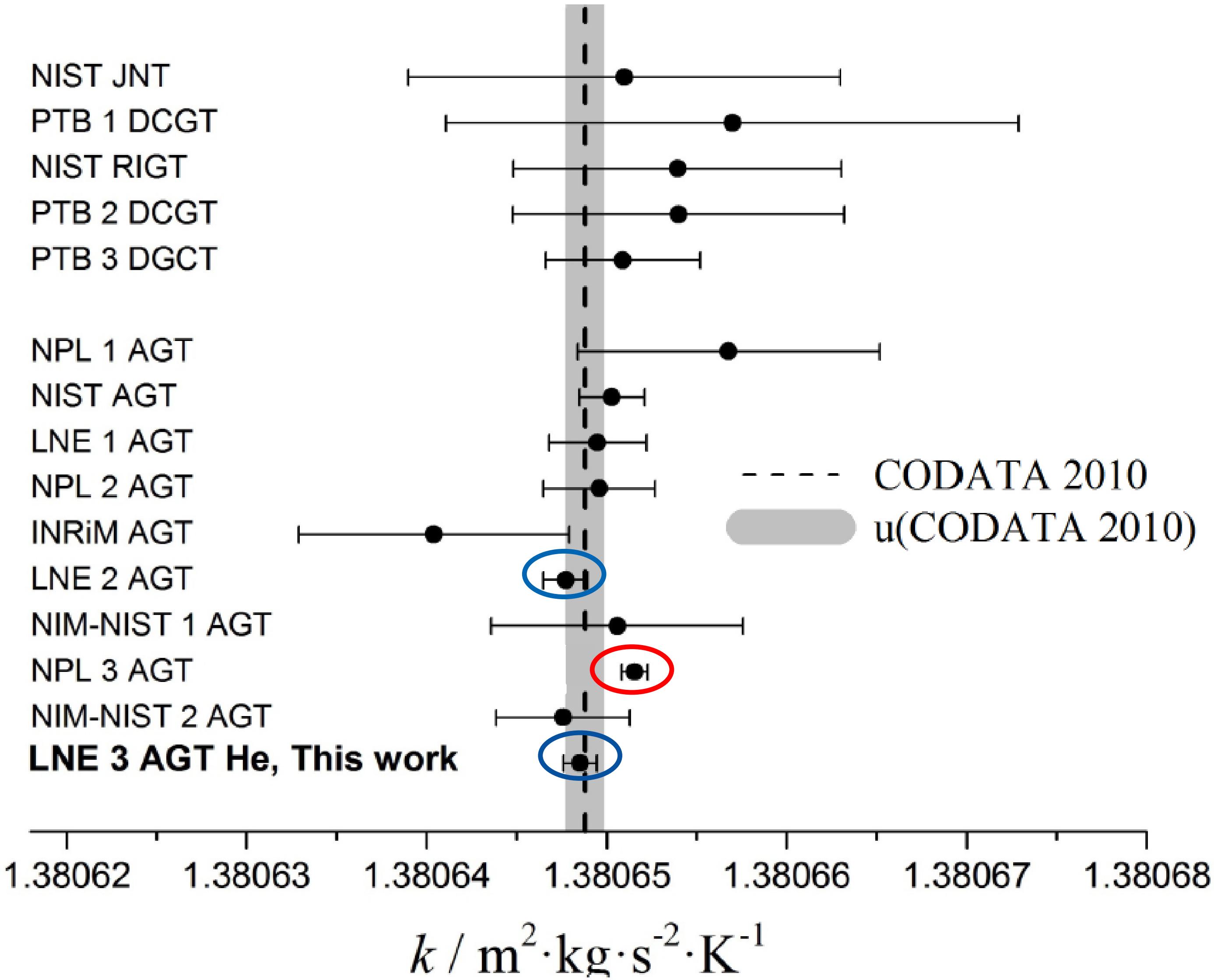
$$k_B = \left\langle \frac{3}{5}\frac{mc_0^2}{T_{tp,water}}\left(\frac{Z_{nl}^{EM}}{Z_{nl}^{A}}\right)^2 \lim_{p\to 0}\left(\frac{< f_{nl}^{A} + \Delta f_{nl}^{A} >}{< f_{nl}^{EM} + \Delta f_{nl}^{EM} >}\right)^2 \right\rangle$$

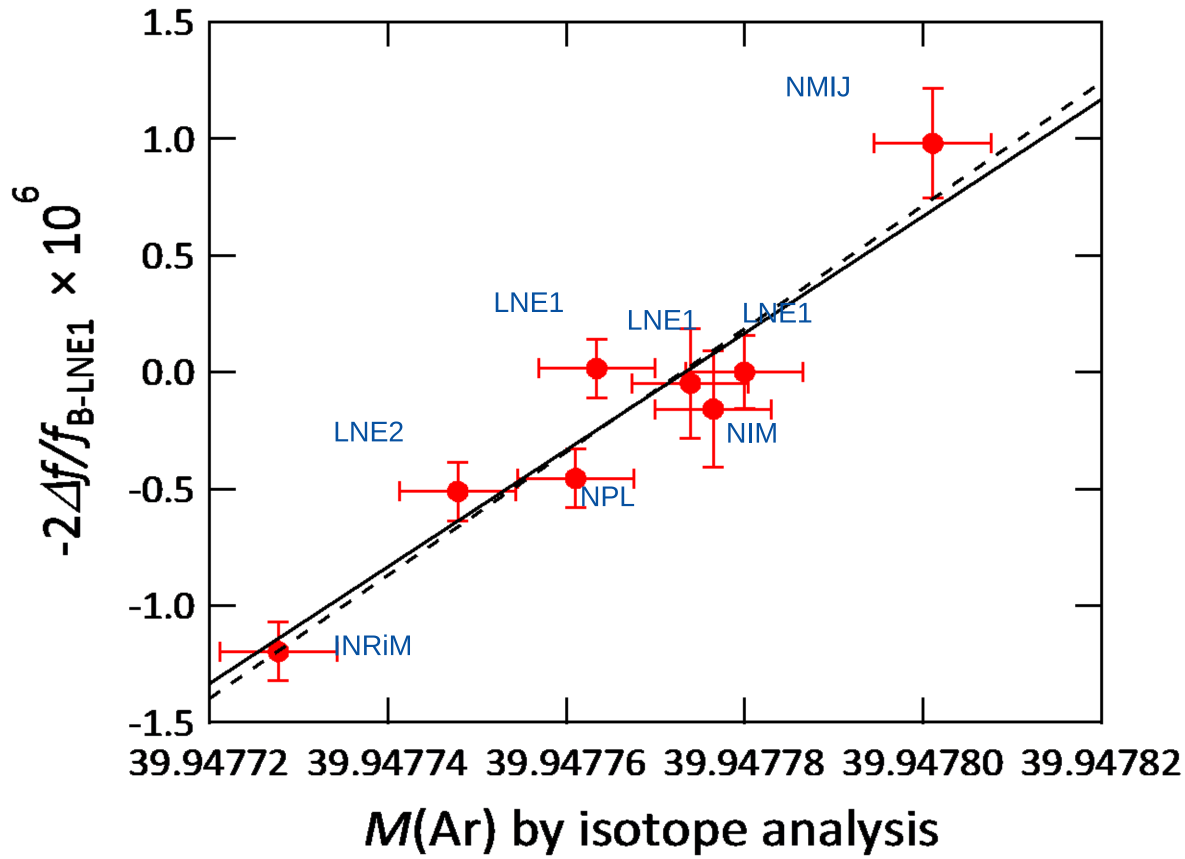
Uncertainty budget on the Boltzmann constant with 3.1 liters cavity in helium

Uncertainty on *k*

	ppm of *k*	March 2017
Volume	**0.19**	Holes and antenna effect, Dispersion over mode-shape, Conductivity, Uncertainty in frequency measurements
Temperature	**0.3**	Calibration, Dispersion over thermometers, Uncertainty in resistance measurements
Molecular weight	**0.3**	Isotopic ratio, Cold trap experiment, Getter experiment, Impurity, Uncertainty in measurements
Zero-pressure limit of $(f_n + \Delta f_n)^2$	**0.51**	Thermophysical properties of argon, Scatter among modes, Accommodation coefficient dispersion,Flow, Tubing acoustic impedance, Shell
Repeatability	**0.05**	Two isotherms
Root of Sum of Squares	**0.69**	

Uncertainty budget on the Boltzmann constant

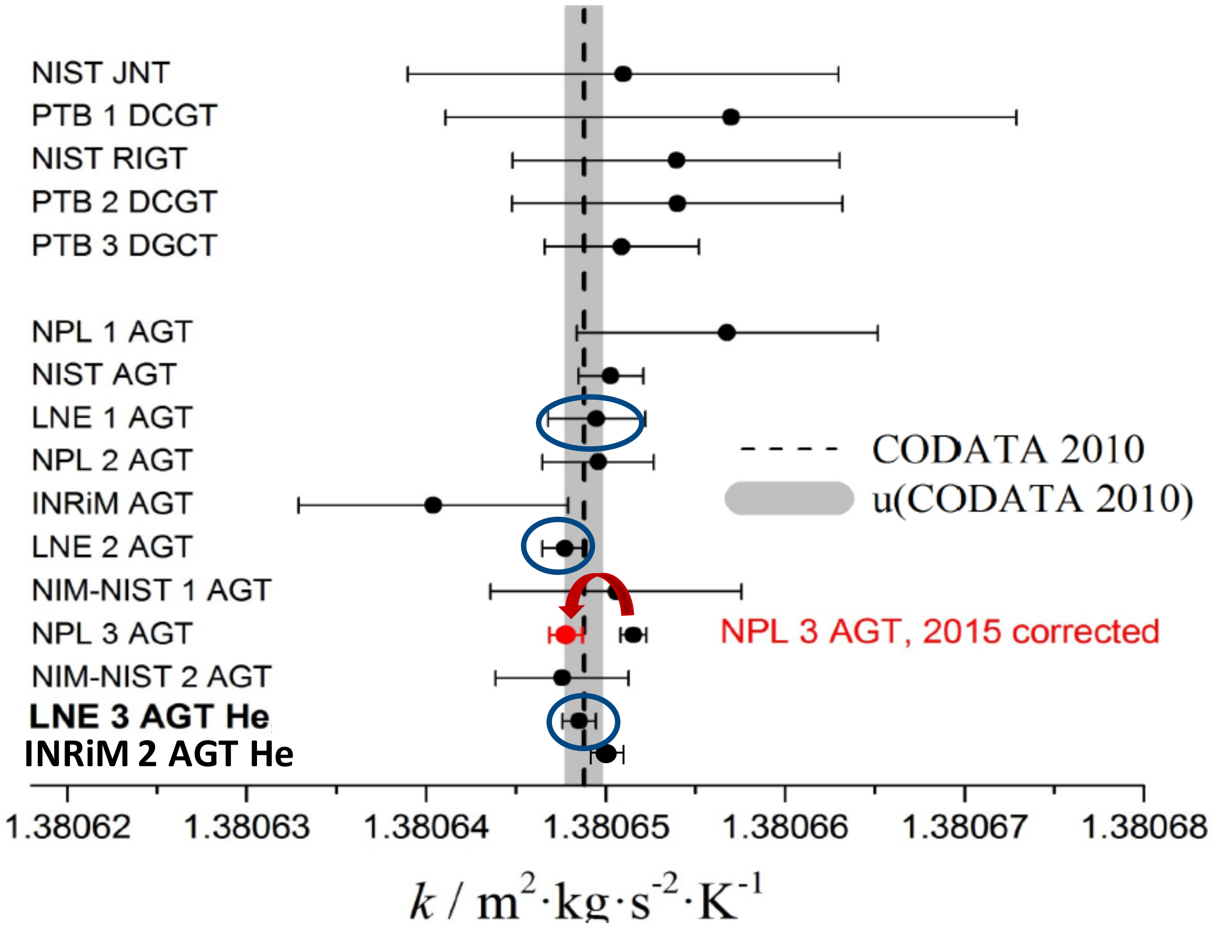




Blind measurement

$$k = \left\langle \frac{3}{5} \frac{m c_0^2}{T_{tp,water}} \left(\frac{Z_{nl}^{EM}}{Z_{nl}^{A}} \right)^2 \lim_{p \to 0} \left(\frac{< f_{nl}^{A} + \Delta f_{nl}^{A} >}{< f_{nl}^{EM} + \Delta f_{nl}^{EM} >} \right)^2 \right\rangle$$

Avoir confiance dans son incertitude

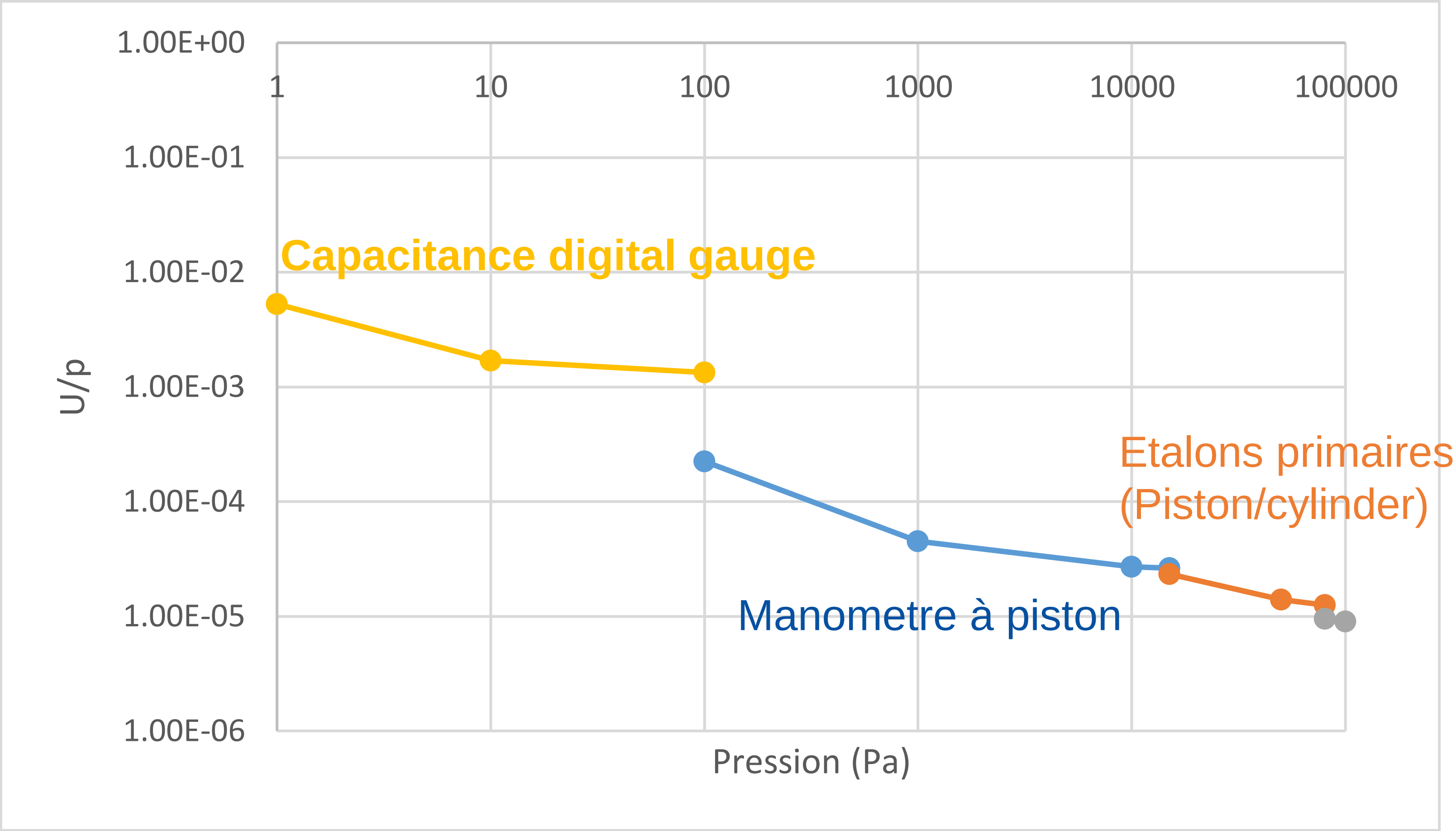


$$k = \left\langle \frac{3}{5}\frac{mc_0^2}{T_{tp,water}} \left(\frac{Z_{nl}^{EM}}{Z_{nl}^{A}}\right)^2 \lim_{p\to 0}\left(\frac{< f_{nl}^{A}+\Delta f_{nl}^{A} >}{< f_{nl}^{EM}+\Delta f_{nl}^{EM} >}\right)^2 \right\rangle$$

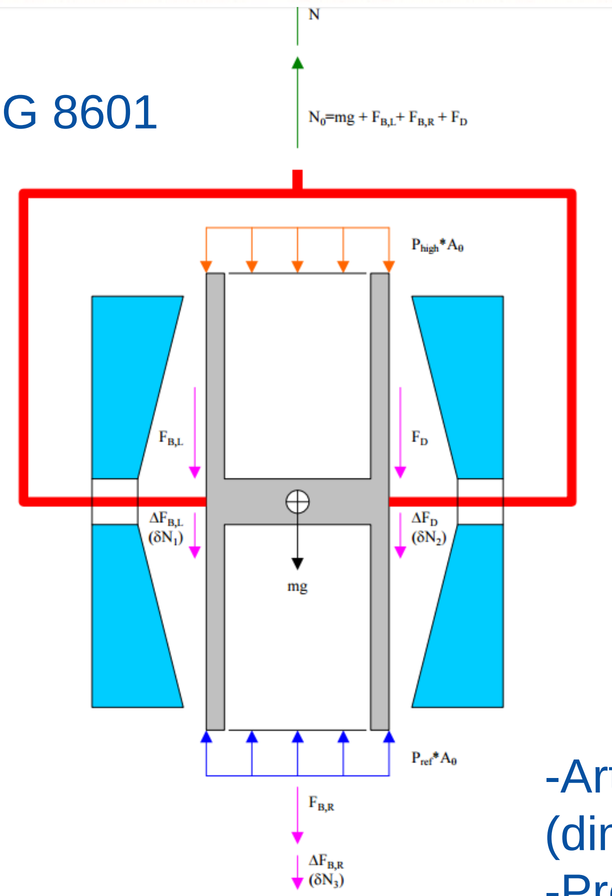
En collaboration avec Roberto Gavioso (INRiM Italie)

État de l'art

Incertitude des références de pression au LNE sur le gamme 1 Pa 100000 Pa



FPG 8601



manomètre à piston basse pression
200 Pa à 20kPa
(U(P) LNE $2.5*10^{-5}*P+2*10^{-2}$)

-Artefact, la surface effective du piston doit être déterminé (dimensionnel ou par comparaison).
-Pression pseudo-statique.
-Gaz dans deux régimes: visqueux et moléculaire.

Les opportunités

Cette nouvelle méthode va tirer parti de quatre avancées :

- La possibilité de réaliser des cavités micro-onde supraconductrices avec des très hautes performances à 7 K
- La possibilité de baser la mesure sur les propriétés de l'hélium, connues à ces températures par des calculs *ab initio*.
- La possibilité de réaliser de manière simple des systèmes fonctionnant à 7 K, grâce au développement des cryogénérateurs.
- Le changement du SI, qui permet de manière plus simple de réduire l'incertitude sur la détermination de la température absolue.

Le principe du changement d'indice

$$n = c_0/v$$

$$\varepsilon_{r\,He}(T,p)\mu_{r\,He}(T,p)=\left(\frac{\left\langle f_{ln}^{em}vacuum+\Delta f_{ln}^{em}\right\rangle(1+\kappa_t p/3)}{\left\langle f_{ln}^{em}+\Delta f_{ln}^{em}\right\rangle}\right)^2$$

Bien établies par les calculs *ab initio*
D'ou le mot quantique

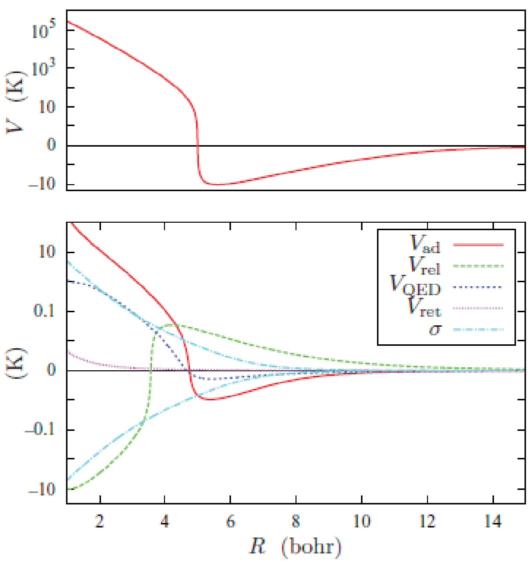
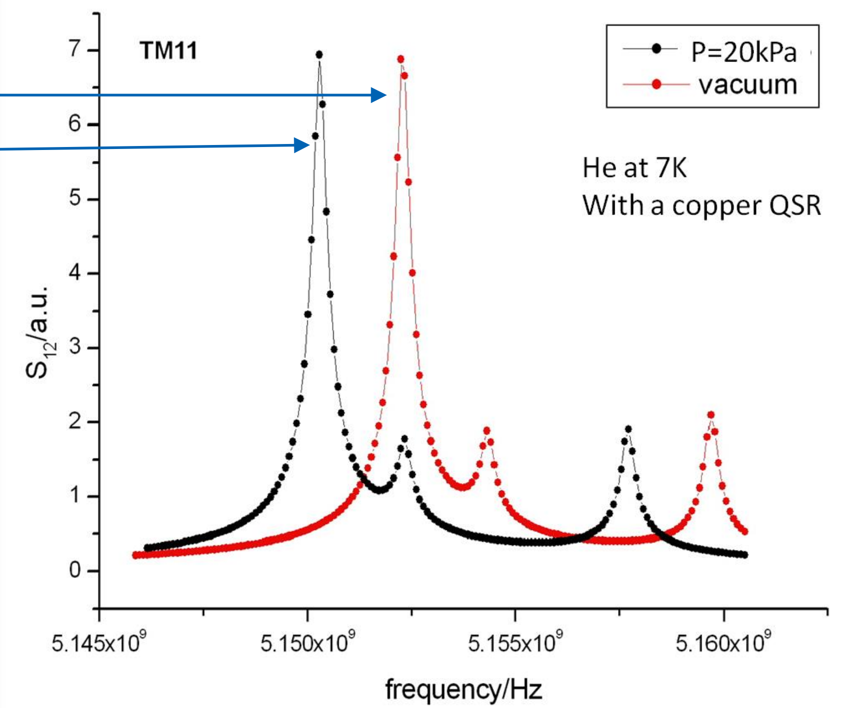


FIG. 5. Potential components at short and intermediate distances R. The ordinate scale is proportional to arcsin($5V$/K), which is approximately linear for small V and proportional to ln $|5V/\mathrm{K}|$ for large $|V|$. Top panel: the potential V of Eq. (2). The potential $V^{+\mathrm{ret}}$ would be optically indistinguishable. Bottom panel: the post-BO components of V and the residual retardation correction V_{ret}. The analytic fit of the uncertainty σ of the potential V is also shown.

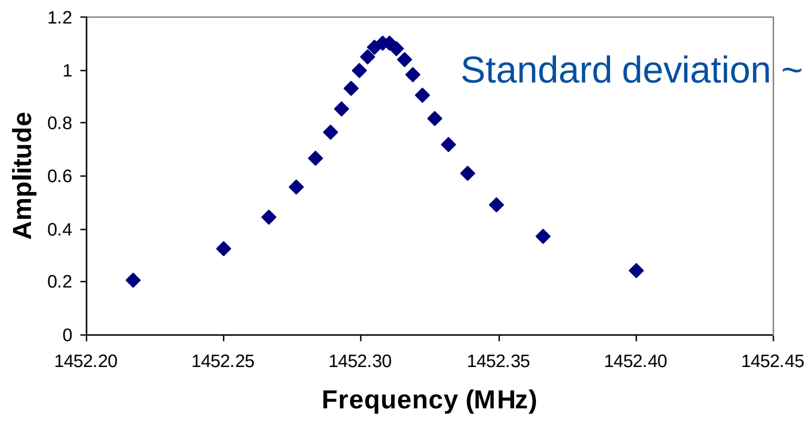


Si la cavité est sphérique et que le gaz soit de part et autres du résonateur, effet de la compressibilité simple a modélisé.

Mesurer une résonance

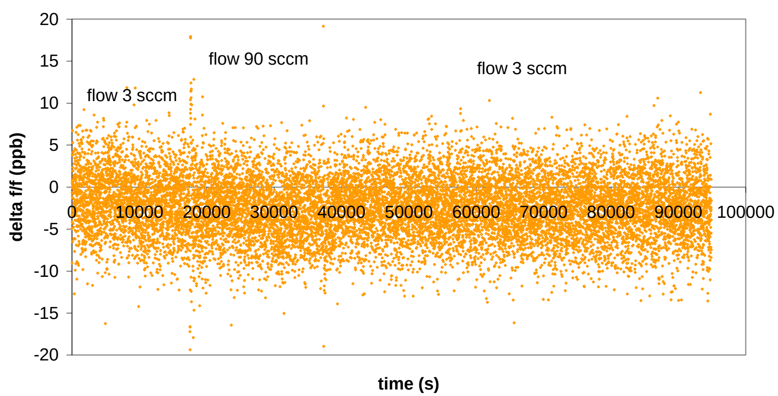
LNE — Le progrès, une passion à partager | le cnam

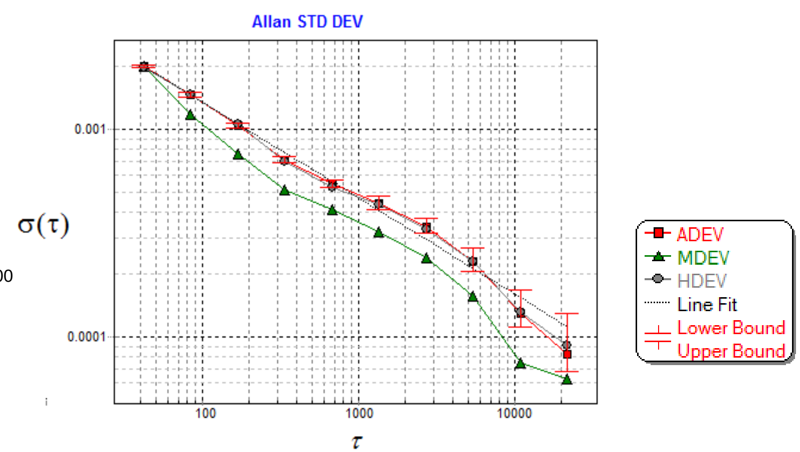
$$\frac{\Delta f_n}{f_n} = \frac{1}{A}\frac{1}{Q}\frac{1}{\frac{S}{N}}\sqrt{\frac{\tau}{t}}$$



Pure He flow @ 6.5 bar at 300K

Microwave change

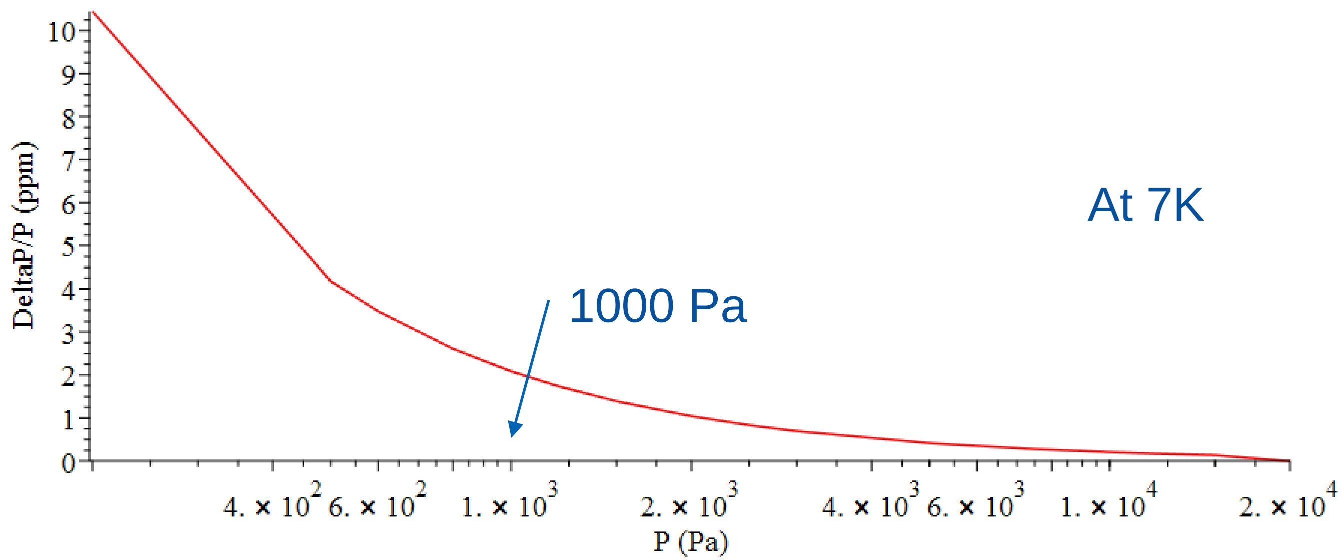




Propagation des incertitudes

Why we shall work at 7K

Uncertainty in Primary Pressure measurement due to an uncertainty of 0.05 $*10^{-9}$ in a frequency measurement
This uncertainty is 10 times better than could be obtained using a copper QSR.

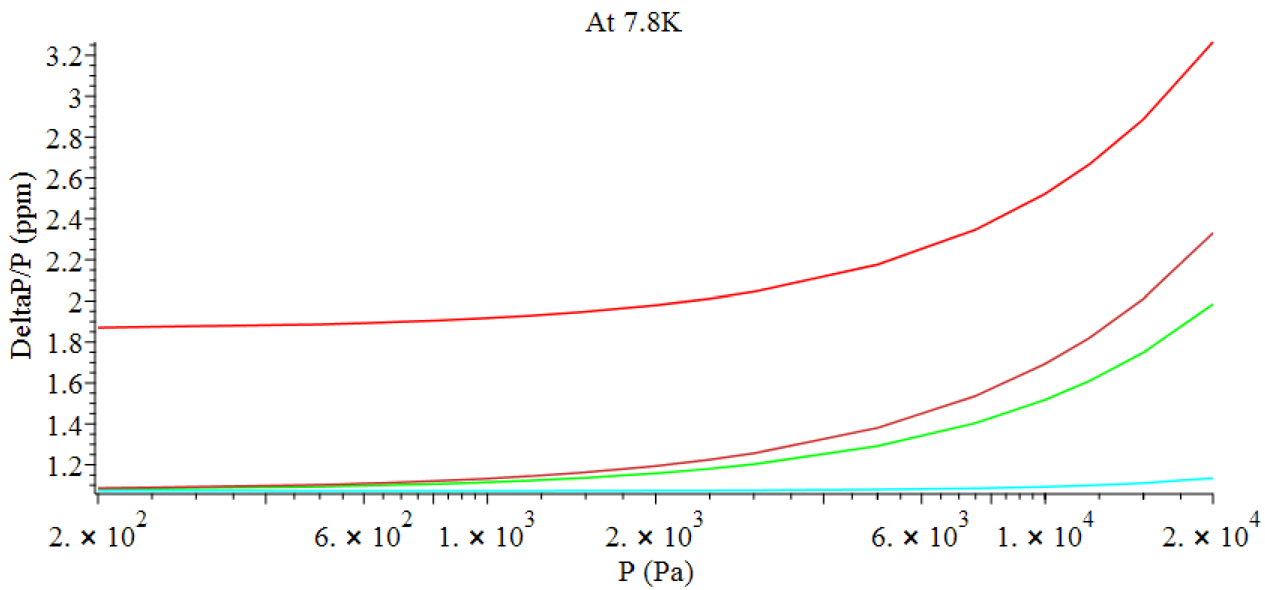


This element is negligible except at low pressure. This is due to the low uncertainty of the measurement . If have 5ppb of uncertainty on the measurement of the frequency resonance This uncertainty is multiply by 100. this graphic show the needs of superconductor cavity

Propagation des incertitudes

How the uncertainty of the virial coefficient propagates and also turns out to be negligible

Uncertainty in Primary Pressure measurement due to an uncertainty of B,C and b from the *ab initio* calculation



At 7 K

Rouge: total
Vert: UB
Bleu :UC
Pourpre: Ub

Uncertainty for B and C taken from Shaul *et al.* J. Chem. Phys. 2012

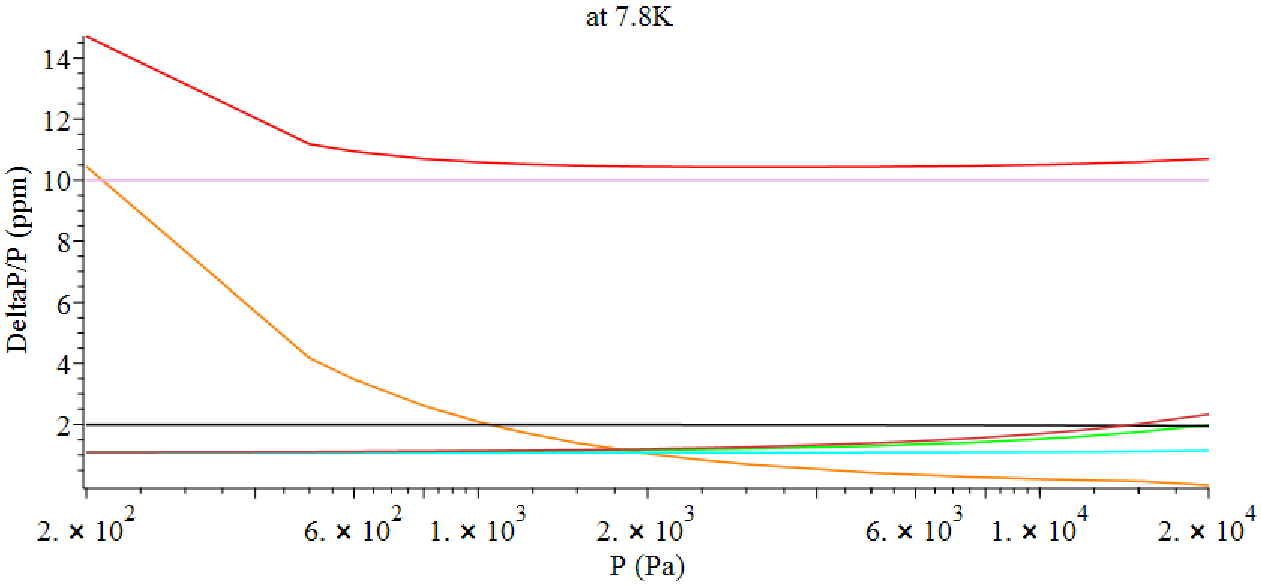
Uncertainty for b_{ε} taken from Rizzo *et al.* J. Chem. Phys. 2002

All the parameters used to get the pressure from the index have un uncertainty propagated to P much smaller than 1 ppm

Propagation des incertitudes

Expected uncertainty due to the model

Uncertainty propagation over the range 200 Pa to 20k Pa



Rouge: total
Orange: measurement
Noir :kp
Vert: UB
Bleu :UC
Pourpre: Ub
Rose: T uncertainty(10ppm)

It is clear from this calculation that the main uncertainty comes from the determination of the thermodynamic temperature (10 ppm)

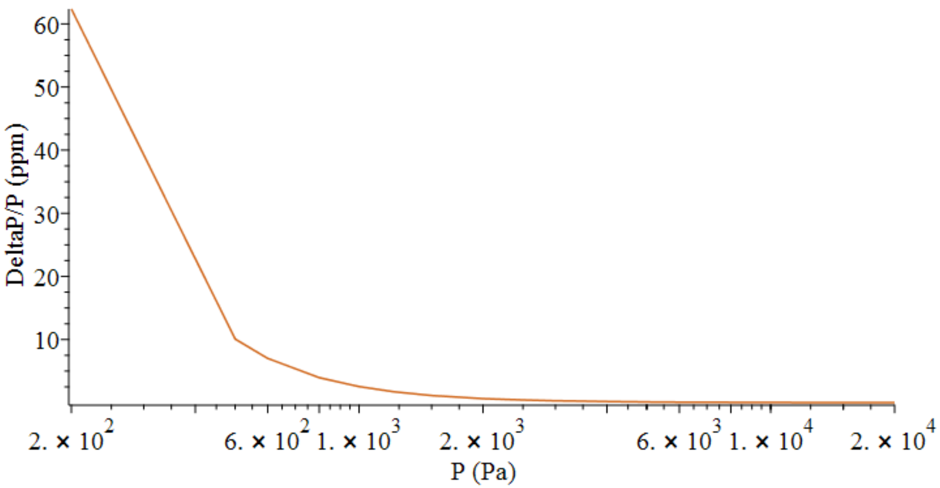
Corrections à prendre en compte

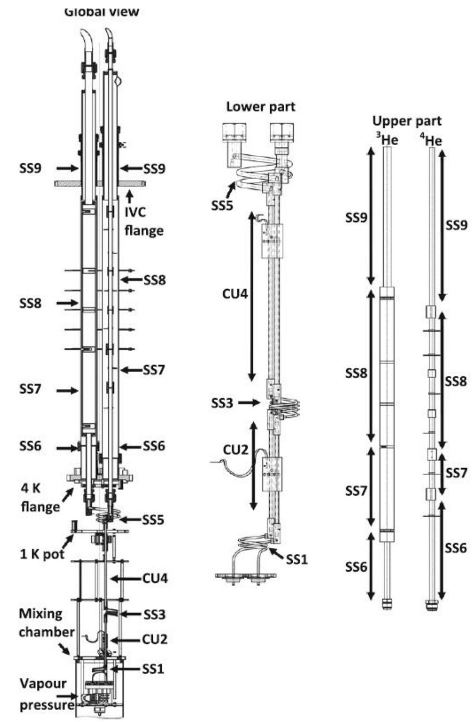
Thermomolecular and hydrostatic pressure corrections

$$(p_\mathrm{H} - p_\mathrm{L})/p_\mathrm{L} = (2\times10^{-9})\,(R\,p_\mathrm{L}/(\mathrm{Pa\cdot m}))^{-1.99}\,((T_\mathrm{H}/\mathrm{K})^{2.27} - (T_\mathrm{L}/\mathrm{K})^{2.27})$$

This relation comes from :Guide to the Realization of the ITS-90 Interpolating Constant-Volume Gas from CCT WG1 Steur, Fellmuth and Tamura

By taking a tube of radius 1.8 cm and a design from F. Sparasci, L. Pitre, D. Truong, L. Risegari and Y. Hermier Realization of a 3He–4He Vapor-Pressure Thermometer for Temperatures between 0.65 K and 5 K at LNE-CNAM *Int. J. Thermophys* (2011) **32**,139–152.

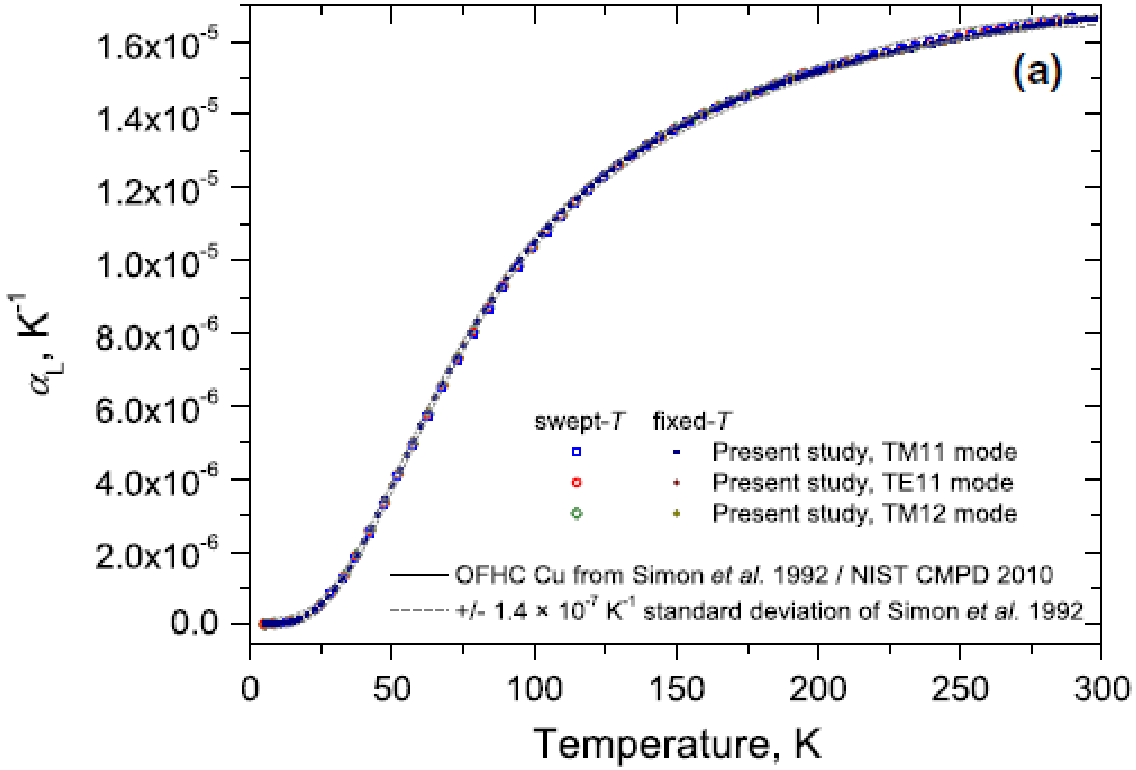




Une sphère très stable

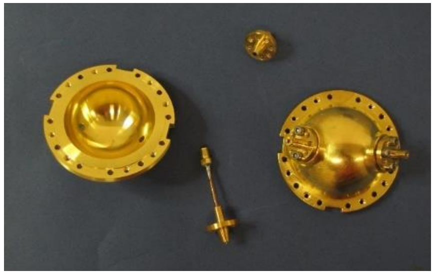
Thermal expansion of the QSR at very low temperature

At 7.8 K the thermal expansion coefficient is very small $< 0.0178 \times 10^{-6}$ K^{-1} or 950 times lower than at room temperature. This very small number will allow us to have a much (100 times) faster measurement. Because the needs to wait for the QSR to come back to the same temperature is reduced compared with room temperature.

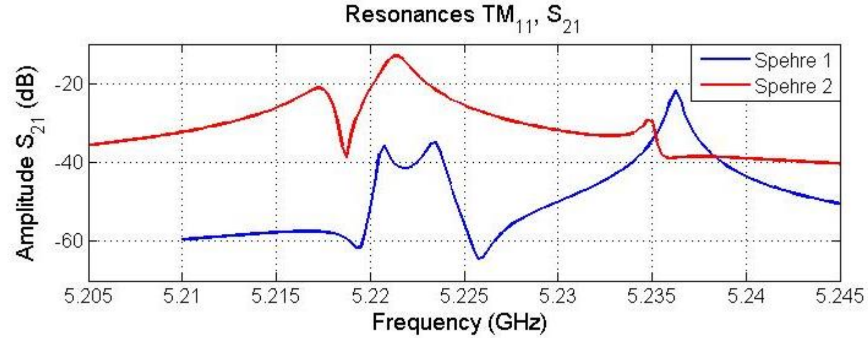


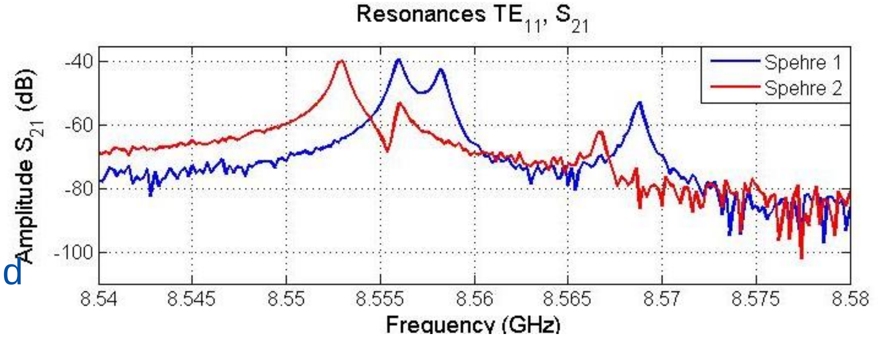
Possible design for QSR with radius of 2.5 cm

- •New design
- •QSR with 2.5 **cm** radius With a possibility to install a microwave antenna with cone.
- • this QSR had a Gold layer 10 µm thick
- • the skin depth was in good agreement compared to the gold metal, gn~200 kHz



Frequency range:
- •TM_{11}: 5.22 GHz
- •TE_{11}, 8.56 GHz

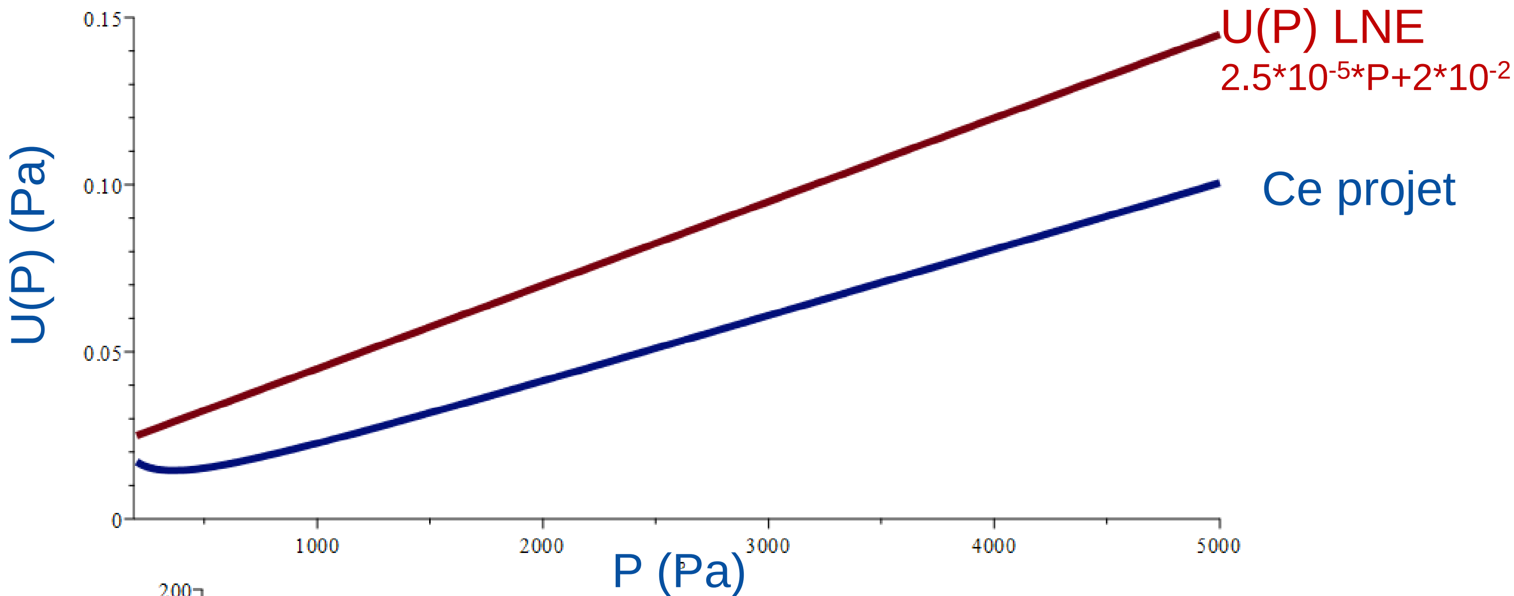


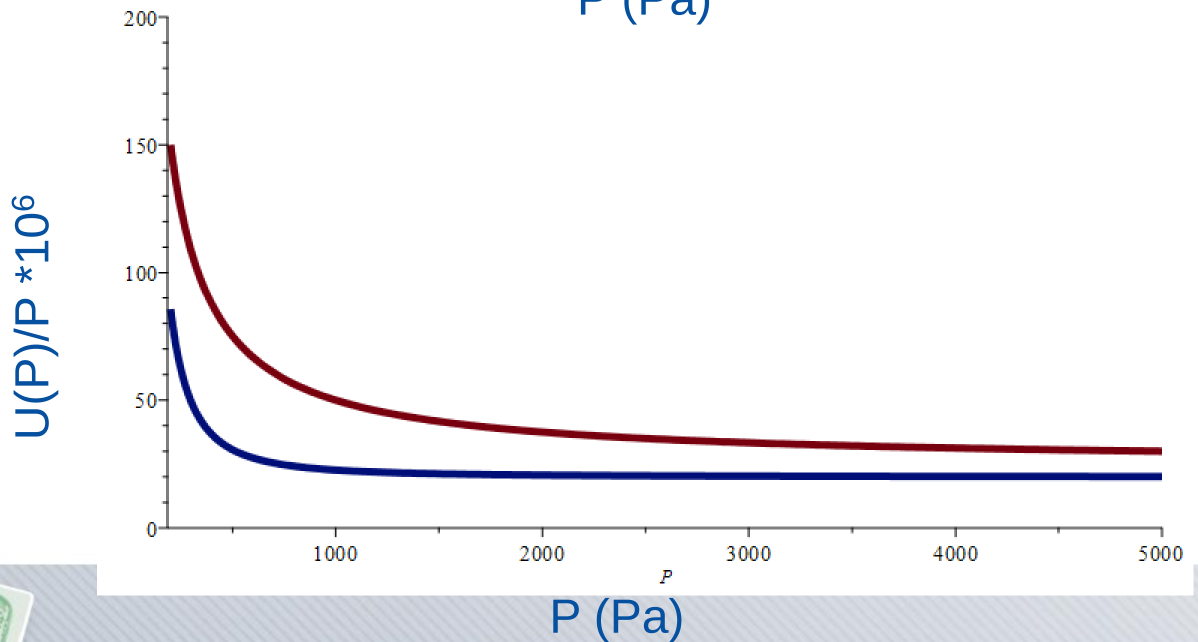


Good Signal under noise ratio with this QSR for the TM and TE

At room temperature we have a Q of 26000 and
A resolution of 5ppb
To get 0.05ppb with a R=2.5. we will need a Q
Of $2.6*10^6$ or an increase of 33 in Q due to the superconductor

Comparaison des incertitudes en k=2





En conclusion

Principaux avantages :

Aucun effet de la pollution (à 7 K) : c'est l'avantage le plus important.
Atténuation du bruit en pression car une partie du gaz se trouve à 7 K, ce qui lui confère une grande masse volumique.
Aucun besoin d'une bonne connaissance de la compressibilité mécanique de la cavité, 1.5% est suffisant.
Calculs *ab initio* pour toutes les propriétés thermo physiques de l'hélium déjà réalisé avec une exactitude suffisante.
Les tubes peuvent être conçus de manière à réduire les effets thermomoléculaires de pression hydrostatique, de façon à les rendre faibles et calculables.
La mesure est rapide.
La gamme de mesure des microondes est facilement atteignable (moins de 10 GHz).
Possibilité de mesure différentielle rapide et précise.

Les difficultés à surmonter :

Le Nb a une température de transition supraconductrice T_c d'environ 9 K.
La température thermodynamique doit être connue avec une incertitude relative de 10^{-5} à 7 K.
Les thermomètres de type capsule doivent être stables au cours des cycles thermiques à mieux que 0,1 mK.
Le gaz et les thermomètres doivent être à la même température, même avec le flux de chaleur venant des micro-ondes (effet de peau).