LA DILUTION FREE : UNE NOUVELLE APPROCHE DES TRES BASSES TEMPERATURES

Patrick Pari, Philippe Forget, Matthieu De Combarieu CEA-SPEC
Alain Benoit CNRS-Louis Néel
Olivier Guia, Julien Paris CRYOCONCEPT

Rappel des principes de la dilution classique et des systèmes sans boîte à 1K

Quelques exemples de systèmes à dilution classiques

La dilution FREE de 1987: STIRLING 1W@20K

Etat de l’art actuel des différents systèmes FREE existants.

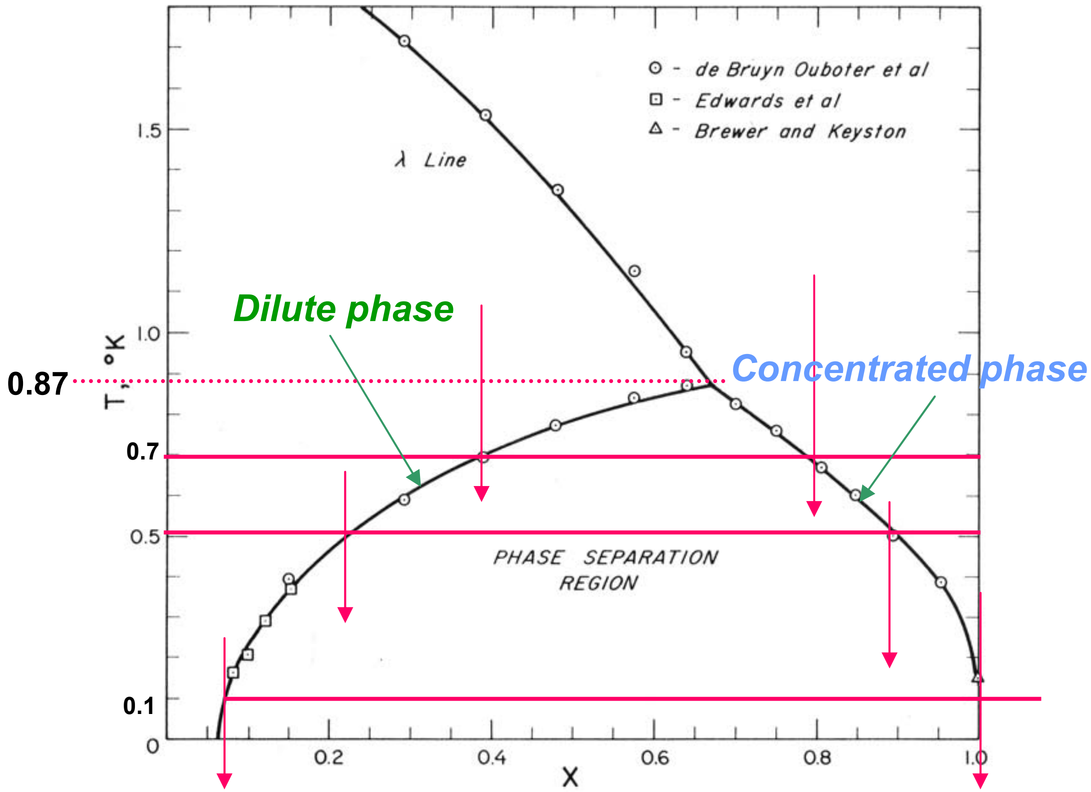
La dilution FREE de 2007 : CRYOMECH PT407: PT 700mW@4.2K

Principe de notre système, original dans sa conception.

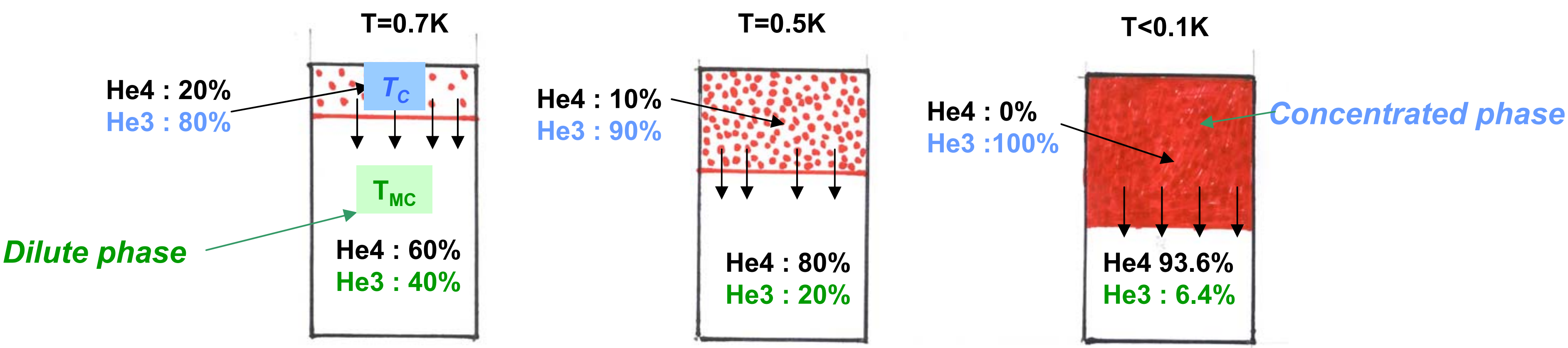
Performances

Analyse thermique et thermodynamique au niveau de la BM.

Conclusions Perspectives

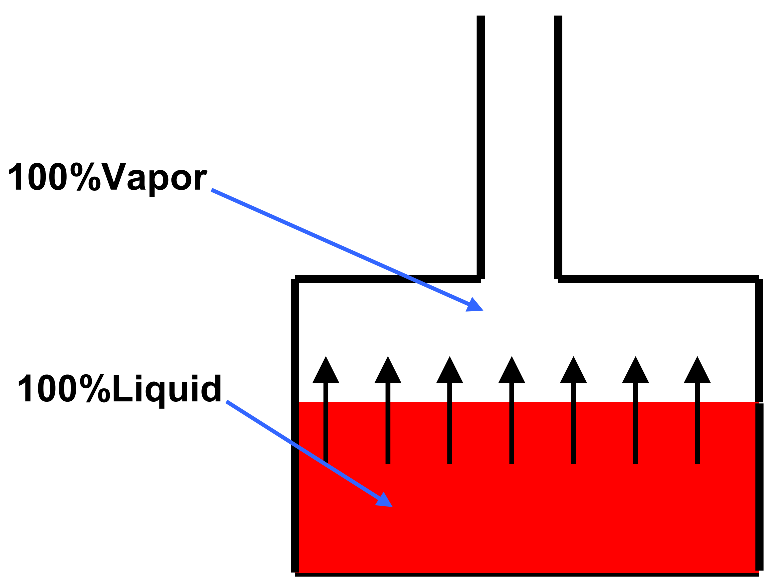


○ - de Bruyn Ouboter et al
□ - Edwards et al
△ - Brewer and Keyston
λ Line
Dilute phase
Concentrated phase
PHASE SEPARATION REGION
0.87
T, °K
1.5
1.0
0.7
0.5
0.1
0
0.2
0.4
0.6
0.8
1.0
X

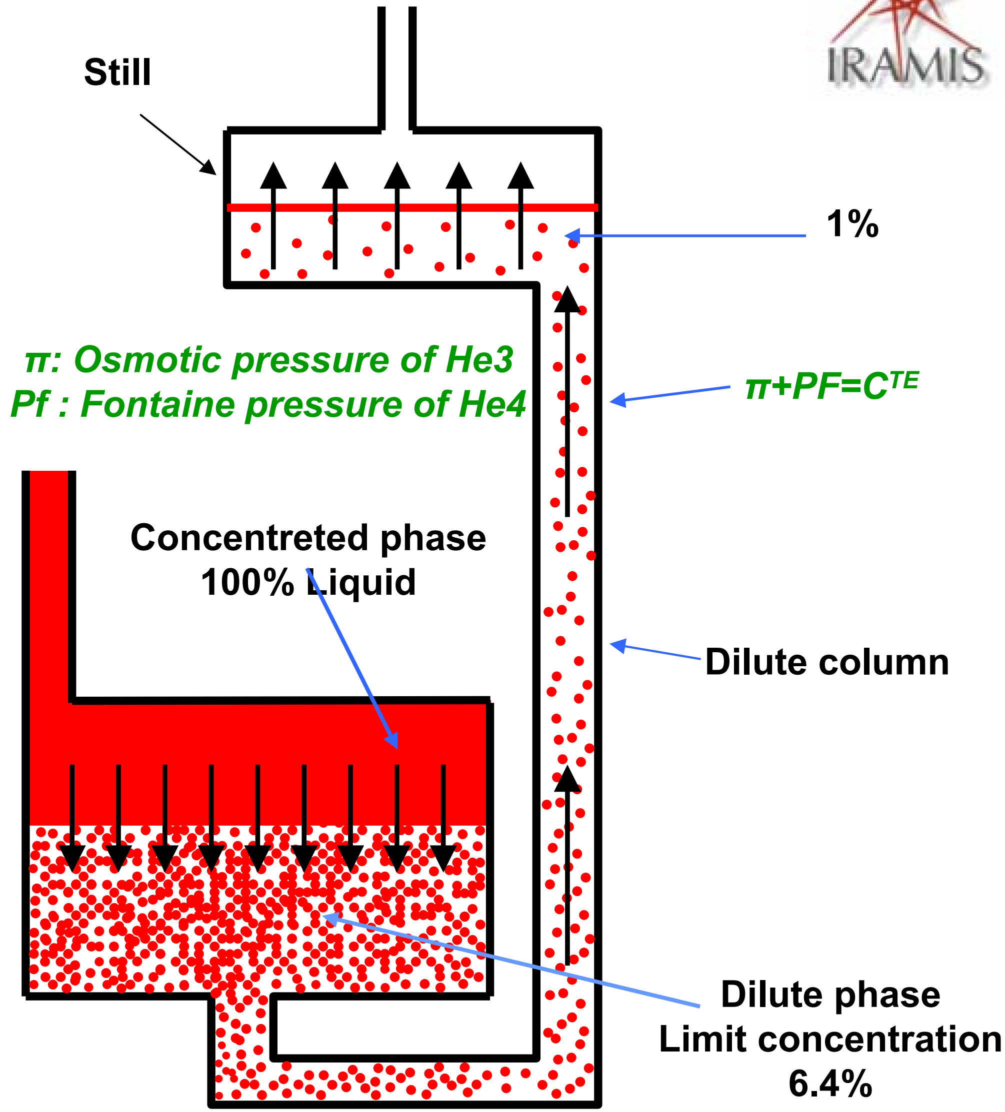


T=0.7K
He4 : 20%
He3 : 80%
T_C
T_{MC}
Dilute phase
He4 : 60%
He3 : 40%
T=0.5K
He4 : 10%
He3 : 90%
He4 : 80%
He3 : 20%
T<0.1K
He4 : 0%
He3 :100%
Concentrated phase
He4 93.6%
He3 : 6.4%

Pur He3 fridge



Dilution



$Q_{EXT}=n_3 \, (96 \, T_{mc}^2-12 \, T_C^2) \quad Js^{-1}$

$Q_{EXT}=84 \, n_3 \, T_{mc}^2 \quad J \, s^{-1}$ *perfect exchangeurs*

At interface :

$$\dot{Q} = \alpha A (T_1^4 - T_0^4)$$

$$\alpha = f(Z_1, Z_2)$$

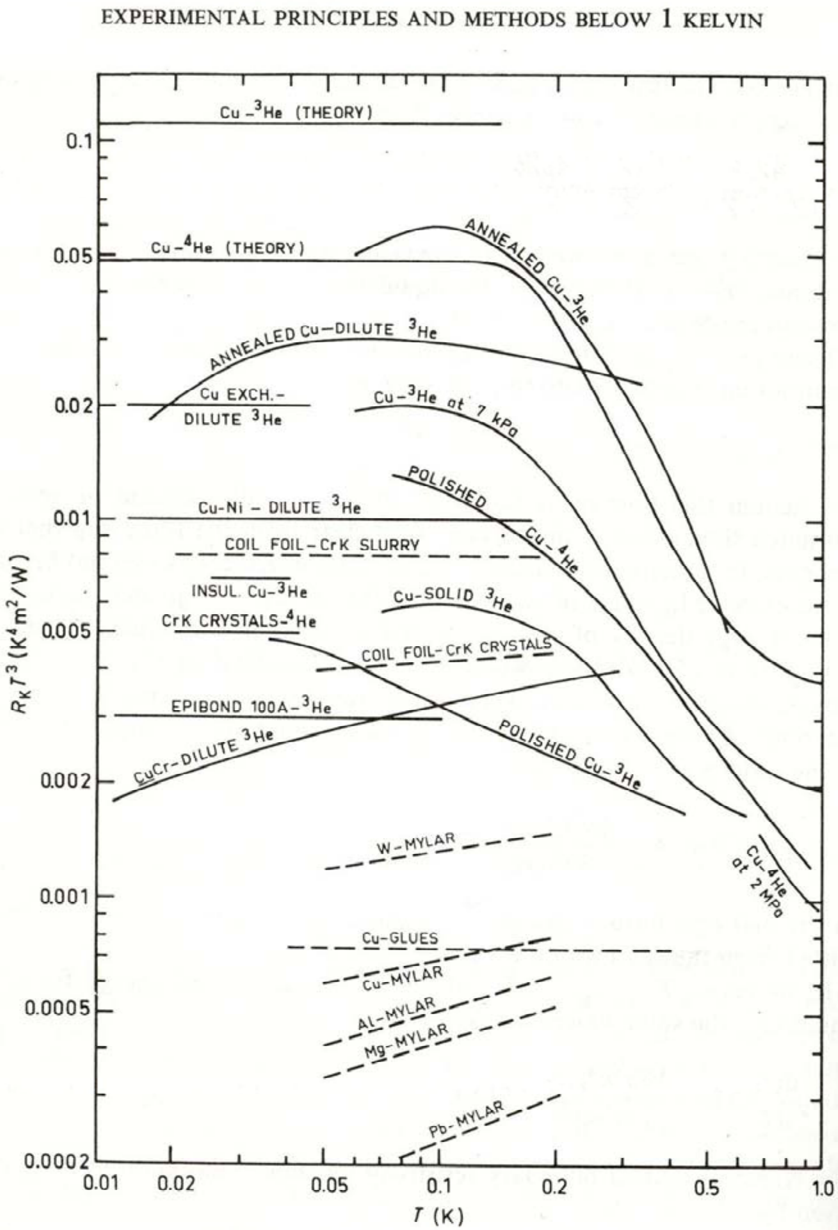
$$Z = \rho \upsilon$$

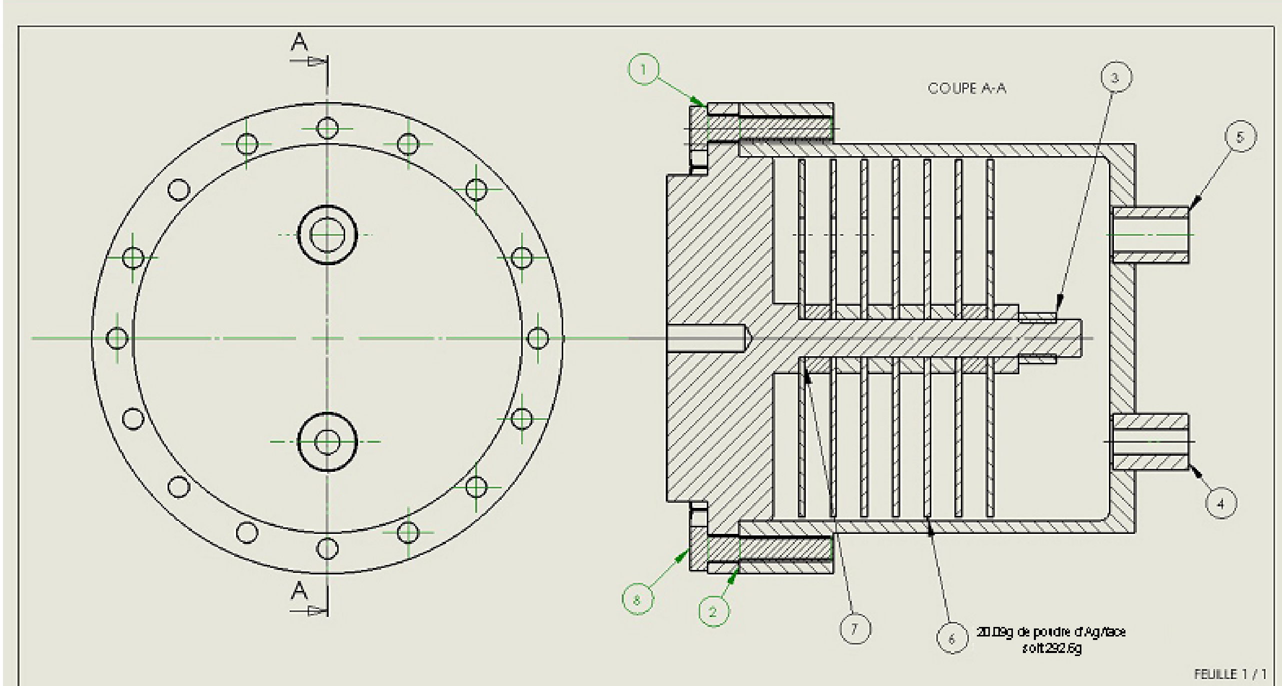
Z = impedence factor
ρ = density
υ = sound velocity

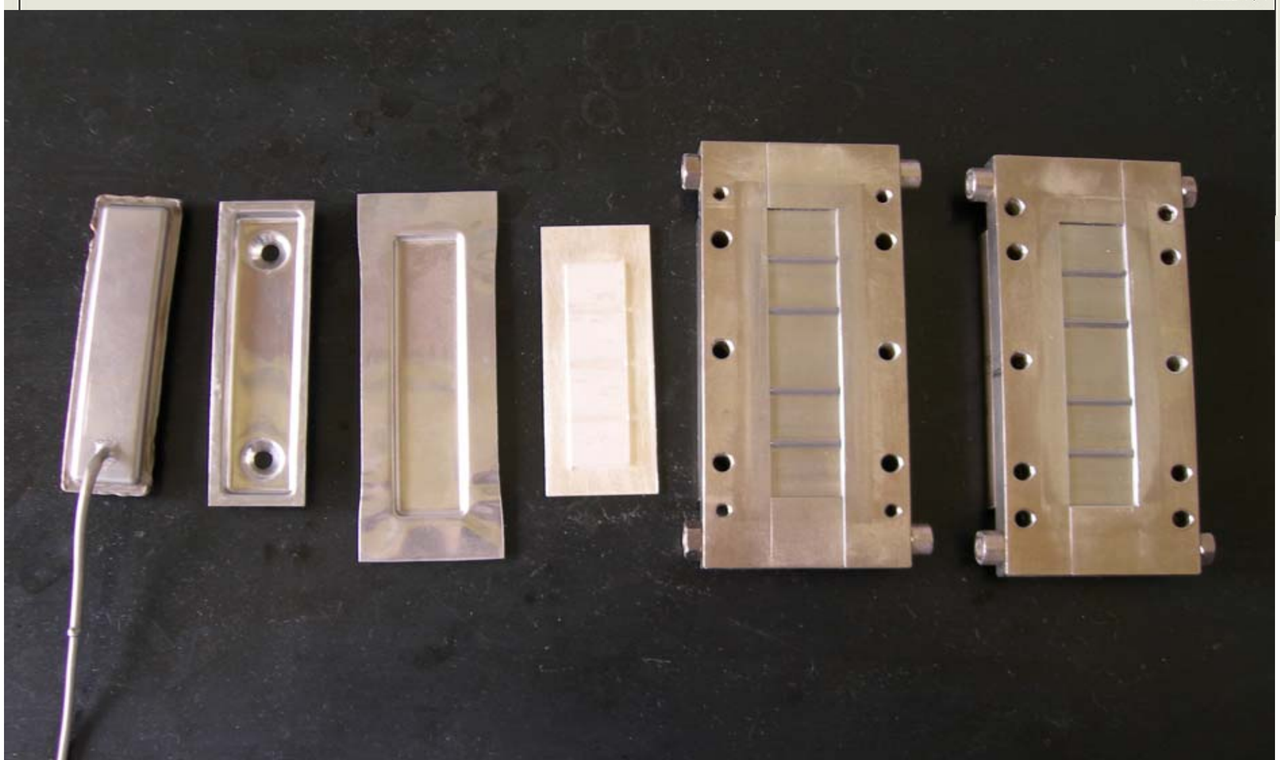
for $\Delta T < T$ $$\dot{Q} = \frac{A T^3 \Delta T}{R_{(K)}}$$

R_K : $K^4 m^2/W$

impedance adjust?

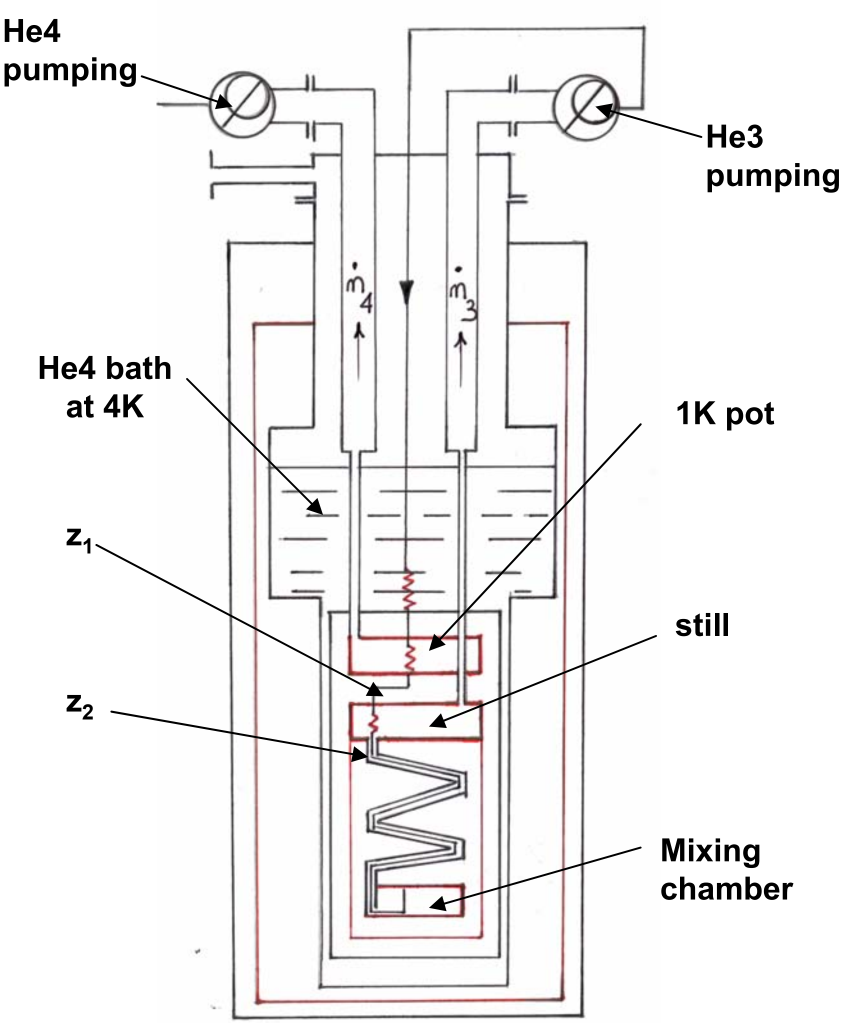



A
A
1
COUPE A-A
3
5
4
8
2
7
6
20.09g de poudre d'Ag/face
soit 292.6g
FEUILLE 1 / 1



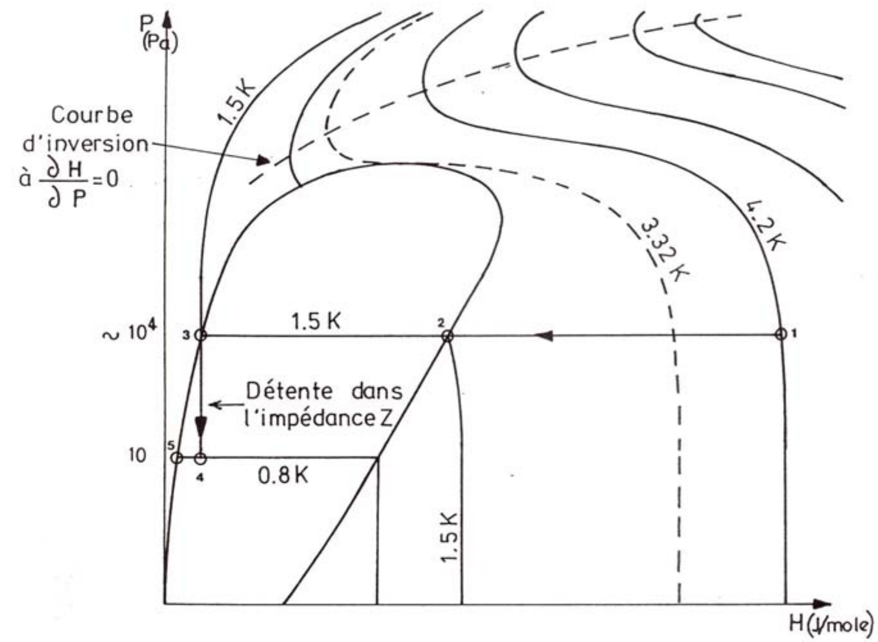
CLASSICAL DILUTION REFRIGERATOR CYCLE AND DIAGRAM

0.2 l/h < liquid helium flow < 1 l/h

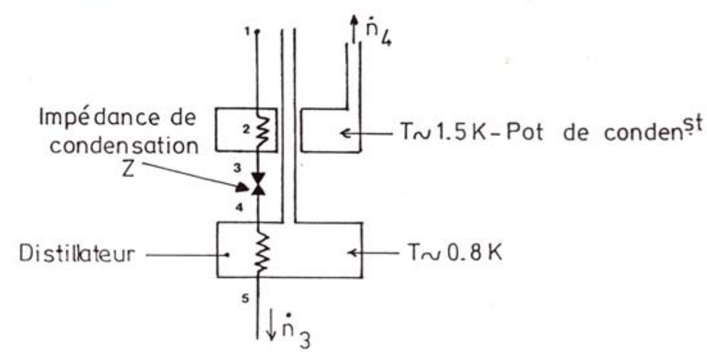


CLASSICAL DILUTION REFRIGERATEUR CYCLE

Diagramme enthalpique de l'He3 pur [21]



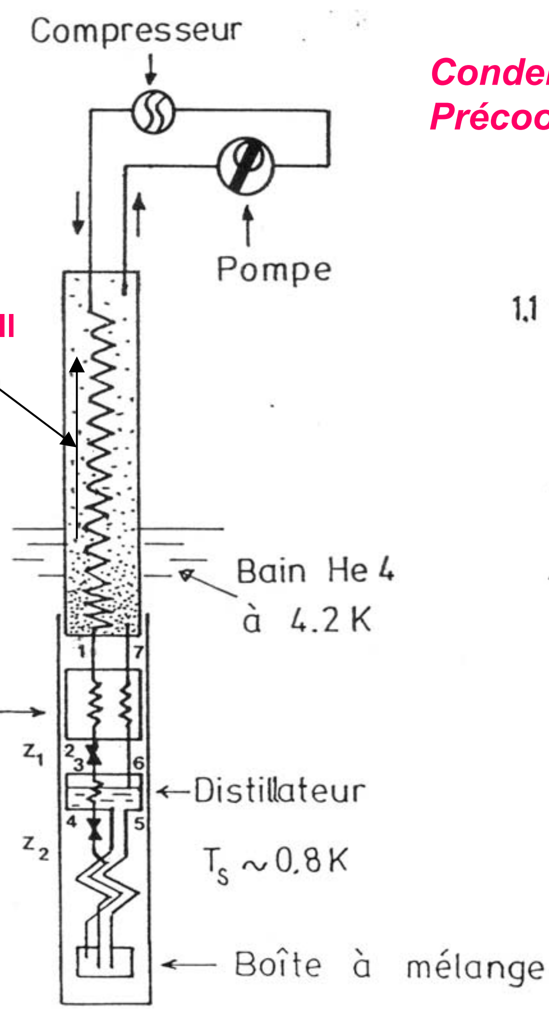
Parcours de liquéfaction dans un appareil à fonctionnement classique



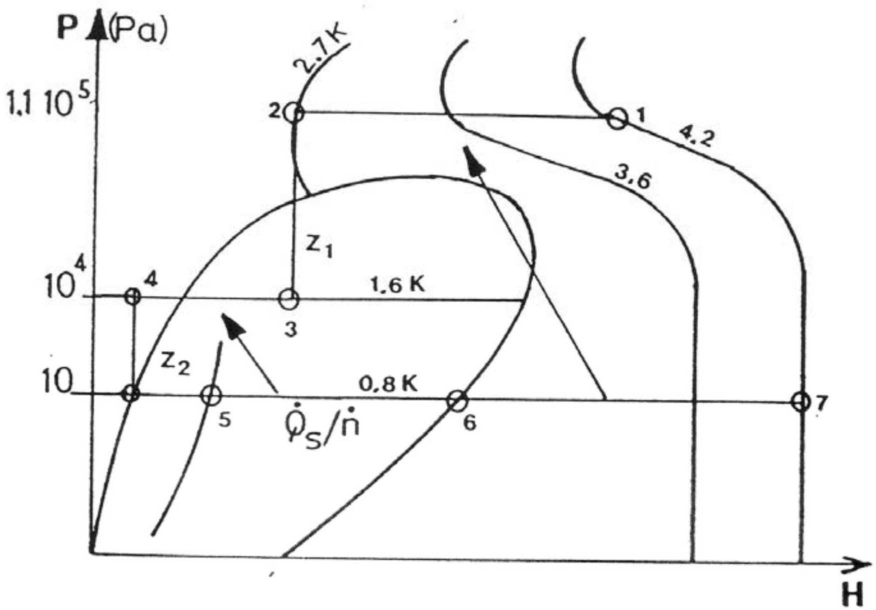
DILUTION STICK

Thèse Michel Caussignac (CNRS)
Thèse Patrick Pari (CEA)

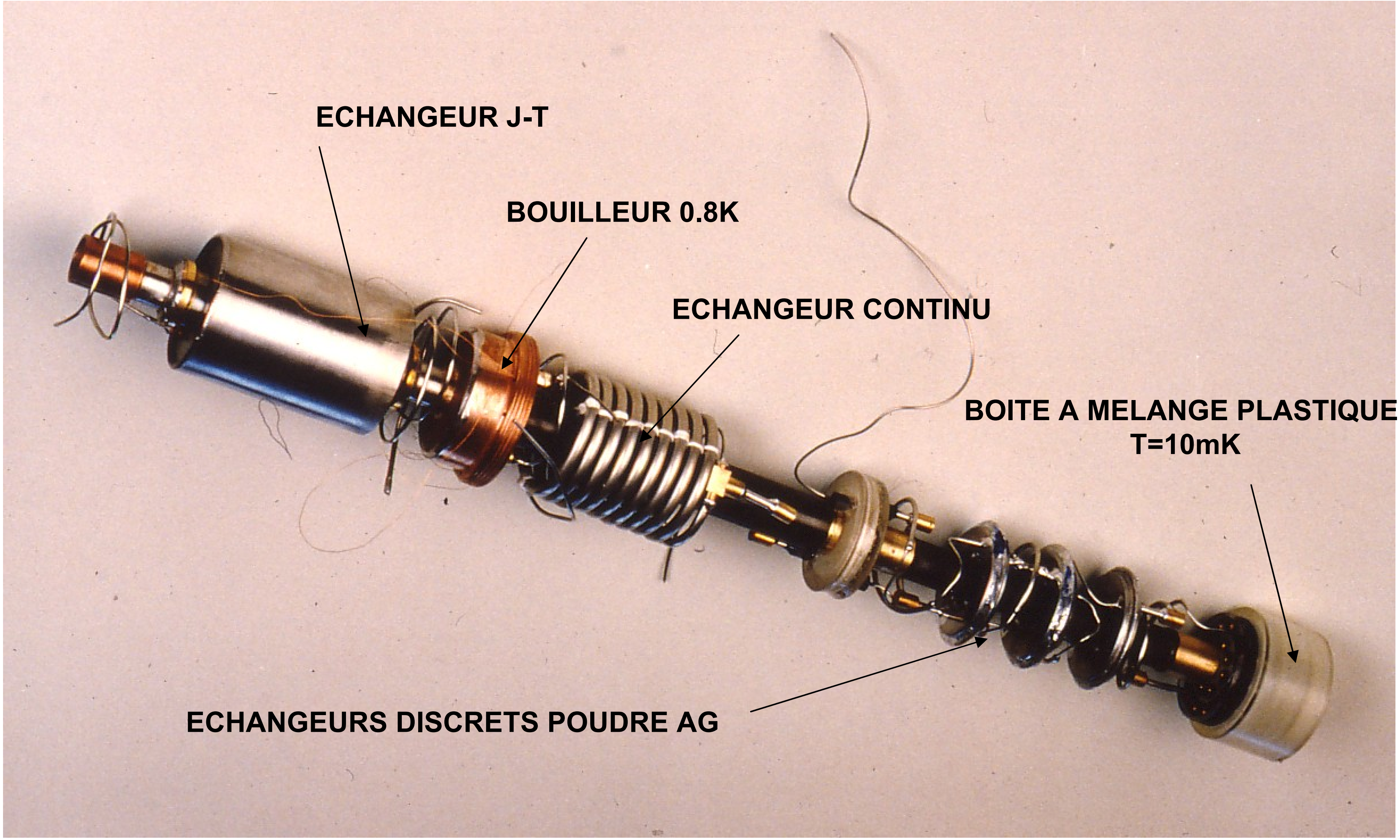
Cold vapors from the still
P=0.1mb



Condensation from 4.2K
Précooling mixture in the Joule-Thomson stage



MINI FRIDJE 10mK
DIAMETRE 30mm



APPAREIL TYPE 200µW

PASSAGES CLIENTS

BRIDE 4K

BOUILLEUR 0.8K

PLATINE CUIVRE DORE 0.8K

ECHANGEUR CONTINU

PLATINE 100mK

ECHANGEURS DISCRETS
POUDRE D4ARGENT FRITTE

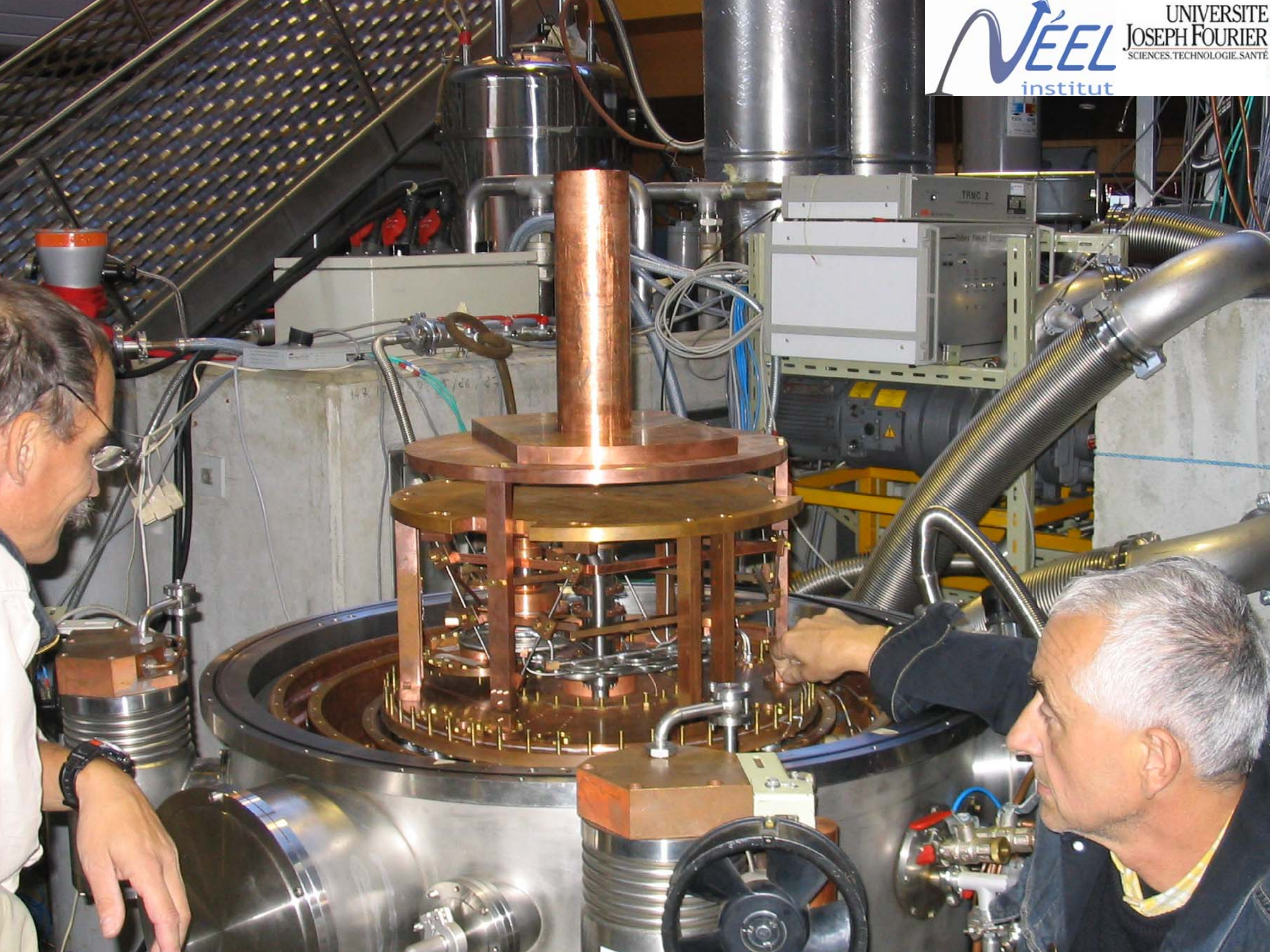
BOITE A MELANGE

PLATINE 10mK

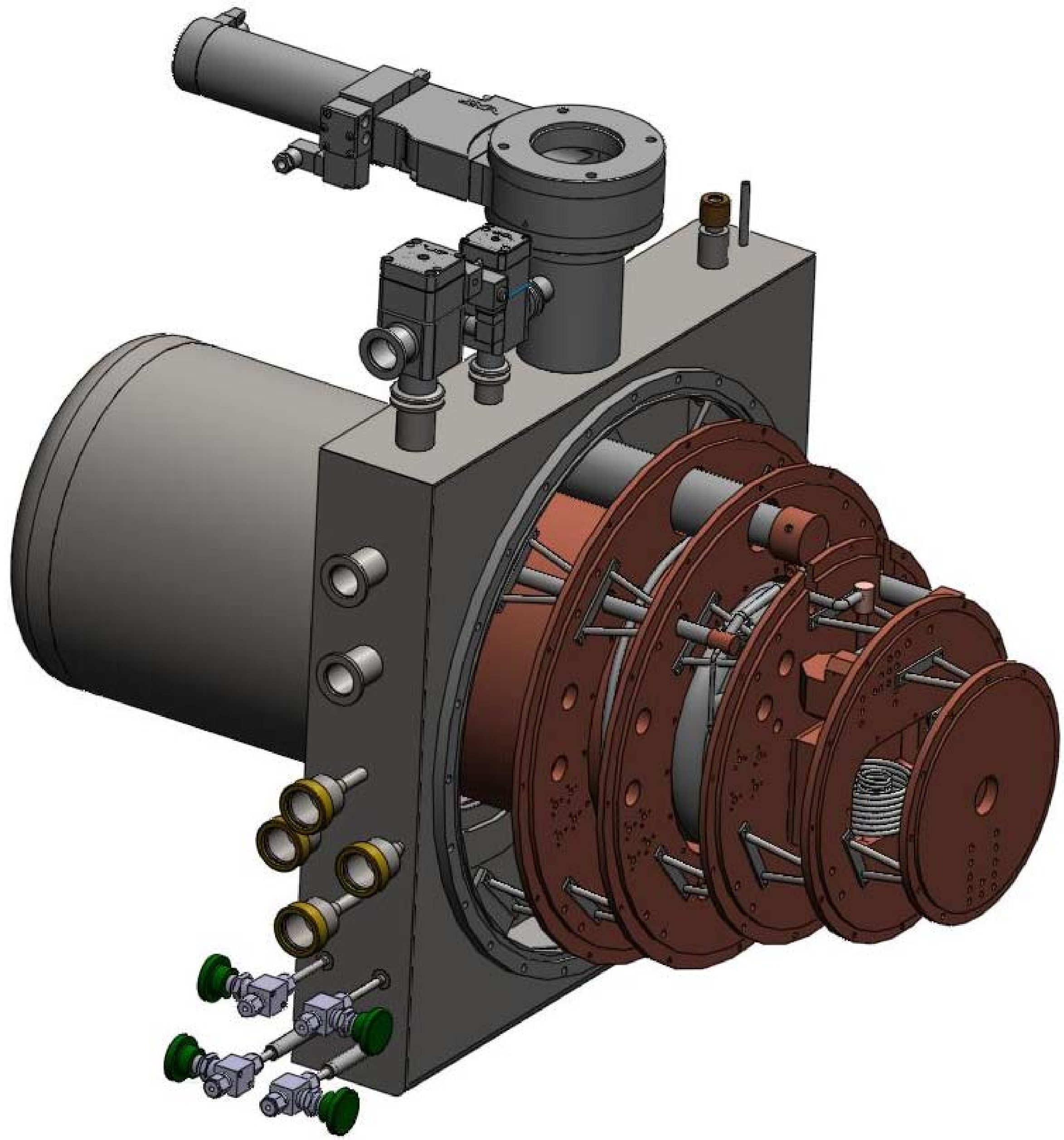
REFRIGERATEUR A DILUTION TYPE 400 -1000µW

REFRIGERATEUR A DILUTION RENVERSE LYON

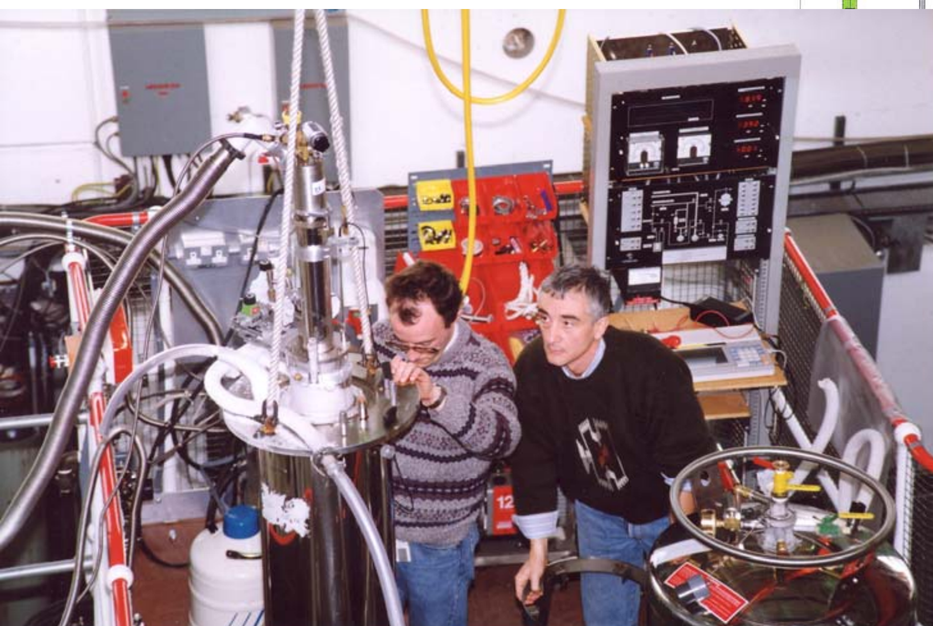
NÉEL institut
UNIVERSITE JOSEPH FOURIER
SCIENCES.TECHNOLOGIE.SANTÉ

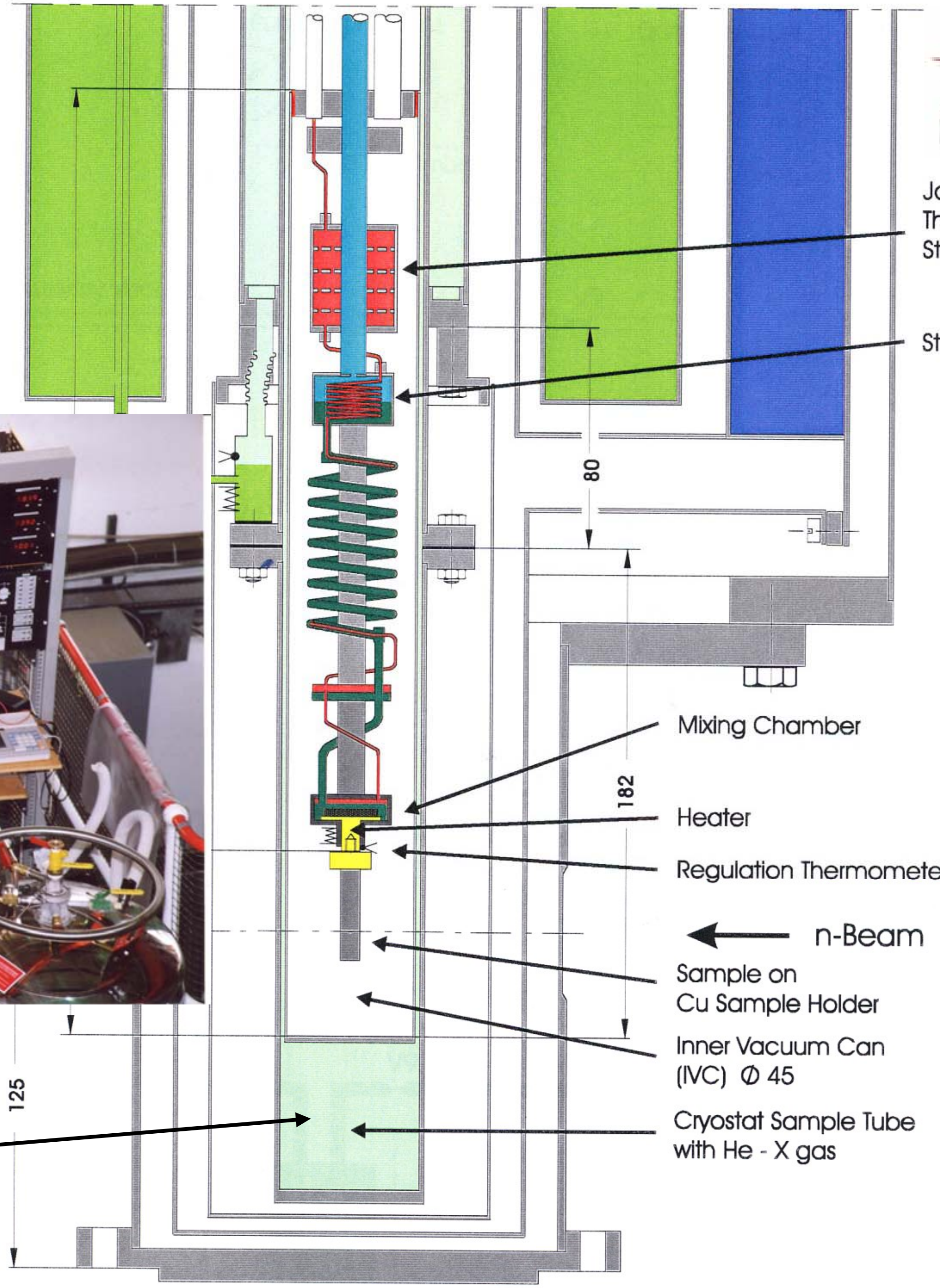


CRYOSTAT A DILUTION HORIZONTAL POUR EXPERIENCES OPTIQUES VIDE UNIQUE, ETAGE JT, DOUBLE BOUILLEUR



Exemple de fonctionnement d'une canne à dilution dans un puit de gaz froid He4





He4 gaz à basse pression
Quelques millibars.

FREE DILUTION REFRIGERATOR OF 1987

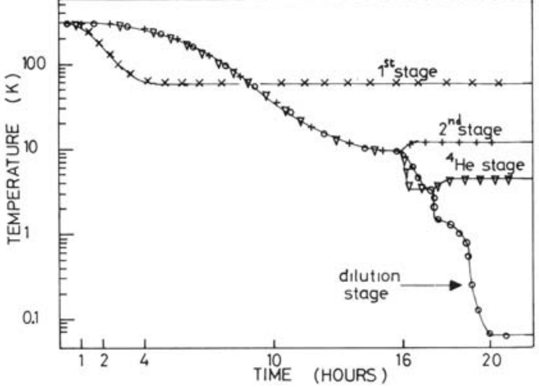
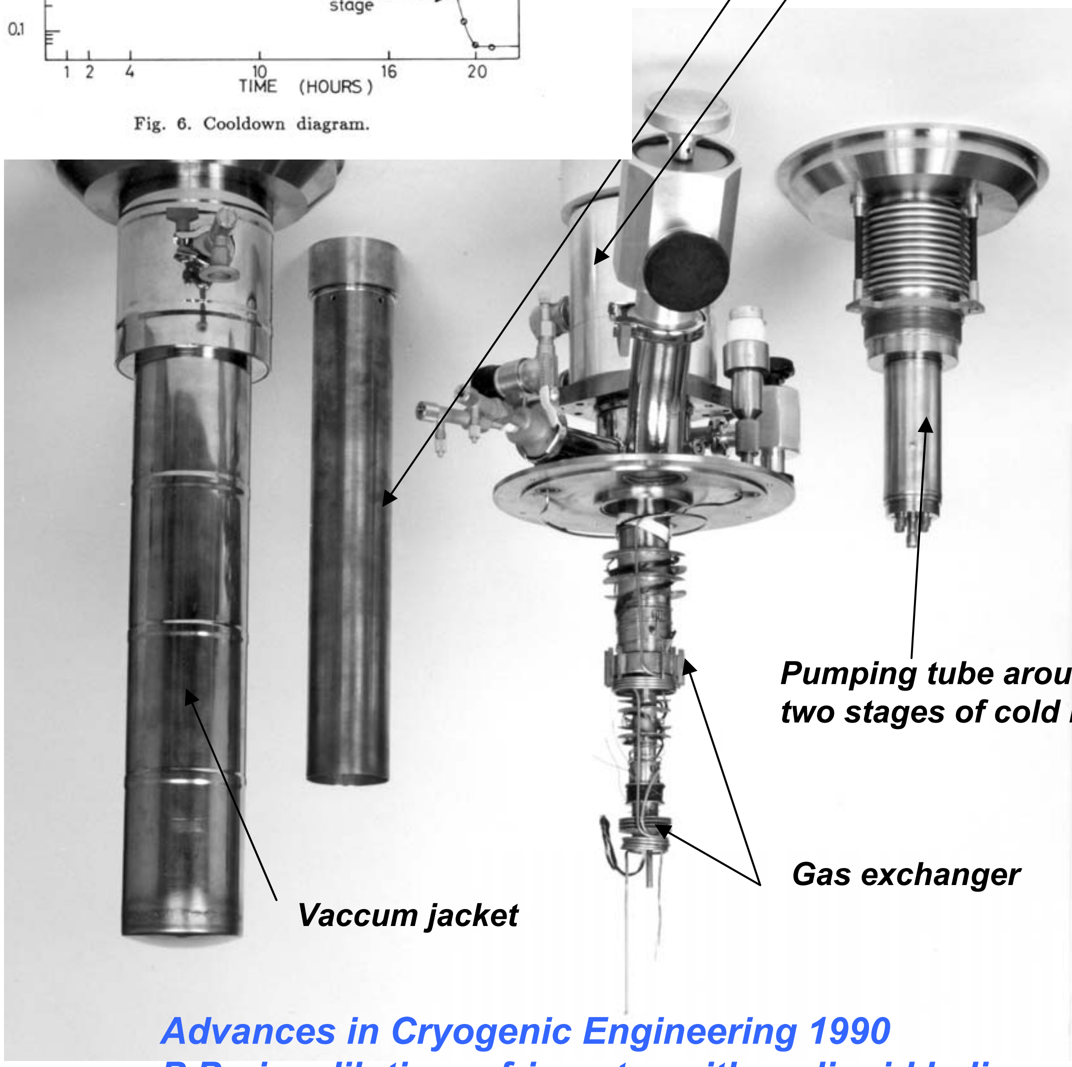
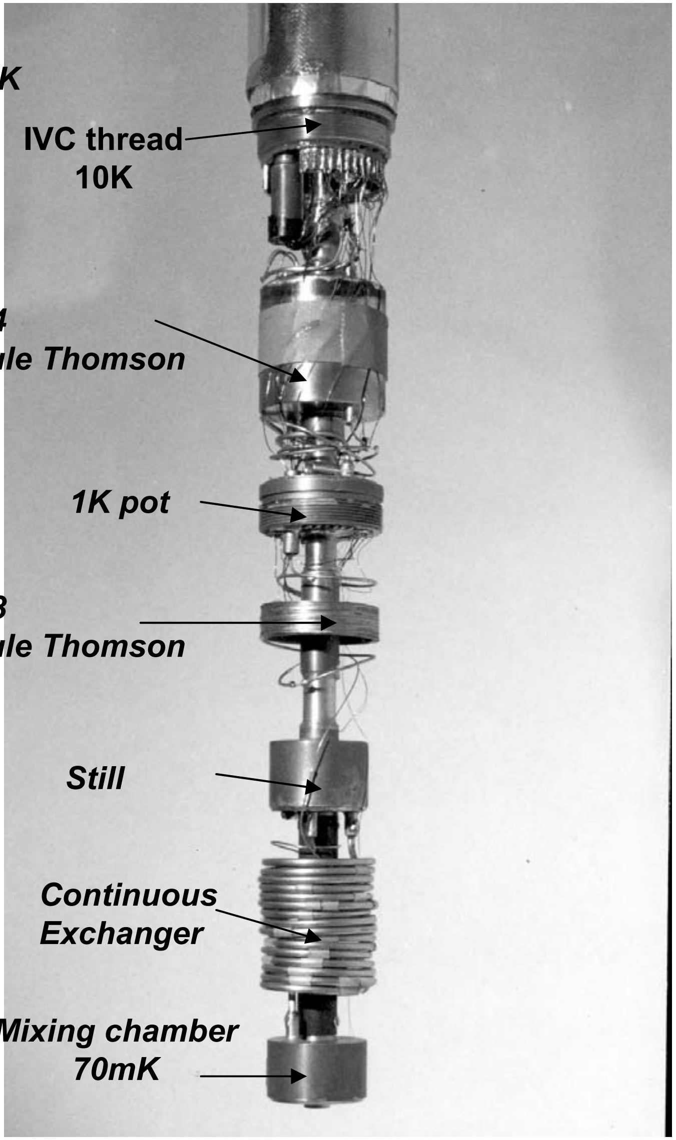


Fig. 6. Cooldown diagram.





Advances in Cryogenic Engineering 1990
P.Pari « dilution refrigerator with no liquid helium supply »

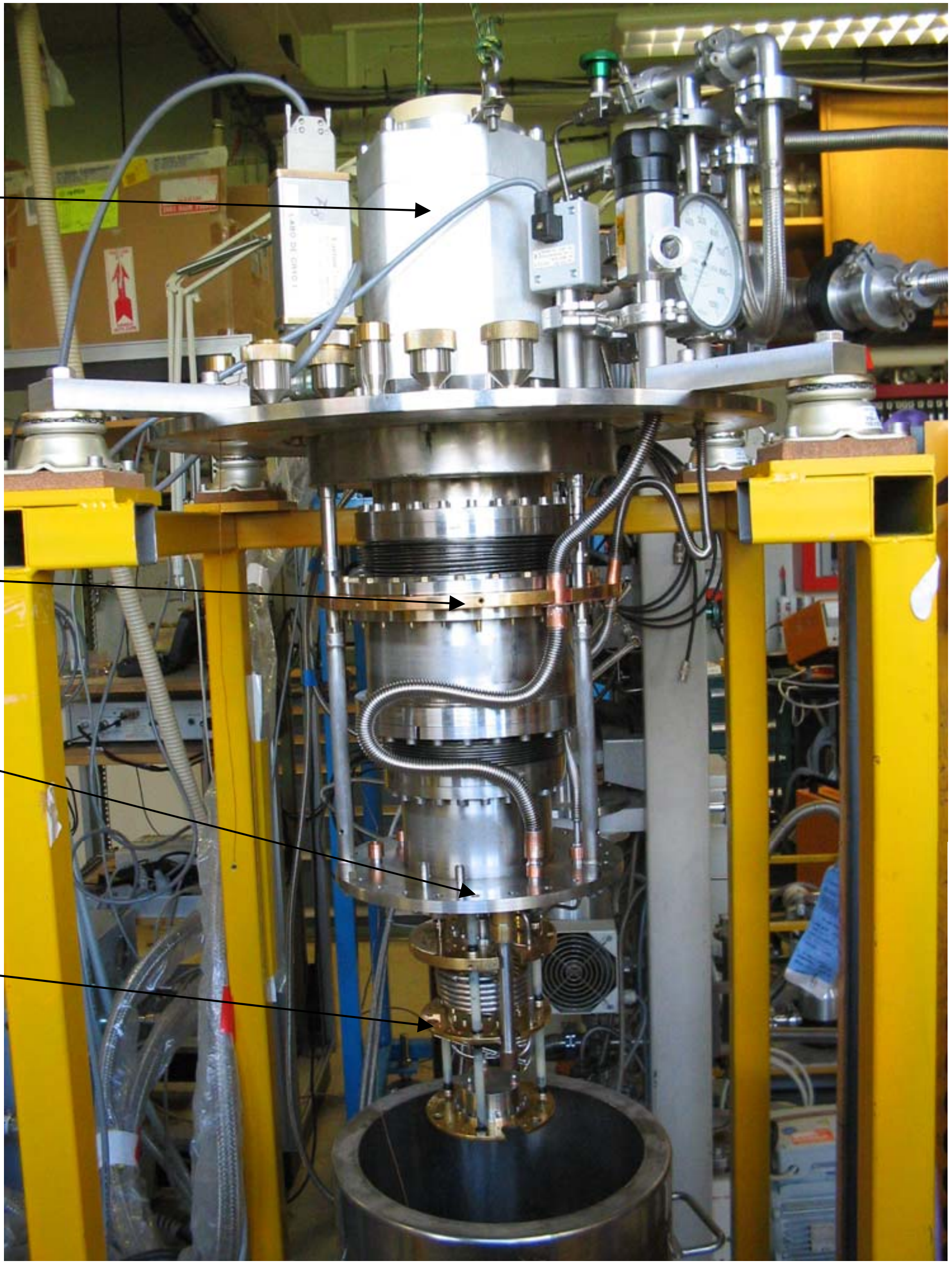
DILUTION FREE 2007

PT 407
700mW @ 4.2K

FIRST STAGE
60k

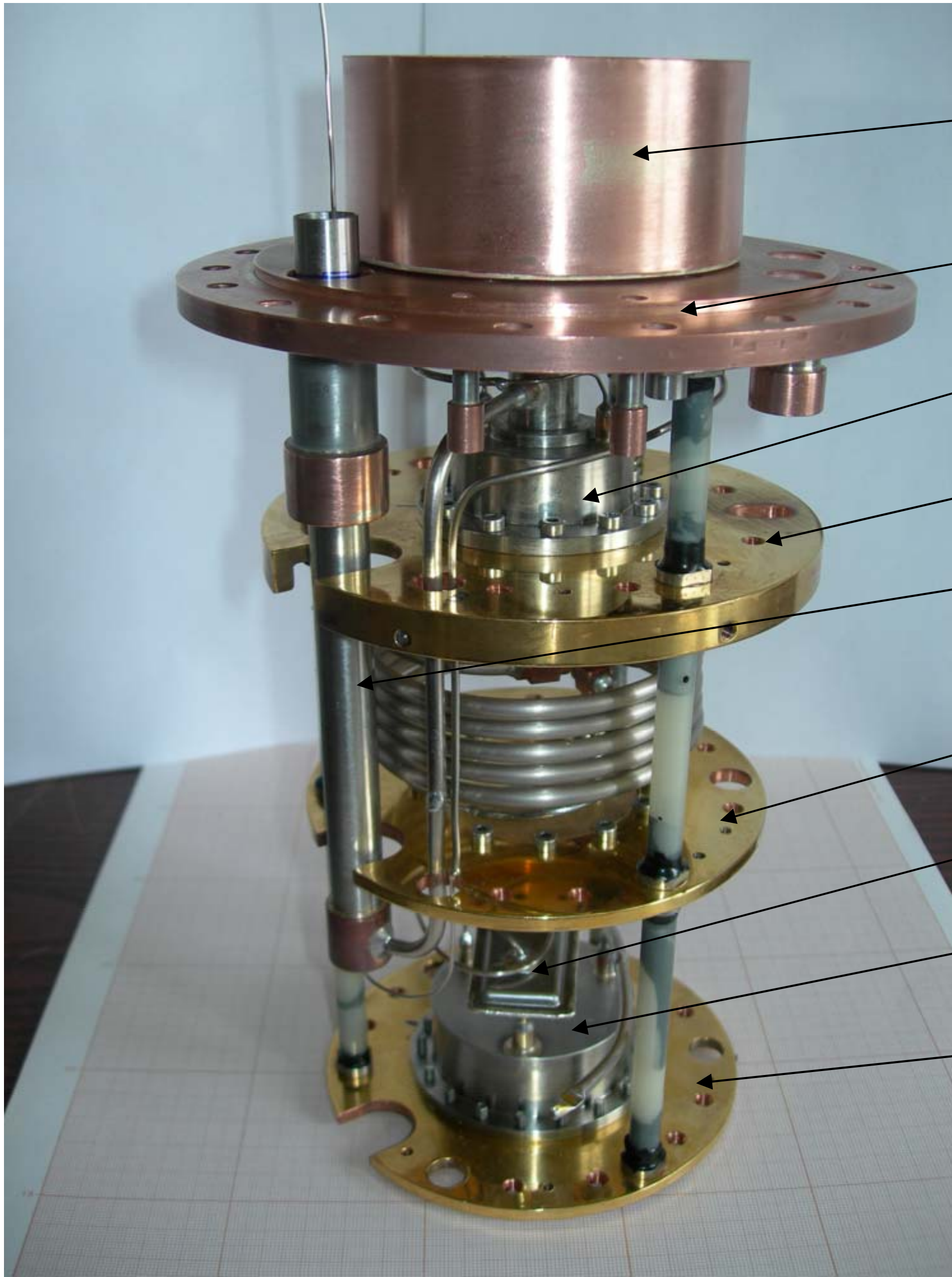
SECOND STAGE
4K

DILUTION STAGE
30 mK
LESS 20 Hours
50mK<T<300K

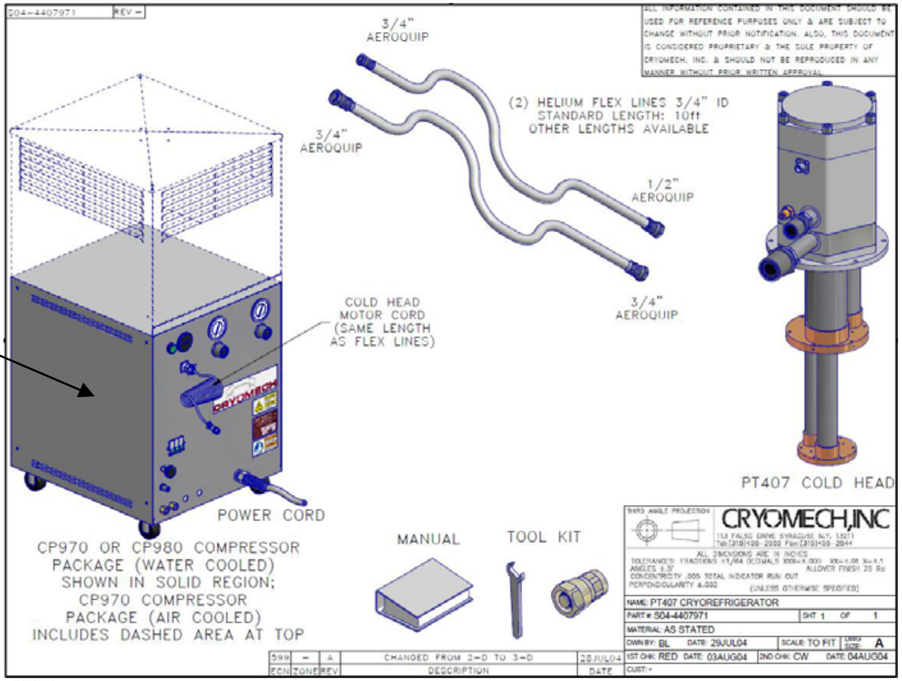


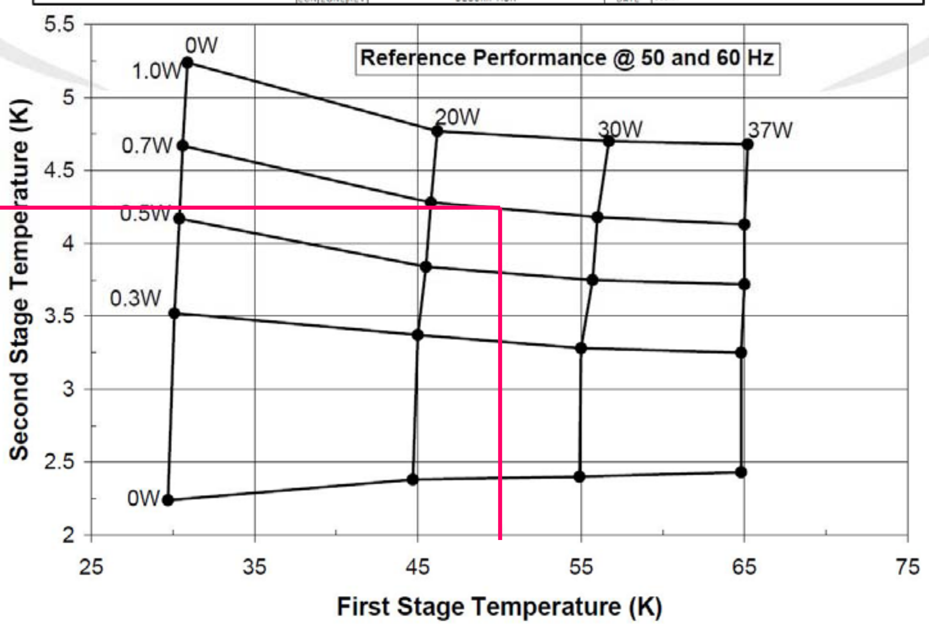
L….

ETAGE A DILUTION FREE 150µW



OUR DILUTION REFRIGERATOR USE A PT CRYOMECH 700mW @ 4.2K





Costs comparaison for one cooling

LIQUID HELIUM DILUTION REFRIGERATOR

Cooling from 300K to 4K : 100L,
Consumption by week : 100L

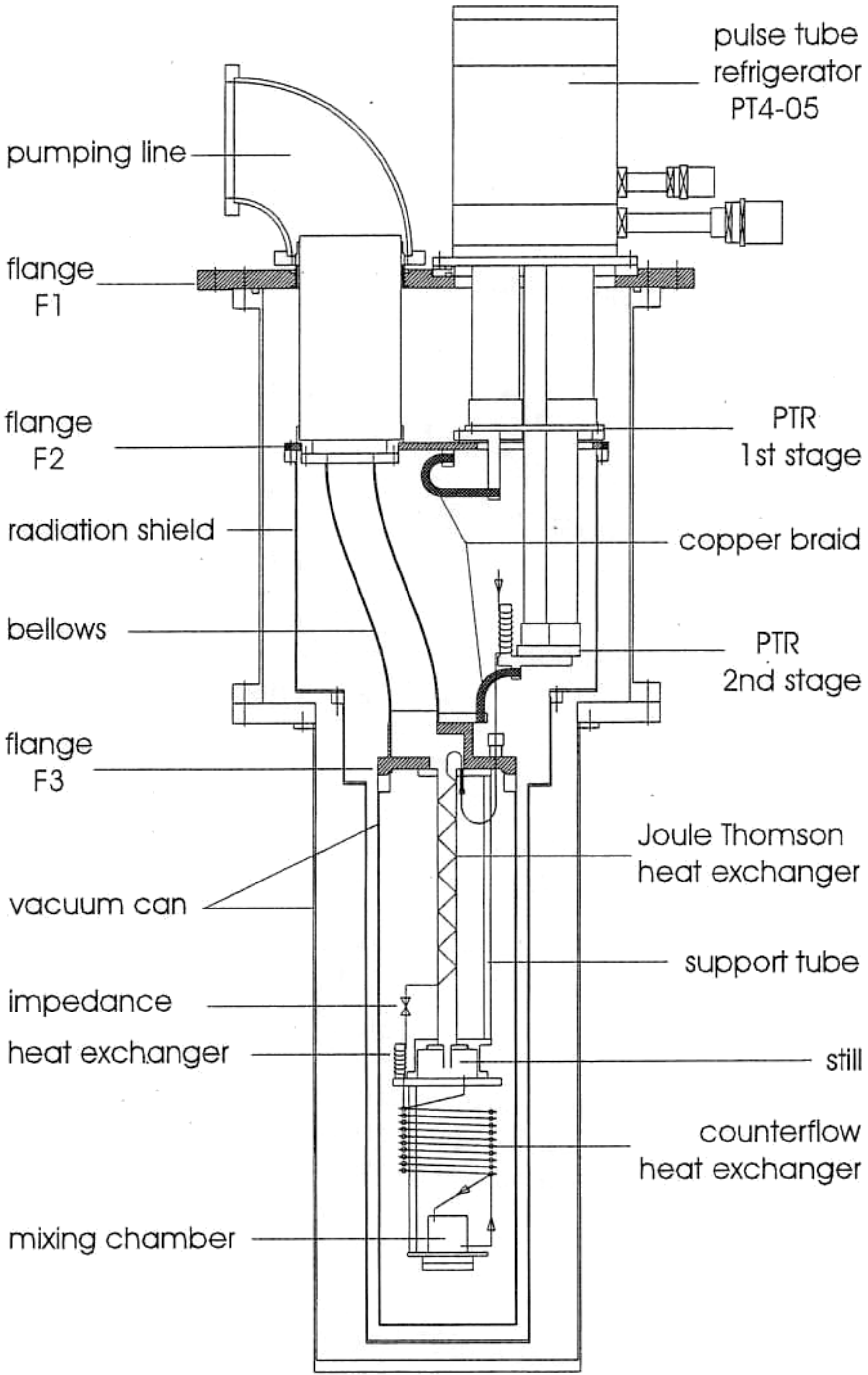
Total cost : 900€ of liquid helium (4.5€/l)

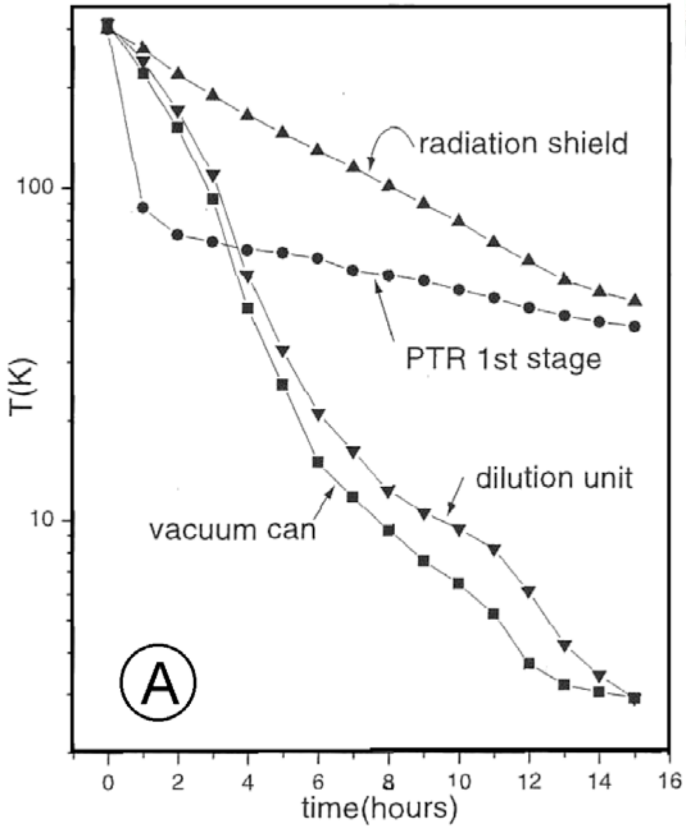
FREE DILUTION REFRIGERATOR

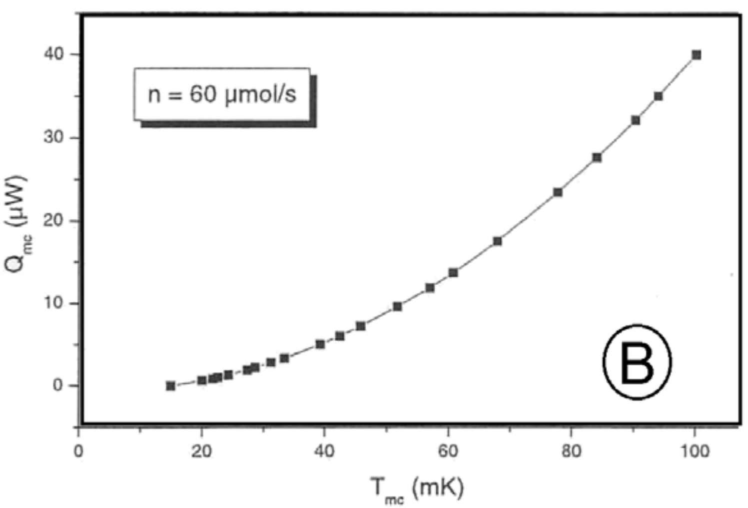
Cooling from 300K to 3K : 14 hours

Total electrical cost by week : 100€

Bibliographie :
many papers by Kurt Uhlig





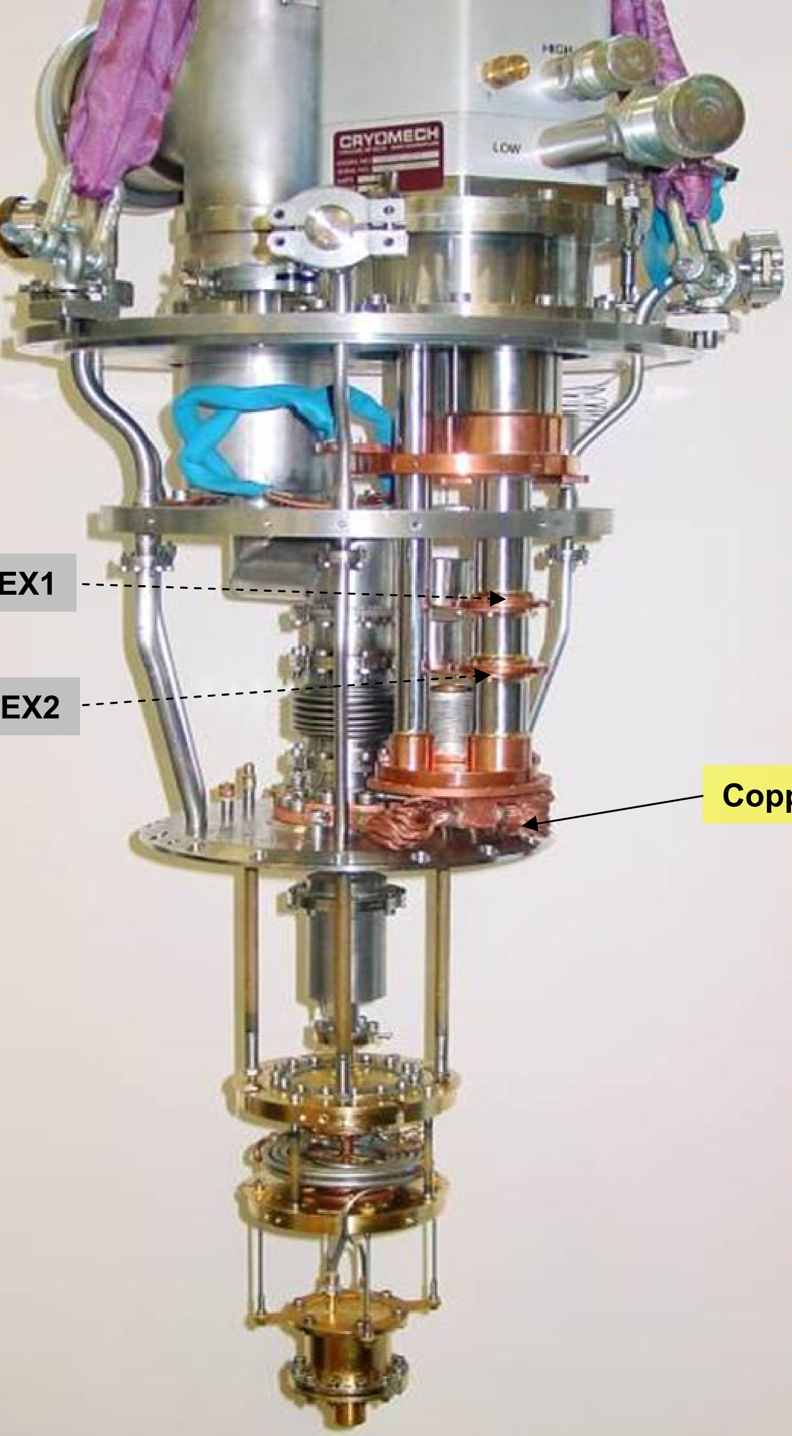


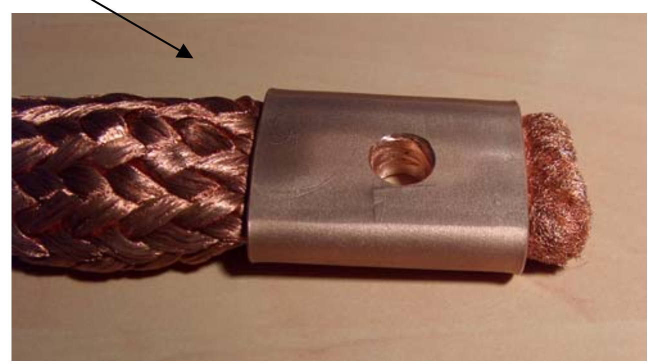
T. Prouvé, H.Godfrin, C.Gianèse. S.Triquenaux, A.Ravex

DEVELOPPEMENT OF DILUTION REFRIGERATOR PRECOOLING BY A PULSED TUBE

Thesis of Thomas Prouvé

(Joseph Fourier University, GRENOBLE) 2007

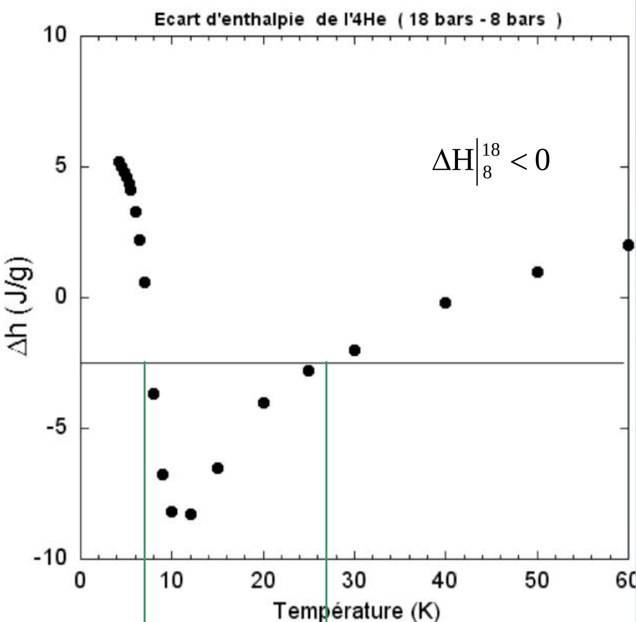




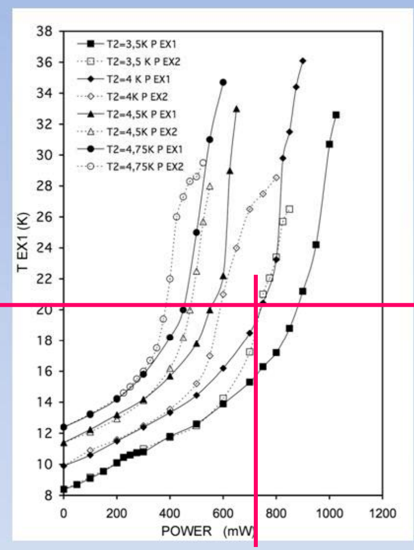
10 000 brins cuivre d = 1/1000^e mm

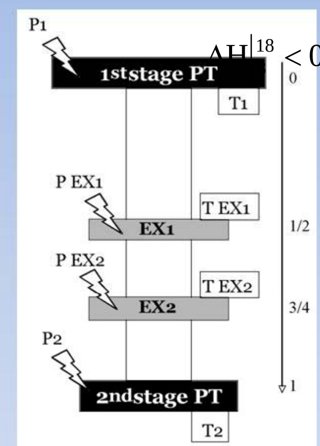
BETWEEN 6K AND 26K $\Delta H\big|_8^{18} < 0$ ITS POSSIBLE TO CHARGE THE REGENERATOR

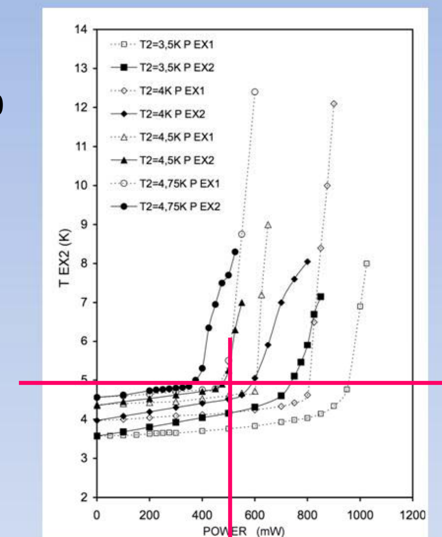
WITHOUT REDUCING THE COOLING POWER ON THE SECOND STAGE OF *PT*



"Free" cooling Power of PT = fast cooling!







Temperature of the first intercept T EX1 as a function of the power P EX1 applied to this intercept (solid lines and symbols), or to the second intercept PEX2 (dashed line and open symbols) for different temperatures T2 of the second stage.

Temperature of the second intercept T EX2 as a function of the power PEX2 applied to this intercept (solid lines and symbols), or to the first intercept P EX1 (dashed line and open symbols) for different temperatures T2 of the second stage.

Pulse-tube dilution refrigeration below 10 millikelvins, T. Prouvé, H. Godfrin, C. Gianèse, S. Triqueneaux, A. Ravex
J. of Low Temp. Phys. 148, 909 (2007)

Pulse-tube dilution refrigeration below 10 mK for Astrophysics, T. Prouvé, H. Godfrin, C. Gianèse, S. Triqueneaux, A. Ravex
J. of Low Temp. Phys. 151, 640 (2008).

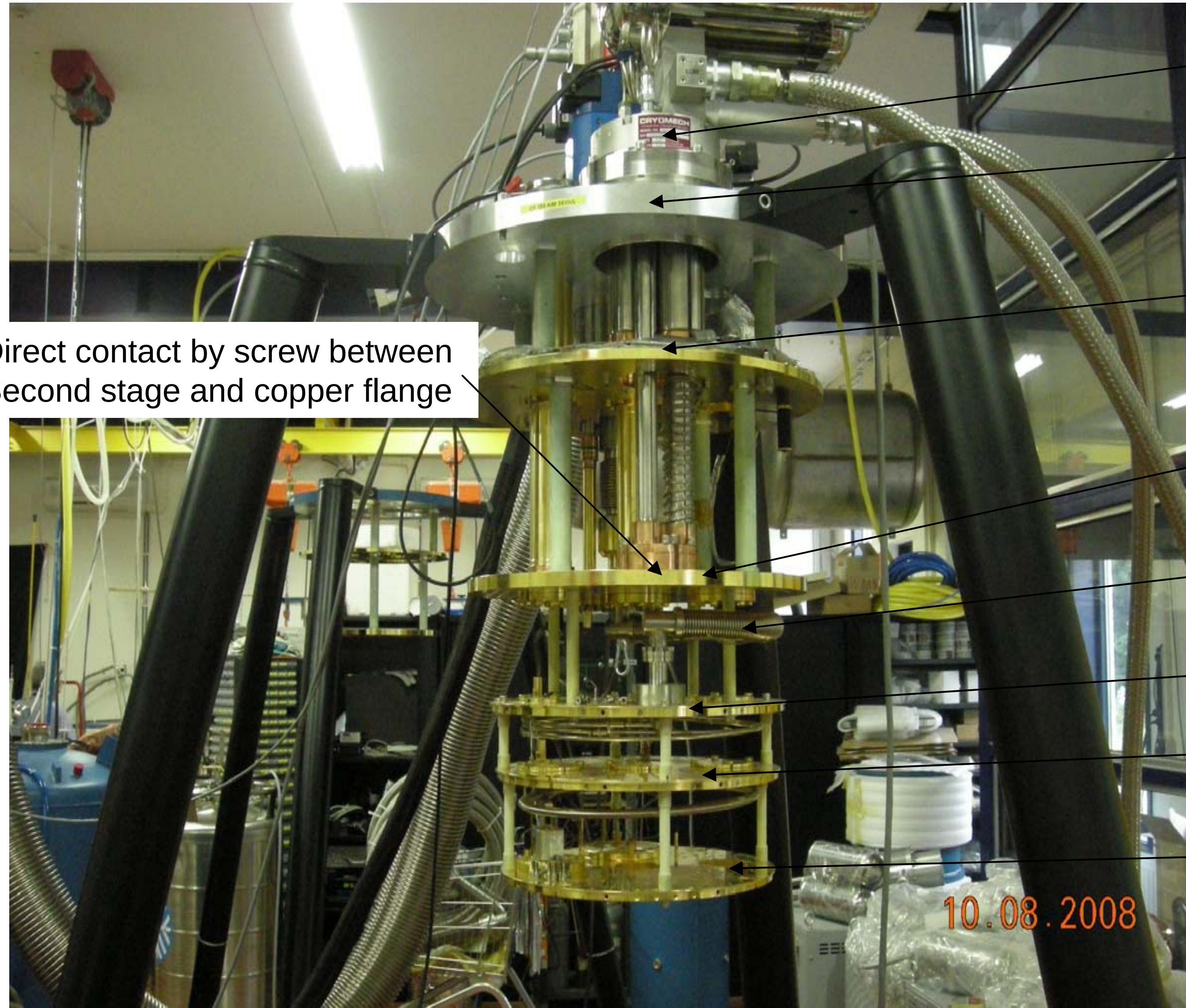
Air Liquide, U.S. Patent 6,915,642 and CNRS-Air Liquide Patent FR07 53945

EX1 700mW@20K

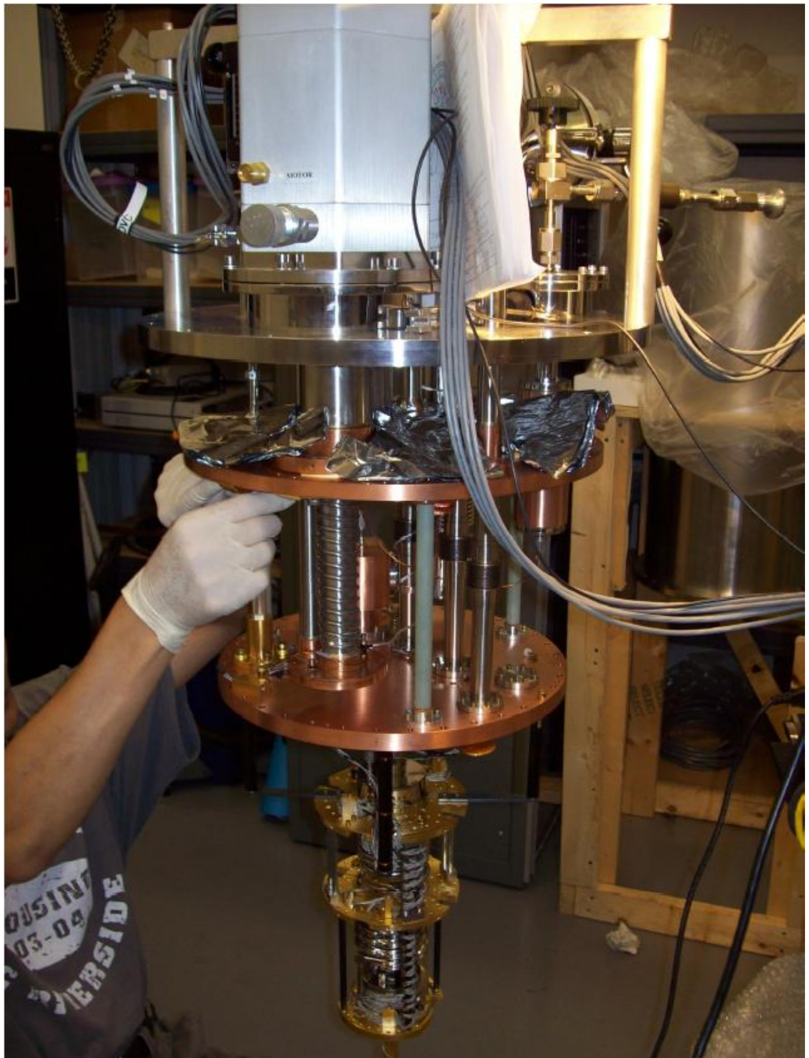
EX2 500mW@6K

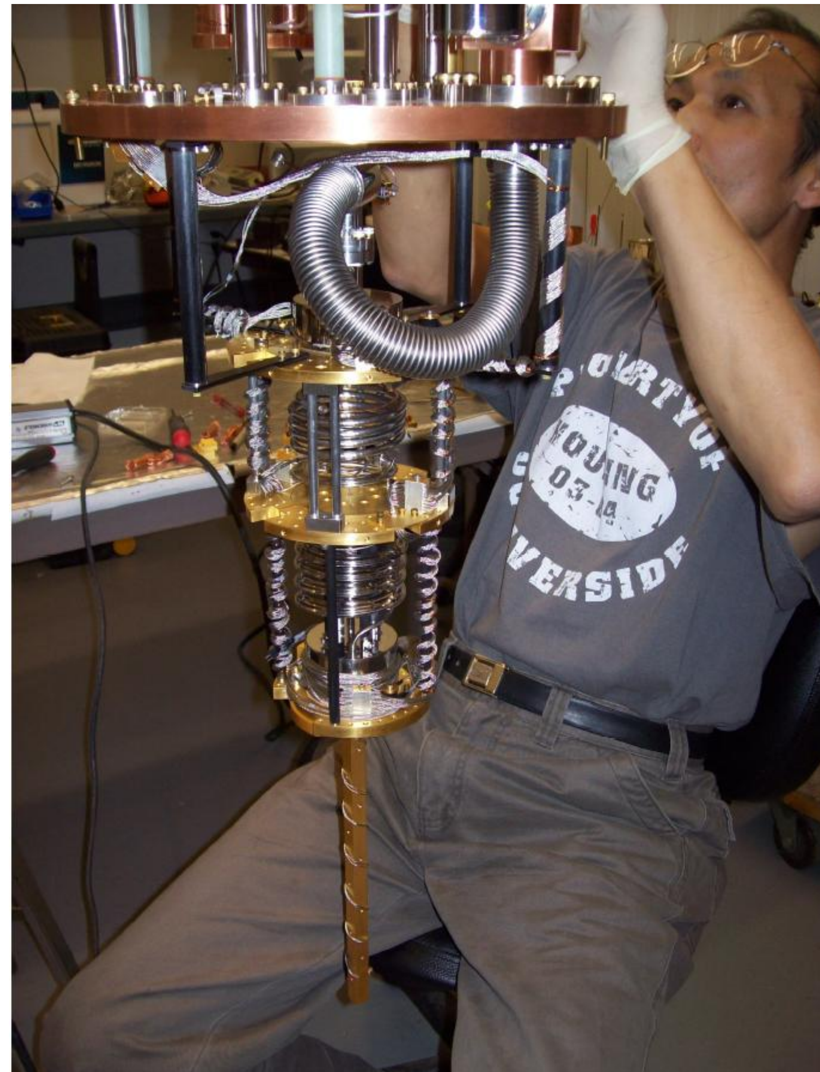
The visit to Giorgio Frossati's company at LEIDEN

COMMERCIAL DRY DILUTION REFRIGERATOR LYDEN CRYOGENIC (*Giorgio Frossati)*



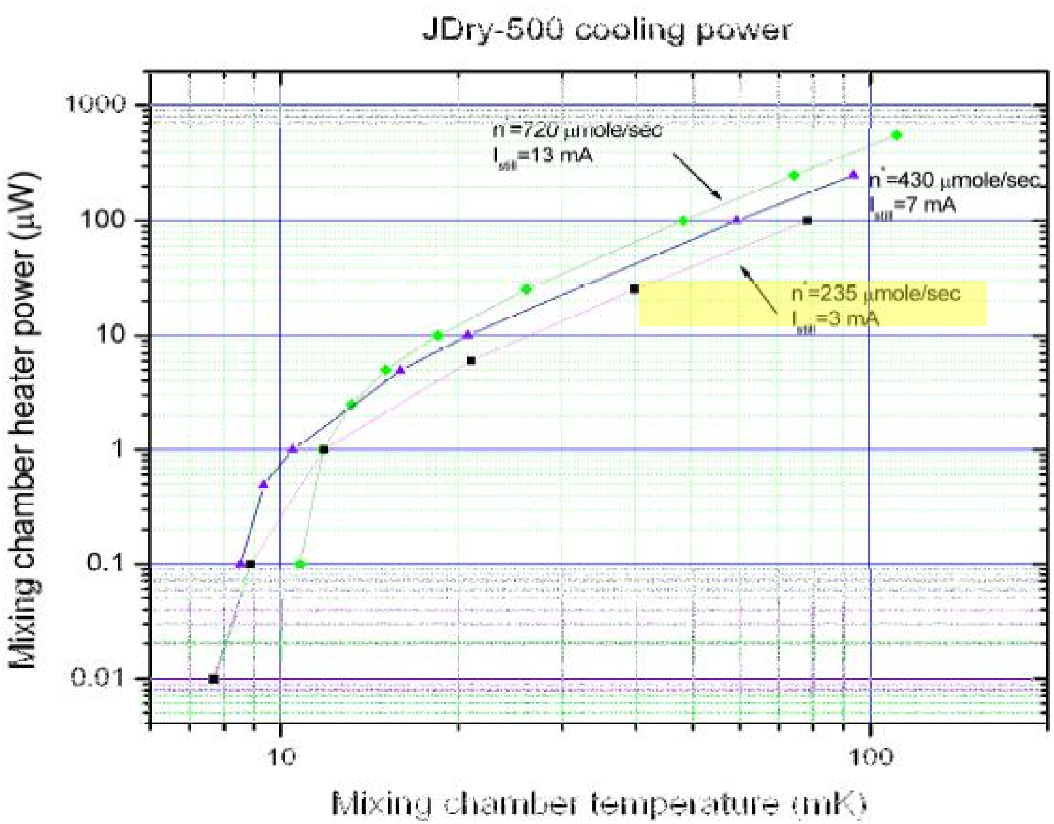
J DRY 500 (JANIS)





Specifications after first cool-down:

1. 36 hours (conservative first cool-down, heat switch not used yet) from start to base temperature using PT-405 pulse tube;
2. 7.5 mK base temperature at optimal circulation rate as measured with compact CMN thermometer, calibrated against superconducting fixed point device;
3. Cooling power up to 450 µW at 100 mK at maximum circulation rate (see plot below);
4. Very quiet oil-free pumping station based on Roots technology (water cooled);
5. No JT compressor required, condensing pressure is always below 0.7 bar absolute (see in Kurt Uhlig, "Condensation stage of a pulse-tube pre-cooled dilution refrigerator", accepted to Cryogenics, 2008);
6. Janis Labview 7 software for resistive and CMN thermometry, as well as pressure and flow control;
7. No change in performance when tilted at 11 degrees;



NOTRE DILUTION FREE

- *REFROIDISSEMENT LE PLUS RAPIDE POSSIBLE 300K>3K*
- *PAS D IVC SEULEMENT ETANCHEITE A TEMPERATURE AMBIANTE*
- *SEULEMENT 3 ECRANS CUIVRE OU ALUMINIUM (60K 4K 1K)*
- *AUCUNE PARTIE METALLIQUE EN CONTACT A FROID ENTRE LE PT ET FRIGO*
- *DEMONTAGE LE PLUS SIMPLE POSSIBLE DU PT POUR REVISION.*
-
- *PRECOOLING*
- *CIRCULATION A DEBIT ELEVE (20 000µmoles/s) ET A CONTRE SENS EN INJECTANT DANS LA GAÎNE PRINCIPALE, SORTIE PAR LE DB*
- *ASSERVISSEMENT DU DEBIT POUR ATTERRIR A 3K AU BOUT DE 8 à 10 HEURES*
- *INVERSION DU DEBIT VERS 3K*

- *COOLING*
- *POMPAGE DU MELANGE PAR LA GAÎNE PRINCIPALE ET LE DB*
- *INJECTION NORMALE ET REFROIDISSEMENT AVEC LE JT DEPUIS 3.5K*
- *COMPRESSEUR OFF EN FIN DE CONTENSATION*

NORMAL JT DILUTION STAGE

DOUBLE STILL JT DILUTION STAGE

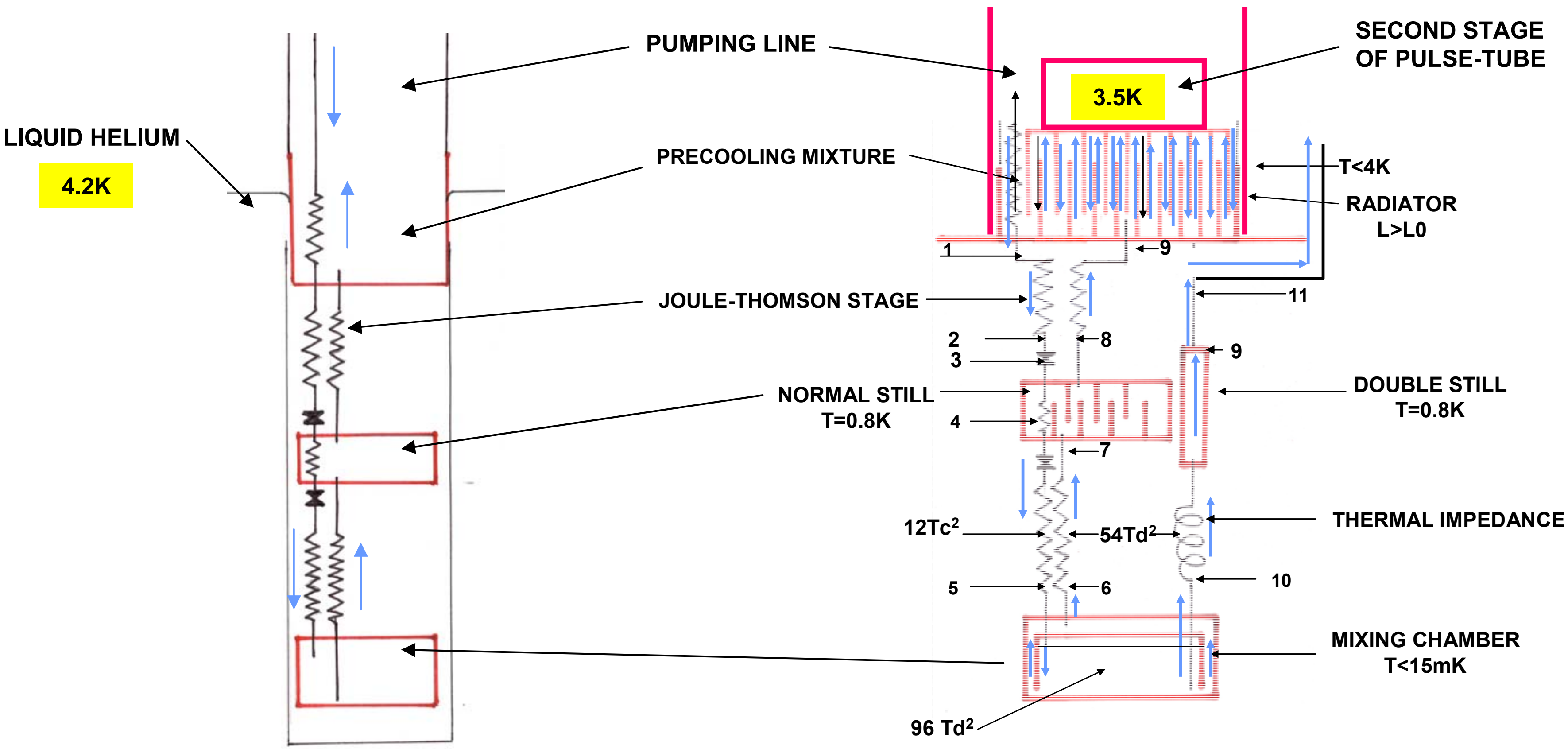


DIAGRAMM OF RAPID COOLING 3<T(K)<300

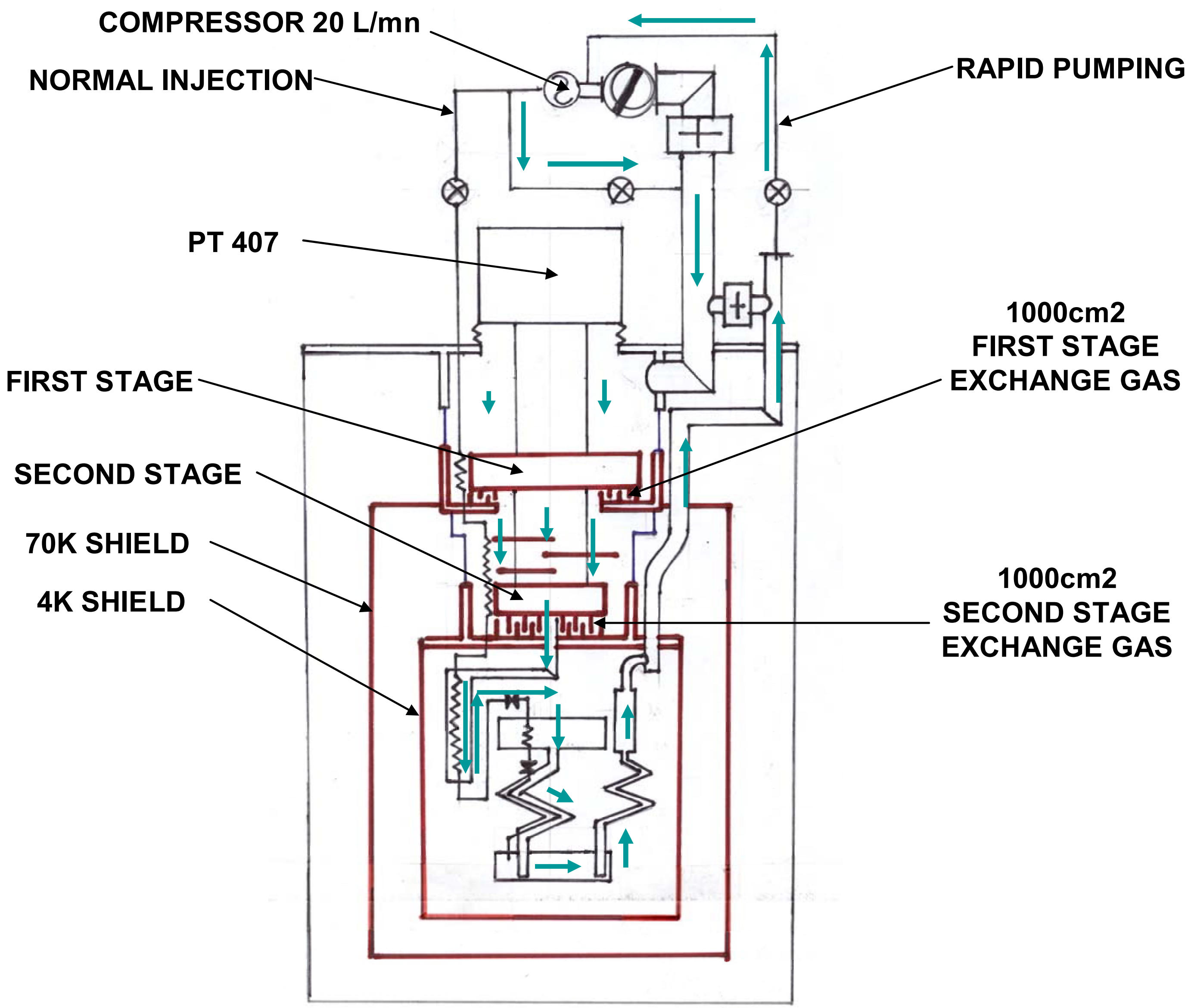
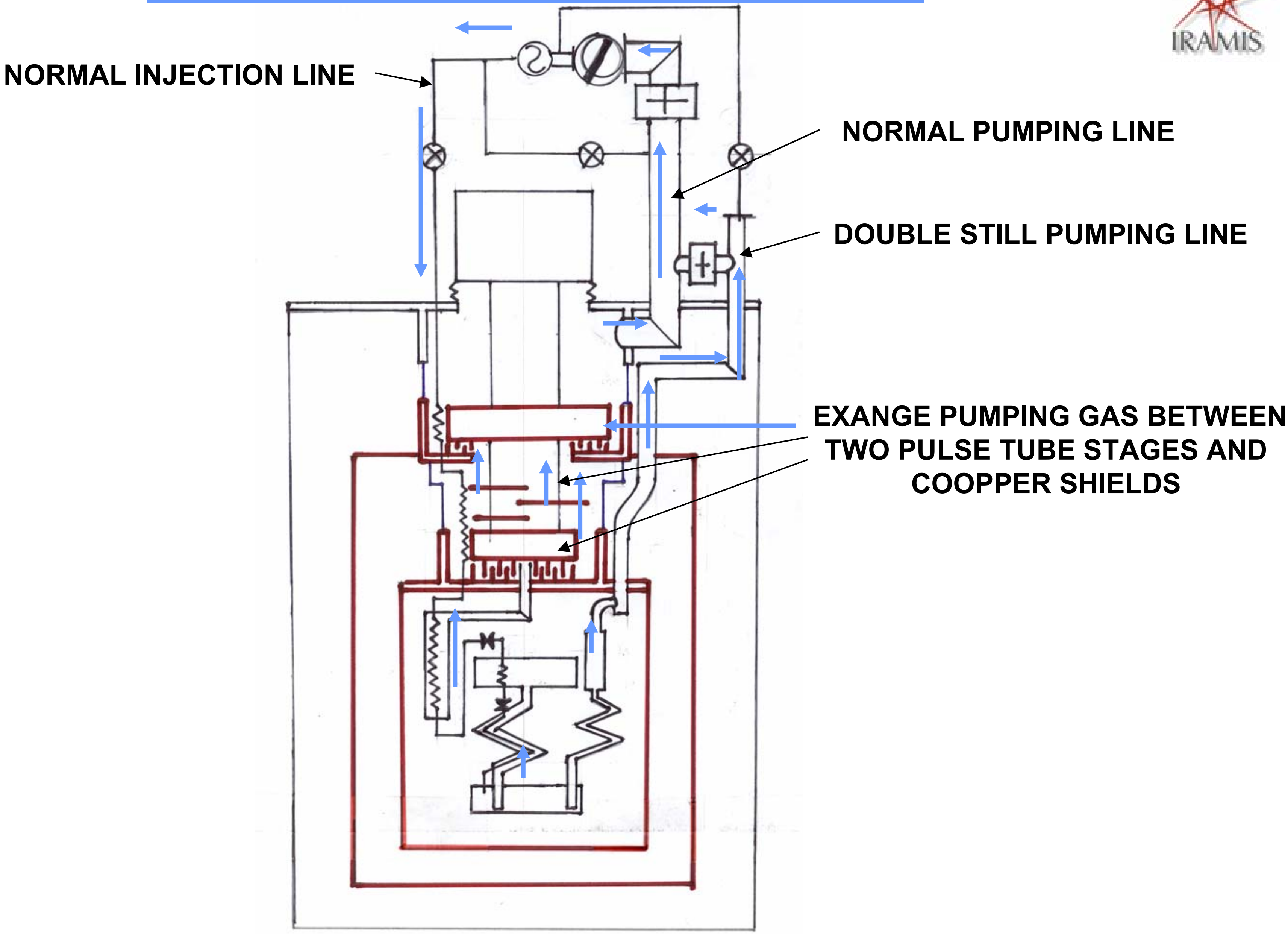
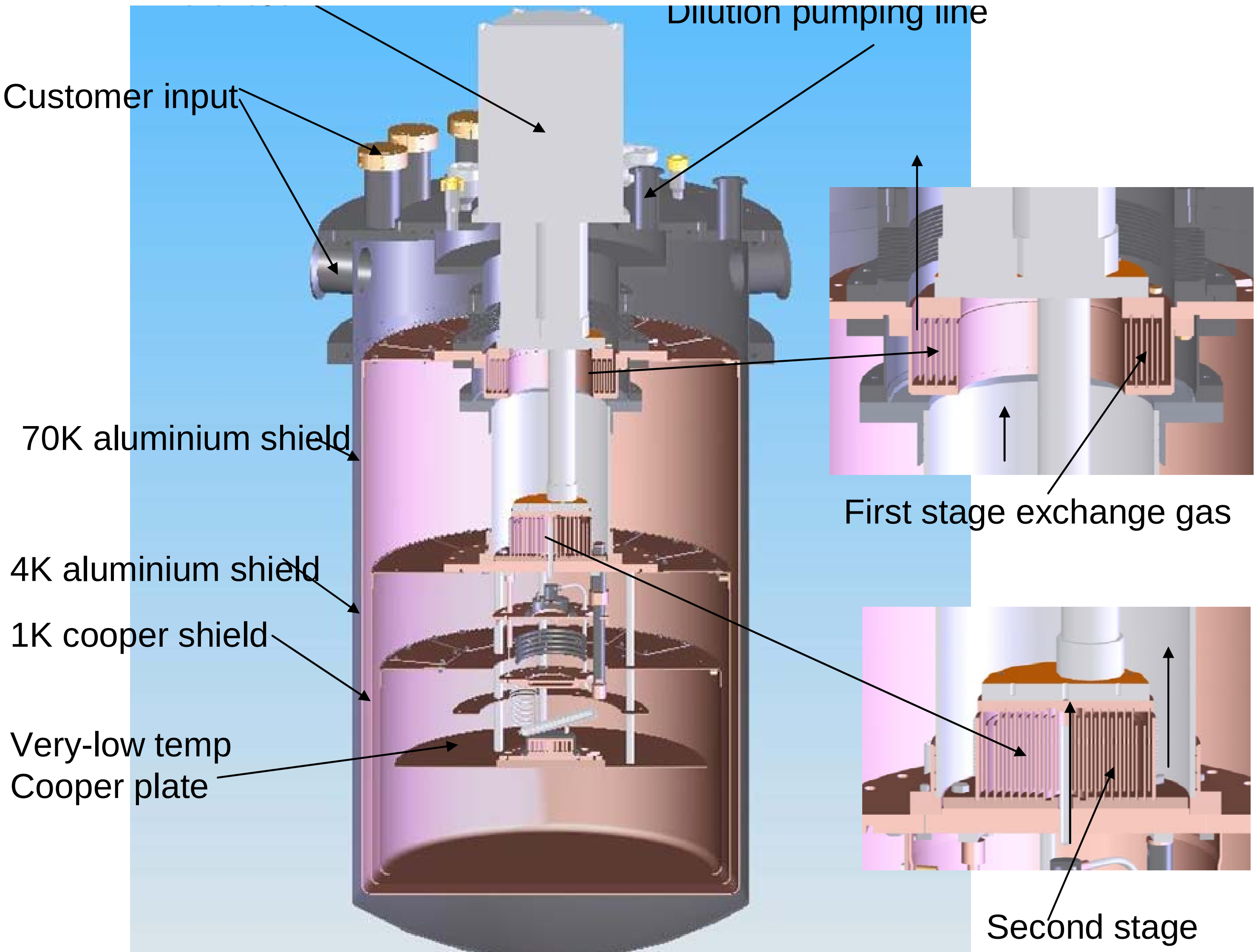


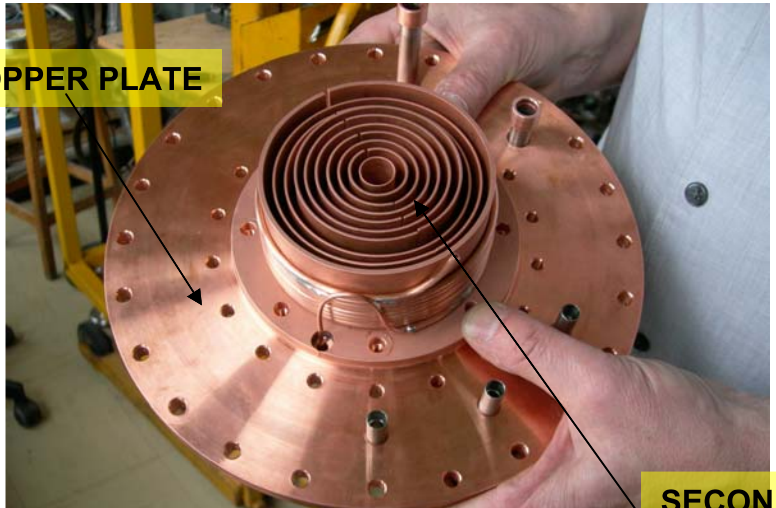
DIAGRAMM OF NORMAL COOLING 16mK<T<3K

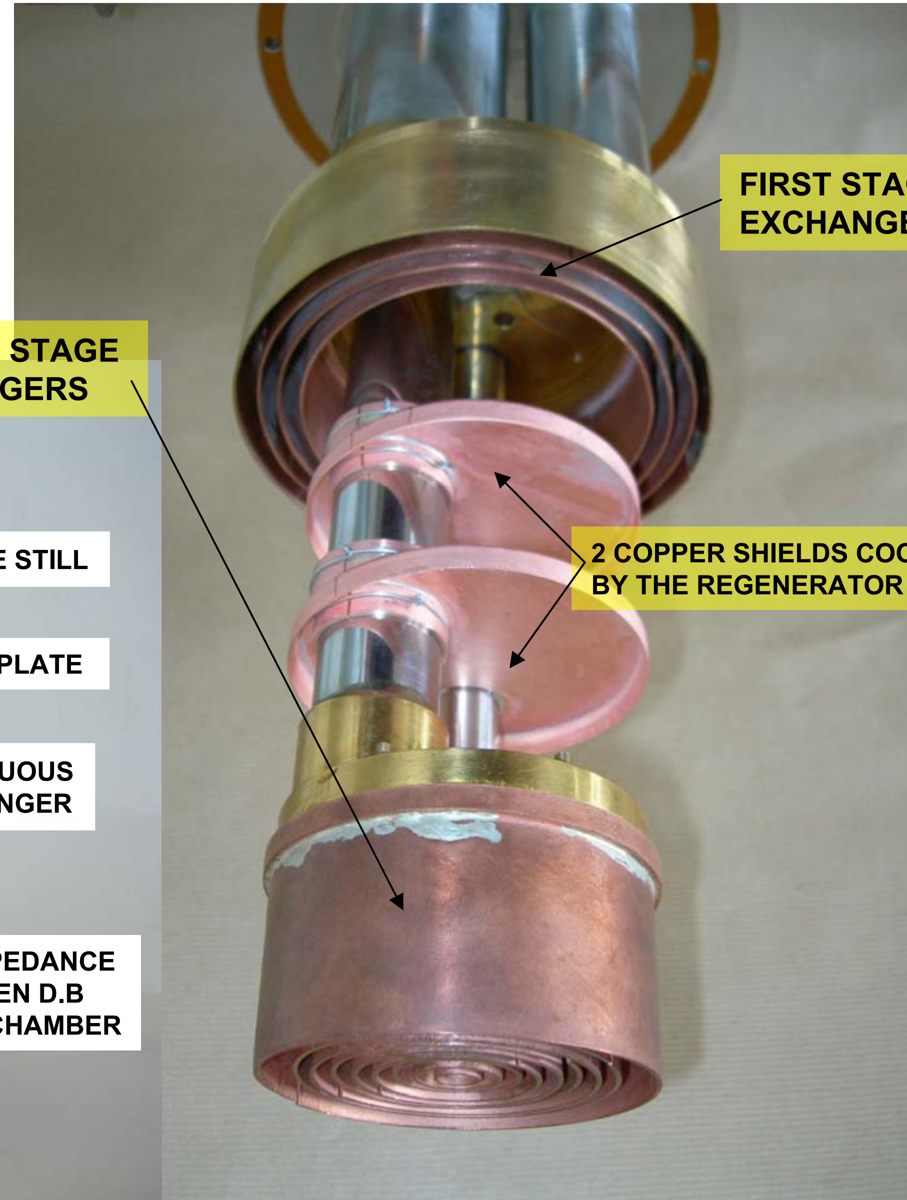


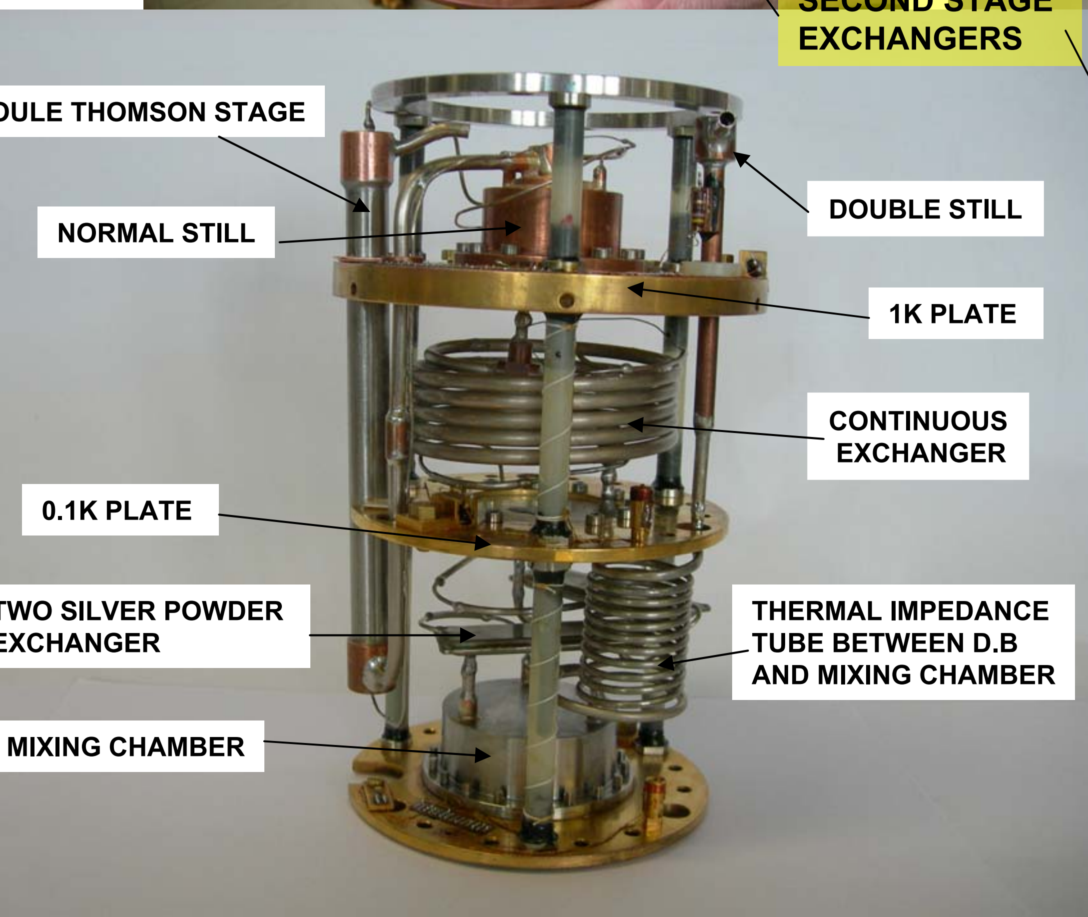


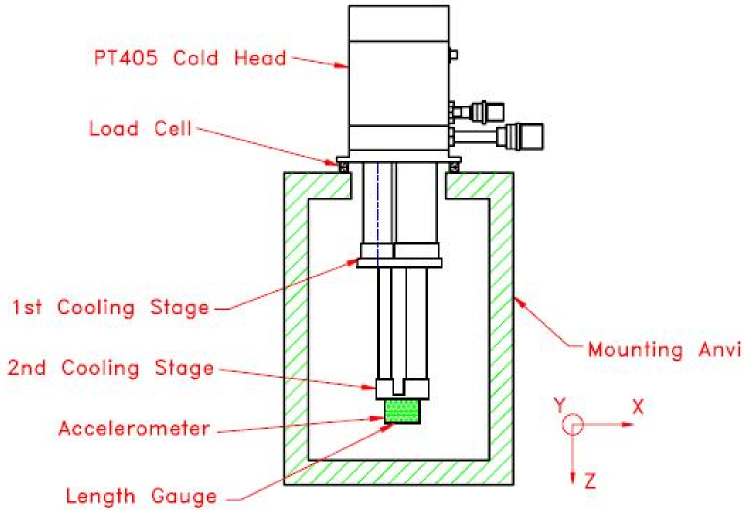
DRY DILUTION REFRIGERATOR
WITHOUT COLD SOLIDS CONTACTS AND NOT IVC

EXCHANGERS BETWEEN FIRST STAGE AND SECOND STAGE OF *PT* .

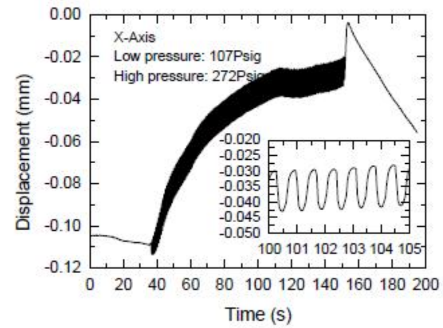


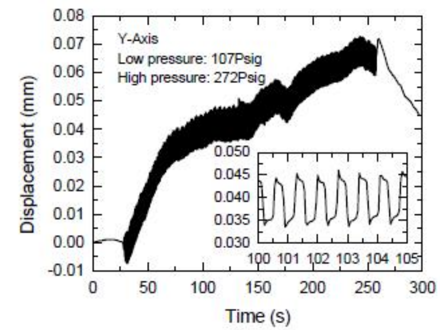






Dynamic displacements on the 2nd stage cold head. The PT405 starts from room temperature.

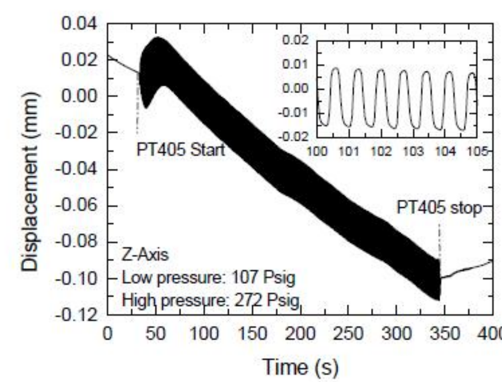




Amplitude of displacement of the 2nd stage cold head on three directions

	X-axis	Y-axis	Z-axis
Length Gauge* Measurement	6 μm	6μm	12μm

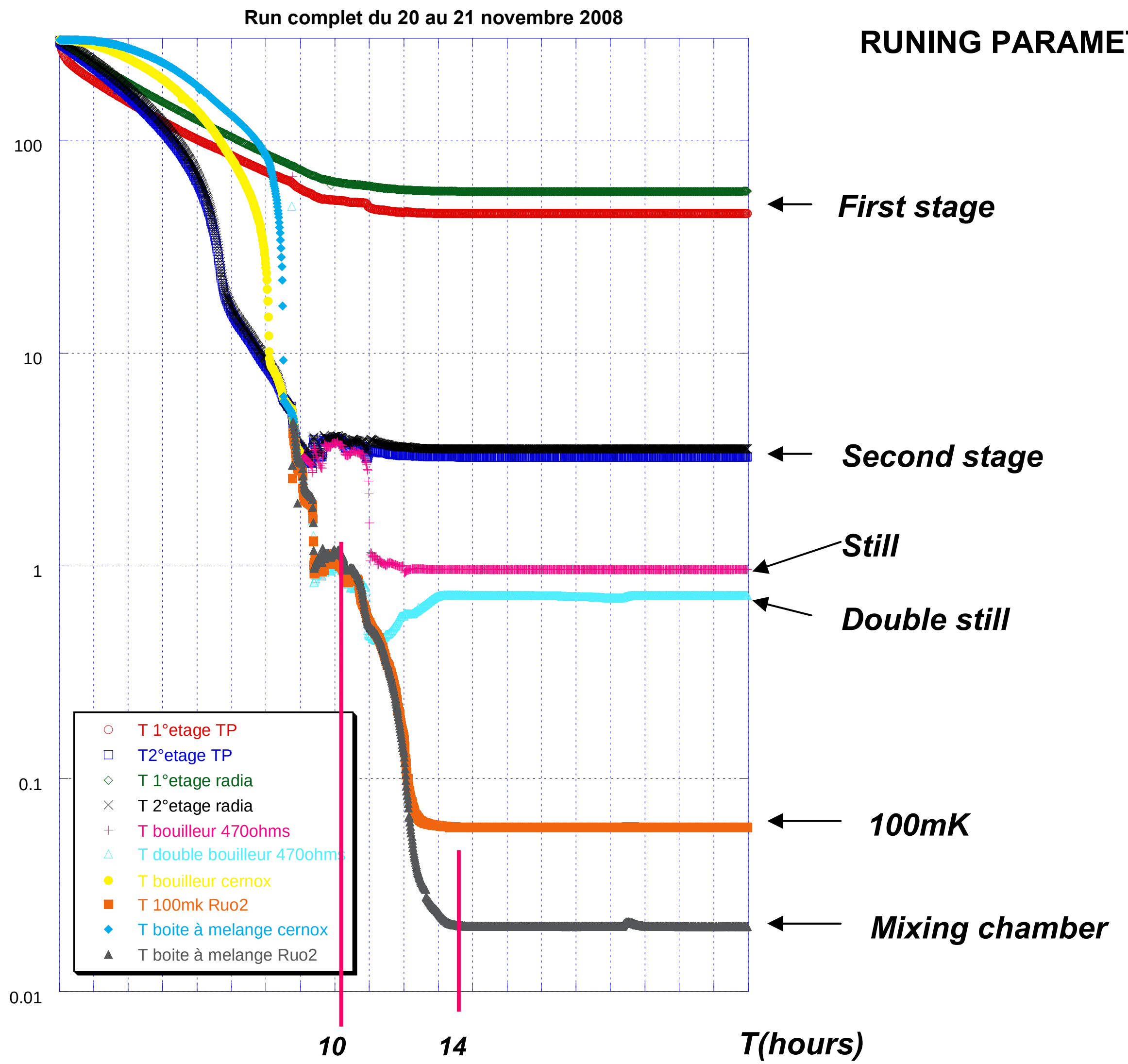
*Suction pressure: 107Psig; Discharge pressure: 272Psig; (4 K operation pressures)



GENERAL VIEW OF FREE DILUTION REFRIGERATOR

PT1	RPT1	PT2	RPT2	Still	Double Still	100mK	MC	P_P	P_{INJ}	n
47K	61.6K	3.34K	3.64K	0.937K	0.72K	60.5m K	16mK	9.8 10^{-2}	410mb	210µmoles

RUNING PARAMETER OF FREE DILUTION REFRIGERATOR

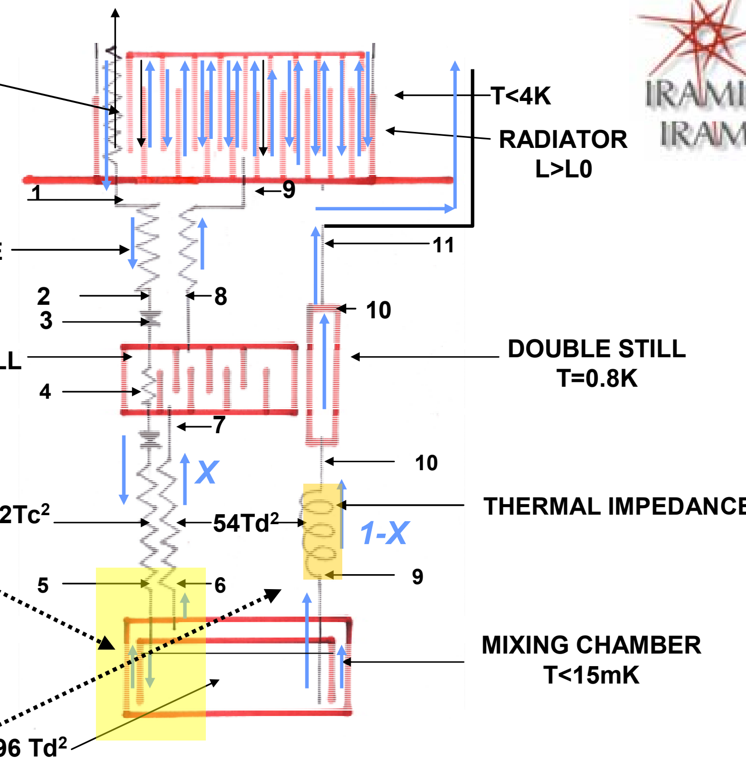


Thermal bilan

$$\dot{Q}_{ext} = \dot{n}_3[X96T_D^{\,2} - 12T_C^{\,2}]$$

$$Pour\ \dot{Q}_{ext} \to 0 \qquad 12T_C^2 = 96XT_D^2$$

$$X = 1 : \frac{T_C}{T_D} = 2.8 \qquad X = 0.8 : \frac{T_C}{T_D} = 2.5$$



The limit temperature is not affected by the circulation in the double still as it is less than 20%

Thermal insulation between mixing chamber and second still

$$L >> L_0$$

$$L_0 = \frac{\pi D^2 K}{4\dot{n}C}$$

cooling power : the same as

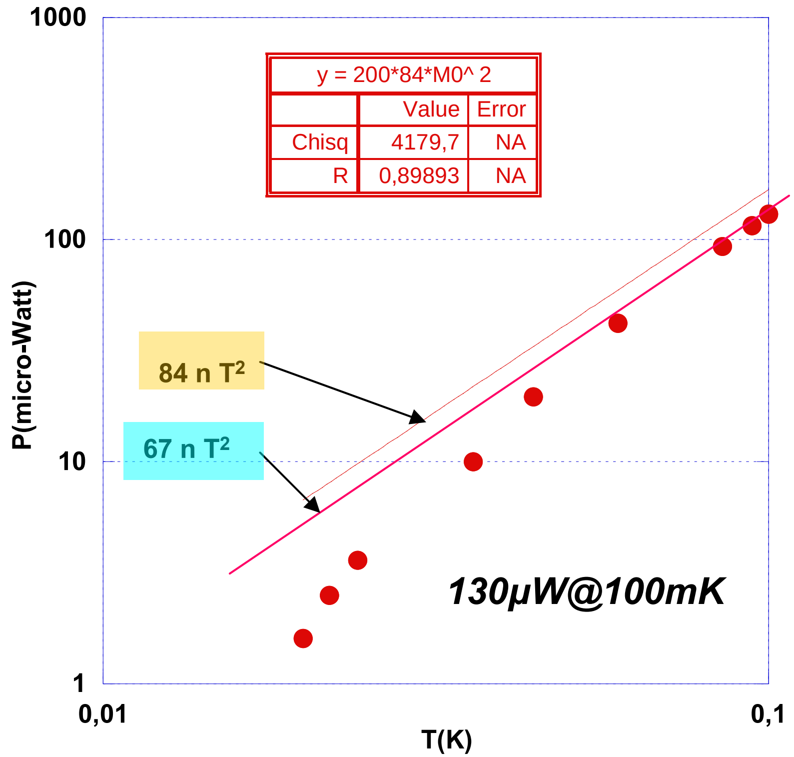
for a system without double still.

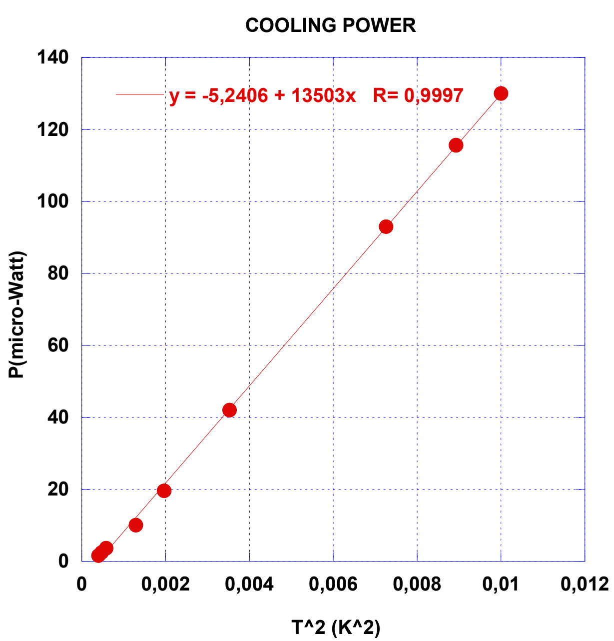
$$\dot{Q}_{ext} = \dot{n}_3 [\ 96 T_D^{\ 2} - 12 T_C^{\ 2}]$$

For $T_D = T_C$

$$\dot{Q}_{ext} = \dot{n}_3\, 84 T_{MC}^2$$

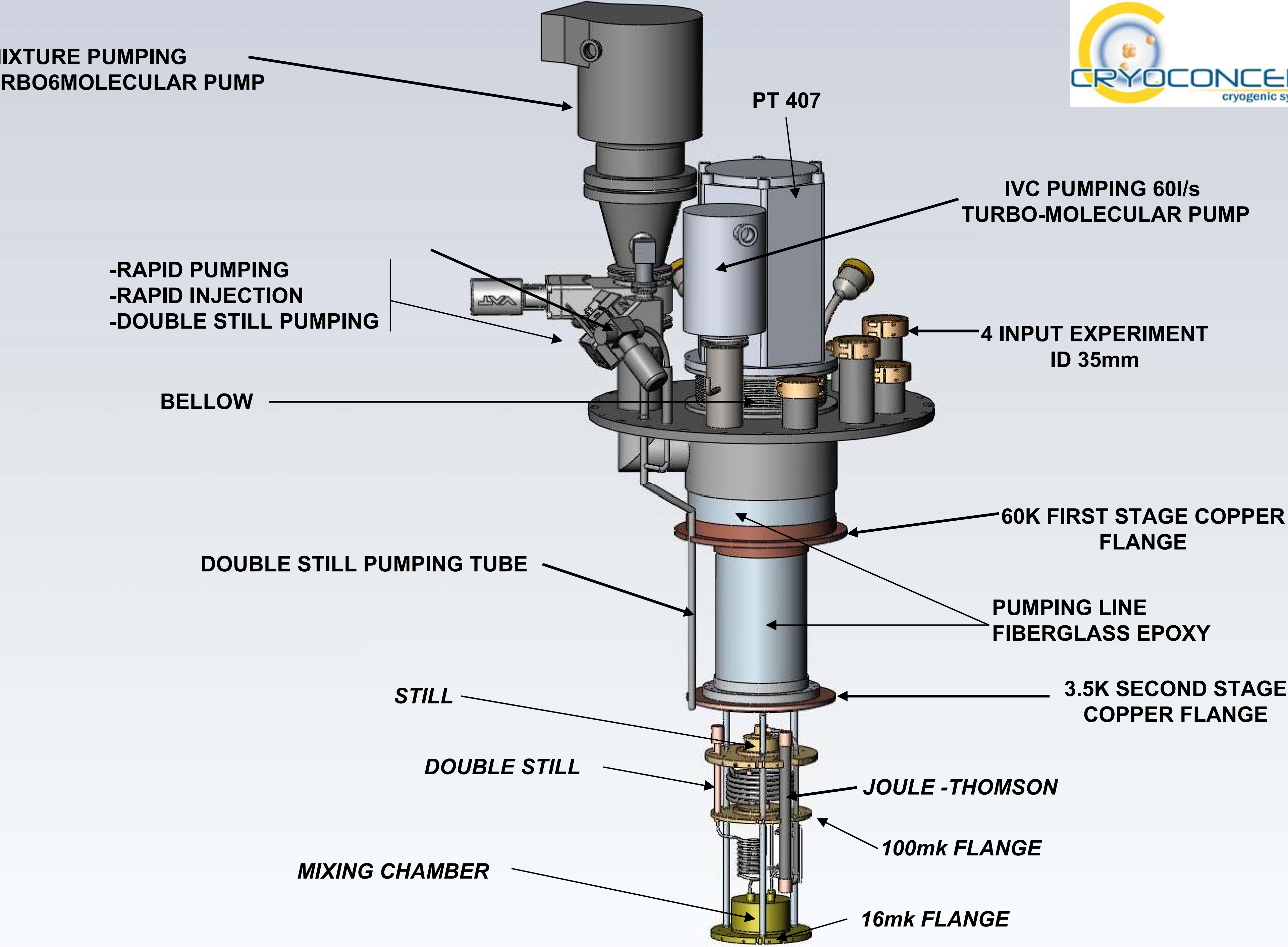
Cooling power with Q_{STILL}=0
12m³/h + turbo-pump 250l/s





Normal still temperature too high
He4 flow-rate : 20%

Normal still	Double still
0.937K	0.72K



IXTURE PUMPING
RBO6MOLECULAR PUMP
PT 407
IVC PUMPING 60l/s
TURBO-MOLECULAR PUMP
-RAPID PUMPING
-RAPID INJECTION
-DOUBLE STILL PUMPING
4 INPUT EXPERIMENT
ID 35mm
BELLOW
60K FIRST STAGE COPPER
FLANGE
DOUBLE STILL PUMPING TUBE
PUMPING LINE
FIBERGLASS EPOXY
3.5K SECOND STAGE
COPPER FLANGE
STILL
DOUBLE STILL
JOULE -THOMSON
100mk FLANGE
MIXING CHAMBER
16mk FLANGE

CONCLUSION

-On démontre la faisabilité du système avec refroidissement rapide, et des paramètres fonctionnels corrects.

-300K------>16mK : 14H00, 130µW@100mK

PERSPECTIVES

-Possibilité d'utiliser un PT à vanne tournante déportée : boucle de masse, rayonnement électromagnétique, vibrations.

-Limiter au maximum de débit dans le double bouilleur Max = 10%

-Améliorer l'efficacité des échangeurs:

1- sur le PT entre le 1^{ER} et le 2^{EME} étage pour diminuer la température du deuxième étage : suppression compresseur.

2- les échangeurs à Ag + performants.

-Conserver le refroidissement rapide même avec des masses plus importantes.

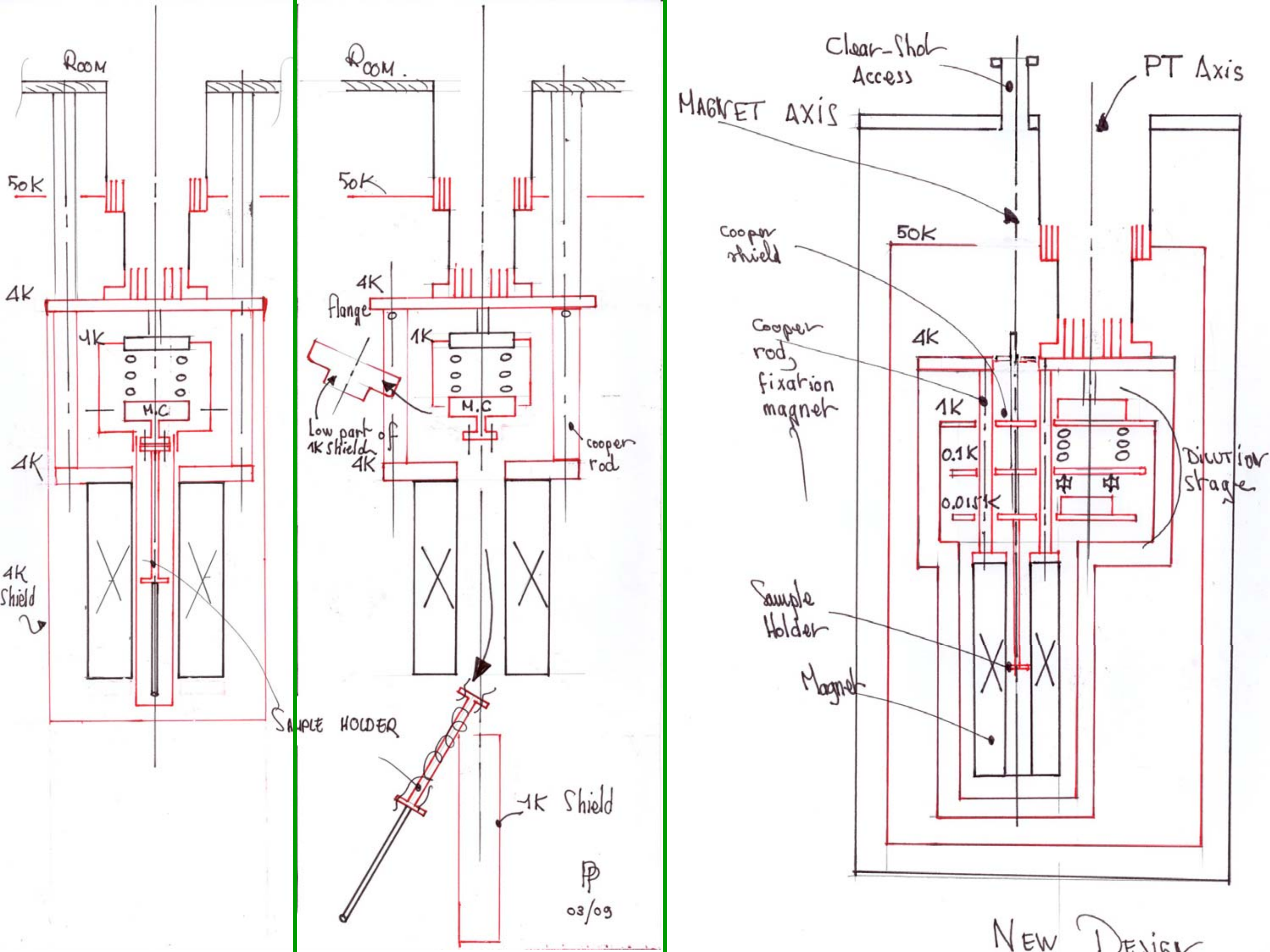
Interrupteurs thermique entre platine 3K et 0.8K, boucles à He4 gaz, voir N2 pour des très gros systèmes.

-Vers l'intégration des bobines FREE à champ magnétique élevé.

-Solutions « réchauffement rapide ».

-Diminuer les vibrations: projet ASTRE collaboration CEA, CNRS, CRYOCONCEPT.

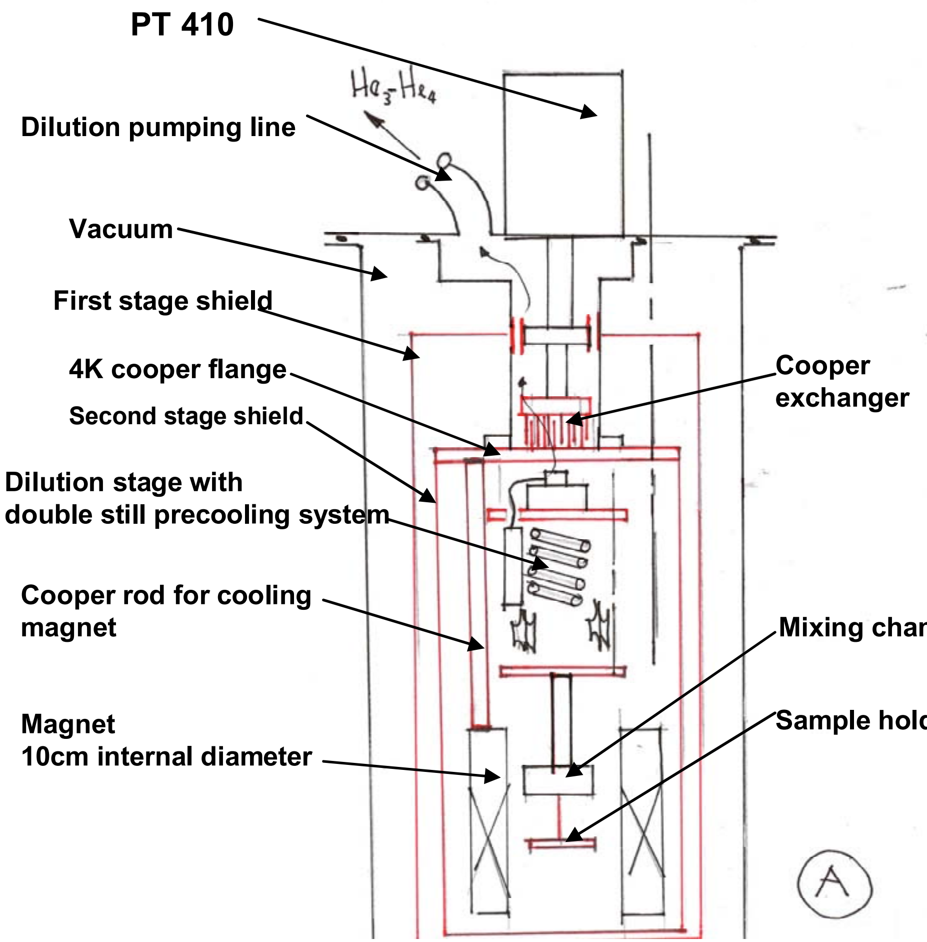
-Augmenter les puissances des systèmes 200µW@100mK, (PT407), 400µW@100mK, (PT410).

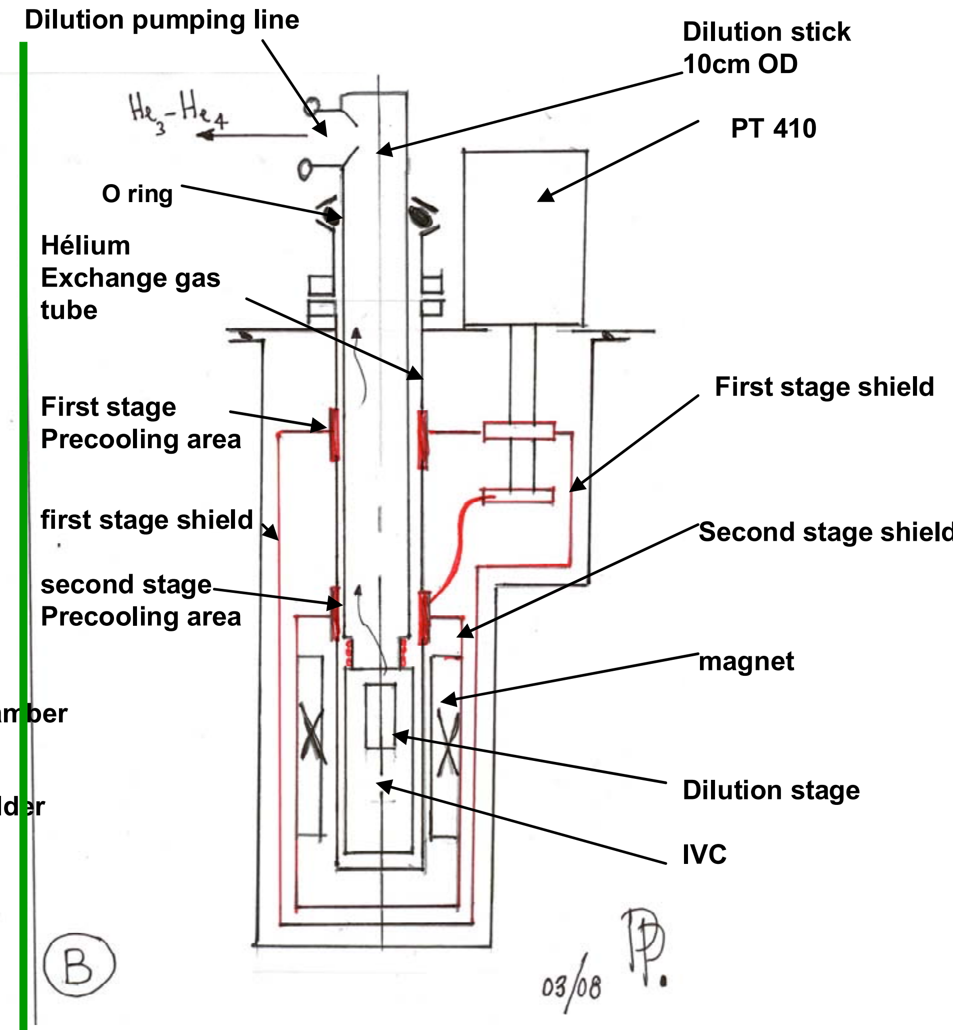

Room
50K
4K
1K
M.C
4K
4K Shield
Sample Holder
Room.
50K
4K
flange
1K
M.C
Low part of 1K shield
4K
cooper rod
1K Shield
03/09
Clear-Shot Access
PT Axis
MAGNET AXIS
50K
Cooper shield
Cooper rod fixation magnet
4K
1K
0.1K
0.015K
Dilution stage
Sample Holder
Magnet
New Design

Two possible designs :

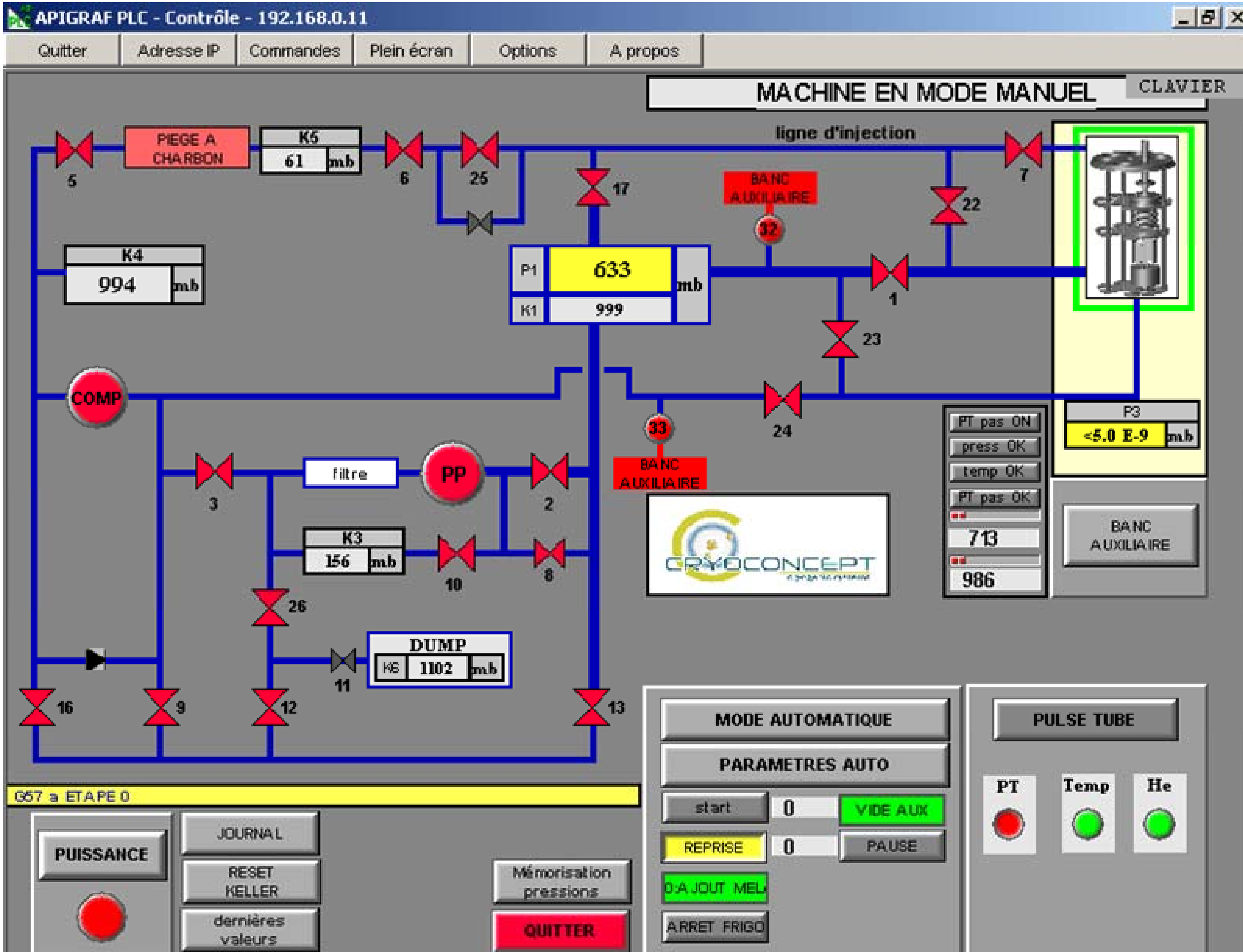
A : the fridge and the magnet are in a single vacuum chamber. (Concept of Mignot fridge without coil). The DR and the magnet are cooled down together with the PT before starting the DR unit. Precooling of the DR is done using a double-still system. There is a single room temperature seal. This system provides a large experimental room.

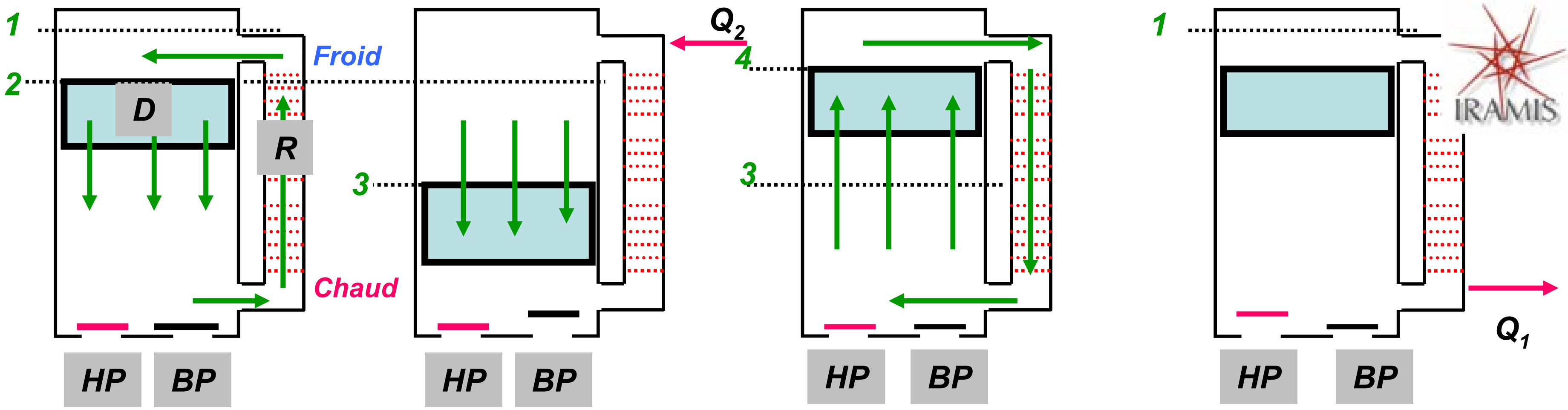
B: **the coil stays cold when heating the fridge** The fridge is installed in a stick with gazeous thermal contacts to the two PT stages. The fridge is precooled using helium exchange gas when inserting the stick. The vacuum can diameter is 10 cm. For precooling, Helium exchange gas is placed in the IVC containing the DR stage and the experiment, and the He3-He4 mixture is present in the stick, which is the pumping line of the DR unit. This system allows to change a sample without heating the magnet.



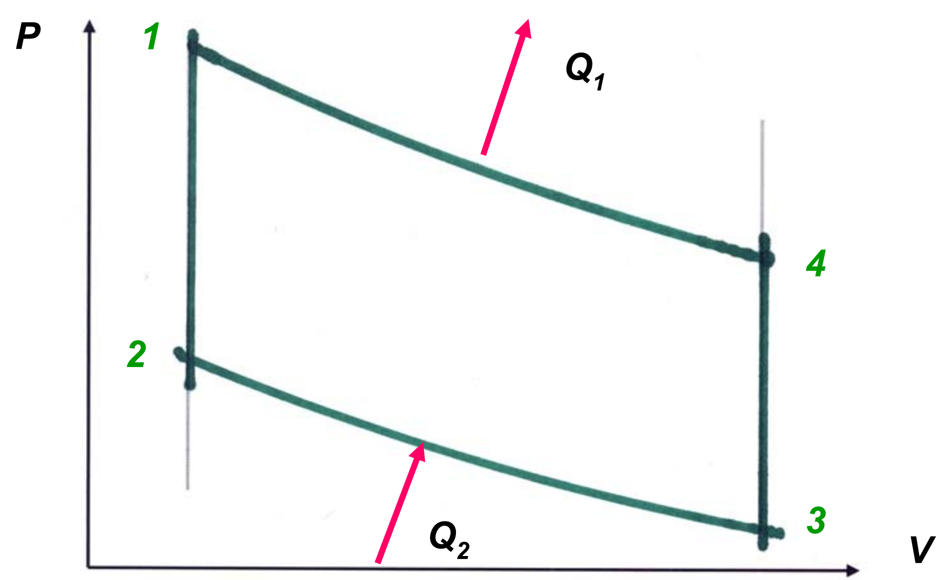


CRYOCONCEPT *GHS*





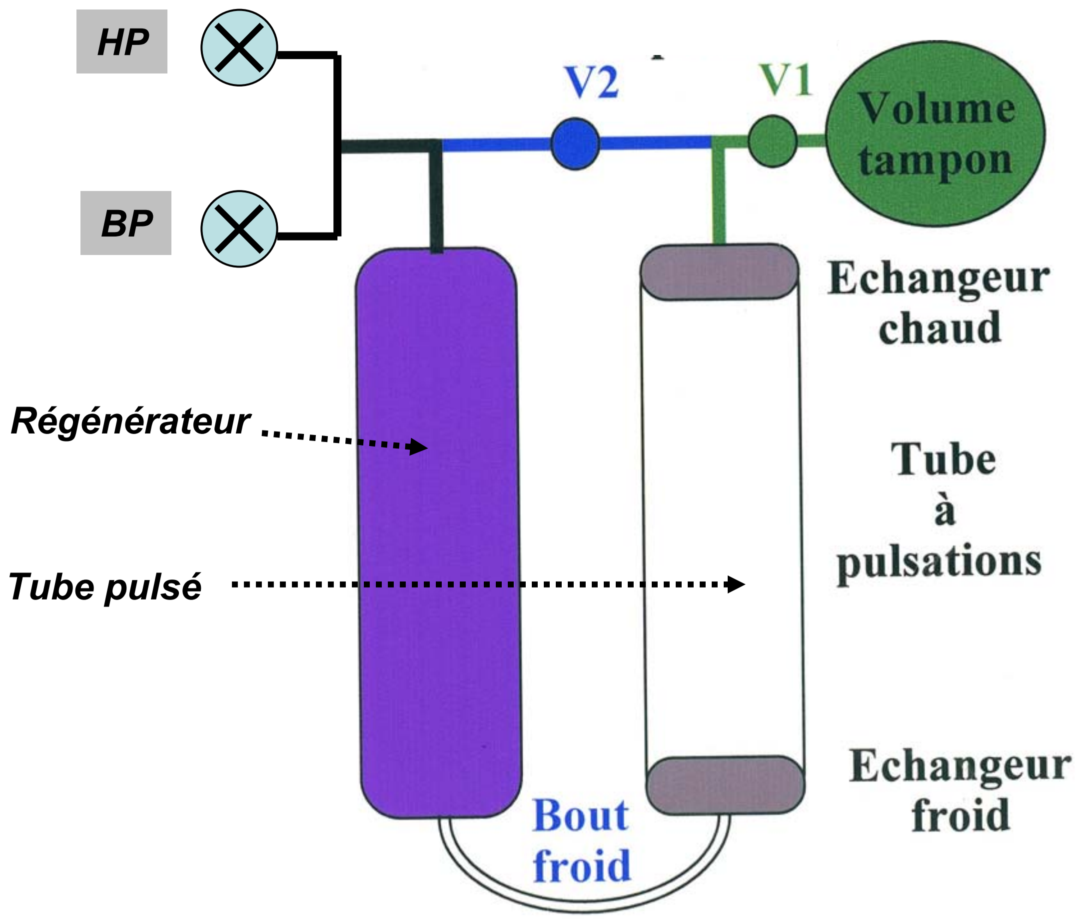
1 > 2 : Refroidissement isochore *(déplacement du piston interne, le gaz traverse le régénérateur)*
2 > 3 : Détente isotherme *(le gaz se refroidit en adsorbant Q_2 via le régénérateur)*
3 > 4 : Réchauffement isochore *(déplacement du piston interne, le gaz traverse le régénérateur)*
4 > 1 : Compression isotherme *(le gaz se réchauffe en évacuant Q_1 via le régénérateur)*



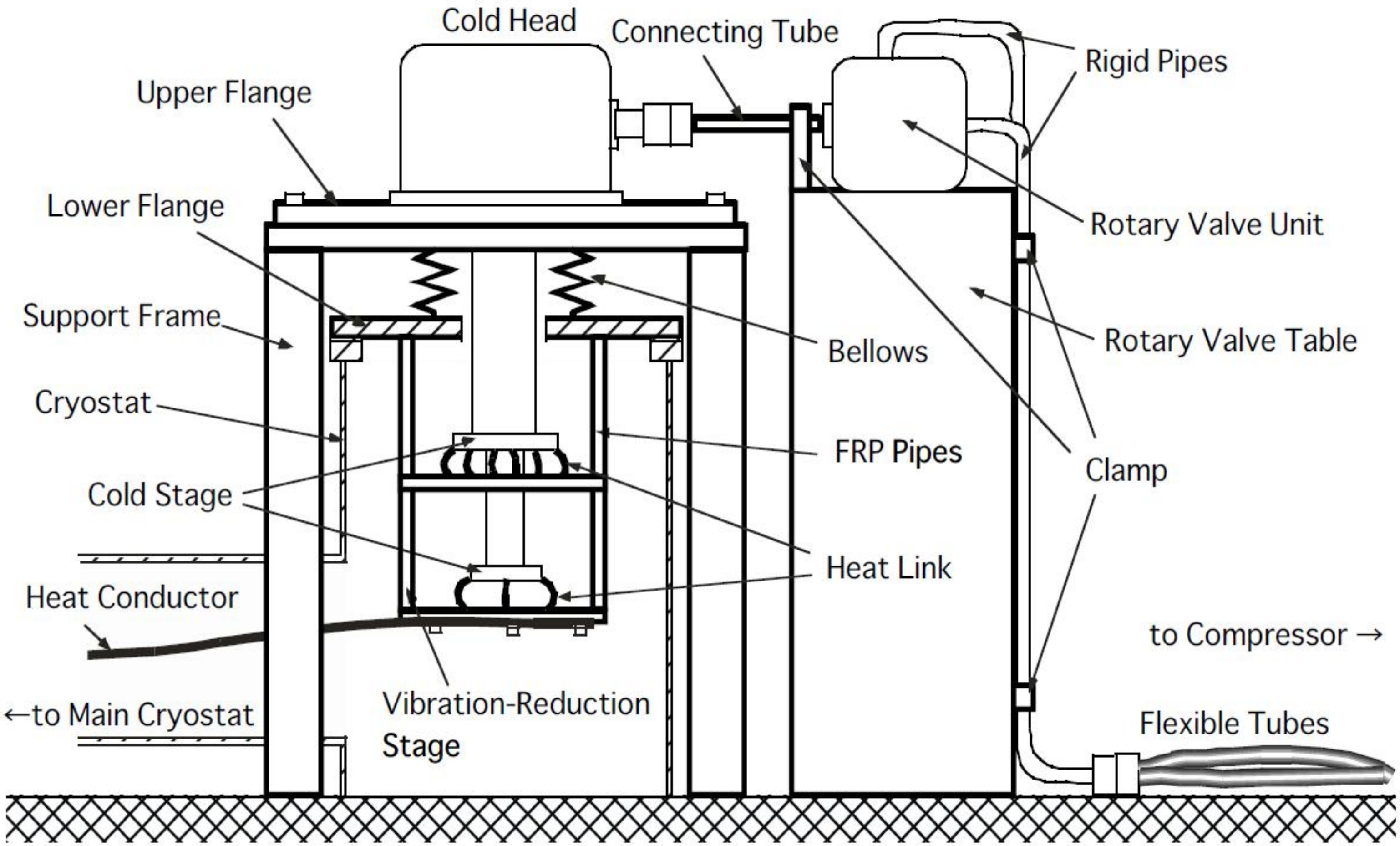
LE TUBE PULSE = MACHINE DE STIRLING SANS DEPLACEUR

REMPLACE PAR UN CIRCUIT RC (VOLUME TAMPON + VANNE V1, V2)

Déphasage du débit masse / onde de pression réalisé grâce au circuit RC

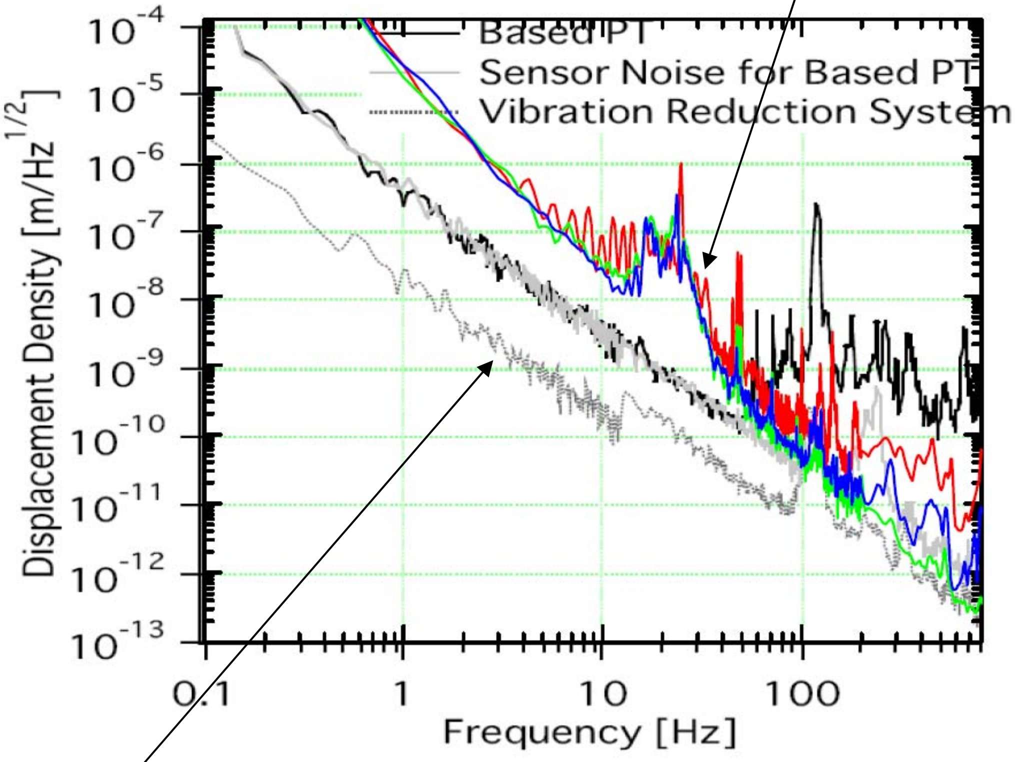


Vibrations : classical design



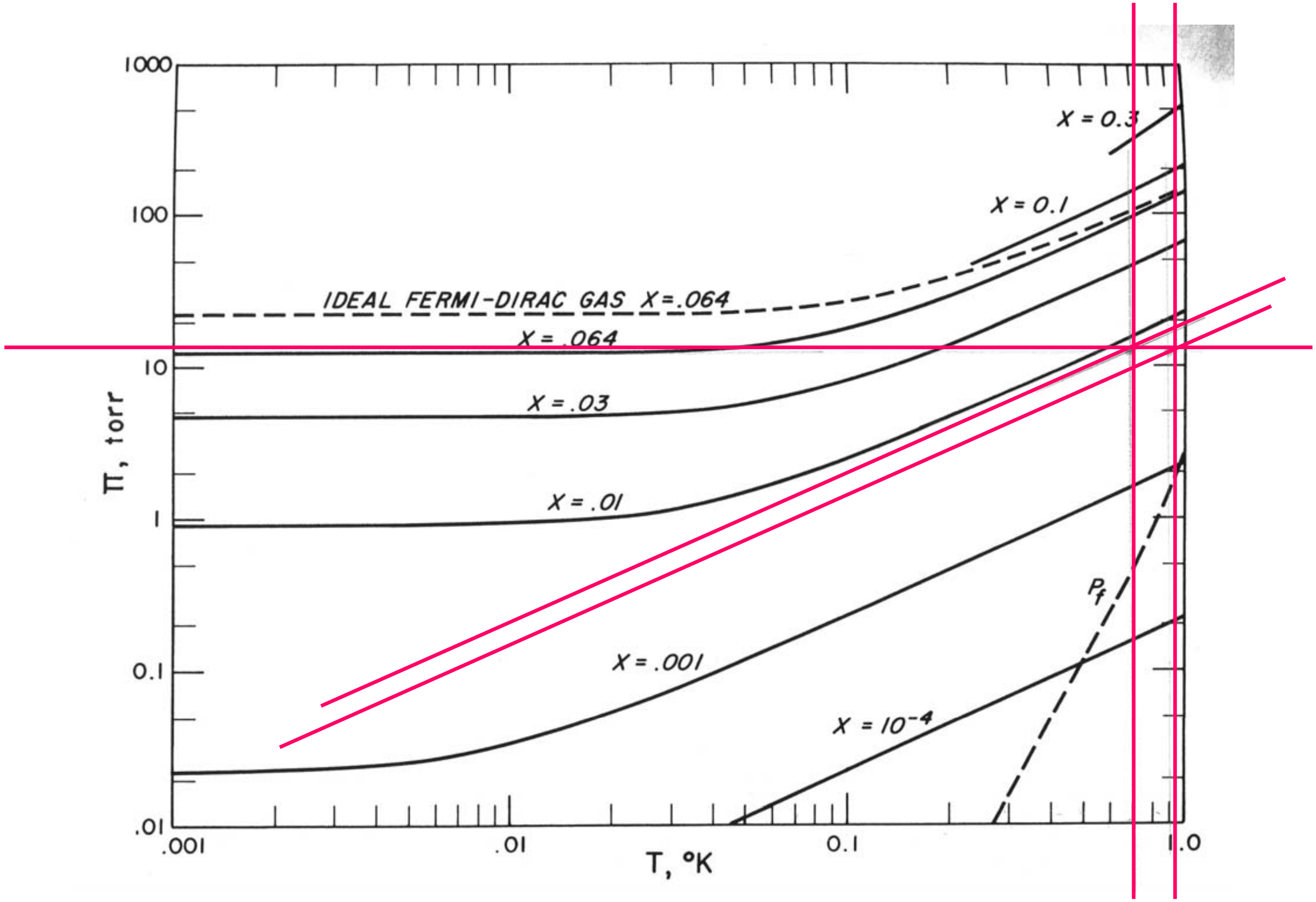
Schematic diagram of the vibration-reduction system for a 4K PT cryocooler.

OUR FRIDGE HAS NO ISOLATION BELLOW

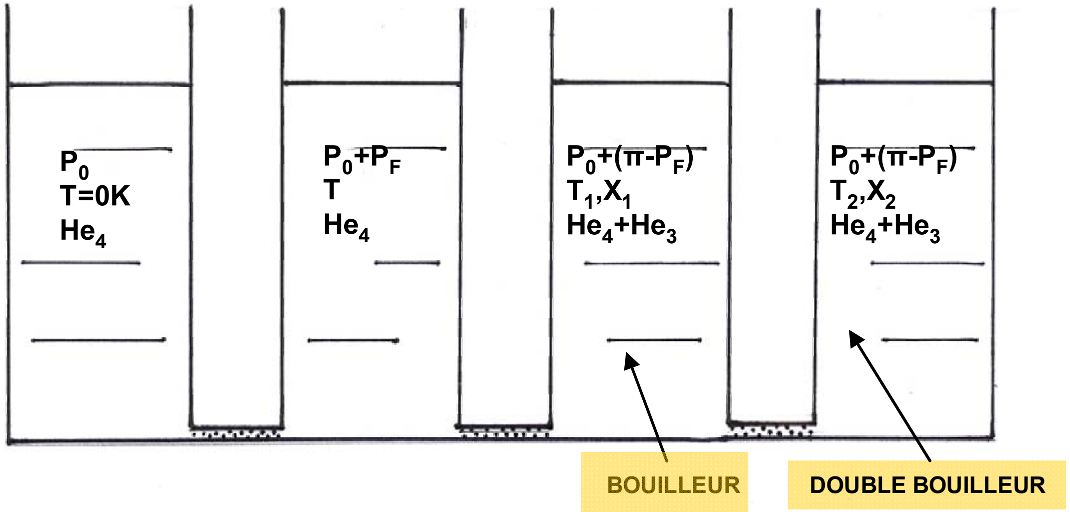


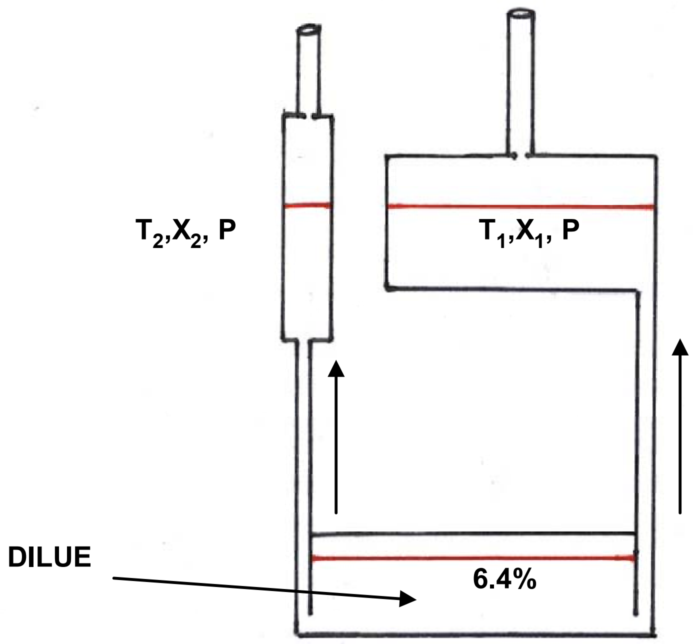
CLASSIC DESIGN

Pression osmotique Vs température de l'He3 dans l'He4 en fonction de différentes concentrations *X* en He3.



Bouilleur et double bouilleur à la même pression mais à deux Température différentes.





$$\mu_{40}(0,P_0) = \mu_{40}(T,P_0+P_F) = \mu_4(T_1,X_1, P_0+(\pi-P_F)) = \mu_4(T_2, X_2,P_0+(\pi-P_F))$$

X: Concentration en He_3 dans la colonne de dilué
π: Pression osmotique des atomes d'He3
P_F: Pression osmotique des atomes « normaux » de l'He_4 superfluide

Dans une colonne de dilué $\pi+P_F=C^{TE}$, en particulier dans Le bouilleur :

$\pi-P_F = X\,R\,T_S / V_{40}$ (Loi de Van'T Hoff)