

PRINCIPE des CRYOSTATS A DILUTION SANS LIQUIDES CRYO (DRY)

Patrick Pari, Philippe Forget, Matthieu De Combarieu CEA-SPEC

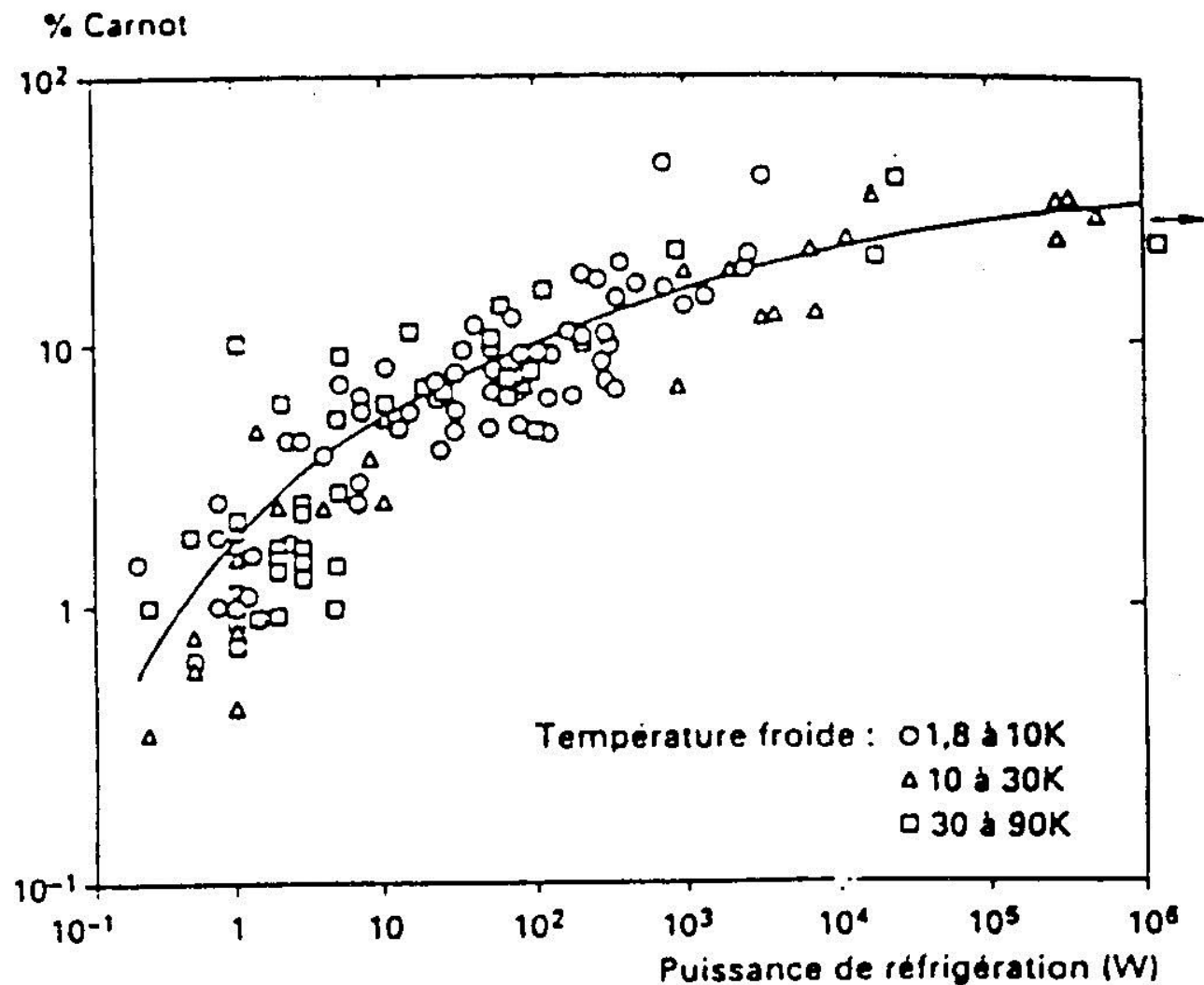
- Comparaison énergie/techno entre systèmes DRY et WET
- Caractéristiques comparées PT : [0.7W@4.2K](#) / [0.9W@4.2K](#)
- La dilution en deux mots. He3 : **4000€ /l (NTP)**. Sport couteux..
- Canne à dilution sans étage 1K, source d'inspiration.....
- La machine historique de 87 (Stirling). Quelle drôle d'idée.....
- Les premiers prototypes de laboratoires.
- Performances de notre première machine DRY (PT) (2008)
- Prototype de la Quantronique (D.Estève) autour d'un PT407
- La machine de Claire et Emiliano. Histoire de famille....
- Les machines commerciales
- Conclusion

COMPARAISON 4K WET/DRY

- **Cryogénie avec hélium liquide (WET) (vase DEWAR)**
- **Liquéfacteur type LPS (Air Liquide HELIAL)**
- 75KWh, 350l/J, Eau : **9 m³/J**

- **Cryogénie sèche (DRY)**
- **Tube pulsé type PT410 CRYOMECH**
- 7.5KWh, 10l/J, (18l/J), Eau : **7 m³ /J**

Rendement de Carnot VS puissance



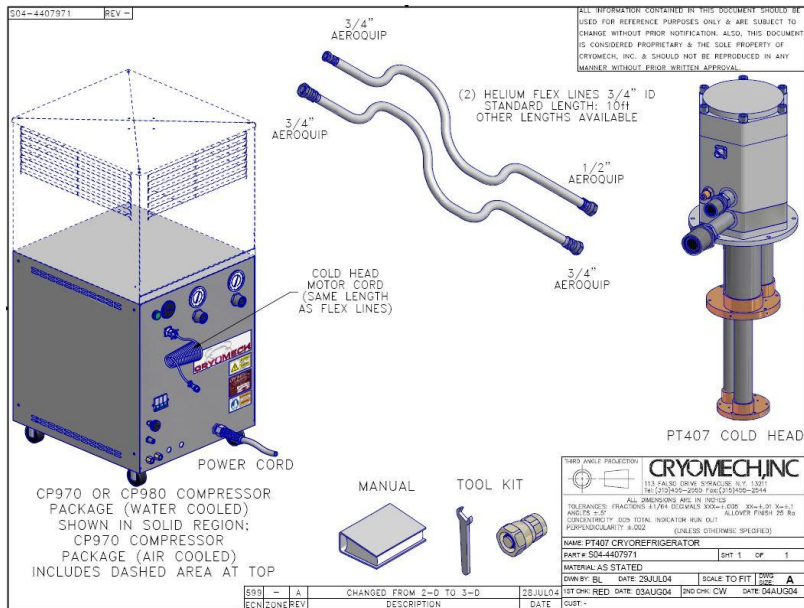
Coût moyen journalier par manip DRY Nécessité d'utiliser une boucle froide

Prix de l'électricité : 0.06 €/KWh

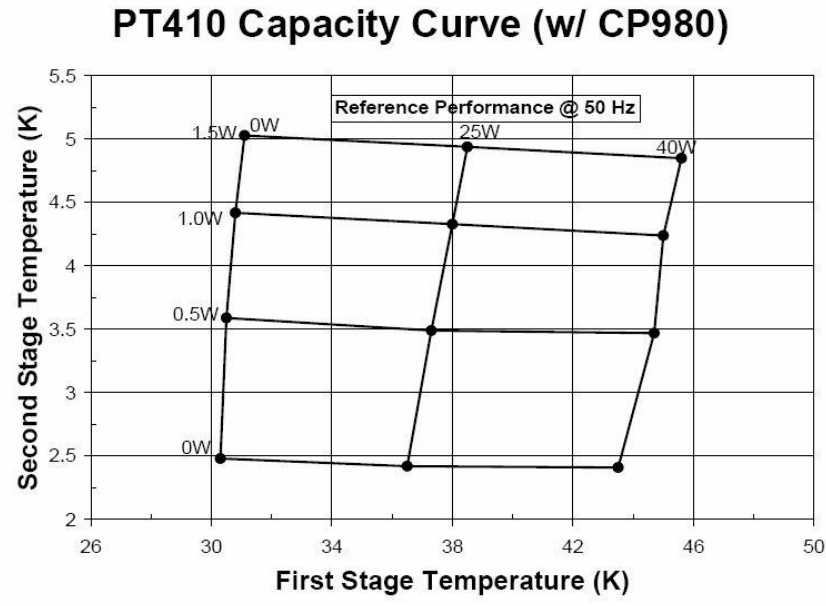
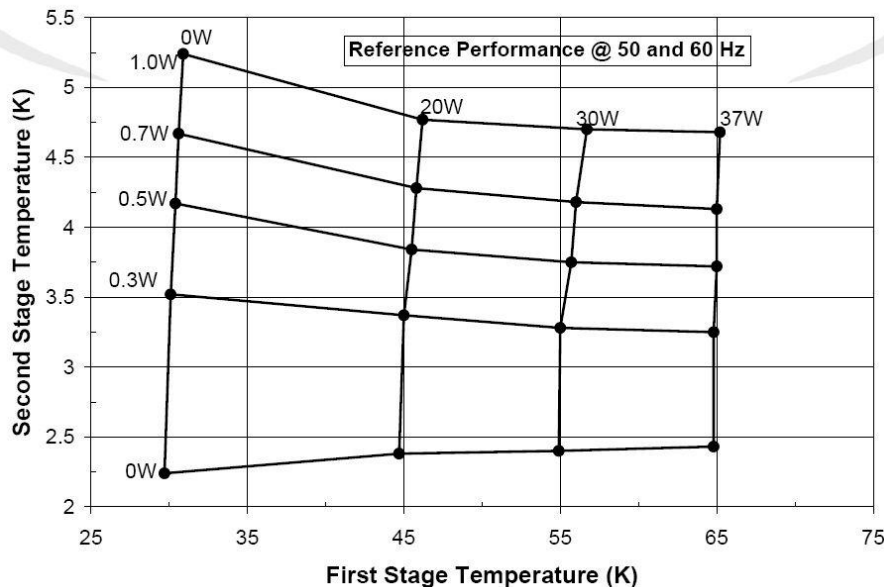
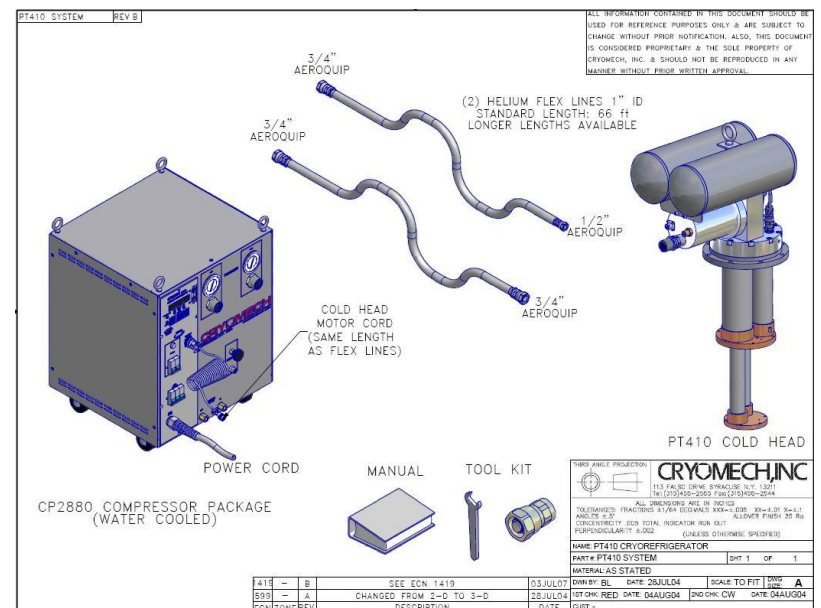
- Prix de l'eau de ville : **3** €/m³ (7 m³ /J)
- Coût journalier en eau perdue:

$$11 \text{ € (électrique)} + \underline{21} \text{ € (eau)} = 32 \text{ €/j}$$

CRYOMECH PT 407, 700mW@4.2K



CRYOMECH PT 410, 1000mW@4.2K

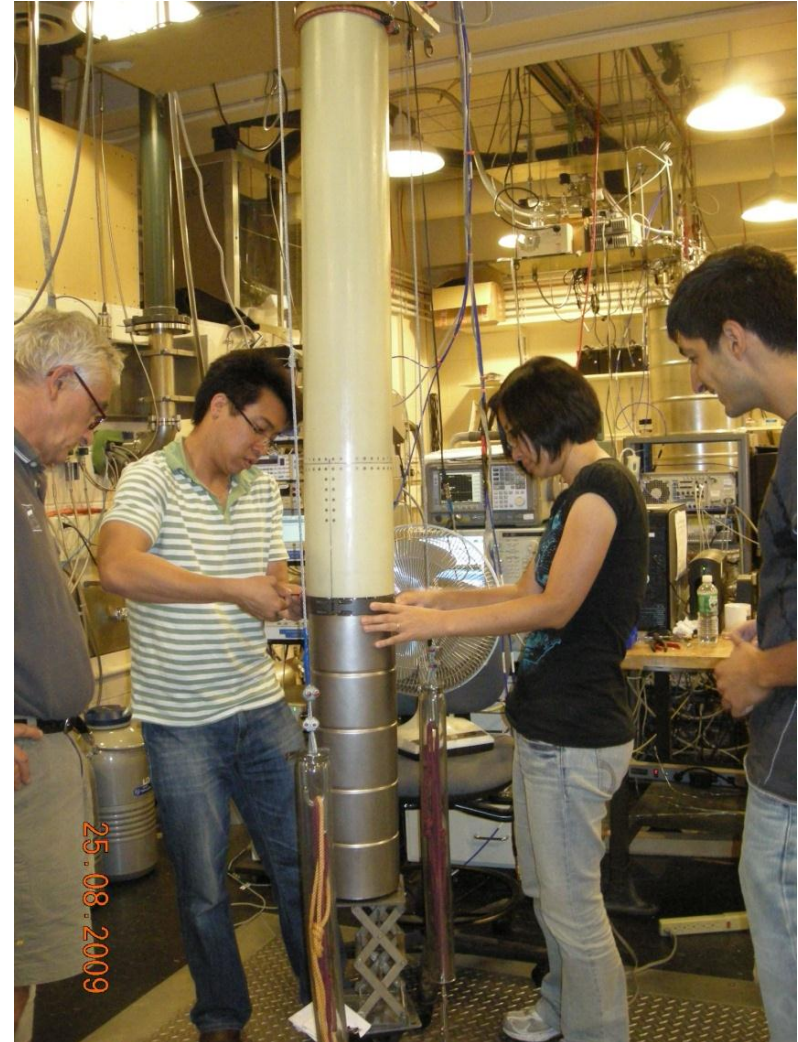


Avantages

- moins d'eau (pompes turbo 2m³/J)
- moins d'électricité (banc de pompage 4KWh)
- moins de vibration et de bruit
- aucune pièce mécanique (pas de maintenance)

Inconvénients 40€/j (base 10l/j à 4€/l)

- Espace expérimental plus réduit (Ø : 150mm H:150mm)
- Mise en œuvre délicate (IVC)
- Fonctionnement Manuel à semi automatique. Donc + de présence.
- Mauvaise utilisation → conséquences importantes sur la consommation He .
- Ne permet pas d'accueillir plusieurs expériences.
- Puissance disponible faible sur l'étage 4K:
-350 mW →12l/j d'hélium



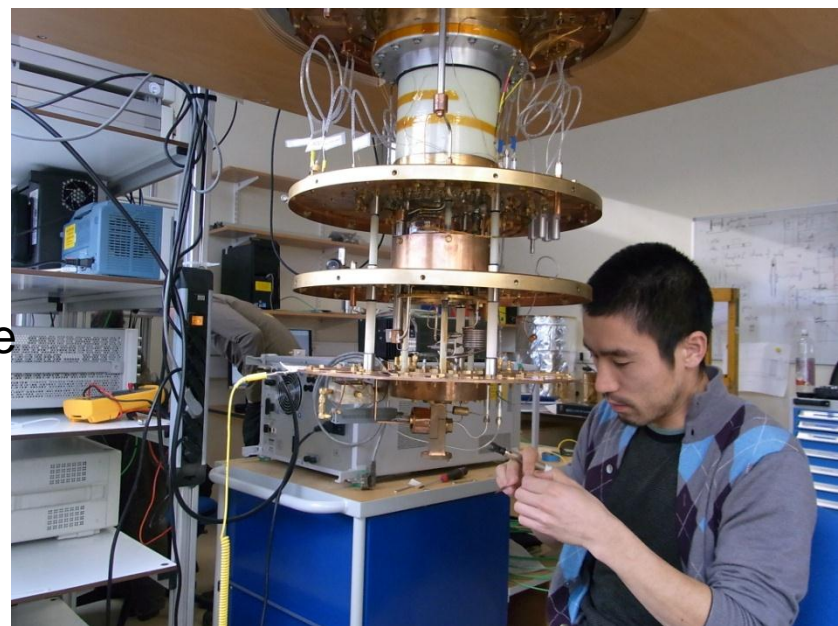
Utilisation avec boucle eau froide

Avantages 11€/j

- Espace très important (\varnothing : 300mm H:300mm)
- Mise en œuvre très simple (pas d'IVC)
- Fonctionnement automatique, contrôle à distance
- Mauvaise utilisation (vide) sans conséquence sur la consommation électrique (7.5KWh).
- Permet d'accueillir plusieurs types d'expériences.
- Puissance disponible importante sur l'étage 4K:
 - 200mW pour un PT407 (0.7W@4K)
 - 350mW pour un PT410 (1W@4K)
- Pas de calorimètre à 4.2K, uniquement des écrans.

Inconvénients

- Vibrations (pb pour les expériences à basse fréquence)
- le bruit
- Maintenance mécanique (compresseur +tête froide)
- Machine thermique donc pas de de température de référence (T=4.2K hélium liquide)

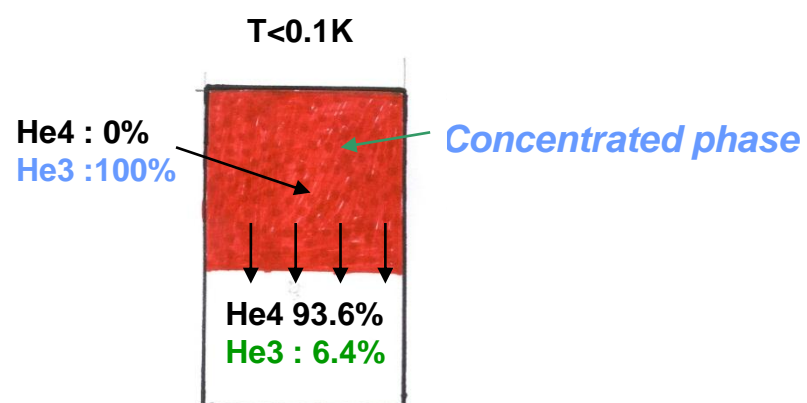
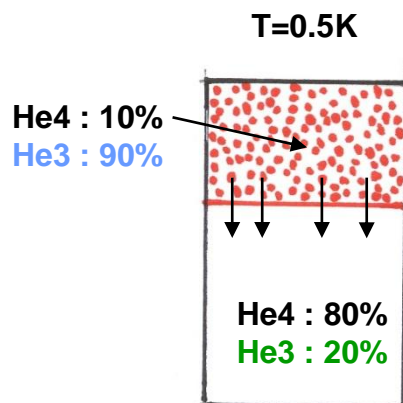
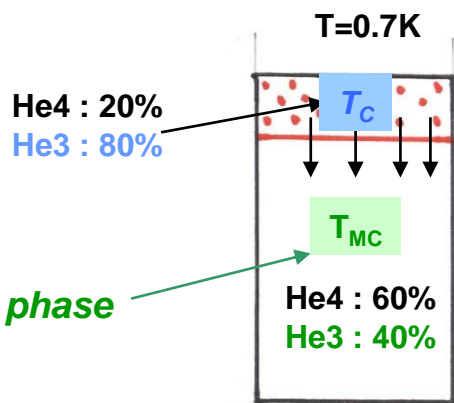
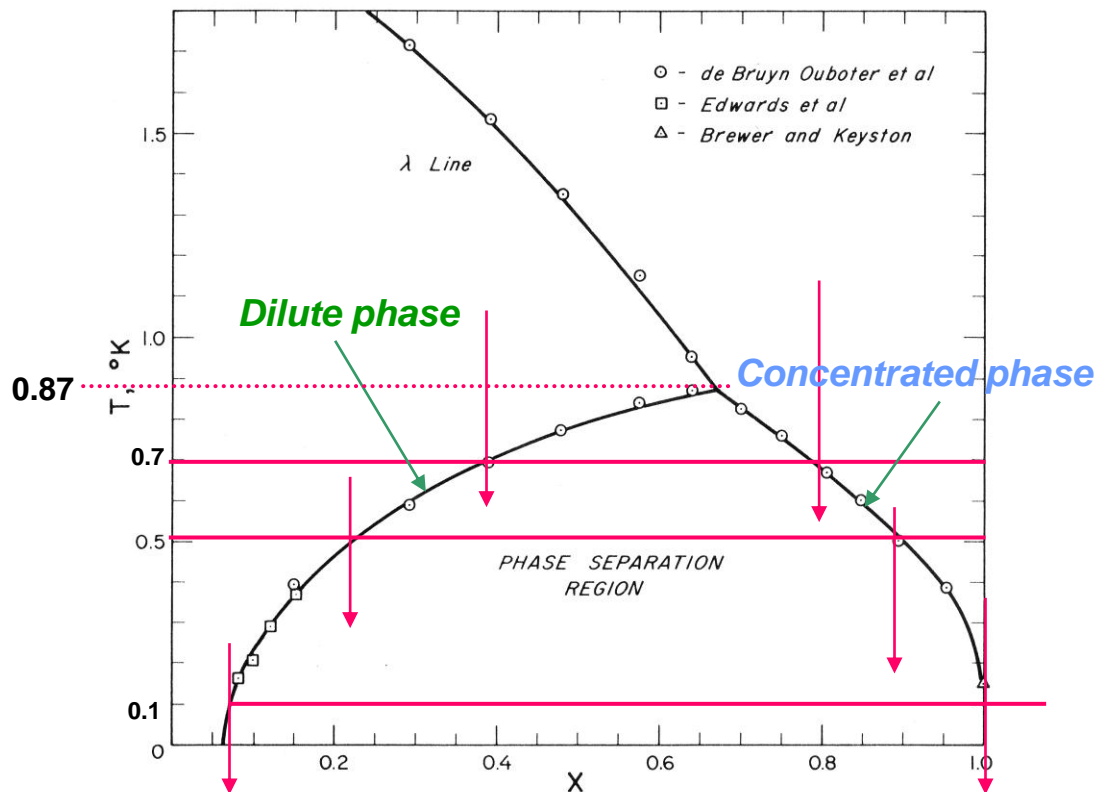


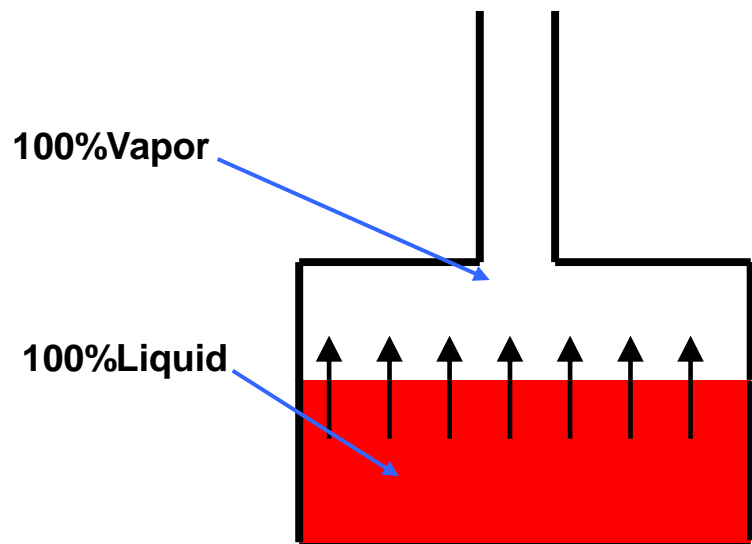
Compresseur 7.5KWh + 7m³/j d'eau



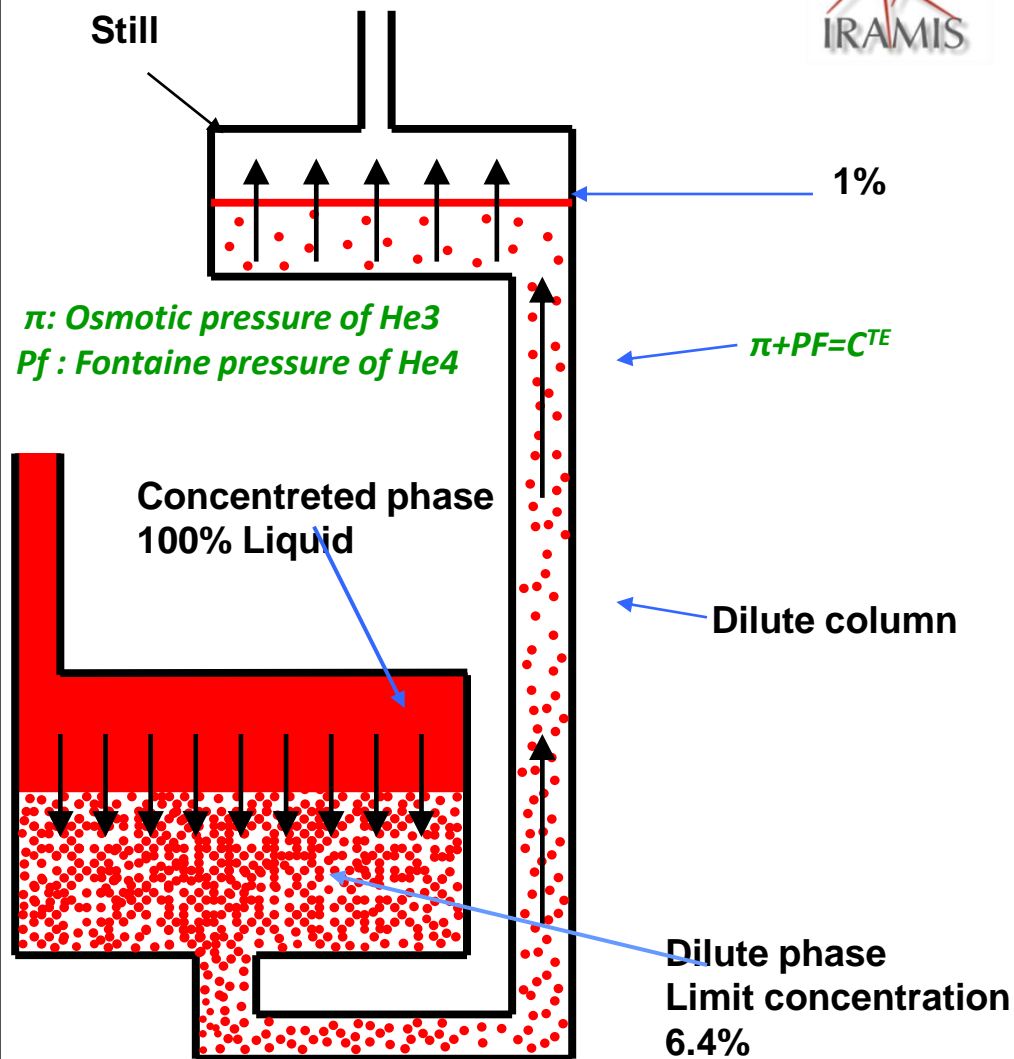
Cryostat à dilution







He3 : plus de 4000€/l (NTP)



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

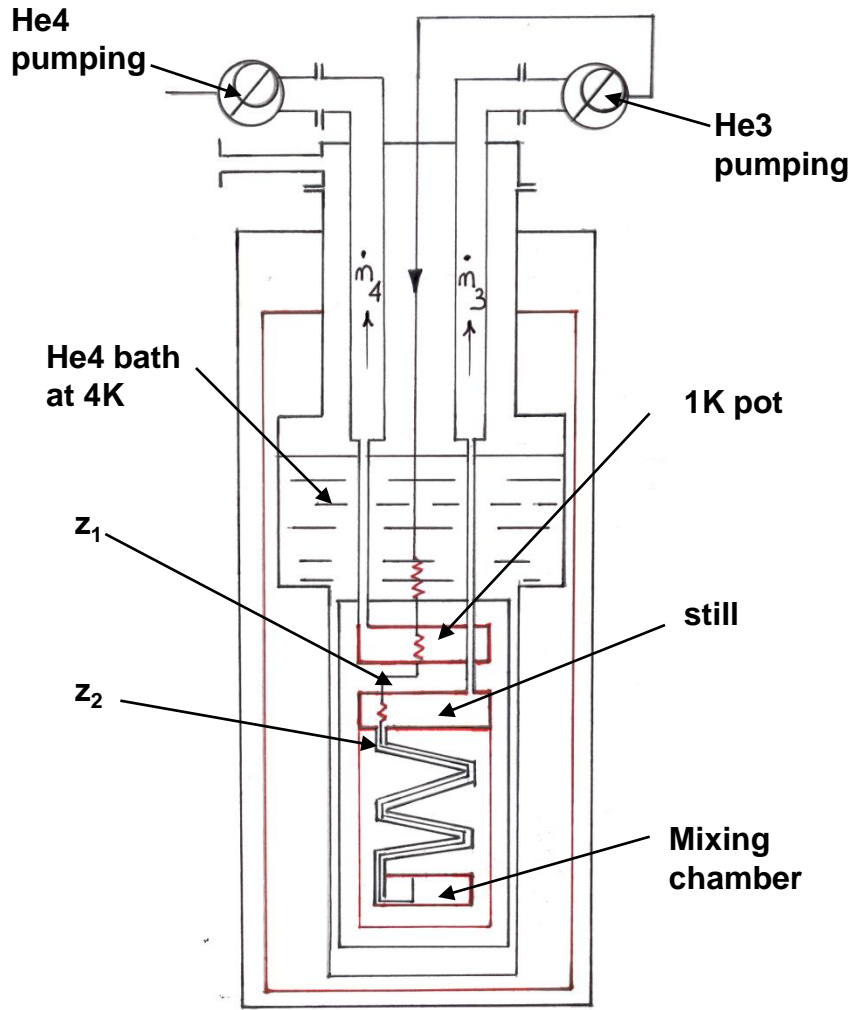
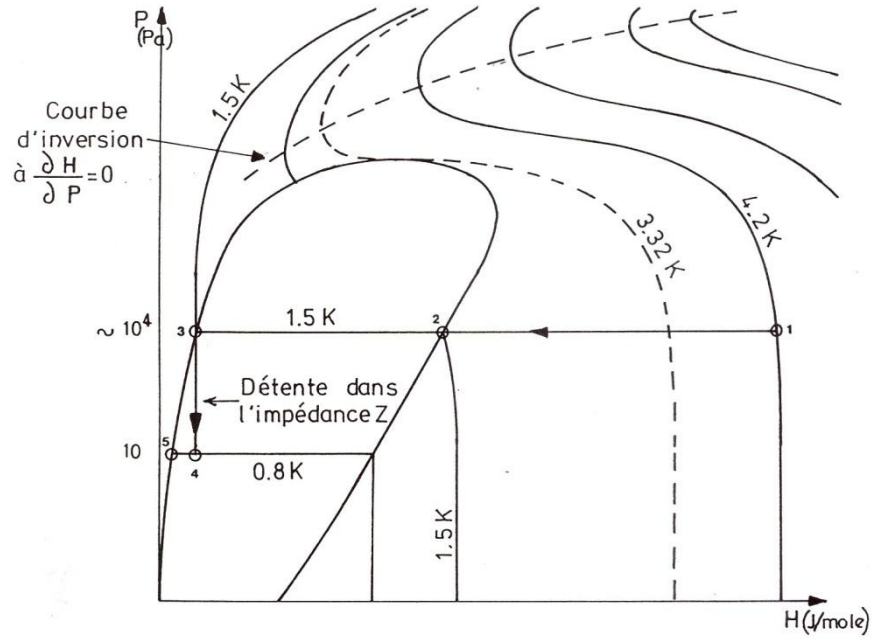
$$Q_{EXT} = 84 n_3 T_{mc}^2 \text{ J s}^{-1} \text{ perfect exchangers}$$

CLASSICAL DILUTION REFRIGERATOR CYCLE AND DIAGRAM

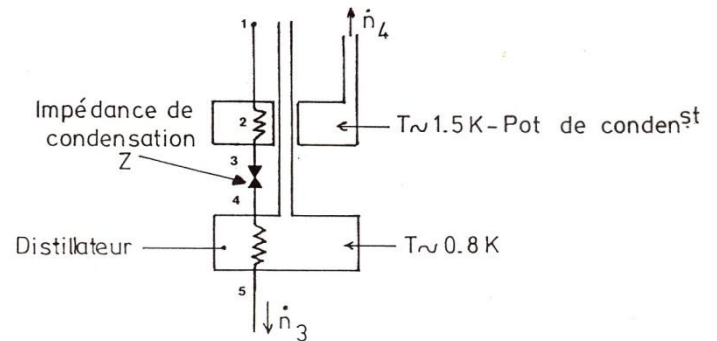


0.2 l/h < liquid helium flow < 1 l/h

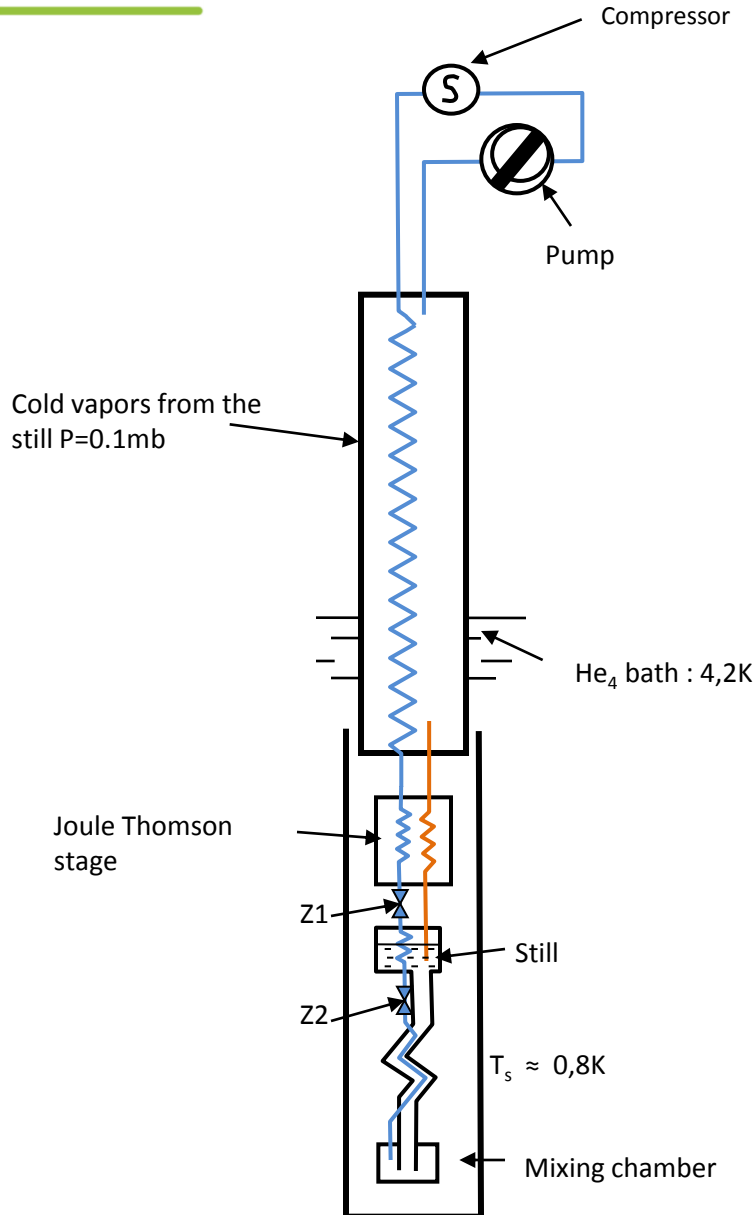
Diagramme enthalpique de l'He3 pur [21]



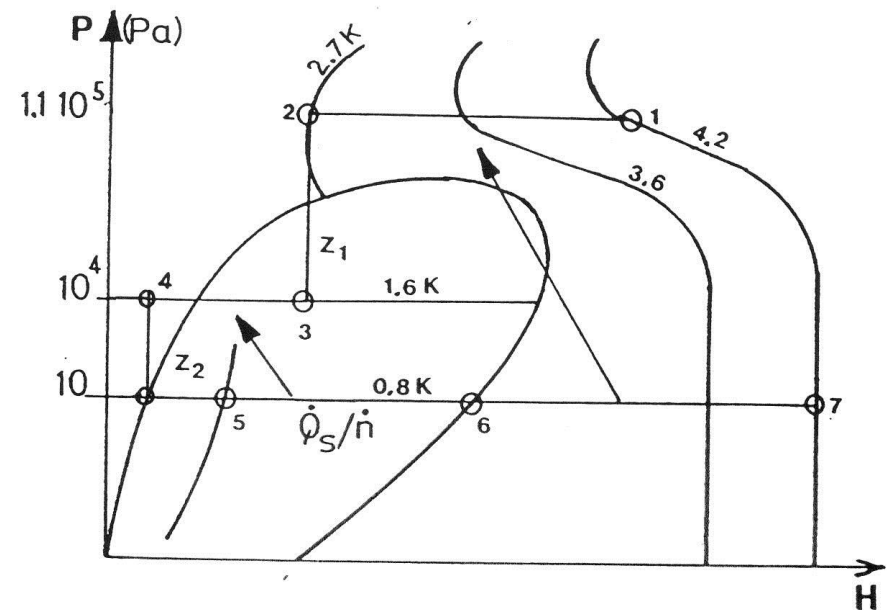
Parcours de liquéfaction dans un appareil à fonctionnement classique



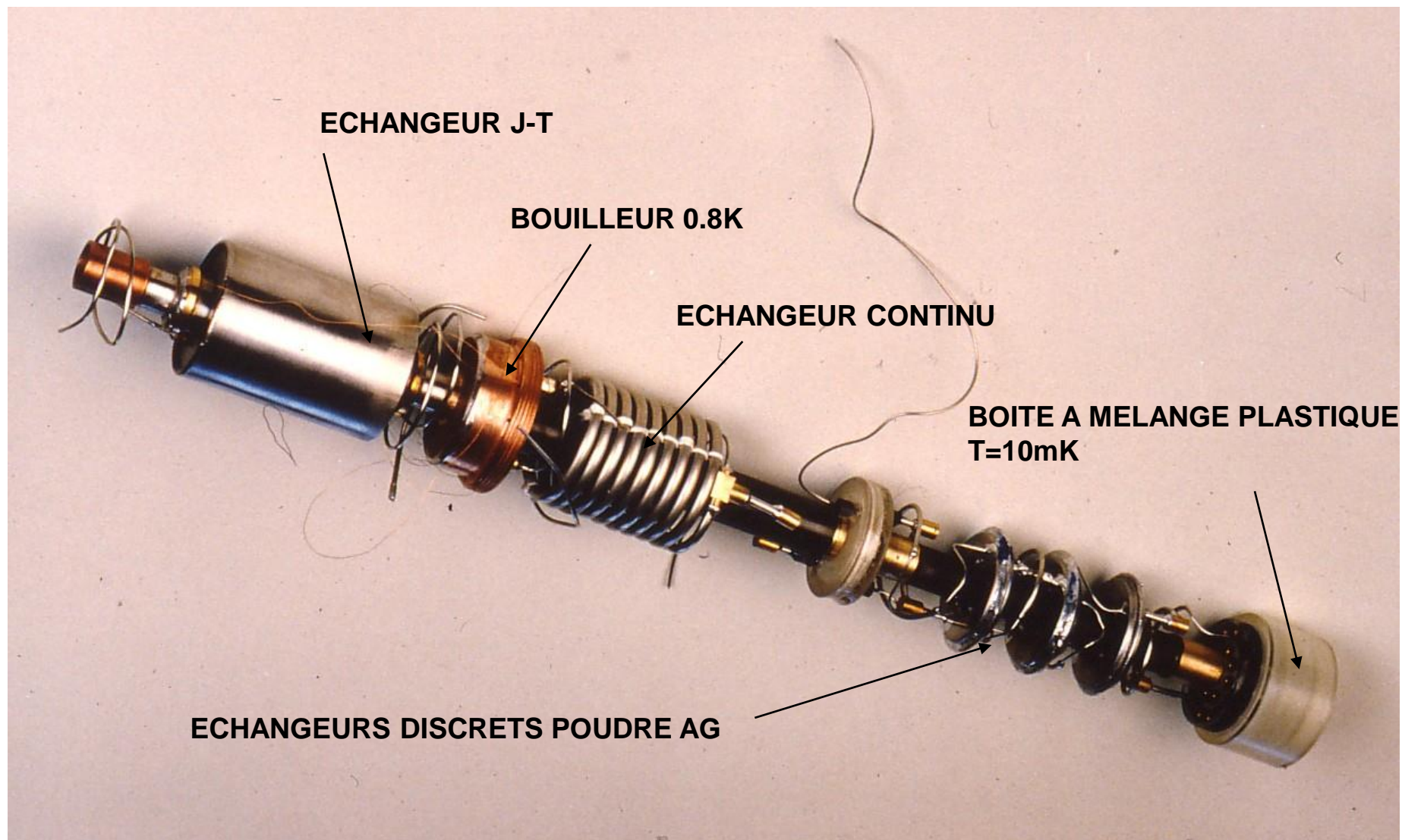
CLASSICAL DILUTION REFRIGERATEUR CYCLE



Condensation from 4.2K
Précooling mixture in the counter-flow exchanger



Moins de 5l d'He3



FREE DILUTION REFRIGERATOR OF 1987

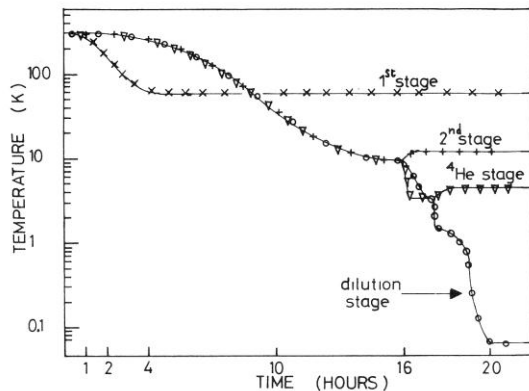
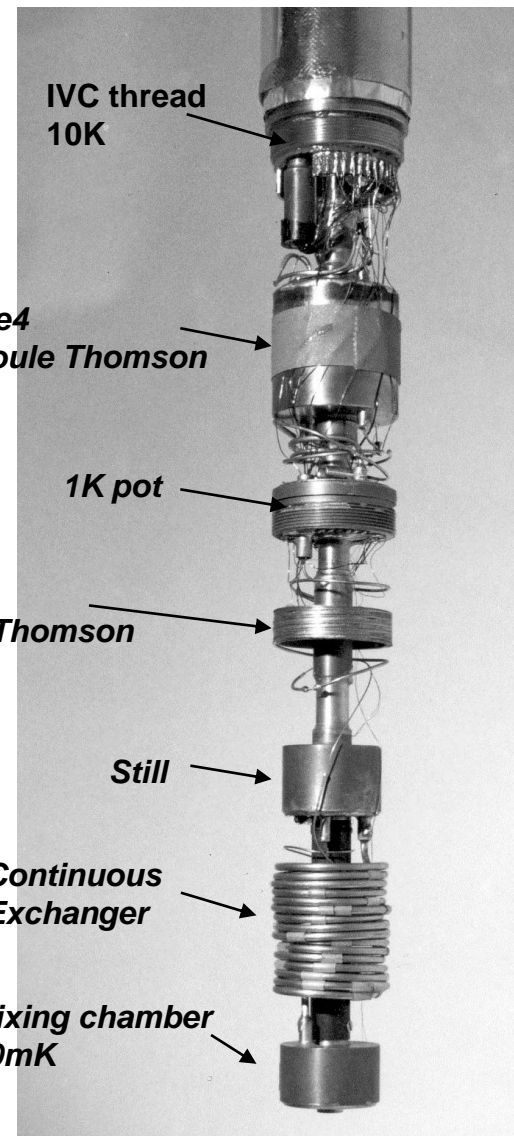
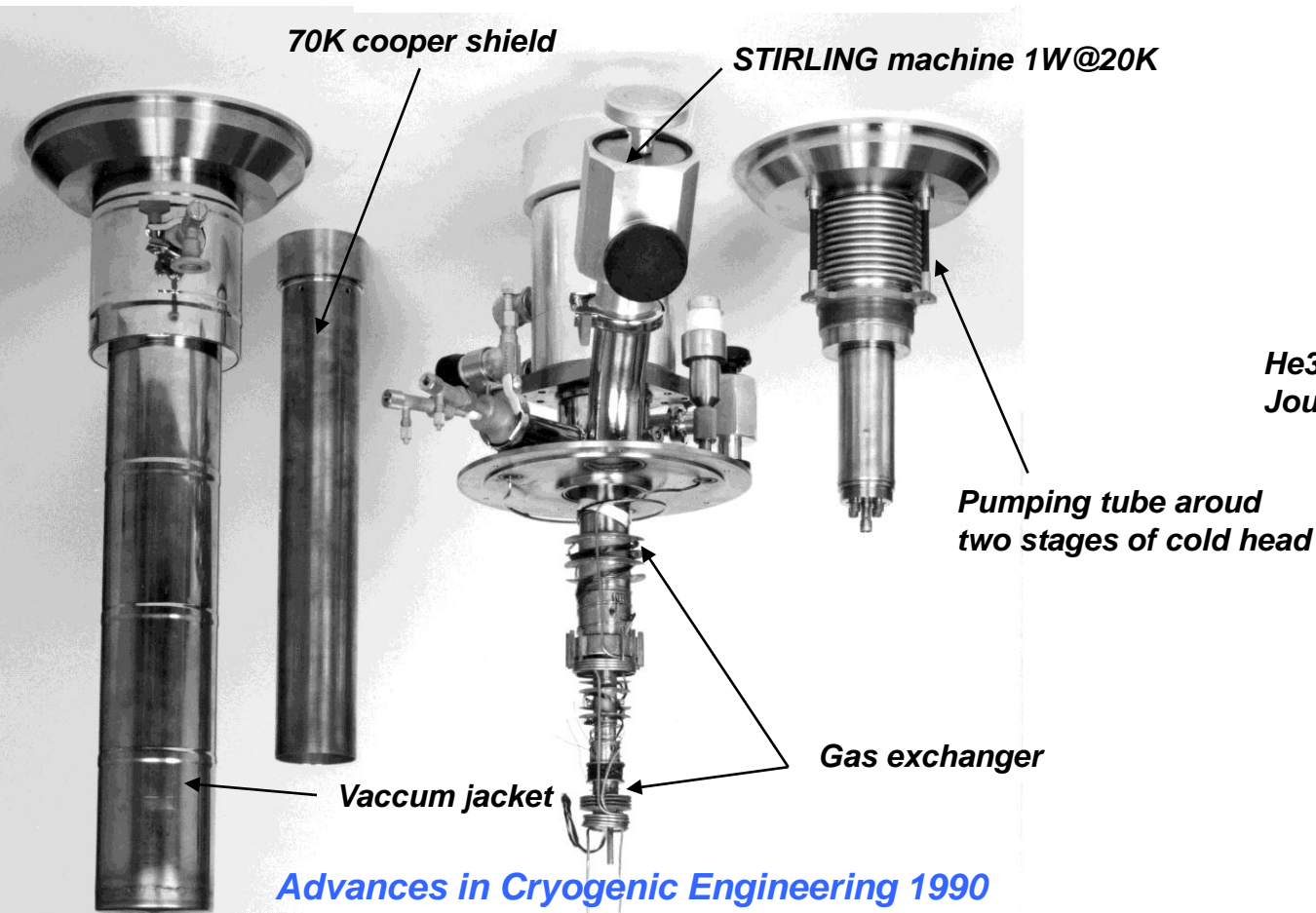
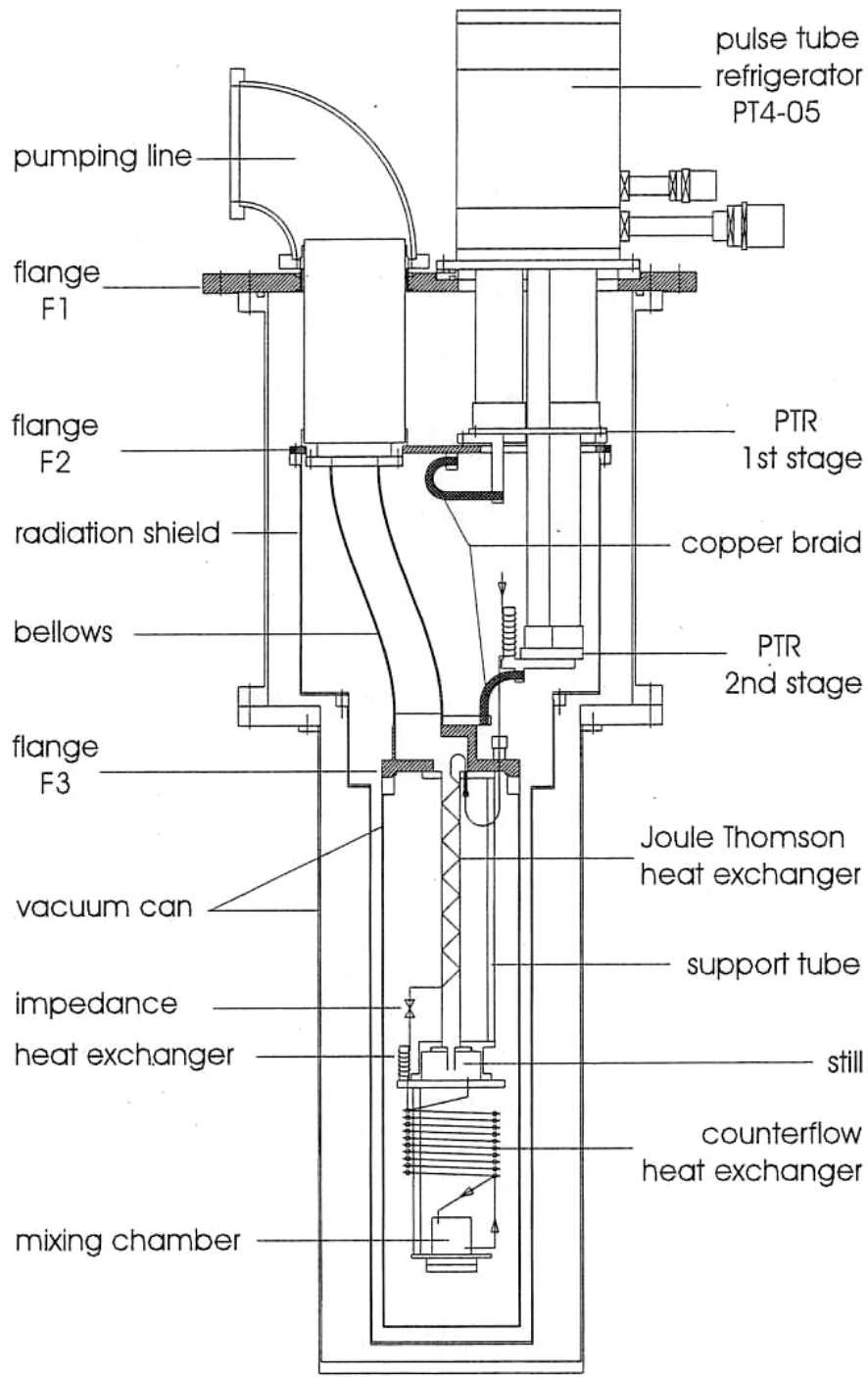


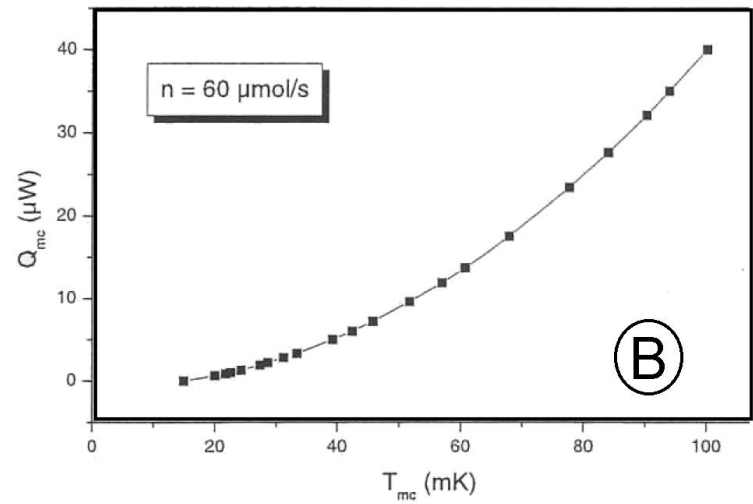
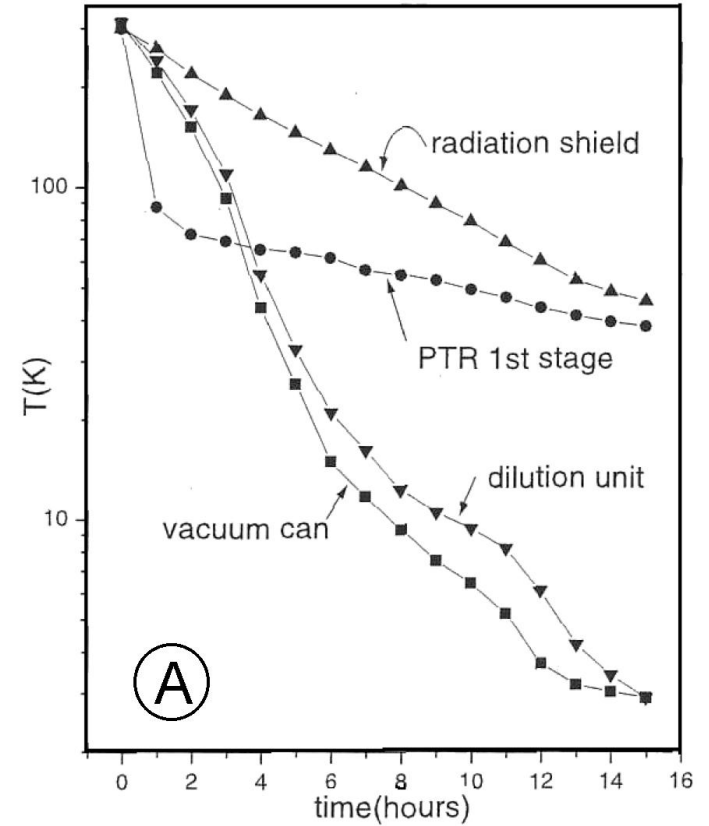
Fig. 6. Cooldown diagram.

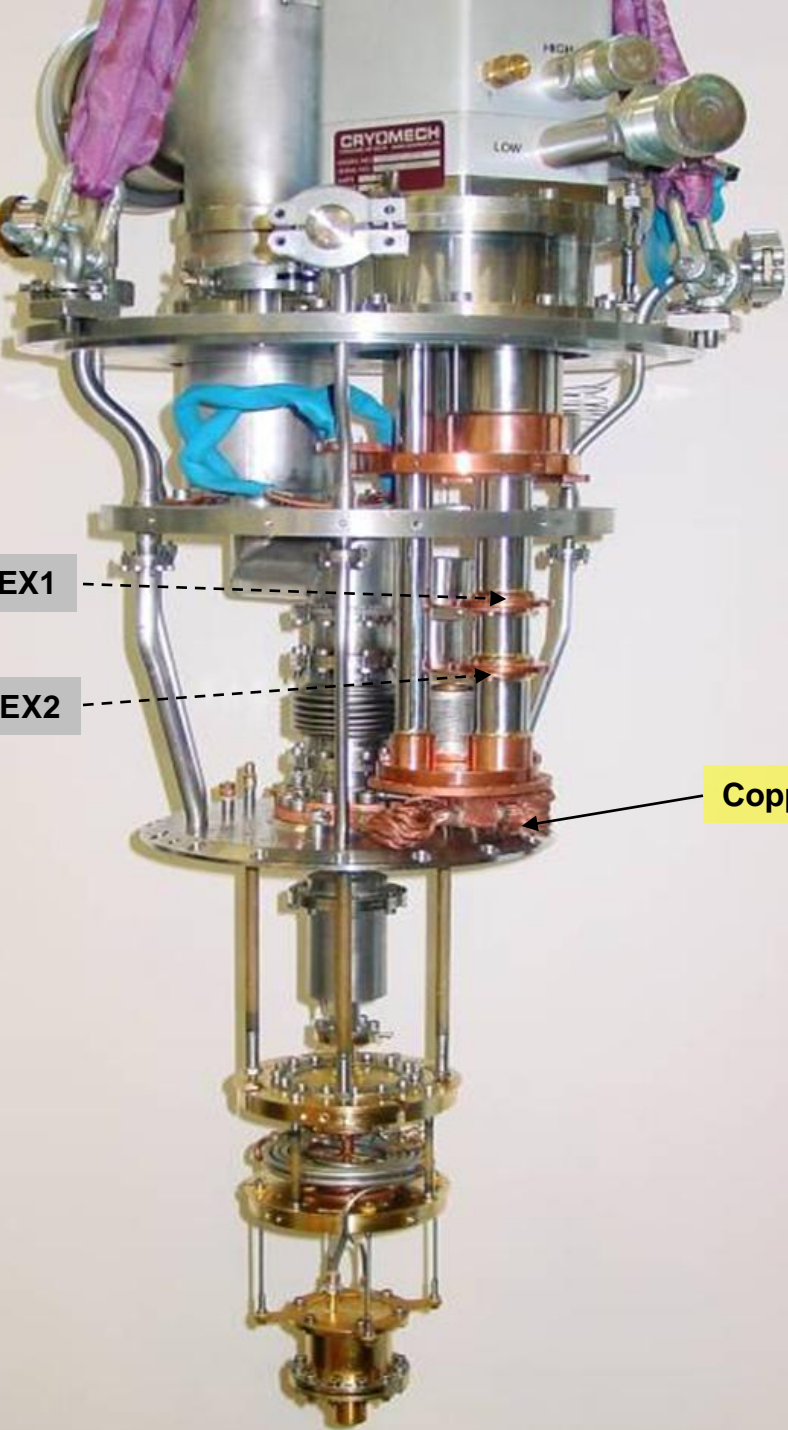


*Advances in Cryogenic Engineering 1990
P.Pari « dilution refrigerator with no liquid helium supply »*



Bibliographie :
many papers by Kurt Uhlig





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

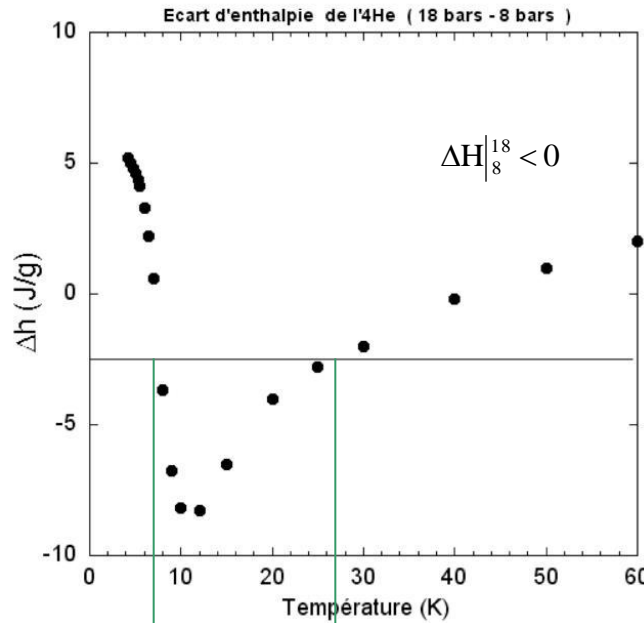
**DEVELOPPEMENT OF DILUTION REFRIGERATOR
PRECOOLING BY A PULSED TUBE**

Thesis of Thomas Prouvé

(Joseph Fourier University, GRENOBLE) 2007

BETWEEN 6K AND 26K $\Delta H_8^{18} < 0$ ITS POSSIBLE TO CHARGE THE REGENERATOR

WITHOUT REDUCING THE COOLING POWER ON THE SECOND STAGE OF PT



$$\Delta H_8^{18} < 0$$

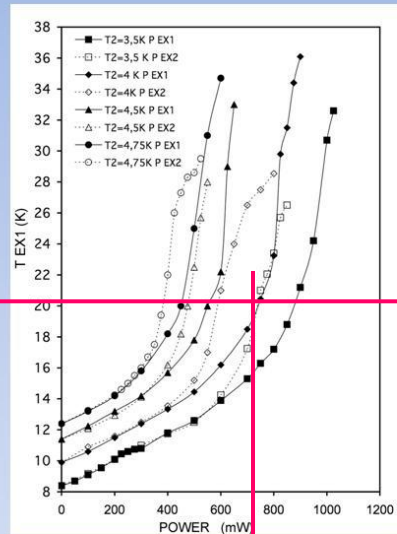
$$\Delta H_8^{18} < 0$$

$$\Delta H_8^{18} < 0$$

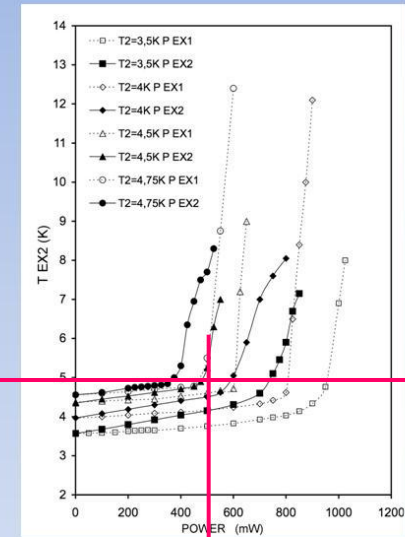
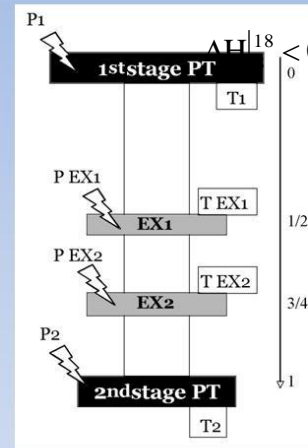
6K

26K

“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 P EX2 (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 P EX2 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).

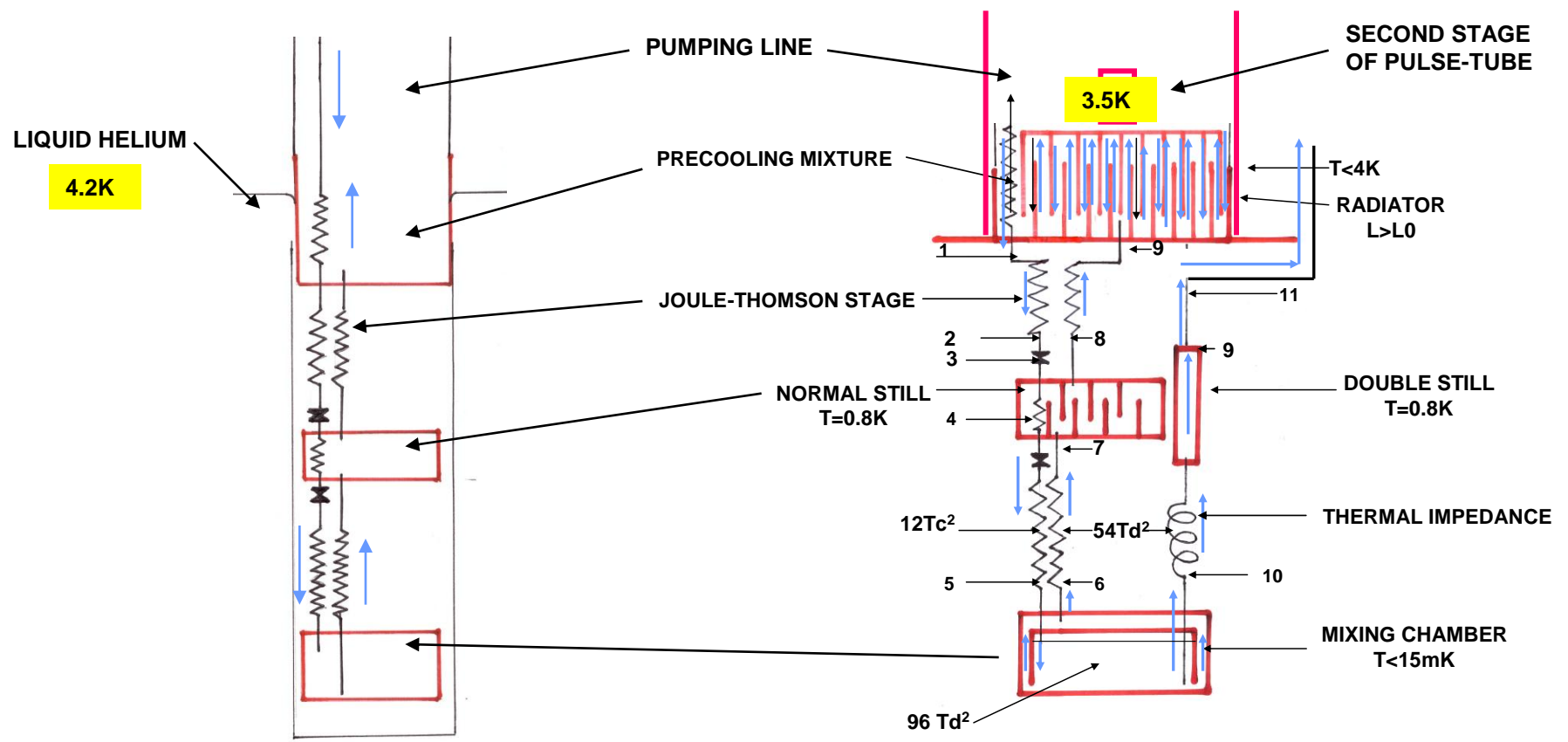
Air Liquide, U.S. Patent 6,915,642 and CNRS-Air Liquide Patent FR07 53945

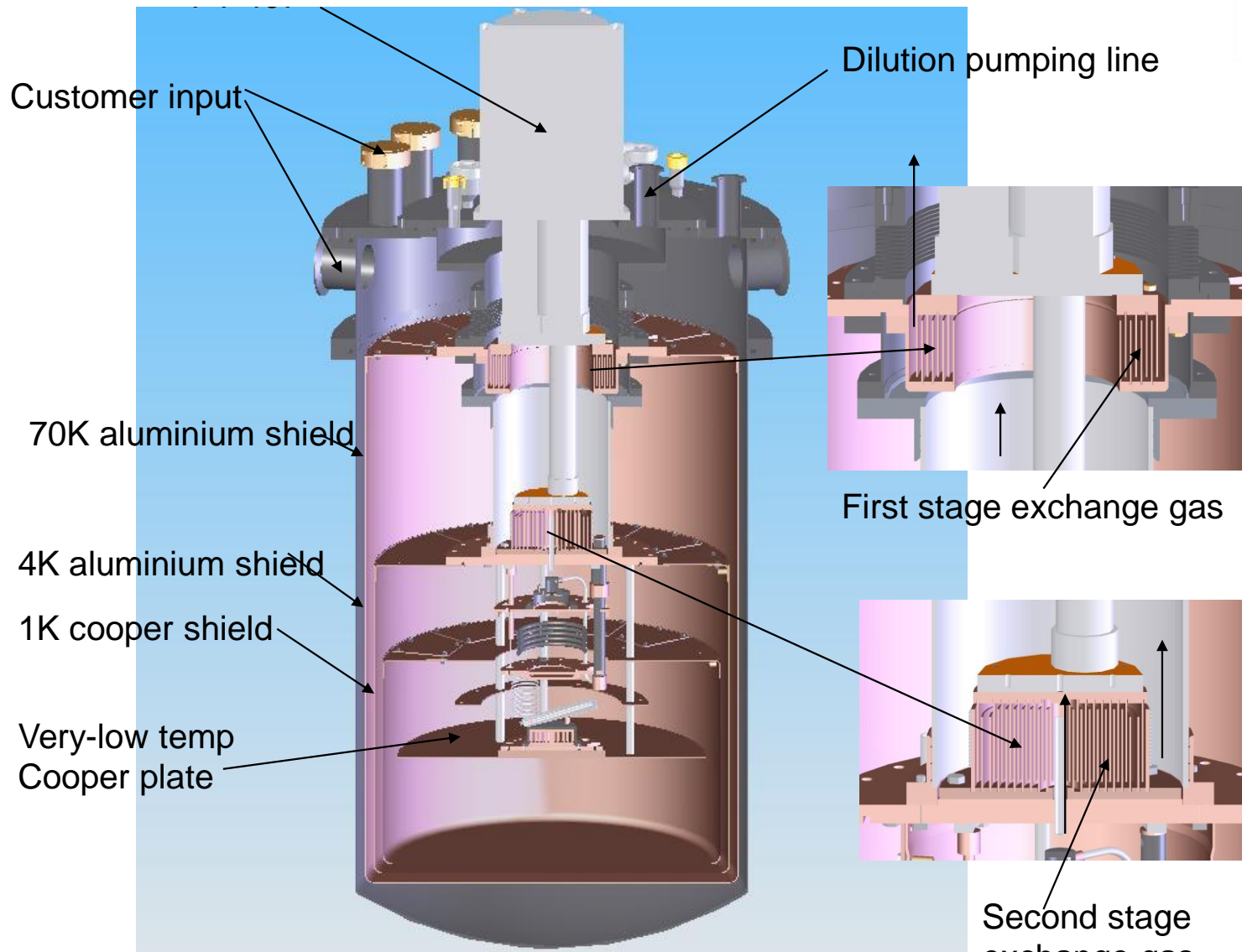
EX1 700mW@20K

EX2 500mW@6K

NORMAL JT DILUTION STAGE

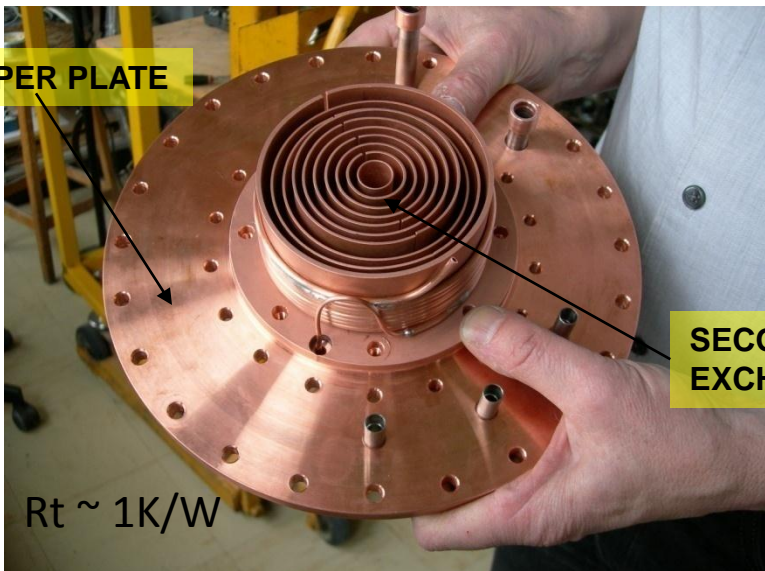
DOUBLE STILL JT DILUTION STAGE





**DRY DILUTION REFRIGERATOR
WITHOUT COLD SOLIDS CONTACTS AND NOT IVC**

4K COPPER PLATE

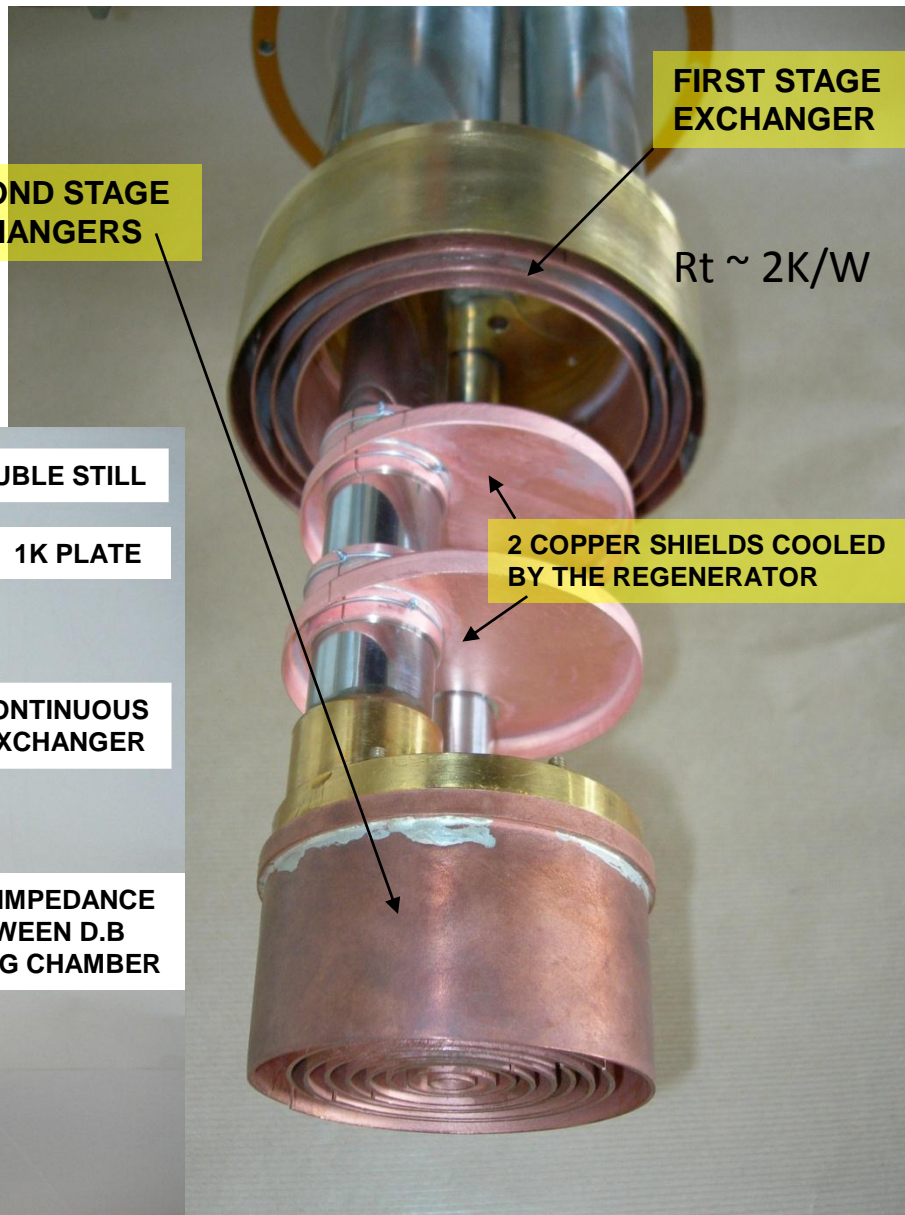


SECOND STAGE EXCHANGERS

$Rt \sim 1K/W$

EXCHANGERS BETWEEN FIRST STAGE AND SECOND STAGE OF PT.

FIRST STAGE EXCHANGER



$Rt \sim 2K/W$

2 COPPER SHIELDS COOLED BY THE REGENERATOR

JOULE THOMSON STAGE

DOUBLE STILL

1K PLATE

NORMAL STILL

CONTINUOUS EXCHANGER

0.1K PLATE

THERMAL IMPEDANCE TUBE BETWEEN D.B AND MIXING CHAMBER

TWO SILVER POWDER EXCHANGER

MIXING CHAMBER

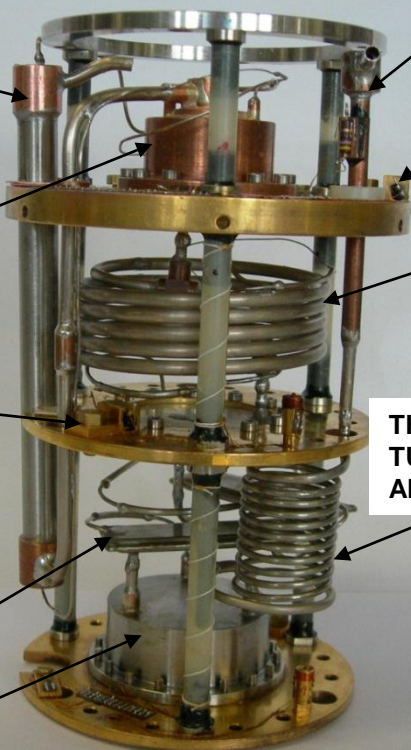


DIAGRAMM OF RAPID COOLING $3 < T(K) < 300$

Start

Click to echap
to quit

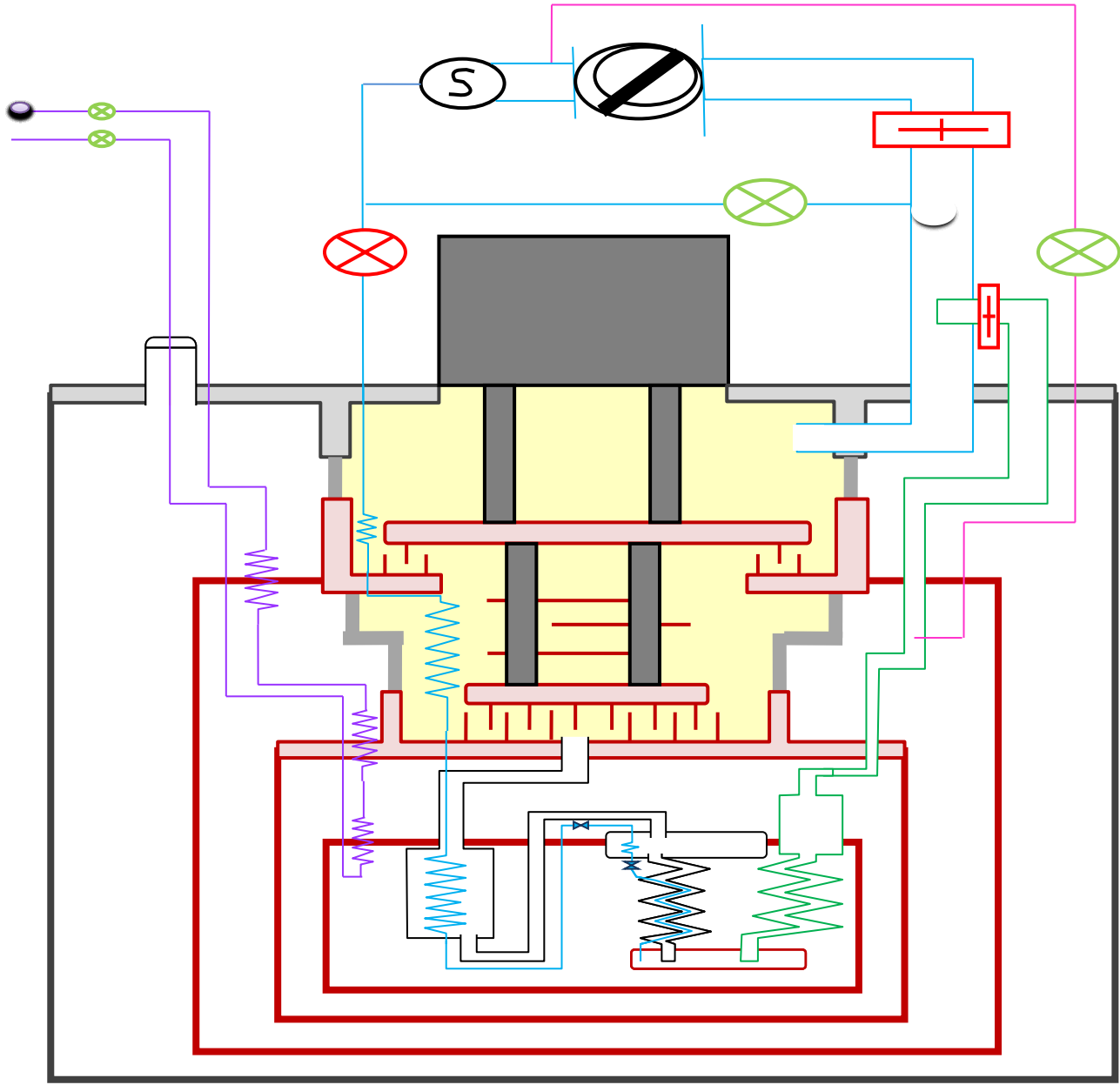
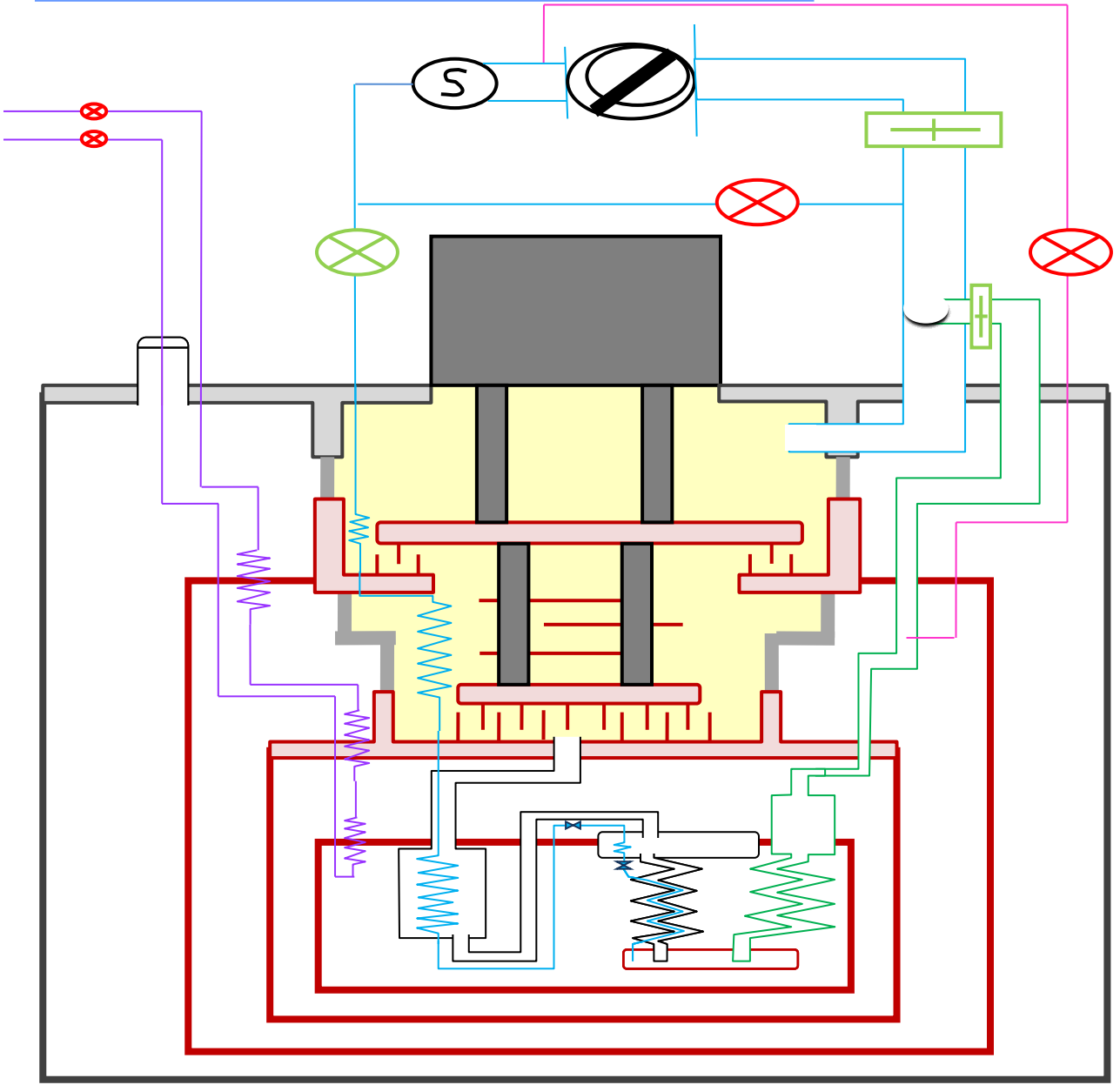




DIAGRAMM OF NORMAL COOLING $16\text{mK} < T < 3\text{K}$

Start

Click to echap
to quit





GENERAL VIEW OF FREE DILUTION REFRIGERATOR PROTOTYPE MODEL WITH PT407



1K COPPER SHIELD

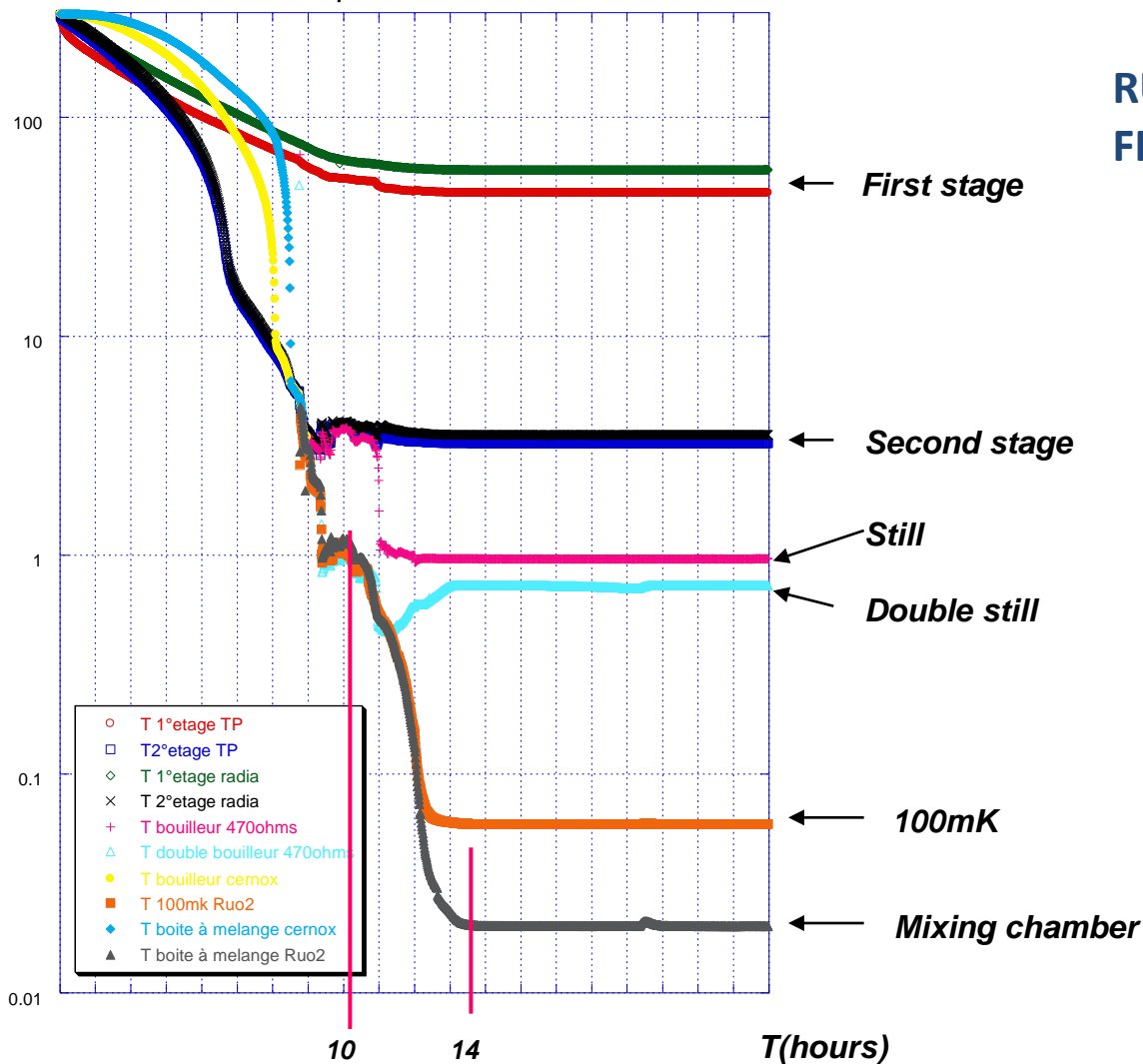
4K COPPER SHIELD
ID : 200mm
Length : 300mm

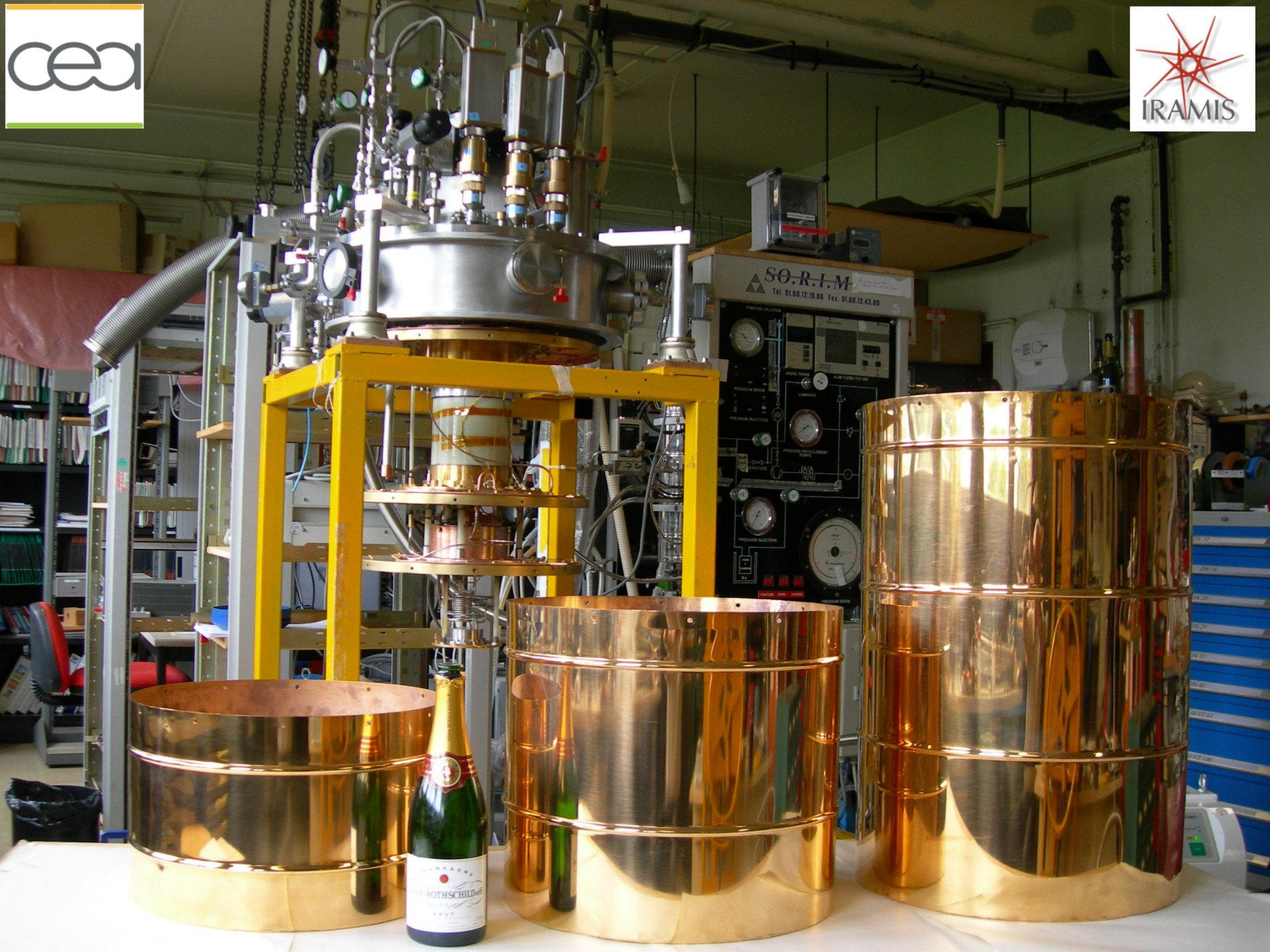
60K COPPER SHIELD

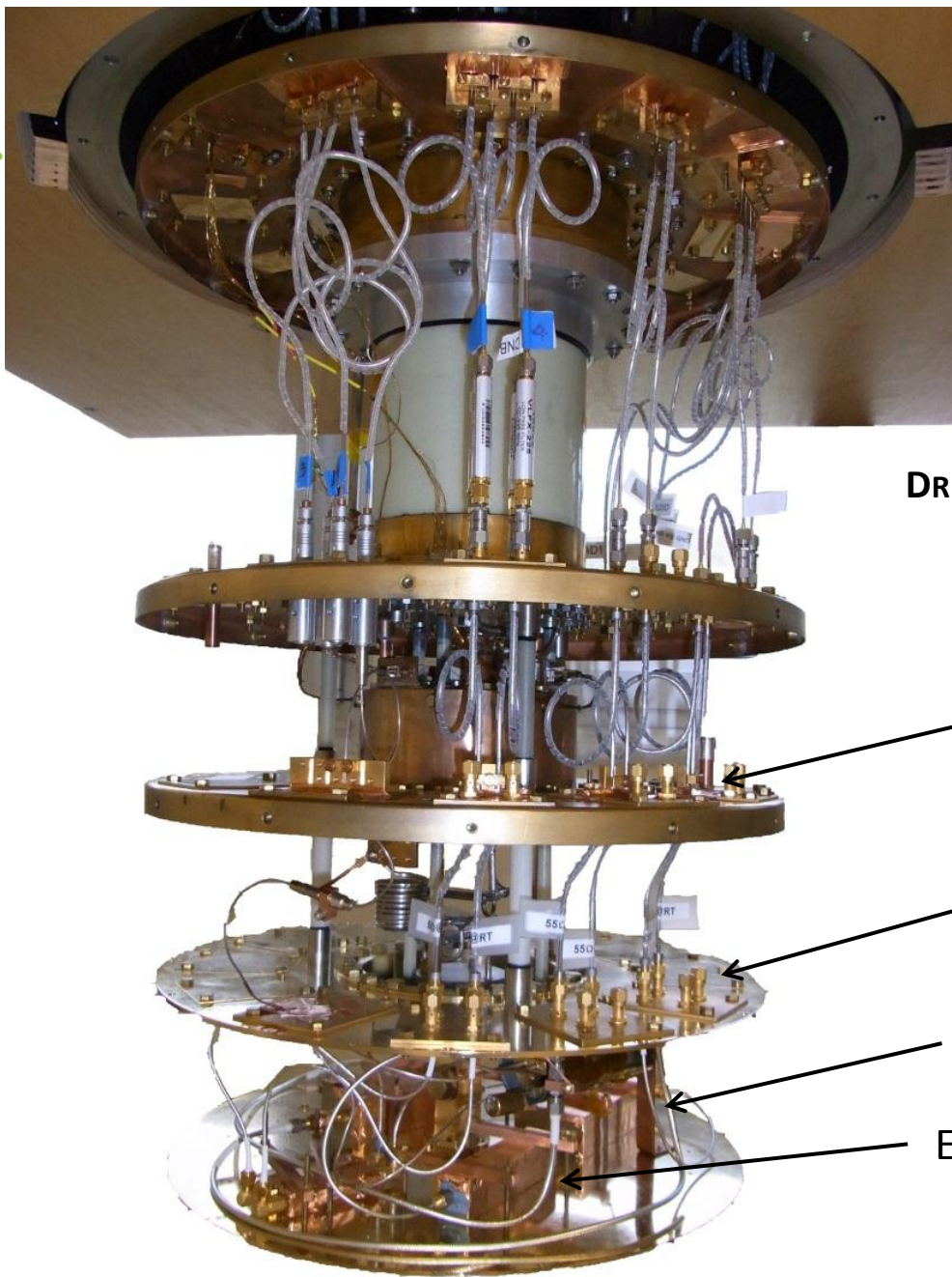
PT1	RPT1	PT2	RPT2	Still	Doub le Still	100m K	MC	P _P	P _{INJ}	n
47K	61.6 K	3.34 K	3.64 K	0.937 K	0.72 K	60.5 mK	16m K	9.8 10 ⁻²	410m b	210μmoles /s

Run complet du 20 au 21 novembre 2008

RUNING PARAMETER OF FREE DILUTION REFRIGERATOR







**DR ESTEVE DEVICE (SPEC/SACLAY) : QUANTUM
ELECTRONIC EXPERIMENTS @ 20 mK**

Electronic devices @ 1K
(pre amplifiers, ...)

Mixing Chamber plate

Additional 20 mK plate

Electronic devices @ 20 mK

Projet Marrache @ CSNSM



Bride 300 K

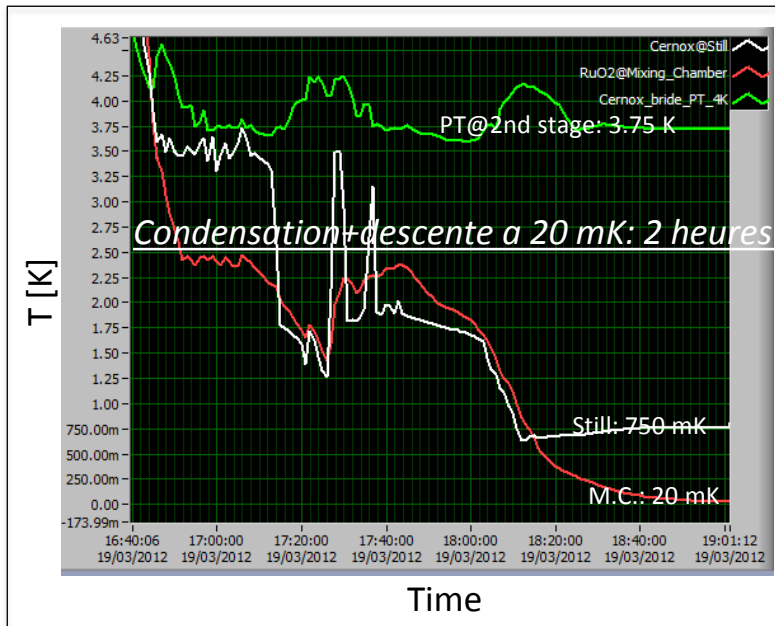
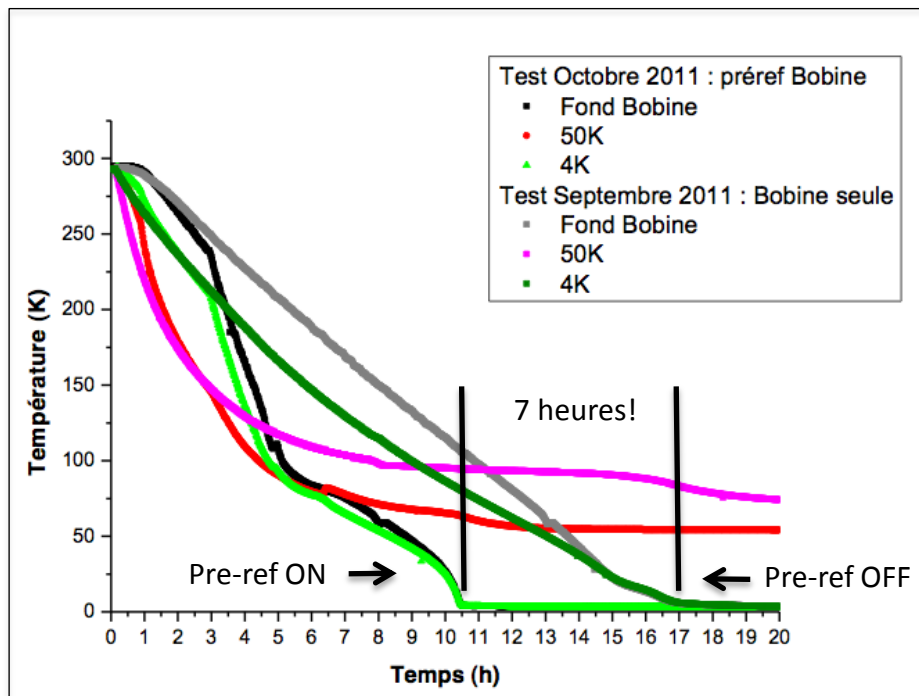
Bride 70 K

Bride 4 K

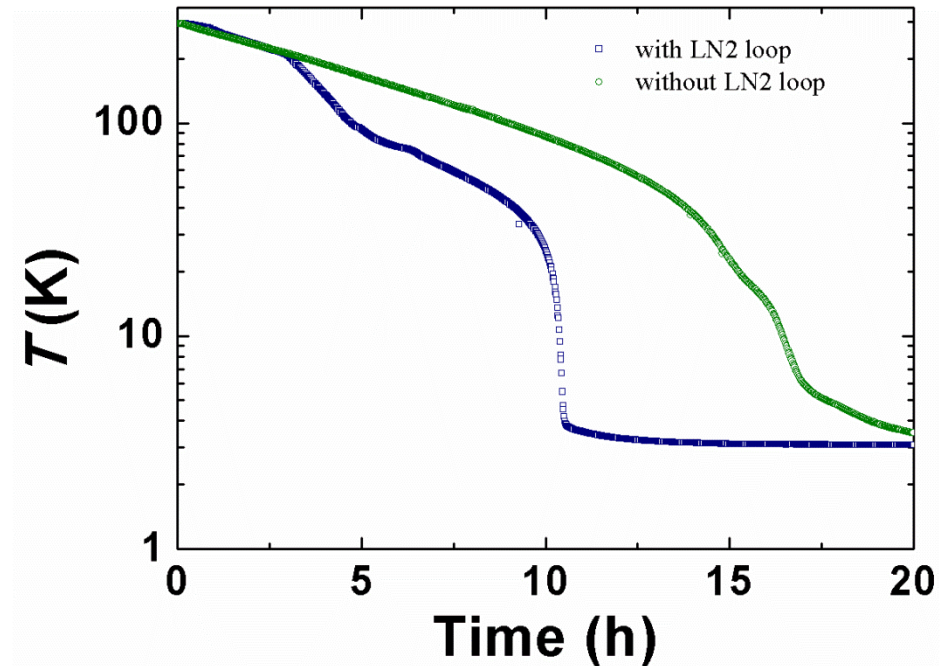
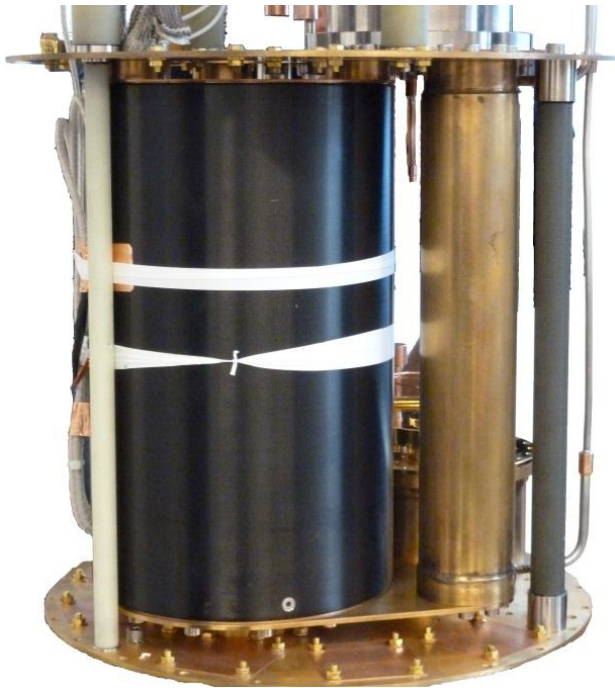
Pre-ref. Azote Liquide

Etage 1 K (Still):
5 mW@850 mK

Etage 10 mK (Mix. Chamber):
5 uW@20 mK, $T_{min} = 13$ mK



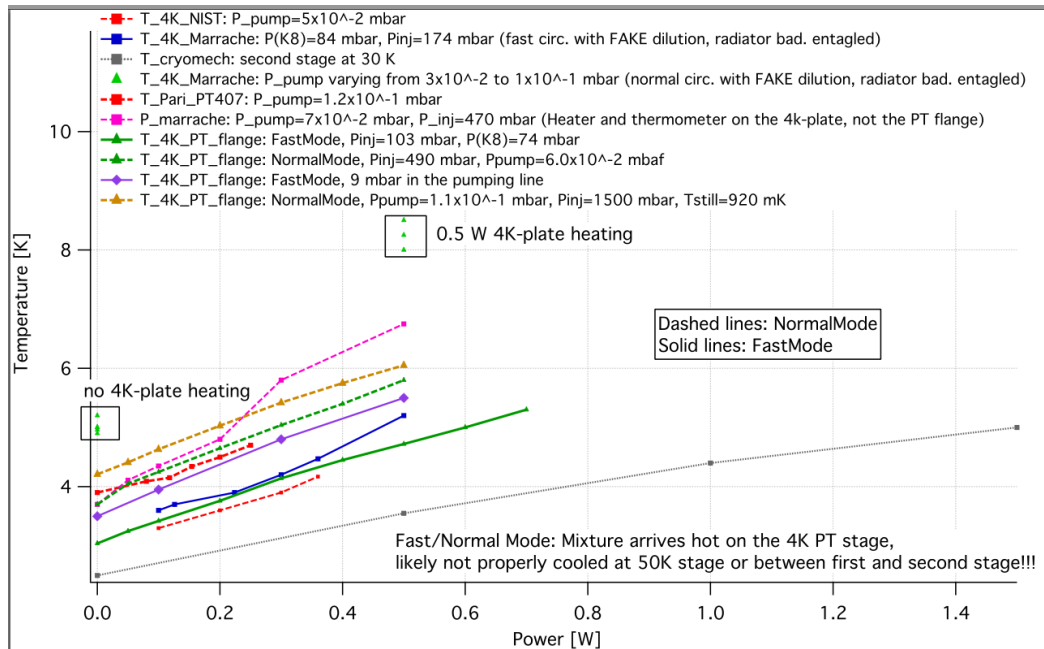
PERFORMANCES – COOLING A 9 T MAGNET



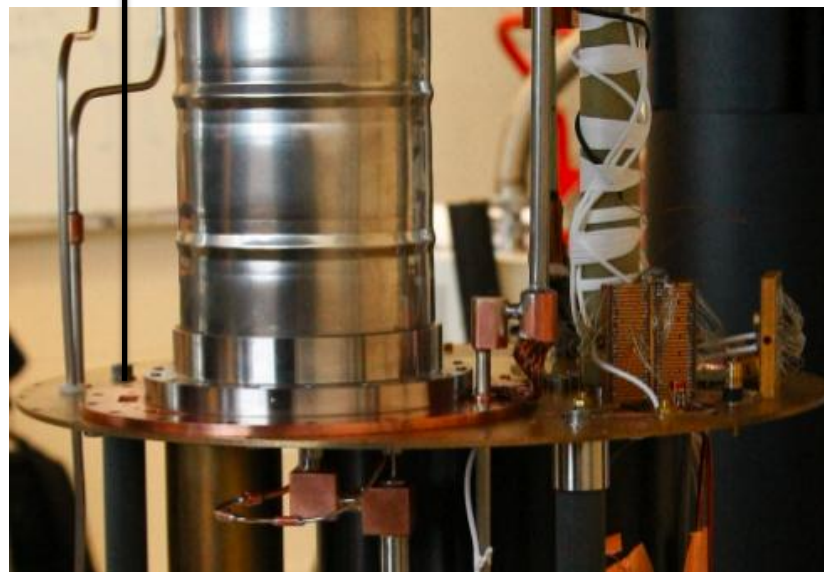
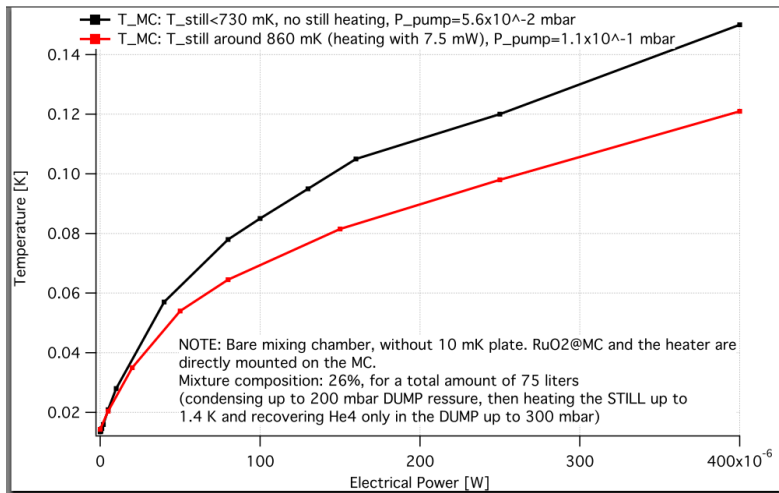
Projet Marrache @ CNSM

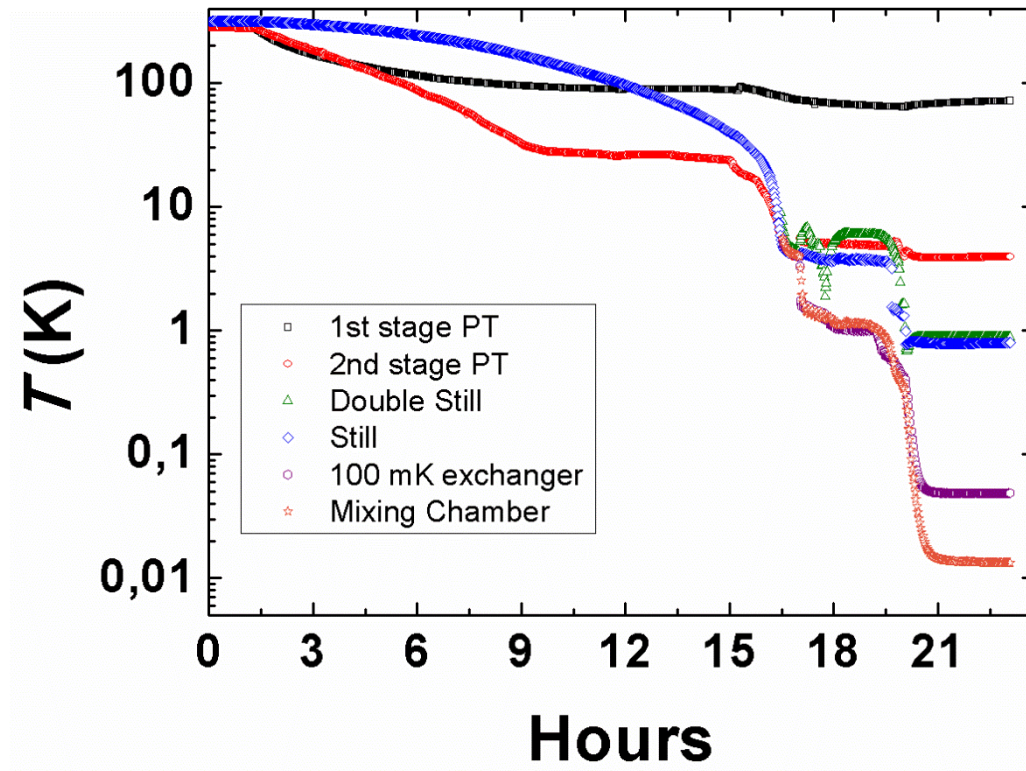


Puissance disponible sur la bride 4 K



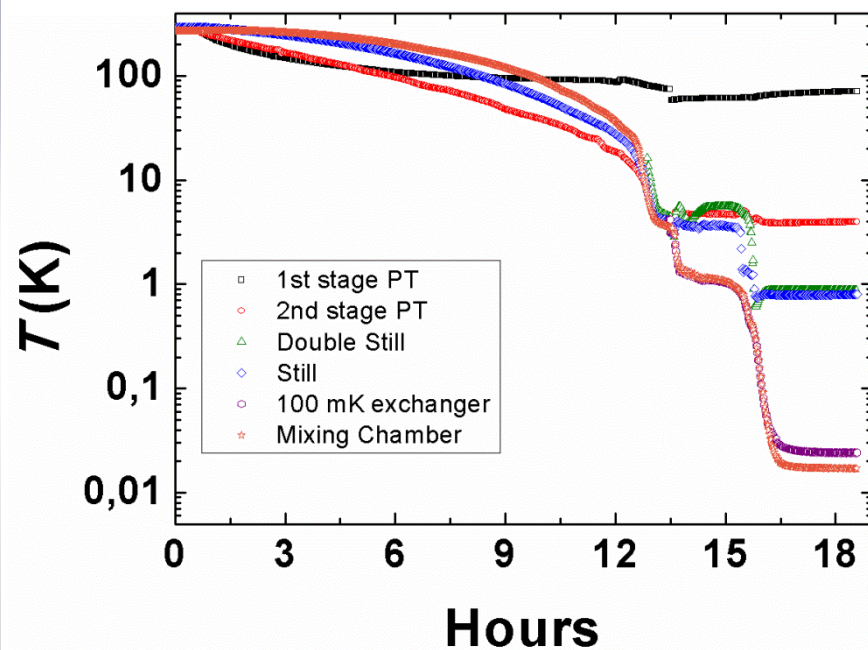
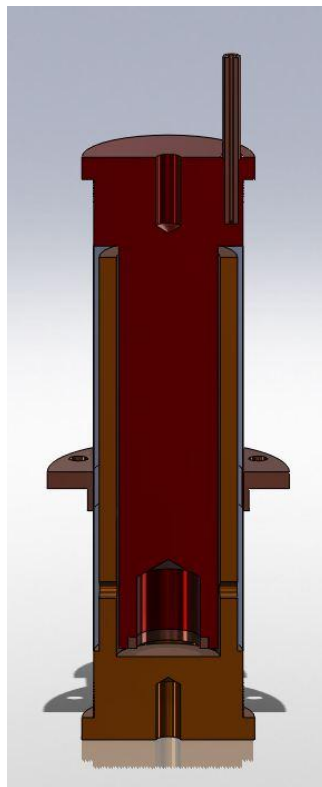
Puissance sur la Mixing Chamber



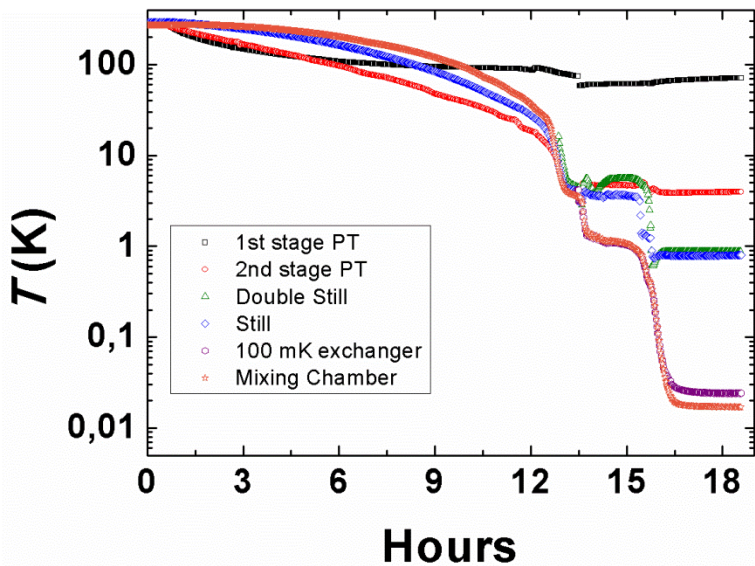


< 21 h from 300 K to < 20 mK

THERMAL SWITCH BETWEEN 4 K STAGE AND 1 K STAGE



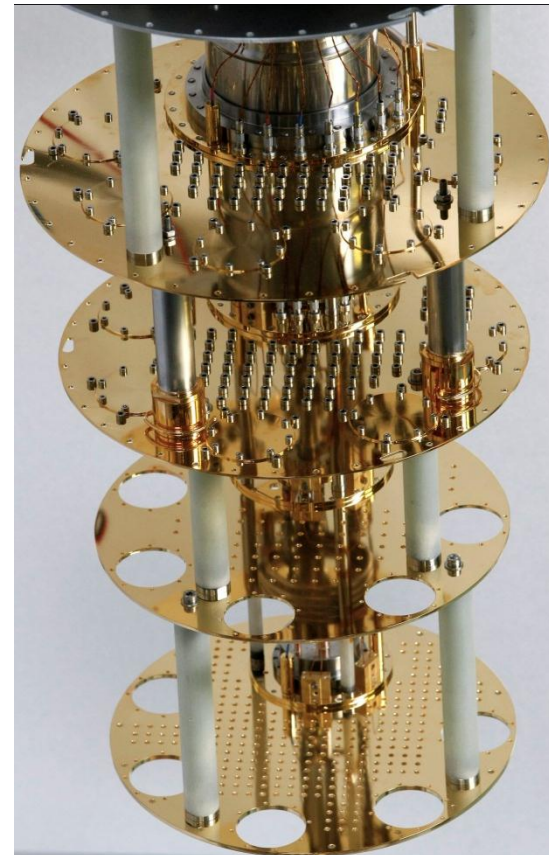
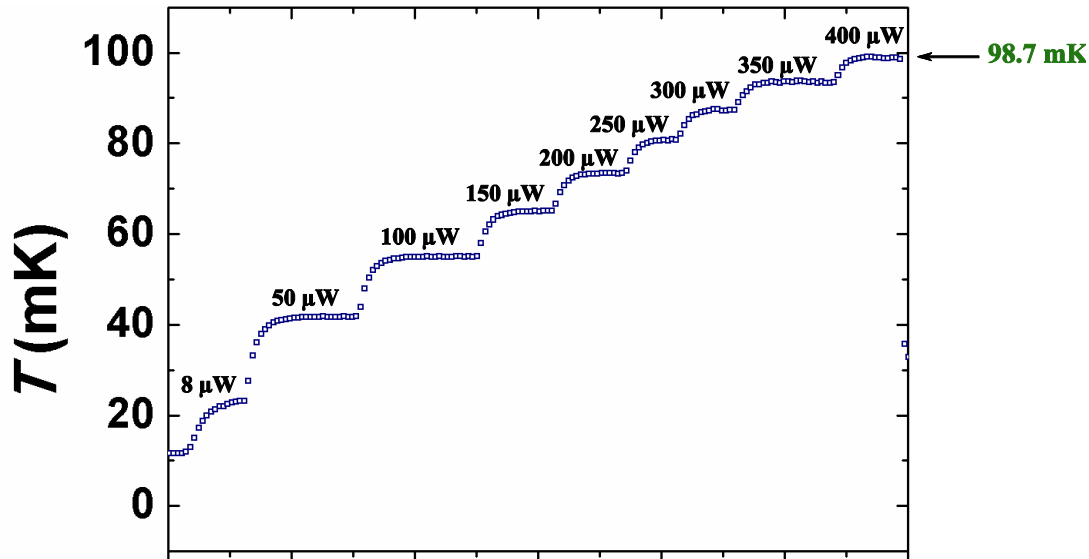
< 17 h from 300 K to < 20 mK



up to $7 \mu\text{W}$ @ 20 mK
 up to $400 \mu\text{W}$ @ 100 mK

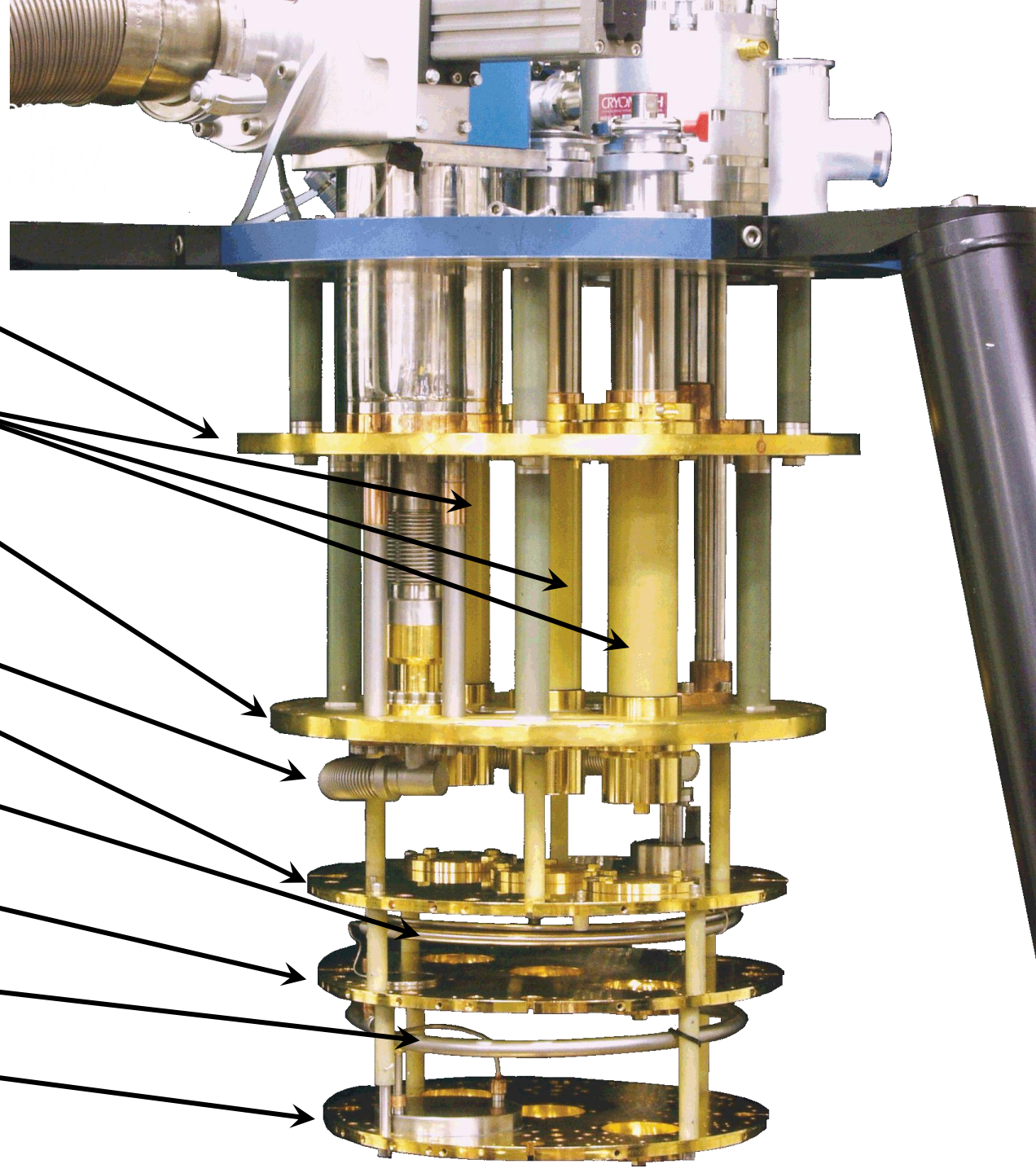
fast cool down (17h from 300 K to < 20 mK)

large experimental space
 (up to $\phi 300$ mm x h=300 mm)





CRYOGEN-FREE Models



50K plate

3 clear 50mm shot tubes

3K plate

JT heat exchanger

Still plate

Continuous heat exchanger

50mK plate

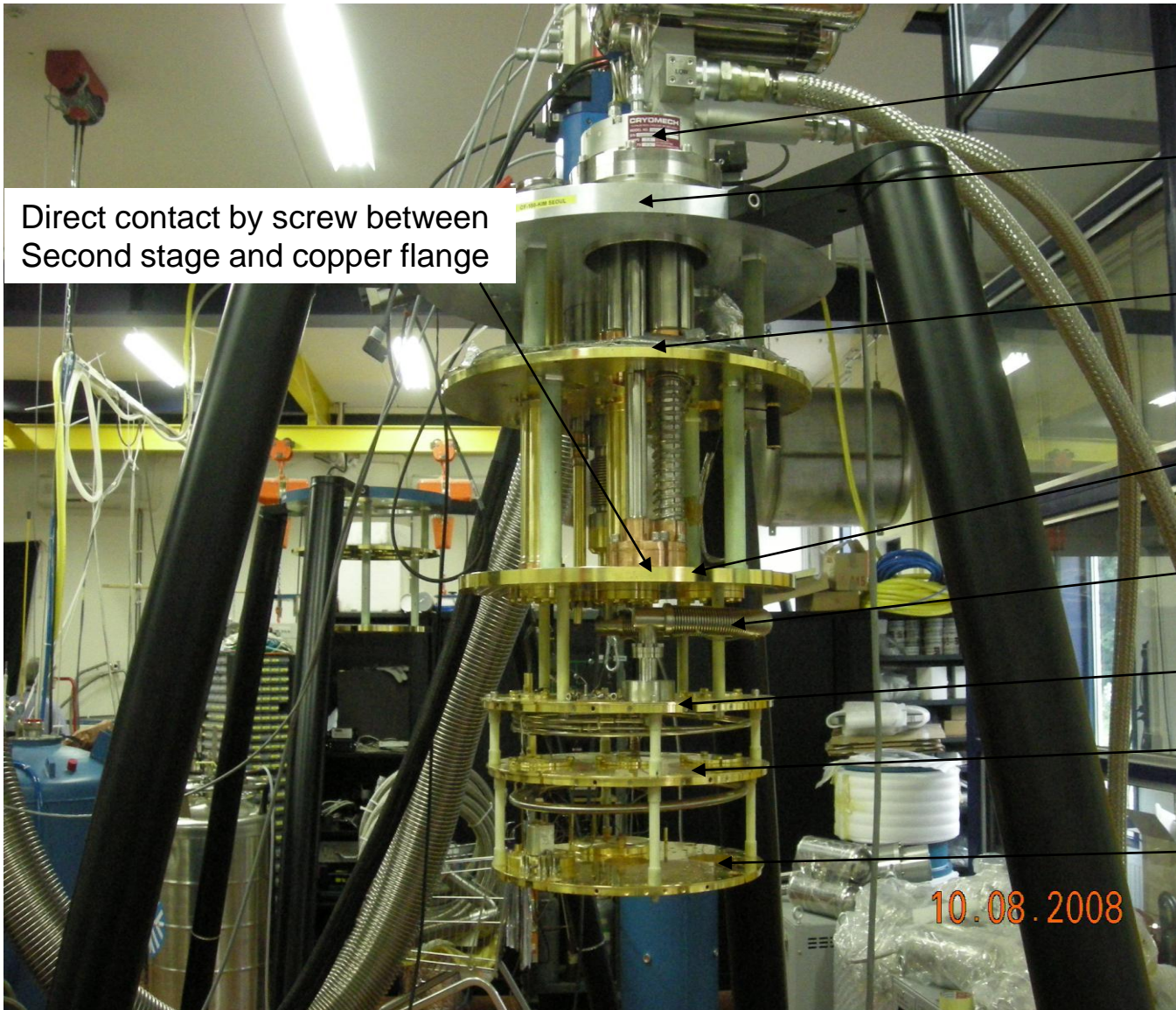
Sintered heat exchanger

Mixing chamber plate



**CRYOGEN-
FREE
Models**

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



Direct contact by screw between
Second stage and copper flange

PT 415 : 1.5W@4.2K

300K flange (dural)

First stage 50K

Second stage IVC 3K

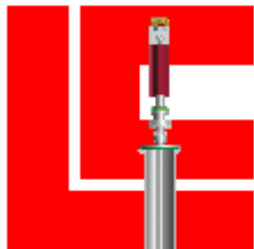
Joule-Thomson

1K flange

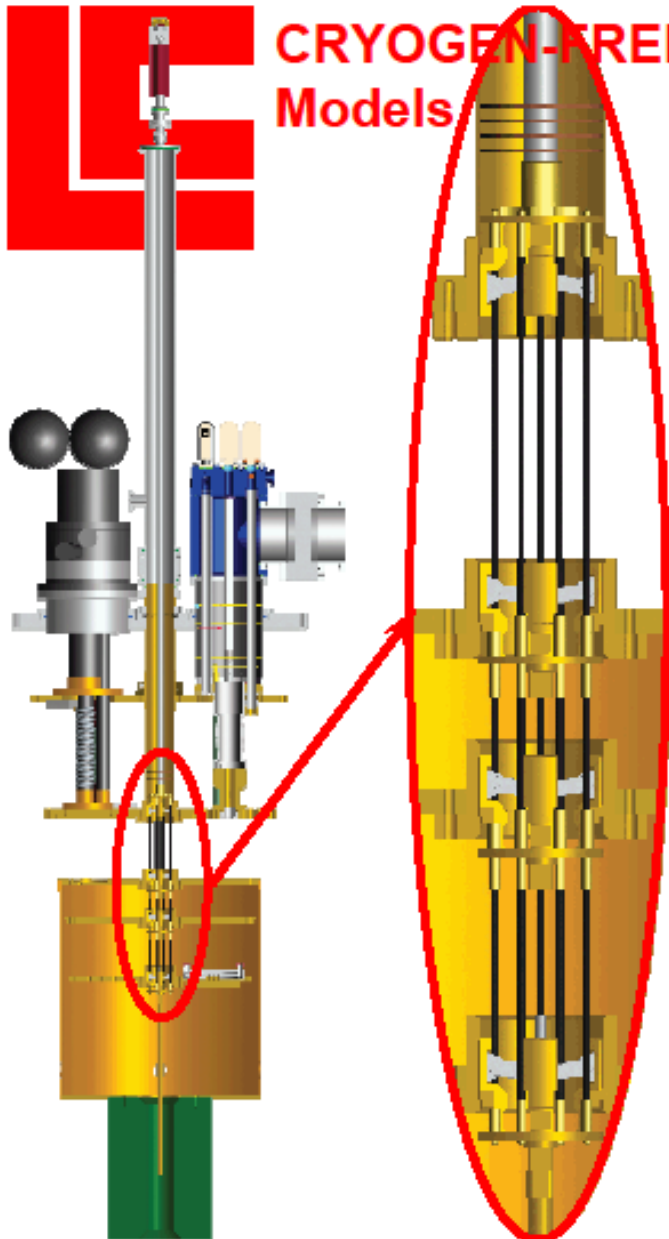
0.1K flange

Mixing chamber flange

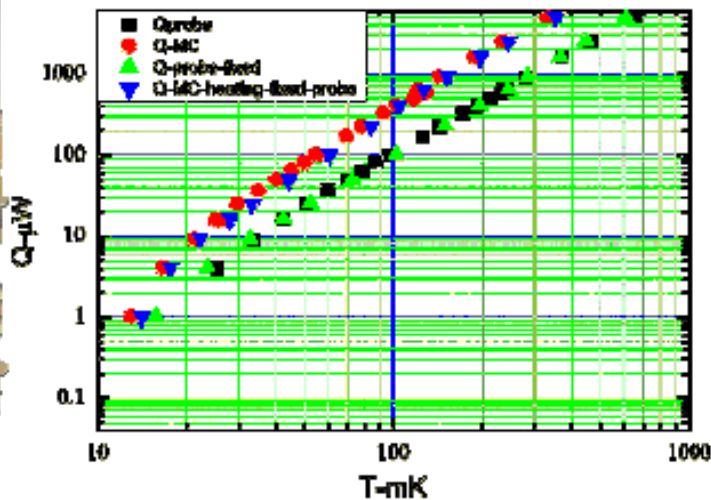
10.08.2008



CRYOGEN-FREE Models



Insertable Probe



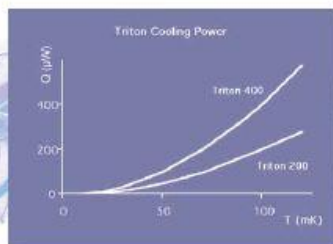
TRITON

Triton Dilution Refrigerator

OXFORD
INSTRUMENTS

10 mK without the need for liquid helium!

- Low vibration pulse tube refrigerator technology
- 10 mK base temperature
- 200 and 400 μ W cooling power at 100 mK
- 3 μ W cooling power at 20 mK
- Fast cooldown times of less than 24 hours
- Excellent temperature stability from 10 mK to 30 K
- Automatic gas handling system
- Oil free circulation pumps
- Single common vacuum, no requirement for exchange gas
- 240 mm x 240 mm sample space enabling integration of complex experimental set-ups
- Built in cryogenic cold trap technology traps contaminants
- Central access for top loading
- Compatible with integrated superconducting magnet systems up to 18 T, wiring services or sample loadlock for a complete Cryofree solution



THE QUEEN'S AWARDS
FOR ENTERPRISE:
[INNOVATION
2010

Award
winning
product

Triton™ family

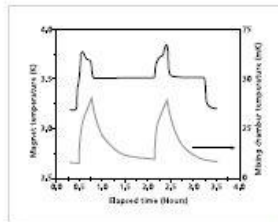
Environment and sample handling



Integrated superconducting magnets provide combined B/T environments cooled by a single PTR

A range of integrated superconducting magnets are available for the **Triton** range.

- 3, 5, 8, 12 and 15 T solenoids available as standard options
- **NEW Triton Ultra** offers magnet options up to 18 T
- HTS current leads for low heat dissipation between 50 K and 4 K
- Fast ramping capability using the latest high field, low loss Nb₃Sn developed by Oxford Instruments
- 70 mm cold bore standard on NbTi coils, 57 mm cold bore standard on Nb₃Sn coils
- 1 in 10³ over 10 mm dsv homogeneity
- 1 in 10⁴/hr stability with persistent mode switch fitted
- Custom designs, split pairs and Vector Rotate coils available on request



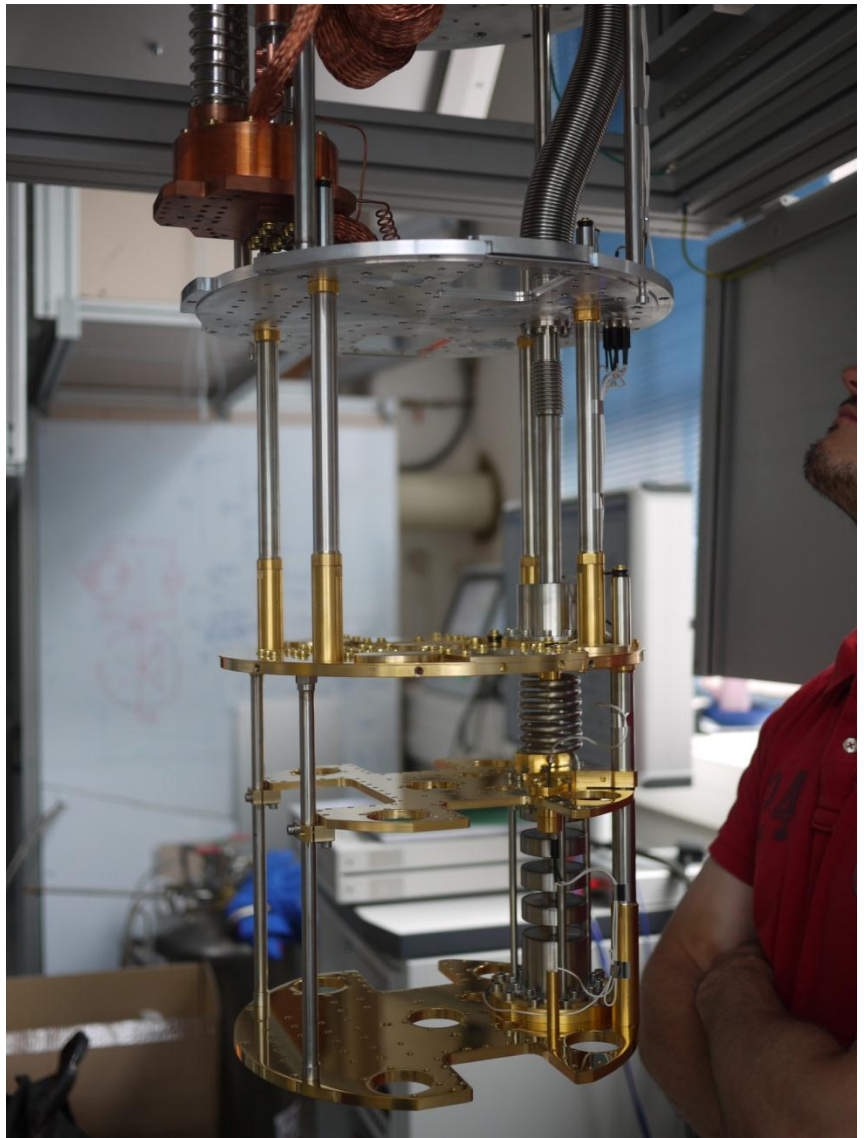
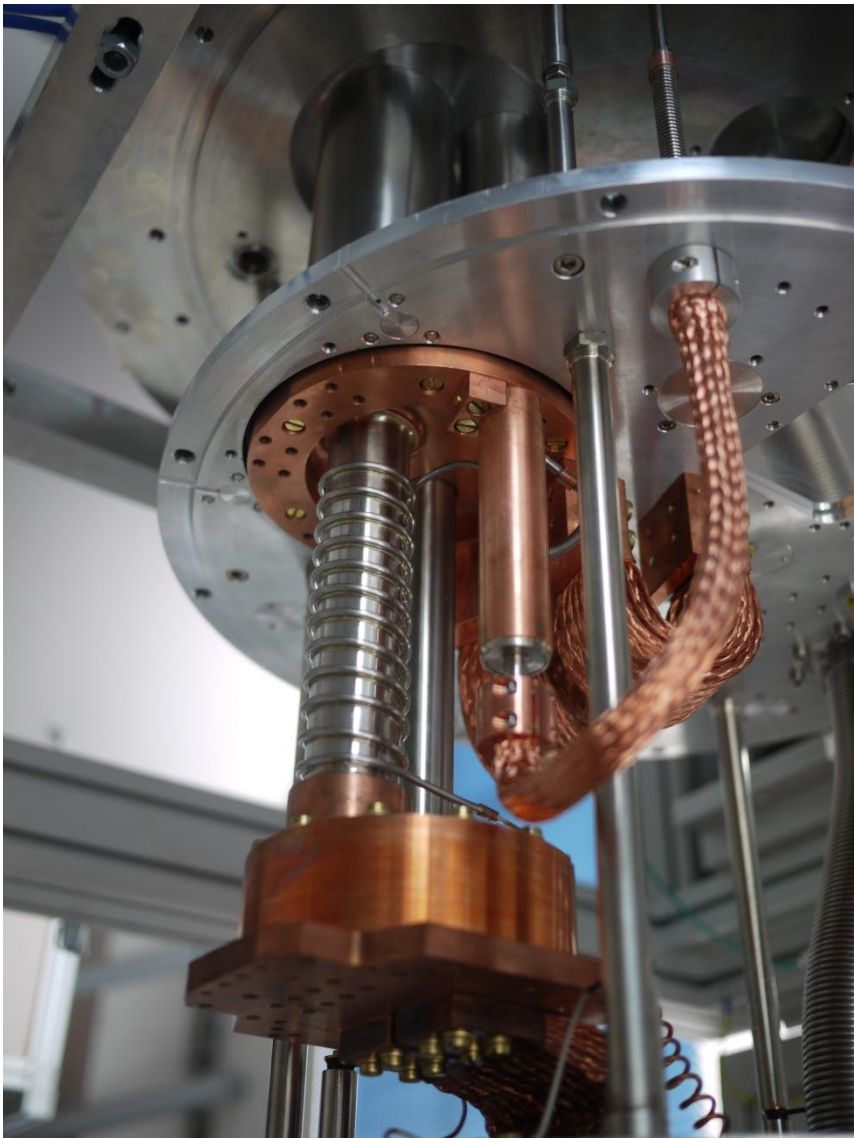
Integrated magnet warming and cooling during 5 T field sweep, hold at field and sweep back to 0 T (sweep rate 0.31 T/min). Local eddy current heating at the mixing chamber also shown during field sweep with time to recover back to base temperature ~ 1 hour. 3 μ W cooling power available at 20 mK.

Top and bottom loading loadlock for fast sample exchange

- Patented design
- 15 min sample exchange
- Cooldown time < 8 hrs
- Top-loading demountable sample carrier cooled to 10 mK
- All wiring efficiently heat sink on the dilution refrigerator



Triton top loading with sample carrier and loading probe



CONCLUSION

- **Dilution DRY 4 à 6 fois moins cher à l'utilisation que le WET.**
- **Maximum de place....**
- **Cryogénie DRY : fonctionne + de 300Jours/ an!**
- **Mais on remplace des récipients par des machines....**
- **Pas trop de recul sur la fiabilité des PT.**
- **Écarts de performances entre différentes machines.**
- **Boucle froide indispensable**
- **Technique au point. Minimum 4 Sociétés sur ce marché.**