Powerful dilution refrigerators for particle physics

From nanowatts to watts below 1 K

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1. Introduction

- 1969 dilution refrigerator (DR) for solid state physics with Mössbauer effect (diploma work); guidance from Olli V. Lounasmaa, Gösta Ehnholm, John Wheatley
- 1970 horizontal DR for polarized target (PT) studies (thesis work), guidance from Michel Borghini
- 1974 powerful DR for large PT's



DR for Mössbauer research built in 1968 – 1969, was still used a few years ago

- Vibrating source holder just above the Mixing Chamber (MC)
- Vibrator drive at RT, connected by a thin tubular shaft
- Base T = 20 mK
- Very stable operation achieved by control of upflow and downflow channels in the concentrated stream
- Was copied and distributed by the SHE Inc.
- Materials choices guided by the experience gained in adiabatic demagnetization refrigerators (thanks to George Pickett)

Horizontal DR insert for Polarized target studies (simplified version with no IVC)



- Built and used for the doctoral thesis works of Willem de Boer and myself in 1970 1971
- Was copied and distributed by a Japanese firm
- Very stable operation in horizontal position
- Maximum power 10 mW, permitted dynamic nuclear polarization (DNP) in MC
- Minimum temperature ≤ 20 mK, with sintered counterflow heat exchanger
- First version was published in Nucl. Instrum. and Meth. **97**, 95 (1971)

Frozen spin polarized target DR

Maximum flow 20 mMol/s Maximum power 100 mW Minimum temperature 16 mK

2. Route map for power

- What were the problems to be solved?
 - Horizontal position
 - Unknown limits to ³He flow
 - Unknown limits to heat transfer from target to He in the MC
 - Method to get the target into the MC without destroying it (loading under LN₂)
 - How to minimize the thickness of cryostat walls around the PT?

Horizontal position

- Was considered as a serious issue by the community, speculating on violent osmotic phenomena and 2-phase convection that were thought to be the cause of instabilities in some previous machines
- Based mainly on my ignorance I first thought that, similar to 2nd law of thermodynamics, pressure gradient must be pointed in the direction of flow of matter, and therefore liquid cannot fall from the dilute stream into the still
- A little later I understood that the surface tension and the capillary forces will keep the liquid inside the machine, even much above the still liquid level, unless a hotspot is available to initiate the nucleation of a vapour bubble

What limits the flow of ³He?

- 1. Heat exchanger(s): optimum design formulas (Section 3)
- 2. Diffusion in the dilute stream: makes a bottleneck in the hot end of the exchanger $\max(\dot{n}_3) \le 0.5 \frac{\text{mMole}}{\text{s}} \times \left(\frac{A_d}{\text{mm}^2}\right)$
- 3. Viscosity in the dilute stream
- 4. Superfluid phenomena below 0.3 K (see de Waele & coworkers)
- 5. Boiling in the still: must stay in nucleate boiling regime (heat flux below 3 mW/cm²)
- 6. Pumps: Root's blowers, often followed by rotary blade pumps; volume flow critical if diffusion limits the flow in the dilute stream (see point 2. above)

Limits to the heat transport inside the MC

- Kapitza resistance gives bead temperature > 50 mK above that of dilute solution, at 200 mK and 0.1 mW/cm³ microwave power dissipation
- Convection of dilute solution: with phase boundary at 200 mK and < 1 mW/cm³ power dissipated in the target by DNP, the dilute fluid surrounding the beads has a temperature at most 10 mK above that of the phase boundary
- Comparing with evaporation refrigerators, the higher heat transport capacity due to convection of dilute solution permits to reach much lower spin temperatures by DNP

Target loading into the MC?

- Vitreous hydrocarbons doped with free radicals devitrify around 130 K
- Irradiated ammonia loses its color centres at 110 K
- Target loading under LN₂ requires specific design features
 - indium seal for horizontal DR's
 - no seal for vertical DR's

Thinning the walls of the cryostat

- Thickness is measured in radiation length [g/cm²]
- Use low-Z materials such as Mylar or epoxy reinforced with Kevlar fibres
- At RT use Al alloys for elastic stability
- In radiation shields use pure Al

3. Optimum design of DR

- Published in LT14 (Otaniemi 1975) and in ICEC6 (Grenoble 1976)
- Exact formulas for maximum cooling power available at optimum ³He flow
- Inequalities for minimising/neglecting the effects of viscous heating and axial conduction in the main heat exchanger

The cooling power

$$\dot{Q}_{\!_{m}}\!\left(T_{\!_{m}}\right) = \dot{n}_{\!_{3}}\!\left[H_{_{\ell}}\!\left(T_{\!_{m}}\right) - H_{_{c}}\!\left(T_{\!_{o}}\right)\right]$$

can be maximized by varying ³He flow while keeping other variables constants:

 $\begin{array}{c} \left(\begin{array}{c}an_{3}\end{array}\right)_{T_{m},T_{s},x_{4}} \\ \text{This yields immediately the optimum flow of }^{3}\text{He:} \quad \dot{n}_{opt} = \frac{H_{l}\left(T_{m}\right) - H_{c}\left(T_{o}\right)}{C_{c}\left(T_{o}\right)\left(\frac{dT_{o}}{d\dot{n}_{3}}\right)_{T_{m},T_{s},x_{4}}} \end{array}$

$$\begin{split} d\sigma_{c} \alpha \left(T_{c}, T_{d}\right) &= d\sigma_{c} \alpha_{c} \left(T_{c}\right) \\ \dot{n}_{3}^{\text{opt}} &= \frac{\sigma \alpha \left(T_{o}\right)}{H_{\ell} \left(T_{m}\right) - H_{c} \left(T_{o}\right)}, \qquad \dot{Q}_{m}^{\max} \left(T_{m}\right) = \sigma \alpha \left(T_{o}\right) \end{split}$$

The outlet temperature of the concentrated stream is solved from the equation

$$\alpha \left(T_{_{o}}\right) \int_{_{T_{_{0}}}}^{_{T_{_{s}}}} \frac{C_{_{c}}\left(T_{_{c}}\right)}{\alpha_{_{c}}\left(T_{_{c}}\right)} dT_{_{c}} = H_{_{\ell}}\left(T_{_{m}}\right) - H_{_{c}}\left(T_{_{o}}\right)$$

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The numeric solution of this equation yields the ratio T_o/T_m that is universal and that is practically constant = 1.9 below 50 mK.

In this region the solution approaches asymptotically

$$\dot{Q}_m^{\max} = \left(\frac{a}{2b}\right)^2 \sigma_c S T_m^4 \cong 12.5 \sigma_c S T_m^4$$

 $\dot{n}_3^{\text{opt}} = \frac{a}{2b^2} \sigma_c S T_m^2 \cong 0.27 \sigma_c S T_m^2 \frac{\text{K}^2 \text{mol}}{\text{J}}$

Above 50 mK it is better to rely on the numeric solution for the temperature ratio T_o/T_m $4L \max{\{\dot{Q}_m\}}$ 1

$$I_c \ll \frac{4L \max\{\dot{Q}_m\}}{3T_c \kappa_c(T_c)} \frac{1}{1 + \frac{1}{3} \frac{\mathrm{d}\kappa_c}{\mathrm{d}T_c} \frac{T_c}{\kappa_c}}.$$

The axial conduction is likely to be most important at the lowest temperature operation; we may then replace the logarithmic derivative by its maximum of 1 and put $\max{\{\dot{Q}_m\}} = \dot{Q}_m^{\min}$, obtaining a simple result:

$$A_c \ll \frac{L\dot{Q}_m^{\min}}{T_c \kappa_c(T_c)} \,.$$

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Neglect of flow heating and axial conduction

Neglect of conduction in concentrated stream (LT end):

$$A_c << rac{4L \max\left\{\dot{Q}_m
ight\}}{3T_c\kappa_c} rac{1}{1+rac{1}{3}rac{T_c}{\kappa_c}rac{d\kappa_c}{dT_c}} \cong rac{L\dot{Q}_m^{\min}}{T_c\kappa_c\left(T_c
ight)}$$

Neglect of viscous heating in the concentrated stream (LT end):

$$\frac{dZ_{c}}{dz} << \frac{b^{2}}{\sigma S \eta V_{c}^{2}} \frac{d\sigma}{\sigma dz}$$

For the dilute stream both terms are smaller and less critical in LT operation, but at high flow rates in HT operation, the dilute flow passages must satisfy the experimental rule

$$\max(\dot{n}_3) \leq 0.5 \frac{\text{mMole}}{\text{s}} \times \left(\frac{A_d}{\text{mm}^2}\right)$$

4. Two examples of DR designs

- Large polarized targets for the experiments of
 - European Muon Collaboration (EMC)
 - Spin Muon Collaboration (SMC)
 - COMPASS muon DIS experiments
 - 2 W cooling power required around 500 mK
- Large DR for the EURECA Dark Matter search
 - Deep underground experiment
 - Extreme radiopurity of DR materials near detector array
 - Array and LT parts of the DR inside water shield
 - 20 μW power to be absorbed at 7 mK



COMPASS PT magnet



SMC DR in COMPASS magnet



COMPASS PT in operation



COMPASS triple-cell cavity



COMPASS double- and triple-cell target holders





Triple-cell cavity mounted CERN Cryolab DR for Dark Matter detector studies (EURECA) Work done by Patrick Wikus, TU Wien 2007









"1 μm " Cu powder sintered at 750 °C, SEM image magnification 1000



Work done by Gerhard Burghart, Thesis, TU Wien 2010:

Thermal resistance between samples of sintered Cu sponges and dilute solution inside the mixing chamber of CCDR:

- Grain size ≈ 1 µm
- Sintering at 750 °C
- Surface area 1.3 m²/cm³

For comparison, results on other fine powders are shown

We are not able to explain why thicker sponges have lower thermal resistance below 30 mK.

The results are important for DM calorimeters, because Ag powder cannot be used due to radiopurity requirements



The vacuum vessel inside the "Pool" housing the detector array experimentary

are the heat shields, C1, C2, C3 are the three parts of the vacu

e PMMA cast around a hermetic, thin-walled copper structure

itanium alloy.

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Desing example for EURECA cryostat (colder part)

- A calorimeter array, h = 1 m, $\phi = 1 \text{ m}$
- B thermal shields
- C OVC made of PMMA clad in thin Cu
- D Titanium alloy support

Vacuum insulated cryoline connects the MC to the still via the tubular countercurrent heat exchanger.

The still and precooler are located at a distance of about 10 m, outside the experimental cavern.

