CONVENTIONAL CYCLE DILUTION REFRIGERATION DOWN TO 2.0 mK

G. Frossati[*],H. Godfrin, B. Hébral, G. Schumacher, D. Thoulouze
Centre de Recherches sur les Très Basses Températures,C.N.R.S. ,
B.P. 166 X ,38042 GRENOBLE Cédex ,FRANCE.

Introduction

Dilution refrigerators are now the most important tool for producing temperatures in the 10 mK region because of their ability to maintain these temperatures continuously. Besides their use to cool all kinds of samples to their minimum working temperatures ,dilution refrigerators are also widely used to precool Pomeranchuk cells, electronic and nuclear demagnetization stages ,all systems which lead to much lower final temperatures.

The importance of reaching lower and lower temperatures needs not to be stressed as was shown by the very fertile fields of superfluid ^{3}He ,solid ^{3}He ,nuclear magnetism ,mixtures of 2 superfluids and so on.

In all cases it is a great advantage to decrease the initial temperatures to which the above cited systems are precooled ,leading to lower final temperatures or smaller initial magnetic fields ,smaller nuclear or electronic cooling stages or longer working times.

In this paper we simplify as much as possible the problem of achieving ultralow temperatures with a dilution refrigerator stressing only those points which we have found important after many years of experiences particularly designing and constructing efficient heat exchangers. We also comment on still and mixing chamber design as well as the influence of the diluted-side size on the flow rate and some possibilities of reaching temperatures lower than our present 2.0 mK in continuous mode.

Obtaining the lowest temperatures with a Dilution Refrigerator

The theory of the dilution of ^{3}He in ^{4}He and its use in refrigeration has been treated extensively in the literature (ref.1 to10) and the reader should refer to one of these papers for a deep understanding of what is going on inside a dilution refrigerator.

Let us consider the expression of the enthalpy balance on the

* Presented by G. Frossati as an invited speaker.

mixing chamber of a dilution refrigerator. The enthalpy of the concentrated phase is $H_c = 12.5\ T^2$ J/mole; that of the dilute phase, on the separation curve :

$H_d = 94.5\ T^2$ J/mole (T in K). If T_c and T_{mc} are respectively the temperatures of the incoming ^{3}He and of the mixing chamber, in Kelvin, we may write the enthalpy balance of the mixing chamber in dynamic equilibrium, for a heat leak $\dot{Q}$ (watts) and a ^{3}He flow rate $\dot{n}$ (mole S^{-1}) as :

$$\frac{\dot{Q}}{\dot{n}} = 94.5\ T^2_{mc} - 12.5\ T^2_c\ \text{J/mole} \qquad (1)$$

$$\dot{Q} = \dot{Q} + \dot{Q}(\dot{n})$$

Figure 1

For a given heat leak $\dot{Q}$, T_{mc} is minimum when $T_c = T_{mc}$, that is

$$T^2_{mc_{min}} = \frac{1}{82}\frac{\dot{Q}}{\dot{n}} \qquad (2)$$

For example, given a reasonable heat leak $\dot{Q} = 0.1\ \mu W$ and a flow rate $\dot{n} = 3 \times 10^{-4}$ moles/sec

$$T_{min} = 2.0\ \text{mK}.$$

Also, from (1), if $\frac{\dot{Q}}{\dot{n}} = 0$,

$$T_{mc} = 0.36\ T_c. \qquad (3)$$

This is to say that if we want to cool the mixing chamber to the lowest possible temperatures, T_c has to be decreased and this is done by means of heat exchangers which remove heat from the low enthalpy concentrated ^{3}He phase and give it to the higher enthalpy diluted ^{3}He.

The most efficient heat exchangers (H.E) are of the continuous counterflow type, in contrast with the step or localized ones and we will only consider the 1st type which certainly leads to better results, as well as being more economical with respect to the volume of ^{3}He required.
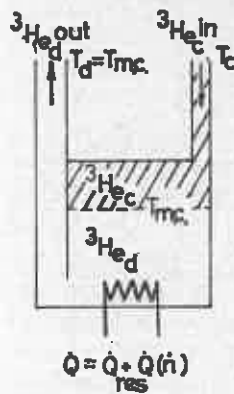
Perfect continuous heat exchangers

A continuous counterflow H.E is described by a system of 2 coupled differential equations containing each one a term due to the heat exchanged between the two phases, through the separating wall, via the Kapitza mechanism, another which takes into account the viscous heating of the liquids due to their motion along the channels and finally a term due to the thermal conductivity of the liquids themselves. This equation has no analytical solution, but in practice it is possible to design H.E. where the viscous heating and the heat conducted axially along the liquids and of course along the body of the H.E. themselves can be made small compared to the heat transfered from the concentrated to the diluted phase. In this case we are left with that is called a "perfect continuous H.E." for which "all the heat given by the concentrated stream is taken by the diluted stream".

In practice

$$\dot{Q}_{visc.} + \dot{Q}_{cond.} \ll \dot{Q}_{exch.}$$

Let us consider a wall separating the concentrated from the diluted stream in a H.E. presenting an area σ_c in contact with the concentrated phase and σ_d in contact with the diluted phase (fig. 2).

Then, if the heat is exchanged through the phonon mismatch process (réf. 11-12) and if T_w is the temperature of the wall

$$\frac{\Delta\dot{Q}}{\Delta\sigma_d} = \alpha_d(T^4_w - T^4_d) \qquad (4)$$

Figure 2

$$\frac{\Delta\dot{Q}}{\Delta\sigma_c} = \alpha_c(T^4_c - T^4_w). \qquad (5)$$

If the exchange areas are equal ($\sigma_c = \sigma_d = \sigma$)

$$\frac{\Delta\dot{Q}}{\Delta\sigma} = \alpha_m(T^4_c - T^4_d) \qquad (6)$$

with α_m, the average heat transfer coefficient

$$\alpha_m = \frac{\alpha_c \alpha_d}{\alpha_c + \alpha_d} = \frac{1}{4\,R_{Km}} \qquad (7)$$

R_{Km} being the average Kapitza resistivity, related to the Kapitza resistance R_m by

$$R_m \; \sigma \; T^3 = R_{Km} \qquad (8)$$

If $\alpha_c = \alpha_d = \alpha$, which is quite possible since there is always some ^{4}He circulated in the concentrated phase, then $\alpha_m = \alpha/2$ and $R_{Km} = 2 R_K$.

The condition of a perfect H.E. is that at each point all heat given by the concentrated phase be taken by the dilute phase :

$$d\,H_c = d\,H_d \qquad (9)$$

or

$$C_{3c}\,dT_c = C_{3d}\,dT_d \qquad (10)$$

For temperatures such that the concentrated ^{3}He and the diluted ^{3}He can be considered respectively as a Fermi liquid and a Fermi gas (below about 50 mK), the heat capacities are proportional to the temperature, with

$$C_{3c} = aT = 25\ T\ \text{J/mol}$$

$$C_{3d} = bT = 107\ T\ \text{J/mol} \qquad (11)$$

if we put $\Delta\sigma = \Delta s\ \Delta Z$, we are left with the system of 2 equations :

$$\frac{\Delta H_c}{\Delta Z} = \dot{n}\ a\ T_c\ \frac{dT_c}{dZ} = \alpha_m\ \Delta s\ (T_c^4 - T_d^4) \qquad (12)$$

$$bT_d^2 = aT_c^2 \qquad (13)$$

$$\alpha_m\ \Delta s\ \{1 - (\tfrac{a}{b})^2\}T_c^4 = \dot{n}\ a\ T_c\ \frac{dT_c}{dZ} \qquad (14)$$

writing

$$\frac{\alpha_m\ \Delta s\ \{1 - (\tfrac{a}{b})^2\}}{a\ \dot{n}} = A \qquad (15)$$

Then (14) becomes

$$A\,dZ = \frac{dT_c}{T_c^3} \qquad (15)$$

Integrating from $Z = 0$, the point where the concentrated ^{3}He enters the mixing chamber, to $Z = \ell$, where ℓ is the total length of the exchanger, one gets :

$$2A\ell = T_c^{-2}\,(0) - T_c^{-2}(\ell) \qquad (17)$$

$T_c(\ell)$ is the temperature of the still, which is known to be about 0.7 K, so that $T_c^{-2}(\ell)$ is negligible compared to $T_c^{-2}(0)$. Now, we have seen from eq. (3) that $T_{mc} = 0.36\ T_c(0)$, if the heat leak to the mixing chamber is small, so that (17) becomes :

$$2A\ell \times 7.7 \simeq T_{mc}^{-2} \qquad (18)$$

Since $\ell\Delta s = \sigma$, the total exchange area, and neglecting $(\tfrac{a}{b})^2 = 5.10^{-2}$, we get the fundamental expression relating the temperature of the mixing chamber to the characteristics of the continuous exchanger, in the ideal case :

$$T_{mc}^2 = 6.4\ R_{Km}\ \frac{\dot{n}}{\sigma} \qquad (19)$$

In the actual case, there is always a heat leak to the mixing chamber so that for an infinite exchange area the minimum mixing chamber temperature is given by (2).

We will then add to (19) the limiting factor given by (2) and the final equation will be :

$$T_{mc}^2 = 6.4\ R_{Km}\ \frac{\dot{n}}{\sigma} + 1.22 \times 10^{-2}\ \frac{\dot{Q}}{\dot{n}} \qquad (20)$$

with T in Kelvin, R_{Km} in m^2K^4/watt, $\dot{n}$ in moles/sec, and σ in m^2.

About heat leaks

The expression (20) allows the calculation of the exchange area necessary to achieve a certain minimum temperature T_{mc} at a given ^{3}He flow rate $\dot{n}$ provided R_{Km} and $\dot{Q}$ are known, supposing also that some geometry for the H.E. has been devised that will make $\dot{Q}_{visc.}$ and $\dot{Q}_{cond.}$ small compared to the heat exchanged $\dot{Q}_{exch.}$.

Let us first examine the heat leak to the mixing chamber $\dot{Q}$. If we measure T_c and T_{mc}, we can determine $\dot{Q}$ from expression (1) when the refrigerator has reached its equilibrium at a given flow rate. $\dot{Q}$ can be divided in two terms, a constant one and another which depends one the flow rate :

$$\dot{Q} = \dot{Q}_{res.} + \dot{Q}(n)$$

$\dot{Q}_{res.}$ is a background heat leak coming from exchange gas, vibrations, heat conduction along the supports, heat coming eventually from plastics, etc... Usually this heat leak decreases with time, either because gas is trapped on the cold surfaces (we observe a factor 10 improvement in the pressure indication at the top of the cryostat, after one week of experiments, typically from 10^{-6} to 10^{-7} torr) or because the epoxy mixing chamber walls come to thermal equilibrium. To decrease the remaining part it is useful to mount the cryostat on a heavy concrete block (about 10^3 kg) resting on rubber pads. All rotary pumps and some large booster pumps could also be mounted on independent concrete blocks, as an additional precaution.

The use of a shield at 30 to 50 mK should decrease the effect of radiation and exchange gas. We use either ^{4}He or ^{3}He as exchange gas, typically 2-3 torr at room temperature and we pump overnight at ^{4} K.

Our background heat leaks are usually smaller than 10 nWatt after a few days that the M.C. has been at low temperatures.

In all cases, the problem of the residual heat leak can be overcome by using large flow rates, at the expense of bigger heat exchangers.

The flow dependent heat leak $\dot{Q}(n)$ is actually much bigger than $\dot{Q}_{res.}$ and we observe it to increase roughly linearly with n, that is $\frac{\dot{Q}}{n} \simeq$ constant. It would be nevertheless necessary to measure T_c more precisely because of the T^2 dependence of the enthalpy balance. This term can be attributed to a combination of viscosity and conduction effects and becomes important usually below 10 mK.

Typically, when $\dot{Q}_{res.}$ is dominating, the refrigerator reaches some minimum temperature upon decreasing the flow rate and further decrease leads to a sharp increase in T_{mc}. When $\dot{Q}(n)$ is dominating, T_{mc} will eventually stabilise for some range of n values, and only when n is low enough that $\dot{Q}_{res.}$ dominates again will T_{mc} start to raise.

$\dot{Q}(n)$ can in principle be made as small as wanted. In fact $\dot{Q}_{visc.} = Z\eta V^2 n^2$ where Z is the geometrical impedance factor (ref. 1) given by $Z = 128\, L/\pi D^4$ for a round tube of length L and diameter D and by $Z = 12\, L/AB^3$ for a rectangular channel of width A and thickness B. η is the viscosity which at low temperatures goes as T^{-2} and V is the molar volume of ^{3}He, in the concentrated ($V_c \simeq 36.8$ cm^3/mole) or diluted phase ($V_D \simeq 430$ cm^3/mole at a concentration of 6.4 %). Then $\dot{Q}_{visc.}$ is proportional to LD^{-4}. Since the heat conduction is proportional to D^2L^{-1}, it is always possible to minimise $\dot{Q}_{visc.} + \dot{Q}_{cond.}$ by increasing both length and diameter, the limit being only the volume of liquid and of course the space available for the dilution unit.

Calculation of continuous heat exchangers

We then calculate the total exchange area needed to reach T_{mc} at a flow rate n through equation 20, using a value of $\frac{\dot{Q}}{n}$ compatible with the minimum temperature chosen.

For instance, if $\sigma = \infty$ and $T_{mc} = 2.0$ mK

$$\frac{\dot{Q}}{n} = 3.2 \times 10^{-4} \text{ J/mole}$$

Of course, we do not want an infinite surface, so we must use a smaller value of $\frac{\dot{Q}}{n}$. Fig. 3 shows the plot of eq. (20) for three different values of $\frac{\dot{Q}}{n}$ using as exchange material sintered Ag powder of grain size smaller than 1 micrometer (ref. 13) called "French powder". From the higher temperature part of the curve we can deduce an R_{Km} value of 8×10^{-2} m^{2}K^4/W from the 1st term of eq. (20) where the effect of the $\frac{\dot{Q}}{n}$ term is negligible. The bending upwards of the curve passing through the experimental points at lower T_{mc} can be attributed to $\frac{\dot{Q}}{n} = 6 \times 10^{-4}$ J/mole. A second set of point using finer powder of average diameter 700 Å (called "Japanese Powder") (ref. 14) is also shown. Here, $\frac{\dot{Q}}{n} \simeq 1.6 \times 10^{-4}$ J/mole.

The French powder once sintered had a specific area of .1 m^2/gm (measured by the B.E.T. method). Details of the sintering process are given in the next section. A sample of the Japanese powder sintered to a filling factor of about 50 % and carefully outgassed at ~ 150°C had an area of 1.8 m^2/g. The exchangers themselves were sintered to a filling factor of 60 % and were not outgassed at high temperature before operation, which might explain the higher $R_{Km} = 4.8 \times 10^{-1}$ m^{2}K^4/W by

an actual loss of the total area. Measurements of the specific area of this powder under different sintering conditions are being done. Recently, values much closer to those of the "French powder" were reported (ref. 15) for the "Japanese powder", when used with pure ^{3}He.

Very fine sintered powders show an increased thermal resistivity at temperatures above about 10 mK (ref. 16-17) due mainly to the decreased thermal conductivity of ^{3}He inside the pores, and it can be seen as a departure from the ideal law at higher temperatures, for the 2nd set of points. This effect can be minimised using relatively thin layers of sinter, possibly with coarser grains for the exchangers which are supposed to work above 8-10 mK.

Bulk materials with low Kapitza resistivities and low thermal conductivities are also good candidates for continuous heat exchangers (ref. 18-20) but it becomes a problem when areas bigger than about 0.1 m^2 are needed because of the big volume occupied.

With $\frac{\dot{Q}}{n} = 1.6 \times 10^{-4}$ J/mole we obtain $\frac{\sigma}{n} = 2.5 \times 10^5 \frac{m^2s}{mol}$ for the French powder and $\frac{\sigma}{n} = 1.5 \times 10^6 \frac{m^2s}{mol}$ for the Japanese one, to go to 2mK or, respectively 50 m^2 and 300 m^2 for a flow rate of 2×10^{-4} moles/second, which means 500 g of French powder or 166 g of Japanese powder. For an average density after sintering of 5 g/cm^3, this gives sponge volumes of 100 cm^3 and 33 cm^3, which implies that 50 cm^3 of ^{3}He are needed in order to fill the pores in the French sinter and 16 cm^3 for the Japanese one. Of course, on the diluted side, the required volume of ^{3}He is roughly 6 % of the above values.

The smaller ^{3}He consumption and total volume occupied do favour the use of the finer powder, but in practive we use both, for the following reasons :

1) Silver powder sticks well only to silver or some silver plated metal and in this respect the French powder sticks much better than the Japanese one which needs a much greater pressure (hence a loss of area). We then use a .2 mm layer of French powder which acts as a "solder" between the metal foil and the Japanese powder.

2) Between 25 mK and 18 mK we use a maximum of .6 mm thick sintered French powder to minimise the effect of the increased R_K.

The best configuration of a continuous H.E. for reasons of optimisation of viscosity and conduction effects is the one shown schematically in fig. 4, where the fluid flows along square channels. Here, for a given viscous heating, heat conduction and liquid volume inside the channels are minimum.

The silver is sintered on both sides of a metal foil in the form of slabs, t = 1 to 2 mm thick and w = 1.5 to 3 cm in width.

Mixing Chamber Temperatures as a function of $\frac{\sigma}{n}$ (m^2 mole s^{-1})
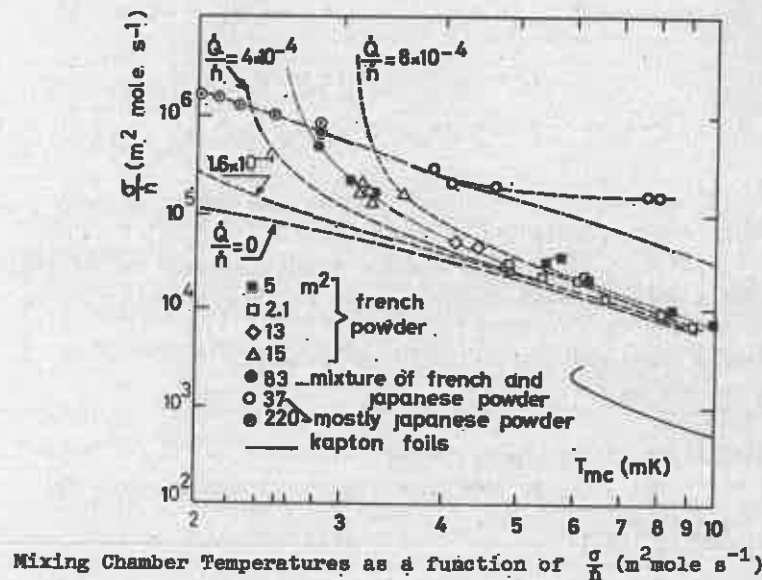
FIGURE 3

Since we have calculated the amount of Ag needed, we can estimate the length of the total exchanger. For example, if t = .15 cm and w = 2.0 cm, then L = 330 cm for the French powder and 110 cm for the Japanese one. We will use for instance 6 elements 25 cm long, which are conveniently made in the form of circular boxes.

Let us now make two assumtions which are somewhat drastic but which work well for the purpose of calculating H.E.

1 - All the heat developed by viscosity and conduction goes to the mixing chamber.

2 - The temperature at any length of a continuous H.E. is the same we would obtain by cutting it there and installing a mixing chamber.

Because of possible instabilities due to partial vaporisation of ^{3}He below the still (ref. 1 and 5) and to decrease the heat conducted backwards in the diluted side, we use a long continuous H.E. made either of CuNi or Brass (see next section for details), which lowers T_c from .7 K to about .70 mK (thus $T_{mc} \sim 25$ mK). From expression 20, with $\dot{n} = 2 \times 10^{-4}$ and $\frac{\dot{Q}}{\dot{n}} = 1.6 \times 10^{-4}$ we get, for $T_{MC} = 25$ mK

$$\sigma_c = \sigma_d = \sigma = .16 \text{ m}^2\text{Ag}$$

$$\text{or} \quad .025 \text{ m}^2\text{CuNi} \ (R_{Km} = 1.25 \times 10^{-2})$$

The 1st H.E. will be calculated in the same way to go from 25 mK to 8 mK with French powder ($R_{Km} = 8 \times 10^{-2}$) : $T_{mc} = 8$ mK, $\sigma = 1.60 - 0.16 \text{ m}^2 = 1.44 \text{ m}^2$ which means about 5 gm or 1 cm^3 of French powder. For a sintered slab 25 cm long and 2.0 cm width, this gives a thickness of .06 cm.

Keeping these points in mind we can calculate the sizes of the channels for the Ag H.E.

Calculation of the channel sizes

If ΔT is small we can take (see fig. 4 for typical geometry) :

$$T = \frac{T_{hot} + T_{cold}}{2}$$

and, as we mentioned earlier

$$\dot{Q}_{visc.} = Z\eta V^2 \dot{n}^2 = \frac{A}{D^4} \quad (21)$$

$$\dot{Q}_{cond.} = \frac{A}{L} \int_{T_1}^{T_2} K_d T = BD^2 \quad (22)$$

$$\dot{Q}_T = \dot{Q}_{visc.} + \dot{Q}_{cond.} = \frac{A}{D^4} + BD^2$$

If we make $\frac{d\dot{Q}_T}{dD} = 0$, then

$$D_{opt} = \sqrt[6]{\frac{2A}{B}} \quad (23)$$

Where $\eta_{3c} = \frac{2 \times 10^{-7}}{T^2}$ N sec K^2/m^2,

Schematic view of Heat Exchanger
FIGURE 4

$$\eta_{3d} = \frac{0.5 \times 10^{-7}}{T^2} \text{ N sec K}^2/\text{m}^2$$

$$K_{3c} \approx K_{3d} = \frac{0.3}{T} \frac{\text{mW}}{\text{m}} \quad \begin{array}{l} T \lesssim 15 \text{ mK for } ^3\text{He}_d \\[8pt] T \lesssim 100 \text{ mK for } ^3\text{He}_c. \end{array}$$

The calculation proceeds as follows :

a) Given $T_{d_{hot}}$ and $T_{d_{cold}}$, $\dot{n}$ and L, calculate D_{opt_d} and $\dot{Q}_{T_d} = (\dot{Q}_{visc.} + \dot{Q}_{cond.})_d$.

b) From (1), using $\frac{\dot{Q}}{\dot{n}} = \frac{\dot{Q}_{T_d} + \dot{Q}_{res.}}{\dot{n}}$ (where $\dot{Q}_{res.}$ is to be estimated) calculate $T_{c_{cold}}$; $T_{c_{hot}}$ is supposed known from the calculation of a previous element or can be taken simply as 2.8 $T_{d_{cold}}$ for the 1st H.E. Remember that we are assuming $T_{d_{cold}} = T_{mc}$ for each point of the H.E. where we would cut it and put a mixing chamber below.

c) Repeat the calculation (a) for the concentrated side to obtain $D_{opt.c}$ and $\dot{Q}_{Tc} = \dot{Q}_{visc.c} + \dot{Q}_{cond.c}$.

For the 2nd exchanger, $T_{c_{hot}}$ and $T_{d_{hot}}$ will be $T_{c_{cold}}$ and $T_{d_{cold}}$ of the 1st one. Determine what will be $T_{d_{cold}}$ for a fraction of the total area calculated. In this case with 5 elements left, $\sigma = \frac{300}{5} = 50$ m^2 or 28 gm. per H.E. Simply add the equivalent area, in Japanese powder of the 1st heat exchanger to the area of the second, that is 6×1.6 m^2 + 50 m^2 = 59.6 m^2. Then from (20) with $\frac{\sigma}{\dot{n}} = \frac{59.6}{2 \times 10^{-4}} = 3 \times 10^5$ s mol^{-1} and a heat leak equal to the one calculated for the 1st H.E. ($\frac{\dot{Q}}{\dot{n}} = \frac{2}{\dot{n}} 1.6 \times 10^{-4}$ J/mol.) obtain $T_{mc} = T_{d_{cold}} = 3.5$ mK. Also calculate $T_{c_{cold}}$, $D_{opt.d}$, $D_{opt.c}$ and $\dot{Q}_{total} = (\dot{Q}_{visc.} + \dot{Q}_{cond.})_c + (\dot{Q}_{visc.} + \dot{Q}_{cond.})_d + \dot{Q}_{res.}$. At some stage it might turn out that $\dot{Q}_{total}/\dot{n}$ is larger than 1.6×10^{-4} J/mol. which means that L is too short for this thermal gradient and the amount of powder should be decreased or L increased. The procedure is identical for the other exchangers, until the minimum temperature is reached.

The total heat leak as a function of the channel size has a rather flat minimum so after finding $D_{opt.}$ this value can be decreased in order to minimize the amount of helium in the channels (see

appendix for typical channel sizes and powder distribution).

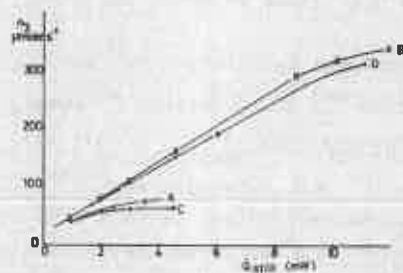### Obtaining high flow rates

As we said earlier, in order to overcome the effects of $Q_{res.}$ and also to have a maximum cooling power at a given T_{MC}, it is necessary to have large flow rates, the largest compatible with the pumping systems. We have observed that for a still designed (and measured) to give high flow rates, it is the high temperature H.E. which limits most the flow.

Fig. 5 shows the ^3He flow rates as a function of power to the still for different impedances of the diluted side, on a dilution refrigerator with a pumping system capable of $\dot{n} = 4 \times 10^{-4}$ moles/sec. (two mercury pumps type Edwards 2M 4A backed by a 12 m^3/n Alcatel pump type 2012 H). Curve A is for a continuous counterflow H.E. of the type described in refs. 1 and 22 and widely used in most of the existing dilution refrigerators. It is made of 2 concentric CuNi tubes 1.50 long respectively 1.0 mm I.D., 1.5 mm O.D. and .2 mm I.D., .4 mm O.D. with an impedance $Z_D = 2 \times 10^8$ cm^3. The maximum $\dot{n}_3$ is limited to $7-8 \times 10^{-5}$ moles/sec. Curve B is for a H.E. of the type we are now using, which leaves to the dilute

^3He flow rate as a function of power to the still

Figure 5

phase a free diameter of 2 mm (see "technical details"). Z_D is typically 3×10^6 cm^{-3} and the ^3He flow rate goes up linearly with Q_{still} until eventually the maximum flow rate possible for the pumps is attained.

Curve C was obtained when we added a short section of 0.5 mm I.D. tube in series with the exchanger B at a place estimated to be at about 300 mK. For this tube $Z = 7 \times 10^7$ cm^{-3}. The decrease in flow rate is drastic.

Curve D shows the effect of a similar value of impedance ($Z = 4 \times 10^7$) but made of 6 m of PVC spaghetti I.D. 1.8 mm placed in series with exchanger B, at 30 mK. There is a very little effect on the flow rate.

These experiments tend to show that it is not the impedance but rather the diameter of the diluted side which is the important parameter on the flow of ^3He quasi-particles, that is, we are facing some critical velocity effect which would have to be more carefully studied in order to derive a quantitative expression for optimizing length and diameter of this H.E. At lower temperatures the tube sizes become large and the ^3He molar volume minimum hence no effect on the flow rate is expected.

Technical details

a) Silver Exchanger Construction

The calculated amount of silver is sintered on both sides of a silver-plated CuNi foil .1 mm thick which is then fitted between two boxes with the profile shown in fig. 4 and welded to them. The boxes are made by pressing or spinning a poorly conducting metal like brass, stainless steel or copper-nickel, typically .3 to .5 mm thick. Inlet and outlet tubes are soldered to each end. This sandwich is then twisted to make a helix and welded.

Japanese powder is first pressed at room temperature to the calculated dimensions, at a density of 45 to 50 % (about 0.5 ton/cm^2 or less), which leads to a self sustaining but fragile sponge. A silver plated CuNi foil is then centered between two silver sponges. A thin uniform layer of French powder between the sponges and the foil acts as a bonding agent. The sandwich is pressed inside a jig with a brass piston using bolts with spring washers to provide pressure during sintering, since the powder shrinks a little. Teflon or vespel pistons, which expand upon heating, can also be used.

The assembly is then taken into an oven consisting of a heated plate and a glass bell jar provided with a viton o-ring (ref. 21). The oven, which was heated to 200°C before introducing the jig is then pumped for 5 min. and 1 Atm. H_2 is introduced after which it is left for about 40 min. The jig is then removed and cooled rapidly with compressed air. Temperature and pressure are quite important for the quality of the bonding and some trials should be done to minimize these parameters in order to obtain a maximum area compatible with good bonding of the sinter to the foil. Exchangers using only (or mostly) French powder or similar can be sintered directly to the foil

although we find easier to first press it.

b) The high temperature continuous H.E.

We make it in the following way : we wrap on a lathe around a
steel rod 2.0 mm in diameter about 5 m of annealed brass tube 1·5 mm
I.D. and 2·0 mm O.D. making a coil roughly 1.5 m long and less than
4 mm O.D. About 1 m of CuNi tube .2mm I.D.,4mm.O.D.is soldered to the
brass tube and the whole is fitted inside a 4mm I.D.,4.5mm O.D. Cu Ni
tube which is then wound in one or two flat spirals. This kind of
exchanger geometry effectively decreases the possibility of gravita-
tional instabilities in the region of temperatures where the concen-
tration of ^{3}He, hence the density of the diluted phase changes most,
that is above 30 mK. The small tube acts as a secondary impedance to
recondense the eventual ^{3}He vapours left on the concentrated side.

The brass tube flattens on being wrapped around the small rod
(which is of course removed afterwards), thus excluding most of the
^{3}He volume, but still leaving a low enough impedance of the order of
10^9 cm^{-3}.

Still and mixing chamber design

In designing an ^{3}He evaporator (still) two points are impor-
tant :

1) It must hold a volume big enough to compensate for changes in he-
lium level due to different volume samples in the M.C. or eventually
some single cycle experiment. In general we like to have a large free
surface for the liquid, typically 50 to 100 cm^2, which gives a rather
flat still thus saving vertical space. It is nevertheless easy to
show that this area can be reduced by at least a factor 10 if needed.

If we pump on liquid mixtures of ^{3}He and ^{4}He the amount of gas
evaporated from a free surface A of liquid is given, using the Lang-
muir formula, by

$$\dot{n}_3 + \dot{n}_4 = 2.48 \ A \left[P_3(T) + P_4(T) - P_o \right] T^{-\frac{1}{2}} \quad \text{mole s}^{-1}$$

where $P_3(T)$ and $P_4(T)$ are the equilibrium pressures of ^{3}He and ^{4}He at
temperature T, and P_o is the pressure of the gas above the bath. With
P in Pascal (1 torr = 133 Pa), A in m^2 and T in Kelvin . Since for
$T < 0.8$ K, $P_3 \gg P_4$, $\dot{n} = 2.48 \ A \left[P_3(T) - P_o \right] T^{-\frac{1}{2}}$ mole s^{-1}.

Thus, for $\dot{n} = 3 \times 10^{-4}$ mole s^{-1} and A = 100 cm^2 = 10^{-2} m^2 at
$T = 0.7$ K , we have

$$P_3(.7) - P_o = 1.45 \times 10^{-2} \text{Pa}$$

since $P_3(.7) = 8.5$ Pa. ,$P_3 \approx P_o$ that is for such an evaporation area
the liquid is practically in equilibrium with its vapour and 10 ti-
mes less area would still be adequate since the pressure drop would
be less than 2% .

2-The amount of ^{4}He recirculated should be kept to a minimum to
avoid instabilities and viscous heating due to superfluid accelera-
tion as well as heat release due to phase separation at temperatures
higher than .1 K,causing a heat load to the tubular H.E.(ref.5).

Also ,superfluid ^{4}He in equilibrium with ^{3}He at T_c will enter
the M.C. in the form of high enthalpy diluted phase thus creating an
extra heat load which can be significant when we are seeking for tem-
peratures of a few milikelvin.As an example,if $\dot{n} = 2.5 \times 10^{-4}$ mole s^{-1}
with 20% ^{4}He and $T_c = 8$ mK ($T_{mc} \approx 3$ mK) .

$$\dot{Q} = 94.5 \times (8 \times 10^{-3}) \times .064 \times 5 \times 10^{-5} = 1.9 \times 10^{-8} \text{W}$$ wich
might be bigger than the background heat leak.We use currently still
temperatures up to 0.85 K .(18% ^{4}He due to its increasing vapour
pressure)when we want maximum cooling power and we observe no insta-
bilities, although the minimum temperatures are all achieved with ^{4}He
concentrations smaller than 5%,that is,still temperatures lower than
.75 K.

The contribution to ^{4}He circulation due to film flow along the
pumping tube is roughly 4×10^{-6} moles sec^{-1} per cm of pumping tube
perimeter. If we use a burning film type of still(ref. 5 and 23)
then we can adopt large pumping tubes (6 to 8 mm i_D) hence negligible
pressure drops thus minimum temperature of the still for a given flow
rate. Nevertheless if the still is to work at flow rates greater than
2×10^{-4} moles/sec., at temperatures where the ^{4}He vapour pressure is
very small compared to ^{3}He,then a polished orifice of 5 or 6 mm
should be adequate since the ^{4}He film flow will be only about 3% of
the total ^{3}He flow.

We observe the best ^{3}He/^{4}He ratios at a given total $\dot{Q}_{still}$ when
the tube which will burn the film is heated with the minimum power
necessary to do so ,and then the rest of the power is applied to a
large area resistance (50 - 100 m^2) inside the bath .The lowest ratios
are obtained when heat is applied to the body of the still.It is thus
convenient that the ^{3}He which is to be cooled to the still temperature
be put in equilibrium directly with the bath by means of a large

area capillary or sintered material so as to decrease as much as possible the temperature jump between the bath and the H.E.,just as with the heating resistance.

The mixing chamber (M.C.) design does not seem to have a great influence on the performance of the dilution refrigerator, as long as the connecting tubes are compatible with those calculated for the last H.E..The ^{3}He inlet tube can stop at the top of the mixing chambers since bringing it down ,besides occupying otherwise useful space inside the M.C. can also lead to heating effects due to phase separation which can eventually exist inside it specially with the large diameters necessary for the lowest temperatures.The outlet tube for the diluted ^{3}He starts from the bottom so as to make the ^{3}He run along all the M.C. thus improving the temperature homogeneity .Fig.6 shows a typical mixing chamber. The construction material is a hot setting epoxy resin (ref. 24) .We actually buy already cured rods(ref. 25) which are then machined and bonded with liquid epoxy (ref. 26).Access into the M.C. is through large conical plugs (total angle about 40°) smeared with Apiezon N or silicon grease.These seals are perfectly reliable and we have made them from about 3 to 60mm in diameter .The joints between epoxy and metal tubing are made by means of Stycast 2850 FT.The temperatures inside the M.C. are automatically stabilised by heating a manganin wire resistance placed in the concentrated side ,close to a sliced speer 100 Ω carbon resistor.(ref. 27)
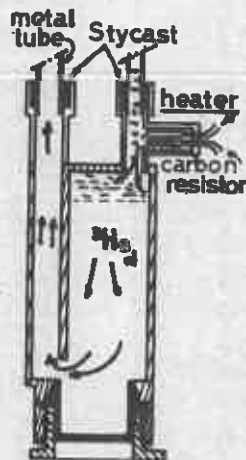
Figure 6
Epoxy mixing chamber

Temperature measurements

The mixing chamber temperatures were measured by means of the A.C. susceptibility of a cylinder of powdered Cerium Magnesium Nitrate ,of diameter equal to height ,from 1K to a few mK ,with a very sensitive mutual indutance bridge (ref.27) .Below 10 mK we have also used the nuclear susceptibility of powdered Pt (ref.28)measured with a continuous wave spectrometer (Q-meter) made with a PAR lock-in amplifier and generator model 124 A and a tuned circuit with the Pt coil resonating at 140 kHz. R.F. voltages up to 1 mV showed no sign of saturation of the signal down to 2 mK .The width of the signal was constant along all the measurements.

The C.M.N. (before turning the field on)was plotted against the height of the signal and surprisingly enough showed no deviation from the Curie law between 3 mK and 11 mK(Fig.7) .Also shown is the cooling power at a flow rate of 2.2 x 10^{-4} moles/sec. The C.M.N. had been previously calibrated against the A, B$_4$ and C transitions of ^{3}He by means of a small Pomeranchuk cell placed inside the M.C. and in equilibrium with it.(ref.29)
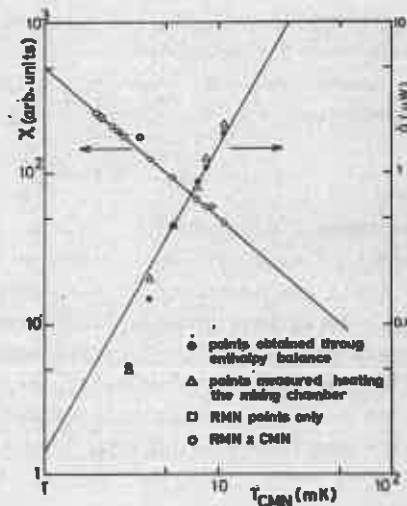
Figure 7
Temperature and cooling power

Conclusion

We have shown that temperatures of the order of 2 mK can be obtained in a continuous way by means of dilution refrigerators using sintered Ag heat exchangers.For the moment we do not think the ultimate performances have been reached and in fact it is possible that using finer powders to increase the area of the last exchangers, or some improved heat exchange mechanism could possibly lead to tempera-

tures around 1 mK ."Direct" heat exchange where the two phases come in direct contact before entering the M.C. thus avoiding liquid to solid Kapitza resistance seems the most attractive possibility .^{4}He circulation and double(^{3}He - ^{4}He) circulation machines (ref.30,31,32) are promising devices using direct exchange but still demand a great deal of work to acheive lower temperatures. Multiple mixing chambers (ref.33,34) when used in connection with a good "classical" dilution refrigerator migth prove to be the best solution although there is the drawback of decreased ^{3}He flow (thus smaller cooling power). Eventually this inconvenience might be overcome by having a last H.E. made of a number of very small holes in the wall separating both phases ,in such a way that the surface tension would inhibit the flow of ^{3}He through the holes.Then all the ^{3}He would flow through the mixing chamber and heat would be conducted from one phase to the other along the column of ^{3}He trapped into the holes according the expression :

$$\dot{Q} = Kn\,\frac{A}{L}\,\ln\frac{T_c}{T_D}$$

(if the thermal conductivity goes as T^{-1}),where A is the area and L the length of each of the n holes and K is the coefficient of thermal conductivity inside the holes.For example ,to go from 2.0 mK to 1.5 mK , .75 kg of Japanese powder area needed on each side of the H.E. at $\dot{n} = 2.5 \times 10^{-4}$ mole s $^{-1}$,to exchange .15 µW.This could be acheived with 1500 holes 50 µ meters in diameter and 10 µm long,if we use for K the coeffeicient of bulk thermal conductivity of ^{3}He .Preliminary experiments are being done in our laboratory to test this possibility.

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Appendix

Calculated values for continuous heat exchangers with

$R_{K_m} = 8 \times 10^{-2}$ m^{2}K^4/W   L = 25 cm and $\dot{n} = 3 \times 10^{-4}$ mole s^{-1}

Nº	Phase	T_{hot}(mk)	T_{cold}(mk)	D_{opt}(mm)	$\dot{Q}_{v+c}$(µW)	σ(m^2)	$\dot{Q}_{exch}$(µW)
1	D	30	9	4.7	.041	1.8	92.5
	C	82.6	24.5	1.7	.008	1.8	
2	D	9	5.5	7.5	.044	3.6	3.28
	C	24.5	14.7	2.8	.009	3.6	
3	D	5.5	3.9	9.3	.0467	6.6	.93
	C	14.6	10.0	3.5	.010	6.6	
4	D	3.9	3.0	10.8	.047	11.5	.39
	C	10.0	7.2	4.1	.011	11.5	
5	D	3.0	2.4	12.0	.050	25.4	.22
	C	7.1	5.1	4.5	.014	25.4	
6	D	2.4	2.0	13.3	.050	84.3	.15
	C	5.0	3.4	5.0	.021	84.3	

Obs.D_{opt} values can be further decreased by about 10-15% for the reasons explained in the text.Furthermore, the actual leaks to the M.C. are somewhat smaller than those calculated,as we could expect.

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Discussion

T. Sugawara: In your experiment $\dot{Q}/\dot{n}_3$ is nearly constant. What is the main reason for this? If this is true, to increase $\dot{n}_3$ seems to have no merit.

G. Frossati: The flow dependent heat leak seems to be a combination of viscous heating and thermal conduction effects. In this sense if a certain temperature is required a sufficiently low $\dot{Q}/\dot{n}$ must be aimed for when calculating the channel sizes. This is always possible by increasing the diameter and the length of an exhanger channel until $\dot{Q}(\dot{n})$ becomes eventually lower than $\dot{Q}$ residual, in which case some work has to be done on vibrational problems, thermal conduction along solids, exchange gas etc.

In minimizing $\dot{Q}(\dot{n})$ by increasing diameter and length we are of course assuming that no convectional instabilities or some other non-trivial phenomena exist. To avoid as much as possible convectional effects the diluted stream is kept horizontal as much as possible, especially above 30 mK, where the concentration of ^{3}He decreases.