

# Pocket Dilution Cooler

T. Prouvé, N. Luchier, L. Duband

CEA/DSM/INAC/SBT

Grenoble , 38054 Cedex 9, France

## ABSTRACT

We have developed a compact, self-contained dilution cooler that can be operated from a 2.5 K or lower heat sink. The system is made of a helium sorption cooler coupled to a closed loop in which the helium mixture flows. The dilution loop operates continuously and is only limited by the autonomy of the sorption unit which needs to be recycled on a regular basis. This first prototype has been designed to provide temperatures below 100 mK for a typical days work. The system can be scaled up within certain limits. Temperatures down to 57 mK and a typical cooling power of 7  $\mu$ W at 100 mK have already been achieved. We expect to reach a nominal useful power of 2.5  $\mu$ W at 50 mK with mixing chamber improvements.

## DILUTION OPERATION AND SELF CONTAINED DESIGN

For a mixture of  $^3\text{He}$  and  $^4\text{He}$  under 850 mK and depending on the  $^3\text{He}$  concentration, there is a phase separation between a “dilute phase” of superfluid  $^4\text{He}$  with saturated  $^3\text{He}$ , and a “concentrated phase” of pure  $^3\text{He}$ .

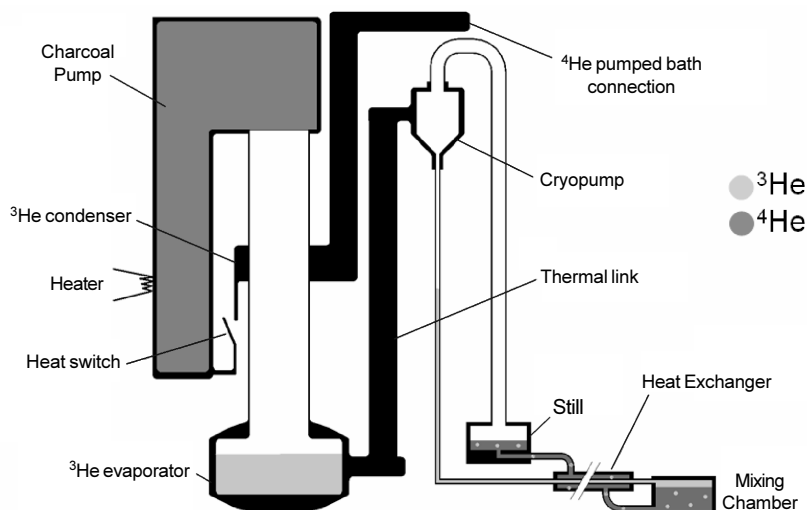
By decreasing the  $^3\text{He}$  concentration of the dilute phase, the equilibrium between the two phases is out of balance and a  $^3\text{He}$  dilution flow occurs from the concentrated phase to the dilute one. Due to the higher enthalpy of dilute  $^3\text{He}$ , dilution of concentrated  $^3\text{He}$  is an endothermic process. By using Radebaugh's<sup>1</sup> works on  $^3\text{He}/^4\text{He}$  mixture properties, Eq. (1) gives the cooling power of the mixing chamber according to the  $^3\text{He}$  dilution flow  $\dot{n}$  (mole/s),  $T_{\text{concentrate}}$  (K), and  $T_{\text{dilute}}$  (K):

$$\dot{Q} = \dot{n} \left( 94.5 T_{\text{dilute}}^2 - 12.5 T_{\text{concentrate}}^2 \right) \quad (1)$$

Since the 1960s, dilution refrigerators have usually been used to achieve temperatures well below 100 mK. With large devices, cooling powers of several milliwatts at 100 mK can be provided, and temperatures as low as 2 mK have been achieved.<sup>2</sup> Nevertheless, those coolers are quite complicated to operate because of their room temperature  $^3\text{He}$  circulation. They are costly, energy intensive, bulky, and not very reliable over time due to  $^3\text{He}$  injection plugging, for instance.

Based on an idea by London et al.<sup>3</sup>, we propose a dilution refrigerator whose  $^3\text{He}$  circulation is provided by a cryopump that condenses the  $^3\text{He}$  vapors coming out of the still. The cryopump operates from a self-contained  $^3\text{He}$  sorption cooler. Figure 1 provides a schematic of our system. In 1972 V.S. Edel'man<sup>4</sup> built such a dilution refrigerator that was operated from a pumped  $^3\text{He}$  bath.

In the mixing chamber, concentrated and dilute phases coexist.  $^3\text{He}$  is driven through a pipe to the still tank by the osmotic pressure gradient. Power applied to the still causes  $^3\text{He}$  to evaporate and also sets the dilution flow. The distillation temperature is driven by the cryopump efficiency,



**Figure 1.**  $^3\text{He}$  sorption cooler and dilution refrigerator mechanism diagram.

which keeps a low pressure at a given flow rate. Liquefied  $^3\text{He}$  at the cryopump temperature goes down in the injection pipe due to gravity. According to Eq. (1), low temperatures can be achieved if the concentrated phase is precooled before entering the mixing chamber. So the injection line features a counterflow heat exchanger cooled by the dilute phase. The dilution operation is gravity driven. That involves some specificity in the design.

Cryopump, still, and mixing chamber locations are not trivial. In order to have an equal pressure between the two phases in the mixing chamber, the  $^3\text{He}$  level of the concentrated phase injection must be rather high. Densities of  $^3\text{He}$  and  $^4\text{He}$  are indeed on the order of  $\frac{1}{2}$ . For a compact design, this requires one to limit as much as possible the distance (height) between the mixing surface and the still.

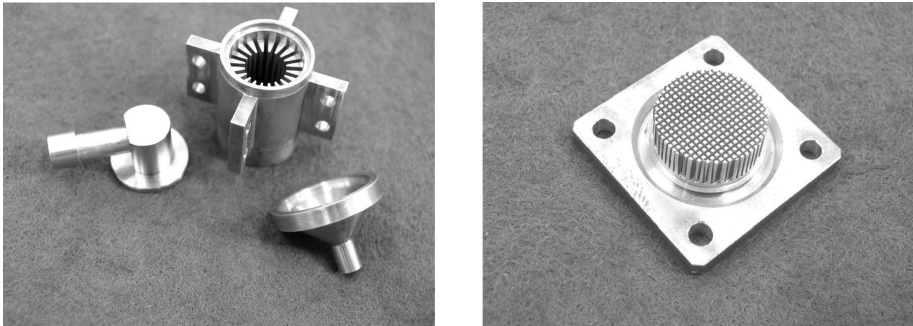
$^3\text{He}$  flow in the injection line produces pressure drops. With standard dilution refrigerators, pressure drops into very long counterflow heat exchangers are not a problem because  $^3\text{He}$  room-temperature pumps can easily produce the input pressure necessary to provide the dilution flow rate. With a cryopump, liquefied  $^3\text{He}$  is just at its saturated vapor pressure, and thus pressure drops are only balanced by an increase of  $^3\text{He}$  level in the injection line (hydrostatic pressure). In a compact design, it is necessary to use a low-impedance exchanger to provide the  $^3\text{He}$  injection.

If no attention is paid to these two special points, the dilution will not operate continuously; instead, all of the  $^3\text{He}$  will be accumulated in the cryopump dead volume, and the mixing surface will be out of the mixing chamber. In our design, the heat exchanger is a flat spiral design that enables the still and the mixing chamber to be placed at approximately the same level.

## DILUTION DESIGN

Our goal is to provide a net cooling power of  $2.5\ \mu\text{W}$  at 50 mK assuming an additional  $0.5\ \mu\text{W}$  heat loss. By using Eq. (1) we have reached a solution with a  $15\ \mu\text{mole/s}$  dilution flow rate. The heat exchanger has a length of 1.5 m with a concentrated side made with CuNi  $1.3 \times 1.7\ \text{mm}$  and dilute side made with CuNi  $2 \times 2.5\ \text{mm}$ .

The average cryopump consumption<sup>1</sup> for a  $15\ \mu\text{mole/s}$  dilution flow rate is almost 500  $\mu\text{W}$ . The pressure into the cryopump is equal to the vapor pressure of liquid condensed to the cold surface. The colder the liquid is, the lower the pressure is, and the better the distillation is. So, according to the condensation power and the Kapitza resistance between cryopump copper's body and liquid  $^3\text{He}$ , we calculated an exchange surface of about  $50\ \text{cm}^2$  to keep the  $\Delta T$  between the cryopump and the condensed liquid below 10 mK. The picture on the left of Figure 2 shows the cryopump with its



**Figure 2.** LEFT: <sup>3</sup>He Cryopump RIGHT: mixing chamber copper heat exchanger.

fins to increase its surface area. The gap between the fins has been designed to avoid liquid accumulation due to capillary forces.

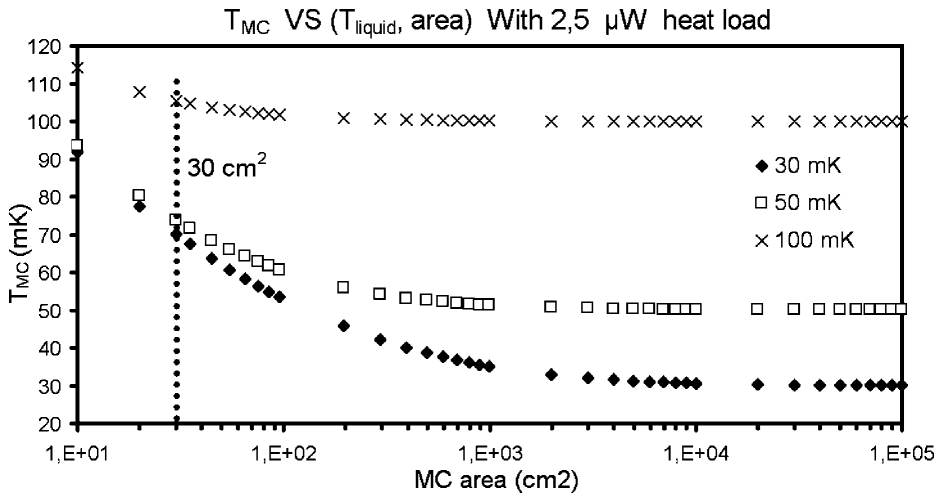
The bottom of the mixing chamber is made of a massive copper heat exchanger. To increase the exchange surface, numerous needlelike fins have been made using an electro-discharge machining technique (see Figure 2). The mixing chamber, with an internal diameter of 1.6 cm and a height of 2 cm, features a 30 cm<sup>2</sup> surface area. This large surface area is enabling for cooldown, but not to achieve very low temperatures and to supply large cooling power.

Figure 3 displays the mixing chamber temperature as a function of exchange surface area and dilute phase temperature. Note that for a 2.5 μW heat-load temperature our present design leads to a mixing chamber at 75 mK when the fluid is at 50 mK. Work is in progress to develop a silver-sintered exchanger to increase the surface to over 1 m<sup>2</sup> for the same mixing chamber volume.

**DEDICATED <sup>3</sup>He SORPTION COOLER**

In order to provide the necessary cooling power at 300 mK for the dilution unit, we have developed a high cooling power <sup>3</sup>He sorption cooler. The dilution refrigerator <sup>3</sup>He flow efficiency depends on two parameters.

First of all, there is the still efficiency. Table 1 gives the percentage of <sup>4</sup>He/<sup>3</sup>He in the circulating vapors. The still temperature should not be over 650 or 700 mK. Circulating <sup>4</sup>He is indeed a power waste and reduces the mixing chamber efficiency. Secondly, there is the <sup>3</sup>He-cooled cryopump efficiency, which determines the operating time for a given dilution flow rate. This is determined by



**Figure 3.** Kapitza boundary resistance effect calculations.

**Table 1.** Comparative table of  $^4\text{He}$  ratio circulated versus still temperature with mixing chamber at 50 mK.

$T_{\text{still}} \text{ (mK)}$	500	600	700	800	900
$^4\text{He}/^3\text{He} \text{ (%)}$	0.08	0.58	2.4	7.2	16.8

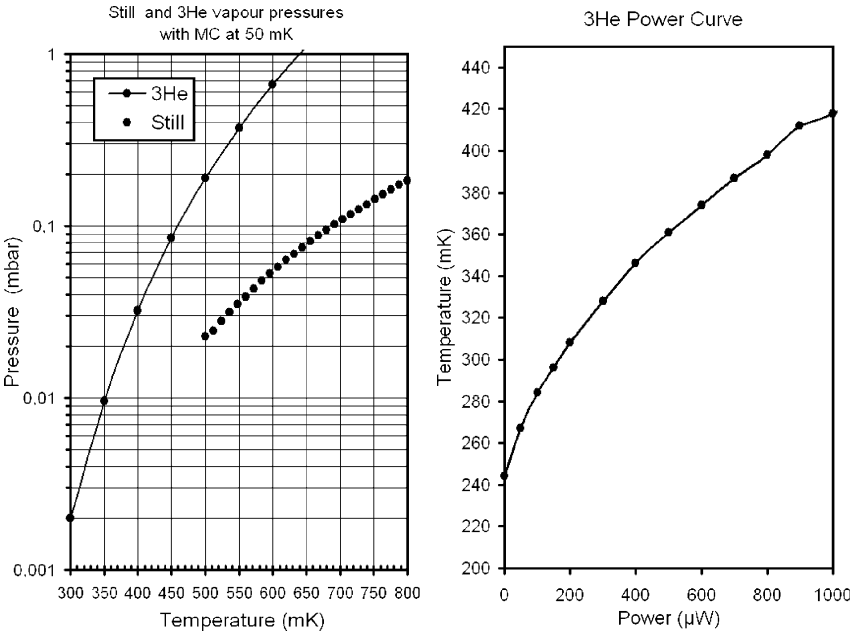
**Table 2.** Comparative table of  $^3\text{He}$  flow between sorption cooler and dilution refrigerator with a mixing chamber at 50 mK.

$T_{\text{still}} \text{ (mK)}$	500	600	700	800
Sorption flow/ dilution flow	1.13	1.17	1.21	1.25

the ratio between the  $^3\text{He}$  vapor condensation rate in the dilution loop, and the sorption cooler  $^3\text{He}$  evaporation rate. The colder the  $^3\text{He}$  vapor is coming out of the still, the lower will be the energetic cost to condense it. However, to have a cold still, the  $^3\text{He}$  sorption cooler has to be even colder. The  $^3\text{He}$  latent heat decreases with temperature, so a lower temperature leads to a reduction in the operating time. Table 2 gives the ratio between these two  $^3\text{He}$  flows for various temperatures. Thus, the warmer the still is, the lower the cryopump efficiency will be. Between 500 and 800 mK, about a 10% increase in operating time can be gained.

So, the  $^3\text{He}$  sorption cooler must be powerful enough at the nominal dilution flow rate to provide the lowest temperature. The left side of Figure 4 shows a diagram of  $^3\text{He}$  and still vapor pressures with a mixing chamber at 50 mK. Note that the sorption cooler should operate below 450 mK to have a still temperature under 650 mK, the temperature required to obtain an efficient  $^3\text{He}$  flow in the dilution refrigerator. The cooler was designed to be able to extract about 500  $\mu\text{W}$  of net heat lift at a temperature below 400 mK. This power corresponds to a 15  $\mu\text{mole/s}$  dilution flow rate.<sup>1</sup>

Large cooling-power sorption coolers have only recently been developed in our laboratory. The first large unit we designed and tested is a 16L STP unit with a 20 mm internal diameter pumping line. This cooler can be operated from a 2.5 K or lower heat sink to condense the  $^3\text{He}$ . With a cold source at 1.5 K, the parasitic heat load is almost 80  $\mu\text{W}$ , but then again, this unit has been designed to operate with very large cooling powers. With a 500  $\mu\text{W}$  applied load, it operates 7 hours and fulfills our initial objective of experiments on a daily basis. The measured cooling power curve is given on the right of Figure 4. The ultimate temperature is near 240 mK, and a temperature of 346 mK is



**Figure 4.** LEFT:  $^3\text{He}$  and still vapor pressures RIGHT: sorption cooler power curve

reached for a cooling power of 500  $\mu$ W. It can be seen on this curve that this sorption cooler can supply temperatures lower than 450 mK for cooling powers up to 1 mW, which enables it to support large  $^3\text{He}$  dilution flow rates.

PRECOOLING AND DILUTION REFRIGERATOR EFFICIENCY

The whole system (sorption cooler + dilution) has been tested on a  $^4\text{He}$  pumped cryostat that provides a temperature of 1.5 K with an operating time of about a day. The  $^4\text{He}$  coldplate is 30 cm in diameter and is surrounded by a first thermal shield at 1.5 K, and then by a second one at 77 K.

Because the sorption cooler is charged with helium and permanently sealed, the internal pressure during most of the cooldown process from ambient temperature is fairly high. As a result convective effects occur in the pumping line and provide an efficient thermal conductance. However, the mixing chamber can not be cooled through the dilution tubing because the thermosiphon effect is not efficient at all. Thus, a thermal switch has been added between the cryopump (thermally linked to the  $^3\text{He}$  cooler through a strong cooper link) and the mixing chamber. During dilution operation, this thermal switch is OFF and leads to a calculated parasitic head load of about 0.4  $\mu$ W.

Our dilution prototype is not designed to contain the  $^3\text{He}/^4\text{He}$  mixture at room temperature, and we are using a 5 L storage tank out of the cryostat. Pressure is about 1.5 bar with 25% of  $^3\text{He}$ . The filling pipe is connected to a 1.5 K cold trap made of stacked copper meshes. Then, the line reaches the dilution on the still using a length of 0.2 mm diameter capillary that limits the superfluid flow, acting just like a diaphragm.

We use Lakeshore thermometers with a  $\text{RuO}_2$  20 mK on the mixing chamber. Heaters on the still and mixing chamber are 402  $\Omega$  glass resistors. A heater and a thermometer are placed on the  $^3\text{He}$  injection capillary between the heat exchanger and the mixing chamber. The heater enables us to measure the gross cooling power in the liquid, and the thermometer enables us to measure the counterflow heat exchanger efficiency.

Figure 5 gives the temperature cooling diagrams. During nitrogen and helium precooling, the sorption pump is kept hot to maintain some internal pressure and consequently convective effect. The

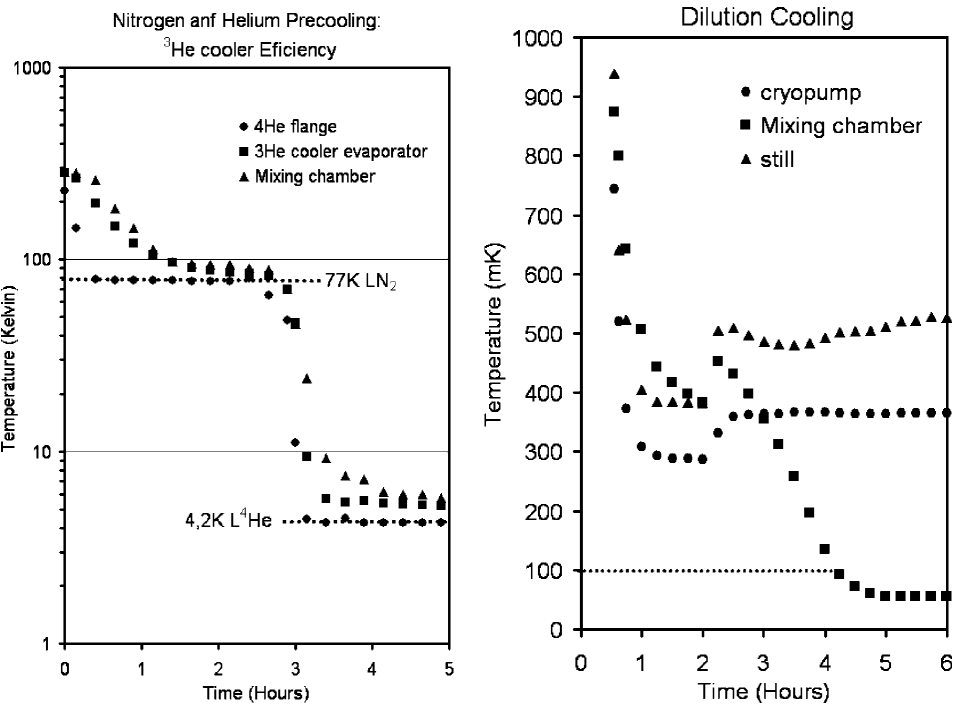


Figure 5. LEFT: LN<sub>2</sub> and  $^4\text{He}$  precooling RIGHT: Dilution cooling 400 mK-55 mK

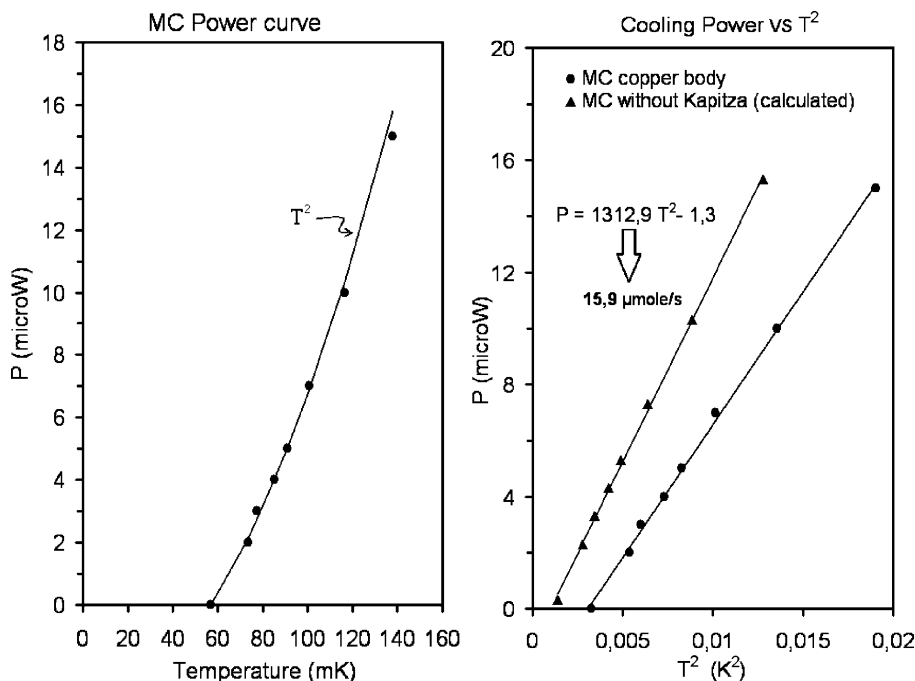


Figure 6. LEFT: mixing chamber power curve RIGHT: Kapitza calculation

heat switch keeps the mixing chamber close to the dilution cryopump temperature. After 5 hours the bath can be pumped.

When the cryostat flange is at 1.5 K, the charcoal pump is cooled. The large sorption pumping line enables an efficient cooling of the dilution refrigerator to 400 mK in 2 hours. At this temperature the thermal switch between the cryopump and the mixing chamber is turned OFF while 400  $\mu\text{W}$  are applied<sup>1</sup> to the still in order to achieve a 15  $\mu\text{mole/s}$  flow rate. The cryopump temperature increases to 380 mK and the still goes to 520 mK.

The strong thermal link between the sorption  $^3\text{He}$  evaporator and the cryopump limits the temperature difference to 20 mK. The  $^3\text{He}$  evaporator at 360 mK takes a heat load of 500  $\mu\text{W}$  (power curve on Figure 4) which gives<sup>1</sup> a flow rate of 15.6  $\mu\text{mole/s}$ . Using the vapor pressures presented in Figure 6, we note that, with a still at 520 mK, the cryopump is very efficient, and the still is well supplied with  $^3\text{He}$ . After 2 hours, the mixing chamber reaches temperatures lower than 100 mK. With a 15  $\mu\text{mole/s}$  flow rate, the operating time is then 3 hours.

REMARK: During the dilution cooldown phase, the mixing chamber initially warms up instead of cooling down. We explain this effect by the fact that during the precooling, the concentrate phase is above the mixture, in the top of the still. As power is applied to the still, the dilution does not start immediately because pure  $^3\text{He}$  in the still has to be evaporated before the distillation of the dilute phase can be initiated.

## POWER CURVE AND EXPECTATIONS

The mixing chamber power curve is given on the left of Figure 6. With 400  $\mu\text{W}$  applied to the still, the mixing chamber achieves an ultimate temperature of almost 57 mK and a cooling power of 7  $\mu\text{W}$  at 100 mK. We notice that the power curve slope is proportional to  $T^2$ , in good agreement with Eq. (1).

We measured the temperature of the  $^3\text{He}$  coming into the mixing chamber. This value was about 90 mK once the ultimate temperature was reached. According to Eq. (1), we should have achieved an ultimate temperature of almost 33 mK without any heat load, and a temperature of 38 mK with 0.5  $\mu\text{W}$  (heat switch, tubing, and wires heat loads) and a 15  $\mu\text{mole/s}$  flow rate.

To analyze the performance, we have represented the power curve as a function of the squared temperature (see Figure 6). We have added on the same diagram a virtual power curve on which the Kapitza boundary resistance between dilute phase and mixing chamber copper body has been subtracted out. The linear fit enables one to calculate a dilution flow rate of 15.9  $\mu\text{mole/s}$  with a heat load of 1.3  $\mu\text{W}$ . These results are in agreement with the data that can be obtained based on the flow rate estimated through the cryopump heat load. The heat load is a bit high, but it could come from the cryostat background heat load.

On this curve a power of almost 2.3  $\mu\text{W}$  at 50 mK can be achieved and corresponds to our expectations. With a 10  $\mu\text{W}$  heat load applied on the  $^3\text{He}$  injection capillary we can compare our Kapitza resistance estimation. We measured 116 mK on the mixing chamber copper body and 97 mK with the power applied to the liquid through the  $^3\text{He}$  injection capillary. These results agree with our estimation to within some mK.

## CONCLUSION

We have developed and tested a compact dilution refrigerator using a cryogenic  $^3\text{He}$  circulation of 15  $\mu\text{mole/s}$  in order to reach a net cooling power of 2.5  $\mu\text{W}$  at 50 mK.

Precooling of the dilution unit is achieved by a  $^3\text{He}$  sorption cooler that can be operated from a 2.5 K or lower heat sink. The 15  $\mu\text{mole/s}$  flow rate is obtained for a precooling temperature of only 520 mK. Higher flow rates can be generated if necessary.

At the nominal flow rate, the ultimate temperature is 57 mK and a power of 7  $\mu\text{W}$  at 100 mK has already been achieved.

As expected, the Kapitza effect limits the ultimate temperature and cooling power. We are currently manufacturing a silver-sintered heat exchanger which will reduce significantly the temperature gap between the dilute phase and the mixing chamber copper body. In this case, as we have shown, powers around 2.3  $\mu\text{W}$  at 50 mK should be achievable.

With a cooling time of 2 hours, the current operating time at low temperature is 3 hours. It could be increased by using a larger  $^3\text{He}$  sorption cooler or could even be made continuous by using two sorption coolers in parallel. Of course this autonomy is also directly linked to the net heat lift of the dilution loop; 7  $\mu\text{W}$  at 100 mK is a significant performance.

The system is gravity driven, and thus cannot be used for a space application. Nevertheless, such compact dilution refrigerator could be used to test detectors on ground applications or as a cooling solution on board balloon flown experiments.

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