

# Concept of a Powerful Cryogen-Free Dilution Refrigerator with Separate 1K Stage

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## ABSTRACT

Helium-3,4 dilution refrigerators (DR) are the workhorses for ultralow temperature scientists. DRs can be continuously operated for unlimited periods of time, and compared to other cooling techniques offer high cooling capacities. Base temperatures are well below 10 mK for well designed DRs. Cryogen-free (CF) DRs precooled by pulse tube refrigerators (PTR) have become standard in recent years.

In a typical CF-DR, the second stage of the PTR runs at temperatures between 2.5 K and 4 K. Its cooling capacity at 4 K is about of 1 W. The next cooling stage is the still of the dilution unit with a typical temperature near 0.7 K. Its cooling capacity is proportional to the He-3 flow and usually well below 20 mW. For many modern applications this is too little to cool and heatsink cold amplifiers, coax lines and electric cables. For these experiments an additional refrigeration stage at an intermediate temperature of about 1 K would be desirable. Several suggestions have been made in the past to address the task.<sup>1</sup> In our CF-DR, we plan to implement a continuous He-4 refrigeration stage with a base temperature of  $\sim 1.2$  K.

The components of the cryostat have been tested separately, but have not been combined in our cryostat so far. The cooling capacity of a newly built 1K-stage has been measured as a function of temperature; it is about 100 mW, whereas the cooling power of the still of the DR is 17 mW. The circulation rates of the DR stage and of the new He-4 stage are about of 1 mmol/s (22 std. cm<sup>3</sup>/s) each. We present details of the planned CF-DR with 1K-stage.

## INTRODUCTION

The DR has become a very basic tool for scientists over the years when temperatures below 0.3 K are needed. Although the cryostat is complex and often has a reputation of being tricky, and although He-3 is expensive and often hard to come by, the dilution of He-3 into He-4 is nevertheless most important as it is the only method to generate ultralow temperatures continuously for extended periods of time. Shortly after PTRs became commercially available, CF-DRs were introduced in 2002.<sup>2,3</sup> Since then, CF-DRs have become very popular with researchers, and quite a number of cryo-engineering firms worldwide offer CF-DRs commercially. Their main advantage compared to conventional DRs is that liquid helium and nitrogen are replaced by a PTR which is operated by just a push-button.

In a CF-DR there are several major cooling steps. The first two cooling steps are given by the two stages of the PTR at temperatures of  $\sim 50$  K (1<sup>st</sup> stage) and 2.5 to 4 K (2<sup>nd</sup> stage). The next heat

reservoir is the still of the DR at a temperature of about 0.7 K, followed by a 100 mK heat sink (heat exchanger of the DR), before the final temperature is reached at the mixing chamber ( $\sim 10$  mK).

Only one flow circuit is necessary for a CF-DR, namely the dilution circuit; this is another simplification compared to conventional DRs where usually a He-4 refrigeration stage is added to the cryostat to condense the back-streaming He-3. This so-called pot typically runs at a temperature near 1.3 K, and usually has spare cooling capacity when needed.

There are low temperature applications where a 1K-stage would be desirable in a CF-DR to cool amplifiers, coaxial cables or current leads; quantum information technology is a contemporary field of research where such powerful refrigerators are required. Usually the still of a DR doesn't offer more than 20 mW of spare cooling capacity; if higher refrigeration capacities are needed, an additional cooling circuit should be available. In this paper, a CF-DR with an additional He-4 refrigerator is described. But unlike in conventional DRs, in our cryostat the 1K-stage is not used to precool and condense the He-3 flow of the dilution refrigerator, and thus 1K-stage and DR can be run independently. The cooling capacity of the 1K-stage is fully available for general cooling purposes. We present experimental results on the cooling power of the 1K-stage.

## HELIUM-4 REFRIGERATION STAGE

### Construction

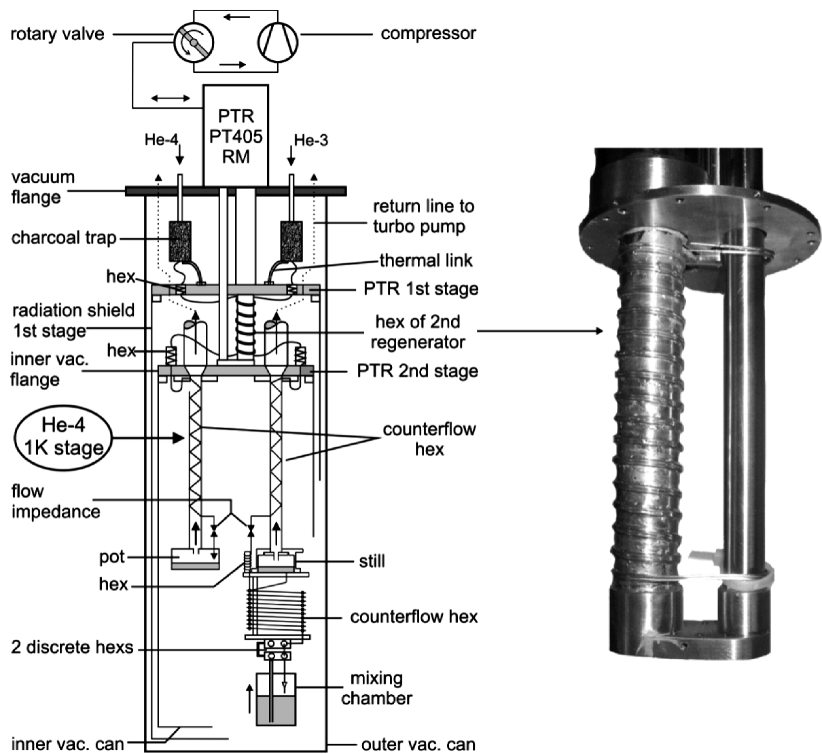
Our cryostat is precooled by a standard PTR (CRYOMECH, model PT405RM)<sup>4</sup> with a refrigeration capacity of 0.5 W at a temperature of the 2<sup>nd</sup> stage of 4 K. Although bigger PTRs with a cooling power of 1 W have become standard in commercial CF-DRs, we decided to go with a small PTR; vibrations are smaller, power consumption is only half, and the purchase price is less with the smaller PTR. On the other hand, cooldown times are longer. But this is irrelevant here as we always precool our cryostat overnight. As long as precooling times remain below 15 hours the small PTR is adequate.

In the projected cryostat (Fig. 1), there are two flow circuits, one for the dilution refrigerator, and one for the 1K-stage. Both circuits have their own charcoal trap to purify the incoming helium gas; the charcoal traps are thermally anchored at the 1<sup>st</sup> stage of the PTR. Then in both circuits the gas streams are further cooled in small heat exchangers at the 1<sup>st</sup> stage of the PTR, before they are cooled in heat exchangers which are soldered to the second regenerator of the PTR.<sup>5-8</sup> The heat exchangers are made from a pair of CuNi-capillaries (1.3 mm i.d.) which were soft soldered in our machine shop to the outside tube of the 2<sup>nd</sup> regenerator (Fig. 1). Depending on the pressure, the He-4 flow may be liquefied in the heat exchanger whereas the He-3 flow of the dilution refrigerator is not.

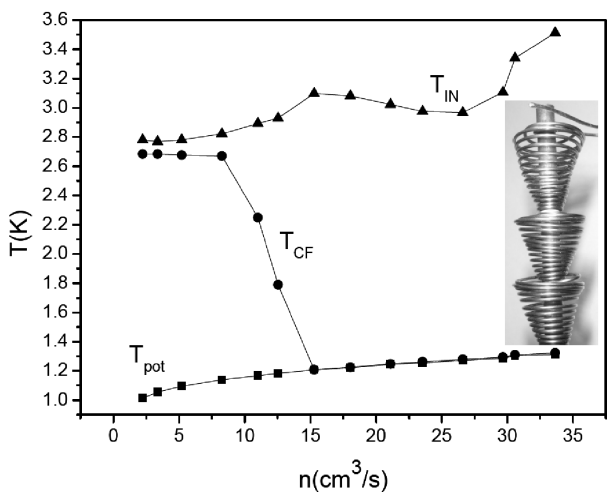
Next, the streams are cooled to the temperature of the 2<sup>nd</sup> stage of the PTR in heat exchangers which are bolted to the flange of the inner vacuum can; the temperature of the He-4 flow after this cooling stage is given in Fig. 2 ( $T_{IN}$ ). From here, the He-3 will flow into the dilution refrigeration unit which has been tested and described before.<sup>9</sup> Its cooling capacity is depicted in the insert of Fig. 3; at a temperature of the mixing chamber of 100 mK, the cooling capacity is 700  $\mu$ W.

The construction of the 1K stage is similar to a JT-stage. It consists of a counterflow heat exchanger, a flow restriction and a vessel ("pot") where the liquid He-4 can accumulate ( $V_{VESSEL} = 53$  cm<sup>3</sup>). The gas is circulated with three rotary pumps (Alcatel 2033H) which are operated in parallel; their combined pumping speed is 100 m<sup>3</sup>/h. One feature we especially like about these pumps is that the outlet pressure can be run up to 0.2 MPa; therefore, a compressor is not needed in the 1K-circuit. After being precooled by the PTR, the He-4 enters the aforementioned counterflow heat exchanger which is situated in the cold gas stream that is evaporated in the vessel. To simplify matters, this heat exchanger is identical to the one in the dilution circuit.

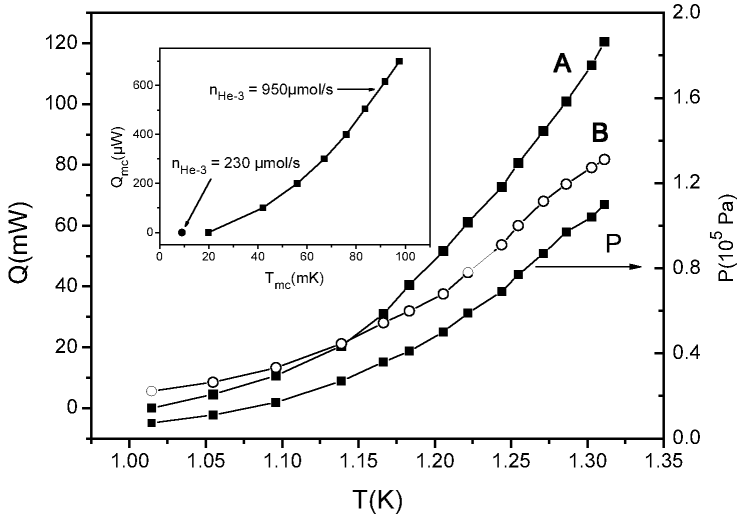
Most recently, we have called attention to DRs where the counterflow heat exchanger of the dilution unit had been omitted;<sup>8,9</sup> this is not advisable here as the temperature of the 2<sup>nd</sup> stage of our PTR is elevated due to the thermal load caused by the He-4 flow of the 1K-stage. If the temperature of the 2<sup>nd</sup> stage of the PTR is above  $\sim 3$  K, a DR without counterflow heat exchanger doesn't work due to thermodynamic reasons. The counterflow heat exchanger of the 1K-stage is made from CuNi



**Figure 1.** Left side: Cross section of the DR. A PTR with remote valve cools a DR and a He-4 cooling circuit. The two independent refrigerators are enclosed by an outer vacuum can, a radiation shield and an inner vacuum can. Right side: Photo of the 2<sup>nd</sup> stage of the PTR with a double heat exchanger soldered to the regenerator.



**Figure 2.** Temperatures of the 1K-stage as a function of the flow rate.  $T_{\text{pot}}$  is the temperature measured at the bottom plate of the vessel,  $T_{\text{CF}}$  is the temperature of the He-4 just before it enters the flow restriction and  $T_{\text{IN}}$  is the He-4 temperature when it enters the counterflow heat exchanger. Insert: Section of the inner element of the counterflow heat exchanger.



**Figure 3.** Refrigeration capacity of the 1K-stage as a function of the temperature of the helium vessel. A: Experimental data, 1K-stage with counterflow heat exchanger. B: Calculated data for a 1K-stage without counterflow heat exchanger. For details see text. P: Condensation pressure of the He-4 flow. Insert: Cooling capacity of the dilution refrigeration stage as a function of the mixing chamber temperature.

capillaries (0.5 mm i.d.) that are bent to 10 little baskets fitting snugly into the pumping pipe (19 mm i.d.) of the 1K-stage (insert of Fig. 2). Contrary to a JT stage, here the He-4 is usually completely liquefied when it leaves the heat exchanger and is expanded in a flow restriction. It is made from a piece of CuNi capillary (100 mm length, 0.1 mm i.d.). Its measured flow impedance is  $z = 3.2 \cdot 10^{16} \text{ m}^{-3}$ . Alternatively, one could use a cold valve with variable flow impedance.

Calibrated thermometers are placed at the gas inlet line and at the outlet of the counterflow heat exchanger to monitor their functioning. A third thermometer is attached to the bottom plate of the pot. All thermometers are RuO chip thermometers.

## Results

In Fig. 3, the refrigeration capacity of the 1K-stage is given (curve “A”). With all three rotary pumps in operation and with the lowest flow rate, the lowest temperature of the pot is close to 1 K. At the highest flow rate of 37 std.  $\text{cm}^3/\text{s}$ , the temperature of the vessel is up to 1.3 K. With the circulation rate, the condensation pressure increases from 0.012 MPa to 0.11 MPa (curve “P”).

Depending on the experimental application, one might consider a simple helium circuit with just a flow restriction after the He-4 is precooled by a PTR. So, for comparison, the calculated cooling capacity<sup>10</sup> of a 1K-stage without the counterflow heat exchanger is included in the figure (curve “B”). For the calculation, the inlet pressure was kept constant at 0.1 MPa, whereas for the helium flow and the inlet temperature the experimental values of curve “A” were used. Thus, the curves are only in part comparable, but it is obvious that, depending on experimental conditions, a circuit without a counterflow heat exchanger would also provide a sizeable refrigeration capacity. In any case, before designing a 1K-stage, its cooling capacity should be derived from the He-4 enthalpy diagram.

In Fig. 2, three temperatures which describe the performance of the 1K-stage, are given as a function of the He-4 flow rate. The temperature of the vessel ( $T_{\text{pot}}$ ) rises with the flow as the inlet pressure of the pumps rises ( $1 \text{ K} < T_{\text{pot}} < 1.3 \text{ K}$ ). The temperature at the inlet of the counterflow heat exchanger  $T_{\text{IN}}$  also rises with increasing flow because it is almost identical to the temperature of the 2<sup>nd</sup> stage of the PTR, and the increasing He-4 flow poses an increasing heat leak to this stage. Finally,  $T_{\text{CF}}$  which is the temperature at the outlet of the counterflow heat exchanger is given.  $T_{\text{CF}}$  drops with increasing flow as the pressure rises. At a flow of 15  $\text{cm}^3/\text{s}$ , the He-4 is liquefied and

precooled to the temperature of the vessel, the coldest temperature possible. At higher flow rates the precooling remains perfect ( $T_{CF} = T_{pot}$ ). From Fig. 2 we conclude that the counterflow heat exchanger is very efficient.

## SUMMARY

In our concept we have explained how a 1K helium refrigerator can be combined with a dilution refrigerator in a cryogen free cryostat. Experimental data show that the helium refrigerator provides temperatures between 1.0 and 1.3 K and cooling capacities of up to 120 mW using a set of rotary pumps with a combined pumping speed of 100 m<sup>3</sup>/h. At the same time a powerful dilution refrigerator can be operated in the cryostat, independent of the 1K-stage. The refrigeration capacity of the dilution unit is 700  $\mu$ W at a temperature of the mixing chamber of 100 mK.

The base temperature of the 1K-stage can be lowered to  $\sim 0.8$  K when turbo pumps are added to the flow circuit; this would correspond to a vapor pressure of 1 Pa at the surface of the liquid He-4 in the pot. In a different application, the 1K-stage may be used with an adiabatic demagnetization refrigerator (ADR) to precool the demag stage. If the price of He-3 remains at excessive levels in the future, it is quite conceivable that ADRs will become more competitive. Precooling temperatures of the demag stage could be lowered by a factor of 2 to 3 compared to precooling with a PTR alone, and holding times and cooling capacities of the ADR improved, accordingly.

## ACKNOWLEDGMENTS

The author thanks VeriCold/Oxford Instruments for support. Discussions with K. Neumaier are gratefully acknowledged.

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