Cryogen-Free Dilution Refrigerator with 1K-Stage

Kurt Uhlig

Walther-Meissner-Institute
Garching 85748, Germany

ABSTRACT

Cryogen-free $^3He/^4He$ dilution refrigerators (DR) precooled by pulse tube cryocoolers (PTC) have become popular with scientists because they are easy to use, reliable and economical. In the cryogen-free DR which was introduced several years ago and, in practically all commercial cryogen-free DRs, there is only one flow circuit, namely the $^3He/^4He$ dilution circuit; a separate condensation stage (formerly referred to as "pot") is not necessary because the condensation of the ^3He flow is taken over by the PTC.

In recent work, a ^4He cooling stage to the DR was added to increase the cooling power of the cryostat at temperatures just above 1 K. Refrigeration powers of up to 100 mW have been reached.

DR (base temperature 8 mK) and 1K-stage are precooled by the same PTC (refrigeration power of 0.5 W at 4 K); DR and 1K-stage have been operated independently of each other, so far. In the work presented here, it is shown how these two cooling circuits can be combined by implementing a heat exchanger (hx) in the 1K-stage where the ^3He stream of the DR is cooled to $T \sim 1$ K. As a result, in addition to the high cooling power of the 1K-stage, a high liquefaction rate in the initial condensation of the $^3He/^4He$ mash is attained (120 std. l/h).

In our presentation, the cryostat assembly is described and cooling powers and low temperature performance of the modified DR are given.

INTRODUCTION

$^3He/^4He$ dilution refrigerators (DR) have become indispensable for experiments below 0.3 K as dilution refrigeration is the only method where temperatures down to 5 mK can be produced continuously and maintained for experimental times of up to a year or possibly longer. In recent years, cryogen-free DRs have more and more displaced traditional cryostats with liquid helium pre-cooling. Ease of operation, cost effectiveness and reliability are outstanding with these refrigerators which are commercially available from several cryo-engineering firms.

Usually cryogen-free DRs have only one flow circuit, the dilution circuit[1]. Therefore, the cooling capacity in the 1K temperature range is given by the cooling power of the still and thus by the relatively small ^3He flow of the DR. For applications where higher refrigeration capacities near 1K are needed to cool amplifiers and electric cables (in our case for experiments on superconducting quantum circuits), a ^4He cooling circuit is added which is similar to a Joule-Thomsom (JT) circuit and is operated at a temperature near 1 K[2,3]. Its refrigeration capacity is almost a factor of 10 higher than the still of the DR. The dilution circuit and the 1K circuit can be run separately, but it is shown how they can be combined advantageously in a cryostat.

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CRYOSTAT DESIGN

A commercial 2-stage PTC (CRYOMECH, PT405-RM)[4] is used to precool the DR and its 1K-stage. The temperature sensor for the 1st stage was a Pt1000 sensor[5], for the 2nd stage a Cernox resistor[6] is used. As reported before, a small PTC (0.5 W at 4 K) is used to minimize vibrations and acoustic noise. As in all cryogen-free DRs, the first stage of the PTC cooled a large radiation shield (see Fig. 1). It was made from copper, no super-insulation was used. The radiation heat load is estimated to be 17 W which caused a temperature rise of the 1st stage from 32 K to an operating temperature of 50 K. The 2nd stage of the PTC reached a base temperature near 2.5 K in our experiments.

In contrast to most commercial DRs, our cryostat has an inner vacuum can (200 mm i.d., made from aluminum) which on cool-downs from room temperature can be filled with exchange gas to cool the dilution unit in the vacuum can to T ~ 8 K. So far, 100 Pa of hydrogen is used; there was no need to pump the hydrogen after cool-downs, it could remain in the vacuum can where it froze. This aluminum can also serves as a radiation shield. Commercial DR manufacturers usually need a separate cooling loop where a compressor circulates helium gas to cool the dilution refrigeration unit from room temperature to T ~ 10 K in their cryostats; this cooling loop, of course, is not required in our design.

To circulate the ^{3}He in the dilution circuit, two (2) turbo pumps (Varian TV-551) and two (2) rotary pumps (Alcatel 2033H) were available and run in parallel; for the 1K-circuit, 2 rotary

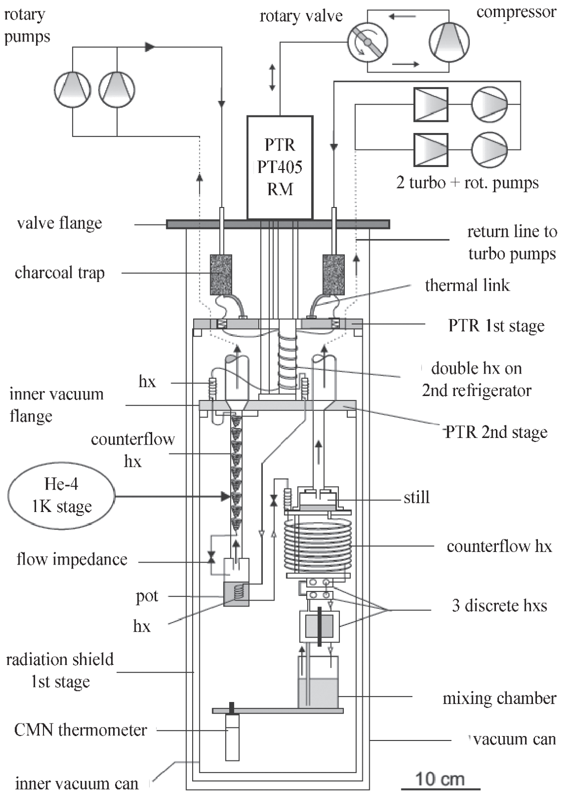

Figure 1. This cross section of the cryostat shows where the 1K-stage and the dilution unit are placed. Note that the condensation line of the DR is run through a hx in the vessel of the 1K-stage. The total length of the DR is 1.2 m. For more details see text.

pumps (Alcatel 2033H) were operated in parallel. The pumps are standard rotary pumps; in commercial DRs usually oil-free pumps are utilized to reduce plugging problems in the DR. In our cryostat, the circulating gas was purified in charcoal traps which were thermally anchored at the 1st stage of the PTC. No nitrogen cooled charcoal traps were necessary.

The 1K-stage is similar to a JT-circuit; the ^{4}He is precooled by the PTC to T ~ 2.5 K, further cooled in a counterflow hx and expanded in a flow restriction (impedance $z = 3.7 * 10^{10}$ cm^{-3}) made from a short piece of capillary. After the expansion, the liquid fraction of the helium can accumulate in a vessel ($V_{vessel} \sim 53$ cm^3) whereas the gas fraction is returned through the just mentioned counter-flow hx to the pumps. A simple gas handling board with a 50 liter storage tank completes the setup of the 1K-stage. An important difference between our 1K-circuit and the classical "pot" of traditional DRs is that the 1K-circuit is a closed flow circuit where a fixed amount of helium circulates and the liquid level in the vessel can be controlled by the amount of helium in the circuit whereas in a helium cryostat the pot takes its helium supply from the helium dewar of the cryostat and is gradually filled up with liquid helium.

The dilution refrigeration unit has been described before[1,7]; in our present setup, there is no counter-flow hx in the pumping line of the still (Fig. 1). Compared to previous work, a third step hx has been added to the dilution unit to reach a lower base temperature. Additionally, a cylindrical capacitor was installed to monitor the height of the liquid level in the still. The condensation line of the DR is run through an hx in the vessel of the 1K-stage so that the ^{3}He after being precooled by the PTC is condensed and further cooled to the temperature of the 1K-stage. The hx is made from a coiled capillary of 0.7 mm i.d. and 0.8 m length (Fig. 1). Alternatively, this capillary was soft soldered to the outside of the helium vessel in a previous setup.

PERFORMANCE

The cooling capacity of the 1K-circuit is depicted in Fig. 2. For thermometry, calibrated RuO resistance thermometers[8] were available and a commercial metal film resistor for heating. The curve on the right was taken with two rotary pumps (Alcatel 2033H) in parallel; this is how the 1K-stage was usually run. With a third pump the middle curve in Fig. 2 was produced, and

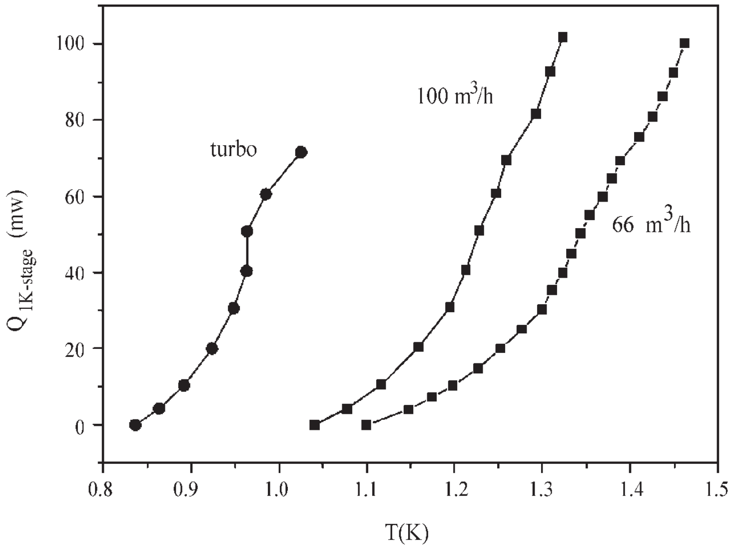

Figure 2. Cooling capacity of the 1K-stage as a function of the temperature of its vessel. Two curves for different rotary pumps are given. In addition the cooling power for operation with a turbo pump is included.

the lowest temperatures near 0.85 K were reached with a turbo pump (Varian TV-551). The ^{4}He condensation pressure of the 1K-circuit was always below 0.1 MPa (Fig. 3).

The condensation rate of the ^{3}He/^{4}He mash with the 1K-stage was usually about 120 std. l/ h with an inlet pressure of the mash of 0.1 MPa. A compressor was not needed for the condensation, neither for the 1K-stage nor for the DR. During condensation, the ^{3}He/^{4}He flow caused a heat load of 22 mW to the 1K-stage which was transferred through the hx in the vessel. A self-regulating increase of the temperature, flow rate and condensation pressure of the 1K-circuit from its lowest levels was noticeable in the condensation process. Later on, with the DR in normal operation, the heat load decreased, e. g., running the DR with a high flow rate of 1 mmol/s (22.4 std. cm^3/s), and brought about a heat leak of 9.4 mW to the 1K-circuit.

Alternatively, the ^{3}He/^{4}He mixture can be condensed without the 1K-stage in operation. Without the 1K-circuit, the condensation rate of the mixture is reduced (≈ 55 std. l/ s) and a pressure of about 0.5 MPa is necessary; a lab compressor is needed. After the condensation, the circulating ^{3}He gas is condensed at the 2nd stage of the PTC at about 2.5 K. At this temperature, the vapor pressure of ^{3}He is 44 kPa; therefore, the condensation pressure has to be at least this high or higher. After being condensed, the liquid ^{3}He (T ~ 2.5 K) flows to the impedance of the DR ($z = 0.39 * 10^{12}$ cm^{-3}). Its molar volume is higher than if it had been condensed with the 1K-stage (T ~ 1.2 K), and therefore the flow rate through the impedance at a given pressure is smaller (Fig. 4).

It was also possible to condense the ^{3}He/^{4}He gas with the 1K-circuit, shut off the 1K-circuit after the condensation process and then run the DR without the 1K-circuit. This feature, in fact, was frequently made use of when the DR was used for thermometry experiments where the 1K-stage was not needed.

In Fig. 4, the cooling capacity of the still is given where the upper curve is for experiments with the 1K-stage in operation and the lower curve for when it was off. With the 1K-circuit running, the enthalpy difference of the incoming ^{3}He between 2.5 K (temperature of the 2nd stage of the PTC) and 1.2 K is dumped to the 1K-stage and is available for cooling at the still. Thus, the cooling power of the still is higher for a fixed throughput with the 1K-stage in use.

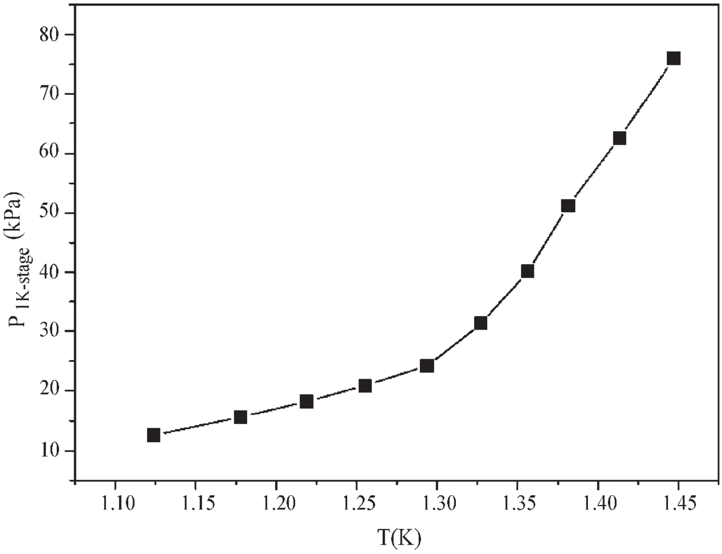

Figure 3. Condensation pressure of the 1K-circuit at the cryostat inlet as a function of the vessel temperature. The condensation pressure was always below atmospheric pressure.

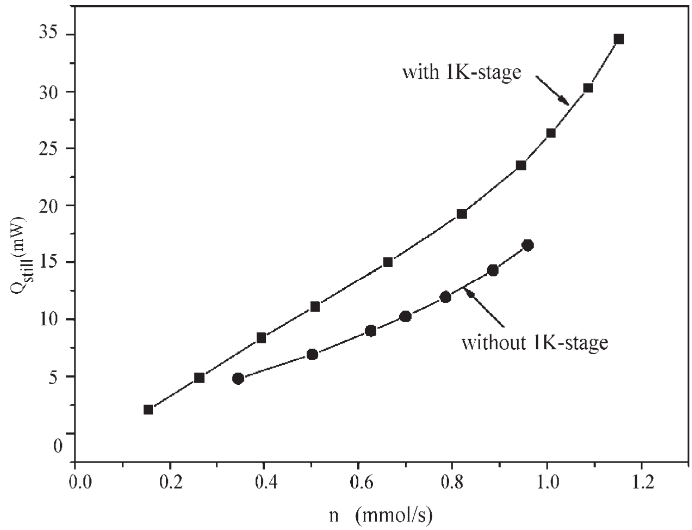

Figure 4. Refrigeration power of the still as a function of the ^{3}He flow. For the upper curve the 1K-circuit was in operation whereas for the lower curve it was off; for the highest data points in both curves the condensation pressure was 0.1 MPa. With the 1K-stage, higher ^{3}He throughputs and cooling capacities were achieved, see text.

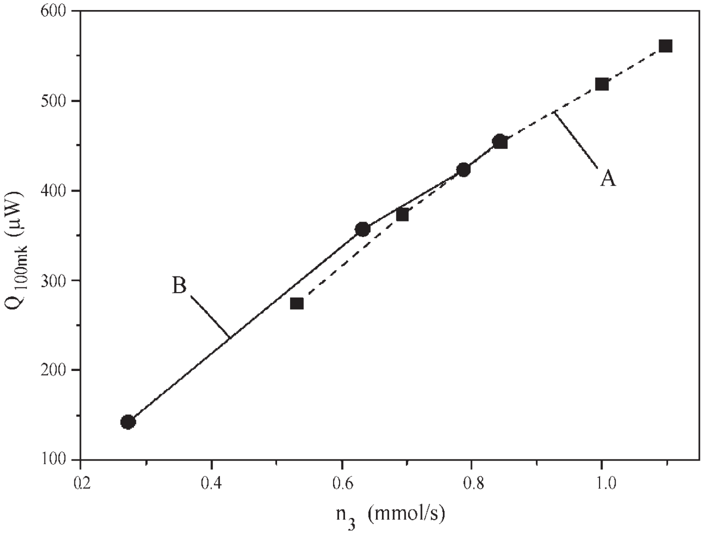

Figure 5. Refrigeration power of the mixing chamber at a fixed temperature of 100 mK. Curve "A" is for operation with the 1K-stage, curve "B" without the 1K-stage.

In Fig. 5, the refrigeration power of the mixing chamber is depicted at a fixed temperature of 100 mK. The maximum refrigeration power is close to 600 μW at a ^{3}He flow of 1.1 mmol/s and an inlet pressure of 0.11 MPa (curve "A"). This is the maximum flow possible with the two turbo pumps used. In earlier work we had reached somewhat higher values with a different set of turbo pumps (2 x Pfeiffer TMH 1601)[1].

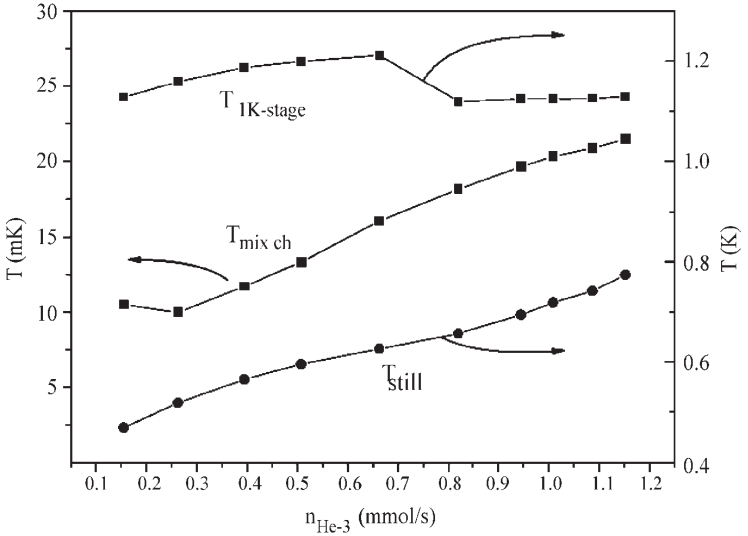

Figure 6. Mixing chamber temperature (left scale), still temperature and temperature of the 1K-circuit (right scale) as a function of the ^{3}He flow of the DR. No heat was applied to the mixing chamber and the 1K-stage.

Curve "B" is the cooling power curve of the DR when the 1K-circuit was off. In our DR, for a condensation pressure of 0.1 MPa the ^{3}He flow was reduced from 1.1 mmol/s to 0.85 mmol/s. The corresponding cooling power was 450 μW; this was the maximum cooling power for the DR when run without the 1K-circuit.

All the temperatures in the DR below the 1K-stage were measured with calibrated RuO sensors. At the mixing chamber, however, a commercial cerium magnesium nitrate (CMN) thermometer was used (Fig. 1)[9]. CMN is a paramagnetic salt whose susceptibility follows a Curie-Weiss-law to very low temperatures of a few mK[10]. A commercial fixed point device which was purchased from the National Bureau of Standards (NIST) was used to calibrate the CMN thermometer.

In Figure 6, the temperatures of the 1K-stage, the still and the mixing chamber are plotted as a function of the ^{3}He throughput. The base temperature of the mixing chamber rose from 10 mK at small ^{3}He flows to about 20 mK at the highest flow rates and the temperature of the still increased from 0.5 K to 0.8 K. The temperature of the vessel of the 1K-circuit remained fairly constant near 1.2 K, independent of the ^{3}He flow in the DR.

SUMMARY

A cryogen-free DR is described which differs in several ways from most commercial cryogen-free DRs. Instead of a precool loop to cool the DR from room temperature to 10 K an inner vacuum can is employed where the dilution unit is precooled in H_2 exchange gas. An additional helium circuit is installed in the cryostat which provides additional refrigeration power of up to 100 mW near 1 K. The dilution circuit is thermally connected to this 1K-circuit by a heat exchanger and thus higher condensation rates are achievable in the initial condensation of the ^{3}He/^{4}He mixture. For mK thermometry a CMN thermometer was used.

ACKNOWLEDGMENT

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