Cryogen-free dilution refrigerators

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Abstract. We review briefly our first cryogen-free dilution refrigerator (CF-DR) which was precooled by a GM cryocooler. We then show how today’s dry DRs with pulse tube precooling have developed. A few examples of commercial DRs are explained and noteworthy features pointed out. Thereby we describe the general advantages of cryogen-free DRs, but also show where improvements are still desirable.

At present, our dry DR has a base temperature of 10 mK and a cooling capacity of 700 µW at a mixing chamber temperature of 100 mK. In our cryostat, in most recent work, an additional refrigeration loop was added to the dilution circuit. This $^4$He circuit has a lowest temperature of about 1 K and a refrigeration capacity of up to 100 mW at temperatures slightly above 1 K; the dilution circuit and the $^4$He circuit can be run separately or together. The purpose of this additional loop is to increase the cooling capacity for experiments where the cooling power of the still of the DR is not sufficient to cool cold amplifiers and cables, e.g. in studies on superconducting quantum circuits or astrophysical applications.

1. Introduction
CF-DRs have become the standard cryocoolers in recent years for many applications in the milli-Kelvin temperature regime. The original motivation for constructing a cryogen-free cryostat came from a very delicate experiment where the varying liquid helium level in the dewar caused offsets in the measuring data. Of course, those were most pronounced after helium transfers. At the time when the first dry DRs were constructed [1, 2], pulse tube cryocoolers were not commercially available, yet. But two-stage Gifford-McMahon (GM) coolers with rare-earth regenerators and base temperatures below 10 K just had become available, and so the first dry DRs were precooled by GM refrigerators.

In figure 1, a cross section of such an apparatus is shown. The GM cooler rested in a trestle which was suspended from the ceiling of the lab, whereas the cryostat had a separate mounting which stood on the floor of the lab. The housing of the GM cooler and the vacuum jacket of the cryostat were joined together by a special soft connection part so that the very heavy vibrations caused by the piston of the GM cooler could not reach the vibration sensitive dilution refrigeration unit. The connection part was made from a rubber bellows tube. Support rings were slid into each convolution of the bellows to keep the tube from contracting while the cryostat was evacuated. The thermal connections between the two stages of the GM cooler and the cryostat were made from copper braided straps, very similar to those in today’s CF-DRs. A big radiation shield was cooled by the 1st stage of the GM refrigerator, and an inner vacuum can with the dilution unit inside was cooled by the 2nd stage. The temperature difference between the 2nd stage of the GM cooler and the
Figure 1. Cross section of one of our GM precooled DRs. The GM-cooler was mounted to the ceiling, whereas the DR stood on the floor of the lab. The two components were joined via a soft connection element so the vibrations of the GM cooler did not reach the dilution unit. For details see text. “A”: hot end of JT circuit, see text.

still was bridged by a JT-stage, consisting of a counterflow heat exchanger and a flow impedance [3].

The base temperature of the GM cooler was 6.2 K. Comparing the enthalpies of the $^3$He inflow and outflow at the hot end of the heat exchanger of the JT circuit yields a value for the cooling capacity $\dot{Q}_{\text{still}}$ of the still:

$$H_3(p_{\text{in}}; T_{\text{in}}) + \dot{Q}_{\text{mc}}/\dot{n}_3 + \dot{Q}_{\text{still}}/\dot{n}_3 = H_3(p_{\text{out}}; T_{\text{out}})$$

(1)

Here $H_3$ is the enthalpy of the $^3$He gas; $p_{\text{in}}, T_{\text{in}}$ and $p_{\text{out}}, T_{\text{out}}$ are pressures and temperatures of the $^3$He at the hot end of the JT circuit (“A” in figure 1). $Q_{\text{mc}}$ is the cooling capacity of the mixing chamber and $\dot{n}_3$ is the $^3$He flow rate. Contributions of the $^4$He circulating with the $^3$He are ignored,
Figure 2. Cross section of a CF-DR with precool circuit and superconducting magnet attached to the 2nd stage of the PTR. Note the wide experimental space in the dilution refrigeration part of the cryostat. “B”: see text.

\( \dot{Q}_m \) is negligible. Inserting reasonable values in equation (1) \( (p_{in} = 10^5 \text{ Pa}, p_{out} = 5 \text{ Pa}, T_{in} = T_{out} = 6.2 \text{ K}, \dot{n}_3 = 0.1 \text{ mmol/s}) \) yields a rather small value of 0.87 mW for \( \dot{Q}_{\text{still}} \), provided the heat exchanger of the JT circuit is working perfectly [4]. Thus the construction of this heat exchanger was crucial, making the task of constructing a dry DR a non-trivial one. The lowest temperature measured with the GM-DR was 7.5 mK.

The situation changed dramatically when pulse tube refrigerators (PTR) became available.

1. First of all, PTRs don’t have a piston inside, and so vibrations are much less of a concern in PTR precooled DRs than in our GM-DRs.
2. The motor/rotary valve unit can be separated from the pulse tube unit (figure 2), and thus vibrations caused by the compressor are decoupled from the pulse tubes. In some commercial PTR-DRs a steel bellows is inserted between PTR and cryostat to decouple the remaining small vibrations of the housing of the PTR, whereas in others the PTR is directly bolted to the cryostat.
3. Another advantageous feature of the PTR is that there are no cold seals inside and thus the pulse tube part of the PTR is practically maintenance free. The compressor does need maintenance, though; absorbers need to be changed after 20,000 hours of running.

4. With better regenerator materials the base temperature of the 2nd stage in closed-cycle cryocoolers has been lowered to about 2.5 K or even lower. This helps tremendously with the construction of dry DRs as will be shown later.

5. Lastly, in PTR precooled DRs the main part of the enthalpy of the back-streaming $^3$He can be dumped in a simple heat exchanger to the $^4$He of the PTR in the 2nd regenerator (“B” in figure 2); thus the heat load caused by the back-streaming $^3$He into the 2nd stage is markedly reduced and the temperature of the 2nd stage becomes almost independent of the $^3$He flow in the DR [5].

![Figure 3](image_url)  
**Figure 3.** Oxford Instruments: Image of a CF-DR with magnet attached at the bottom. The vacuum can and the radiation shield of the first stage of the PTR are detached. Here, PTR and turbo pump are directly mounted to the top of the DR. The refrigeration capacity of the dilution unit is 400 µW at $T_{\text{mix, ch}} = 100$ mK; its base temperature is below 10 mK.
It became clear quickly that the PTR was perfectly suited to construct a much improved version of a CF-DR.

2. Cryogen-free DRs

Today’s commercial CF-DRs are clearly further developed compared to our GM precooled DRs and our first DR with pulse tube precooling [6]. The most significant improvement concerns the experimental space available. The diameter of the mixing chamber plate has about been tripled; this was quite easily possible as there is no longer a dewar with liquid helium around the DR. For DRs with large diameters, it is more practical to dismiss the concept of an inner vacuum can (figure 1) and to replace the latter by a radiation shield. Thus there is only one vacuum space in the cryostat; cold seals and cold electrical feedthroughs can be omitted. On the other hand, for the cooldown from room temperature to a temperature of ~ 10 K, exchange gas precooling is no longer possible. Instead, an additional cooling loop is necessary in the cryostat (figure 2) where a forced flow of helium gas cools all the components in the DR to the base temperature of the PTR [7]. A separate lab compressor may be used to circulate the helium in the loop, or a small fraction of the helium of the PTR is branched off into it (figure 2) [8]. This gas loop has to be evacuated after the precool process to avoid a superfluid film in the loop which would cause a thermal short between the components of the DR at low temperatures. Alternatively, gas gap switches have been successfully used for precooling [9,10]. Precool times vary roughly between 10 and 30 hours, depending on the size of the PTR and the amount of material to be cooled. Our DR can be efficiently precooled overnight.

![Figure 4. Big CF-DR made by Leiden Cryogenics BV. This CF-DR has been built for the CUORE experiment (neutrino-less double-beta decay). Five PTRs with a refrigeration power of 1.5 W each at 4 K can be installed in this cryostat. 10 t of lead have to be cooled to 1K. The weight of the detector is 1.5 t; its intended base temperature is 10 mK.](image-url)
The very low base temperature of ~ 2.5 K of PTRs is a big advantage during the condensation phase of the DR. The liquefaction rate of the $^3$He mixture is high with this good precooling condition. E.g., in our cryostat we reach a gas liquefaction rate of 100 std.l./h. Also, during operation the refrigeration power of the still is high. Using equation (1) and the same pressures as in the example above ($p_{in} = 10^5$ Pa, $p_{out} = 5$ Pa, $T_{in} = T_{out} = 2.6$ K, $\dot{n}_{\lambda} = 0.1$ mmol/s), we now calculate a value of 2.9 mW for the cooling capacity of the still (compared to 0.87 mW of our GM-DR described above which had an inlet temperature of 6.2 K) [4]. We have shown that due to the low base temperature of the PTR the counterflow heat exchanger can be omitted altogether [11], but in this setup the condensation rate of the helium mash was reduced to ~ 50 std.m$^3$/h.

CF-DRs can be combined with dry superconducting magnets (figures 2,3). At the time of this writing, the highest field commercially available with a dry DR is 15 T with a 57 mm bore of the magnet [7]. Top loading and bottom loading ports are available with these DRs, and even the exchange of cold samples (~ 4 K) is technically feasible.

The pressure variations in the pulse tubes are between 0.6 MPa and 1.7 MPa during operation. They bring about an alternation in length of a few µm of the stainless steel tubes which the pulse

Figure 5. BlueFors Cryogenics: CF-DR. This DR has seven line-of-sight access ports between the 300 K flange and the mixing chamber. The mixing chamber plate (bottom plate) has a diameter of 290 mm. The cooling capacity of the DR is 400 µW at $T_{mix, ch} = 100$ mK.
Figure 6. This compact CF-DR of Janis Inc. is tiltable (-20° to 40°). It will be utilized for an astrophysical experiment.

tubes are made of. These alternations in length and vibrations are compensated by copper braided straps or ropes between the two stages of the PTR and the DR (figure 2). The copper ropes also compensate small thermal expansion effects of the cryostat during cooldowns. Vibrations of the cold head are compensated by steel bellows between the housing of the PTR and the vacuum jacket of the DR. Vibration amplitudes measured at the location of the mixing chamber are less than 0.1 µm (Oxford Instruments and BlueFors Cryogenics).

In our DR, the charcoal trap to purify the back-streaming ³He is installed in the DR and thermally connected to the 1st stage of the PTR by copper ropes. In addition, we have a zeolite trap and a filter filled with cotton in the gas handling board. In most commercial dry DRs the charcoal trap is outside of the DR and cooled with liquid N₂.

Figures 3 to 6 show examples of commercial cryogen-free DRs. Comments to these cryostats are given in the figure appendant captions.

3. CF-DR with additional 1K cooling stage.
There are experimental applications at milli-Kelvin temperatures where the cooling capacity of the still of a DR at a temperature ~ 0.7 K is not enough to cool cold amplifiers and coax lines. To enhance the cooling capacity of our CF-DR, we have added a ⁴He circuit to the dilution circuit. A CF-DR with a 1K condenser has been proposed before [12]; in this work it is suggested to use the 1K stage to condense the back-streaming ³He of the DR. In our report the DR and the 1K stage can be operated independently of each other. It would be conceivable to operate the 1K stage with ³He instead of ⁴He if lower temperatures would be required [13].

A cross section of our CF-DR is given in figure 7. Our DR has an inner vacuum can (200 mm i.d.) which can be filled with exchange gas (usually H₂), so a precool loop or gas gap switches are not necessary. The cold stage of the ⁴He refrigerator consists of a counterflow heat exchanger, a flow restriction and a vessel where liquid ⁴He can accumulate. To circulate the ⁴He, we used a rotary pump with a pumping speed of 80 m³/h (Edwards E2M80). The helium flow is cooled and liquefied in heat exchangers which are attached to the two stages and to the second regenerator of
Figure 7. Cross section of our CF-DR with 1K cooler (left side of the sketch). DR and 1K stage can be run simultaneously and separately. Total length of the vacuum can is 1.20 m.

Our PTR (CRYOMECH PT405-RM). A charcoal trap which is connected to the first stage of the PTR purifies the helium flow [14].

The dilution unit has been described in [15]. It is equipped with a counterflow heat exchanger and two discrete heat exchangers. The bottom plate of the mixing chamber has a large silver sponge which provides the thermal contact to the $^3$He inside. The $^3$He gas, after it enters the DR, is cooled by the PTR in three heat exchangers which are identical to the ones of the 1K stage. The heat exchanger at the 2nd stage of the PTR is a double heat exchanger made from a pair of CuNi capillaries which are soft soldered to the outside of the regenerator tube. The temperature gap between the 2nd stage of the PTR and the still is bridged by a counterflow heat exchanger. To circulate the $^3$He, we have used several pump combinations in the course of time. Recently, we had 2 (sometimes 3) Alcatel 2033H rotary pumps in parallel, combined with 2 turbo pumps in parallel (Pfeiffer TMH 1601).
Figure 8. Refrigeration capacity of the 1K stage as a function of its temperature for a cooling loop with (curve A) and without (curve B) counterflow heat exchanger. In the insert the cooling capacity of the mixing chamber is given for a high $^3\text{He}$ circulation rate.

In figure 8 the cooling capacities of the 1K stage and the DR (insert) are given. Both cooling circuits were run simultaneously in this experiment, but they can also be operated separately. Two curves are plotted for the 1K cooler; the upper curve is for a cooling stage with a counterflow heat exchanger in its circuit as shown in figure 7. The lower curve is for a 1K loop where the counterflow heat exchanger has been omitted [16]. Whereas for both cases the base temperature is nearly the same, the cooling capacity of the loop with counterflow heat exchanger is clearly improved. At the highest temperature measured, the cooling capacity of the 1K circuit with counterflow heat exchanger was 100 mW. In all of the experiments reported, the pressure at the inlet of the 1K stage was below 0.1 MPa. The cooling capacity of the DR is 700 µW at a mixing chamber temperature of 100 mK and a high $^3\text{He}$ circulation rate of 0.95 mmol/s. At a lower $^3\text{He}$ flow of approximately 0.2 mmol/s the lowest temperature in the mixing chamber was 10 mK.

4. Summary
The most important advantage of CF-DRs compared to traditional DRs with liquid helium is probably the ease of use during cooldowns and during operation. Cooldowns can be either semi-automatic or computer controlled; no time is wasted with the handling of cryogens. There are no measuring errors due to varying levels of liquid helium. Another advantage is that the experimental space in dry DRs can be increased as much as needed as there are no size limitations due to helium dewars; in a dry DR there is only a vacuum can. CF-DRs can be compactly designed which is an advantage when the ceiling height is limited. The original investment in a PTR is recovered after about one year of operation from the savings on liquid helium. Vibration amplitudes in CF-DRs at
the mixing chamber can easily be kept at a small value of about 0.1 µm, although it seems that we should aim at even lower values for delicate applications like cooling STMs.

In our recent work we have demonstrated experimentally how an additional cooling loop can be added to a CF-DR to increase the cooling power available near 1 K to about 100 mW. In addition, the cooling capacity of the still (up to 30 mW in our DR) is unchanged and also available for experiments. The DR is only slightly affected by the newly added 1K refrigeration stage.

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