Sample Loading and Accelerated Cooling of Cryogen-free Dilution Refrigerators

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Abstract. We present results from system tests of top and bottom loading cryogen-free dilution refrigerators which enable cool-down times from room temperature to mK temperatures in 6 to 8 hours. The loading and unloading processes take only a few minutes to perform and the cooling procedure is fully automated. Sample temperatures of less than 10 mK have been achieved with up to 8 coaxial cables and 25 DC-wires connected to the sample holder. We also present cool-down tests of a beam line dilution refrigerator with a 35 kg mass installed on the mixing chamber. A heat pipe was developed to accelerate the cooling of large experimental payloads and the mass was cooled from room temperature to 30 mK in less than 28 hours.

1. Introduction

In the interest of minimizing heating effect caused by relative motion of mK sample stages with respect to superconducting magnets in integrating systems cooled by a pulse-tube refrigerator (PTR), it is advantageous to mount both the magnet and the dilution refrigerator (DR) rigidly to a single shared platform.

One consequence of such a shared platform is that the entire system including magnet needs to be warmed up when changing the sample. A top or bottom sample loading mechanism is therefore essential in order to reduce turnaround times. Unfortunately it is not trivial to achieve good thermal contact in a cold-assembled joint at mK temperatures. Any heat load to the sample such as that conducted through wires can cause a significant temperature gradient between the mixing chamber and the sample. These problems can be avoided by loading the sample directly into the liquid in the mixing chamber, but such systems require large amounts of $^3$He: a commodity which recently has become exceedingly expensive. A vacuum loaded exchange mechanism is therefore preferable.

However some experiments – such as those requiring large and/or massive sample stages – are not suitable for top or bottom loading and other methods must be employed to reduce the cool-down time of the entire system. Using liquid nitrogen is cheap but rather labor-intensive since it requires the user to be present throughout the pre-cooling process. Utilizing some of the spare cooling capacity of the PTR first stage would be a more convenient alternative if it can be coupled to the low-temperature stages. Heat pipes, with their simple design and superior heat transfer characteristics, appear to be the ideal technology for this application.

2. The sample loading mechanism

Figure 1 shows a sample carrier that can be vacuum loaded into a dilution refrigerator using a demountable loading stick. The carrier makes electrical contact with a docking station via push-fit
connectors for both semi-rigid coaxes and DC wires installed on the refrigerator. Thermal contact is then affected by bolted contact. This concept offers two great advantages. Firstly, installing ancillary equipment (filters, attenuators, circulators, amplifiers, etc) and anchoring wires becomes easy due to the large amount of space available on the refrigerator. Secondly, other than a very small amount of radiation, there is no heat load on the sample carrier itself. This means that virtually no temperature gradient will develop between the mixing chamber and the sample irrespective of the number of wires.

2.1. Design details
The design and operation of this dilution refrigerator has been described elsewhere [1]. Although both a bottom and top loading version of the same concept have been developed by the authors, only the top loading version will be discussed here.

A 60 mm diameter central line-of-sight port through the refrigerator’s platform allows the carrier to reach into the bore of the magnet. The sample carrier, can house up to eight BMA coaxial push-in connectors which are rated up to 20 GHz and one 25-way DC connector. In order to allow electrical contact with the sample when it is loaded in and out of the cryostat, an additional 25-way DC connector is located on the top side of the carrier.

Thermal contact is achieved through the three bolted conical contacts. A hexagonal key located on each of the three drive rods mates with an M5 socket head and allows torques of up to 10 Nm to be applied on each bolt. The threaded section just above the hex key allows the sample carrier to be pulled out after the fasteners have been loosened.

Radiation baffles (copper pads) which can be opened or closed by rotating a dedicated drive rod, are installed at each plate of the refrigerator. To guarantee the elimination of radiation heat loads, each baffle is spring loaded to push it up against the experimental plate.

A vacuum lock with a double ‘O’-ring sliding seal protects the main vacuum chamber during loading and unloading. Both sides of the seal are evacuated using a turbo molecular pump. The gate valve can then be opened and the bottom valve closed. The top valve stays open to allow the pump to remove any small amount of air that leaks through the primary seal as the drive rods slides up or down.

![Figure 1: Sample carrier (left), loading stick with drive rods (top middle) and vacuum lock (right).](image)

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2.2. Operation and results
The sample carrier, assembled inside the vacuum lock, is mounted on top of the cryostat and evacuated for typically 20 min. In the meantime, the mixture is pulled out from the fridge and collected in the room temperature storage tank, a process which is fully software automated. The gate valve is then opened and the baffle stick rotated to allow free passage for the sample carrier.

Unlike most other top loading systems, the sample carrier loads directly from room temperature to the mixing chamber without any pre-cooling. Although the total cool-down time may become slightly longer this way, it saves significantly on manual labor since the cooling process is fully automatic all the way to base temperature.

Figure 2 shows the sample carrier cooling from room temperature to 100 mK, which was the limit of the resistance thermometer calibration during that cool down. The final temperature was 9.8 mK and was measured on the sample flange with nuclear orientation (NO) primary thermometry. At the time of writing, this is the coldest sample temperature ever achieved on a cryogen-free top loader. This system is currently being used for quantum electrical research at the National Physics Laboratory (NPL) in London.

Figure 2: Cooling of the sample carrier, pulse-tube second stage (PT2) and mixing chamber (MC).

3. Gravity-driven heat pipes
Most cryogenic heat pipes circulate a mixture of liquid and high pressure gas through small capillaries to achieve thermal contact between a ‘hot end’ (evaporator) and a ‘cold end’ (condenser) [2, 3]. We have opted for a different concept, shown in figure 3, which is gravity driven rather than pressure driven. This makes the heat pipe particular useful for pre-cooling applications where the pipe needs to ‘switch off’ when the system has cooled to a certain temperature. When the condenser cools below the melting point of the working gas, all liquid solidifies at the upper end leaving the rest of the pipe completely dry. This is important because of the large heat capacity of the solid (in this case N2) which would slow the cooling of the mixing chamber very noticeably at mK temperatures. Dilution refrigerators operate over a wide temperature range so it is imperative the pipe can remain in its off state when the evaporator is heated to temperatures above the melting point of the working fluid.

Heat transfer along the pipe occurs in two different temperature regions: at high temperatures (above the evaporation point) due to natural convection and at the evaporation point due to the latent heat of the liquid. Heat transfer in the liquid region is more or less independent of the charging
pressure provided there is enough gas to maintain an efficient formation of droplets. Heat transfer in the convection region, however, is pressure dependent so the overall ‘on’ conductance will improve with pressure.

Figure 3: Schematics of the heat pipe concept (left) and a cooling curve of the evaporator (right).

3.1. Design details
The internal volume of the pipe is approximately 100 cc and the room temperature expansion volume 1000 cc, both of which are charged to a room temperature pressure of 10 bar (abs) of N₂ gas. To maximize the heat transfer in the liquid region, the condenser has a concave shape which should help the liquid to form droplets near the center of the pipe. Also, the evaporator has a developed surface to aid the metal-to-fluid heat exchange.

To make the most of the cooling power available on the PTR first and second stages, the pipe is connected between both cold heads and the mixing chamber. A radiation baffle (5 mm orifice) is incorporated near the second stage. The pipe is also equipped with two edge-welded bellows to decouple vibrations from the cold heads from the mixing chamber.

The pipe was first tested on its own (not connected to the DR) and without the orifice installed to be able to study the heat pipe effect better. In the final configuration, the evaporator was connected to the mixing chamber together with a 35 kg copper payload.

3.2. Operation and results
The operation of the pipe is self-regulated in the sense that the heat of condensation is large enough to prevent the pulse-tube heads from cooling below the melting point of N₂ until the mixing chamber has cooled below 100 K. This means that no additional heaters or software control is required to run the system. The results of the initial test are shown in figure 3 and clearly demonstrate the two different regions of operation.

The overall cool-down time of the final configuration was 24 h to 10 K, which was approximately 8 h shorter than without the heat pipe installed. The time to condense the mixture and run the DR to base was 4 h which was the same as without heat pipe installed, which proves that the N₂ must have solidified in the upper end of the pipe as intended. This system is currently in use at the neutron beam line facility Institut Laue Langevin (ILL) in Grenoble.

References
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