A Tilttable Single-Shot Miniature Dilution Refrigerator for Astrophysical Applications

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Abstract

We present a 3He/4He dilution refrigerator designed for cooling astronomical mm-wave telescope receivers to around 100 mK. Used in combination with a Gifford-McMahon closed-cycle refrigerator, 4He and 3He sorption-pumped refrigerators, our cryogen-free system is capable of achieving 2 μW cooling power at 87 mK. A receiver attached directly to the telescope optics is required to rotate with respect to the downward direction. This scenario, of variable tilt, has proved difficult for typical dilution refrigerators, but our design has a geometry chosen to allow tilt to 45° and beyond.

Keywords: Dilution, Sorption coolers, 3He-4He mixtures, Instrumentation

1. Introduction

For several decades astronomers have used cryogenic systems to cool receivers operating in the microwave and mm-wave bands. Cooling to around 10 – 20 K, for example, is necessary for state-of-the-art receiver sensitivity at 10 mm wavelength in systems with active amplification. At shorter wavelengths (mm-wave) bolometric detectors are often employed, where a cryogenically-cooled absorber is illuminated with the incoming mm-wave signal and its temperature measured to determine the signal power. Depending upon details of the detection system and local conditions, for example sky emission, this may typically require a cold stage at around 100 mK for optimum sensitivity. Such a temperature may be routinely provided in the lab by 3He/4He dilution. However, when there is a requirement for the receiving system to be mounted upon a telescope that will follow astronomical targets across the sky this can be problematical for traditional implementations of the dilution refrigerator (DR) because of their complex gas-handling system and limitations in tilt angle (although ± 30° operation is claimed by Batey et al. [1] and the Janis ASTRA dilution refrigerator is specified to operate over an asymmetric tilt range). A novel design was developed by Benoit and Pujol [2], that dispensed with the need for a still to draw 3He out of the mixing chamber, instead employing a “vortex pump” effect. Our approach, for astrophysical applications, is to use a single-shot diluter.

Single-shot 3He refrigerators are commonly employed in mm/sub-mm astronomical instruments for several reasons (see for example [3,4]). When used in combination with pre-cooling stages – either 4He or 3He/4He refrigerators – these systems are characterized by having no pumping on the 4He liquid helium bath, excellent duty cycle and durations (typical 24 hours operation versus 2–4 hours recycling). They are also usually very compact with no external gas lines. This last characteristic is welcomed when designing systems destined to be operated remotely on turning and rotating telescopes. The single-shot dilution refrigerator described in this paper is an evolution of the triple-stage sorption refrigerator described in [4], which used a double stage 3He/4He fridge to pre-cool a third 3He single-shot fridge capable of achieving a few μW of cooling power below 300 mK. One of the authors has presented a similar design for continuous circulation using condensation pumping [5]. However, the geometry of the heat exchanger (a helix of concentric tubes) do not permit a large tilt range.

2. Theory

In conventional DR systems, single-shot operation is normally achieved by disabling the 3He return line. Under these conditions, the 3He gas leaving the still is pumped away and not returned to the mixing chamber. Removing the heat input produced by the returning 3He allows the mixing chamber to achieve lower temperatures for a limited time. Very few attempts can be found in the literature describing systems designed to be operated in single-shot mode [6,7].

The cooling power $\dot{Q}$ of a conventional fridge can be calculated by using the first law:

$$\dot{Q} = n_3[H_{3D} - H_{3C}]$$  \hspace{1cm} (1)

where $n_3$ is the molar rate at which 3He is passing from the concentrated to the dilute phase and $H_{3D}$ and $H_{3C}$ are the enthalpies in the diluted and concentrated phases. We may equivalently express this in terms of entropies:

$$\dot{Q} = n_3 T[S_{3D} - S_{3C}]$$  \hspace{1cm} (2)
The enthalpies of the $^3$He in the concentrated and diluted phases can be determined by integration of values for the specific heat.

At low temperatures, where specific heat capacities vary linearly with temperature, eq. 2 leads to a simple approximation (see for example [8] or [9], eq. 7.30):

$$\dot{Q} = 84n_3T^2$$  \hspace{1cm} (3)

Radebaugh [10] quotes a fit to experimental data to within 1\% at temperatures below 40 mK, but by 100 mK this can be seen (his fig. 14b) to have increased to approximately 15\%. Further departure from this model is to be expected at the higher temperatures extant during the initial cool-down of a single-shot DR.

In the case of a continuous-circulation DR these equations assume that the enthalpies / entropies of $^3$He leaving and reentering the mixing chamber cancel (although in the case of an inefficient heat exchanger this will not be true). For the single-shot DR we have no contribution from the enthalpy of returning $^3$He. However, because the mass of fluid in the mixing chamber is balanced by that in the still there is a net flow of $^4$He into the MC as $^3$He is removed. In our model we therefore track both $^4$He and $^3$He levels in both phases as time passes.

In a single-shot DR we must optimize the amount of $^3$He left in the concentrated phase after the minimum operating temperature has been reached. This will determine the run-time and therefore the duty-cycle of the fridge.

This means that it is important for us to model the cooling power of the fridge during the cool-down phase. Therefore we need to evaluate entropies or enthalpies at temperatures above the range of the normal approximation. Also note that we may start with $^3$He concentrations in both phases close to 50\%. The normal approximation of a “concentrated” phase that is pure $^3$He over a very dilute phase is not valid.

A knowledge of specific heat capacities for helium mixtures is enough to evaluate the enthalpy or entropy balance, as we can obtain these values through integration. Experimental specific heat data, as collected by Radebaugh [10] and Kuerten et al. [11] have been taken by Chaudhry [12] to make polynomial fits. We employ these as the input to our model.

Starting with our helium mixture at an initial temperature determined by the $^4$He sorption fridge, given a set molar flow rate of $^3$He (determined by the pumping speed of the sorption pump and conditions in the still) out of the mixture we may calculate the rate of $^3$He transport between phases, and the associated rate of cooling. Onto this is added a load power, comprising a power representative of an attached load, plus any contributing heat leaks (see below). This calculation is performed for each time step, with the molar quantities in each phase tracking the helium transfer. The mixture temperature is adjusted at each step according to $\Delta Q$ and the heat capacity of the mixture according to the polynomial model.

This results in a minimum-available temperature and the fraction of the initial $^3$He remaining once a running temperature of 100 mK is reached, given the starting temperature. These are plotted in figures 1 and 2, for three values of initial $^3$He concentration. The balance of extraction rate and load affects the result; for example slower cooling increases the effect of a given load due to the higher total energy input during the cool-down. A $^3$He transfer rate of $20 \mu$mol.s$^{-1}$ has been employed, with 1 mole of mixture at the start and a simulated total load power of 1$\mu$W applied constantly, which is taken to include all heat leaks. These plots illustrate that a low initial temperature is strongly desirable and that the single-shot scheme very much favours higher concentrations of $^3$He than would be typical for a continuous dilution cooler.

Having established that we need a high $^3$He concentration and a low initial temperature we examined the effect of applying power to the mixing chamber during the cool-down. The resulting plot is given in figure 3. The $^3$He / $^4$He volumes and $^3$He transfer rate were set to values typical for our system.
The test installation for the miniature dilution refrigerator (MDR) is built around an RDK-415D Gifford-McMahon (GM) cryo-cooler, manufactured by Sumitomo Heavy Industries Ltd. This has specified cooling capacities of 35 W at 50 K and 1.5 W at 4.2 K for the first and second stages respectively, when operated from a 50 Hz supply. The GM cooler is fixed to a vacuum can machined from aluminium alloy.

A lower-vibration pulse tube cooler would be preferred. However, our PTC was dedicated to another experiment at the time.

Each GM stage cools a radiation shield fabricated from oxygen-free copper. The first-stage radiation shield is wrapped within multi-layer insulation — 20 layers were used. Typical running temperatures are 23.9 K and 1.95 K for the first and second GM stages respectively. However, these temperatures are elevated by operation of the various cryo pumps. For example, the first stage temperature may increase to approximately 25 K, and the second stage to 5 K during operation of the large 4He cryo pump. A system schematic is presented in figure [4].

The second and third stages of cooling are respectively provided by a 4He and a 3He adsorption cooler. In each case a charcoal-loaded cryo pump (shaded grey in figure [4]) is mounted between the first and second GM stages, and connected by thin-walled stainless-steel tube to an evaporator within the second-stage space. The cryo pumps are fabricated from 50-mm stainless steel tube with a volume of approximately 200 ml, filled with activated charcoal of approximately 2-mm average grain size. Heating the cryo pump causes 4He / 3He to be de-adsorbed; this then condenses in the evaporator. When the heater is switched off and the cryo pump cooled by means of a heat switch to the GM second-stage plate, the charcoal starts to re-adsorb helium vapour, thus pumping on, and consequently cooling, the condensed liquid.

To prevent the formation of a 4He super-fluid film a film breaker is inserted at the bottom of the tube to the 4He evaporator. This consists of an aluminium disc with a pinhole. An undesirable side-effect of the film breaker is that it prevents free-circulation of helium vapour during cool-down, which would leave the evaporator too hot to condense helium. Our approach to solving this problem is to pre-cool the 4He evaporator with a twin-tube linkage between the 3He cryo pump and evaporator. During pre-cooling of the 4He fridge this allows convective circulation of 3He gas between the GM second stage plate and the 4He evaporator connection. Convective circulation of 3He gas from the cold 4He evaporator connection also pre-cools the 3He evaporator, allowing efficient condensation of 3He.

The final temperatures of the 4He and 3He evaporators are approximately 0.96 K and 0.43 K respectively for a typical cooling cycle.

The heat switches used to cool the cryo pumps employ vapour convection. A small cryo pump supplies 4He vapour to a twin-pipe circuit between the large cryo pump and a copper connection (not shown) to the GM second stage. This provides sufficient heat transfer to cool the cryo pump by 40 K (from 55 K to 15 K) in approximately 35 minutes. This switch design will be the subject of a future paper.

The construction of the MDR is similar to that of the adsorption fridges. Its adsorption cryo pump is located between the first and second GM stages, with a thin-walled (0.2 mm wall thickness, 1/4 inch diameter) tubular stainless steel connection through the second stage cold plate to the still. An intermediate cooling connection is made from the tube to the 3He evaporator. Since the still is run at a higher temperature (800 mK) than the connection to the 4He fridge (500 mK) the temperature change of helium rising up the tube is inverted. This is to inhibit any 4He super-fluid film, with its associated large conductance.

The still is constructed from copper, providing thermal connections to a ruthenium oxide thermometer and a heater, supplied with Voltage $V_{\text{still}}$ from the Dewar controller. Its internal volume is 8 ml. Entering the still at the bottom is its stainless steel capillary connection to the mixing chamber. The capillary is long and thin (0.8 mm internal diameter, 0.1 mm wall thickness, by 200 mm length), within the limits imposed by consideration of the large pressures that develop when the system is at ambient temperature, to reduce thermal conduction to the mixing chamber.

The mixing chamber is constructed from copper, with thermal connections for thermometers (germanium resistance) and a heater for simulating a load. Its internal volume is 15 ml. The capillary connection is to the bottom of the mixing chamber. The chamber is filled with oxygen-free copper braid to improve the thermal connection from the chamber walls to the mixture.

At the top of the mixing chamber two further stainless steel tubes (110-mm length, 3.175-mm outside diameter and 0.1-mm wall thickness) are connected to a small copper chamber above, in thermal contact with the 3He link. When the MDR is warm and its cryo pump is heated the tubes of the MDR circuit contain 4He / 3He gas. Working by convection the twin tubes act as a passive heat switch or thermal diode between the mixing chamber and the 3He fridge. The mixing chamber otherwise has very good thermal isolation and will not cool without this.

Once mixture has been condensed within the mixing chamber, pre-cooled to the 3He fridge temperature by the action of
the passive heat switch, the cryo pump is cooled and the still warmed to start the dilution process. The mixing chamber then cools further. When it is colder than the $^3$He fridge convection through the heat switch tubes stops (the switch opens).

The thermal conduction through the metal tubes of the heat switch plus that through the capillary to the still is estimated to be 0.1 $\mu$W. Conduction through the residual gas at the saturated vapour pressure of the condensed helium mixture, is estimated to be below 0.1 $\mu$W. Other contributing heat leaks are neglected, such as conduction through helium between the still and the mixing chamber.

There are no additional radiation shields inside the GM radiation shields, or around the still.

The MDR circuit is charged with 7 dm$^3$ (STP), or 0.3 moles, of $^3$He / $^4$He mixture in a 1:1 ratio. Two 0.4-dm$^3$ expansion volumes are attached to the gas lines for $^3$He and $^3$He / $^4$He mixture reduce the gas pressure when the system is warm.

### 4. Results

Figure 3 shows the temperature of the MDR mixing chamber during several coolings. The different curves show the results for different levels of power applied to the mixing chamber during the cooling, to simulate different load conditions. The cooling that reaches the lowest temperature and stays cold the longest is for zero applied power. The warmest / shortest cooling had 18 $\mu$W applied. Thus we illustrate that we can achieve temperatures below 70 mK and can hold below 100 mK for up to approximately 3 hours. The total time taken to cycle the sorption fridges and the MDR is approximately 7.5 hours. These coolings are with the system oriented level.

Comparison with our model suggests that after the initial cool-down there is $^3$He remaining in the mixing chamber. This leads to the flat portion of the cooling curve. The temperature starts to rise when the $^3$He is exhausted.

Examination of figure 3 suggests that our lowest temperature of approximately 63 mK implies a heat leak power of 9.2 $\mu$W applied to the mixing chamber. This is far higher than our estimates of residual conduction (0.2 $\mu$W) or radiation. Whilst we cannot be certain of the actual $^3$He flow rate, and the composition of the mixture following the pump-down by the cryo-pump, this large discrepancy suggests that there is some other significant source of heating.

Next we examine the effect of still temperature. For our measurements the fridge was operated at constant $T_{\text{still}}$ rather than constant $V_{\text{still}}$. This has the advantage that as the level of mixture in the still and/or the evaporation rate of the mixture varies, there will not be a catastrophic change in still temperature. Under a constant $V_{\text{still}}$ it is possible for temperature fluctuations in the mixture, particularly towards the end of a run when the $^3$He is nearing exhaustion, to cause a run-away increase in $T_{\text{still}}$.

$T_{\text{still}}$ is held constant by a software PID running on the Dewar controller. This increases or decreases $V_{\text{still}}$ accordingly as $T_{\text{still}}$ falls or rises.

As shown in Radebaugh [10] the effective latent heat of evaporation for the helium in the still varies as a function of both the still and mixing chamber temperatures. But for reasons of simplicity we have adopted a constant value of 24 J.mol$^{-1}$ for our model. The model input power to the still is taken from real values of an example cooling. The resulting cooling curve is shown in figure 3 along with the corresponding experimental cooling curve. Also shown is the $^3$He flow rate used in the model.

We see that whilst the model reproduces the general shape of the cooling curve, including the constant-temperature holding period, it predicts a faster initial cool-down and a lower minimum temperature. To some extent this reflects the uncertainty in initial conditions within the mixing chamber (MC) — we do not know how much of the mixture is in the MC when we start running the still, as some will have evaporated following operation of the cryo-pump. However, we find that the model can be made better to agree with the experimental cool-down slope by rescaling the $^3$He by a factor of 0.75, and that adding an extra 4 $\mu$W to the load power improves agreement between $T_{\text{min}}$ values. As noted above, conduction and radiation are not expected to account for heat leaks of this order. One unmodelled contribution would be back-flow of $^4$He from the still, through imperfect heat exchange with the out-flowing $^3$He or by frictional heating. However, we strongly suspect that our passive switch, using mixture from the mixing chamber, might not be

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**Figure 4:** Schematic of the MDR system. Thermometers are indicated by a T and heaters by an H.
Figure 5: Mixing chamber cooling curves for applied powers of 0 (lowest), 1, 2, 3, 4, 5, 6, 8, 10, 12, 14, 16 and 18 µW (highest). For no load the mixing chamber can stay below 100 mK for approximately 3 hours.

Figure 6: A comparison of an experimental cooling curve with 1 µW applied and our modelled results. The modelled curve with 1 µW applied uses the 3He transfer rate shown. A better fit is obtained by reducing the transfer rate to 75% of this and increasing the load to 5 µW.

switching “off” fully once cold, probably due to film flow. We intend to replace this with a separate switch for a future version. Also, we may be suffering from inefficient thermal transfer between the MC walls and the mixture, with the MC temperature measured by a thermometer bolted to the outside face. No allowance is made for Kapitza resistances in our measurements. In future we intend to install a sintered heat exchanger to improve this.

Figure 7 shows load curves for values of $T_{\text{still}}$ from 775 to 900 mK up to $P = 12$ µW, and 18 µW in the case of our adopted $T_{\text{still}} = 800$ mK.

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effect on performance. Therefore in the experiments that follow only a change in tilt in the perpendicular direction, i.e. keeping the still and mixing chamber on the same level, was explored. In each case the cool-down was performed at the tilt recorded, rather than changing the angle once cold.

Figure 9 shows the effect of tilt angle on the minimum temperature and hold time achieved. For this plot the hold time is measured as the time spent below 100 mK. As shown by the solid line there is a general trend of increasing $T_{\text{min}}$ as the fridge is tilted further from level. However, there is also a general trend of increasing hold time.

To verify that the fridge was still capable of cooling to a useful temperature under a load condition at these tilts we ran tests at 30° and 60° tilt with applied loads of 1 $\mu$W and 2 $\mu$W. Resulting load curves may be seen in Figure 10 and hold times in Figure 11. As the system is tilted away from level both the running temperature and hold times increase.

### 5. Conclusions

We have demonstrated a system consisting of a novel $^4$He + $^3$He refrigerator combined with a prototype single-shot miniature dilution refrigerator, capable of cooling to 63 mK with a hold time below 100 mK of nearly 3 hours. We have further demonstrated that this system can operate when tilted to 30° and beyond in one direction.

This is itself useful for applications on telescopes, so long as the geometry allows for the mixing chamber and still to be kept level. To allow more flexibility of orientation, with a tilt in any direction about the MDR, we are developing a new concentric design. This has the still built around the mixing chamber. It will be the subject of a future paper. Enlarging the working volumes and using more mixture should also improve performance. We are hopeful that a significant improvement in cooling power will be realized by replacing the passive pre-cooling heat switch.

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Vitae

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