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A completely self-contained cryogen-free dilution refrigerator, the TritonDR^{TM}

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Oxford Instruments have developed a new dilution refrigerator for ultralow temperatures down to below 35 mK. The TritonDR^{TM} system is a continuous cryogenic cycle dilution refrigerator. The refrigerator is driven by a closed cycle cryocooler and hence requires no liquid cryogens. The system has a dedicated electronic control unit and software that provides full control of operation.

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Introduction

It is difficult to overestimate the importance of ^{4}He liquefaction, first achieved by Kamerling Onnes 100 years ago at the University of Leiden. The availability of liquid ^{4}He has had a huge impact on cryogenics and many other areas of physics. However, there is currently a major trend in cryogenics to remove the need for external liquid cryogens by using closed cycle coolers (such as Gifford–McMahon and pulse tube coolers) to create so-called cryogen-free systems. This trend has been driven by the increasing cost of liquid ^{4}He and the need for skilled staff to handle the liquid cryogens. There is now a high demand for cryogen-free alternatives to liquid cryogen cooled refrigerators from scientists working with ultralow temperatures (ULT). Traditional ULT cryogenic instruments, such as ^{3}He evaporation refrigerators and ^{3}He–^{4}He dilution refrigerators, require liquid ^{4}He for filling cryostats and liquid N_2 for pre-cooling and cold traps.

The first cryogen-free refrigerators arrived at the beginning of the 1990s [1,2]. It was another decade before research scientists and commercial companies began using and producing cryogen-free technology [3–5]. In 2004, Oxford Instruments, first among the commercial companies, responded to the demand for cryogen-free ULT refrigerators by developing the Heliox AC-V^{TM}. This is a single-shot adiabatic expansion ^{3}He refrigerator that is capable of cooling to 280 mK [6]. The Heliox AC-V^{TM} refrigerator is driven by a Cryomech PT403 pulse tube refrigerator (PTR) and uses the adiabatic expansion process to provide additional pre-cooling and liquefaction of compressed ^{3}He gas (at an initial temperature ~ 3 K) as some of the gas is allowed to expand into a cold volume (held at a temperature ~ 50 K). Liquid ^{3}He collects in a copper chamber (the ^{3}He pot) and an activated charcoal adsorption pump is then used to pump the vapour above it and cool the ^{3}He pot to base temperature. The quantity of ^{3}He liquefied during the multi-expansion processes is ~ 50%, which is sufficient to enable the refrigerator to run at base temperature for more than 2 days. When the ^{3}He liquid in the pot has evaporated and the pot starts to warm, the refrigerator can be cooled to base temperature again by completing a regeneration cycle which includes warming the adsorption pump to ~ 30 K before re-cooling the pump to below 5 K.

At this point it is worthwhile addressing the question of why we do not just use simple condensation of ^{3}He gas by cooling below its critical temperature of 3.32 K, since many PTRs (including the PT403) are capable of providing useful cooling powers below 3 K. Analysis shows that because the ^{3}He condensation pressure at 3 K is very high, the overall efficiency of the thermodynamic process at such temperatures is low. A large adsorption pump would be needed to accommodate the volume of ^{3}He gas required to reduce the vapour pressure from the high condensation value to its final value of 10^{-3} mbar. This in turn would significantly increase the heat load to the cold stage of the PTR (PT2) during the adsorption pump regeneration cycle. For example, the thermal cycling during

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regeneration of an adsorption pump with mass ~ 0.5 kg would use the full 4.2 K cooling power of the PT403 cryocooler (which is ~ 250 mW). It would be possible to overcome this problem by using a larger cooling power PTR, but there are significant advantages related to using the PT403 PTR: It only requires a small air cooled or water cooled compressor which runs from single phase mains voltage and the mechanical vibration is less than that of more powerful PTRs. An additional advantage of using adiabatic expansion of ^{3}He gas is the reliability of fridge operation if the PTR performance deteriorates. Using the adiabatic expansion method can provide ^{3}He liquefaction from initial temperatures > 3.3 K.

Dilution refrigerator design

The experience gained during development of the Heliox AC-V™ allowed us to construct a continuous cycle ^{3}He refrigerator, with a cooling power of ~1 mW at T ~ 0.45 K, by employing two single-shot ^{3}He refrigerators operating together so that one refrigerator is running at base temperature whilst the other is regenerating. The continuous ^{3}He refrigerator operates from the cold stage of a Cryomech PT403 PTR. Having built a powerful ^{3}He refrigerator it was a quite natural decision to use it to cool a condenser and realize a dilution refrigerator based on Edelman's method of condensation pumping [7]. The basic principle of condensation pumping is that a cold condensation surface is kept significantly colder than the still and so as ^{3}He evaporates from the still it can condense on this cold surface and be returned to the dilution unit under the action of gravity.

It is important to note that a small number of papers have been published related to cryogenic cycle dilution refrigerators (CCDR) based on condensation pumps. The pioneering work in this area was performed by Edelman in 1972 [7], with later improvements by the same author to reach temperatures below 50 mK [8]. However Edelman's system used a single-shot ^{3}He platform, which prevented continuous fridge operation. The first continuous CCDRs were built at the end of the 1980s by some groups: Group from Erevan [9], Yu. Bunkov in Moscow [10], and V. Sobolev in Kharkov [11,12]. These dilution refrigerators were based on the original idea from [7], but included two ^{3}He refrigerators. These CCDRs had condensation pumps at the lower surfaces of the ^{3}He pots, which allowed continuous pumping of the still. The fridges in Moscow and Kharkov have achieved temperatures below ~12 mK.

One should emphasize that all the above continuous CCDRs used liquid ^{4}He for ^{3}He liquefaction and cooling of the adsorption pumps.

There are some similarities between our continuous cycle CCDR and those described in Refs. 10–12. One of them is that we also use two ^{3}He adsorption refrigerators as shown in Figs. 1 and 2. The crucial difference between our design and designs [10–12], is the ^{3}He heat pipe (HP) that provides thermal switching between the two single-shot ^{3}He refrigerators (Fig. 1). The cold end of the ^{3}He HP cools the condensation pump of the dilution unit, where the ^{3}He evaporated from the still is condensed. The two upper tubes of the HP provide a variable thermal link to the two ^{3}He pots. The HP performs two important functions: Firstly, it provides reasonably stable condensation pump temperatures during cycling of the ^{3}He refrigerators and secondly, it forms a powerful cold radiation shield at T < 0.4 K, which is used to significantly reduce the heat leak transmitted to the mixing chamber by experimental services such as electrical wiring, drive rods and optical fibres etc.

A block diagram of the TritonDR is shown in Fig. 1. This shows the arrangement of the two ^{3}He refrigerators and the ^{3}He HP that thermally links the two ^{3}He pots to the condensation pump. Also shown schematically are the key components of the dilution refrigerator – the still, the continuous heat exchanger and the mixing chamber. Note that gas heat switches provide controllable thermal links between the ^{3}He adsorption pumps and the cold stage of the PTR.

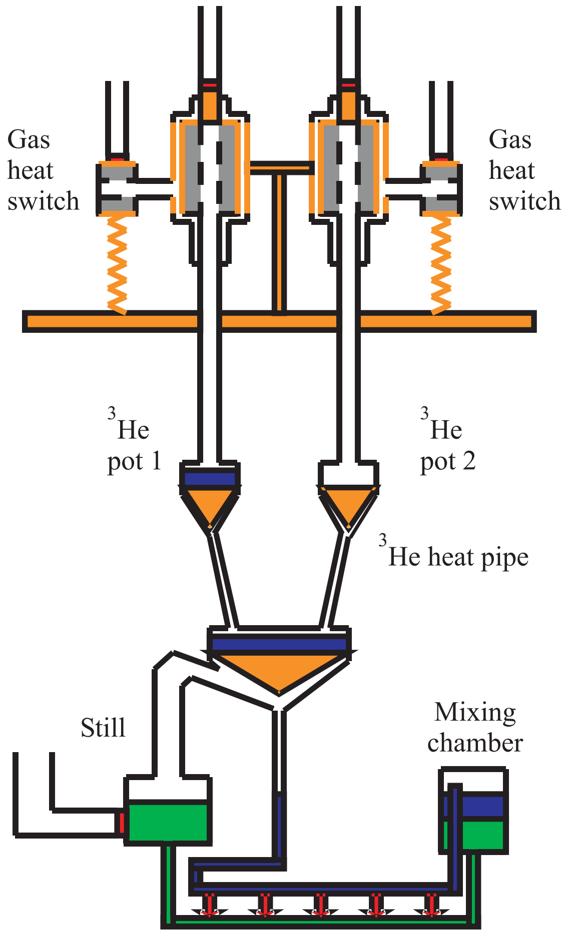

Fig. 1. Principle block diagram of TritonDR.

Fig. 2. Transparent view of TritonDR, showing a conceptual design of the support structure, enabling the cryostat to be tilted and positioned for easy access to the sample platform on the mixing chamber.

The ^{3}He HP plays an important role in the stability of condensation pumping because it reduces the harmful effect of dew evaporation and recondensation during ^{3}He refrigerator swap over for regeneration. Dew, which covers all cold surfaces, is evaporated when the ^{3}He pots warm up and this can partly condense directly on to the still in Refs. 10–12. Condensation of the ^{3}He rich evaporating dew into the still can disturb the osmotic pressure balance between the still and mixing chamber. This effect is undesirable as it leads to instability of the mixing chamber temperature.

Using the ^{3}He HP, the level of temperature stabilization that can be achieved depends on the amount of dew held on the ^{3}He pots, which is a direct function of the surface area of the lower surfaces of the ^{3}He pots, and the total heat capacity of the HP. The effectiveness of the HP can be seen in Fig. 3, which shows the temperature fluctuations of the ^{3}He condensation pump during regeneration mode swapping of the ^{3}He pots. Also shown are the temperature oscillations of the PTR cold stage. The typical temperature fluctuations of the condensation pump are below ~ 25 mK, which is acceptable. The final effect of these fluctuations on the still and mixing chamber temperatures is shown in Fig. 4.

Dilution refrigerator control and operation

The control system for the TritonDR is based on the field programmable gate array (FPGA) architecture [14]. Use of this system means all the control algorithms are defined in software before being compiled to the FPGA or

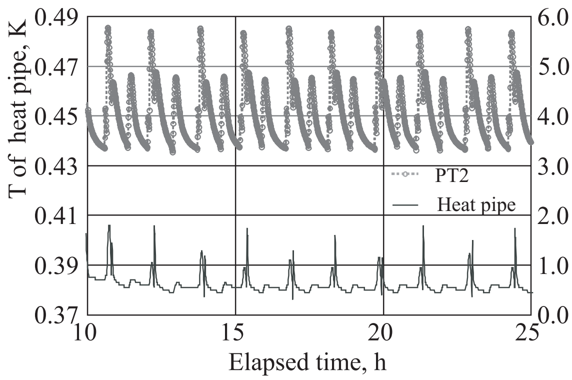

Fig. 3. The temperature of ^{3}He Heat pipe during the cycling of the ^{3}He refrigerators for CCDR continuous operation. Upper curve shows pulse tube disturbance.

real-time control modules. As a result, modifications to the operation of the TritonDR system become a simple software exercise. Using the FPGA control architecture we can finely tune the pump regeneration cycles to ensure that the PT2 temperature remains as cold as required while cooling and regenerating elements of the ^{3}He refrigerator.

Measurement of the resistance thermometers, also called resistance temperature detectors (RTD), fitted to the PTR, ^{3}He refrigerator components and the still uses two FPGA modules and an Oxford Instruments developed reference printed circuit board (PCB). Current is supplied from a 16-bit dc current source to the reference PCB. A fraction of the current is filtered and supplied to the sensor circuit to better than 10 nA resolution. The sensor circuit contains the RTD and precision, low-temperature coefficient resistor. The voltage across the RTD and precision resistor are measured on adjacent channels of a 24-bit voltmeter. The system contains a real-time driven feedback loop which adjusts the RTD excitation current to keep the voltage across the RTD within pre-defined limits and prevent excess power dissipation in the RTD. The level of

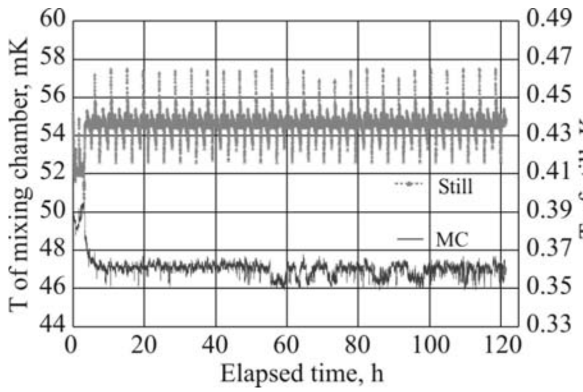

Fig. 4. Triton-DR: Five days of continuous running. The mixing chamber $T \sim 47$ mK with no electronic control.

noise suppression applied can also be software configured with the usual trade-off with response time.

Temperature control of the various ^{3}He refrigerator components is handled by multiple parallel PID loops. Using the FPGA architecture and real-time modules, genuine parallel control loops can be easily configured all using independent control parameters. The heaters are directly driven from 16-bit current source modules. Additional control conditions, as required during adsorption pump regeneration, are written into the control loops, again with real-time variable parameters, so control can be optimized during final system test. For control of the still heater, an additional Oxford Instruments PCB incorporating range switching is used, to increase the operational range of the control loop.

The mixing chamber RTD is a RuO_2 chip driven by Oxford Instruments FemtoPower PCB which keeps the sensor excitation less than 10 fW. This established Oxford Instruments system uses a quasi-dc, 3-step measurement cycle consisting of 2 offset nulling steps followed by a measurement step. The procedure is referenced to the ac supply to ensure the sequence always occurs in an integer number of power line cycles enabling pick-up to be minimized. All sensor cables pass through a pi-filter to suppress higher frequency pick-up. For the TritonDR system the output voltage of the FemtoPower PCB is amplified to supply half of a 24-bit voltmeter module. The system gives 0.1 mK temperature resolution with an absolute electrical measurement accuracy of 1% in temperature.

The mixing chamber heater power is supplied from a digital i/o module via another Oxford Instruments developed PCB. Digital lines from the FPGA are used to switch between heater ranges. This gives 5 ranges, each a decade apart, from 0 to 2 μW, up to 0 to 20 mW. Scaling within each range is provided by a pulse-width modulated (PWM) digital line referenced to the 40 MHz real-time clock and feeding an integrator circuit on the Oxford Instruments PCB. Control of the mixing chamber heater power can be either direct or by a real-time based PID loop driven by the mixing chamber temperature. A final digital line switches a relay to completely isolate the mixing chamber heater when it is switched off to ensure there is no leakage current erroneously heating the mixing chamber when base temperature is required from the fridge.

System performance

The first TritonDR system has been designed to achieve temperatures below 50 mK. Hence the dilution unit is only fitted with a simple tube-in-tube continuous heat exchanger. We have now successfully built and tested a number of 50 mK fridges. In Fig. 4, one can see five days of continuous operation for one of these units without any human intervention. The upper curve shows still temperature and the lower curve is the mixing chamber (MC) temperatures. The MC base temperature was below 47 mK and the temperature stability was better than ± 0.5 mK without electronic temperature control. On a later unit we increased the surface area of the continuous heat exchanger and achieved temperatures below 35 mK with a natural stability better than 0.5 mK (Fig. 5).

Cooling power of TritonDR

It is well known that the cooling power of the mixing chamber is a function of the ^{3}He circulation rate and the efficiency of the heat exchangers. The circulation rate is determined by the still heater power. However it is important to maintain a low still temperature in order to minimize the circulation of ^{4}He. The temperature of the still depends on the efficiency of the condensation pump, which is related to the pump's surface area and the quality of the surface. The heat load due to condensation of ^{3}He from the still has to be transmitted back to the ^{3}He refrigerator via the HP. Hence the temperature of the condensation pump will not only depend on the cooling power of the ^{3}He refrigerator but also on the Kapitza resistance between the condensation pump, heat pipe and the two ^{3}He pots. All heat loads associated with running the dilution refrigerator must eventually be absorbed by the cold stage of the PTR. Analysis shows that the maximum circulation rate with a PT403 can be up to $\sim 3\cdot10^{-5}$ mole/s, which limits the cooling power to ~ 20 μW at 100 mK for the PT403. This value of cooling power may look quite modest, but it is typical of many dilution refrigerators produced at the end of the 1960s [15], which were used in many areas of physics research. It is important to note that the MC cooling power required for typical experimental services on the TritonDR is significantly less than that for traditional dilution refrigerators. This is because of the presence of the powerful shield at $T \sim 0.35$ K provided by the HP, which is used for thermal dumping of diagnostic and experimental services. Consequently, even at modest ^{3}He circulation rates, it is possible to provide stable low

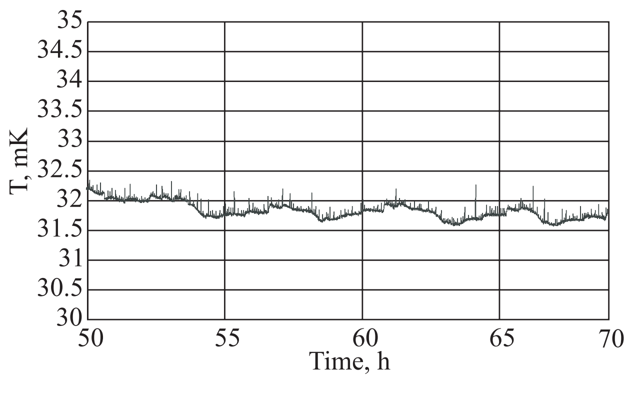

Fig. 5. The mixing chamber base temperature intrinsic stability for TritonDR with the improved continuous heat exchanger.

temperature MC operation with reasonable customer experimental wiring as shown below.

The general equation for the heat load to the MC associated with the thermal conductivity of a solid of uniform cross sectional area (S) is

$$\dot{Q} = \frac{S}{L} \int_{T_{MC}}^{T_d} \kappa(T) dT, \qquad (2)$$

where T_d is the temperature of the thermal dumping point before the MC, L is the length and $\kappa(T)$ is the thermal conductivity coefficient of the solid.

The heat load through metals at low temperatures is determined by conduction electrons and so

$$\dot{Q} \propto \frac{S}{L} T_d^2, \qquad T_{MC} << T_d, \qquad (3)$$

and for insulators, where the conductivity is determined by phonons

$$\dot{Q} \propto \frac{S}{L} T_d^4, \quad T_{MC} << T_d, \quad T_d < \frac{\theta_{\text{Debye}}}{10}. \qquad (4)$$

The typical thermal dumping points on a traditional dilution refrigerator are the 1 K pot and the still, which has typical temperature ~ 0.8 K. The TritonDR has a dumping point on the PTR cold stage (~ 3 K), which is an analogue of the 1 K Pot. However, it also has a powerful ^{3}He HP platform at ~ 0.35 K, which is significantly colder than the still (Fig. 3). In this case it is much better to dump services on the ^{3}He HP rather than the still.

The benefit of this dumping procedure can be seen from the ratio of the heat load to the mixing chamber for services fitted to a traditional fridge compare to the same services fitted to our CCDR, which is given by

$$\frac{\dot{Q}_{\text{Tr}}}{\dot{Q}_{CCDR}} \approx \left[\frac{0.8\,\text{K}}{0.35\,\text{K}}\right]^{\geq 2}, \qquad (5)$$

which for metals is > 5 and for insulators is > 25. This is an important effect as it reduces the need for high MC cooling powers. This result is supported by the experimental data in Fig. 5, which shows the mixing chamber temperature for a TritonDR which has 3 carbon-fibre support legs from the HP along with standard diagnostic wiring plus additional customer wiring consisting of 48 constantan wires of 0.1 mm diameter. One can see that the MC cools to < 35 mK.

Conclusions

1. Oxford Instruments have developed a novel dilution refrigerator that requires no liquid cryogens. The fridge can cool to < 35 mK using only a simple continuous heat exchanger.

Fig. 6. TritonDR and electronic control box. The core team members are shown to give an idea of the scale of the system.

2. The system has no cryogenic valves, no external helium pumps and uses only a single phase air or water cooled compressor to drive the PTR.

3. The system is self-contained and fully automated using a dedicated electronic control system and software.

4. To avoid a long description of the overall size of the TritonDR we present a photograph of the system along with some of the authors (Fig. 6).

All authors are employees of Oxford Instruments NanoScience.

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