Top loading cryogen free cryostat for low temperature sample environment.

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Today almost a quarter of all neutron scattering experiments performed at neutron scattering facilities require sample temperatures below 2 K. However, a global shortage of helium gas can seriously jeopardise low temperature experimental programmes at neutron scattering laboratories. Luckily the progress in cryo-cooler technology offers a new generation of cryogenic systems with significantly reduced consumption and in some cases nearly a complete elimination of cryogens. Here we discuss design and test results of a new cryogen free top-loading cryostat developed ~~by the~~ through an ISIS and Oxford Instruments collaborative project. The cryostat provides neutron scattering sample environment in the temperature range 1.4 – 300 K. High cooling power (0.23 W at temperature less than 2 K) achieved at the cryostat's variable temperature insert heat exchanger allows operation of a standard dilution refrigerator insert in a continuous regime. From a user perspective, the system offers operating parameters very similar to those of an Orange cryostat but without the complication of cryogens.

Neutron scattering, sample environment, dilution refrigerators

ULT: ultra low temperature; SE: sample environment; PTR: pulse tube refrigerator;

VTI: variable temperature insert.

Introduction

According to ISIS[1] beam-line proposal statistics almost 25% of all experiments require a sample temperature below 2K [1]. Such sample environment is usually provided by conventional top-loading cryostats. The temperature range can be extended below 1K by inserting a dilution refrigerator or ^{3}He evaporation insert. There are fundamental reasons for such a high demand. Firstly, the thermal motion of atoms is drastically reduced at low temperature, significantly improving

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[1] ISIS – pulsed neutron and muon source at the Rutherford Appleton Laboratory in Oxfordshire, UK, operated ~~by STFC.~~

the precision of structural measurements. Secondly, the low temperature range allows the study of phase transitions at temperature below 2K.

Conventionally this sample environment is provided by an Orange cryostat [2] or similar liquid helium bath cryostat with all the consequences typical for liquid cryogen based cryogenics. Hence the extensive use of liquid cryogen based equipment requires significant resources, creates a number of logistical issues including the considerable cost of the cryogens and can also pose health and safety problems. For example the cost of liquid helium in the UK has doubled in the last five years and continues to grow. There is also a very strong environmental aspect of this problem. Helium gas is a limited natural resource, which in the case of release rises through and escapes from our atmosphere [3].

Fortunately the progress in closed cycle refrigerator technology offers a new generation of cryogenic systems with significantly reduced consumption and, in some cases, completely eliminates the use of cryogens [1]. This breakthrough became possible due to a new generation of commercial cryo-coolers developed during the last decade. The most successful representative is the pulse tube refrigerator (PTR). The unique feature of the PTR is the absence of cold moving parts, which considerably reduces the noise and vibrations generated by the cooler [4, 5]. It also increases the reliability of the cold head because no expensive high-precision seals are required.

Here we discuss the new 50 mm diameter top-loading cryogen-free cryostat which provides sample environment for neutron scattering experiment in the temperature range 1.4 – 300K. Thanks to the high cooling power of the cryostat’s variable temperature insert (VTI): 0.23W @ 1.9K, the temperature range of the system can be extended below 1K by employing an Oxford Instruments’ Kelvinox VT dilution refrigerator insert. In our test the base temperature of the dilution insert, 50 mK, has been achieved in the continuous circulation regime. From a user perspective, the system offers operating parameters very similar to those of a standard top-loading liquid helium bath cryostat. This system has been developed ~~by~~ through an ISIS and Oxford Instruments collaborative project.

Design of the cryostat

The design of the system is based on the idea of combining a top-loading cryogen-free system described in Ref. [4] with a helium condensation loop [6], which has been successfully realized in the first prototype described in [7]. The PTR selected for the top-loading system is a Sumitomo RP082 B with cooling power of 1 W @ 4.2 K and an electrical input power to the compressor of 7 kW.

The cryostat utilizes a proprietary Oxford Instruments design of heat exchanger on the first stage of the PTR which gives enhanced thermal contact with the circulating gas. An ISIS developed heat exchanger is used on the second stage of the PTR to provide greater efficiency at high flow rates preferred for beam line operation.

The fixed capillary impedance used in the previous design [7] has been replaced by a needle valve that enables change of the helium circulation rate in a broad range.

The major change in comparison with the design of the previous system [7] is the replacement of the cryostat's top-loading insert with a VTI based on that used in Oxford Instruments' Cryofree product range (TeslatronPT and SpectromagPT) with beam line specific modifications. The annulus design of the VTI significantly improves the recuperation of cooling power stored in the cold helium vapor pumped out of the VTI's 1K pot heat-exchanger. The helium vapor is evacuated through the annular space of the VTI by an Edwards XDS35i dry scroll pump. After that the helium vapor is released into the environment. Two pressure relief valves (adjusted to 0.3 MPa relief pressure) together with two standard pressure gauges have been fitted at the inlet of the circulation loop and at the pumping line in order to prevent over-pressurizing of the system in the event of a blockage in the cryogenic parts of the circulation loop.

Thermal contact between the VTI 1K pot heat exchanger and the sample is achieved by cold exchange gas. The sample space is isolated from the VTI circulation loop so that the sample exchange gas is completely separate from the circulating helium gas. A temperature range from 1.4 K to 300 K can be achieved by the appropriate use of exchange gas and sample heating. This range can also be extended to ultra low temperature by using a dilution refrigerator insert [8]. One of the main advantages of the VTI is the absence of liquid helium in the sample horizontal plane which makes it ideal for neutron scattering experiments.

The system thermometry consists of four fully calibrated Cernox temperature sensors: at the sample holder, at the VTI heat exchanger and at the 1st and 2nd stages of the PTR. There are also two heaters. These are mounted on the sample holder and the VTI heat exchanger.

Operation of the cryostat and test results

After activating the PTR the Helium gas is released to the inlet of the circulation loop. The pressure of helium gas at the inlet is set at 0.1 MPa. After reaching equilibrium within a few minutes the circulation rate is settled at $2 \cdot 10^{-3}$ Moles per second. At lower temperatures the circulation rate increases reaching a maximum 10^{-2} Moles per second. Initial cryostat cool-down data is shown in Fig.1. Here the curve entitled “Sample” corresponds to time dependence of the temperature of the sample; “VTI” to the temperature of VTI heat exchanger; “PT1” and “PT2” to the temperatures of the 1st and 2nd stages of PTR. The total time taken by the sample holder to cool down from room temperature to base temperature (below 2K) is less than 7 hours. Once cooled, and with a continuous flow of helium, the cryostat can remain continuously at base temperature.

The sample changing procedure is very similar to that used in conventional top-loading cryostats. The helium circulation is stopped. The VTI is heated quickly and filled with helium gas at just above atmospheric pressure. After that the sample stick can be withdrawn and the sample changed. Once the sample stick is loaded back into the VTI the heater is turned off and the sample tube evacuated. A small pressure of helium exchange gas (~ 1 kPa) is required for sample cooling. The helium gas circulation is restored and the system can be left to re-cool to the base temperature. The temperature data collected during a typical sample change is presented in Fig.2. The labels correspond to the temperature curves in the same way as in Fig. 1. The total time required for a sample change, starting and finishing at the sample base temperature, is less than 2 hours.

The performance of the VTI heat exchanger agrees well with a classical 1K pot model [9] with a maximum cooling power 0.23W achieved in continuous regime at 1.9K.

The procedure of loading and cooling a KelvinoxVT dilution insert is also similar to one employed in conventional cryostats. The cooling down procedure consists of three stages. Stage one is pre-cooling of the insert with heat-exchange helium gas in the dilution insert’s vacuum can from room temperature down to below 2K. After that the VTI is warmed up to 5K and the exchange gas is pumped out from the insert for approximately one hour. Once good vacuum in the vacuum can is achieved, the VTI heater is switched off and the dilution refrigerator insert starts helium mixture condensation and, eventually, circulation in the automated regime. In Fig. 3 the first stage of the dilution insert pre-cooling is presented. The labels correspond to the temperature curves in the same way as in Fig. 1 except “Sample” is changed to “Kelvinox” – the temperature of the Kelvinox 1K pot heat exchanger. As we can see the insert can be cooled from room temperature down to 2K in less than 4 hours. The condensation of the ^{3}He/^{4}He mixture, initiation of ^{3}He circulation and cooling of the refrigerator down to base temperature is shown in Fig. 4, where (a) represents the behavior of the pressures in the dilution insert condensation line “G1”, before the nitrogen trap “G2” and in the ^{3}He pumping line “P1”; and (b) the time dependence of temperatures of the PTR second stage “PT2”, the 1K pot heat exchanger of the dilution insert “1 K pot” and at the mixing chamber ”M/C”. The refrigerator operates in fully automated mode. The time taken from the beginning of the condensation to reaching base temperature is little more than an hour. The total cool down time of the dilution insert starting from inserting the refrigerator into the VTI is less than 7 hours, which is a typical time for a conventional cryogen based top-loading cryostat. After achieving the base temperature the refrigerator has been left in circulation mode for 16h without significant fluctuations of the mixing chamber temperature.

In the test the base temperature of the fridge was 55mK a little bit higher than the technical specification of 35mK. This can be explained by a massive sample attached to the mixing chamber, which has not been removed after the previous neutron scattering experiment, conducted in a standard Oxford Instruments Variox cryostat. In this experiment a similar base temperature of 60mK was achieved.

Future development

Currently the cryostat has only been tested with the VTI operating in open loop mode where the helium gas is released into the environment. However, the flow circuit incorporates Oxford Instruments’ patent pending helium purification system which protects the cryogenic parts of the circulation loop from contamination related blockages. This purification system is already used successfully for closed loop circulation within Oxford Instruments’ Cryofree product range. The new beam line optimized cryostat described in this paper will undergo further testing at the ISIS facility to investigate fully closed cycle operation.

Conclusion

We have presented the design and test results of a new 50 mm diameter top-loading cryogen free cryostat for neutron scattering sample environment. Thanks to the high cooling power of the cryostat’s VTI heat exchanger (0.23W @ 1.9K) the cryostat temperature range 1.4 – 300K can be extended to milliKelvin temperatures by inserting a dilution refrigerator insert into the VTI. In our tests with a Kelvinox dilution insert a base temperature of 55 mK has been achieved in the continuous circulation regime. All the parameters of the new system are very close to that of conventional cryogen based cryostats, such as an Orange cryostat for example. However the new system does not require cryogenic liquid top-ups which can be challenging from the logistical, economical, operational and safety points of view.

We are very grateful to members of the ISIS sample environment group involved in the ISIS cryogen free development project. We also greatly appreciate the fruitful collaboration with colleagues from Oxford Instruments.

Conclusion

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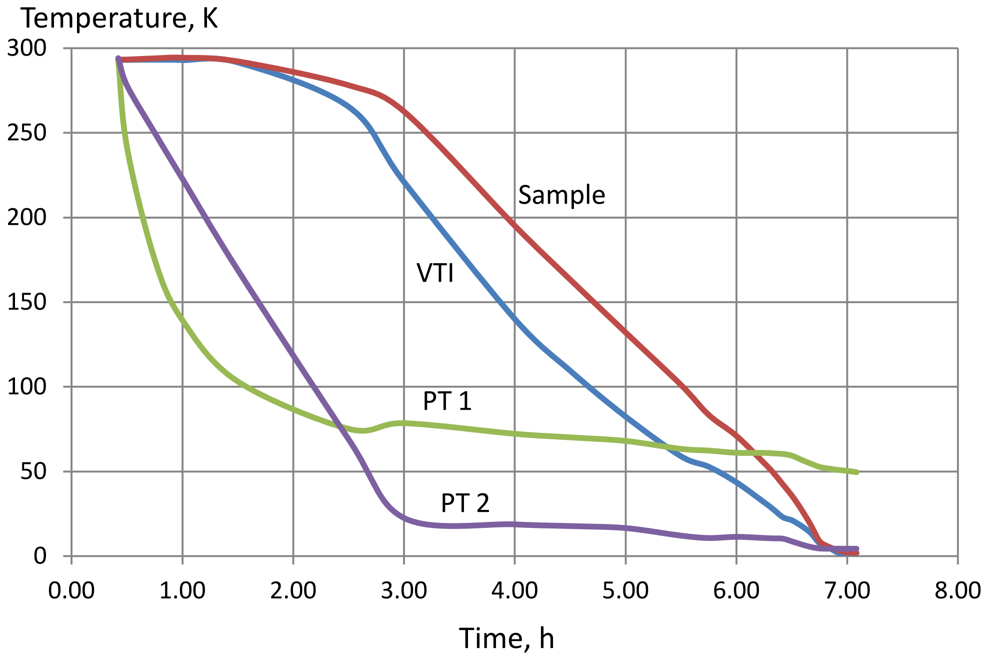
(color online) Figure 1. Initial cryostat cool-down data: “Sample” is the temperature of the sample; “VTI” is the temperature of the VTI heat exchanger; “PT1” and “PT2” are temperatures of the 1st and 2nd stages of the PTR respectively.

(color online) Figure 2. Typical sample change data. The labels correspond to the temperature curves in the same way as in Figure 1.

(color online) Figure 3. The first stage of the dilution insert pre-cooling data. Labels correspond to the temperature curves in the same way as in Figure 1 except “Sample” is changed to “Kelvinox” – the temperature of the Kelvinox 1K pot heat exchanger.

(color online) Figure 4 In this picture we present the process of condensation of the He mixture, initiation of ^{3}He circulation and cooling of the Kelvinox to base temperature, where (a) represents the behavior of the pressures in the condensation line “G1”; before the nitrogen trap “G2”; and in the ^{3}He pumping line “P1”; and (b) the time dependence of temperatures at the 2nd stage “PT2”; at the 1K pot heat exchanger “the 1K pot”; and at the mixing chamber “M/C”.

Figure1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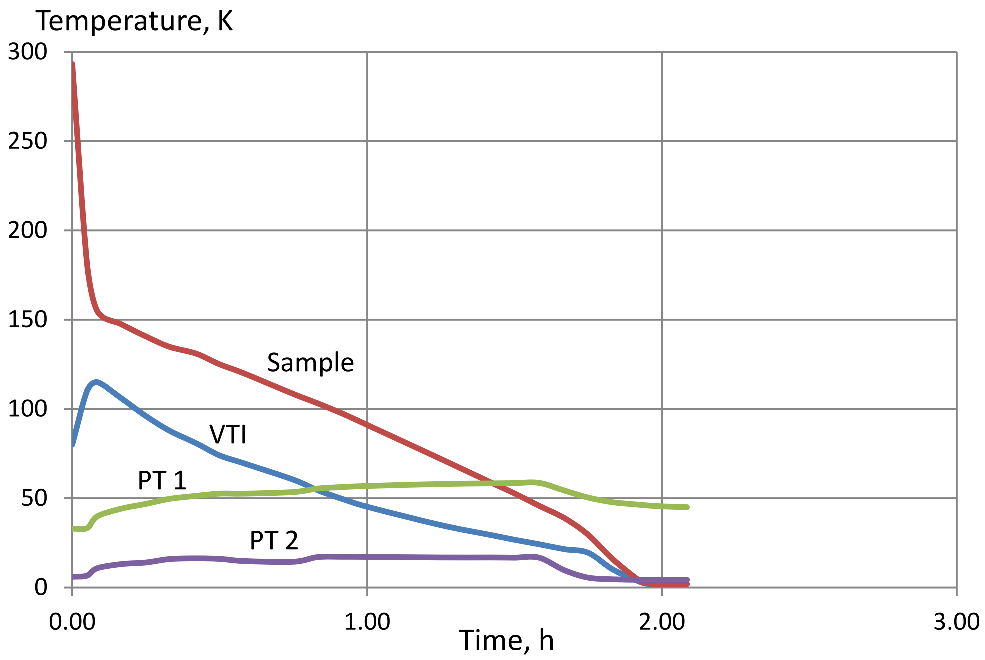
Time, h
Sample
VTI
PT 1
PT 2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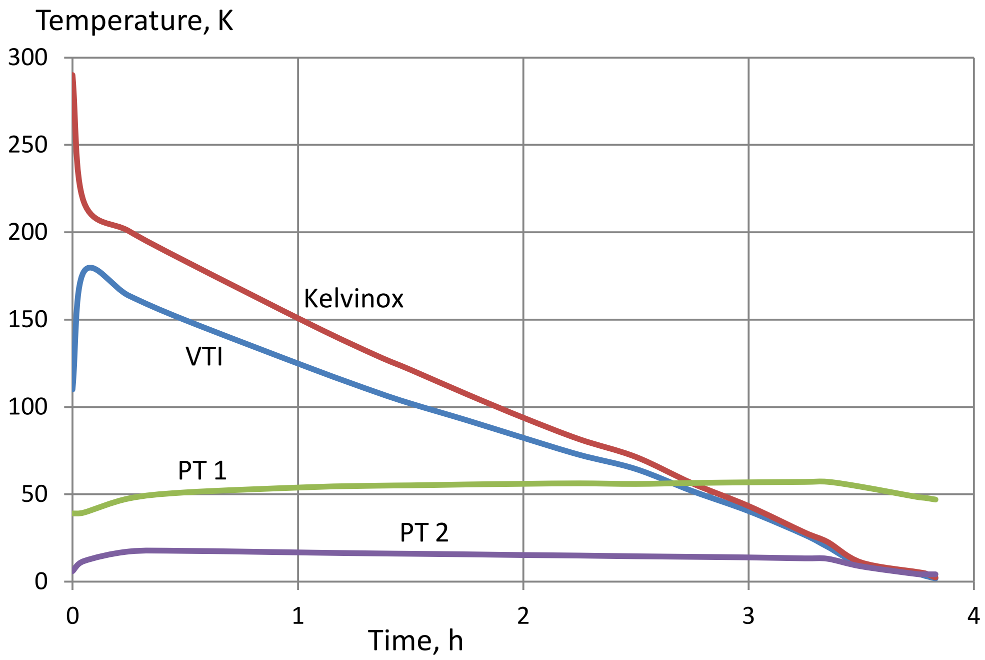
PT 1
PT 2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Kelvinox
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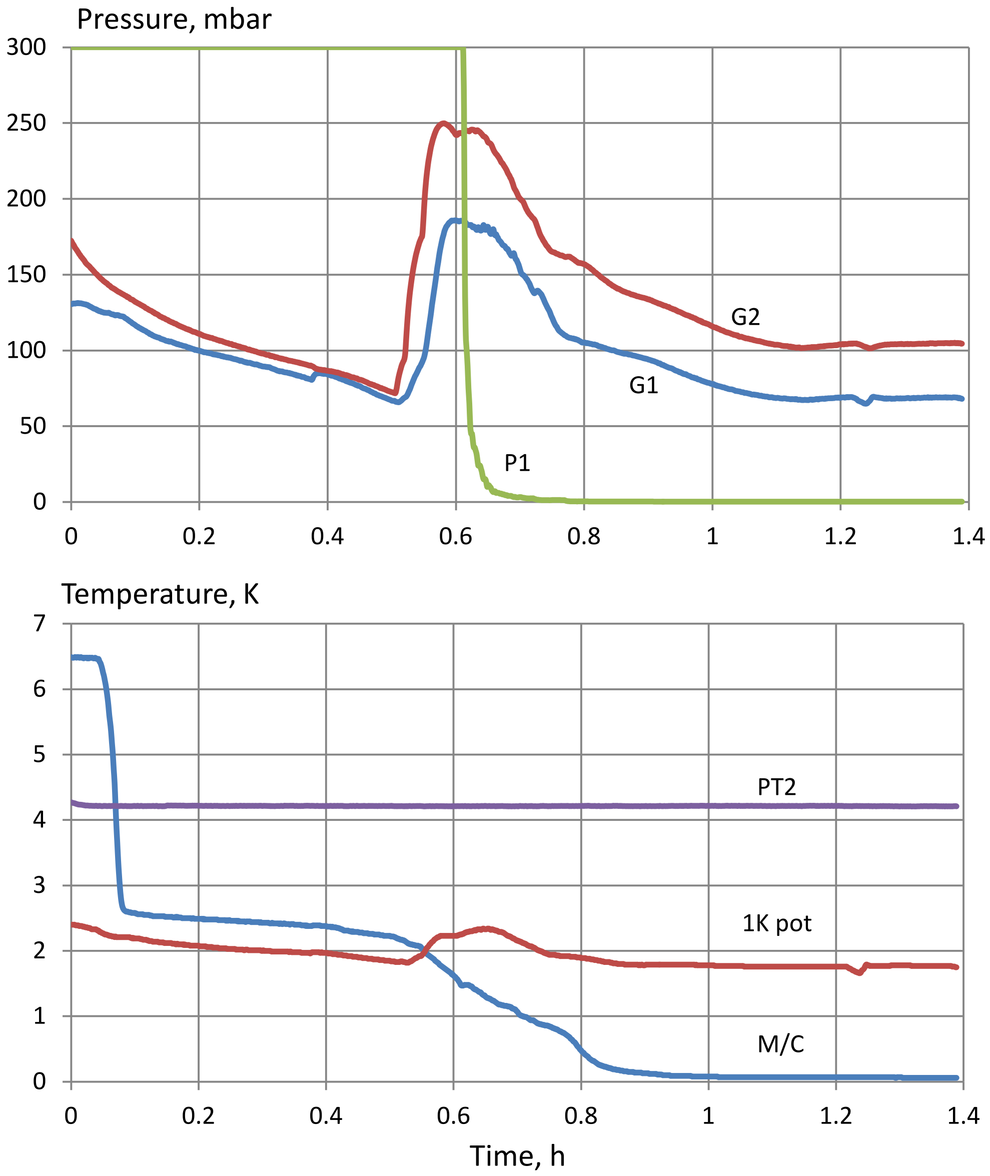
PT 1
PT 2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0.8
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G2
G1
P1
Temperature, K
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6
5
4
3
2
1
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0
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0.4
0.6
0.8
1
1.2
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Time, h
PT2
1K pot
M/C