Cryogen free high magnetic field sample environment for neutron scattering.

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Abstract. Cryogenic equipment can be found in the majority of neutron scattering experiments. Recent increases in liquid helium cost caused by global helium supply problems lead to significant concern about affordability of conventional cryogenic equipment. However the latest progress in cryo-cooler technology offers a new generation of cryogenic systems in which the cryogen consumption can be significantly reduced and in some cases completely eliminated. These systems also offer the advantage of operational simplicity, require less space than conventional cryogen-cooled systems and can significantly improve user safety. At the ISIS facility it is possible to substitute conventional cryogenic systems with cryogen free systems.

1. Introduction
It is probably no exaggeration to say that cryogenic equipment is used in the majority of neutron scattering experiments. Most neutron facilities have a fleet of various cryostats providing low and ultra-low temperature sample environment and superconducting magnets for high magnetic fields [1]. Such large scale usage of cryogenic equipment requires significant resources and creates a number of logistical problems including health and safety issues and the considerable cost of the required cryogens.

The latest progress in cryo-cooler technology offers a new generation of cryogenic systems in which the cryogen consumption can be significantly reduced and in some cases completely eliminated [2, 3]. These systems also offer the advantage of operational simplicity, require less space than conventional cryogen-cooled systems and significantly improve user safety.

At the ISIS facility it is possible to substitute conventional cryogenic systems with cryogen free systems. A preference has been given to systems based on the pulse tube refrigerator (PTR) which
possesses no cold moving parts. This fact reduces generated noise and vibration [4], increases reliability and decreases service efforts.

In this presentation we discuss the design and test results of two superconducting split pair magnet systems supplied recently to the ISIS by Oxford Instruments [5]. These systems were successfully tested at Oxford Instruments and currently are being prepared for commissioning at the ISIS facility.

![Figure 1. Wide angle chopper instrument 9T superconducting magnet in the cryostat with helium re-condensed by PTR.](image)

2. **PTR actively-cooled superconducting magnets**

The design of re-condensing magnet cryostats is usually based on the design of similar bath cryostats [2]. As shown in Fig.1 the superconducting magnet is immersed in the liquid helium. The radiation shield is cooled by the cooler’s 1\textsuperscript{st} stage and the 2\textsuperscript{nd} stage re-condenses helium directly in the helium vessel. Thus the re-condensing system does not consume any liquid helium in normal operation.

The main advantage of this system is that all magnet operating procedures, for example cooling, running up to the field and quenching remain the same as for a standard magnet in a bath cryostat. This approach also provides a homogeneous temperature distribution, which is crucial for optimum magnet performance. All standard system accessories like current leads, magnet power supply, helium transfer siphon, etc. can be used with this system. One more important advantage is the ability of the magnet to stay at field for more than forty eight hours before quenching in the case of a cryo-cooler power failure. This feature also makes it possible to move the cold magnet between different neutron scattering instruments and/or the cryogenic laboratory.

3. **Factory test results at Oxford Instruments**

Both the wide angle chopper magnet for spectrometry and the 14T magnet for diffraction measurements share the similar top parts of the re-condensing cryostat with Variable Temperature Inserts (VTI) but have different bottom parts accommodating high field split pair magnets and sample space. Some comparative magnet characteristics are given in the table below.

In order to achieve maximum re-condensation and provide spare cooling power for possible future VTI flow re-condensation the cryostat is equipped with one of commercially available 1W @ 4.2K PTR manufactured by Cryomech Inc. This PTR has substantial spare cooling power for the size of the system and therefore in normal steady state operation this spare cooling power needs to be counteracted by built-in electric heaters in order to stabilize system pressure, temperature and liquid level. Alternatively the heater can be switched off and the system will reach equilibrium state at sub atmospheric pressure and lower temperature when PTR cooling power (which is dropping with lowering temperature) will be balanced by the heat load. Experiment shows that equilibrium
temperature in this case is of order of 3.5K however this operational mode is not desirable due to inconvenience of topping the system up and danger of atmospheric air ingress into helium volume.

**Table 1.** The main features of split pair superconducting magnets.

|                      | Chopper Magnet | 14T Magnet |
|----------------------|----------------|------------|
| Central Field Strength at 4.2K | ±9T            | ±14T       |
| Central field homogeneity (over 10mm x 10mm cyl.): | <0.4%          | <0.4%      |
| Neutron aperture in horizontal plane | 2 x 90° & 2 x Ø40mm | 340°       |
| Neutron aperture in vertical plane | ±15°           | +10° and -5° |
| Current decay in persistent mode | 30 ppm/hr   | 20 ppm/hr  |
| Sample space diameter | 50mm           | 25mm       |
| Sample temperature range | 1.5-300K    | 1.5-300K   |

The system is equipped with an automatic pressure control system keeping the He bath pressure at a set level (usually 15 mbar above atmospheric pressure) by means of supplying required power to the built-in heater. It was demonstrated during the system test that temperature, pressure and liquid He level can be kept constant in steady-state operation with approximately 600-650mW supplied to the heater. The plot of the above mentioned parameters in steady state operation is shown on Fig.2.

![Figure 2. System parameters in steady-state controlled operation.](image)

One of the very useful operational modes of the system available due to substantial spare cooling power and provision of special gas return path is external gas re-condensing. Gaseous He at room temperature and a constant flow rate of approximately 2.5 gL/min was supplied to the system via a set of filters and a cold trap. The system was capable of re-condensing its own boil-off plus additional external gas, still controlling bath pressure at a set level using the heater (though apparently with less power). The He level rising was observed confirming the fact of room temperature gas re-condensation. The plot of the system parameters in the described operational mode is shown on Fig.3. The VT1 performance exceeding specification was demonstrated in the course of system test as well. Because the dry scroll 35 m³/hr pump supplied by Edwards-BOC is used for gas circulation in the VT1 it was possible to demonstrate a few modes with He gas flow re-introduction back to the system from...
the pump exhaust. This mode is similar to the external gas re-condensing described above but He level remains stable – i.e. the system works in zero-boil-off regime with the VTI running at a set temperature. Some further work in assessing necessity of gas purification for long term operation is still to be done but the first results are very promising and indicate that the system is capable of working in fully re-condensing mode under different operational conditions.

![Figure 3. System parameters in external gas re-condensation mode.](image)

Currently ISIS uses a number of dilution and $^3$He refrigerator inserts used with standard VTI. All these systems can easily be made cryogen-free if used in a re-condensing cryostat with VTI [4].

### 4. Conclusions

Driven by global helium supply problems and by the desire to optimize technical resources, the ISIS facility is carrying out an internal development program to substitute gradually all conventional cryogenic systems with cryogen-free systems. As explained above the helium consumption of these systems can be dramatically reduced and in some cases completely eliminated. These systems are also more convenient to operate and require less maintenance. First two large split pair superconducting magnet systems are received at the ISIS facility after successful factory tests at Oxford Instruments. Full gas re-condensation in steady state and external gas supply was demonstrated during the tests as well as the possibility to fully re-utilise the VTI flow.

### References

[1] Bailey I F 2003 Z. Kristallogr. 218 84
[2] Kirichek O, Carr P, Johnson C and Atrey M 2005 Rev. Sci. Instrum. 76 055104
[3] Evans B E, Down R B E, Keeping J, Kirichek O and Bowden Z A 2008 Meas. Sci. Technol. 19 034018
[4] Kirichek O, Evans B E, Down R B E and Bowden Z A 2009 Journal of Physics: Conference Series 150 012022
[5] Oxford Instruments plc: [http://www.oxford-instruments.com](http://www.oxford-instruments.com)