Cryogen free sample environment for neutron scattering experiments at ISIS

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Abstract. Most neutron facilities have a fleet of cryostats providing low temperature and high magnetic fields for sample environment. This large scale usage of cryogenic equipment requires significant resources and can create a number of problems including health and safety issues and the considerable cost of the required cryogens. The last problem has become more significant due to the increasing costs of liquid helium caused by global helium supply problems. The ISIS facility has an internal development programme intended to gradually substitute all conventional cryogenic systems with cryogen free systems preferably based on the pulse tube refrigerator. The programme includes a number of development projects which are aiming to deliver a range of cryogen free equipment including a top-loading 1.5 K cryostat, superconducting magnets in re-condensing cryostats and cryogen free dilution refrigerators. Here we are going to describe the design of these systems and discuss the results of prototype testing.

1. Introduction
The increasing costs of liquid helium caused by global helium supply problems raises significant concerns about the affordability of conventional cryogenic equipment based on the evaporation of liquid cryogens. This problem poses a serious threat to the future experimental programmes of large scientific facilities. However the progress in closed cycle refrigerator (CCR) technology offers a new generation of cryogenic systems with significantly reduced consumption and, in some cases, near complete elimination of cryogens. 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. It also increases the reliability of the cold head because no expensive high-precision seals are required and the cold head can be operated without any service inspection.

There are two options available in order to reduce or, in the ultimate case, eliminate the usage of cryogens: so-called “dry” systems that do not contain liquid cryogens at all, built around a cryo-cooler that utilizes the cooling power from the cold head [1, 2] and re-condensing systems in which evaporating helium is cooled and returned back to the cryostat by a CCR [3]. The ISIS facility is carrying out an internal development programme intended to gradually substitute all conventional cryogenic systems with cryogen-free systems preferably based on the PTR. This paper will describe the design of these systems and discuss the results of prototype testing.
2. Top-loading cryostat based on a CCR

Since the late 1970s the Orange Cryostat designed at the ILL [4] has remained the workhorse of all neutron scattering facilities. This is a top-loading liquid helium bath cryostat with a base temperature of ~1.5K. However today the Orange Cryostat is losing its popularity due to its high helium consumption and the significant resources required for routine operation.

Almost a decade ago ISIS started a development programme aimed at developing a cryogen-free top-loading system based on the CCR as an ideal substitute for the Orange Cryostat. This project consists of two phases. During the first phase, top-loading systems based on the Gifford McMahon (G-M) cold head and the PTR have been designed and tested [2, 5]. These top-loading systems are fitted with rotation stages which allow the rotation of a sample around its vertical axis with precision better than ±0.5°. The photos of both systems are presented in Fig. 1 and comparisons of technical specifications achieved in tests are presented in Table 1. It can be seen that the top-loading system based on the PTR, reaching a base temperature of 3.5K in under three hours, cooled faster than the G-M CCR based system. In addition, the vibration level measured in the PTR cryostat was also an order of magnitude less than that measured in a similar system based on a standard G-M cold head. Low level of mechanical vibrations is essential for high resolution neutron reflectometry and SANS experiments. For a rough estimation of the CCR-based top loading system’s cost efficiency one can compare the cost of liquid helium required to run a standard Orange Cryostat (~30 £/day) with the cost of the electric energy consumed by a CCR (~8 £/day). This difference is only likely to increase in the foreseeable future.

| Type of system | Base sample temperature | Cool down time | Sample change time | Number of systems available |
|---------------|-------------------------|----------------|--------------------|-----------------------------|
| PTR Based     | 3.5K                    | <3h            | <1.2h              | 2                           |
| GM Based      | 4.4K                    | ~4h            | <1.25h             | 8                           |
| Orange cryostat | ≤1.5K                  | ~ 24h          | ~ 2 h               | 21                          |

During the second phase of the project a 1.5K cryogen-free system based on a PTR, with a 50 mm diameter top-loading sample access for neutron scattering experiments has been developed and successfully tested [6]. The system design is based on the combination of a top-loading system [2] with a helium condensation loop, which consists of three heat-exchangers mounted on the cryocooler, stainless steel impedance and 1.5K heat-exchanger on variable temperature inserts (VTI). The sample temperature range of this system is 1.45 – 300K in the continuous flow regime. A sustainable cooling power of 55 mW at 1.82K and temperature stability of ± 0.1K in temperature range 5 - 200K, and ±0.01K below 5K has been achieved. From a user perspective, the system offers operating parameters very similar to those of an Orange cryostat, but without the complication of cryogens. The system may also be used with ultra-low temperature inserts such as the Kelvinox VT dilution refrigerator. Recent preliminary tests have successfully demonstrated the operation of a Kelvinox VT insert at 75 mK.

3. Cryogen free dilution refrigerators

The ISIS facility uses a number of dilution and ³He refrigerator inserts which can be used with the VTI of a conventional cryostat or superconducting magnet. These systems consume cooling power produced at the VTI heat exchanger and can easily be used with cryogen-free, or reduced cryogen, cryostats. This can be achieved by inserting the refrigerator either in the top-loading system based on the PTR discussed above or in a re-condensing cryostat with a VTI. However the range of applications of conventional ultra-low temperature inserts is limited by their relatively low cooling power (~30 µW at 100mK) and small sample space (OD ≤ 37 mm and height ≤80 mm).

A growing number of neutron scattering experiments now require a combination of extreme conditions such as high pressure, high magnetic field or large sample size, with ultra-low temperatures. In order to satisfy these emerging requirements one needs a powerful dilution
refrigerator with a sample space big enough to accommodate a high pressure cell or a small superconducting magnet. Oxford Instruments, in collaboration with ISIS, have developed a powerful cryogen-free dilution refrigerator (E-18) for neutron scattering experiments. The design of the refrigerator is based on a prototype of a “dry” dilution unit with PTR pre-cooling [7]. The refrigerator is capable of cooling large (OD 200 mm; height 250 mm) and heavy (up to 20 kg) samples and provides access for the neutron beam through a set of thin aluminium alloy windows. The base temperature of the refrigerator is 15mK and the cooling power is approximately 370 μW at 100mK. The cool down time of E-18 is ~36 hours, which means using only one sample during single experiment. Currently Oxford Instruments, again in collaboration with ISIS, is developing a split pair superconducting magnet for neutron scattering experiments; the magnet is going to be attached to the 4K shield of the E-18 refrigerator. The design of the magnet is based on a prototype successfully tested by Oxford Instruments [8].

Fig. 1 Top loading CCRs based on PTR (left); and on GM cryo-cooler (right)

4. Superconducting magnets actively cooled by a PTR
Neutron scattering experiments at high magnetic field and ultra low temperatures consume significant amount of the total cryogens spent by ISIS user program. In 2006 ISIS commissioned three superconducting magnets for use in neutron scattering experiments [5, 9]. All three magnets have used
re-condensing technology based on the PTR. The design of re-condensing magnet cryostats is usually based on similar helium bath cryostats [3]. The superconducting magnet is immersed in the liquid helium. The radiation shield is cooled by the cooler’s first stage and the second stage re-condenses helium directly in the helium vessel. Thus the re-condensing magnet cryostat 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 are identical to those of a standard magnet in a bath cryostat. All standard magnet system accessories like current leads, power supply, helium transfer siphon etc. can be used. This system also provides a homogeneous temperature distribution, which is crucial for optimum magnet performance. Another important advantage is the ability of the magnet, in the case of a cryo-cooler power failure, to stay at field for more than 48 hours before quenching. This feature also makes it possible to move the cold magnet between different neutron scattering instruments and/or the cryogenic preparation laboratory.

Zero helium boil-off operation of the re-condensing 9T superconducting magnet cryostat with a Kelvinox VT dilution refrigerator insert running at a base temperature of 63mK during a 24 hour period has recently been demonstrated at ISIS [10]. This combined system allows a cryogen-free high magnetic field and ultra-low temperature sample environment for neutron scattering experiments. The system offers a significant reduction in liquid helium consumption as well as the advantage of operational simplicity and a significant improvement in user safety. Estimations based on half a year of 14T magnet cryostat operation statistics show that one re-condensing magnet cryostat should save us ~7500 £/year.

5. Conclusion
The ISIS facility is carrying out an internal development programme with the intention of gradually substituting all conventional cryogenic systems with cryogen-free systems preferably based on the PTR. This paper has described the design of ISIS cryogen-free and re-condensing systems and discussed the results of prototype testing and operational experiences of these systems in the ISIS user programme. This approach offers a significant reduction in liquid helium consumption as well as the advantage of operational simplicity and a significant improvement in user safety. The ISIS facility plans a gradual substitution of all conventional cryostats with cryogen free systems in the next decade.

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