

# Cryogen free low temperature sample environment for neutron scattering experiments

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**Abstract.** Recent increase in liquid helium cost caused by global helium supply problems rose significant concern about affordability of conventional cryogenic equipment. Luckily the progress in cryo-cooler technology offers a new generation of cryogenic systems with significantly reduced consumption and in some cases nearly complete elimination of cryogens. These cryogen-free systems also offer the advantage of operational simplicity and require less space than conventional cryogen-cooled systems. The ISIS facility carries on an internal development program intended to substitute gradually all conventional cryogenic systems with cryogen free systems preferably based on pulse tube refrigerators. A unique feature of this cryo-cooler is the absence of cold moving parts. This considerably reduces vibrations and increases the reliability of the cold head. The program includes few development projects which are aiming to deliver range of cryogen free equipment including top-loading cryostat, superconducting magnets and dilution refrigerators. Here we are going to describe the design of these systems and discuss the results of prototypes testing.

## 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. This last problem has significantly worsened recently due to the increase in liquid helium costs caused by ongoing global helium supply problems [2].

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 [3, 4]. These systems also offer the advantage of operational simplicity, require less space than conventional cryogen-cooled systems and significantly improve user safety.

In order to explore this opportunity the ISIS facility is carrying out an internal development program intended to substitute gradually all 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 considerably reduces the generated noise and vibration and increases the reliability of the cold head. Another important advantage is that expensive high-precision seals are no longer required and the cold head can be operated uninterrupted by service inspection.

In this presentation we discuss the design and test results of a few cryogen-free systems including a top-loading cryostat based on the PTR, superconducting magnets, a dilution refrigerator (DR) and a stress rig for measurements of bulk stress in engineering components at cryogenic temperatures.

## **2. Top-loading cryostat based on the PTR**

During the last three decades the Orange Cryostat designed at the ILL [5] has remained the workhorse of neutron scattering centers world-wide. This is a liquid helium bath cryostat with a base temperature of 1.5K, with a top-loading sample holder. The Orange Cryostat also has a set of thermal radiation shields containing neutron-transparent aluminium or vanadium windows. Despite its enviable reputation the Orange Cryostat is now losing its popularity due to its high helium consumption and demands on maintenance and operation resources.

One aim of the ISIS development program is to develop a cryogen-free top-loading cryostat based on the PTR as an ideal substitute for the Orange Cryostat. This project consists of two phases. The first phase is the design and testing of a simple top-loading system based on the PTR. This phase was successfully completed and the results published [6]. This top-loading PTR cryostat reached a base temperature of below 3K in under three hours. Vibration levels measured in the PTR cryostat were an order of magnitude less than those measured in a similar system based on a standard G-M cold head. The second phase of the project is the addition of a  $^4\text{He}$  circulation loop to the top-loading cryostat to attain a base temperature below 2K, allowing use with a dilution insert. Tests on a prototype design have reached a base temperature of 2.3K and further development is underway.

## **3. PTR actively-cooled superconducting magnets**

In 2006 ISIS received funding for the development of high magnetic field sample environment equipment. This project includes the purchase of three superconducting magnets: a wide angle chopper magnet for spectrometry; a 3D magnet for the reflectometry and a 14T magnet for diffraction measurements. The ISIS supplier tender specified that all magnets must have zero cryogen boil-off to be achieved by the re-condensation of helium by the PTR [3].

The design of re-condensing magnet cryostats is usually based on the design of similar bath cryostats. As shown in Fig.1 the superconducting magnet is immersed in the liquid helium. The radiation shield is cooled by the cryo-cooler first stage and the second 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. The quench venting mechanism can also be the same as for a bath cryostat. In fact, for the user this system is almost identical to a conventional one but does not need regular cryogen supply, instead only electrical power is required to keep the system cold.

One more important advantage is the ability of the magnet to stay at field for more than ten 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.

Another superconducting 5T magnet, based on “dry” cryogen-free technology, has been designed for the new ISIS muon instrument HiFi by Cryogenic Ltd. The cooling power for the magnet is provided solely by pulse tube refrigeration. This kind of “dry” system is usually more compact than re-condensing systems [4, 7, 8]. They do not require a helium reservoir, making the design of the system simpler. As in the re-condensing system, the radiation shield is cooled by the first stage of the cryo-cooler, and the superconducting magnet is thermally linked directly to the second stage. The cool

down of “dry” systems may take tens of hours and in the case of cryo-cooler power failure the approximate time until the magnet quenches is less than one minute.

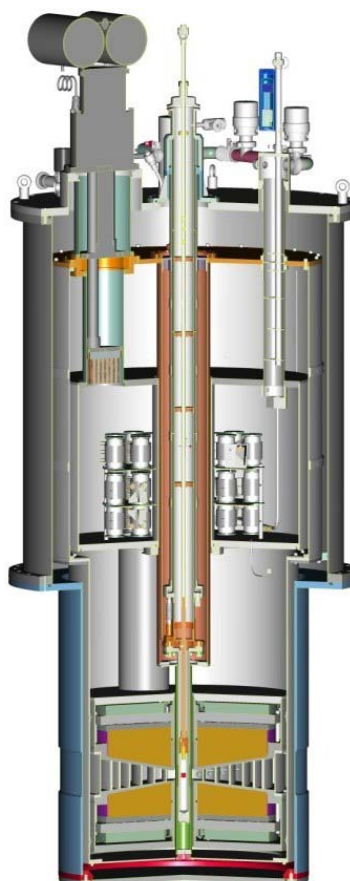


Fig. 1 The 14T superconducting magnet in a cryostat with helium re-condensed by PTR (by courtesy of Oxford Instruments)

#### 4. Cryogen free dilution refrigerators

Currently ISIS uses a number of dilution and  $^3\text{He}$  refrigerator inserts which can be used with standard variable temperature inserts (VTI) of cryostats or superconducting magnets. These systems consume cooling power produced at the VTI heat exchanger. For Oxford Instruments Kelvinox<sup>®</sup> and Heliox<sup>®</sup> as well as Leiden Cryogenics DR the temperature of the heat exchanger should be less than 2K. For the TBT<sup>®</sup> fridge, which incorporates Joule-Thompson stage [9], the VTI temperature can be around 4K. All these systems can easily be made cryogen-free if used with cryogen-free cryostats. This can be achieved by inserting the refrigerator either in the top-loading cryostat based on the PTR discussed above or in a re-condensing cryostat with a VTI. Thus all new ISIS magnets are capable of providing a cryogen free regime for refrigerator inserts. However the range of applications of the systems mentioned is limited by their relatively low cooling power ( $\sim 30 \mu\text{W}$  at 100 mK) and small sample space (OD of the sample space is  $\leq 37\text{mm}$  and height  $\leq 80 \text{ mm}$ ). These parameters are crucial for a number of neutron experiments which require a combination of extreme conditions like high pressure, high magnetic field or large sample size with ultra-low temperatures.

VeriCold Oxford Instruments in collaboration with ISIS have developed a powerful cryogen-free DR for neutron scattering experiments. The design of the fridge is based on a prototype of a “dry” DR with PTR pre-cooling [10]. The fridge is capable of cooling large (OD 200mm; height 250mm) and heavy (up to 20 kg) samples and provides access for the neutron beam through the set of vanadium (or aluminium) windows. The main specifications of the VeriCold DR are presented in Table 1 in comparison with the specifications of the standard Kelvinox<sup>®</sup> VT. The results of the system test on a neutron beamline will be published elsewhere.

**Table 1.** The main specifications of the VeriCold DR in comparison with the standard Kelvinox<sup>®</sup> VT

	VeriCold OI Cryo-free DR	Kelvinox <sup>®</sup> VT sample-in-vacuum DR
Cooling power at 100 mK	$\geq 400 \mu\text{W}$	$30 \mu\text{W}$
Base temperature	$\leq 15 \text{ mK}$	$\leq 30 \text{ mK}$
Sample space	$\varnothing 250 \text{ mm}$ ; height 250 mm	$\varnothing 38\text{mm}$ ; height 80mm

## 5. 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. The ISIS facility has also developed a cryogen-free stress rig for measurements of bulk stress in engineering components at cryogenic temperatures [11] which falls out of scope of this publication.

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