Cryogenic Sample Environment Systems at ISIS

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A cryogen free cryostat for neutron scattering sample environment

The design combines a tor-loading cryogen-free system with a helium condensation loop. The Pulse tube refrigerator (PTR) is a Supergen cryo 7.8 K. High purity helium gas is supplied to the system via a liquid nitrogen cold trap. The gas passes through a filter and is pre-cooled on the 1st stage of the PTR, from here it is cooled further on the 2nd stage regenerator heat exchanger; this heat exchanger sits on the reservoir tube of the PTR. After the regenerator heat exchanger the helium gas flows through the 2nd stage heat exchanger. After pre-cooling in the 2nd stage heat exchanger the helium gas condenses in a liquid helium chamber which is attached to the PTR 2nd stage. From the chamber the liquid helium is fed into the VITI heat exchanger via an automated cryogenic valve, the liquid helium enters the VITI heat exchanger where it evaporates and is evacuated through the VITI annular pumping space.

The system has a temperature range from 1.80K up to 300 K with cooling power in excess of 200mW at 1.8K. The systems temperature range can be extended to ultra low temperature by using a Kelvinox dilution refrigerator insert.

Cryogenic stress rig for measuring bulk stress in engineering components

Coolina is provided by two RDK415 closed cycle refrigerators (CCRs); thermal conduction is minimised by a high vacuum in the cryostat; the radiation shield is linked to the first stage of the CCRs, the sample spaces are linked to the second stage of the CCRs through welded flexible copper shims, thermal conduction is minimised by using insulating ceramic spaces within the two rig arms.

Base temperatures of approximately 6.5 K. 8K in approximately two hours, with high power heaters assisting in a cold to cold sample change in 2 hours.

Helium Cryostats

ISIS has approximately 30 static exchange gas cryostats for its scientific program, many of these systems are fitted with special tails that ensure the best data can be obtained during an experiment.

The systems are a combination of Orange and Oxford Instruments Varian cryostats that can offer a range of temperature form 1.5K up to 300K with exchange gas and by the use of a hot stage sample stick the upper range can be extended to 700K without exchange gas.

Superconducting Magnets

ISIS has 5 high field magnets, which include a 3D Vector magnet and 4 vertical field magnets.

Shown left is the 97T wide angle spectrometer magnet with a ±10° opening in the vertical plane and two 90° openings in the horizontal plane. All surfaces in the direction of incident and scattered beam are clad in boron nitride tiles that give this magnet a very low background. Shown right is the 13.5T high field magnet for diffraction, it has a ± 5° ± 10° opening in a total of 27mm of Al between the magnet formers, the magnet has a low background as the upper and lower surfaces of the formers are coated in cadmium.

The 4K Top Loading Closed Cycle Refrigerator

The 4K Top Loading CCR has a 100mm diameter sample volume that is linked to the second stage of the CCR that delivers a temperature of approximately 4.5K. The first stage is connected to a radiation shield that is of the order of 40K. Sample cooling is via static exchange gas that is set through a conventional 3 way valve.

The unit has an operating temperature range between 4.5K and room temperature, temperature is measured by a rhodium iron sensor and temperature control is achieved by two 128 Watt heaters. The temperature range can be increased to 700K by using a thermally isolated hot stage mounted to a sample stick that enables temperatures as high as 700K to be reached.

Extending the use of top loading CCR and wet systems

Sample rotation can be offered by using the rotation stage above on the left; this is a VG Scienta rotator ofalinear (ISIS pattern) that utilises a Renishaw rotary encoder ring. The platform is controlled by a McLeenan motor drive and can rotate to a LabVIEW 6.1 and can deliver rotational accuracy of 0.01° per step and 0.01° feedback. The Hot Sticks shown above centre is produced in house and consists of a copper sample holder that houses 2 Watox Fire red 100W 100V heaters and a PT100 sensor, the heating element is thermally insulated by ceramic spacers and 2 thermal shields can be attached to reduce temperature gradient. The device has been successfully used up to 700K; it should be noted that exchange gas needs to be removed from the top loading CCR before any hot work begins.

The 1K insert shown above right is supplied by Ice Oxford, it is a continuous flow device that uses liquid Helium. The 1K insert is installed into the top loading CCR and cooled to 100K by exchange gas, the exchange gas is then removed. A low loss transfer line fed from a 100L liquid Helium Dewar is then coupled to the insert and cooling begins. Helium liquid is fed to a 4K reservoir which in turn feeds a 3K pot. The 3K pot is then pumped upon to achieve continuous temperatures of 1.25K.

Ultra Low Temperature

The devices shown above on the left are parasitic inserts that can be used in any Ø50mm compatible system, ISIS has 3 sorption refrigerator inserts that can offer temperatures as low as 250mK and 3 dilution refrigerator inserts of this type that can offer temperatures as low as 30mK.

The inserts can offer a wide range of diverse sample environments with additions like Atto Cube systems for manipulation or by the installation of capillaries for condensed gases, as well as single crystal work and fine rotation. Shown above in the centre is the pulse tube based E18 cryogen free high powered dilution refrigerator; the system has a base temperatures of 20mK and has a cooling power measured at 40mW at 120mK.

This frigde takes on challenging work that current dilution refrigerator system at ISIS struggle with; examples of the science studied using this frigde are the further exploration of the Helium 4 super solid phenomenon and high pressure studies using clamped cells.

The E18 dilution fridge has been constructed with reinforcing supports from the 3rd stage of the pulse tube to the mixing chamber. The frigde itself has been built to take samples of up to 20Kg in mass.

The system has further benefits in that it is completely oil free, its operation is fully automated and computer controlled.