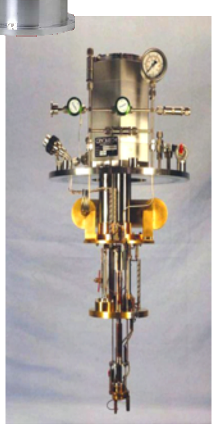
Ultra Low Temperature Environments for Neutron Scattering

Oxford Instruments' superconducting magnet and cryogenic technologies are at the heart of instrumentation driving applications in drug discovery, life science and materials science.

A key technique used to probe nano-materials, biological species and novel states of matter is Neutron Scattering.

Creating extreme sample environments for Neutron Scattering is at the leading edge of magnet and cryogenic design from Oxford Instruments.



HelioxAC-V

Ultra Low Temperature Design

Integrated Sample Environments

It is typical to incorporate Ultra Low Temperature (ULT) sample environments into high field split pair magnets for Neutron Scattering. As helium absorbs neutrons it is desirable to have no He in the beam path. This rules out conventional 3He and dilution refrigerator designs, requiring the sample space to be immersed in liquid He. One solution is to use Cryofree® technologies to offer sample in vacuum arrangements. However, these type of systems do not lend themselves to integration with high field magnets. Heliox™VT (3He) and Kelvinox™VT (dilution refrigerator) inserts designed to condense using a VTI 1 K stage high in the bath away from the beam path and to only have a very diffuse level of exchange gas in the beam path. These achieve temperatures from 300 K to 300 mK and 300 K to 30 mK smoothly and continuously.

As these inserts use liquid He to condense during normal operation, they require a supply of helium from the main bath. This can be achieved using helium recondensing options which significantly reduce helium consumption and the dependence on liquid cryogen maintenance. It also allows integration of conventional ULT sample environments with high field split pair magnets for the best available extreme sample environments for Neutron Scattering applications.

KelvinoxVT dilution refrigerator insert

Pulse Tube Refrigerators

PTR cooling is based on periodic pressure variations and displacement of helium gas within a cylinder with heat exchangers at both ends. The PTR cooling process begins with a compression phase in which the cooled gas from the regenerator flows into the pulse tube. In a subsequent expansion phase, the temperature of the gas is further reduced thus cooling a load attached to the cold heat exchanger.

Sumitomo Heavy Industries PTR

The main advantage of PTRs over Gifford-McMahon cryocooler designs is that they have no moving parts in the low temperature region. Crucially this significantly reduces vibration and the need for maintenance.

High Temperature Performance

It is desirable to be able to measure multiple decades in temperature when characterising a sample, without moving the sample or changing cryostat. For wide temperature ranges, there is a variety of options:

In close collaboration with the new neutron source FRM-2 in Garching, VeriCold Technologies, an Oxford Instruments company, has developed a **pulse tube based 4 K closed cycle refrigerator** compatible with various inserts. Recently the system has been redesigned for use with a confocal microscope.

Full automation and fast sample cooldown times maximize your productivity. For sample characterisation, magnetic fields up to 9T can be applied at the sample position. Windows for direct sample characterisation (e.g. optical, X-ray, neutron) can be integrated into the system as an option.

For **low temperature requirements**, the **Heliox**AC-V offers Cryofree operation at the touch of a button. Offering base temperatures < 300 mK and hold times in excess of 50 h, this 3He system is ideally suited to Neutron Scattering applications. This system is fitted with thin wall, seamless aluminum tails and operates up to 300 K as standard.

The patented 3He technology developed on the **Heliox**AC-V has led Oxford Instruments to our latest low temperature system, the **Triton**DR.

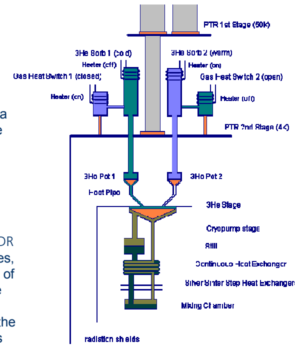
Cryofree® Dilution Refrigerators

Triton™DR Dilution Refrigerator

Fully automated and under computer control, TritonDR cools to 4 K using a PTR as the cooling platform and then utilizes patented 3He technology to cool to < 50 mK.

A reciprocating dual sorption pumped 3He stage cools from 4 K to < 400 mK. While one of the sorbs is cooled and pumps one of the 3He pots, the other is warmed and regenerates its 3He charge ready to switch and maintain continuous cooling. The 3He stage operating continuously at 400 mK acts as a cryopump circulating the self contained 3He/4He mixture, cooling the mixing chamber < 50 mK.

Having additional cooling at 400 mK allows the interception of heat from experimental services more efficiently than in traditional dilution refrigerators. This reduces the need for large footprint, high cooling power inserts. The TritonDR Is particularly well suited for experimental regimes, like Neutron Scattering, dissipating low amounts of heat at base temperature but requiring moderate service access to the mixing chamber. As the refrigeration cycle is closed loop and internal to the cryostat, no pumps or gas handling equipment is required making the system both compact and portable.



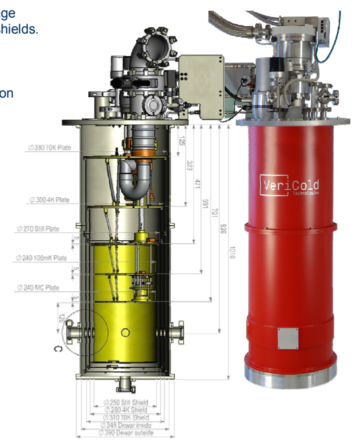
Summary

- 50 mK Cryofree base temperature, 10 μW at 100 mK cooling power
- 300 K operation
- High cooling power intermediate stage at 400 mK
- Low vibration pulse tube refrigerator
- No pumps or gas handling system
- Patented self-contained cryogenic cycle for leak-free reliable operation
- No needle valves or impedances

VeriCold DR 200-10 Dilution Refrigerator

In September 2007 VeriCold Technologies GmbH became part of Oxford Instruments. This has introduced a second Cryofree dilution refrigerator to the OI product portfolio. Like TritonDR, the VeriCold DR 200-10 uses a 4 K PTR to provide 1st and 2nd stage cooling of the refrigerator and radiation shields.

Unlike TritonDR, the VeriCold products operate using a conventional dilution circulation process with a Joule-Thompson stage, providing cooling of the returning 3He mixture from 4 K to below 1 K. This means that the system has conventional gas handling and pumping systems, however, it reaches a lower base temperature of 10 mK.



Summary

- 10 mK Cryofree base temperature,
- 200 μW at 100 mK cooling power
- 300 K operation
- Oil free operation
- Single vacuum
- No IVC, no Indium seal

VeriCold Technologies
An Oxford Instruments Company

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Split Pair Magnets for Neutron Scattering

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A key technique used to probe nano-materials, biological species and novel states of matter is Neutron Scattering.

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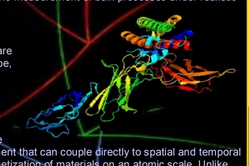
Neutrons scatter from materials by interacting with the nucleus of an atom rather than the electron cloud. This means it is easier to sense light atoms, such as hydrogen, in the presence of heavier ones. Neighboring elements in the Periodic Table can be distinguished and isotopic substitution can be used to label different parts of the molecules making up a material.

The interaction of a neutron with the nucleus of an atom is weak, making them a highly penetrating probe. This allows the investigation of the interior of materials, rather than the surface layers probed by techniques such as X-ray scattering, electron microscopy or optical methods. This feature also makes the use of complex sample environments such as cryostats, furnaces and pressure cells quite routine, and enables the measurement of bulk processes under realistic conditions.



Because of this weak interaction, neutrons are a non-destructive probe, even to complex and delicate biological or polymeric samples.

Neutrons are spin-1/2 particles and therefore have a magnetic moment that can couple directly to spatial and temporal variations of the magnetization of materials on an atomic scale. Unlike other forms of radiation, neutrons are ideally suited to the study of magnetic structures.

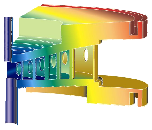
Interleukin-12 protein molecule: DR TIM EVANS / SCIENCE PHOTO LIBRARY
Cloud Chamber Showing Neutrons: LAWRENCE BERKELEY LABORATORY / SCIENCE PHOTO LIBRARY

Magnet Design

Finite Element Analysis

Finite element modelling of the magnet structure allows our engineering team to predict and control the electro-magnetic forces within the coil structure.

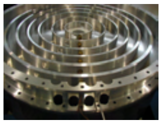
Forces and stored energies within split pair magnets can be extremely high making fundamental coil design a key step in providing magnetic environments for Neutron Scattering.



Former Design

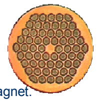
Once modeled the next step in creating a magnet structure is the manufacture of the magnet former. The former is custom built in collaboration with the customer and end users. This is to ensure beam access and scattering angles are suitable for the application and within the design tolerances allowed by the finite element analysis.

For Neutron Scattering, aluminium rings are used to space the coil sets with nested coils providing scattering angles in both horizontal and vertical directions. A dark angle of a few degrees is required to provide access for cryogens and services to the coil structure.



Wire

The best high field performance attainable in Nb_3Sn is provided by Oxford Superconducting Technology's Rod Restacked Process (RRP^{TM}) wire. A focused development effort, over the last 10 years, has resulted in a 3-fold increase in critical current density at 20 T in this wire. This performance has enabled two world-record setting magnets: a 950 MHz NMR magnet, and a 16 T dipole magnet.



Quench Management

Great care is taken to understand the stored energy and quench mechanisms in high field split pair. During a quench, currents can be induced in the opposite coil from the quenched one, generating local currents well in excess of the wire's short sample limit. These localized high currents can also generate stress in the wire well beyond the design limit of the magnet. To avoid such failure modes quench heaters are fitted to force even quenching throughout the magnet and safe decay to zero field through the protection circuit.

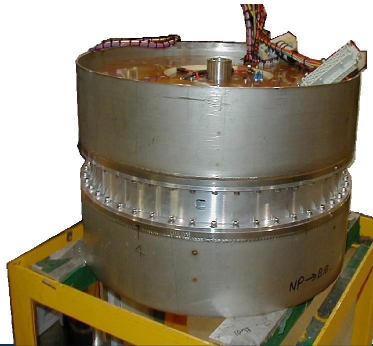
Coil Structure

Typical performance specifications are:

- Field strength of up to 15 T at 2.2 K and 13.5 T at 4.2 K
- Field orientation: Vertical with split geometry
- Homogeneity over 10 mm DSV: 0.5%
- Split at magnet centre line: 20 mm
- Split angle ±2 degree
- Neutron access in the horizontal plane: 330 degree

Other coil geometries are possible and have been demonstrated at lower fields. Examples are 4 coil large access magnets, asymmetric split pairs for polarised neutron studies and actively shielded coils for minimised laboratory stray field.

Almost 50 years of experience has shown us that with a combination of skilled design, the highest quality wire and careful manufacturing processes, superconducting split pair coils with fields up to 15 T can operate in extreme environments purpose built for Neutron Scattering. These coil structures represent the state of the art in high field split coil design.



Cryogenic Design

Recondensing Systems for Neutron Scattering

ActivelyCooled™ magnet technology is a helium recondensing technology that reduces dependency on liquid cryogens. The principle of using cryocoolers to recondense cryogens and eliminate LHe boil off is well established. Low vibration Pulse Tube Refrigerators (PTR) are now commercially available.

The alternative of using cryocoolers for direct magnet cooling is also well established. However, a "dry" configuration does not offer the security of a liquid helium volume in the event of a power or other failure and presents additional challenges in the vibration isolation of the magnet.

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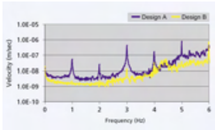
Sumitomo Heavy Industries PTR

Vibration Isolation

Vibration is an issue of central importance in most measurement techniques. The PTR, must not only be coupled closely to the magnet system to function cryogenically, but also have vibration isolation.

In order to understand the transmission paths for vibration, accelerometers were attached at various points around a development cryostat. The most significant transmission path was found to be from the PTR 'sock' through to the upper part of the Helium vessel. Alternative configurations of PTR sock and cryostat were evaluated in order to reduce the low frequency vibrations. As can be seen Design B resulted in the transmission of lower magnitude accelerations in the critical low frequency range.



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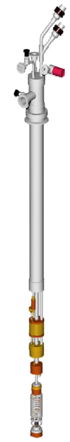
KelvinoxVT dilution refrigerator insert

Summary

- Split coil magnetic fields up to 15 T
- Sample temperatures from 300 K to 30 mK
- Customised Al-ringed coil formers for Neutron Scattering
- Recondensing over Cryofree® technology to minimise helium usage without compromising performance.

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