



Sample Environment in ISIS Spallation Neutron Source

Oleg Kirichek & Sample Environment Group

*ISIS Facility, STFC, Rutherford Appleton Laboratory,
Harwell, Didcot, UK*



Science & Technology
Facilities Council

NMI3 JRA6 Sample Environment High Pressure Task

Inert Gas Cells

Current situation:

- **6 kbar** automated gas handling system for inert gases
- **13.8 kbar** 'oil' intensifier for hydraulic testing
- gas cells up to **5.5 kbar**

Objectives:

1. **15 kbar** 'oil' intensifier for hydraulic testing
2. **10 kbar** automated gas handling system for inert gases
3. Design and produce cells and test seal systems up to **8 kbar** (LT–300 K)
4. Design and prototype **10 kbar** cells for 300 K



Hydrogen Cells

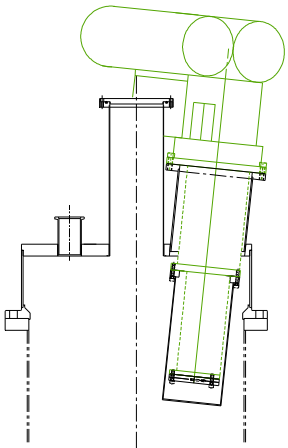
Current situation:

- **3 kbar** H₂ intensifier and gas handling system
- Inconel cell **2 kbar** and up to **700 K**,
- Inconel cell **3 kbar** at **300K**

Objectives:

- Materials – H₂ compatibility/neutron transmission properties
- Sourcing and assembly of **8 kbar** H₂ intensifier and gas handling system
- Produce and test cell for **4 kbar** and up to **700 K**
- Produce and test cell for **6 kbar** at **300 K**
- Design and prototype **8 kbar** cells for **LT – 300 K**

Cryo-free cryostat 1.5 – 600K based on Pulse Tube Refrigerator



1st Stage: *Pulse Tube Refrigerator* (completed):

Base temperature: 2.41 K; Cooldown time: 54min; Level of vibrations was considerably less than that produced by GM coolers.

2nd Stage: *Conventional Top Loader* (completed):

Base temperature on the sample holder 3.7 K

3rd Stage: *1.5K Top Loader*:

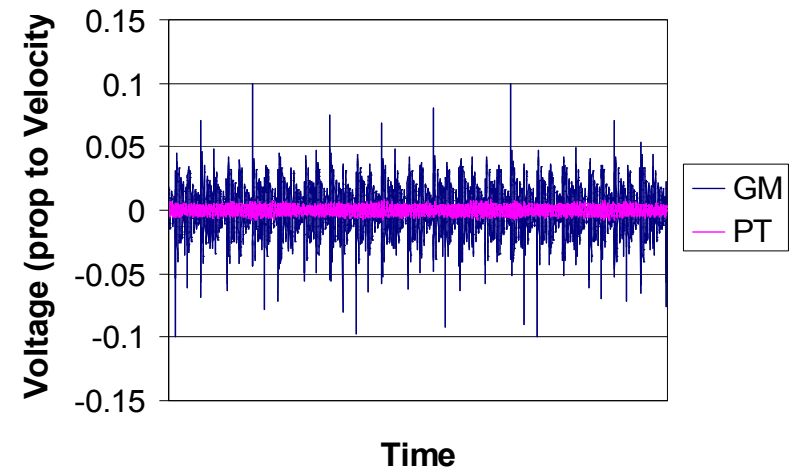
First prototype achieved *2.3K*. After the test the design has been modified. The test of the prototype 2 is planned for the summer of 2008

Comparison between GM & PT CCR Toploaders (GM: Sumitomo 1.5W@4.2K)

Meas. Sci. Technol. **19** (2008) 034018

Property	PT	GM
Base sample temperature	3.6 K	4.4 K
Time to cooldown to base	<3 hrs	≈4 hrs
Cooldown after sample change	<1.25 hrs	<1.25 hrs
Initial cost of CCR system	\$38,000	£19,000
Maintenance costs - coldhead	--	£5,000 @ 9,000 hrs
Maintenance costs - compressor	£2,500 @20,000hrs	£2,500 @20,000hrs

Vibration
measurements





ISIS Advanced Magnets Project

The *ISIS Advanced Magnets Project* started with a submission to the facilities development fund of the CCLRC. In July 2005 a positive response was received and funding was confirmed in June 2006. The project was split into two parts to spread the costs:

Part-I four magnets:

Wide Angle Chopper Instrument Magnet for Spectrometry

14T Superconducting Magnet for Diffraction

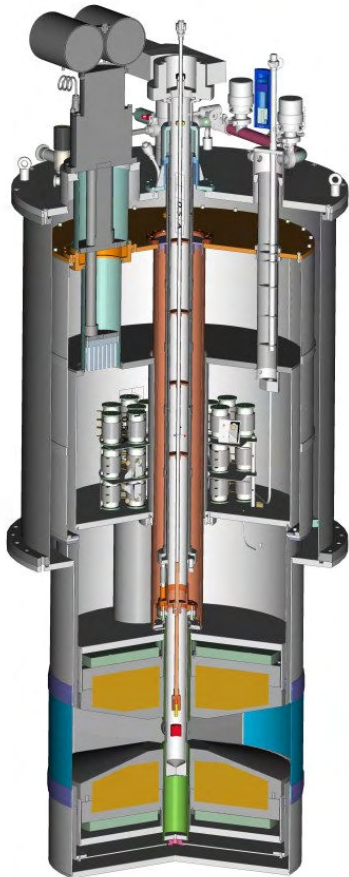
3D Magnet for the reflectometers and lowQ Instruments

Pulsed Magnet Development

Part-II will further expand facilities at ISIS and will be submitted after making some progress on Part-1.



9T Wide Angle Chopper Magnet

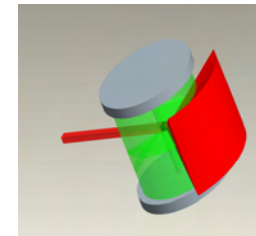
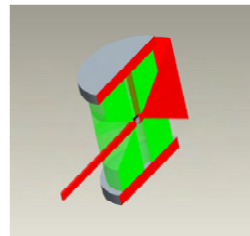


9T Wide Angle Chopper Magnet with wide detector coverage $\pm 15^\circ$ in the vertical plane and $\pm 40^\circ$ in horizontal plane.

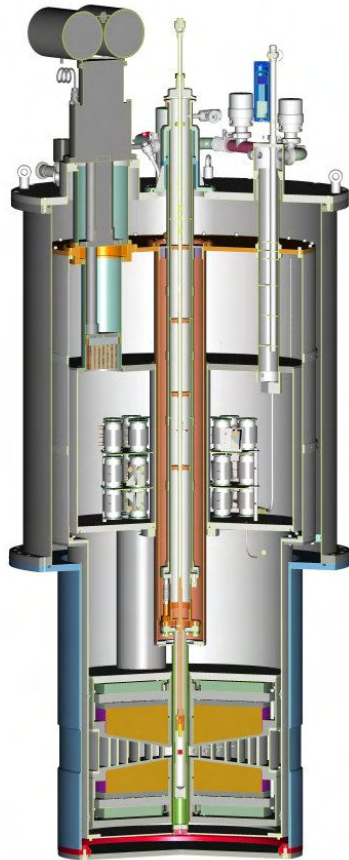
Our aim is to achieve the widest aperture possible with the highest achievable field.

Contract has been given to *Oxford Instruments*.

We expect to have the magnet available for use in 2009.



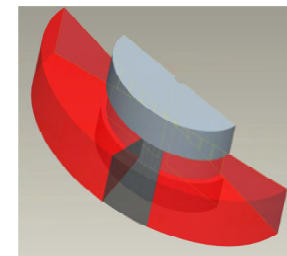
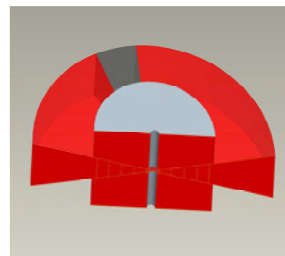
14T Superconducting Magnet



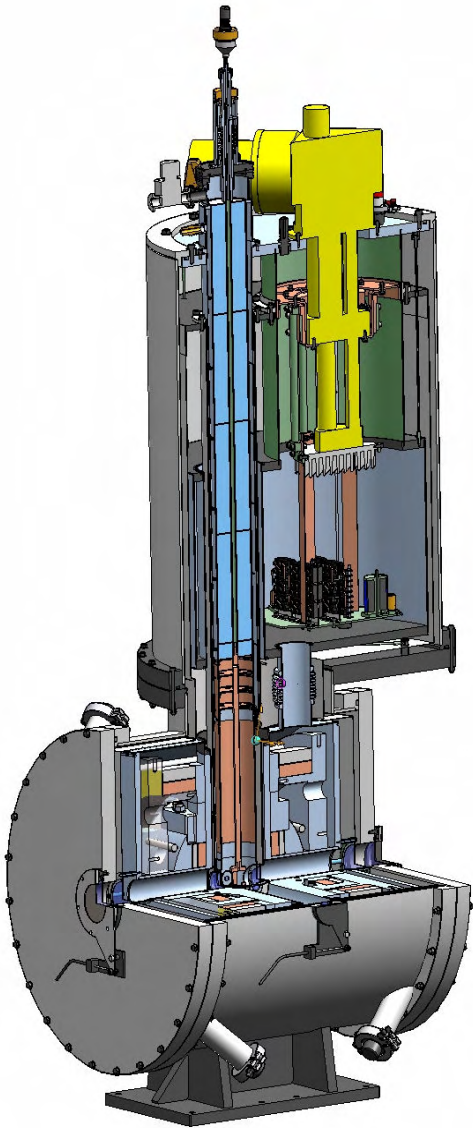
The state of the art for a split pair magnet is 15T, but by reducing the maximum field to *14T* it is possible to increase the *detector viewing angle from $\pm 3^\circ$ to $+10^\circ$ and -5°* . The addition of dysprosium booster would enable fields up to *$\sim 16T$* .

Contract has been given to *Oxford Instruments*.

This magnet expected to be delivered early in 2009.



3D magnet

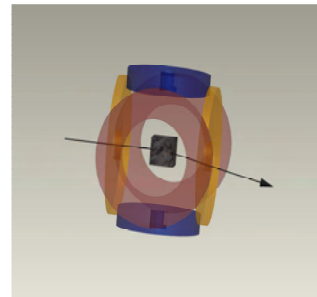


3D magnet will allow the application of a magnetic field in any direction.

The maximum magnetic field is expected to be $2T$ in all directions.

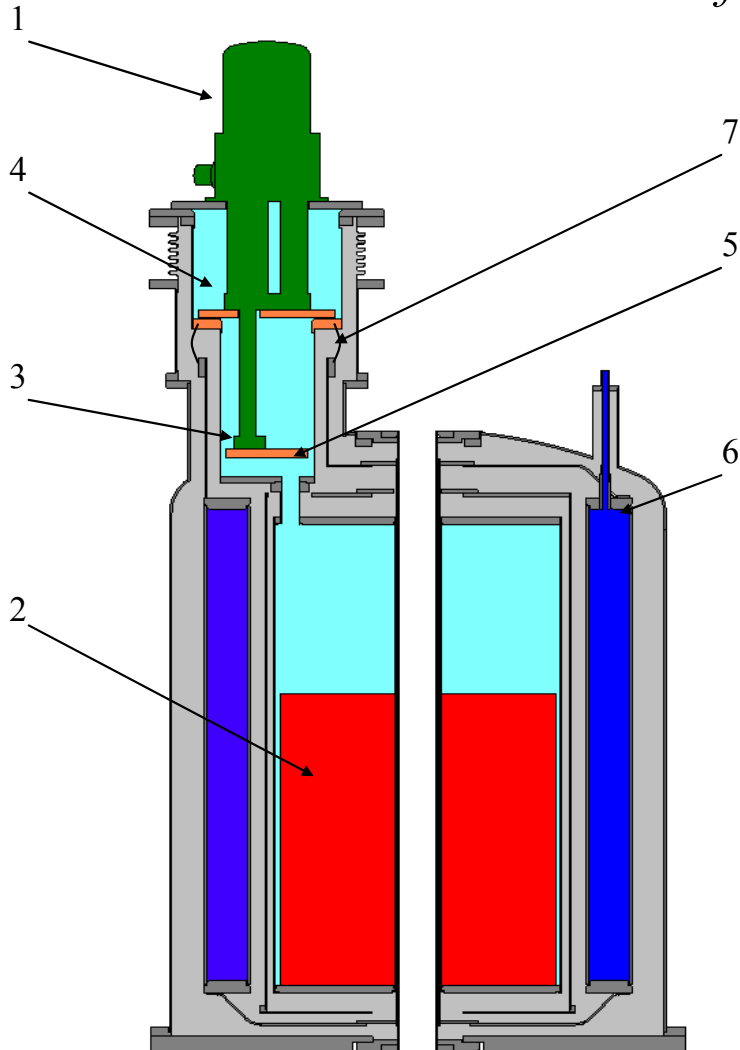
Contract has been given to *Scientific Magnetics*.

We hope to run this magnet before December 2008.



Pulse Tube Refrigerator re-condensing technology

Review of Scientific Instruments **76**, 2005, p. 055104



1. Pulse Tube Refrigerator (PTR)
2. Superconducting magnet
3. Second stage of PTR (~ 4K)
4. First stage of PTR (~ 40K)
5. Condenser
6. Infrared radiation shield (~ 50K)
7. Flexible thermal links (copper braids)

Pulsed Magnetic Fields

(Joint project: Oxford University and ISIS Facility)

Within 3 years Oxford University will develop and build a prototype **Pulsed Magnet** running at **>20T** and **2Hz** with **2ms** pulse width.



Science Case:

- magnetic field induced structural transformations
- excitations in high temperature/high magnetic field superconductors
- dynamical and structural properties of complex organic and biological systems e.g. alignment of polymers and self-assembly of biological macromolecules.

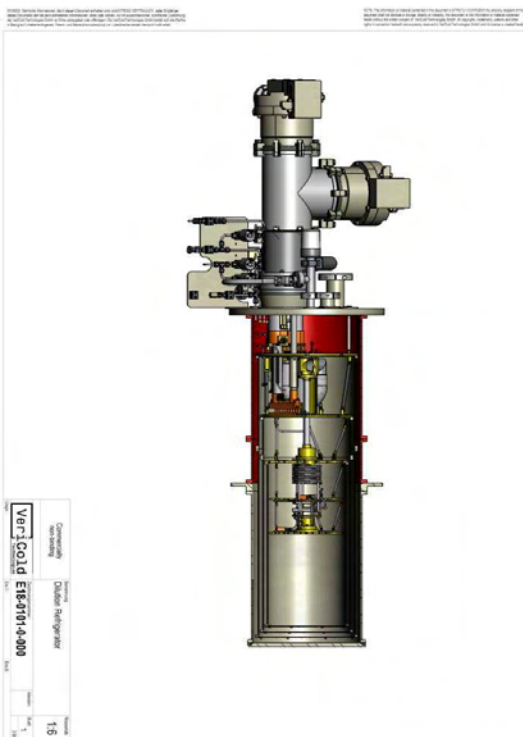
ISIS Instrument:

TS2 instrument: **eXess** the extreme sample environment instrument (the instrument may require some changes to make it operational with pulsed magnetic fields).

Powerful Cryo-free Dilution Refrigerator for Low Temperature Sample Environment

**VeryCold Oxford Instruments
Cryo-free Dilution Refrigerator**

**Kelvinox VT
A sample-in-vacuum Dilution Refrigerator**



Cooling power $\geq 400 \mu\text{W}$!

Base temperature $\leq 20 \text{ mK}$

Sample space: Vertical Cylinder
 $\text{\O} 250 \text{ mm}$; height $\sim 250 \text{ mm}$

Fully automated operation!



Cooling power $30 \mu\text{W}$

Base temperature $\approx 25 \text{ mK}$

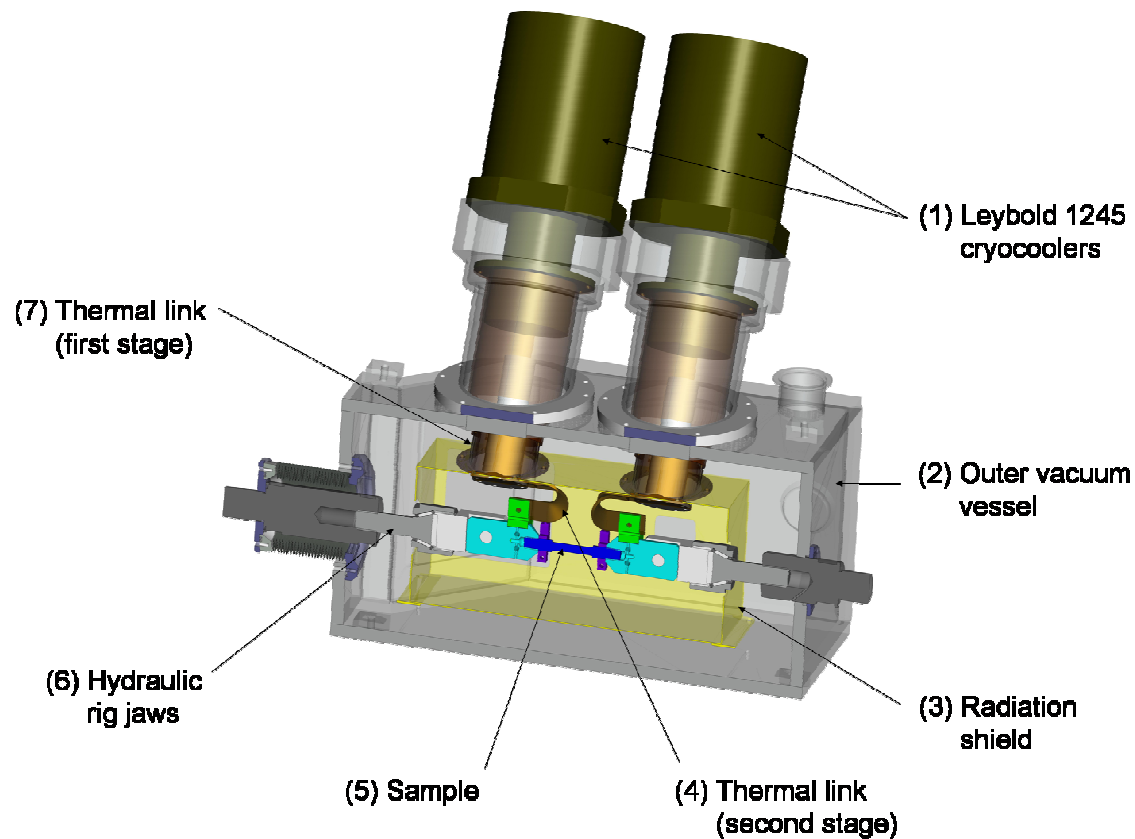
Sample space: Vertical Cylinder
 $\text{\O} 38 \text{ mm}$; height 80 mm

Expected delivery - June 2008

ISIS Sample environment group has:
4 off Kelvinoxes VT + 1 off TBT DR

Stress rig for neutron scattering measurements of bulk stress in engineering components at cryogenic temperatures

Meas. Sci. Technol. **19** (2008) 034019



The achieved technical specification:

- sample cooling to 30K;
- applied loads up to 50kN;
- access for incident and diffracted neutron beams at 45° to the tensile loading axis.