

# International Workshop on Sample Environment at Neutron Scattering Facilities



#### Workshop booklet assigned to:

. ,



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## **General Information**

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#### **Onsite Logistics**

#### **Registration Hours**

Sunday, May 25: the registration desk is located in the main lobby area of the ILL building ILL4. Registration is available from 16:00 to 20:00.

Monday, May 26: the registration desk is located in the main lobby area of the ILL building ILL4. Attendees are invited to register from 8:00 to 8:30.

#### **Workshop Pack**

Each attendee receives a pack upon check-in at the Registration Desk. This pack contains this workshop booklet, a badge, an ILL tee-shirt, a pen and the ILL 2007 Annual Report.

#### Food

On Sunday, May 25, dinner items will be provided in the main lobby of the ILL4 from 19:00 to 20:00.

Breakfast, lunch and dinner meals will be provided for registered workshop attendees at the conference hotel "Le Grand Hôtel de Paris" in Villard de Lans. A continental breakfast will be served on Tuesday and Wednesday from 7:00 to 8:00. Lunch time is from 12:30 to 13:30, Monday through Wednesday. Pick up a cup of coffee or tea during the workshop breaks.

On Monday, May 26, a dinner will be served at the conference hotel in the Vercors lounge.

On Tuesday, May 27, the workshop attendees will be invited to a special workshop dinner that will take place at the "Auberge des Allières".

The refreshments will be offered at the bar of the "Grand Hôtel de Paris" on Monday evening.

Special meals are available by prior arrangement.

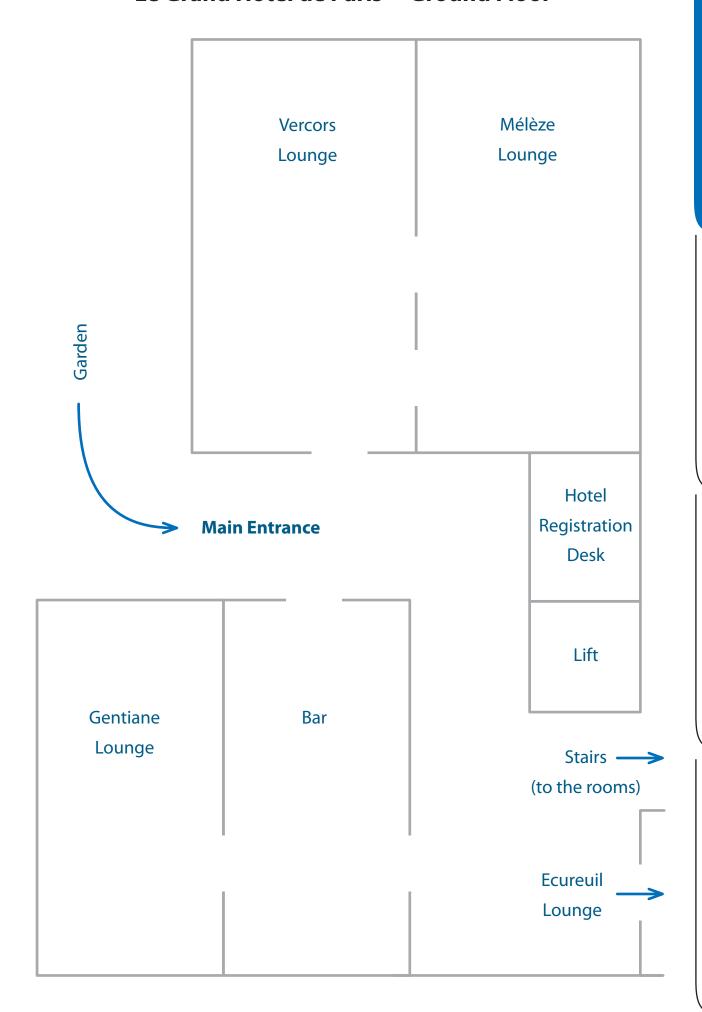
#### Lost and Found

The workshop organisers and the hotel management team are not responsible for lost, damaged, or stolen property or equipment. If you have lost an item, check with the staff at the registration desk of the hotel and with the workshop organiser. If you find anything, please turn it to the staff at the hotel registration desk.

#### **Network Connectivity**

SE@NSF 2008 attendees will have a free access to a wireless network to bring them a convenient and ready Internet access. Please go to the registration desk of the hotel, give your room number and ask for a WiFi Access card. The WiFi network is available in the ground floor and in some rooms of the first and second floors.

#### "Le Grand Hôtel de Paris" - Ground Floor



#### **Transportation and Directions**

#### Travelling to the Institut Laue Langevin

By car - <a href="http://www.mappy.com">http://www.viamichelin.com</a>

Arriving by motorway from Paris/Lyon, Milan/Geneva, Barcelona/Marseille, at Grenoble, follow signs for "Gare EUROPOLE" or "Polygone scientifique Louis Néel". Please use the Mappy or Michelin web sites for getting a map.

By train - <a href="http://www.voyages-sncf.com">http://www.voyages-sncf.com</a>

You can consult the up-to-date timetables and book your tickets on the SNCF (French railway service) web-site. Please note that seat reservations on the TGV are obligatory; they should be made when you purchase your ticket. It is also possible to phone the SNCF for further details: +33/0 8 36 35 35.

By air to Lyon - Saint Exupéry airport (LYS) - <a href="http://www.lyon.aeroport.fr">http://www.lyon.aeroport.fr</a>

You can phone the airport from France: 0 826 800 826, or from abroad: +33 426 007 007. When travelling from Lyon-Saint Exupéry airport to Grenoble, tickets can be bought either from the "Satobus" counter inside the airport, or directly from the driver. A single ticket costs about 20€, a return ticket about 30€ (see <a href="http://www.satobus-grenoble.fr/">http://www.satobus-grenoble.fr/</a>).

The bus leaves from the coach situated between Terminals 1 and 2, Level 1. There is an official stop at the "Place de la Résistance" (near the "Pont d'Oxford" roundabout), approximately 5 minutes walk distance from the ILL.

By air to Grenoble St. Geoirs (GNB) then bus to Grenoble - http://www.grenoble-airport.com

The airport bus meets each scheduled flight. For departures the bus leaves from the bus station 75 minutes before each scheduled departure. There are fewer buses during the summer months (end March to end October). Please check with the ILL Travel Office for any changes (travel@ill.eu). A single ticket costs 13 Euros, but it is cheaper to buy a return ticket (20 Euros).

By air to Paris then TGV train to Grenoble - <a href="http://www.voyages-sncf.com">http://www.voyages-sncf.com</a>

There are a number of TGV connecting Paris airports to Grenoble. You can consult the up-to-date timetables and book your tickets on the SNCF (French railway service) web-site. Please note that seat reservations on the TGV are obligatory.

By air to Geneva (GVA) then by train or bus - <a href="http://www.gva.ch">http://www.gva.ch</a>

Take one of the trains from the airport to Geneva Central Station (Cornavin) and change at Cornevin for a train to Grenoble. AEROCAR runs 3 coaches a day. See information (in French only). You can also book your tickets on line at <a href="http://www.altibus.com">http://www.altibus.com</a>.

#### Local Grenoble buses

Take the #34 marked "Polygone Scientifique" or "Sassenage Le Chateau", from the bus stop across the road from Grenoble railway/bus station. This will take you right to the ILL entrance.

You can also take the #30 from behind the station - Europole side, destination "St Egrève". This drops you off at the "Place de la Résistance" roundabout, where you have a few minutes walk to the site.

Journey times for both buses is about 10 minutes and there is a bus every 10-15 minutes throughout the day until about 20:15. Tickets can be purchased singly from the bus driver or in a booklet ("carnet") at tobacconists and major tram stops. For more information, you can visit the SEMITAG web site: <a href="http://www.semitag.com/index.php?id=3">http://www.semitag.com/index.php?id=3</a>.

#### Travelling back home

By air to Lyon - Saint Exupéry airport (LYS) - http://www.lyon.aeroport.fr

To go to the airport the Satobus coach leaves from the central bus station ("gare routière") next to Grenoble train station. It can also pick up passengers at the "Place de la Résistance" roundabout close to the ILL. The pick-up point is situated on the bridge-side of the roundabout. You will see "Satobus" written on the sign at the bus stop - it is the same stop as for the #30 and #715 buses to St-Egrève and Voiron. Do not confuse this stop with the others near the roundabout which are used by the local buses.

Please make a sign to the driver as the coach approaches (approx. 5 minutes after departure from the railway station). Tickets can be bought from the driver with cash or by cheque (French banks). For further information call "Cars Faure" +33/0 4 76 27 89 29.

#### **Workshop Hotel**

The workshop takes place at the hotel "Le Grand Hôtel de Paris" - <a href="http://www.ghp-vercors.com">http://www.ghp-vercors.com</a>. It is located at 35km from Grenoble in the Vercors mountain. At the very centre of Villard de Lans, in a 3 hectare private park, this is a beautiful place for enjoying the nature and sharing lively discussions. Within the hotel, you will find a restaurant, a bar with a pleasant atmosphere, a wireless network, lifts, parking places and a tennis court.

About 150m away from the hotel, there is a shopping area, a water center (sauna, hammam, jacuzzi), a casino, a bowling and a disco. There is also a 18-hole golf course located at the entrance of the Natural Reserve of the Vercors area (7km).

#### Transportation to Villard de Lans

On Monday, May 26, after a quick tour of the ILL, attendees will be invited to take a shuttle bus that will transport them to the Workshop Hotel. The coach will leave at 11:00 from the entrance of the ILL site.

#### Transportation to Grenoble

On Wednesday, May 28, attendees will be transported back to Grenoble with a shuttle bus. The coach will leave at 15:00 from the hotel "Le Grand Hôtel de Paris".

#### **Workshop Policies**

#### Presenting

The size of the posters to be displayed at the Poster Sessions is A0-Portrait. Tools will be provided for their fixation on the panels. Attendees are asked to send original electronic versions in PDF format to <a href="mailto:SEatNSF@ill.eu">SEatNSF@ill.eu</a> so that they can be included in the Workshop DVD.

Speakers will use a computer connected to a projector for their presentation. The formats of the files that can be displayed are: Keynote (Mac OS), PowerPoint (Mac OS, Windows) and Acrobat. Electronic copies of the presentations will be included in the Workshop DVD.

#### **Smoking**

Smoking is not permitted anywhere inside the Institut Laue Langevin and the hotel. Indeed, in France, it is not permitted to smoke in public areas except outside.

#### Taping and Photographing

Videotaping and audiotaping of workshop sessions will be performed by the organisers. By registering to the workshop, attendees accept to appear in the Workshop DVD that will be pressed after the workshop.

#### **Dress Code**

There is no specific dress code. For the Workshop Dinner, as we will be above 1400m altitude in the Vercors mountain, it is strongly advised to take a woollen article, may be a rain coat, and good shoes so that the 20-30min walk planned does not become a pain.

#### **Sponsors**

We thank very much the following entities for their active support without which this workshop could not have been organised:

Advanced Research Systems Inc.

http://www.arscryo.com

Bruker BioSpin AG

http://www.bruker-biospin.com

Institut Laue Langevin

http://www.ill.eu

Integrated Infrastructure Initiative for Neutron and Muon Scattering (NMI3 - FP6)

http://www.neutron-eu.net

Oxford Instruments NanoScience

http://www.oxinst.com

SDMS

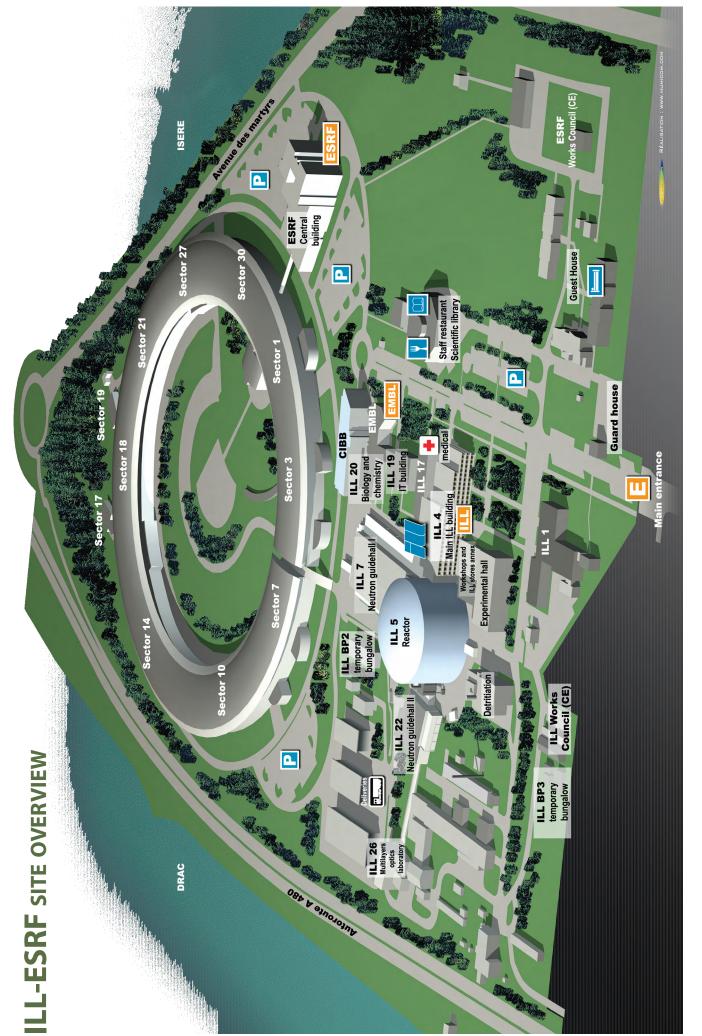
http://www.sdms.fr

SNLS

http://www.snls.fr

TransMIT GmbH

http://www.cryo.transmit.de





## Workshop Features

Take a Break
Exhibit Fair
Agenda at a Glance

#### Take a Break

#### **Coffee Breaks**

The coffee breaks served in the Gentiane lounge or in the garden of the Grand Hôtel de Paris are offered by the following company representatives:

Monday, May 26 16:00 Advanced Research Systems, Inc. Dr. Ravi BAINS rbains@arscryo.com

http://www.arscryo.com

Tel: +1 610 967 2120 Fax: +1 610 967 2395

Tuesday, May 27 Bruker BioSpin AG Dr. Raffaele GILARDI 10:00

http://www.bruker-biospin.com

raffaele.gilardi@bruker.ch Tel: +41 44 825 9561 Fax: +41 44 825 9260

16:00 **Oxford Instruments Nanoscience** Mr. Phil PICKERING

> http://www.oxinst.com phil.pickering@oxinst.com Tel: +44 1865 393 434

Fax: +44 1865 393 333

Wednesday, May 28 TransMIT GmbH 10:00 Prof. Dr. Guenter THUMMES http://www.cryo.transmit.de

thummes@transmit.de Tel: +49 641 99 33460 Fax: +49 641 99 33409

#### **Workshop Dinner**

The workshop dinner will take place in the "Auberge des Allières" near Lans en Vercors (altitude 1476 m - http://www.aubergedesallieres.com/auberge/auberge.php). After a Champagne apéritif, attendees will be invited to share a meal made from local specialities. The Champagne labels shown below are offered by the producer Claude Carré and the Workshop Dinner is offered by the participating company:

SDMS / SNLS

http://www.sdms.fr / http://www.snls.fr

Mr. Xavier SAUGE

sauge@sdms.fr Tel: +33 476 649 973 Fax: +33 476 649 998



#### **Exhibit Fair**

Located in the Gentiane lounge of the Grand Hôtel de Paris, the Exhibit Fair is a great place to see and discuss about the latest products, services and technologies. Participating companies include the following:



#### Advanced Research Systems, Inc.

7476 Industrial Park Way, Macungie, PA 18062, USA Tel: +1 610 967 2120, Fax: +1 610 967 2395 http://www.arscryo.com rbains@arscryo.com



#### A S Scientific Products Ltd

2 Barton Lane, Abingdon Science Park, Abingdon, Oxfordshire OX14 3NB, UK Tel: +44 1235 533 060, Fax: +44 1235 554 125 http://www.asscientific.co.uk enquiries@asscientifc.co.uk



#### **Bruker BioSpin AG**

Industriestrasse 26, 8117 Fällanden, Switzerland Tel: +41 44 825 9111, Fax: +41 44 825 9626 http://www.bruker-biospin.com sales@bruker-biospin.ch



The Business of Science®

#### **Oxford Instruments NanoScience**

Tubney Woods, Abingdon, Oxfordshire OX13 5QX, UK Tel: +44 1865 393 200, Fax: +44 1865 393 442

http://www.oxinst.com NanoScience@oxinst.com



#### **SDMS**

ZI Les Condamines, BP 4, 38160 Saint-Romans, France Tel: +33 476 649 999, Fax: +33 476 649 998 http://www.sdms.fr sdms@sdms.fr



#### **SNLS**

ZI Les Condamines, BP 3, 38160 Saint-Romans, France Tel: +33 476 381 833, Fax: +33 476 382 991 http://www.snls.fr snls@snls.fr



#### **TransMIT Center for Adaptive Cryotechnology and Sensors**

Heinrich-Buff-Ring 16, 35392 Giessen, Germany Tel: +49 641 99 33460, Fax: +49 641 99 33409

http://www.cryo.transmit.de thummes@transmit.de

#### Agenda at a Glance

#### Sunday, May 25

16:00RegistrationInstitut Laue Langevin<br/>ILL4 - main lobby area19:00Welcome reception<br/>dinner items providedInstitut Laue Langevin<br/>ILL4 - main lobby area

#### Monday, May 26

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08:00	Registration	Institut Laue Langevin ILL4 - main lobby area
08:30	<b>Opening Session</b> by E. Lelièvre-Berna	Institut Laue Langevin ILL4 - Chadwick Amphitheater
09:00	<b>ILL Tour</b> organised by I. Te Gröen	Institut Laue Langevin Experimental Halls
11:00	<b>Departure for Villard de Lans</b> with "Les Cars du Vercors"	Shuttle Bus Grenoble - Villard de Lans
12:30	Lunch	Grand Hôtel de Paris Vercors lounge
14:00	Session "Projects, Facts and Figures from Neutron Scattering Facilities" - chaired by E. Lelièvre-Berna	Grand Hôtel de Paris Mélèze lounge
	"A Review on 20 years of Sample Environment: Men & Machines - 1988 to 2008"	<i>M. Meißner</i> HMI - Germany
	"Sample Environment in ISIS Spallation Neutron Source"	O. Kirichek ISIS RAL - UK
	"Recent Sample Environment Innovations for SANS"	S. Olsen ANSTO - Austrialia
	"Sample Environment at FRM-II"	J. Peters FRM-II - Germany
16:00	Break offered by Advanced Research Systems, Inc.	Grand Hôtel de Paris Gentiane lounge
16:30	Session "Projects, Facts and Figures from Neutron Scattering Facilities" - chaired by Z. Bowden	Grand Hôtel de Paris Mélèze lounge
	"Sample Environment at the Swiss Spallation Source SINQ"	<b>M. Zolliker</b> PSI - Switzerland
	"Experience with Cryofurnaces at Oak Ridge National Laboratory"	<b>L. Santodonato</b> ORNL - USA
	"Modernising the ILL Sample Environment Suite"	<i>E. Lelièvre-Berna</i> ILL - France
19:00	Dinner	Grand Hôtel de Paris Vercors lounge

#### Tuesday, May 27

07:00	Breakfast	Grand Hôtel de Paris Vercors lounge
08:30	Session "In the Field" chaired by L. Santodonato	Grand Hôtel de Paris Mélèze lounge
	"Self-Shielded Magnets for Neutron Scattering"	A. Church SNS - USA
	"High Pressure for Neutron Scattering at Low Temperature and High Magnetic Field"	<b>R. Sadykov</b> INR, IHPP - Russia
	<i>"1000°C in a Superconducting 5T Magnet"</i>	<b>C. Redmon</b> HFIR - USA
	"Sample Cooling in the forthcoming 25T-Hybrid-Magnet - Requirements, first Sketches and Approaches"	<i>J. Heinrich</i> HMI - Germany
10:00	Break offered by Bruker BioSpin AG	Grand Hôtel de Paris Gentiane lounge
10:30	Session "Opening Scientific Fields" chaired by J. Peters	Grand Hôtel de Paris Mélèze lounge
	"Sample Environment in Soft Condensed Matter at ILL"	<b>G. Fragneto</b> ILL - France
	"Aerodynamic levitation and laser heating — Current and future developments at the ILL"	<i>L. Hennet</i> CNRS-CEMHTI - France
	"Superconducting Vortices in CeCoIn <sub>s</sub> : Toward the Pauli- Limiting Field"	<i>M. Kenzelmann</i> PSI - Switzerland
	"Neutron Scattering Investigations in Extreme Sample Environments"	<i>T. Chatterji</i> ILL - Grenoble
12:30	Lunch	Grand Hôtel de Paris Vercors lounge
14:00	<b>Session "Projects, Facts and Figures from Companies"</b> <i>chaired by M. Meißner</i>	Grand Hôtel de Paris Mélèze lounge
	"Cryocooler and Cryostat Options for Neutron Scattering"	<b>R. Bains</b> Advanced Research Syst. , Inc USA
	"World's Highest Field Actively Shielded Split Coil Magnet for Neutron Scattering"	<b>R. Gilardi</b> Bruker BioSpin AG - Switzerland
	"Oxford Instruments NanoScience"	<i>P. Pickering</i> Oxford Instruments - UK
	"Pulse Tube Cryocoolers: an Option for Cooling without Cryogenic Liquids"	<b>G. Thummes</b> TransMIT GmbH - Germany
16:00	Photo Session	Grand Hôtel de Paris Garden
16:15	Break offered by Oxford Instruments NanoScience	Grand Hôtel de Paris Gentiane lounge
16:45	Poster Session #1	Grand Hôtel de Paris Gentiane lounge
19:00	Departure for the Workshop Dinner	from the Grand Hôtel de Paris

#### Wednesday, May 28

07:00	Breakfast	Grand Hôtel de Paris Vercors lounge
08:30	Session "Under Control" chaired by S. Olsen	Grand Hôtel de Paris Mélèze lounge
	"Electronic Developments in ISIS Sample Environment in 2007"	<b>D. Cowdery</b> ISIS RAL - UK
	"Portable Automated Pressure and Temperature Control System"	<b>D. Maierhafer</b> HFIR - USA
	"Reconsidering ILL Cryostats Electronics"	<b>N. Belkhier</b> ILL - France
	"Window Wars: Quartz vs. Titanium"	<b>E. Fitzgerald</b> NIST - USA
10:00	Break offered by TransMIT GmbH	Grand Hôtel de Paris Gentiane lounge
10:30	Poster Session #2	Grand Hôtel de Paris Gentiane lounge
12:30	Lunch	Grand Hôtel de Paris Vercors lounge
14:00	Closing Session	Grand Hôtel de Paris Mélèze lounge
	"Comments on the fifth edition"	<b>M. Meißner</b> HMI - Berlin
	"Final conclusions and remarks - sixth edition"	<i>E. Lelièvre-Berna</i> ILL - Grenoble
15:00	<b>Departure for Grenoble</b> with "Les Cars du Vercors"	Grand Hôtel de Paris
16:15	Arrival at Grenoble	

## **Abstracts**

Oral Contributions
Poster Contributions

## A Review on 20 Years of Sample Environment: Men and Machines - 1988 to 2008

M. Meißner - Hahn Meitner Institut (Berlin, Germany)

meissner@hmi.de

Oral Contribution - Monday, May 26 - 14:00

will give a personal summary on the past two decades at BENSC where sample environment (SE) equipment has been developed for the new instruments at the refurbished 10-MW-reactor BER-II. The story begins with the early equipment at ILL and ISIS in the '80s of which BENSC benefited for a successful start in the early '90s. Later on, BENSC found its own field of expertise with the development of very low temperature devices which could be combined in a modular concept with various types of cryomagnets.

With the new millennium, the upgraded and new neutron scattering facilities provided financial budgets and man power resulting in new achievements in sample environment technology (i. e. cryogen-free, extreme high temperature and pressure). In addition, cooperation projects shared by various SE-teams and financed by international agencies will open new frontiers.

The increasing interaction among the SE community worldwide was triggered by the ENSA survey on instrumentation in 1995-1997 and initiated the first SE workshop in Berlin 1999. Now, facing the 5<sup>th</sup> workshop upcoming in Grenoble I am confident that sample environment equipment continues to be an essential and exciting part of neutron scattering instrumentation.

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#### **Sample Environment in ISIS Spallation Neutron Source**

O. Kirichek - ISIS Rutherford Appleton Labs (Didcot, United Kingdom)

o.kirichek@rl.ac.uk

Oral Contribution - Monday, May 26 - 14:30

Sample environment equipment such as cryostats, pressure cells, furnaces, magnets, stress rigs etc are an essential to most neutron scattering experiments to induce the sample being studied into a phase or state of particular interest. We are going to give a brief overview of the new sample environment equipment available for neutron scattering experiments in ISIS which should considerably expand the range of physical parameters such as high pressure, high magnetic field and ultra low temperature.

The equipment includes new top loading cryogen free cryostat based on pulse tube refrigerator, powerful cryogen free dilution refrigerator produced by Verycold Oxford Instruments, three advanced superconducting magnets for pulsed neutron scattering at ISIS and a stress rig for measurements of bulk stress in engineering components at cryogenic temperatures. All systems mentioned above are expected to be either helium re-condensing or completely cryogen free. Here we are going to describe the design of these systems and discuss the results of prototypes testing.

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#### **Recent Sample Environment Innovations for SANS**

S. Olsen, S.A. Pullen, E.P. Gilbert, F. Klose, M. Perry, J.C. Schulz & R.A. Robinson - Australian Nuclear Science & Technology Organisation (Sydney, Australia)

sol@ansto.gov.au

Oral Contribution - Monday, May 26 - 15:00

Over the last 18 months 2 new Sample Environments have been developed for use on the Quokka Small Angle Neutron Scattering machine. The first device is a joint development between ANSTO and New Zealand company HTS-110, a 5 Tesla Horizontal field Closed-Cycle High Temperature superconducting magnet, made of BSSCO 2223 material. This magnet mounts on tilt stages and has the sample mounted in a closed-cycle cryo-furnace. An electric field can be added to the sample whilst inside the magnet.

A device for rapidly heating and cooling samples, the RHQC, has been developed, and tested at IPNS. This device allows samples to be quenched at rates of up to 11K/sec and heated at 19K/sec in a temperature range of -120°C to 300°C. This device contains a cold cell and a hot cell and the sample is moved, pneumatically, between the cells. The device is mounted on a translation stage allowing either cell to be in the neutron beam. Multiple heating and cooling cycles can be run in the one experiment.

Notes:

#### **Sample Environment at FRM II**

J. Peters - Forschungs-Neutronenquelle Heinz Maier-Leibnitz (Berlin, Germany)

juergen.peters@frm2.tum.de

Oral Contribution - Monday, May 26 - 15:30

Cryogen-free 7.5 T Magnet – Two Years of User Operation: In 2007 the FRM II Cryogen-free 7.5 T Magnet was operated on various instruments under user operating conditions. Different experimental setup for low temperature applications were field tested.

More over the talk addresses some new projects of sample environment equipment at FRM II. The FRM II Closed Cycle Cryostat with Ø50 mm sample tube has got a big brother. The new Ø80 mm sample tube allows increased sample sizes and minimized parasitic neutron reflections in e.g. case of powder diffractometers. For use at standard cold heads, we developed a heat switch based on a shape memory alloy. To intensify the high pressure activities at FRM II, a double action press (press capacity 450kN, tensile force 220kN) is available. The press allows static and dynamic applications up to 10 Hz by remote controlled servohydraulics. First design studies for high pressure cryostats and furnaces are in progress.

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#### Sample Environment at the Swiss Spallation Source SINQ

M. Zolliker - Paul Scherrer Institut (Villigen, Switzerland)

markus.zolliker@psi.ch

Oral Contribution - Monday, May 26 - 16:30

The swiss spallation source SINQ is a continuous neutron source. As it has only a medium flux, its strengths lie both in sophisticated neutron instruments, and in the wide range of available sample environment equipment.

In addition to the standard equipment available at neutron scattering facilities, different combinations are offered to the experimentalists, some of them are unique. Dilution inserts allowing access to temperatures well below 100 mK may be used in the two high field magnets: the 15 Tesla vertical field cryomagnet for diffraction and spectroscopy and the 11 Tesla horizontal field cryomagnet for small angle scattering. In the latter case, the field may be perpendicular or parallel to the beam. MilliKelvin temperatures may also be combined with MuPAD, a device for neutron polarization measurements in 3 dimensions. High pressures up to 100 kbar may be performed down to 4 K, or 12 kbar down to 1.5 K. High temperature up to 1400 K may be performed in controlled atmosphere.

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## **Experience with Cryofurnaces at Oak Ridge National Laboratory**

L. Santodonato, J. Wenzel, and A. Church - Oak Ridge National Lab. (Oak Ridge, USA) santodonatol@ornl.gov

Oral Contribution - Monday, May 26 - 17:00

Cryofurnace here refers to any sample environment that operates from cryogenic temperatures to above 400 K. Four different types used at Oak Ridge National Laboratory are compared (hot stage Displex, hot exchange gas Displex, hot stick liquid helium, and hot exchange gas liquid helium).

The basic design of each cryofurnace is described, and performance data are presented. It is emphasized that none of them are fully optimized, especially the in-house designed hot exchange gas Displex. A process of continual testing and upgrading is essential to realizing the full potential of any of these devices. Specific tests and upgrades are described, including design modifications to enhance thermal contact and improve "cold valve" regulation. Upgrades in operating procedures and temperature control schemes are also described, which are especially relevant for off-the-shelf systems.

This leads to the question: does it make sense for a sample environment team to design its own cryofurnace (or sub-system) when off-the-shelf options are available? In-house design carries the risk of wasting time and money. But the team is sure to learn some valuable lessons, has a chance at advancing the state-of-the-art, and could save money in the long run.

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#### **Modernising the ILL Sample Environment Suite**

E. Lelièvre-Berna, N. Belkhier, E. Bourgeat-Lami, J.-P. Gonzales, J.-L. Laborier, F. Marchal, P. Martin, Y. Memphis, P. Mendes, O. Losserand, C. Payre, X. Tonon, S. Turc - Institut Laue Langevin (Grenoble, France)

lelievre@ill.eu

Oral Contribution - Monday, May 26 - 17:30

or about three years, we upgrade or renew the sample environment suite made available to +40 instruments within the framework of the ILL Millennium Programme. We report on the progress accomplished during these years by our rejuvenating staff.

Among the many tasks presented, we will see that the thermometers of about half of our cryostats have been replaced with Cernox sensors calibrated at ILL. We have built several compact cryogen-free cryostats for Eulerian cradles and two new dilution inserts, developed the cryofree Orange cryostat and new electronics for controlling the cryogenic devices. ILL have acquired a second 100kbar Paris-Edinburg cell and a Triton dilution refrigerator. We have ordered an asymmetric zero-boil-off 10T cryomagnet for three-axis spectrometers and some diffractometers. Three other cryomagnets are also being discussed for the reflectometers and the SANS and TOF spectrometers. A safer and very accurate 2kbar gas handling system for continuously loaded gas pressure cells is being designed in collaboration with the CNRS and six copies of a fully automatised and much safer power-rack for the 1900K ILL-designed furnaces are in production.

On the instruments side, we will soon start the installation of the new electronics that will be connected to the main control workstation using CORBA. A computer installed in the cabinet will ensure this link, record the data collected by the numerous controllers and program them from a shared database.

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#### **Self-Shielded Magnets for Neutron Scattering**

A. Church - Spallation Neutron Source (Oak Ridge, USA)

churchaj@ornl.gov

Oral Contribution - Tuesday, May 27 - 08:30

n 2004, we conducted a survey of the magnetic interference issues at neutron scattering facilities worldwide. We found numerous incidents where the fringing field of a sample magnet (1) interfered with field-sensitive instruments such as Neutron Spin Echo spectrometer; (2) interfered with polarized neutron components; and (3) interfered with detectors that use photo-multipliers; and (4) magnetizing instrument components such as sample table and radiation shielding. The interference resulted in substantial loss of beam time and in some cases damages to the instruments and/or magnets. Several recommendations were made to the SNS as a result of the survey. One of the suggestions was to develop self-shielded sample magnets to reduce the fringe field at its source. While self-shielded solenoid magnets are used, e.g. in Nuclear Magnetic Imaging, self-shielded split-pair magnet with opening for neutron passage and asymmetric field profile for spin-transport needs to be developed.

SNS then decided to adopt a stray field policy soon after in late 2004, being 5 Gauss @ 0.5 meter, 0.05 Gauss 5 meter, but provision for exceptions. By developing self-shielded magnets, we will minimize the negative impact of magnetic interference in our facilities.

SNS is the first in the world to specify actively shielded neutron magnets, the 5T vertical field system SLIM SAM (Shielded Asymmetric Magnet) was delivered in March 2008 and is ready to be commissioned on a beam line during May 2008. We are also looking at a 16T vertical field system, which has had its design finalized and now has a 2009 delivery.

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## High Pressure for Neutron Scattering at Low Temperature and High Magnetic Field

R. Sadykov - Inst. Nuclear Research RAS (Moscow, Russia), Institute for High Pressure Physics RAS (Troitsk, Russia)

rsadykov@inr.ru

Oral C	ontribu	tion -	Tuesday,	, May	y 27 -	08:50
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ydrostatics: We have tested a number of Fluorinert types and found the limits of solidifications for each. The maximum is obtained for FC87 at 23kbar.

Clamp cells: High pressure clamp cells was made from TiZr zero alloy, hard Al and hard non magnetic HNU (NiCrAl) alloys for TOF-method on the pulsed and continuous neutron sources are presented in this report. These cells are designed for powders and single crystals for diffraction and inelastic neutron scattering studies. It is possible to put them in standard cryostats (even in dilution fridge inserts) and in high magnetic field cryomagnets up to 6-14T. Single crystal or powder NaCl (a pressure calibrant) and Fluorinert (a pressure medium) were used in all experiments on the neutron sources SINQ (Swiss), ISIS (UK), HMI (Germany) and ILL (France). Some of these cells were used for the investigations of the magnetic spiral in ZnCr<sub>2</sub>S<sub>4</sub>, CsCuCl<sub>3</sub> and MnSi under pressure. For investigating the form-factor dependence of magnetic localized moments in CePd(Rh)<sub>2</sub>Si<sub>2</sub> under pressure up to 40kbar and 10T with polarized neutrons on D3(ILL), we made a new nonmagnetic composite piston/cylinder type cell from TiZr+HNU.

<u>Presses</u>: Two different geometries of low temperature press for "in situ" investigations by neutron scattering are presented for discussions.

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#### 1000°C in a Superconducting 5T Magnet

C. Redmon - High Flux Isotope Reactor (Oak Ridge, USA)

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Oral Contribution - Tuesday, May 27 - 09:10

For the first time, in-situ, time-resolved measurements of the shift in the equilibrium phase transformation temperatures were achieved in the use of a superconducting magnet for neutron scattering, used in conjunction with a custom induction furnace. The materials studied required magnetic fields of 4.8T and sample temperatures >900°C. This pioneering research, using neutron diffraction methods at the WAND, will allow for future understanding and development of the next generation of structural and functional Fe-C binary alloys. Predictions had been made in the past by studying the phase equilibrium on samples no longer under applied field that are now proven to be true through this new capability.

The design and testing of the insert were initially full of catastrophic 'what ifs'. The failure of the insert isolation or protection circuits would permanently damage the 5 Tesla cryomagnet. Many design difficulties were overcome to allow a sample to be at 1000°C while inserted into a 4.2K magnet system with a dynamic flow VTI. Also, prior experience at the National High Field Magnetic Laboratory, with the same compositions and furnace, also proved that extreme RF fields generated by the induction heater reeked havoc on most every piece of electronic equipment around it. Problems during the experiment included temperature sensor offsets, 'virtual' magnet quenches, and the inability to ramp field in any configuration. The insert was successfully run in laboratory to 1000°C at 5 Tesla, and on instrument to 900°C at 4.8 Tesla.

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## Sample Cooling in the forthcoming 25T-Hybrid-Magnet - Requirements, first Sketches and Approaches

J. Heinrich - Hahn Meitner Institut (Berlin, Germany)

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Oral Contribution - Tuesday, May 27 - 09:30

The HMI is already in the process of preparing the sample environment equipment for its planned High Field Magnet at the EXED instrument. For testing purposes, a sample cooling system for the existing vertical warm bore magnet VM-5 was built. The targeted parameters were very soft: a lowest temperature of approx. 50K and a later use in combination with a high temperature top part for a temparature range of some hundred Kelvin were considered.

The major difficulty for sample cooling in this magnet is the same as expected for the High Field Magnet: the distance of about 1.5 m between sample position and possible positions of the cryo cooler. Additionally we have a nearly horizontal arrangement in the High Field Magnet. To see what can be achieved at least in the High Field Magnet, a very basic, transferable design was chosen and common workshop materials instead of special cryogenic materials were used.

Moreover the design was modelled in a computer simulation to investigate the used software's possibilities. The cool down behaviour will be shown and some comparisons between experimental and simulated data will be discussed.

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#### Sample Environment in Soft Condensed Matter at the ILL

G. Fragneto - Institut Laue Langevin (Grenoble, France) fragneto@ill.fr

Oral Contribution - Tuesday, May 27 - 10:30

Current activities in soft matter and plans for the future regarding sample environment for soft matter experiments at the ILL will be presented. Because of their fragile nature, soft-matter samples are often temperature sensitive or evolve (age) on long time-scales. Many samples are thus ideally prepared and pre-characterized immediately prior to neutron scattering experiments for an efficient use of neutron beam-time. For this reason the creation of a Partnership for Soft Condensed Matter (PSCM) on the ILL-ESRF site has been considered recently and strongly supported by the ILL management, following the successful example of the Partnership for Structural Biology and also given the strong support of the Scientific Council. Optimization of sample environment for soft matter experiments is to be dealt within the Partnership as well as facilities for sample preparation and characterization. Development of in-situ complementary techniques will be also pursued.

Existing specialised ILL sample environment for soft matter experiments include: Stopped Flow (D22), Humidity Chambers (D16, D17, IN11), Rheometer (D11), Pressure cell (D11), Solid/Liquid Cells (D17), Syringe pumps (D17), Langmuir trough (FIGARO)

For in-situ complementary techniques, a Brewster Angle Microscope is available (FIGARO). There is also a project for complementary Light Scattering and SANS on D11.

The requirements for more specialised sample environment items as well as equipment for the Soft Matter Laboratory will be presented and the need of a strong interaction with the ILL Sample Environment Group will be discussed.

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## Aerodynamic Levitation and Laser Heating — Current and Future Developments at the ILL

L. Hennet - CNRS-CEMHTI (Orléans, France)

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Oral Contribution - Tuesday, May 27 - 10:50

Studies of the liquid state present an obvious fundamental interest and are also important for technological applications since the molten state is an essential stage in various industrial processes.

At very high temperature, it is difficult to use conventional furnaces, which present major problems. In particular, the sample can be polluted by the container and the structural properties of the materials can be affected by the crucible. This has led to the development of containerless techniques and their use at synchrotron and neutron sources to study the structure and the dynamics of high temperature liquids.

Different levitation techniques have been developed by various groups around the world and at the CEMHTI, we have chosen to work with the aerodynamic levitation which is a simple way to suspend samples by using a gas flow. The advantages of this technique are the simplicity and compactness of the device, making it possible to integrate it easily in different kinds of experiments

Over the past 10 years the CEMHTI has developed various devices for neutron investigations at ISIS and ILL. In this presentation, I will give an overview of the aerodynamic levitation setups currently available at the ILL.

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#### Superconducting Vortices in CeCoIn<sub>5</sub>: Toward the Pauli-Limiting Field

M. Kenzelmann - Paul Scherrer Institute (Villigen, Switzerland)

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Oral Contribution - Tuesday, May 27 - 11:10

Many superconducting materials allow the penetration of magnetic fields in a mixed state in which the superfluid is threaded by a regular lattice of Abrikosov vortices, each carrying one quantum of magnetic flux. The phenomenological Ginzburg-Landau theory, based on the concept of characteristic length scales, has generally provided a good description of the Abrikosov vortex lattice state.

We conducted neutron-scattering measurements of the vortex lattice form factor in the heavy-fermion superconductor CeColn<sub>5</sub> and found that this form factor increases with increasing field - opposite to the expectations within the Abrikosov-Ginzburg-Landau paradigm. We propose that the anomalous field dependence of the form factor arises from Pauli paramagnetic effects around the vortex cores and from the proximity of the superconducting state to a quantum critical point. Our experiment can be seen as the first evidence of a novel superconducting vortex structure in solid matter. This study is a good example of the importance of specialized low-temperature and high-field sample environment in the study of condensed matter using neutron scattering.

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### Neutron Scattering Investigations in Extreme Sample Environments

T. Chatterji - Institut Laue Langevin (Grenoble, France)

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Oral Contribution - Tuesday, May 27 - 11:30

shall review the results of neutron scattering investigations on several magnetic materials at millikelvin temperatures, under high magnetic field up to 14 T and also under hydrostatic pressure up to about 30 kbar carried out at ILL and HMI. I shall describe and discuss the experiments where some of these extreme sample environments were combined.

The results of our neutron scattering investigations of hyperfine-induced nuclear spin ordering and nuclear spin waves in Nd<sub>2</sub>CuO<sub>4</sub> and related compounds at millikelvin temperatures will be discussed in some details. I shall also describe the results of our recent neutron scattering investigations on the magnetic structures, magnetic (H,T) phase diagram and spin waves in newly discovered multiferroic meterials TbMn<sub>2</sub>O<sub>5</sub>, YMn<sub>2</sub>O<sub>5</sub>, DyMn<sub>2</sub>O<sub>5</sub> and CuO. The pressure-temperature phase diagram of the Kondo lattice compound CeSb will be discussed. Finally I wish to discuss the new physics that can be investigated if the present sample environment limits could be extended to a reasonably moderate amounts.

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# **Cryocooler and Cryostat Options for Neutron Scattering**

R. Bains - Advanced Research Systems, Inc. (Macungie, USA)

rbains@arscryo.com

Oral Contribution - Tuesday, May 27 - 14:00

Over the past 10 years ARS has worked closely with the NS user groups to develop and offer CCR's for neutron scattering experiments.

We have the unique distinction of manufacturing the cryocooler as well as the customized cryostat to user specifications. Cryocoolers are available from the specialized 1.7K cooler developed at ILL and licensed to ARS, to the standard 4K, 30K and 80K systems which have been available for almost 10 years now.

Cryostat design is based on the experiment and can be offered as sample in vacuum and sample in vapour configurations, each design has its advantages in sample exchange/throughput and temperature range.

The presentation will cover the advantages and disadvantages of each type of cryostats as well the proper selection of cryocoolers.

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# World's Highest Field Actively Shielded Split Coil Magnet for Neutron Scattering

R. Gilardi, J. Hinderer - Bruker BioSpin AG (Fällanden, Swtizerland) raffaele.gilardi@bruker.ch

Oral Contribution - Tuesday, May 27 - 14:30

A new actively-shielded 16T split-coil magnet system is being developed and constructed by Bruker BioSpin. This will represent the first high-field actively shielded split-coil magnet for neutron scattering experiments. The magnet is a geometrically symmetric split coil, which can be operated in both symmetric and asymmetric field modes (for experiments with polarized neutrons).

In order to minimize the stray fields, a superconducting shield coil is integrated in the magnet assembly. The design of an actively shielded split coil magnet for neutron scattering is different from previously manufactured actively shielded NMR and laboratory magnets, which are standard products of Bruker, and poses some new specific questions, such as the design of the shield coils and of a suitable supporting structure. Moreover, the materials used in the split region have been evaluated with respect to mechanical stability, neutron transparency and activation in close collaboration with the Paul Scherrer Institute. The first magnet system is in the construction phase and will be installed at the Spallation Neutron Source (SNS) in USA in 2009.

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### **Oxford Instruments NanoScience**

P. Pickering, A. Catalfamo - Oxford Instruments NanoScience (Oxford, United Kingdom) phil.pickering@oxinst.co.uk

Oral Contribution - Tuesday, May 27 - 15:00

Oxford Instruments NanoScience supports the neutron scattering community by providing low temperature (as low as 10 mK) and high magnetic field (up to 15T) sample environments, tailored to neutron scattering applications.

Oxford Instruments has a unique mix of expertise gained over the years by working in partnership with every major neutron scattering facility to provide state of the art magnets and cryogenic systems. We are also proud to have pioneered the design of magnet systems for neutron scattering. We can combine field orientation, field strength, neutron access and low temperature environment to provide you with a uniquely powerful measurement platform. More recently, to overcome the growing problem of helium shortage, Oxford Instruments has been developing Cryogen-free solutions. As a result came the Triton<sup>TM</sup>DR, pump free Cryofree® dilution refrigerator providing ease of operation and flexible configuration. Magnet and low temperature insert systems with integrated helium recondensing options are also now available. Come and see us at our booth and we will be delighted to discuss your specific requirements.

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# Pulse Tube Cryocoolers: an Option for Cooling without Cryogenic Liquids

G. Thummes - TransMIT GmbH / Centre for Adaptive Cryotechnology and Sensors (Giessen, Germany)

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Oral Contribution - Tuesday, May 27 - 15:30

Among all types of regenerative cryocoolers, only the pulse tube cooler (PTC) operates without moving cold parts. This unique feature of the PTC results in high reliability, reduced vibrations, and reduced magnetic interference, which makes it very attractive for cooling without the need for liquid helium or nitrogen. For more than one decade, the TransMIT-Centre in Giessen has been developing and manufacturing various types of PTCs for operating temperatures between 2.2 K and about 150 K. Two-stage PTCs are available that supply cooling powers at 4.2 K ranging from 0.15 to 1.1 W with electrical input powers from 2 to 10 kW. Demonstrated applications of these PTCs are, for example, "dry" cooling of Josephson-voltage standards, superconducting magnets, laboratory cryostats, and pre-cooling of sub-Kelvin coolers. Other types of TransMIT-coolers are lightweight, single-stage PTCs that operate on linear compressors with only 100 W power consumption, and which deliver about 2 W of cooling power at 80 K. All of the above coolers can be adapted to the particular requirements of the user.

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# **Electronic Developments in ISIS Sample Environment in 2007**

D. Cowdery - ISIS Rutherford Appleton Labs (Didcot, United Kingdom) d.cowdery@rl.ac.uk

#### Oral Contribution - Wednesday, May 28 - 08:30

This talk describes progress in four areas of electronics made during 2007. The areas covered are motion control, thermometry, experiment control and instrumentation.

Motion control covers the use of the ISIS Standard Motion Controller in many varied locations, its degree of adoption and an analysis of failures since installation began. An example of a 5 axis goniometer is shown.

In thermometry a description is given of the next generation triple Eurotherm 3504 controller. Full electrical isolation is maintained between the three channels, each of which has a 2  $\mu$ V resolution and a 5 digit display to the microvolt range. We use a novel system to control power at low levels but retaining high power capability.

The need for more versatile experiment control has forced us to develop a Universal Interface Box. Its function is to connect various interface types between pieces of experimental equipment. It has the capability to cross connect interfaces of the following types:- NIM, serial string generation & parsing, TTL, fibreoptic, analog and relays. Advanced control will enable it to be configured by remote script file. The prototype has already given good service using NIM to ASCII serial strings with timed gaps and analog output. Production of these units us now underway.

Instrumentation issues have centred around low frequency vibration measurements, particularly in Pulse Tubes. A laser based method of analysis is described with a frequency response down to mHz.

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# Portable Automated Pressure and Temperature Control System

D. Maierhafer - High Flux Isotope Reactor (Oak Ridge, USA)

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Oral Contribution - Wednesday, May 28 - 08:50

A portable automated pressure and temperature control system has been developed to control the environment of fluid confinement cells used for neutron scattering research. This system was built from commercially available parts to minimize cost and increase reliability. It is rated to operate at pressures from 0 bar (0 PSIA) to 87.2 bar (1264 PSIA), and measures pressure with 0.0031% full scale accuracy, which corresponds to  $\pm 0.003$  bar ( $\pm 0.039$ PSI) at maximum pressure.

Pressure control is accomplished by dithering fast response (5 ms) solenoid valves to move packets of gas. Valve control can be switched between auto and manual mode through the use of a keyswitch. Temperature control is accomplished using a closed loop PID controller. Heating power is 1200W maximum with temperature measurement accuracy at the sensor of  $\pm 51$  mK at 300K. The software algorithm is implemented using LabVIEW and steps through a predefined sequence of temperature and pressure setpoints to automate fluid dosage. Temperature and pressure are continuously logged and the SANS measurement can be started once the predefined stability criteria for temperature and pressure are met (i.e., the thermal and sorption equilibria are reached). This mechanism allows measurement with high accuracy and can make efficient use of an instrument 24/7. The software runs as stand-alone, and is controlled through an Ethernet connection. The system contains a reference volume for simultaneous volumetric sorption measurements.

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## **Reconsidering ILL Cryostats Electronics**

N. Belkhier - Institut Laue Langevin (Grenoble, France)

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Oral Contribution - Wednesday, May 28 - 9:10

For more than 20 years, ILL use the combination of a Carbon probe and a platinum probe for measuring the temperature in Orange cryostats (1.5 - 320K). It was assumed that all Carbon sensors are almost identical (one common calibration curve) and it necessitated the use of a controller able to regulate the temperature from a weighted sum in the range 20 - 50K where both sensors are taken into account. For this purpose, the ILL had developed and used its own temperature controller featuring sensors auto-identification, 110W heating power and enough memory for storing 140 calibration curves and PID parameters. ILL had also developed its own cryogen level monitors at a time where there was no available commercial units.

Today, things have changed and ILL have therefore started the ambitious project to replace the Carbon probes mounted on  $\approx$ 70 cryostats and  $\approx$ 150 sample sticks with Cernox sensors, the ILL temperature controllers with LakeShore 340 units, and the cryogen level monitors and cold-valve controllers with programmable units made from commercial parts. We present these new products as well as a simple micro-controller based system that sets automatically the LakeShore controllers after connecting the leads to a cryostat or a stick.

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## **Window Wars: Quartz vs. Titanium**

E. Fitzgerald - NIST Center for Neutron Research (Gaithersburg, USA)

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Oral Contribution - Wednesday, May 28 - 09:30

At the NCNR we have one horizontal field superconducting magnet for use on two SANS and one USANS instruments with a maximum field of 9T. The original design for the bottom of the sample well used quartz windows epoxied into a titanium block. On the first experiment, this arrangement blew out. Although this case was definitely user error, in the following years we have had an increasing number of problems with the windows leaking unexpectedly. Recently it became such a problem that the quartz windows were removed and titanium windows were permanently welded in. Knowing that this was an irreversible change we also began work on a new quartz "cup" design that would combine the low background of quartz windows with the robust nature of titanium windows. This talk will cover the development, implementation and results of the quartz "cup" design as well as draw comparisons with the previous designs.

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# Wet or Dry....That is the question

#### R. Down - ISIS Rutherford Appleton Labs (Didcot, United Kingdom)

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#### **Poster Contribution**

There has been a steady increase in the amount of dry cryogenic systems available on the market and most facilities will have responded with the odd investment here and there; suddenly at ISIS the odd investment has now grown and there are a considerable number of Neutron and Muon instruments that are using or planning to use dry systems.

We will take a look at what dry systems ISIS has and how the face of our cryogenic support and equipment base is changing; what range of temperature we can offer and what ancillary equipment we can supply to ensure that our dry systems are indeed as good as the wet systems that they replace.

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# Low Temperature Remote Pressure Measurement in Sapphire Anvil and other Cells

B. Evans, J. Dreyer, C. Bull - ISIS Rutherford Appleton Labs (Didcot, United Kingdom)
B.E.Evans@rl.ac.uk

#### **Poster Contribution**

This poster will describe the ongoing development of a system for low temperature pressure measurements on clamp cells with optical access. The system makes use of ruby fluorescence, the principle of which is based on the shift in wavelength of laser light as it is absorbed and re-emitted by a fragment of ruby under pressure.

At ISIS we have had the capacity for bench-top pressure measurements in sapphire anvil and Paris-Edinburgh cells for some time. We have now made successful remote measurements on a sapphire anvil cell at low temperatures using an adapted EasyLab Optiprexx ruby fluorescence system and a fibre optic assembly. This system has also been used to make remote pressure measurements on a Paris-Edinburgh cell with diamond anvils.

A new development to provide hydraulically driven high-pressure cells with optical access for pressures ≥10 GPa with in-situ ruby fluorescence measurements at low temperatures will also be described.

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# A Compact System for the Production and Quality Measurement of Para-Hydrogen

B. Evans, C. Goodway, J. Bones, A. Ramirez-Cuesta - ISIS Rutherford Appleton Labs (Didcot, United Kingdom)

B.E.Evans@rl.ac.uk

#### **Poster Contribution**

Normal hydrogen gas contains 25% para-hydrogen and 75% ortho-hydrogen, but may be converted to a maximum possible 99.8% para-hydrogen at low temperatures. The percentage of para-hydrogen in a gas sample can be verified by neutron transmission methods during experiments, but it is usually preferable to know the para-hydrogen percentage of the gas before adding it to a sample on the beam line.

The ISIS para-hydrogen production rig contains a 10 K cold head assembly, a pumping set, a gas handling system, a para-hydrogen generating cell and a temperature control system. A gauge for measuring the percentage of para-hydrogen is incorporated into the assembly with supporting electronics. The principle of operation of the gauge is based on conductivity measurements.

This poster will describe the design and testing of the para-hydrogen rig and gauge.

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# Variable Temperature Insert for Paris Edinburgh Cell

D. Francis<sup>1</sup>, W. G.Marshall<sup>1</sup>, M. G.Tucker<sup>1</sup>, J. W Dreyer<sup>1</sup> and G. Kouzmenko<sup>2</sup> - ISIS Rutherford Appleton Labs<sup>1</sup> (Didcot, UK), Oxford Instruments NanoScience<sup>2</sup> (Abingdon, UK) d.j.francis@rl.ac.uk

#### **Poster Contribution**

A prototype variable temperature (v-T) insert for the Paris-Edinburgh (P-E) press has been designed and developed at ISIS which is capable of varying the sample temperature over the  $110 - 500 \, \text{K}$  interval with excellent control characteristics. The insert utilises a liquid nitrogen cooling circuit to cool just the sample and the WC anvils, with the latter incorporating the resistive heaters used for temperature control and heating.

The insert assembly is thermally insulated from the P-E cell body by zirconia-cored seats and backing disks and PTFE insulation. The temperature of the P-E cell cylinder housing is maintained close to ambient temperature by means of a separate constant-temperature circuit. The whole installation in mounted on a standard Tomkinson flange and runs in a standard ISIS instrument vacuum tank. The sample temperature and pressure can be monitored and controlled remotely from the ISIS instrument computer used for neutron data acquisition.

Cool-down times of the previous low-temperature setup – which required cooling the entire P-E press using liquid nitrogen inside a large vacuum-walled tank – were typically 4-5 hours from ambient to 120 K. Recent commissioning tests of the v-T insert demonstrated the ability to cool the sample to 100 K and warm to 473 K from ambient temperature within ~45 minutes. In addition to a more efficient and productive use of neutron beamtime, this insert now makes possible high-pressure neutron diffraction experiments over the 100-500 K and 0-10 GPa intervals with a single P-E cell sample loading, an entirely new capability.

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## **New Developments on Orange Cryostats**

S. Gerischer - Hahn Meitner Institut (Berlin, Germany)

gerischer@hmi.de

#### **Poster Contribution**

In the last years two major improvements have been made on our standard Orange cryostats. The first concerns the sample space and the cool down time. We replaced the 50mm diameter lower part of the sample space by a bottle shape 68mm can. This will reduce the aluminium background scattering especially for the neutron instruments with radial collimators. In addition we removed some useless masses which interrupt the cool down at 20K for half an hour.

The second is a cheaper and better needle valve controller. The new controller contains a small LCD display and an Atmel micro-controller. Compared to the current design the electromagnetic noise caused by the driving of the stepper motor is strongly reduced. It uses a simple two-level controller and will shut down the driving current when the motor is not moving. The open design allows us to connect an absolute position encoder if it is needed to monitor the absolute movement. It will even be possible to use this controller as an independently running Lambda controller und needle valve controller on the Oxford Instruments Magnets.

A new pressure gauge even allows us to regulate the needle valve below 1 mbar. Thus the base temperature of the Orange cryostat can be lowered to 1.3K with the help of a roots pump.

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# **Micro-Furnace for Transmission X-Ray Diffraction Imaging**

B. Gorges - European Synchrotron Radiation Facility (Grenoble, France) gorges@esrf.fr

#### **Poster Contribution**

A new transmission furnace has been developed to work under gas flow up to 800°C. Most X-ray furnaces offering wide angle access work in reflection mode; X-ray topography measurements have to be done in transmission mode but still necessitate a wide angle. The reflection setup also gives a larger distance between the sample and the detector. This transmission furnace had to comply with a number of additional constraints: thermalisation of a low thermal conducting crystal sample, stressless sample mounting and small temperature gradients over a 7x7mm² vertical sample surface. The construction also had to take the possibility of chemical reactions at high temperature between the sample, the construction materials and the gas into account.

The validation of the new setup has been done on ESRF beamline BM05 through the characterization of the ferro-electric phase transition of  $BaTiO_3$ .

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## A.S. Scientific Products Limited

C. Hiller, P. Wiggins - (Abingdon, United Kingdom)

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#### **Poster Contribution**

A S Scientific Products design and build sample environment apparatus. Under licence from the ILL, we build the 'famous' Orange Cryostat and the High Temperature Furnace (up to 1800°C).

A large part of our sample environment business now involves the use of mechanical cold heads. We have OEM relationships with all the major manufacturers (Sumitomo (SHI) Cryogenics, Leybold, Cryomech, Edwards). This allows us to make a number of different Top Loading Cryostats with either Pulse Tube or GM machines. We also manufacture a 1.8K cryogen free unit utilising a standard 10K GM machine.

A S Scientific Products can supply all the usual accessories needed to build a complete system including:

- Sensors
- Temperature controllers
- LHe and LN<sub>2</sub> transfer lines
- Bespoke tail sets with vanadium, sapphire windows.
- Vacuum pumps.

We can also design and build Bath Cryostats, Flow Cryostats, and 1 off style cryogenic apparatus.

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# Progress and Future Developments involving High Pressure Hydrogen at ISIS

M. Kibble - ISIS Rutherford Appleton Labs (Didcot, United Kingdom)

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#### **Poster Contribution**

Within the Experimental Operations Division of ISIS, the Furnace, Pressure & Special Systems Section are developing their range of Sample Environment Equipment to meet the ever advancing demands of the visiting scientists and scientific community.

Scientific interest is progressively demanding further advancements of our equipment to enable the development of scientific information and knowledge and this includes the requirement of ever increasing pressures. With interest growing in areas such as fuel replacement, Hydrogen storage has become the hot topic. At present ISIS can support experiments involving Hydrogen of pressure ranges of up to 3kbar within a temperature range of 10 to 773 Kelvin.

The Furnace, Pressure & Special Systems Section are currently progressing and developing equipment with the aim to provide an experimental environment achievable of providing pressures of up to 8kBar with a Hydrogen pressure medium within neutron and Hydrogen compatible materials. To enable us to meet these parameters the section needs to source companies who will develop equipment in collaboration with ISIS. This has been our initial aim and at present we have been able to compile a portfolio of companies willing to develop and modify existing inert gas systems with the aim to produce a system that is manufactured from Hydrogen safe materials.

Our resent purchases include a 7kbar Hand Intensifier, 10kbar Transducers and 10kbar rupture disc assembly, all of which are Hydrogen friendly.

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# The Laboratory for Magnetic Measurements at BENSC

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#### **Poster Contribution**

Since recent years, the Hahn-Meitner-Institute operates a variety of research laboratories for sample characterisation. In order to install a service for BENSC users providing access to complementary measurements at extreme conditions, these laboratories have been combined to form the new Laboratory for Magnetic Measurements at BENSC (LaMMB). At present, the possibilities of LaMMB offer four different measurement systems with magnetic fields up to 14.5 T and temperatures down to 260 mK. The services of LaMMB are strongly requested by internal as well as by external research groups.

Starting in 2008, a new cryogenic system with magnetic fields up to 17 T and temperatures down to below 10 mK will greatly extend the temperature and magnetic field range of the measurements at LaMMB.

In order to give an example for a successful measurement program the organic S=1/2 spin ladder material  $(C_5H_{12}N)_2CuBr_4$  will be presented. The phase diagram of  $(C_5H_{12}N)_2CuBr_4$  was determined by measurements of the specific heat, the magnetocaloric effect and the magnetization in magnetic fields up to 14.5 T and down to 300 mK.

Notes:	

# **Polarimetric Neutron Spin-Echo demonstrated**

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#### **Poster Contribution**

The multifaceted dynamics of antiferromagnets and helimagnets require more than the conventional neutron spin echo set-up (NSE). Indeed, the neutron beam polarisation is not necessarily flipped upon scattering. When the magnetic interaction vector is complex. In the presence of nuclear-magnetic interferences the rotation of the incident polarisation can be of any angle around or toward a specific direction; it is impossible to distinguish between a simple depolarisation due to e.g. magnetic domains and a rotation of the polarisation vector.

One way of overcoming these difficulties is to implement a zero-field polarimeter [FT] on an NSE spectrometer [FM]. This has now been achieved using the  $3^{rd}$  generation Cryopad [ELB] and a modified version of the Intensity Modulated variant of NSE [BF]. To do so, two additional  $\pi/2$  flippers were installed near the sample space. These flippers, combined with the other two  $\pi/2$  flippers, define the Larmor precession regions before and after the sample position. A solid-state polariser was added after the first Larmor precession region to repolarise the beam. This is necessary for handling the polarisation vector in Cryopad. The last step involves the installation of the Cryopad cryostat with its two rotating nutators and the Orange cryostat used to cool the sample.

We present the pioneering Polarimetric Spin-Echo experiment performed at HMI with the ILL Cryopad. This technique is now available on IN15 at ILL.

[FT]: F. Tasset, Physica B 157 (1989) 627

[FM]: F.Mezei, Z. Phys. 255 (1972) 146; F. Mezei (ed.), Neutron Spin Echo, Lecture Notes in Physics Series, Vol. 128 (Springer, Heidelberg, 1980)

[ELB]: E. Lelièvre-Bernaa, P.J. Brown, F. Tasset, K. Kakurai, M. Takeda, L.-P. Regnault, Physica B 397 (2007) 120–124

[BF]: B. Farago, F. Mezei, Physica B 136 (1986) 627

Notes:	

# SDMS/SNLS: a Valuable Long-Term Industrial Partner for Neutron Scientists in Nuclear, Vacuum and Cryogenics Technologies

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#### **Poster Contribution**

Over the last 40 years, SDMS and its subsidiary SNLS have grown to become one of the most highly regarded names in the fabrication business (manufacturing and often contribution to designing) in Nuclear, Vacuum and Cryogenics technologies worldwide. The main areas of work are Energy, Research, Defence & Space.

Located in St-Romans, halfway between Grenoble and Valence in South East of France, SDMS/SNLS employs a total staff of 120 people, of which 25 engineers. SDMS/SNLS profession is the well-documented production, in clean workshops, of specific noble metal sets and assemblies for customers with a high technology profile.

Through its involvement in all the major scientific projects and installations, the company has honed its skills and won widespread renown amongst highly demanding customers. With its well adapted workforce and equipment, SDMS/SNLS offers the required quality on time and to budget. Particularly within the frame of Neutrons Sources and their Experimental Environment (Research Reactors and Spallation Accelerators as well), the company SDMS/SNLS provides specific UHV Chambers, customized LN<sub>2</sub>, LHe or LH<sub>2</sub> Cryostats, Lines or Loops, reactors Incore Equipments, even reactors Core Vessels, experimental fully-equipped Gloves Boxes, special nuclear wastes or samples Tanks, Casks and Containers, particles Accelerating Cavities, particles Beams Collimators, instrumented Irradiation Devices or Targets, structures or parts of Particles Detectors, etc.

Notes:	\

# **Compact 1100K Furnaces for Cradles & Automatic Power Racks for 1900K ILL-Designed Furnaces**

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#### **Poster Contribution**

Single crystal diffractometers do need compact devices covering large temperature ranges and which can be mounted on a cradle. In order to satisfy our users, we have built a 1.8K cryogen-free cryostat and more recently a 1100K furnace. This furnace is very compact and compatible with the cold-head fixation. Its geometry is also compatible with the large solid-angle required on four-circle diffractometers. We present the performance of the furnace and show how easy it is to replace the heater.

We also present the new power racks that will be used to control our 1900K furnaces. They are fully automatised, much safer than the preceding ones, and as fast as possible without destroying the equipment.

Notes:

# Management of Liquid Helium, Nitrogen and Argon at ILL

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#### **Poster Contribution**

or many years, ILL sign contracts with CEA and CNRS for the delivery of liquid helium, liquid nitrogen and argon. Since the 80's, a lot of efforts have been made to recover the helium gas with the aim to decrease the recurrent costs. Indeed, scientists and technicians use in average 500 liters of liquid helium per day while a kilogramme of gas, i.e. about 8 liters of liquid, cost more than 30€.

We present the organisation of the delivery, usage and recovery phases and explain how helium losses are minimised down to less than 20%. We also present the evolution of the usage of the cryogens and try to evaluate the future needs considering the arrival of cryogen-free and zero-boil-off systems.

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	Notes:	

# **How to Keep a Sample Stick out of Your Face**

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#### **Poster Contribution**

We have designed and tested a mechanism to improve the safety of top-loading closed-cycle refrigerators and liquid helium cryostats. Accidental introduction of air into the sample space can create a situation in which excessive pressure may build inside the sample space of the cryostat. Ice that forms near the top of the cryostat creates a blockage that prevents the relief valves typically located at the top of the cryostat from being able to function.

If the pressure rises high enough, the sample stick can be launched. In extreme cases, the body of the cryostat can be damaged or even explode. This presents a dangerous situation to the unsuspecting researcher, can result in the expensive loss of equipment, loss of beam time, and can damage the sample. We have devised a means of venting the bottom of the sample well through the sample stick itself. We present this mechanism and report on its tested performance.

Notes:	\

### **CCR Helium Hose Swivels**

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#### **Poster Contribution**

The SNS beamline 4a Magnetism Reflectometer has space constraints regarding a 4 Kelvin CCR and a concrete wall. The requirement is that the CCR rotates through 360 degrees horizontally about the cold finger axis as well as rotate through 90 degrees about a vertical axis to bring it out of the magnet coil for sample changes.

In order to maintain this movement, which sweeps the helium supply hoses past the wall, swivels were designed in house. This design allows the hoses freedom of motion without concern of kinking the hoses. Further development has yielded back-to-back swivel pairs which allow full freedom of movement for the CCR with no concern for flexure of the high-pressure helium hoses. The original pair of fittings has been in operation for 6 months now with no noticeable drop in system pressure. ORNL is currently investigating the option of patenting these fittings as no off-the shelf solutions to this need have been found.

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	Notes:	

# **High Pressure High Temperature (HPHT) Sapphire Cell**

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#### **Poster Contribution**

Lord temperature high pressure sample environment schemes are commonly used in twenty-first century neutron scattering experiments. The road less traveled, however, is high temperature high pressure sample environment schemes, especially on inelastic neutron scattering instruments. The challenge to address issues of material selection, cell and insert design, testing, and user approval coupled with the process of approving SNS' first high pressure cell, bunker, and testing procedure were nothing short of ground breaking.

Notes:	1

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This Workshop is the fifth of a series of biannual events launched in 1999 at the Hahn Meitner Institut (Berlin, Germany) by M. Meißner. Following events took place at the ISIS / Rutherford Appleton Laboratory (Didcot, UK), the Paul Scherrer Institut (Switzerland) and the Argonne National Laboratory (Chicago, USA). Participants are mostly staff members in charge of the sample environment equipment at neutron scattering facilities and scientists using neutron beams worldwide.

A wide range of topics related to the establishment and control of sample environments is discussed. The fifth edition starts with an overview of the projects running at different facilities and continues with sessions on specific technical issues and scientific needs. A special session gives the companies participating in neutron scattering technical projects the opportunity to present their latest products and projects that might be of interests to neutron scatterers.

Eddy Lelièvre-Berna SE@NSF 2008 Organiser

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