

ISIS Sample Environment and Support Laboratories Newsletter

Greetings and welcome to a new update from the Support Labs and the Sample Environment teams! Everyone has been busy over the long shutdown; some of that work has recently been highlighted at the 'Shutdown Showcase' event. The Neutron and Muon Source User Meeting (NMSUM) (25/04/22 – 27/04/22) is another opportunity to get new insights and highlights from our various groups!

I'd like to thank all the contributors for taking the time to inform everyone of the good work your teams have been engaged with behind the scenes during shutdown and cycle! – *Editor, Daniel Nye*

ERAS AND SAMPLE SAFETY

The ISIS Sample Safety Team has been busy rolling out a new Chemistry Lab Test. Completion of this test will be a requirement for any user to gain access to the Chemistry and Pre. Labs. The system is now live, and it is advised to complete the test before arriving on site for future experiments. [The test can be found on the RAL facility page.](#)

The ISIS Sample Safety Team has liaised with IT to update many of the back-end elements of the Experimental Risk Assessment (ERA) system. Most of these changes will only result in minor changes to the way in which users use the system, but the overhaul of the system will now result in more accurate risk assessments, improving the overall safety of these experiments.

The ISIS Sample Safety Team would like to take this opportunity to remind users and staff that while high hazard experiments can be supported, the team will need to be consulted beforehand. It is strongly advised that you get in contact with the Sample Safety Team as early in the process as possible to ensure the ISIS Sample Safety Team have sufficient time to make all the necessary arrangements.

Should you have any questions about online or offline experiments at ISIS then please discuss these with the ISIS Sample Safety Team who can be contacted at ISISSampleSafety@stfc.ac.uk.

BIOLOGY LABS



We are delighted to welcome the new BioLab manager, Ludmila Mee, to the Support Labs group! Ludmila started on 14/03/22. Immediately prior to her position here, she ran the life sciences labs at Oxford Brooks University, for almost a decade. These included the Biochemistry Lab and the Cat-2 Microbiology Lab there.

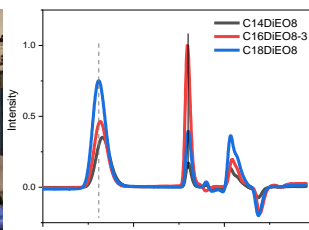
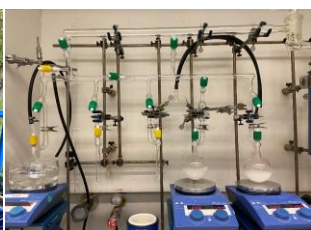
Ludmila has a PhD in Biochemistry from the Novosibirsk Institute of Bioorganic Chemistry and an MSc in Molecular Biology from Novosibirsk State University. She has multiple papers published under her maiden name of Bozhenok, including one in Nature Cell Biology: DOI: [10.1038/ncb1196](https://doi.org/10.1038/ncb1196)

Ludmila has experience with many techniques, including gene cloning, gene-specific PCR, sub-cellular fractionation, protein purification and protein expression in bacteria and insect cells. She is experienced with primary cells and in-vitro cell-based assays. Ludmila has vast experience in tissue culture: approximately 60 different cell lines. These cell lines were mostly related to a project looking at the development of antibody-based cancer therapeutics for solid tumors. Ludmila worked on the biochemical purification of plasma membranes from cancer tissues for subsequent analysis of proteins by Mass Spec.

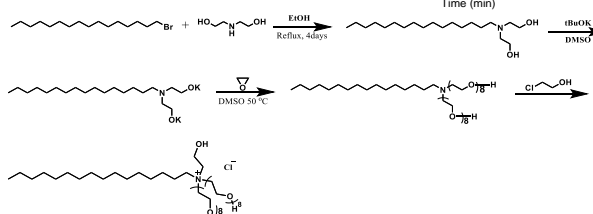


Please contact the lab manager, Ludmila Mee (ludmila.mee@stfc.ac.uk) for instrument bookings and access

DEUTERATION FACILITY



The Deuteration Facility is continually looking to expand on the profile of materials that we offer. Deuterium labelling has enabled polymer scientists to address many questions such as polymer structures and dynamics through neutron techniques that are unachievable by other means. Development of deuterated monomers, oligomers and polymers at ISIS is inspired by the increasing request from user community in recent years.



To build up the polymer deuteration skills we have collaborated with the polymer synthesis lab in the Jülich Centre for Neutron Science (JCNS), Germany. The collaborative project includes deuterated Polydimethylsiloxane PDMS, Polyethylene glycol PEG, and PEG derivatives. These deuterated polymers have been applied in the neutron experiments of ISIS users, which significantly advanced the experimental design and characterisation.

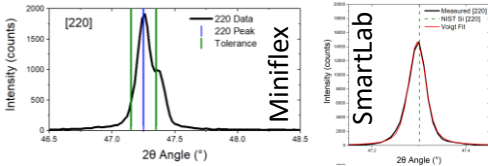


For D-Lab publications, please visit <https://www.isis.stfc.ac.uk/Pages/Deuteration-publications.aspx>

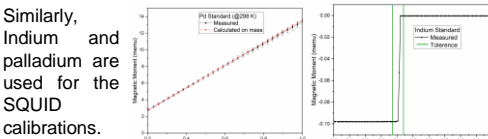
Please contact the D-Lab team to discuss requirements (ISISDeuterationFacility@stfc.ac.uk).

MATERIALS CHARACTERISATION LAB

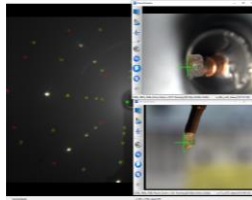
The MCL team are continuously improving the reliability of measurements through thorough calibrations and measuring documented standards.



For the powder diffraction instruments, this involves measuring against a NIST standard and then comparing multiple measured peak positions with the theoretical peak positions. The reports are available on request.

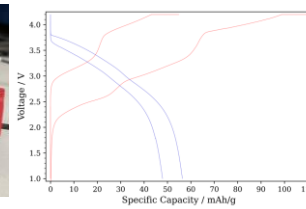


Similarly, Indium and palladium are used for the SQUID calibrations.

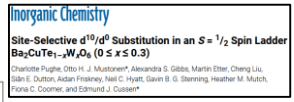
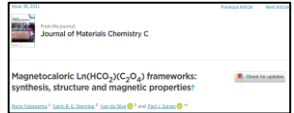


The Laue camera is regularly calibrated internally to ensure the crosshairs are correctly positioned, and with an external crystal to check the camera-to-sample distance, ensuring a good staging point for measurements.

A new Battery testing cell is being commissioned in the lab. Further work will be required to optimise the measurements, but the initial testing with the cell looks promising.



There have been many new publications with the MCL referenced; a small selection is highlighted below:



Please contact the lab manager, Gavin Stenning (gavin.stenning@stfc.ac.uk) for instrument bookings, access and inductions.

CHEMISTRY AND SAMPLE PREP. LABS

Work is underway to remove the legacy chemicals from the non-active cupboards, with assistance from the relevant instrument scientists.

While waste chemicals are regularly removed from the Prep. Labs, there had been a gradual build-up of longer term samples and legacy chemicals which were not initially identified as waste. These items have been monitored on a regular basis and the long shutdown allowed for the diversion of time and resources to their removal. The owners needed to be contacted to determine if they were required to be returned or else disposed of through the proper routes.

The Covid-19 restrictions in the Prep. Labs regarding occupancy numbers have been removed and are now monitored by CO₂ level. As such, it is no longer necessary to book time in the Prep. labs, and there are no longer set 'workstations'; the markings are currently being removed.



It is still possible to have chemicals and samples delivered to Sarah Langham, and to ask Rachel and/or Sarah to ship samples; this option is being continued indefinitely.

There are a variety of chemicals available in the prep labs: [Please check here](#) before ordering chemicals to see if we already have what you need.

Please contact Sarah Langham (sarah.langham@stfc.ac.uk) for any information about the Chemistry labs.

HYDROGEN AND CATALYSIS LAB

The space previously occupied by the high-throughput catalyst-screening robot is in the process of being repurposed to accommodate the rapidly growing research area of battery materials. Focus on this area has been increasing across multiple departments on site and beyond, so providing more space for this research will keep the lab well-placed to meet user expectations and needs in the future.

Bill David's group, working with the company *Reaction Engines*, are intending to use one of the fume hoods long term for research into generating hydrogen from ammonia for energy storage.

Methanol absorption methodology has been used for the first time in the lab. Specifically a vapour methanol absorption onto zeolite samples was measured from the methanol absorption isotherms. This was achieved using the *Autosorb* equipment. The *Autosorb* is an instrument that can measure the physisorption or the chemisorption of a sample in a gaseous environment. In this instance, the gas has been replaced with a vapour.

This was the first instance of a vapour-absorption experiment in the R79 Hydrogen and Catalysis lab; The success of this experiment has significantly boosted the confidence in running similar measurements in the future, increasing the variety of experiments which can be supported.



Please contact the lab manager, James Taylor (james.taylor@stfc.ac.uk) for instrument bookings and access

PRESSURE AND FURNACE

Along with many other teams, the Pressure and Furnace Team recently had an article featured on the website, which has been summarised here. Please check out the article in full at the link below.

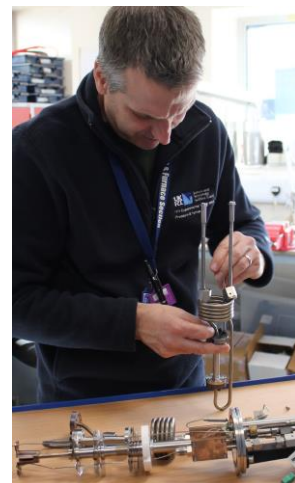
<https://ukri.sharepoint.com/sites/theforce-stfc/SitePages/Behind-the-scenes-20220302.aspx>.

The Pressure and Furnace Team provide sample environment equipment and technical support to ISIS users and instrument scientists for experiments that involve high temperatures, high pressures and gas handling. This includes the logistics of getting experiments scheduled as equipment and staff time need to be booked in advance.

This cycle is just for Target Station 2 instruments, which has provided the opportunity for instrument scientists to schedule more complicated experiments while the equipment availability is higher.

Time has also been used to run some offline measurements for users in advance of their beamtime to check the feasibility of the methodology for more complicated experiments.

The long shutdown has allowed more time to be invested in equipment maintenance. Gas systems, furnaces and pressure cells have been stripped down, rebuilt and leak tested.



Please contact Team Leader [Chris Goodway](#) for more information about the support the Pressure and Furnace group can offer you.

CRYOGENICS

The helium recovery project at ISIS is extremely successful, with over a 90% helium recovery rate. This serves to decrease costs and waste at the same time.

With this success, the cryogenics team have branched out to recover helium used at Diamond Light Source (DLS); a 600m long pipe has been laid to transport high pressure helium from DLS to the recovery building. As with ISIS, it is re-liquefied and transported to DLS in Dewars.

The Cryogenics "Great Northern Tour" took place in 2021, with the Cryogenics team visiting various institutions in the north of England with strong cryogenics departments:

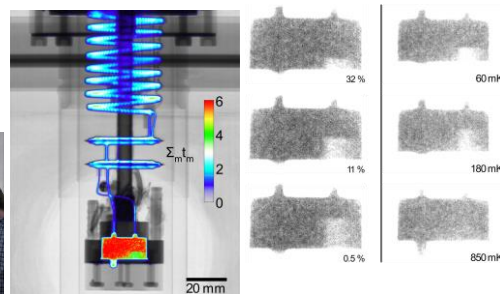
Daresbury Lab, Lancaster University and Manchester University specifically, allowing them to trade techniques and experiences, provide an opportunity for networking and gain a broader perspective on the state of cutting edge cryogenics in the UK.

The cryogenics team have published a paper: "Neutron imaging of an operational dilution refrigerator", detailing their studies of the dilution process in-operando by neutron radiography. The published paper can be accessed here:

<https://doi.org/10.1038/s41598-022-05025-0>



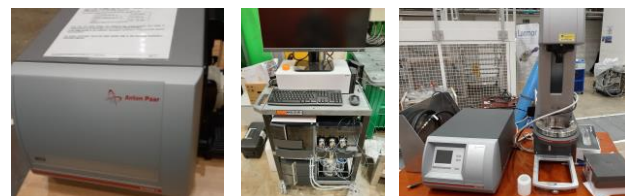
Not only does this increase sustainability on site as a whole, but this kind of collaboration improves the links between DLS and ISIS.



Please contact Richard Down (richard.down@stfc.ac.uk), Cryogenic Team Leader for information.

SOFT MATTER

The new Surpass instrument will provide a unique capability in surface analysis, which is not currently available at ISIS (& RAL). This will provide crucial information for planning and optimising expensive neutron reflectometry experiments.



In-line size exclusion chromatography (SEC) is now routinely used on small-angle x-ray scattering (SAXS) instruments, particularly those which focus on protein solution scattering. The feasibility of a SEC-SANS was recently demonstrated on the time of flight SANS2d instrument at ISIS target station 2, in collaboration with the B21 Team at Diamond Light Source, on a number of systems including proteins, liposomes and membrane proteins in copolymer nanodiscs, demonstrating the utility of SEC-SANS on a pulsed source for classical bio-SANS and more generic biophysical systems of interest.

The SANS group and Soft Matter section very recently acquired a HPLC system and will be commissioning SEC-SANS at ISIS, with the aim of making SEC the de facto sample environment for biological molecules.

The original Anton Paar rheometer has given ISIS a number of high impact publications, often in areas relevant to industry as it is possible to subject samples to shear and temperature cycles which mimic industrial or biomedical processes. A companion unit exists at Diamond for use by the SAXS instruments, and there are shared components. However, the unit has been in service for over 10 years with an increasing number of faults and in need of modifications to allow the reflectometry beamlines to make use of it, which would be problematic for the SANS program. The new rheometer, also from Anton Paar will have all the capabilities of the previous instrument, with some important enhancements and new capabilities:

- A control unit separate from the main body which will make arrangement on the beamline more straightforward.
- Improved thermal control, reducing reliance on separate water baths and liquid N2 circuits.
- For the first time at ISIS, a strain attachment has been purchased, which will allow samples to be stretched inside the neutron beam. We believe we may be the first institute to have done this.

Please contact Team Leader [Andy Church](#) for more information about the Soft Matter group.