NEUTRONS
FOR SOCIETY

Annual Report 2023

Institut Laue Langevin

The world's leading facility in neutron science and technology

Ipoustéguy's sculpture at the Institut Laue Langevin (ILL) in Grenoble is a monumental work that seamlessly integrates with its surroundings. It depicts an evocative sequence of human history and its challenges, from humorous beginnings to darker representations of the consequences of the discovery of atomic fire. The artwork also expresses hope through symbols of maternity and the continuity of life. The depiction of man, portrayed vividly, embodies the duality of his existence, illustrated by symbolic forms and a door evoking uncertainty about the future. This captivating sculpture invites deep reflection on the journey of humanity.

annual report 2023

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The Institut Laue Langevin (ILL) is an international research centre providing world-leading facilities in neutron science and technology. Neutrons are used at the ILL to probe the microscopic structure and dynamics of a broad range of materials at molecular, atomic and nuclear level.

The ILL delivers the most intense, continuous neutron beams in the world from its 58.3 MW nuclear reactor designed for high brightness. The reactor normally functions round-the-clock for three 50-60 day cycles per year. The reactor supplies neutrons to a suite of 40, continuously upgraded, state of the art instruments.

The ILL is owned by its three founding countries - France, Germany and the United Kingdom. These three Associate countries contributed some 80 M€ to the Institute in 2023, a sum complemented to the level of 20 M€ from the ILL's Scientific Member countries (collectively know as the fourth Associate!) - Austria, Belgium, the Czech Republic, Denmark, Italy, Poland, Spain, Slovakia, Slovenia, Sweden and Switzerland. The ILL's overall budget in 2023 amounted to about 120 M€.

As a service institute, the ILL makes its facilities and expertise available to visiting scientists. It has a truly global user community: over 1600 researchers from 40 different countries came to work at the ILL in 2023. The 1062 experiments they performed were pre-selected by a scientific review committee. During 2023, 461 scientific papers were published, following the treatment and interpretation of data obtained from the use of our facilities. 108 of these articles were published in high-impact journals.

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FOREWORD


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2023 is the year which marked the return of ILL to full neutron production and scientific productivity, after a long shutdown for installing the main components of the H1-H2 guide system to support the Endurance programme. We are back with a rejuvenated, renovated and world-leading suite of instruments. Nowhere else provides the breadth and quality of neutron instrumentation that we do and nowhere else delivers the same breadth and quality of science impact. The long shutdown was preceded by the COVID years, which were both disruptive and instructive in totally different ways. It feels like the return to normal operations and science was long overdue. It feels good to be back!

It feels good to be back for me personally as well. I left ILL in 2010 to join the then fledgling ESS project and many things have happened since then. Returning to ILL after a break of 13 years has been both exciting and eye-opening. Many aspects of the working culture are unchanged or at least very much recognisable. Many of my old colleagues are still here, a bit older and often wiser, but also very much recognisable, and I have really enjoyed reconnecting with my old friends and colleagues. However, the part of the Institute which has changed the most dramatically is the instrument suite. The Endurance programme has been implemented over two rounds and followed directly on from the Millennium programme. The result has been a continuous series of activities to upgrade old instruments and build new instruments for more than 20 years, delivering a suite of instruments that would make a brand-new facility proud, let alone one which recently celebrated its 50th year of operation. One of the first times I walked around the instruments after returning here in October, I ended up getting lost in the labyrinth of instruments, shielding walls and control areas at the back of ILL7. That is how completely the instrument suite has been modernised and rearranged over the last 10 years or so.

The year was also marked by changes in ILL Management (the Director position being filled by Paul Langan, then Jérôme Estrade, then myself), but with no interruption in ILL's forward momentum, or in our strategy moving forward. We need to complete Endurance and we need to focus on delivering world-leading science that addresses the most important and impactful scientific and societal challenges of today and the coming years. I would like to thank all of my predecessors and colleagues for the work they have put into placing us in the position of strength in which we find ourselves today.

It has been more than a year since we returned to on-site work post-COVID. In many ways, things have returned to how they were before, but there are some things that have changed. Some of these changes are improvements. We have developed new tools to manage remote users, working from home has become more common, many meetings take place via Zoom. These are all useful additions to our capabilities, but they have side effects which we need to watch out for. Rebuilding and maintaining the social climate, strong relationships with our colleagues, informal networks, supporting each other. These all require us to be present at our place of work in person. There is a strong case to be made for maintaining our physical presence in the office, workshop, laboratory or instrument as our main place of work. In my view, the new normal should look a lot like the old normal.

The year ahead contains many challenges, not least demonstrating the reliable reactor operation that a strong science programme needs. Another long(ish) shutdown is planned to allow essential maintenance and the safety upgrades required by our regulators. We have engaged in a process to establish a Science Strategy which will help guide future resource allocations to ensure maximum scientific and societal impact. We will continue to demonstrate the importance of our science impact through the user programme. We are all seeing the increasing effects of anthropogenic climate change. We play an essential part in meeting the global need to accelerate the development and deployment of green technologies to achieve carbon neutrality and environmental sustainability. We deliver critical new understanding in human health, engineering, quantum materials and particle physics. As you read through the scientific highlights of this report, I would like you to reflect on the breadth and quality of the science that we support, and the importance to society of us continuing to deliver that science.

Ken Andersen
ILL Director


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ABOUT NEUTRONS

WHY NEUTRON SCATTERING IS USEFUL

Neutrons can behave as particles, waves or microscopic magnetic dipoles. With their very specific properties, neutrons can provide information that is often impossible to obtain using other techniques.

WAVELENGTHS

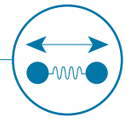
Comparable to the spacing of atoms and molecules.

Ideal for probing the **STRUCTURE** of a variety of materials, from single atomic species to complex biopolymers.

ENERGY

Comparable to the time scales of molecular diffusion, vibrations and rotations.

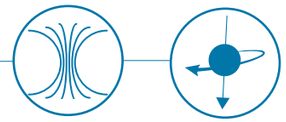
Ideal for probing the **DYNAMICS** of many processes; energy exchanges from 1 μeV to 1 eV can be detected.



SPIN AND MAGNETIC DIPOLE MOMENT

Make neutrons interact with the magnetic fields generated by unpaired electrons in materials.

Sensitive to the Microscopic **MAGNETIC PROPERTIES** of materials that can be studied in exquisite detail with polarised neutrons.



INTERACTION WITH ATOMIC NUCLEI

Cross-sections vary almost randomly with atomic number; complementary to X-rays, which interact with the electron cloud.

LIGHT ELEMENTS can be studied in the presence of heavier ones (sensitivity to hydrogen atoms is essential, from energy engineering to biochemistry).

ISOTOPES of the same element and periodic table neighbours can be distinguished (substitution, e.g. H/D, can be used to highlight specific features).

ELECTRIC CHARGE

Neutrons are electrically neutral.

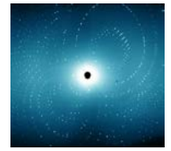
NON-DESTRUCTIVE and with **LARGE PENETRATION POWER**, well suited for complex samples and sample environments (e.g. extreme temperatures and pressures, delicate or precious samples).

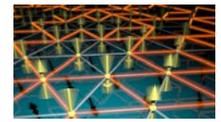
Neutron techniques, science and impact

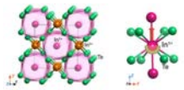
Neutron techniques are vital research tools in science and innovation. They help us to understand and develop a diversity of materials and processes in a range of societally relevant areas, including environment, energy, health and ICT, covering a range of scientific fields such as nuclear, particle and condensed matter physics, chemistry, biology, materials science, engineering and more. Over the years, advances in instrumentation have continuously improved signal quality and reduced sample requirements (size and composition), opening up fundamentally new research opportunities.

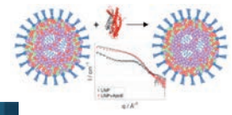
NEUTRON DIFFRACTION

When neutrons are scattered by a sample, their direction changes, giving information on the internal structure of the sample. Neutron diffraction is a powerful and often unique tool for studying the structure. It is in fact a very precise technique for measuring the structure of ordered and disordered materials, ranging from the simplest to the most complex.



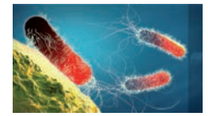






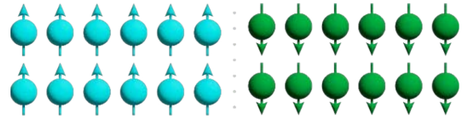
SMALL-ANGLE NEUTRON SCATTERING (SANS)

Small-angle scattering does not attempt to see atoms but is interested in the organisation of macromolecular complexes. As scattering elements are large (polymers, micelles, foams..), diffraction occurs at very small angles.



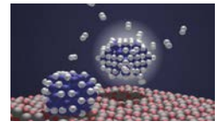
NEUTRON REFLECTOMETRY

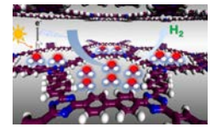
Neutron reflectometry gives information on the structure of thin films (depth-dependent composition and in-plane information) and of surfaces. It is also a powerful technique for studying interfaces (solid/solid, solid/liquid, liquid/liquid) that can be buried in multilayer systems.



NEUTRON SPECTROSCOPY

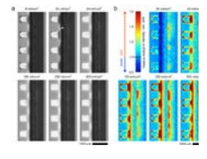
Neutron spectroscopy, or inelastic neutron scattering, observes the change in the energy of the neutron as it scatters from a sample and can be used to probe a wide variety of different physical phenomena, such as the motions of atoms (diffusional or hopping), the rotational modes of molecules, sound modes and molecular vibration, recoil in quantum fluids, magnetic and quantum excitations or even electronic transitions.





IMAGING

Neutron imaging is a non-destructive technique, highly complementary to X-ray imaging, that can see inside materials and examine processes therein. White beam imaging is based on the attenuation of the neutron beam, due to absorption or scattering, through an object. Tomography is performed by rotating the sample and reconstructing the 3-dimensional volume from a series of images.



SCIENTIFIC HIGHLIGHTS

As a service institute, ILL makes its facilities and expertise available to its 'users' (visiting scientists). At the ILL, scientists, engineers, technicians and administrators are organised in 'groups' that run the facility, develop it, and provide support to the users, making it all possible – from the reactor to the instruments, from neutron optics to detectors, from sample environment to computing, from the support laboratories to scientific coordination.

1 062
ILL and **CRG**
EXPERIMENTS

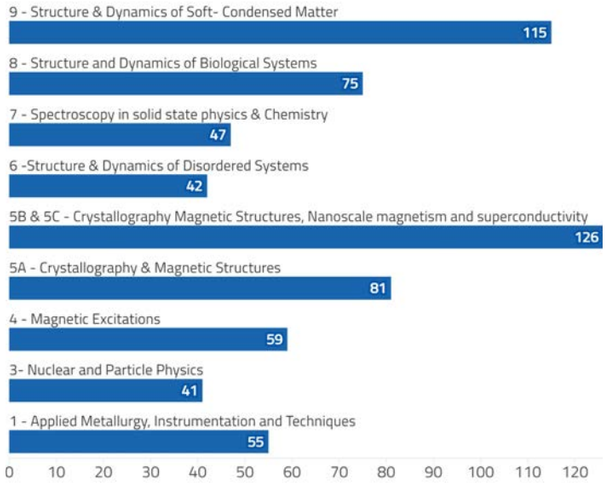
including **107** CRG
147 EASY
38 DDT
22 internal research experiments

165 DAYS OF NEUTRONS
4 262 DAYS FOR SCIENCE

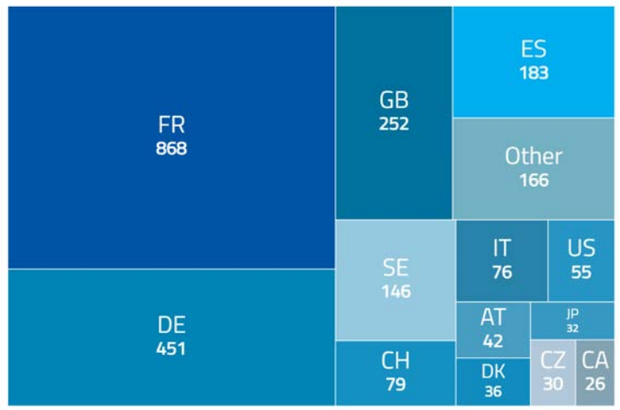
1 673
INDIVIDUAL USERS

2 442
USER VISITS
FROM **48** COUNTRIES

Distribution by college
A total of 641 accepted proposals



National affiliation of ILL users in 2023
Number of user visits

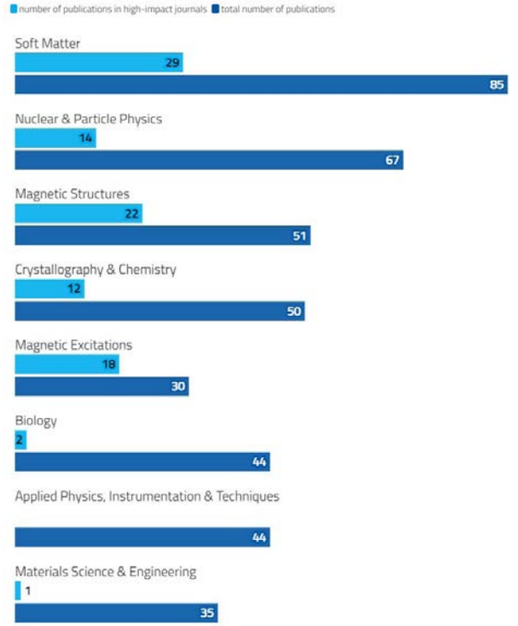


Scientific life at the ILL is organised in 'colleges', each dealing with a particular field of research and the associated neutron methods and instrumentation. ILL scientists belong to one or more colleges, depending on their personal research interests. The areas covered include condensed matter physics, chemistry, biology, materials science and engineering, Earth sciences, and nuclear and particle physics. The impact of the neutron science carried out at ILL ranges from scientific discovery and excellence to addressing societal challenges in the fields of health, the environment and climate change, energy and advanced materials for IT and computing technologies.

The scientific highlights in the following pages are grouped in terms of these impact areas.

461
ILL publications
recorded in 2023 of which **108** published in high-impact journals*

461 publications in 2023
of which 108 published in high-impact journals



TRAINING

GRADUATE School

26 full-time-equivalent ILL PhD students
41 PhD students working on ILL PhD projects
24 successfully defended ILL PhD theses

HERCULES School

115 participants of
32 different nationalities
working in **24** different countries

* This set of journals is identified by ILL as being of high impact (IF ≥ 7) or particular relevance to the neutron scientific programme.

A. Kovalevsky

Neutron Scattering Division, Oak Ridge National Laboratory, Oak Ridge, Tennessee USA - kovalevskyay@ornl.gov

My current research focuses on designing antivirals against SARS-CoV-2 and other RNA viruses, designing antidotes against nerve agent and pesticide poisoning, and studying cancer-related vitamin B6-dependent enzymes. My approach combines innovative biophysical and computational methodologies, such as X-ray and neutron crystallography at near-physiological conditions, neutron vibrational spectroscopy, biomolecular simulations, and structure-based virtual reality (VR)-assisted drug design.

Neutrons help design novel hybrid antivirals against COVID-19

Coronavirus disease 2019 (COVID-19), caused by a highly infectious severe acute respiratory syndrome coronavirus-2 (SARS-CoV-2), triggered the worst pandemic in the past one hundred years. SARS-CoV-2 continues to circulate in the human population, creating variants capable of escaping the current vaccines, threatening their effectiveness. The design of small-molecule antivirals targeting viral proteins can alleviate this problem. Detailed structural and functional knowledge of these drug targets is of critical importance for designing effective antiviral treatments and preparing for future pandemics.

Highly transmissible respiratory viruses can cause devastating pandemics, exemplified by the recent COVID-19 pandemic that has taken nearly 7 million lives and left many more individuals suffering from long COVID complications. Although vaccines to protect from the disease are now generally available, many people will remain unvaccinated due to various reasons. This allows SARS-CoV-2 to mutate, selecting for escape variants that threaten the vaccine effectiveness and necessitating a constant requirement to develop new vaccines. Small-molecule antivirals can be designed to target specific viral proteins, or drug targets, not involved in vaccine-virus interactions, helping alleviate this problem. One such drug target is SARS-CoV-2 main protease enzyme (MPro), a viral protein involved in one of the crucial steps in virus replication – the maturation process that makes the viral particles infectious. Inhibiting the MPro function therefore stops the virus replication in its tracks and can cure COVID-19.

The MPro enzyme is an established drug target for antiviral therapy. MPro functions by cleaving the peptide bonds connecting non-structural proteins within the large polyprotein chains synthesised from the SARS-CoV-2 RNA genome when the virus highjacks the cell's protein production machinery. X-ray crystallography has provided answers concerning the enzyme's overall structure and interactions with the polyproteins and inhibitors, but questions remain unresolved regarding its catalytic properties and binding of inhibitors because the hydrogen atoms that determine protonation states, electrical charges and intermolecular interactions are not visible in X-ray structures.

Neutron macromolecular crystallography can help resolve these biochemical issues because hydrogen atoms are directly located in neutron structures, providing an accurate model for drug design. Using neutron crystallography, we designed and investigated novel hybrid inhibitors of MPro that combine the chemical features of clinical hepatitis C protease inhibitors and a feline infectious peritonitis drug. This work was undertaken in collaboration with the National Institutes of Health and the University of Tennessee Health Science Center.

The neutron structure of a hybrid inhibitor BBH-1 established a complete picture of the BBH-1 intermolecular interactions with Mpro, visualising the hydrogen-binding networks and creating an accurate map of electrical charges around the inhibitor, all of which govern its ability to bind to the enzyme. We observed that BBH-1 is negatively charged, but this charge is compensated by a positively charged histidine residue that makes a hydrogen bond that is crucial for inhibitor recognition by the enzyme. Importantly, several ionisable residues near the bound inhibitor change their protonation states, hence their electrical charges, when BBH-1 binds compared to their states in the inhibitor-free enzyme.

The detailed structural information provided by the neutron structure was used in the design of novel hybrid MPro inhibitors that are derivatives of BBH-1. The new inhibitors bound tightly to the enzyme and demonstrated robust antiviral properties when investigated in human cells using live SARS-CoV-2 virus. The outcome of this study demonstrates that neutrons are indispensable for resolving biochemical issues and can provide essential information for structure-assisted drug design.

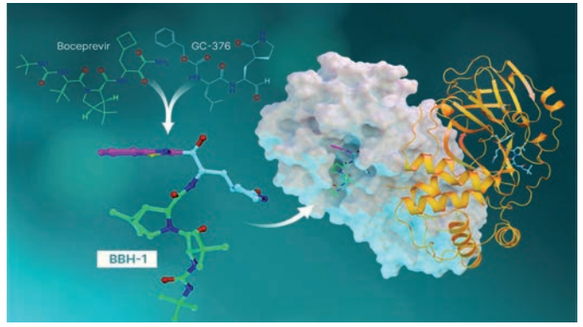


Figure 1: The drug boceprevir, a hepatitis C drug, was combined with GC-376, a feline infectious peritonitis drug, to create an experimental hybrid molecule BBH-1. BBH-1 bound to SARS-CoV-2 MPro with high affinity and the neutron structure of the complex provided essential information for designing novel hybrid MPro inhibitors which showed favorable properties for stopping the SARS-CoV-2 virus from replicating and preventing it from hijacking healthy human cells.

Original publication: Nat Commun (2022)— 10.1038/s41467-022-29915-z.
ILL contact: Matthew Blakeley, blakeleym@ill.fr
Instrument: Quasi-Laue diffractometer **LADI**

Andrea Scotti

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I am currently a Senior Lecturer at Malmö University. In November 2023, I was appointed as a Wallenberg Academy Fellow and I will continue my research in the Division of Physical Chemistry at Lund University starting in spring 2024. The main subject of my research is experimental soft matter, with particular focus on the relationship between the microscopic architecture of soft building blocks, e.g. microgels, and the macroscopic response of a soft material, both in three and two dimensions.

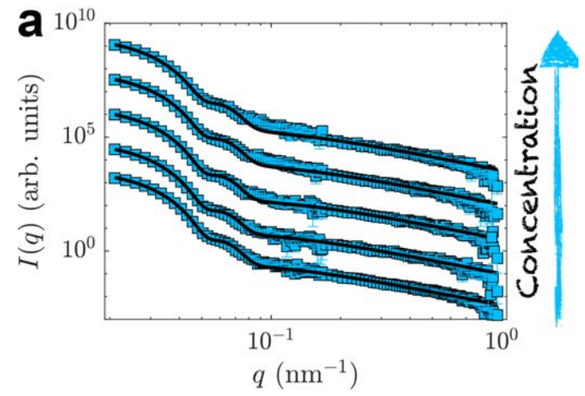
Self-healing of charged microgels in neutral and charged environments

Microgels are crosslinked polymeric networks swollen in a good solvent. One of their most remarkable properties is their responsiveness to external stimuli, such as changes in temperature, that leads to the collapse of the particle. Therefore, these colloids, which are originally soft and deformable, can become hard and compact once they are collapsed. Their capability to respond to changes in their environments, such as changes in the suspension osmotic pressure, by adapting their size is the origin of the so-called self-healing properties of microgels: when a few large microgels are mixed with a majority of small microgels, the large microgels adjust their size and fit into the crystallin lattice former by the smaller microgels without causing defects in the crystal.

Here, we study the response to crowding of ionic microgels. These particles are embedded in a matrix of either neutral or ionic microgels with comparable sizes. The microgels used for the matrix are synthesised from a deuterated monomer and can be contrast-matched during small-angle neutron scattering (SANS) experiments. The combination of SANS performed on D11 with contrast variation and small-angle X-ray scattering (SAXS) allowed us to probe simultaneously the response to crowding of individual particle and structure formed by the microgels in the samples. In particular, SANS with contrast variation allowed us to directly measure the form factor of the microgels in crowded environments (**Figure 1a**).

By changing the pH values of the suspensions, four different situations are studied: (i) uncharged ionic microgels in a matrix of neutral microgels; (ii) charged ionic microgels in a matrix of neutral microgels; (iii) uncharged ionic microgels in a matrix of uncharged ionic microgels; (iv) charged ionic microgels in a matrix of charged ionic microgels. In situations (i) and (iii), a moderate deswelling is observed, and faceting plays a role only at high packing fractions. When the charged ionic microgels are immersed in a matrix of smaller neutral microgels, these softer and larger 'defects' preferentially facet, while isotropic deswelling is minimal (**Figure 1b, left**)In contrast, faceting is not affected by the charges in the microgel polymeric network, i.e. the shear modulus can be assumed constant (**Figure 1b, right**).

Our results show that the type of microgel that forms the matrix (neutral or ionic) plays a pivotal role in determining the main response of the microgels to crowding, namely faceting or isotropic deswelling. A study of this kind was only possible thanks to the combination of X-ray and neutron scattering with contrast variation performed on D11.



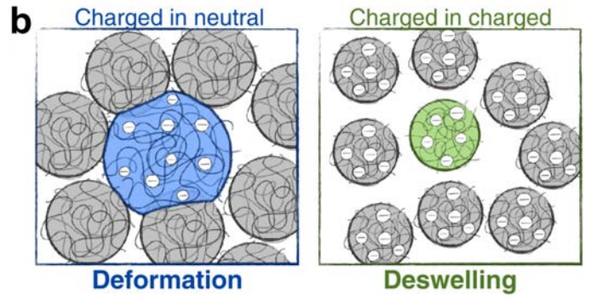


Figure 1: SANS intensity measured using contrast variation. Sketch of the response of ionic microgels to crowding.

Original publication: Langmuir— 10.1021/acs.langmuir.2c03054
ILL contact: Ralf Schweins, schweins@ill.fr
Instrument: Lowest momentum transfer & lowest background small-angle neutron scattering instrument **D11**

Robert Bradbury

Helmholtz Zentrum Berlin, Germany - robert.bradbury@helmholtz-berlin.de

I am currently employed as a Post-Doctoral Researcher at the Helmholtz-Zentrum Berlin working on solid-state Li-S batteries. I am originally from the United Kingdom and have spent the majority of my research career utilising a range of neutron techniques.

Visualising lithium-ion transport in solid-state Li–S batteries using ^{6}Li contrast-enhanced neutron imaging

Solid-state lithium-sulfur batteries offer an alternative to the currently prevalent lithium-ion batteries for use in applications ranging from electric vehicles and consumer electronics to large-scale energy storage. However, there are still performance barriers that need to be resolved before they can enter widespread use. One such limitation is the performance degradation related to changes in the solid electrolyte microstructure under operation which affects the diffusion of lithium ions through the cell.

X-ray imaging has been extensively used in battery research but it has not been possible to directly visualise the lithium itself using this technique due to the low electron density and thus low X-ray cross-section of lithium. Neutrons, on the other hand, do not have this issue and the neutron cross-section of lithium is considerably larger than many of the elements found in such batteries. As such, it is possible to directly visualise discrete changes in the lithium distribution across a cell such as formed Li_2S in a cathode, given an optimised instrument setup. However, a neutron image by itself cannot differentiate between particular lithium ions, such as those that are trapped and those that are mobile. Fortunately, the two most abundant isotopes of lithium, ^{6}Li and ^{7}Li, have significantly different neutron beam attenuation coefficients. Neutron absorption by the ^{6}Li isotope is much greater than the ^{7}Li isotope, while the naturally occurring mixture of the two isotopes is dominated by the ^{7}Li (≈92.4%).

Here, we have taken advantage of this utility to directly observe the dynamics of Li^+ ion diffusion through the solid electrolyte separator of a Li-S solid-state battery using *operando* neutron radiography and *in situ* neutron tomography. Through enrichment of a Li-In anode with ^{6}Li, the contrast with respect to the Li elsewhere in the cell was altered to the extent that it was possible to differentiate, during the initial discharge, the Li^+ ions diffusing through the separator from the anode from those that were initially located in the solid electrolyte separator. This information was not previously accessible when measurements were made with a similar cell that did not vary the lithium isotopic ratio. The 2D radiography, performed *operando*, and which concentrated on the initial discharge when attenuation changes compared to the pristine state are most clear, required an instrument capable of recording high-resolution images very quickly to demonstrate dynamically the attenuation changes across the solid electrolyte. Additionally, 3D tomography performed *in situ*, required the highest possible resolution available in order to visualise the degree of homogeneity of the lithium percolation through the separator region and thus highlight the changing ^{6}Li distribution between the discharged and charged states. The high neutron flux of the ILL reactor combined with the high-resolution setup at the NeXT imaging instrument enabled the short image collection times (6 s) and pixel sizes (14.2 µm) necessary for such a system where both time and spatial resolution are vital.

This work marks an important initial step in the characterisation of lithium-ion transport through solid electrolyte separators combining neutron imaging with selective lithium isotope enrichment. The potential is clear for future study of the dynamics of lithium-ion transport though solid electrolytes, with the aim of identifying transport pathways an important next step.

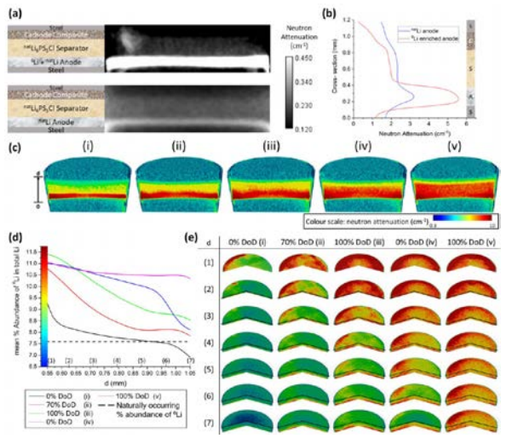


Figure 1: **a)** Radiography images showing neutron attenuation for two cells. One has a Li–In anode where the Li has the naturally occurring isotopic mix (**bottom**). In the other, the Li has been enriched with ^{6}Li (**top**). **b)** Cross-section of the median neutron attenuation (Σ_{median}) across the images displayed in (**a**). **c)** 3D tomographies for each degree of discharge (DoD, i–v) showing the ^{6}Li progression through the cell. Each tomography has been sectioned vertically for clarity. The deep red at the bottom of (i) clearly shows the position of the anode. **d)** Mean percentage abundance of the ^{6}Li isotope in the lithium contained within the solid electrolyte. Data is calculated using the tomographies from (**c**) since ^{6}Li isotope abundance is related to neutron attenuation. The colour scale pertains to the images shown in **(e)**. **e)** 3D representations showing the inhomogeneity of the percentage abundance of the ^{6}Li isotope, for charge states (i)-(v), at intervals through the solid electrolyte (1–7). Each row shows the full depth of the electrolyte separator at that *d* starting from the anode—separator interface at the bottom of the image to the current *d* position at the top, with the black line marking the edge of the vertical/ horizontal plane.

Original publication: Adv. functional mat. 2023— 10.1002/adfm.202302619
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Instrument: Neutron and X-ray tomography **NeXT**

Roberto Fernández de Luis

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My research focuses on the synthesis and chemical encoding of new and already know porous Metal-Organic Framework materials used for environmental remediation, prevention, revalorisation and monitoring purposes.

MOFs inspired by nature for environmental remediation of heavy metals

The recovery of metals from aqueous solutions targets both hazardous substances – heavy metals (e.g. cadmium, lead or mercury) – as well as industrially valuable metals which enable the generation of a parallel profit to reduce the cost of environmental remediation. Metal-organic frameworks (MOFs) – advanced sorbents which are composed of metal ions or clusters interconnected by organic linkers - are considered a promising emerging solution for environmental remediation purposes.

MOFs offer exceptional porosity, and in particular, an outstanding chemical versatility that enables their pore space to be decorated with natural acids (malic, mercaptosuccinic, succinic, fumaric and citric) and amino acids (histidine, cysteine and asparagine). These essential building blocks of life make it possible to modulate the metal-adsorption capacity and affinity of the parent mesoporous MOF-808 employed in this work, and hence, adapt its properties to the selective adsorption of specific ions (**Figure 1**).

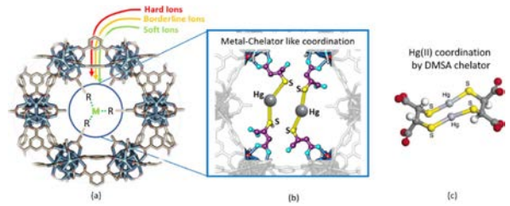

Figure 1: **(a)** Illustration of hard, borderline and soft metal-chelator-like traps within the MOF-808 scaffold. **(b)** Illustration of tentative placement of the mercaptosuccinic molecules into the MOF-808 pore space and their coordination to Hg(II). **(c)** Coordination modes of dimercaptosuccinic acid chelators over Hg(II) ions.

The MOF-808 scaffold was synthesised and post-synthetically functionalised with the amino and natural acids, then characterised using X-ray diffraction (XRD), infrared (IR) spectroscopy, thermogravimetry (TGA) and N2 adsorption measurements.

Unravelling how the various adsorbed substances interact with the different MOF variants enables the most promising versions of the MOF-808@amino acid system to be identified, in addition to guiding future modifications and improvements. However, the IR, EPR, UV-Vis and Raman spectroscopies employed to unravel the adsorption mechanisms of the amino acid-decorated pore space towards metals were insufficiently fine to enable an understanding of the materials and processes at the fundamental level. The more conventional spectroscopic techniques of IR and Raman spectroscopy were therefore combined with advanced inelastic neutron spectroscopy (INS) carried out using the ILL's IN1-Lagrange neutron spectrometer (**Figure 2**), which offers an exceptionally high flux, energy range and energy resolution, ideal for measuring intramolecular vibrations in very small samples.

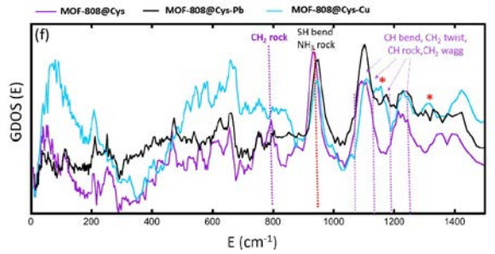

Figure 2: Inelastic neutron scattering spectra of MOF-808@Cys before and after Cu(II) and Pb(II) adsorption.

Neutron vibrational spectroscopy allows us to highlight the vibrations of hydrogen atoms that were involved in the MOF-808 functionalisation and metal-adsorption processes. Among the INS bands affected by the metal adsorption (**Figure 2**), the intensity of the signal located at 950 cm^{-1} energy is reduced in comparison to the initial MOF- 808@ Cys. The band was initially assigned to the combination of the bending vibration of SH and the rocking vibration of NH3 groups. In addition to the subtle intensity loss, adsorption of Pb(II) and Cu(II) induces a slight displacement of the signal to higher-energy values. The metal coordination by sulfide groups of Cu(II) or Pb(II) ions (**Figure 1b**) would induce a deprotonation to S-anions and, thus, a reduction of the INS signals associated with the SH vibrational modes (~950 cm^{-1}). This conclusion is based on an active participation of the –SH groups in the metal-chelation of both Pb(II) and Cu(II) ions that finally would lead to a significant reduction of the intensity of the signal.

By combining the spectroscopic techniques with Density Functional Theory (DFT) calculations, it was possible to suggest the final metal-amino acid species stabilised within the MOF after adsorption. The immobilisation of amino acid functions in the pore space of MOF-808 endows the material with metal complexing mechanisms where single or cooperative metal binding modes give rise to the stabilisation of isolated, clustered, and even partially reduced species for some specific metals such as copper. We anticipate that amino acid encoding, and in particular multivariate decoration of MOFs with multiple amino acid residues, will become an important tool for engineering artificial metal-chelator functions with improved selectivity to trap metal ions from the complex mixture.

Original publication: Chem. of Materials— doi.org/10.1021/acs.chemmater.2c02431
ILL contact: Mónica Jiménez-Ruiz, jimenez@ill.fr
Instrument: Large Area GRaphite ANalyser for Genuine Excitations **IN1-Lagrange**

Ren Qingyong

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I obtained my PhD from the University of New South Wales, Canberra in 2016, followed by three and a half years of postdoctoral research at Shanghai Jiao Tong University. In 2020, I joined IHEP, CAS and CSNS to work on the first inelastic neutron scattering TOF spectrometer in China. My research focuses on lattice dynamics of condensed matter and functional materials.

Phase transition and ionic diffusion in superionic Ag_8SnSe_6 thermoelectrics

Superionic phase transition is an important issue in condensed matter physics and functional materials because superionic materials always exhibit ultralow lattice thermal conductivity and excellent ionic conductivity. Understanding the details of phase transition can guide better design for thermoelectric materials and solid-state electrolytes. We used the hot neutron 4-circle diffractometer D9 to resolve complex crystal structures and to track their changes across phase transition. This work revealed that Se atoms play a key role in the superionic phase transition.

Superionic phase is a novel state of matter. It usually consists of two sublattices, one of which forms a rigid framework while the other one can move or diffuse within the framework. Most superionic materials have ultralow lattice thermal conductivity, which is beneficial for thermoelectric energy converters. In addition, the excellent ionic conductivity property makes them a promising group of solid-state electrolytes for energy storage batteries. Although these materials have been extensively studied for energy conversion and storage applications, the atomistic mechanisms for their superior thermal transport properties remain elusive due to a lack of comprehensive understanding of their crystal structures across the superionic phase transition. One of the typical superionic materials are the argyrodite-type compounds, such as Ag_8SnSe_6.

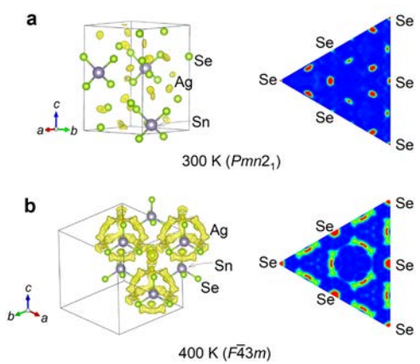

Figure 1: **Left)** The crystal structures of the Ag_8SnSe_6 below and above the superionic phase transition temperature (~355 K) and maximum entropy method (MEM) analysis on the atomic distributions (yellow iso-surface). **Right)** The nuclear density of Ag and Se atoms in a certain cutting plane.

To explore the crystal structures of Ag_8SnSe_6, we prepared single crystals and used the hot neutron 4-circle diffractometer D9 at the ILL. Refinements of the single crystal data revealed an orthorhombic structure with a space group of $Pmn2_1$ at 300 K (below the superionic transition temperature T_C ~355 K) and a cubic structure with $F\text{-}43m$ at 400 K. Then, the model-free maximum entropy method (MEM) is adopted to reveal a realistic nuclear density distribution. As shown in **Figure 1**, the Ag ions in the low-T phase are constrained around localised potential energy. On the other hand, they are distributed over a large and interconnected region in the high-T phase, as marked with the yellow iso-surface in the left panel or the banana-like contours in the right panel of **Figure 1**. More interestingly, the comparison of the crystal structures below and above T_C indicates that the Se_4 and Se_5 atoms in the orthorhombic structure, corresponding to the Se_1 and Se_2 atoms in the cubic structure, present obvious changes in the atomic positions and atomic displacement parameters (**Figure 2**). These unusual behaviours suggest that these two special Se atoms play a key role in the superionic phase transition, which was further supported by our quasi-elastic and inelastic neutron scattering measurements as well as the machine-learned molecular dynamic simulations.

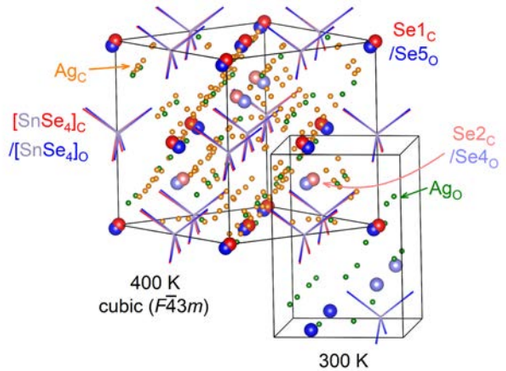

Figure 2: Comparison of the crystal structures below (orthorhombic, $Pmn2_1$) and above (cubic, $F\text{-}43m$) the superionic phase transition (from the refinement of single crystal diffraction data).

Original publication: Nat. Mater. 22, 999 (2023)— 10.1038/s41563-023-01560-x

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Instrument: Hot neutron four-circle diffractometer **D9**

Leonardo Chiappisi

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Scientific Coordinator of the Partnership for Soft Condensed Matter at the ILL.

My research delves into the captivating realm of colloids. My interests encompass a wide spectrum, ranging from fundamental studies on surfactant and polymer solutions to their intricate mixtures, foams and responsive coatings.

Pressure-induced phase transitions of nonionic polymer brushes

In the realm of smart materials, the temperature-responsive properties of nonionic polymers have been extensively studied. They are recognised as key mechanisms in materials science and are exploited in various applications, such as drug delivery, sensors, mechanical actuators and optoelectronic devices. However, the pressure response of such films has largely remained unexplored, primarily due to the technical challenges associated with high-pressure experiments. In this research, we delve into the conformational transitions of nonionic brushes and semi-dilute solutions triggered by hydrostatic pressure.

Our investigation, employing neutron reflectometry to probe the coil-to-globule transition of brushes, reveals an intriguing alignment between the pressure–temperature phase diagrams of brushes and semi-dilute solutions. Performing neutron reflectometry experiments under high-pressure conditions of up to 2 kbar represents a significant technical challenge, which was successfully overcome thanks to the expertise in high-pressure techniques at the ILL and the use of the high-flux neutron reflectometer FIGARO.

Remarkably, we demonstrate that the phase behaviour of nonionic polymers can be understood and predicted using simple thermodynamic concepts traditionally applied in protein denaturation studies. To achieve this, an extensive exploration of the free energy landscape for the coil-to-globule transition of nonionic polymers was conducted using calorimetry, density and sound velocity measurements—techniques made available by the Partnership for Soft Condensed Matter (PSCM) on the EPN campus.

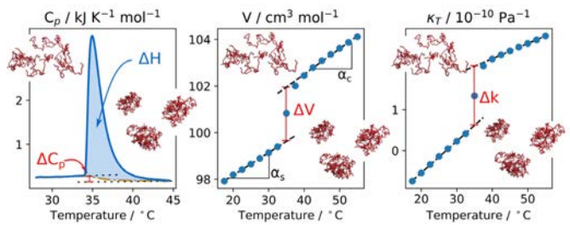

Figure 1: From **left** to **right**: Heat capacity as a function of temperature, obtained by differential scanning calorimetry allowing the determination of the entropy and heat capacity change of dehydration; Partial molar volume of PNIPAM as a function of temperature obtained from density measurements allowing the determination of molar volume and expansibility change of dehydration; Isothermal compressibility as a function of temperature, obtained from sound velocity measurements allowing the determination of compressibility change of dehydration.

This predictive power facilitates the design of polymer-based additives and coatings for high-pressure applications.

Further insight into the pressure-responsive behaviour was obtained from molecular dynamics simulations at the fully atomistic level, conducted at the Jožef Stefan Institute in Ljubljana. Our combined experimental and simulation approaches unveil a universal phenomenon, where the hydration of the polymer by water molecules determines the temperature and pressure-responsive behaviour at low-pressure values. Conversely, pressure-induced dehydration at high pressure emerges as a polymer-specific phenomenon and arises from the reorganisation of the hydrogen-bond network upon polymer collapse.

These findings are applicable across a broad spectrum of nonionic polymers, offering a valuable framework for designing responsive coatings with tailored pressure-responsive behaviours. This work contributes to advancing our understanding of the intricate interplay between pressure and polymer conformation, paving the way for innovative applications in materials science.

Shifting our attention to more biologically relevant questions, the pressure-induced hydration of polymer chains may explain the low friction coefficients found in synovial fluids, offering implications for poly(zwitterionic) brushes. Moreover, our experimental approach, combining structural and thermodynamic studies, holds promise for unravelling desolvation mechanisms in more complex polymer systems, including those with salts or low molecular weight organic compounds, or in non-aqueous solvents.

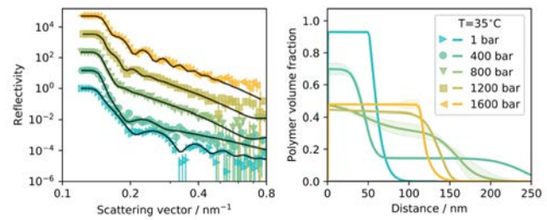

Figure 2: Neutron reflectivity data on PNIPAM brushes (on the **left**) and the corresponding brush volume fraction profiles (on the **right**). The full lines represent the average value of the obtained volume fraction profile, while the shaded areas indicate the standard deviation of the neutron reflectivity data.

Original publication: Macromolecules— 10.1021/acs.macromol.2c01979

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Instrument: Fluid Interfaces Grazing Angles ReflectOmeter **FIGARO**

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I completed my EngD in the Structural integrity assessment of WAAM steel components for marine applications at the University of Strathclyde in 2023. I currently work as a Research Associate at the University of Bristol.

The surface treatment effects on fatigue performance, residual stress distribution and texture of wire arc additively manufactured low-carbon steel components

Fatigue significantly impacts engineering structures subjected to cyclic loading. To address this, various mechanical and surface treatments have historically aimed to enhance fatigue life. Wire Arc Additive Manufacturing (WAAM) offers cost-efficient large-scale component production but introduces challenges such as residual stresses. This study investigates the impact of surface treatments (rolling and laser shock peening) on fatigue crack propagation in WAAM-built ER70S-6 and ER100S-1 steel specimens, addressing fatigue performance, residual stress, microstructure and texture. The study aims to unlock WAAM's potential for offshore renewable energy structure manufacturing, re-manufacturing and repair.

A series of fatigue crack propagation tests were performed on surface-treated ER70S-6 and ER100S-1 WAAM-built specimens, and the results were analysed and compared with the untreated materials tested under the same loading conditions. The obtained results were interpreted in terms of the sensitivity of the cracking behaviour to the specimen orientation and extraction location. For ER70S-6 steel, both treatments extended test duration for vertical specimens, while horizontal specimens saw a 15% reduction. Laser peening on ER70S-6 vertical specimens achieved the longest duration, but horizontal specimens showed faster crack propagation. ER100S-1 specimens displayed less pronounced effects, with laser peening enhancing fatigue life in vertical specimens. Surface rolling was less effective than laser shock peening. FCG rates correlated with the stress intensity factor range were lower for treated ER70S-6 specimens, and laser peening demonstrated significant improvement for ER100S-1 bottom specimens.

Furthermore, the residual stress profiles were measured using a monochromatic strain diffractometer SALSA at the ILL (**Figure 1a**), before and after applying the surface treatment techniques, and the effects of locked-in residual stresses on the fatigue performance of WAAM-built components were discussed. **Figure 1b** reveals the pronounced tensile residual stresses in the untreated ER100S-1 vertical top specimen, shifting post-laser peening to significant compressive stresses, benefitting fatigue life. In contrast, the ER100S-1 horizontal bottom specimen initially had compressive stresses, reversed by surface rolling to damaging tensile stresses, leading to the shortest fatigue life. Through-thickness distribution showed asymmetry, with laser peening inducing higher compressive stresses near the surface compared to rolling. Laser peening demonstrated higher efficiency on ER100S-1 specimens, confirming consistency with similar studies on WAAM-built components.

This study unveils crucial insights for enhancing fatigue performance in Wire Arc Additive Manufacturing (WAAM) components, aiming to broaden industrial applications. Assessing laser shock peening and surface rolling effects on fatigue crack growth, residual stress and material texture in low-carbon steel revealed advantages and limitations. Both treatments improved ER70S-6 fatigue life, with laser peening excelling. Laser-peened vertical specimens from ER70S-6 bottom and ER100S-1 top WAAM walls showed superior durability. Conversely, uniformly applied rolling on ER100S-1 proved inefficient, increasing FCG rates and shortening fatigue life. Surface rolling induced detrimental near-surface residual stresses, while laser peening introduced beneficial compressive stresses, reducing FCG rates. Residual stress measurements revealed slight asymmetry, probably due to non-uniform treatment.

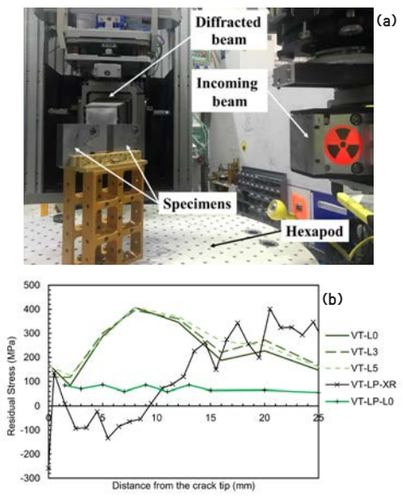

Figure 1: a) Neutron diffraction test set-up at SALSA instrument; **b)** Residual stress distribution in ER100S-1 vertical specimen before and after laser shock peening.

Original publication: Journal of Materials Research and Technology— 10.1016/j.jmrt.2023.03.227
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Instrument: Strain imager for engineering applications **SALSA**

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I'm a postdoctoral fellow working in the Theory group at the ILL. My scientific interests lie at the interface between physics, chemistry, and recently, biology. I'm currently interested in modelling disordered proteins that are vital for cell functioning and material design applications. In collaboration with researchers at the ILL and in Germany and Sweden, I combine theoretical, numerical and scattering methods to investigate the structural and dynamic properties of a variety of disordered protein assemblies.

Structure and dynamics during the hydrophobic collapse of elastin-like polypeptides

Elastin is a key extracellular matrix protein that provides resilience and elasticity to biological tissues such as lungs, blood vessels and skin. These properties are encoded in its monomer - tropoelastin, which is composed of alternating cross-linking and hydrophobic domains. Inspired by these hydrophobic domains, elastin-like peptides (ELPs) are a minimal model to understand the early stage of self-assembly of tropoelastin into fibers and are sought after in biomedical applications due to their stimuli-responsive nature and biocompatibility. In particular, ELPs feature a hydrophobic collapse upon crossing a critical temperature. Even though elastin and ELPs have been extensively studied, their structure remains controversial.

Here, we employed state-of-the-art atomistic modelling combined with advanced sampling methods to resolve the early stage of assembly of short ELPs in a wide range of peptide concentrations and temperatures, including their critical temperature. We found that already at the single chain level, the peptide shows signs of a hydrophobic collapse as the temperature increases. We additionally constructed a stochastic model of a peptide near its conformational transition using Markov state modelling (MSM). The simulation trajectory was successfully mapped into two Markov states shown in **Figure 1a.** The first state is rich in conformations with a transient secondary structure formed by valine residues, while the latter state is fully disordered. Next, the calculations of the potential of mean force between two peptide chains displayed a change in the interactions from repulsive to attractive with temperature (**Figure 1b**) consistent with the expected phase behaviour. In multi-chain systems, we observed the formation of dynamical aggregates with a coil-like conformation of the chains. Analysis of contacts between chains revealed that central valine residues play a key role in contact formation, and sequence modification with residues of a lower hydrophobicity substantially decreased the probability of contact formation.

One example of the sequence modification is shown in **Figure 1c.** Interestingly, these valine residues are the ones that engage in the secondary structure motifs depicted by the MSM for a single-chain system (state S1), highlighting the similarity between intra- and interpeptide interactions. Finally, the peptide translational and internal motions were slowed down by an increase in peptide concentration and temperature. For the latter, we computed the self-part of the van Hove correlation function for hydrogen atoms belonging to the peptide backbone for future comparison with experiments (**Figure 1d**).

Our results are valuable for designing applications employing thermo-responsive polymeric materials. Currently, we are complementing our findings with experimental results obtained by small-angle neutron and x-ray scattering, as well as quasi-elastic neutron scattering performed on the same peptide sequence. Additionally, we are extending our numerical work to other chain lengths to probe the generality of our findings.

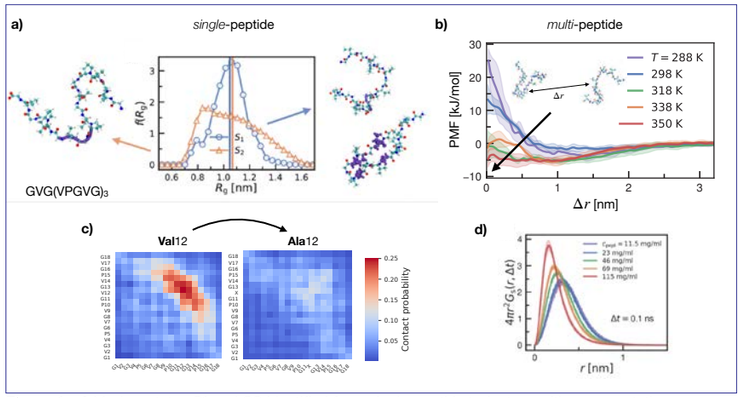

Figure 1: a) Markov states S1 and S2 identified for a single-peptide trajectory with representative snapshots of the peptide's conformations; **b)** Potential of mean force for a pair of two peptides as a function of temperature; **c)** Interpeptide contact maps for the original sequence (**left**, Val12) vs the sequence modified with alanine (**right**, Ala12); **d)** van Hove self-correlation function Gs(r, Δt) computed using the backbone hydrogen atoms at T = 298 K for the time windows Δt = 0.1 ns.

Original publication: Biomacromolecules— 10.1021/acs.biomac.3c00124
ILL contact: Olga Matsarskaia, matsarskaia@ill.fr
Instrument: Small-angle neutron scattering **D11**

Stanislav Nikitin
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I completed my PhD at the Max Planck Institute CPfS in Dresden in 2020 working on strongly correlated systems. My current research focuses on exotic phases and excitations in quantum magnets and topological materials. Neutron scattering is a powerful tool that helps us to study this kind of phenomena with outstanding resolution.

Thermal evolution of Dirac magnons in the honeycomb ferromagnet $CrBr_3$

Graphene owes most of its famed physical properties to its honeycomb lattice structure. We have investigated its magnetic equivalent, chromium tribromide $CrBr_3$. This material, which possesses identical lattice geometry to graphene, is still magnetic when slimmed down to a single atomic layer. Using advanced neutron scattering, we probed the magnetic band structures in a single crystal of $CrBr_3$ at unrivalled resolution. The complex magnetic topology revealed extends our fundamental understanding of magnetism and may lead to exciting technological applications.

Electrons in graphene behave like the massless quasiparticles described by the Dirac equation, because their bandstructure exhibits Dirac cones at the K point of the Brillouin zone (BZ). The same type of bandstructure governs bosonic quasiparticles in systems such as 2D ferromagnets (FM). The topology of magnon bandstructures has become a subject of active theoretical and experimental research due to possible applications in spintronic devices. We have performed a comprehensive study of the temperature-induced renormalisation of the magnon self-energy in the model honeycomb FM $CrBr_3$ using modern neutron spectrometers (PANTHER at the ILL and EIGER at PSI).

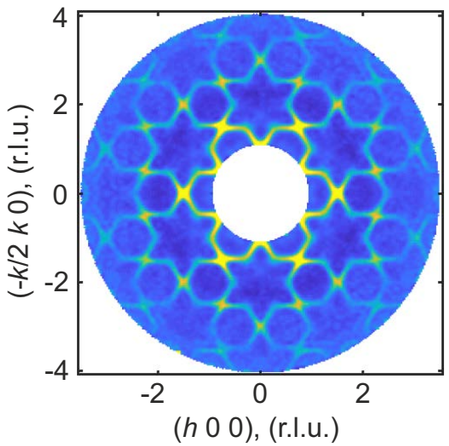

Figure 1: Constant-energy slice taken at E = 10±0.2 meV showing clear well-defined magnon excitations measured on PANTHER at T = 2 K with E_i = 15 meV.

We first used low-temperature INS data **(Figure 1)** to refine the magnetic spin Hamiltonian and determine the weak next-neighbour interactions. We proved that the magnon dispersion has nearly perfect Dirac cones at the K point with no discernible gap. A recent report to the contrary is shown to be an artifact of the data treatment **(Figure 2)** which arises because of inaccurate integration due to limited counting statistics. We also found that the topological consequences are reflected in the intensity winding. For temperatures up to 40 K, there is a considerable downward renormalisation of the magnon dispersion and growing linewidths, whose T^2 form we characterise to high accuracy, but whose variation across the BZ is demonstrably not well captured by the available theory. In this way, our results establish the experimental standard for the temperature-induced modification of the spin dynamics in a honeycomb ferromagnet.

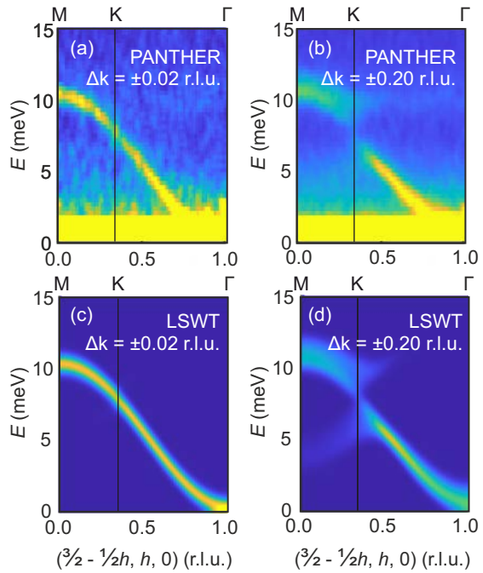

Figure 2: **(a,b)** INS spectra from PANTHER obtained by integrating the measured intensities over different widths (Δk) in the orthogonal direction; Panels **(c,d)** show the analogous results modelled within LSWT. The spectrum is entirely continuous as Δk is taken to zero, whereas increasing Δk causes an apparent spin gap due to the small magnon density of states at the K point.

Original publication: PRL (2023) — 10.1103/PhysRevLett.129.127201
Contact author: Björn Fåk, fak@ill.fr
Instrument: Thermal neutron time-of-flight spectrometer **PANTHER**

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Laboratory for Neutron Scattering and Imaging, Paul Scherrer Institut, Switzerland - Jennifer.graham@psi.ch

I completed my PhD in Chemistry this year at the ILL and the University of Birmingham (UK), where I mainly used (diffuse) neutron diffraction to understand quantum magnetic materials. I have now moved to PSI to start my first postdoc in the muon group.

Experimental evidence for the spiral spin liquid in $LiYbO_2$

Spiral spin liquids are an exotic class of correlated paramagnets with an enigmatic magnetic ground state composed of a degenerate manifold of fluctuating spin spirals. In this study, we prove the existence of the spiral spin liquid ground state in $LiYbO_2$ – the first such example in a distorted diamond structure – through advanced reverse Monte Carlo analysis on diffuse neutron scattering data.

Even within the realm of quantum materials, a spiral spin liquid is a rare magnetic ground state. Born out of the continuous fluctuations of moments that connect in short-range spiral structures, these materials are interesting from a fundamental perspective but also for their potential applications in emerging technologies, such as spintronics. However, finding a spiral spin liquid is an experimental challenge, mainly due to their sensitivities to structural distortions.

Recently, a new model supported the formation of a spiral spin liquid in a distorted diamond structure, with $LiYbO_2$ being identified as a prime candidate for the model. We confirmed the degree of distortion in the diamond sublattice of $LiYbO_2$ was sufficient to fit the model through high-resolution powder diffraction data on the ILL instrument D2B.

Next, we turned our attention to the magnetic structure, which requires both average and local structure methods due to the partially ordered nature of the spiral spin liquid. We started by confirming the long-range order with high-resolution neutron powder diffraction data from WISH (ISIS). We found that an incommensurate helical structure exists below 450 mK, which crucially had the correct phasing of moments to validate the spiral spin liquid model from an average structure perspective **(Figure 1)**.

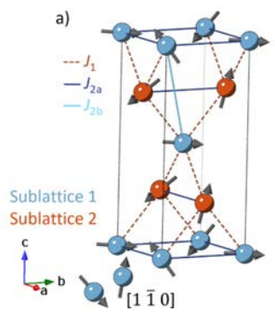

Figure 1: Visualisation of the average magnetic structure of $LiYbO_2$, as determined by WISH.

However, there was one element of the story that didn't quite add up - the calculated moment size from WISH was less than half of what was expected. We believed that this discrepancy could be explained through the partial ordering of the spiral spin liquid model. Specifically, part of the moment should be static, which gives rise to the long-range incommensurate order, and part should be contained in the dynamic motion of the fluctuating spin spirals which will result in diffuse scattering.

To test this hypothesis, we conducted diffuse neutron scattering measurements on D7 at the ILL. Our data showed a mixture of Bragg and diffuse features concomitant with what is expected for a spiral spin liquid. We fit these data with a modified version of the reverse Monte Carlo program, SPINVERT, to simultaneously model the magnetic Bragg and diffuse features, and in doing so could account for the full magnetic moment. From the fits to the powder data, we were then able to reconstruct the expected single-crystal neutron scattering planes. **Figure 2** shows the (h h l) plane in which a continuous ring of diffuse scattering, representing the contoured spiral surface, can clearly be seen. As this is the expected manifestation of single-crystal magnetic neutron scattering, these results therefore confirm that $LiYbO_2$ has a spiral spin liquid ground state.
In proving that the elongated diamond lattice structure of $LiYbO_2$ can host a spiral spin liquid, these findings improve our fundamental understanding of magnetic phenomena and pave the way for the design of new magnetic systems with advanced technological applications.

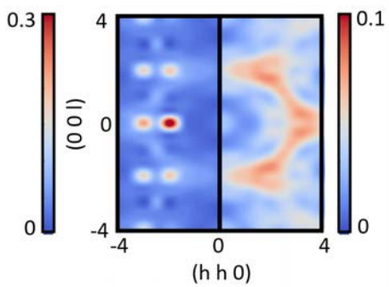

Figure 2: Reconstruction of the expected single-crystal diffuse neutron scattering from powder data analysis at 50 mK. (h h l) scattering plane shows a continuous ring of diffuse scattering, the experimental signature of the spiral spin liquid (**left**: total magnetic scattering, **right**: diffuse ring only).

Original publication: PRL (2023) — 10.1103/PhysRevLett.130.166703
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Instrument: Diffuse scattering spectrometer, **D7** and high-resolution powder diffractometer, **D2B**

Andrew Wildes
Institut Laue Langevin, Grenoble, France

I'm an ILL instrument scientist, co-responsible for the diffuse scattering spectrometer D007. I was recruited to the ILL last century and have the dubious achievement of having subsequently worked in all the neutron scattering instrument groups in the Science Division.

Understanding the properties of 'magnetic graphene'

The discovery that graphite could be thinned down to an atomic monolayer, creating graphene, led to a Nobel Prize and has enormous potential in technology from health to energy production to engineering. Graphite has this ability due to its crystal structure, consisting of strongly bound atomic layers that have weak interplanar binding. Many other compounds have similar crystal structures and can also be delaminated to an atomic monolayer. Some of them are magnetic, promising great benefits in electronics, optics and data storage.

$NiPS_3$ is one such magnetic compound, belonging to a broader family of magnetic transition metal thio- and selenophosphates containing Mn, Fe and Co. Their atomic structures are quasi-two-dimensional, similar to graphite, being composed of layers of strongly bound atoms that, in turn, are weakly bound through van der Waals forces. The compounds are antiferromagnets at low temperature and the magnetism is also quasi-two-dimensional. Most of the family members will maintain their magnetic structure when thinned to a single atomic monolayer.

The magnetic structures and dynamics vary markedly with the metallic ion species, and $NiPS_3$ has a number of properties that make it particularly interesting. The Ni^{2+} ions have the smallest magnetic moment of the family, and $NiPS_3$ might be expected to host exotic quantum effects. It has strong coupling between its electronic and magnetic properties, which could be exploited in technology, and it has been used to make workable devices such as field-effect transistors. The compound may host bound electron-hole states that are also coupled to the magnetism, making it a potential model compound for the study of magneto-electron physics in low dimensions. $NiPS_3$ is the only one of the thiophosphate compounds that does not magnetically order when thinned to a monolayer, suggesting a strong dependence of the dynamics on anisotropy and dimensionality. Conversely, $NiPS_3$ has, at T_N = 160 K, the highest magnetic ordering temperature of the thiophosphate compounds, which is somewhat surprising considering its small spin and anticipated quantum-like behaviour.

A detailed knowledge of the spin dynamics is required to understand the magnetic properties of $NiPS_3$, and neutron inelastic scattering is the experimental method of choice. Experiments using a variety of neutron instruments at ILL, ISIS and ANSTO revealed a number of remarkable results.

The magnetic structure, shown in **Figure 1**, comprises ferromagnetic chains that are antiferromagnetically coupled. Representative inelastic neutron scattering data are shown in **Figure 2.** The data show clear features associated with four magnon branches, as expected for the magnetic structure, whose energies could be reproduced using linear spin theory. Gaps at the Brillouin zone centres are due to a strong planar anisotropy, and there is a relatively strong dispersion between the *ab* planes, meaning that $NiPS_3$ is more three-dimensional than its sister compounds. Both observations are consistent with the loss of long-ranged order when $NiPS_3$ is thinned to a monolayer, as the Mermin-Wagner theorem explains that such order cannot exist in two dimensions with planar anisotropy.

However, linear spin wave theory could not accurately reproduce the neutron intensities, particularly at the Brillouin zone centres. Neutrons are a quantitative probe, and the fact that semi-classical theory could not reproduce the intensities suggests that more unusual, possibly quantum, physics are at play.

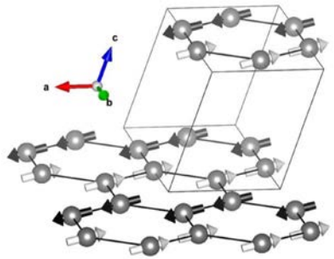

Figure 1: The magnetic structure of $NiPS_3$

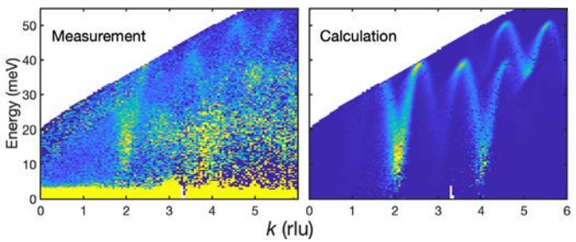

Figure 2: Neutron scattering data from MERLIN showing spin waves in $NiPS^3$ (**left**) with a calculation showing the expected scattering from the best-fitting model (**right**).

Original publication: Phys. Rev. B— 10.1103/PhysRevB.106.174422
ILL contact: Andrew Wildes, wildes@ill.fr
Instrument: Thermal neutron three-axis spectrometer **IN8**

David Lhuillier
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I'm a physicist in the Nuclear Physics Department at CEA-Saclay (DPhN), specialising in reactor neutrinos. I'm behind the work that revealed the reactor antineutrino anomaly, and spokesperson for the STEREO experiment.

Reference neutrino spectrum of ^{235}U fission established by the STEREO experiment

Reactors are very intense sources of antineutrinos, allowing us to probe the rich physics of these elementary particles. The STEREO experiment has just made a precise measurement of the spectrum of antineutrinos associated with ^{235}U fission. It rejects the hypothesis of the existence of sterile neutrinos to explain the "reactor antineutrino anomaly", much debated in recent years, and provides reference data to improve the quality of nuclear databases.

In 2011, a reassessment of the prediction of the neutrino spectra emitted by nuclear reactors led to the "reactor antineutrino anomaly" (RAA): an average deficit of 6.5% was observed between measured neutrino fluxes and this prediction.

The hypothesis of a new type of neutrino, called sterile, was put forward, with potentially important repercussions in many fields of physics. The STEREO experiment aimed to test this hypothesis unambiguously, by exploiting the fact that if the RAA is caused by a sterile neutrino, the deficit would only be the average of an oscillating pattern, which should become visible when approaching very close to a compact reactor core. The ILL's high flux reactor offered a promising site, even if STEREO didn't want neutrons! The collaboration (CEA-Irfu, CNRS-LPSC, CNRS-LAPP, ILL and MPIK-Heidelberg) therefore developed a dedicated detector: a neutrino target volume divided into 6 identical cells to scrutinise any oscillations in counting rates induced by the sterile neutrino. It was filled with 2 m^3 of scintillating liquid to sign the interaction of antineutrinos. The assembly was isolated from external background by 100 tons of shielding and inserted under the transfer canal on level C of the reactor, 10 m from the core, to provide additional protection against cosmic rays. Thanks to the strong support of ILL staff, data collection could begin at the end of 2016 and lasted until the end of 2020, with over 100,000 neutrinos detected. Combined with a high degree of precision in understanding the detector's response, our experiment has become a leader in the field. Published in Nature, our final results reject the sterile neutrino hypothesis with a high degree of confidence (**Figure 1**), as the spectra of all 6 cells are measured to be compatible with each other. By merging all these data, we have also obtained a spectrum of neutrinos emitted by ^{235}U fission that is a reference in the community (**Figure 2**). It confirms the deficit with respect to the prediction and highlights a local distortion in shape between 5 and 6 MeV.

Thus, the success of STEREO has led to a complete study of the RAA, leading to the conclusion that it does not stem from the existence of a sterile neutrino, but from biases in the nuclear data involved in the prediction. We are witnessing a paradigm shift: after having long relied on nuclear data, precise and model-independent measurements of reactor neutrinos are now being used as a reference to improve the quality of these data.

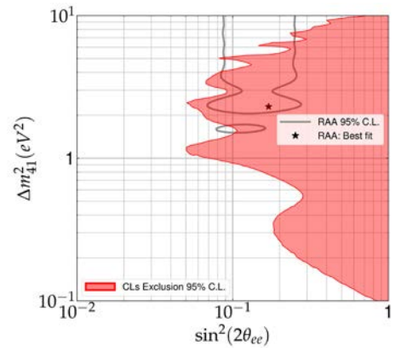


Figure 1: Contours in the plane of the frequency of the oscillation (vertical axis) versus its amplitude (horizontal axis). The STEREO results exclude all parameters in the red-shaded area at 95% confidence level, rejecting most of the domain of existence of the sterile neutrino deduced from the RAA (black curve). The remaining uncovered domain at high frequency is in strong tension with cosmological models.

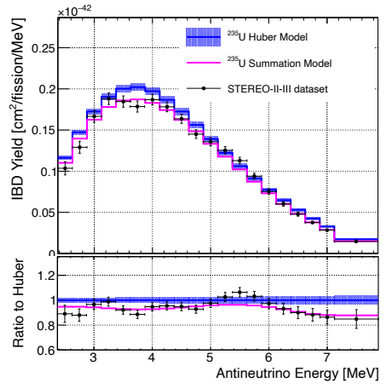
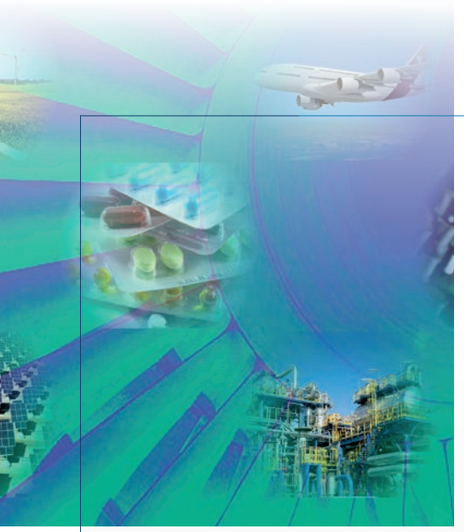


Figure 2: Neutrino spectrum of ^{235}U fission as measured by STEREO (black points). The comparison with the initial prediction (blue) shows the mean deficit as well as a gaussian-like local distortion in the 5-6 MeV range. The latest calculations (magenta), based on corrected nuclear databases, recover a good agreement with the total rate, while still missing the local bump.

Original publication: Nature— 10.1038/s41586-022-05568-2
ILL contact: Torsten Soldner, soldner@ill.fr
STEREO Detector

INDUSTRY



The Industrial Liaison Unit (ILU) acts as the interface between the ILL's scientific activities and industry. Its role is to promote the ILL's advanced characterisation capabilities to potential industry clients, and facilitate access. Working with industry is important for the ILL as a way of demonstrating its broader societal relevance and impact. Whilst the main focus of the ILL will always be scientific excellence, industrial collaborations generate important direct and indirect socio-economic benefits that cannot be achieved without industry's participation. This reinforces the ILL's philosophy of producing 'neutrons for society'.

2023 was a busy year for industrial access at the ILL. There were 141 scheduled experiments within the user programme that self-declared some form of industrial relevance. Within this group, twenty-two user experiments took place during the year that involved at least one proposer from industry. These correspond to non-proprietary access through the ILL's standard user programme. A further twenty proprietary industry experiments took place in 2023 permitting our industrial clients dedicated access, scientific support and data confidentiality.

Almost every industrial sector was represented within the broad range of companies using the ILL's state-of-the-art facilities in 2023. This included pharmaceuticals, materials science, consumer products, electronics, aerospace and renewable energies – specifically hydrogen power technologies and battery storage. This gives a holistic view of where the advanced capabilities of the ILL and the expertise of its staff can bring value to industrial activities, such as new product development, industrial R&D, process optimisation and quality control. Amongst the many companies who accessed the ILL's instruments through our standard user programme in 2023 were BASF, AstraZeneca, Proctor & Gamble, Seagate and Siemens Gamesa. Companies accessing through the user programme do so through an open-call proposal system where the proposal details are in the public domain.

2023 saw some significant staff changes within the ILU with the departure of Caroline Boudou and the arrival of Richard Davies. Richard brings to the role a strong background in industrial innovation, new product development and R&D, having held leadership roles within industry in those areas. 2023 also saw the departure of Manon Letiche. Although Manon was not directly part of the ILU, she supported industrial access at the ILL through her role as the TENIS instrument responsible (an instrument dedicated to industrial applications in the electronics industry, and funded through IRT Nanoelec). Manon's replacement, Emmanuel Atukpor, joined the ILL in September and has already delivered a successful campaign of industrial experiments on TENIS.

A final point worth mentioning regarding the ILU is its role supporting the ILL's participation in national and international projects involving industry. The aforementioned IRT Nanoelec is one such example, with others including ReMade, RADNEXT, BIG-MAP and EASI-STRESS. Many of these high-profile projects receive funding through the European Union.

45 Experiments involving industry

141 Experiments with industrial relevance

40 **Publications** with industrial co-authors

RADIONUCLIDE PRODUCTION FOR MEDICINE

Radionuclides* are the main ingredients of the radio-pharmaceuticals that are required for all nuclear medicine applications. For many years, this discipline focussed mostly on diagnostic applications, using Tc-99m and other γ-ray emitters for molecular imaging applications with so-called gamma cameras or SPECT (Single Photon Emission Computed Tomography) or using F-18 and other positron emitters for imaging by PET (Positron Emission Tomography). Therapeutic applications are also possible, but for a long time remained restricted to niche areas, e.g. the treatment of metastasised thyroid cancer, a relatively rare type of cancer, with I-131. In recent years, the emergence of novel radiolabelled molecules that can target other types of cancer cells has enabled a significant broadening of the clinical applications of therapeutic radionuclides. In particular, the use of Lu-177-PSMA (Prostate Specific Membrane Antigen) compounds has led to a breakthrough in the treatment of metastasised prostate cancer, a very common type of cancer. After successful clinical trials, the first compound of this class was approved in 2022 by the medical agencies in the US, UK and EU under the brand name Pluvicto©, marketed by Novartis.

Pharma companies have the necessary expertise to run clinical trials, synthesise drugs, perform quality control and organise drug distribution to pharmacies and hospitals. However, the primary ingredients of the drugs, in this specific case the radionuclides, are usually provided by other players.

So-called "no-carrier-added" lutetium-177, the highest quality of Lu-177 that is required for such applications, is produced by irradiating enriched Yb-176 targets, a neighbouring lanthanide element, under high neutron flux. After neutron capture to the short-lived intermediate Yb-177, the latter beta decays to Lu-177. In a complex radiochemical process, Lu-177 is then separated from the remaining target material to eventually provide a solution of pure Lu-177 of radiopharmaceutical quality, so-called EndolucinBeta© produced by ITM Radiopharma in Garching, a longstanding partner of the ILL. More recently, other radiopharma companies have also been moving towards performing radiochemical separation of Lu-177.

While Lu-177 is already being used in approved drugs, vigorous research and development continues at different levels to develop new radiopharmaceuticals against other types of cancer and/or including novel radionuclides that provide more favourable decay characteristics for specific applications. For example, terbium 161, another radiolanthanide with similar chemical properties to lutetium but prolific emission of short-range low-energy electrons, is produced by irradiating enriched Gd-160 targets. The subsequent radiochemical separation is performed at the Paul Scherrer Institut (Villigen, CH). A clinical trial using the radiopharmaceutical Tb-161-DOTATOC against neuroendocrine tumours is currently ongoing at Basel University Hospital.

Preclinical and clinical research is being supported by the EU project PRISMAP in the framework of which the ILL, together with many European partners (CERN, PSI, ARRONAX, SCK-CEN, DTU, NCBJ, etc.), supply research users with a large variety of novel radionuclides that are not available commercially.

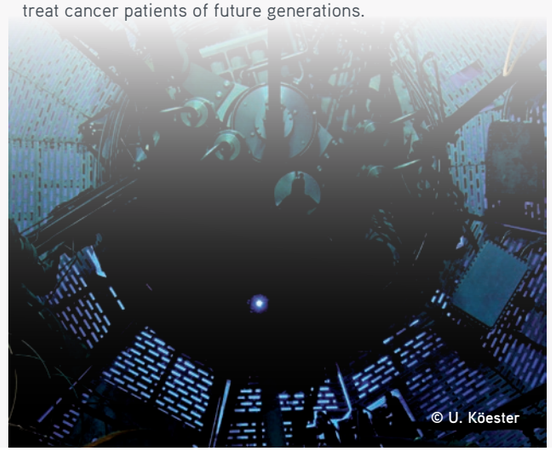
SECURE, another EU project with ILL involvement, focuses on developments to streamline the production and supply of beta- and alpha-emitting radionuclides, e.g. W-188, Au-199, etc.

The approval of Pluvicto© and the start of its large-scale marketing in 2023 is a true game changer for the entire radiopharmaceutical supply chain: suddenly more enriched target material, more irradiation capacity, more radiochemical separation capacity and more radiopharmaceutical labelling capacity are needed, along with the corresponding logistics network, to bring the short-lived Lu-177 (6.65 days half-life) quickly from one player to the next.

The revolution of the radiopharmaceutical market triggered by the approval of Pluvicto© places more emphasis on a capacity enhancement, i.e. the possibility of producing more Lu-177 more often without neglecting the capability to continue to produce radionuclides for research. The ILL is therefore implementing a project with a conventional loading-/unloading procedure and will double its irradiation capacity by adding a second dedicated beam tube to the existing beam tube.

Before being sent to radiochemical laboratories for separation and purification of the radioisotopes, the irradiated samples have to be removed from the irradiation shuttles and placed in shielded casks. This so-called "decanning" process is currently carried out in the ILL's large general purpose hot cell and is only possible thanks to the tremendous commitment of our colleagues from the Reactor Block and Health Physics groups. To enhance the availability and improve the efficiency and ergonomics of the process, a new compact hot cell dedicated to radionuclide shipments will be constructed.

Thanks to the incredible farsightedness of the engineers who designed the ILL reactor two generations ago and the commitment of the Institute's scientific, technical and administrative staff, the ILL's high-flux reactor will be able to produce radioisotopes to treat cancer patients of future generations.



* Often the term radioisotope is used synonymously with radionuclide, but when no chemical element is specified the more general term radionuclide is more appropriate

ENDURANCE

Endurance: Approaching completion

C. Dewhurst & A. Meyer

The Endurance programme (2016 – 2023) has seen the upgrade, modernisation or complete renewal of more than 30 instruments and infrastructure projects, many of which have now been delivered and are available to users. During the H1-H2 long shutdown from October 2021 to February 2023 we rolled out the build of our modernised infrastructure and instrument suite in parallel with major maintenance, security and safety work on the reactor. The new **H24** guide and instrument suite was completed and commissioned with the reactor restart on 27th February 2023. The **H15** guide and associated instrumentation will be completed and enter the user programme in 2024. The Millennium and Endurance programmes over the last 20 years have ensured ILL has a fully modernised suite of world-class instruments ready to operate for the decade to come.

60 M€
ENDURANCE

8 YEARS (2016-2023)

more than 30 instrument and infrastructure projects

H24 GUIDE & INSTRUMENT SUITE COMMISSIONED IN 2023

D10+
IN13
XtremeD
CT2

ENDURANCE PHASE I: (2016 – 2018)

The first phase of Endurance saw the delivery of the new fission-fragment gamma ray spectrometer FIPPS, the upgraded IN5 cold time-of-flight (TOF) spectrometer, the upgraded IN20 thermal triple-axis spectrometer (TAS), the new thermal TOF spectrometer PANTHER and an upgraded D3 hot-neutron diffractometer. The SuperSUN source of ultra-cold neutrons has begun commissioning with the aim of producing the highest densities of UCNs and allowing science to begin on the PanEDM experiment. The BASTILLE and NESSE infrastructure projects have continuously delivered software for data treatment and analysis and new sample environment capabilities throughout Endurance.

H24 guide and instruments D10+, IN13, XtremeD, CT2, FIPPS

The new H24 guide brings dedicated thermal neutron guides to upgraded D10+, IN13 (CRG) and the new extreme conditions powder and single-crystal diffractometer XtremeD (CRG) as well as providing neutrons to the renewed test instrument CT2 and a repositioned FIPPS. Each instrument benefits from a dedicated end-of-guide position, allowing optimised beam-shaping and monochromator optics. The new guide has a high critical angle coating (m=3) and exploits the two radii of curvature of the H241 (R=14000m) and H242 (R=8000m) down-stream sub-branches to naturally curve and expand the guide over a distance of 22 m. We have measured a total gain in count rate of 11 times on the new D10+ single crystal diffractometer (Cu monochromator at 1.26 Å) with respect to the old D10 instrument thanks to the efficiency of the new detector (x1.6) and neutron flux (x6.6) and due to the increased divergence, guide and monochromator size. Similar gains in intensity have been measured on IN13, primarily due to the performance of the new H24 guide coupled with a new temperature gradient monochromator, which is in the process of being optimised. New capabilities and capacity are available with the CRG powder and single crystal diffractometer XtremeD, which has a neutron intensity to rival that of D20c.

ENDURANCE PHASE II: (2019 – 2023)

In a first round of funding for Endurance 2, we were able to rapidly construct detectors for the SANS instruments D22 and D11. A second protein crystallography station, DALI, was installed while the secondary spectrometer of the D16 cold-neutron diffractometer has also been upgraded, including a new wide-angle detector. The neutron imaging facility, NeXT, has now seen a full re-build of the instrument backbone allowing state-of-the-art imaging and a second (monochromatic) imaging station, MOTO, to be operational in 2024.

H15 guide and instruments T3, D(00)7, D11, SAM, SHARP+

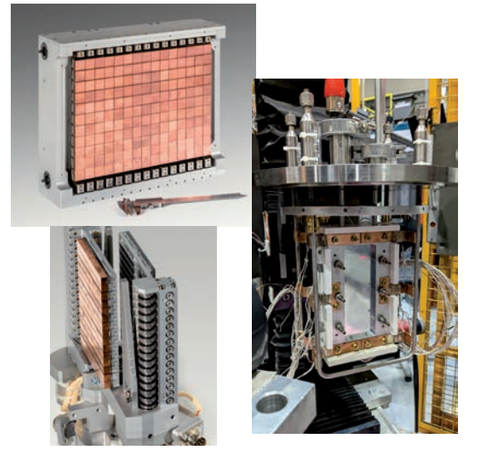
The H15 guide brings dedicated cold neutron guides to upgraded D(00)7, D11, and to the new SANS instrument SAM and TOF spectrometer SHARP+ as well as providing neutrons to the renewed test instrument T3.

The H15 guide has a rather complex opposing-curved expanding section, referred to as 'the trumpet'. The trumpet serves two important purposes: The first is to spatially expand the neutron guide, thus converting the upstream high divergence to a lower divergence but extended over a greater area. This allows the guide to be split into multiple individual guide branches, allowing five dedicated end-of-guide beam positions for instrumentation. The second is that the opposing curve leaves a 'fingerprint' correlation between the divergence profile and spatial position at the end of the trumpet. In other words, neutrons on the left of the guide have a divergence distribution on average pointing to the left and conversely for the right side of the guide. Importantly, this allows the guide branches to be more widely separated in angle, therefore allowing space for substantially more instrumentation downstream. Civil engineering works for H15 were completed during the H1-H2 shutdown, allowing the cold-neutron instruments on the Vercors side of ILL7, excluding the H15 instruments, to restart with the ILL reactor. The H15 works were scheduled intentionally so as to allow continued construction of the out-of-casemate H15 guide and instruments during reactor operation in 2023. The winter shutdown 2023-2024 has allowed the completion of the new H15 guides, finally connecting the new guide to its instrumentation suite ready for commissioning and user operation during the second half of 2024. We expect significant gains in performance from instruments such as D(00)7 and SHARP+ (CRG) compared to their predecessors D7 and IN6 due to the new high-performance H15 guide, dedicated focussing guide and beam optics and improved or larger detectors. In the case of D(00)7, the expected order of magnitude increase in flux will make polarisation and spectroscopic analysis measurements realistic as well as massive gains for the more usual diffuse scattering measurements. On the cold TOF spectrometer SHARP+ we expect more than ten times the count rate of its predecessor IN6. D11 is in the process of being rebuilt in a relocated position with an optically cleaner collimation and should regain its theoretical brilliance to match that of D22 and D33, as should that of the new CRG SANS instrument SAM, which includes a MIEZE option. The test instrument T3, essential for the testing of polarised supermirrors produced in-house, is being rebuilt and upgraded and completes the H15 instrument suite, offering increased flux and beam polarisation.

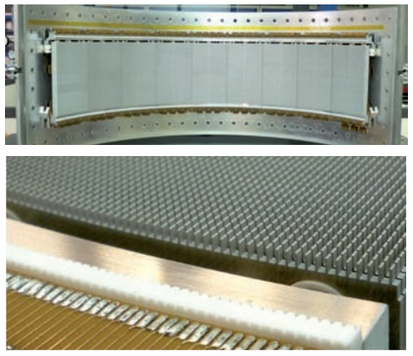
In-house technologies

In-house developed technologies have been key to successful and efficient instrument upgrades. Both single- and double-focussing monochromators have been produced for various instruments, some of which are shown in the figures. The detector technology used for D16 and XtremeD is an excellent example of the high-quality research, development and manufacturing carried out in-house by ILL's services.

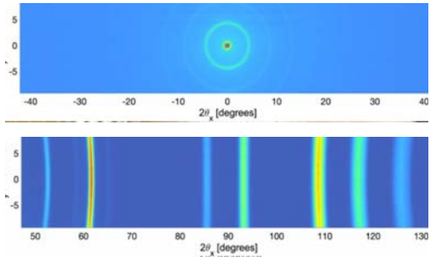
The so-called Trench Multi-Wire Proportional Counter (TMWPC)



Top left: Double-focussing copper monochromator for PANTHER. **Bottom left:** Single-focussing back-to-back copper and graphite (HOPG) monochromators for D10+. **Right:** Final alignment of the new temperature gradient monochromator for IN13 on T13c with the first neutrons of 2023.



ILL-designed and manufactured Trench Multi-Wire Proportional Counter (TMWPC) detector technology for XtremeD, D16 and D20.



D16's new 'banana' detector has a radius of 1.15 m and 1152 x 192 pixels of resolution 1.5 mm and 2 mm [x,y]. **Top**: Small-angle scattering from silver behenate on D16. **Bottom**: Wide-angle diffraction from Yttrium-Iron-Garnet powder on D16.

is a novel concept in detector technology, allowing high count rate and spatial resolutions down to 1.5 mm using ^{3}He detection gas. This same technology is being used to provide a new high-performance detector for D20c.

Completed Endurance projects

We have already delivered many new or upgraded instruments and infrastructure packages that are already in user operation. These are:

• **FIPPS**: Fission-fragment gamma ray spectrometer for identification of prompt short-lived fission products and entirely complimentary to the existing Lohengrin mass spectrometer. (2016)

• **RAINBOWS**: A re-scoped project to upgrade the D17 reflectometer with a new focussing guide and chopper system to pursue an alternative high-flux mode of operation using the so-called 'coherent summing' method. (2018)

• **IN5 / H16**: Cold-neutron TOF spectrometer boasting huge gains in intensity, in particular at shorter wavelengths while focussing onto much smaller samples. (2019)

• **NESSE 1 & 2**: Stimulus in sample environment capabilities covering all scientific domains such as low- and high-temperatures, high pressure, magnetic fields, humidity environments, and equipment for soft-matter and biological sciences. (2019, 2023)

• **BASTILLE 1 & 2**: Stimulus in scientific computing - Modern data reduction and analysis software tools via the Mantid project. Mantid is now well developed and fully deployed over our suite of TOF spectrometers and is starting to be adopted on our powder diffraction, reflectometry and SANS instruments (solution scattering). (2019, 2023)

• **PANTHER**: New thermal-neutron TOF spectrometer replacing IN4 and with performance (signal / noise) approximately 60 times higher than that of its predecessor. (2020)

• **IN20**: Upgraded thermal triple-axis spectrometer. A velocity selector for wavelength filtering and allowing much greater flexibility in instrument use and accessible energies. (2020) New graphite monochromator and analyser and a new Heussler monochromator. (2023)

• **Orient Express**: Strategic move of instrument to H23 to allow uninterrupted access for sample alignment during the H24 works and to free up an end-of-guide position for the future relocation of FIPPS. (2020)

• **DALI**: Second protein crystallography instrument with an increased neutron flux and flexibility due to the use of a velocity selector. (2020)

• **D3 Liquids**: The hot-neutron diffractometer has been upgraded with a new position-sensitive area detector and polarisation components, allowing efficient measurement of hydrogen-containing liquid samples with precise discrimination of incoherent scattering. (2021)

• **D11 Detector**: Replacement of the aging multidetector with a modern, increased area, efficiency and count-rate detector. (2021)

• **D22 Detector**: An additional high-angle detector, massively extending the instrument's dynamic q-range and allowing more rapid measurements with a reduced number of instrument configurations. (2021)

• **D16**: New secondary spectrometer, including a new wide-angle detector bank with approximately four times the angular coverage (85 degrees) of the previous detector. (2023)

• **NeXT**: A full re-build of the neutron imaging instrument, allowing state-of-the-art imaging with advanced contrast techniques, high spatial resolution, intense neutron flux and combined X-ray imaging. A second (monochromatic) imaging station, MOTO, for measurements such as Bragg-edge and dark-field imaging as well as technique development is under installation. (2023)

• **H24 guide**: New thermal-neutron guide providing dedicated end-of-guide positions to D10$^+$, IN13, XtremeD (CRG), CT2 and FIPPS. (2023)

• **D10$^+$**: Renewed thermal single-crystal diffractometer with count rates more than ten times that of the previous D10 instrument due to the new H24 guide, new and larger focussing monochromators and improved efficiency detector. (2023)

• **IN13**: New primary spectrometer, including new temperature gradient monochromator, optimised for the dedicated end-of-guide position on the new H24 guide. (2023)

• **XtremeD**: New CRG thermal neutron powder and single crystal diffractometer with performance to rival that of D20. (2023)

• **CT2**: Renewed test instrument for the characterisation of neutron detectors. (2023)

• **SuperSUN**: The new ultra-cold neutron (UCN) source is in the commissioning phase, has begun to produce high densities of UCNs and will allow science to begin on the PanEDM experiment. (2023)

Ongoing Endurance projects

A number of Endurance projects remain in execution such as the H15 guide and associated instrument suite. We have also been able to launch several new instrument upgrade projects in the last wave of independent Endurance projects. The ongoing Endurance projects are:

• **H15 guide**: New cold-neutron guide providing dedicated end-of-guide positions to T3, D(00)7, D11, SAM (CRG) and SHARP$^+$ (CRG). (2024)

• **T3**: Renewed reflectometry test instrument for the characterisation of supermirrors. (2024)

• **D(00)7**: Upgraded diffuse scattering diffractometer and spectrometer. The expected increased flux will make polarisation and analysis spectroscopic studies realistic. (2024)

• **D11**: SANS instrument relocated onto the new H15 guide and upgraded with reliable and optically clean beam collimation. (2024)

• **SAM**: New SANS CRG instrument with MIEZE option. (2024)

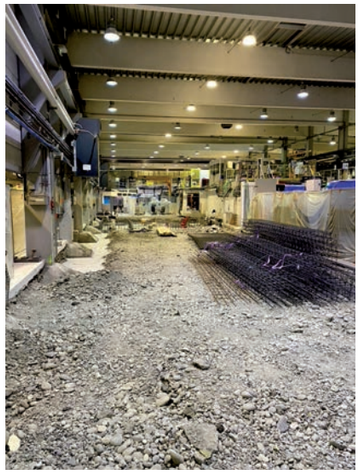
• **SHARP$^+$**: Relocation of the SHARP instrument (previously IN6) onto the new H15 guide and upgrade of the primary spectrometer with focussing guide, monochromator and chopper cascade. (2024)

• **D20c Detector**: Replacement of the existing aging microstrip detector with a new banana detector based on TMWPC technology. (2024)

• **MARMOT**: Multiplexed energy and angle analysis on the cold-neutron TAS instrument ThALES. Detector elements and bent silicon analyser crystals for the spectrometer will use in-house technologies and fabrication. (2024 / 2025)

• **WASP**: The wide-angle spin-echo instrument will receive its full complement of detectors, with completion of the first additional detector bank due in 2024. (2024 / 2025)

Maximum destruction February 2022



ILL7 guide hall - Chartreuse

ILL7 guide hall - Vercors

ILL22 guide hall - NeXT

H24, H15 and ILL22 instruments rebuilt by February 2023

ILL7 – H24: D10+ & XtremeD

ILL7 – H15: D11 & SAM

ILL22: NeXT & D16

List of instruments

The instrument facilities at the ILL are listed in the table and shown in the layout drawing on p. 32.
In addition to the ILL instruments, there are 8 Collaborating Research Group (CRG) instruments.
CRGs have the possibility of building and operating instruments at the ILL for the purpose of carrying out their own research programmes. They provide additional capacity (typically 50% of their beam time) and, in many cases, unique capability for the ILL instrument suite.

Details about the framework for the operation of CRGs can be found at:
https://www.ill.eu/users/instruments/crgs

ILL INSTRUMENTS		DAYS REQUESTED	DAYS ALLOCATED	NUMBER OF ACCEPTED EXPERIMENTS
D2B *	powder diffractometer	191	107	52
D3 *	single crystal diffractometer	104	93	14
D4 (50% with IN1-LAGRANGE) *	liquid diffractometer	143	57	20
D007 ***	diffuse-scattering spectrometer			
D9 *	single crystal diffractometer	128	69	10
D10+ *	single crystal diffractometer	137	79	12
D11 ***	small-angle scattering diffractometer			
D16 *	small momentum-transfer diffractometer	120	77	17
D17 *	vertical reflectometer	225	118	33
D19 *	single crystal diffractometer	149	88	12
D20 *	powder diffractometer	244	117	54
D22 *	small-angle scattering diffractometer	188	106	68
D33 *	small-angle scattering diffractometer	228	115	51
DALI *	quasi-laue diffractometer for biological macromolecules	57	31	8
FIGARO *	horizontal reflectometer	158	110	41
FIPPS *	fission product prompt gamma-ray spectrometer	435	97	9
IN5 *	time-of-flight spectrometer	332	135	38
IN8 *	three-axis spectrometer	145	113	19
IN15 *	spin-echo spectrometer	246	103	26
IN16B *	backscattering spectrometer	315	120	37
IN20 *	three-axis spectrometer	136	63	10
LADI *	Laue diffractometer	226	110	7
Lagrange (50% WITH D4) *	neutron vibrational spectrometer	142	49	13
PANTHER *	time-of-flight spectrometer	162	71	26
PF1B *	neutron beam for fundamental physics	294	147	11
PF2 *	ultracold neutron source for fundamental physics	160	159	13
PN1 *	fission product mass-spectrometer	247	208	20
SALSA *	strain analyser for engineering application	153	98	20
SuperSUN **	ultracold neutron source for fundamental physics			
ThALES *	three-axis spectrometer	239	131	22
WASP *	wide-angle spin-echo spectrometer	193	67	18

CRG INSTRUMENTS		DAYS REQUESTED	DAYS ALLOCATED	NUMBER OF ACCEPTED EXPERIMENTS
D1B - CRG-A *	powder diffractometer	127	72	29
D23 - CRG-B *	single crystal diffractometer	106	48	9
IN12 - CRG-B *	three-axis spectrometer	143	37	7
IN13+ - CRG-A **	backscattering spectrometer	63	33	5
IN22 - CRG-B *	three-axis spectrometer	100	33	6
SAM - CRG-B ***	small-angle scattering diffractometer			
SHARP+ - CRG-A ***	time-of-flight spectrometer			
SuperADAM - CRG-B *	reflectometer	86	48	13
S18 - CRG-B *	interferometer	125	83	6
XtremeD - CRG-B *	powder diffractometer			
JOINTLY FUNDED INSTRUMENTS				
NeXT 75% *	imaging instrument operated with Ni-Matters composed of HZB, UGA and ILL	312	83	33

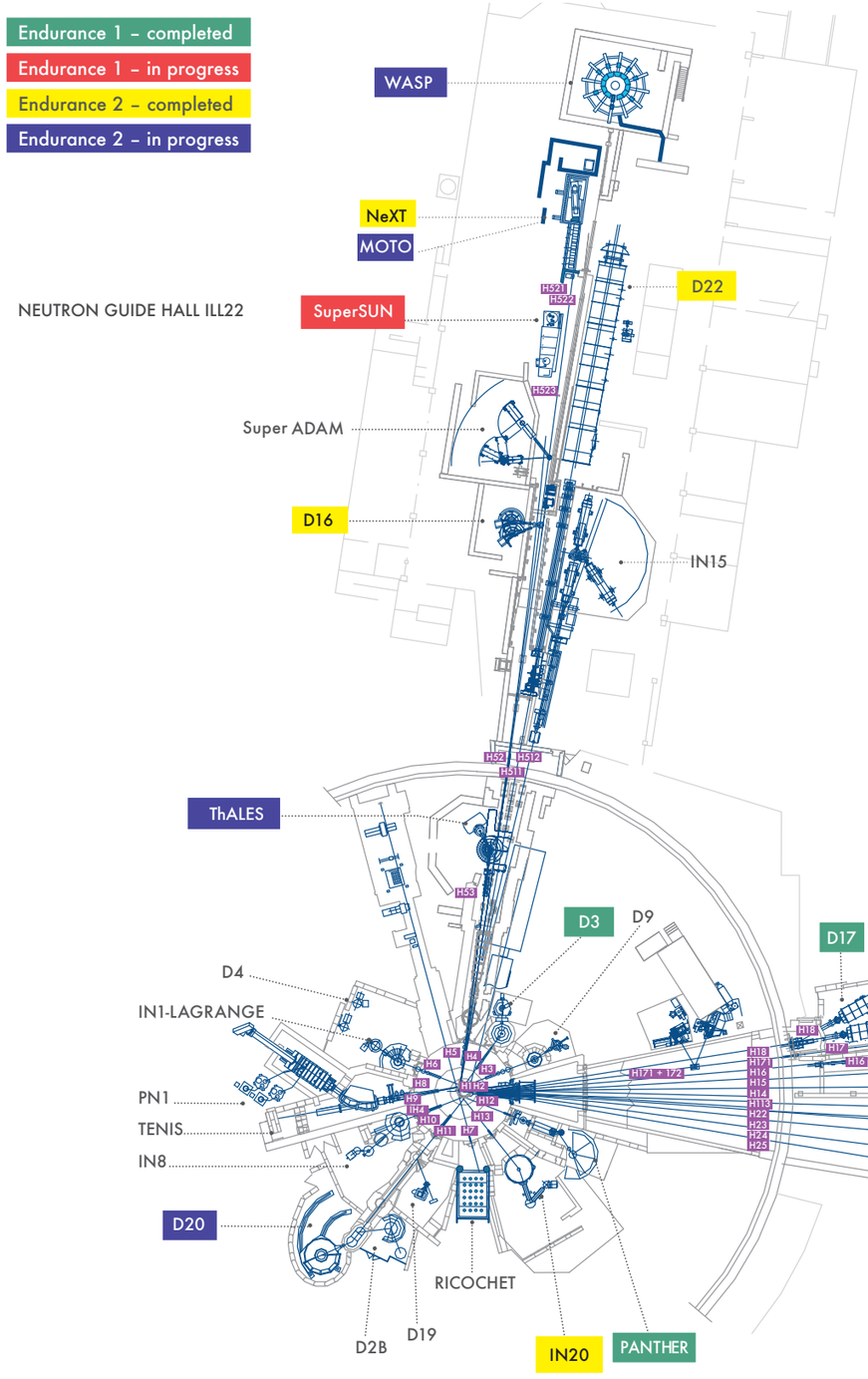
TEST AND CHARACTERISATION BEAMS	
CT1, CT2	detector test facilities
OrientExpress	Laue diffractometer
TENIS	neutron irradiation position
T3 (under construction)	neutron optics test facility
T13A, T13C	monochromator test facility

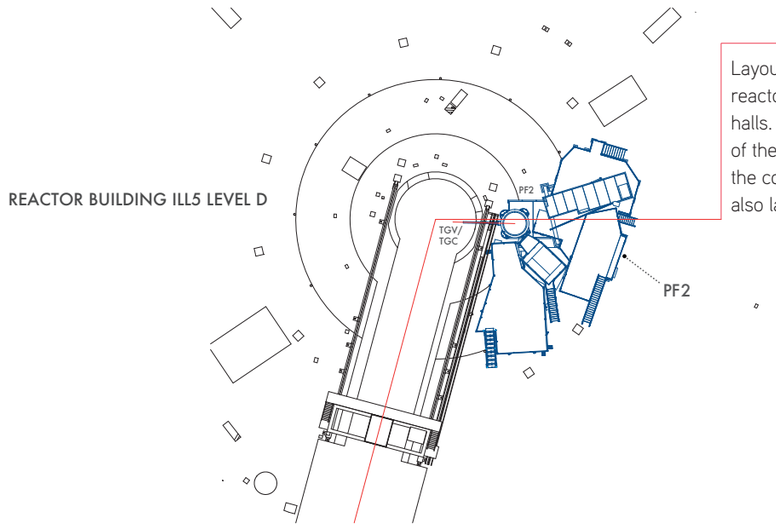
The number of proposals submitted/accepted corresponds to the figures from the November and April 2023 scientific council subcommittee meetings, with a total of 789 accepted experiments scheduled in 2023. A total of 1062 ILL and CRG experiments were performed (including 38 DDT - Director Discretion Time - 147 EASY and 22 internal research experiments). Beam days available for science (used for users, EASY, DDT and internal research) amounted to 4 262.

Status of instruments
* operational
** commissioned
*** under construction

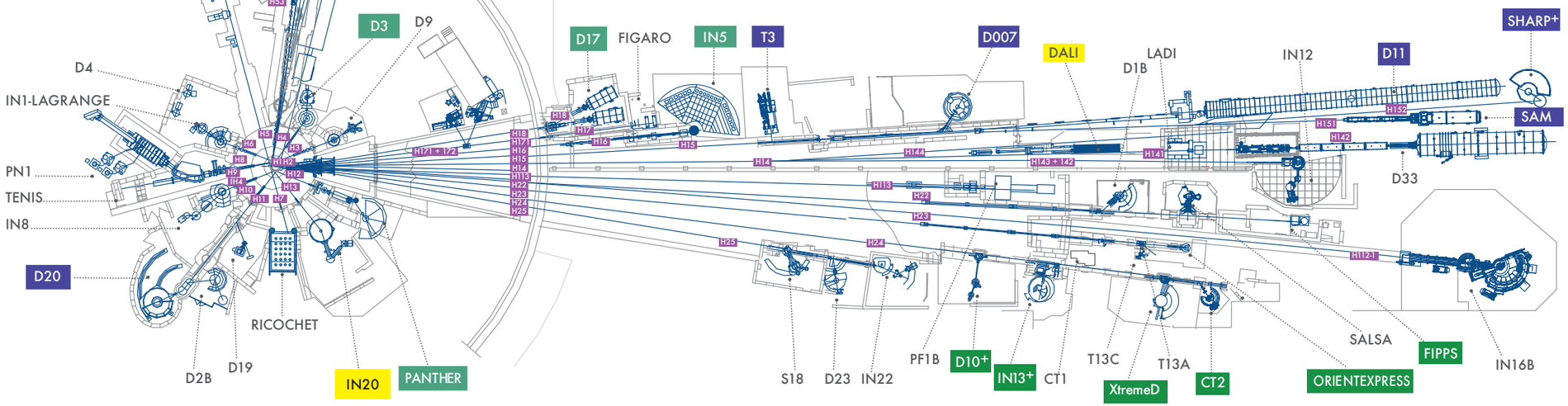
Note: D3, D9 and IN8 will operate for 50% of the time from 2024
D19 is no longer available for users from 2024
PF2 consists of different set-ups where several experiments are running simultaneously. The values given are averages for these positions (and normalised by a factor of 5).

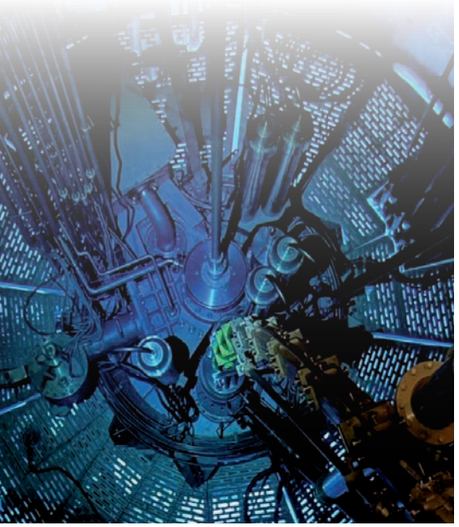
Details of the instruments can be found at: https://www.ill.eu/users/instruments/instruments-list





Layout of the ILL instrument suite covering the reactor building ILL 5 and the ILL 7 and ILL 22 guide halls. Instruments completed or in-progress as part of the Endurance programme are highlighted as per the colour code given, and the neutron guides are also labelled.





REACTOR

THREE
CYCLES AND
165
DAYS OF
OPERATION
IN **2023**

~58.3 MW

1.5 x 10^{15} n/s cm^2

A SINGLE
HIGHLY
ENRICHED
URANIUM
FUEL ELEMENT

Following the successful completion of an intense 16-month programme of upgrade and maintenance work within the framework of the H1-H2 shutdown, the reactor was restarted at the end of February 2023. However, on restart, fluctuations in power were experienced due to the overheating of the V4 beam tube, which had been modified during the shutdown to install additional fission chambers closer to the core to offset the decrease in the flux from the start-up source due to the long shutdown. The reactor therefore had to be shut down again so that the V4 water cooling system could be reinstalled. It was then restarted on 29 March, and by the end of 2023 three operating cycles had been successfully delivered.

Problems with the supply of materials and, above all, delays with completion by suppliers of the detailed design studies for the work to be carried out during the next long shutdown have led the ILL to review its operating schedule for 2023 and 2024. 2023 and the first half of 2024 are to be devoted to the user programme, and the next long shutdown for refurbishment of the polar crane and installation of fire sprinklers will start in the second half of 2024.

Overall, the Reactor Division delivered a total of 165 operating days in 2023, out of the 172 initially planned, at different power levels to optimise the fuel cycle and the user programme.

Over the past few years, the ILL has been producing radioisotopes, in particular lutetium-177, which is highly effective in treating certain forms of cancer. Production of these radioisotopes was stepped up in 2023.

As has been the case for the last few years, the ILL's relations with the French nuclear safety authority (ASN) continue to be good, as shown by the confidence expressed by the ASN at the annual review in January 2023. Before the start of the first cycle of 2023, the ASN inspected our requalification tests for the reactor restart, drawing rather positive conclusions. In fact, the feedback received from all the ASN inspections in 2023 was encouraging. Finally, a national exercise was held in November 2023 to test the ILL's emergency response organisation. The feedback we received was again positive.

Concerning the RPP project to reinforce the physical protection of the installations, which is being conducted under the supervision of France's security authority, the HFDS (*Haut Fonctionnaire de Défense et de Sécurité*), a new strategy has been defined by the ILL which includes the deployment of armed guards. Starting in April 2024, they will be stationed within the ZAC to reinforce the security of both the ILL installations and everyone on the site.

The main activities carried out by the Reactor Division during the inter-cycle shutdowns in 2023 were:

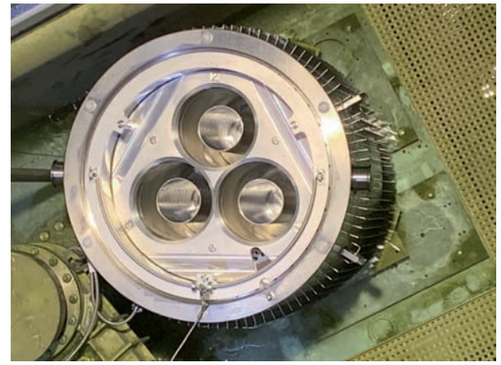
- Maintenance work and periodic testing
- Testing of the anchorage points installed for removing the trolley of the polar crane on Level D
- Replacement of the pump on the CEN groundwater circuit
- Shipment of irradiated fuel using a TN-MTR cask
- Refurbishment of the plugs on beam tubes H10 and H12
- Replacement of the safety valve of the hot neutron source

Moreover, as part of preparations for the upcoming long reactor shutdowns in 2024 and 2025, the Reactor Division:

- continued work on the clean-up of the detritiation facility by removing certain components/equipment in order to prepare the facility for the installation of a recombiner unit. The purpose of this recombiner unit will be to recombine the tritium and deuterium with oxygen at very low concentration, in order to produce heavy water for the primary circuit.
- prepared the replacement of the trolley and the reinforcement of the polar crane on Level D (a request for authorisation was sent to the ASN and the 1t lifting system was dismantled).
- continued work on preparing for the installation of a fire sprinkler system (completion of fire column in the annular space, start of work on installing additional pipes to supply the sprinklers from a water tank which will be located outside the reactor building,...).
- prepared the reinforcement of pipe support structures located at the bottom of transfer canal n°2.
- prepared the reinforcement of the casemates H1H2 and H5 to protect the reactor building penetrations.

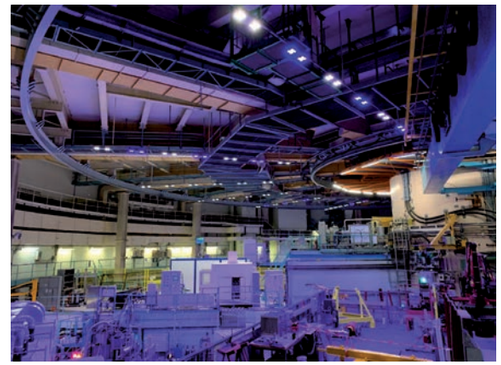
Concerning the fuel cycle, the ILL continues to participate in the HERACLES fuel conversion programme with the aim of using LEU in the reactor within the next ten years. Work on presenting a feasibility study review at the beginning of 2024 is ongoing and will be followed by the launch of the detailed design phase, with the first irradiation of an LEU fuel assembly planned for 2029 before full-scale conversion. This scenario is dependent on receiving a positive decision from the Associates concerning the future of the ILL.

Jérôme Estrade
Head of the Reactor Division



TN-MTR cask to evacuate the irradiated fuel.

Replacement of trolley and reinforcement of polar crane on Level D.

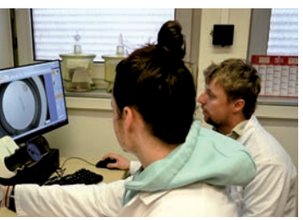


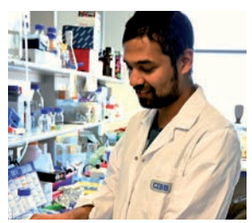
Installation of a fire sprinkler system on Level C.

Hot cell for radioisotope production.

LABORATORIES

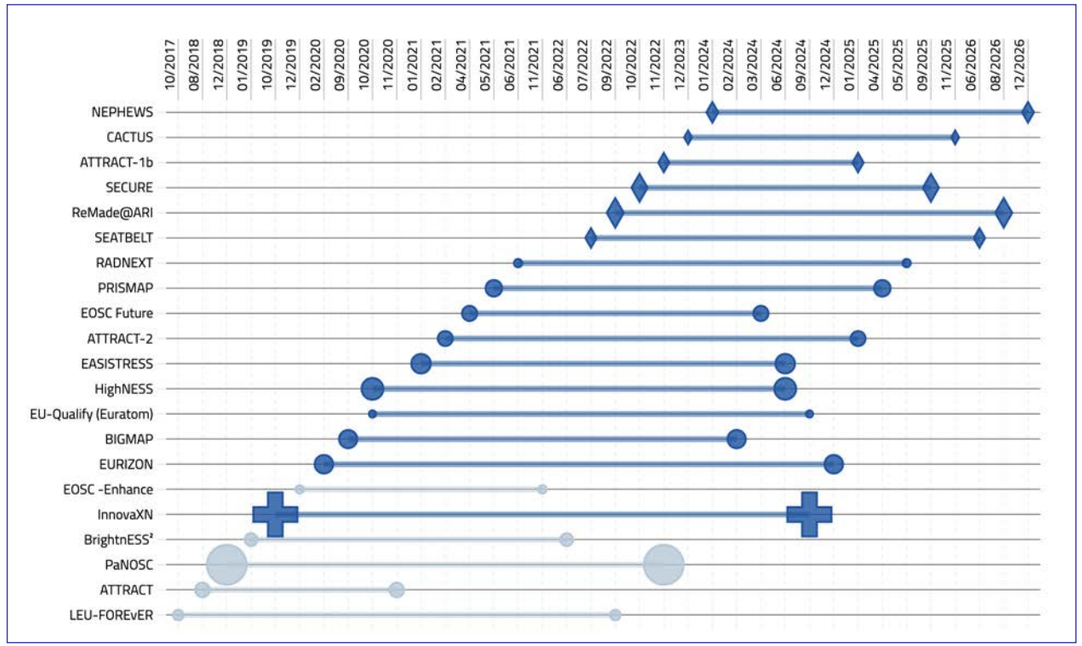
In order to maintain their ranking at international level, European research infrastructures must optimise their resources and develop synergies at every level. The ILL is firmly committed not only to building high-performance instruments, but also to offering the best possible scientific environment for its user community. On-site sample preparation laboratories provide space and equipment to prepare and characterise samples before, during and after neutron experiments. They are available to the user community and empower in-house research. Over the years, we have established close collaborations with neighbouring institutes and launched a number of successful scientific support partnerships, such as the Partnerships for Soft Condensed Matter (PSCM) since 2012, and for Structural Biology (PSB) since 2002. Within these partnerships, dedicated platforms and laboratories (D-Lab and L-Lab) provide the ILL user community with a variety of deuterated biological molecules, including proteins and lipids. These activities are now grouped together in the newly founded BDCS (Biology, Deuteration, Chemistry and Soft Matter) group, headed by Frank Gabel since 1st December 2023.



Photos: V.G

EU project activity at the ILL



Externally-funded projects, and in particular, those funded by the European Commission through the Horizon Europe programme, are an excellent opportunity to develop new areas of science and technology with partners in Europe and enhance training. Such projects reinforce the core business of ILL but also create synergies with and lead to common solutions among facilities, enhancing the provision of new services for nomadic neutron scatterers in Europe.

ILL is involved in an increasingly wide range of EU projects - currently 17 (with annual revenue of more than 1 M€) – that, in view of their importance, are reviewed twice a year by ILL's directors. The current portfolio of projects covers, for example, the circular economy (ReMade@ARI), batteries for clean energy (SEATBELT and BIG-MAP), radio-isotopes for health (PRISMAP and SECURE) and the development and integration of ILL data and software services in the European Open Science Cloud (EOSC). One major project close to finishing is InnovaXN, the Marie-Curie (MSCA) funded PhD programme, hosted by ILL and ESRF, in which 40 research projects will have been completed with industry partners. Two new projects are now starting: NEPHEWS to provide trans-national access to neutron and X-ray facilities and reach out to new communities and CACTUS to improve solar photovoltaic performance for extreme climate conditions.

The portfolio of projects must be continually renewed by identifying the best opportunities in the EC work programmes, with two more to be published in Horizon Europe for 2025 and then 2026 – 2027. The next Framework programme (FP10) is being prepared and will start in 2027. In the current work programme (2023 – 2024) about a dozen projects were submitted in autumn 2023 and spring 2024, covering subjects like artificial intelligence, high magnetic fields, neutron optics and detectors and hydrogen research for clean energy. We look forward to seeing the outcome of these proposals, bearing in mind that success rates are about 30%.

All EU-funded projects address EU priority areas, which are often close to major societal challenges. In many cases they also require collaboration with industry in order to bring fundamental and applied research closer together with innovation and new products and processes. These directions are fully aligned with the ILL vision for research with neutrons and the science strategy that will be published in 2024. In this context, ILL would like to enhance its portfolio of science-driven, EU-funded projects.

For more information about EU projects at ILL, including future participation, please contact europe@ill.eu.

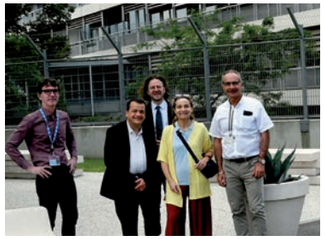
EVENTS & WORKSHOPS

30 **SCIENTIFIC EVENTS** AND **VIP** VISITS

50 GENERAL SEMINARS


Visit of A. Petit & CNRS delegation.
27 October ©VG


Visit of the French Minister of Research
S. Retailleau. 22 May ©VG


Visit of K. Amram, DGRI, & her colleagues.
8 June ©VG


Visit of S. Schmidt & HZDR delegation.
1 June ©SMonfront


UK 50 years @ILL. 29 November ©VG

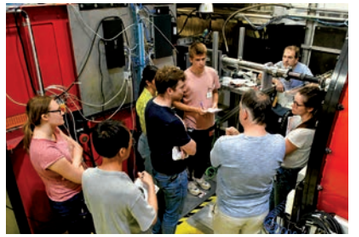

20th Anniversary of the Partnership for Structural Biology (PSB). 31 May


Visit of JAEA, Kyoto University. 6 March ©MJ


PhD clip session. 15 June ©VG


Visit of Deputy Head of Mission, British
Embassy, T. Rycroft. 29 November ©VG


Visit of ITM Radiopharma. 5 September ©S. Monfront


FANs school. 20 June ©S. Monfront


Visit of American delegation of the Fulbright
programme. 10 October ©VG

Symposium in honour of Philippe Nozières. 20 October. ©E. Ryan


Visit of the International Atomic Energy Agency (IAEA)
& members of the CEA. 28 February ©S. Monfront


Visit of the German Ambassador S. Steinlein.
7 November ©VG

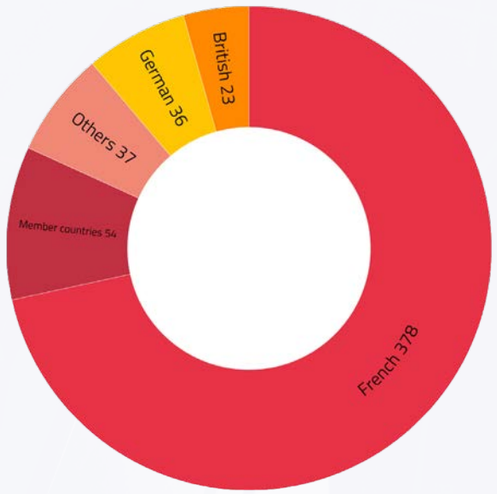

Neutrons for delivery systems (NDS). 11 July ©S. Monfront

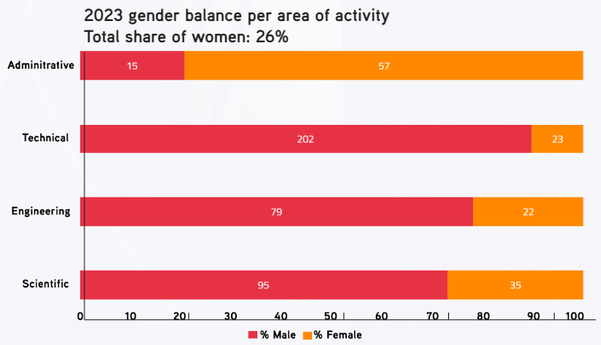
FACTS & FIGURES

Staff on 31 December 2023

The ILL employs 527 staff, including 90 experimentalists in the scientific sector and 37 thesis students.
In total, the workforce comprises 30 different nationalities: 378 French, 36 German, 23 British, 54 Scientific Member countries, and 37 others.

Distribution of staff by nationality - 31 December 2023





Associates

France
Commissariat à l'Energie Atomique et aux Energies Alternatives (CEA)
Centre National de la Recherche Scientifique (CNRS)
Germany
Forschungszentrum Jülich (FZJ)
United Kingdom
United Kingdom Research & Innovation (UKRI)

Countries with Scientific Membership
Spain: MCIN Ministerio de Ciencia e Innovación
Switzerland: Staatssekretariat für Bildung, Forschung und Innovation (SBFI)
Italy: Consiglio Nazionale delle Ricerche (CNR)
Belgium: Belgian Science Policy Office (BELSPO)
Sweden: Swedish Research Council (VR)
Denmark: Danish Agency for Science, Technology and Innovation
Poland: Consortium of Polish Scientific and Research Institutions (NDPN)
Slovenia: The National Institute of Chemistry

CENI (Central European Neutron Initiative) Consortium composed of:

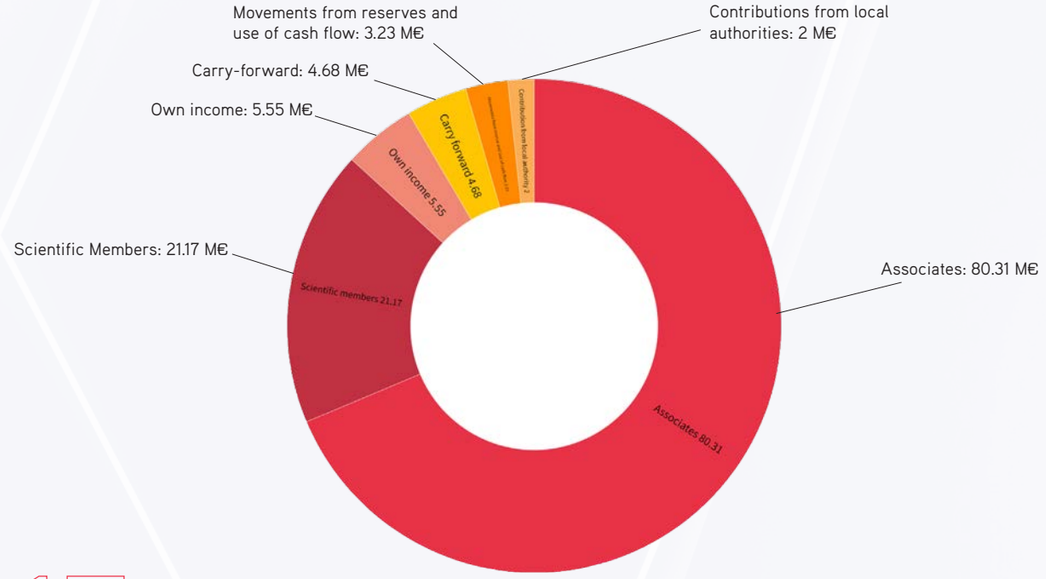
Austria: Österreichische Akademie der Wissenschaften
Czech Republic: Charles University, Prague
Slovakia: Comenius University, Bratislava

Review panels - January 2024

Key **Chair/focus group chair**
ILL college secretary/focus group secretary

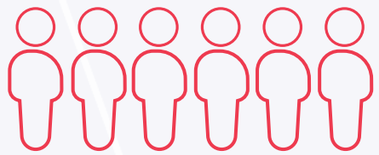
Applied metallurgy, instrumentation and techniques
Monica Ceretti (University of Montpellier, FR)
Arnold Paecklar
Nuclear and particle physics
Guillaume Pignol (LPSC, Grenoble, FR)
Estelle Chanel
Magnetic excitations
Rasmus Toft Petersen (KGS Lyngby, DK)
Lucile Mangin-Thro
Crystallography
Marc Widenmeyer (TU Darmstadt, DE)
Oscar Fabelo
Magnetic structures
A. Gibbs (Rutherford Appleton Laboratory, UK)/ D. Lott (GKSS Forschungszentrum, Germany)
A. Rodriguez Velamázan/N. Steinke
Structure and dynamics of liquids and glasses
D. Morineau (University of Rennes, France)
M. Appel
Spectroscopy in solid state physics and chemistry
F. Juranyi (PSI, Switzerland)
J. Ollivier
Structure and dynamics of biological systems
J.R. Lu (University of Manchester, UK)
O. Matsarskaia
Structure and dynamics of soft condensed matter
E. Dubois (Pierre and Marie Curie University, Paris, France)/R. Campbell (University of Manchester, UK)
O. Czakkel/L. Chiappisi

Operating budget 2023: 116.696 M€ (excluding taxes)
The ILL's Associate countries contributed some 80 M€ to the Institute in 2023, a sum enhanced by significant contributions from the ILL's Scientific Member countries.

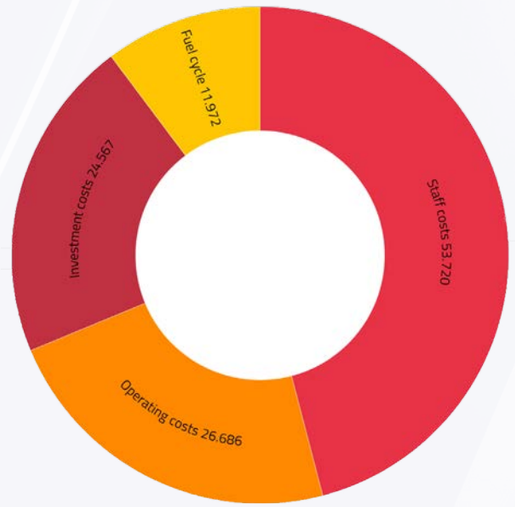


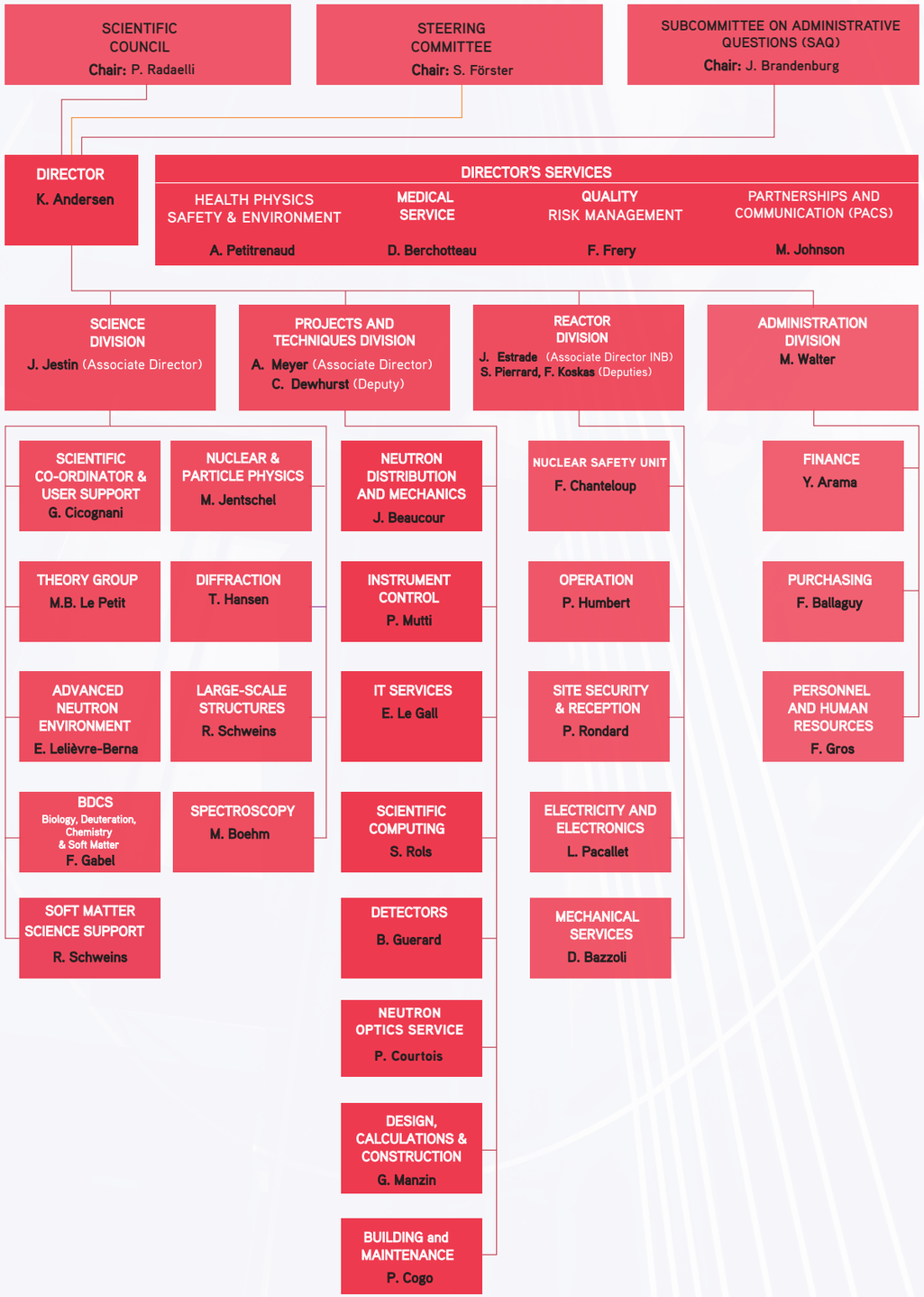
117 M€
Annual income
69% from the Associates
18% from the Scientific Member countries

527 members of staff
of **30** different nationalities



2023 Expenditure: 116.696 M€

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P. Cogo

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EDITORS AND PRODUCTION TEAM
Mark Johnson and Giovanna Cicognani

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