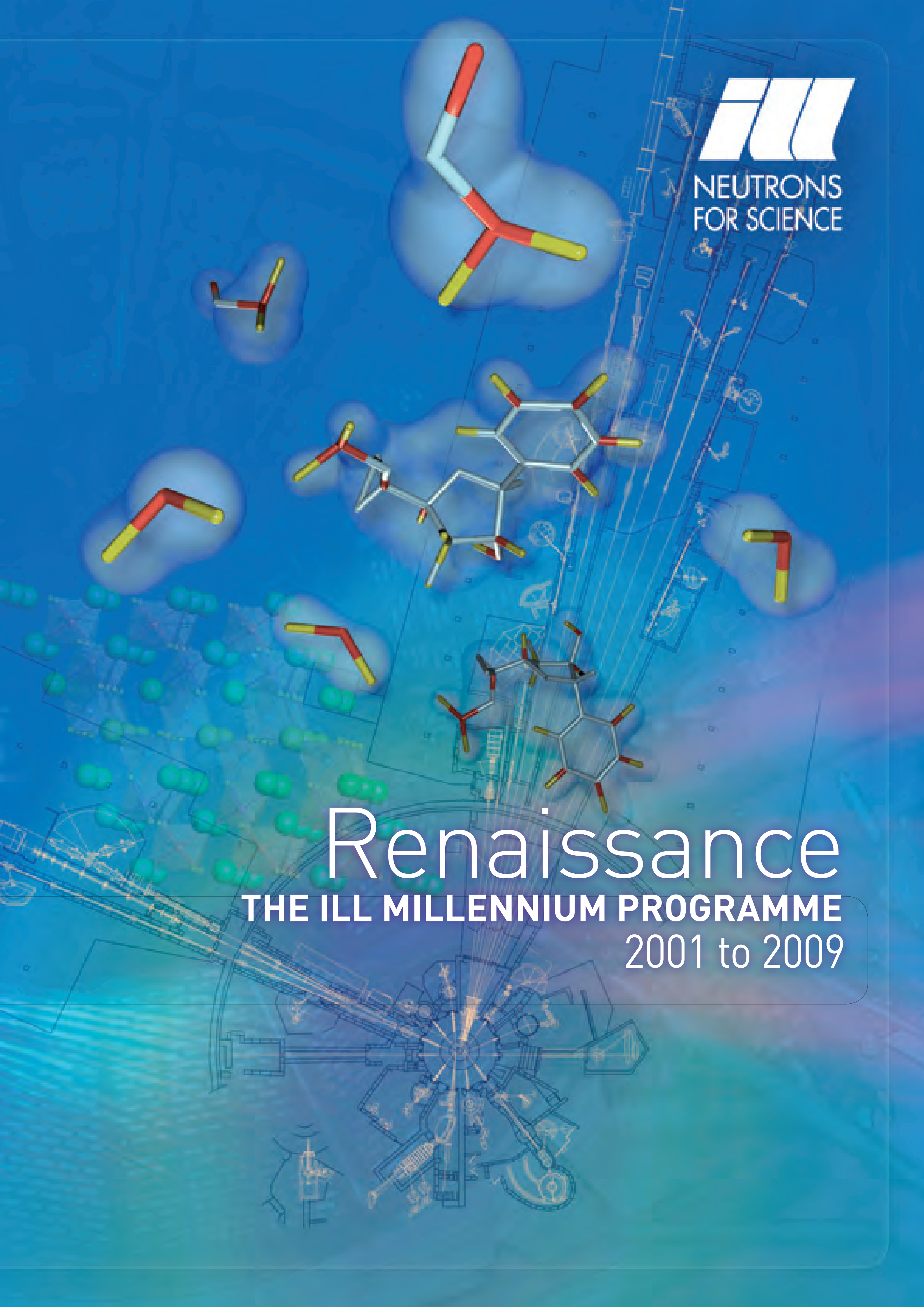


ill
NEUTRONS FOR SCIENCE

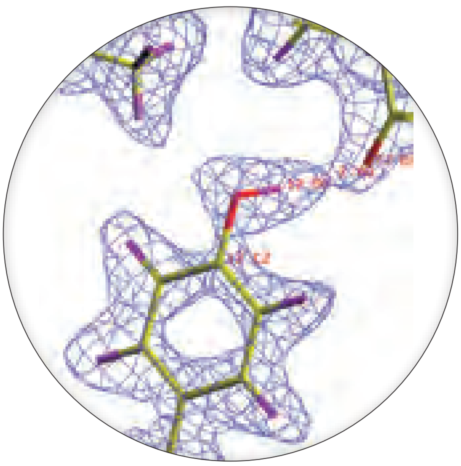
Renaissance

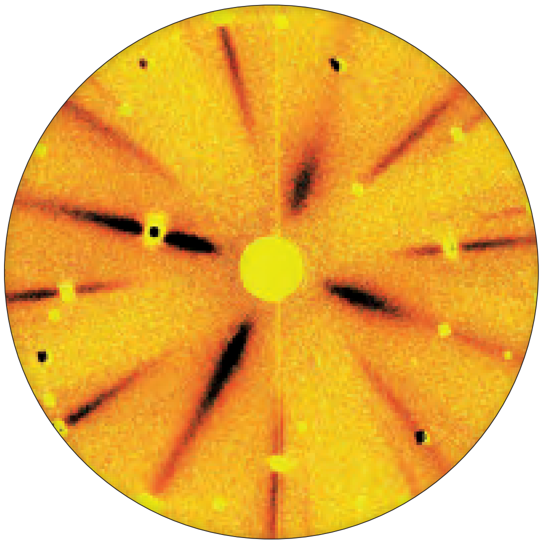
THE ILL MILLENNIUM PROGRAMME

2001 to 2009

CONTENTS

UPGRADED INSTRUMENTS





NEW INSTRUMENTS

NEW INFRASTRUCTURE

FOREWORD

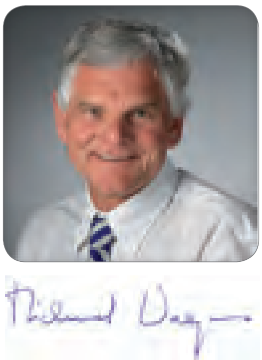
The Institut Laue-Langevin (ILL) first opened its doors to visiting scientists almost 40 years ago, and ever since then, we have led the world in neutron-scattering science and technology. A key factor in this success has been the continuous renewal or development of our instruments to ensure that they are world-leading, and the latest such initiative has been the first phase of the Millennium Programme, officially launched in 2000 and completed in 2008.

Generous funding by our Associates has enabled us to provide our user community with 14 new or refurbished instruments that, together with marked improvements in infrastructure, have hugely increased the average detection rate across the instrument suite.

This achievement is made even more remarkable by the fact that the Millennium Programme ran in parallel over the years 2002 to 2007 with another large and ambitious project – the Refit Programme – whose aim was to reconstruct much of the reactor and surrounding buildings, primarily to conform to more demanding nuclear-safety standards in relation to seismic shock. We also established interface laboratories in partnership with the neighbouring European Synchrotron Radiation Facility (ESRF). These provide complementary facilities that optimise the exploitation of X-rays and neutrons in biology, engineering and soft-condensed matter. Remarkably, despite all this activity, there has been very little disruption to the user programme: over the period 2001-2009, 32 out of a total of 36 cycles were run, based on the traditional 'four cycles a year' objective.

This brochure highlights some of the successes of the first phase of the Millennium Programme, presenting the various new or upgraded instruments and some of the new science that they have facilitated. It also looks forward, embracing the next phase launched in 2007 and due to run until 2014, as well as setting the scene – in the framework of our ILL2020 vision – for developments yet further into the future. We are greatly helped in this through funding from ESFRI (European Strategy Forum for Research Infrastructures) to explore technical innovations that may underpin new instruments.

We very much look forward to working with our users to develop a well-rounded case for next-generation instruments and infrastructure to serve their science to 2025 and well beyond.



Professor Richard Wagner
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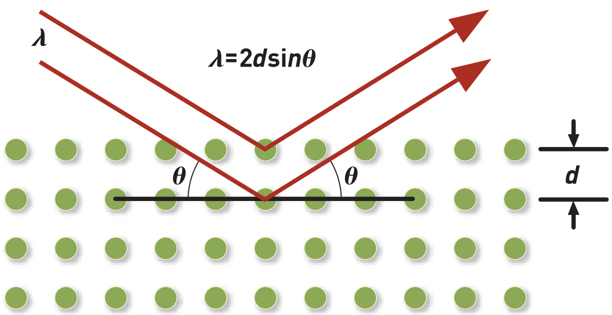
THE ELEMENTS OF NEUTRON SCATTERING

Neutrons are incredibly versatile in the information they can provide for research in physics, chemistry, biology, materials and earth sciences, and engineering.

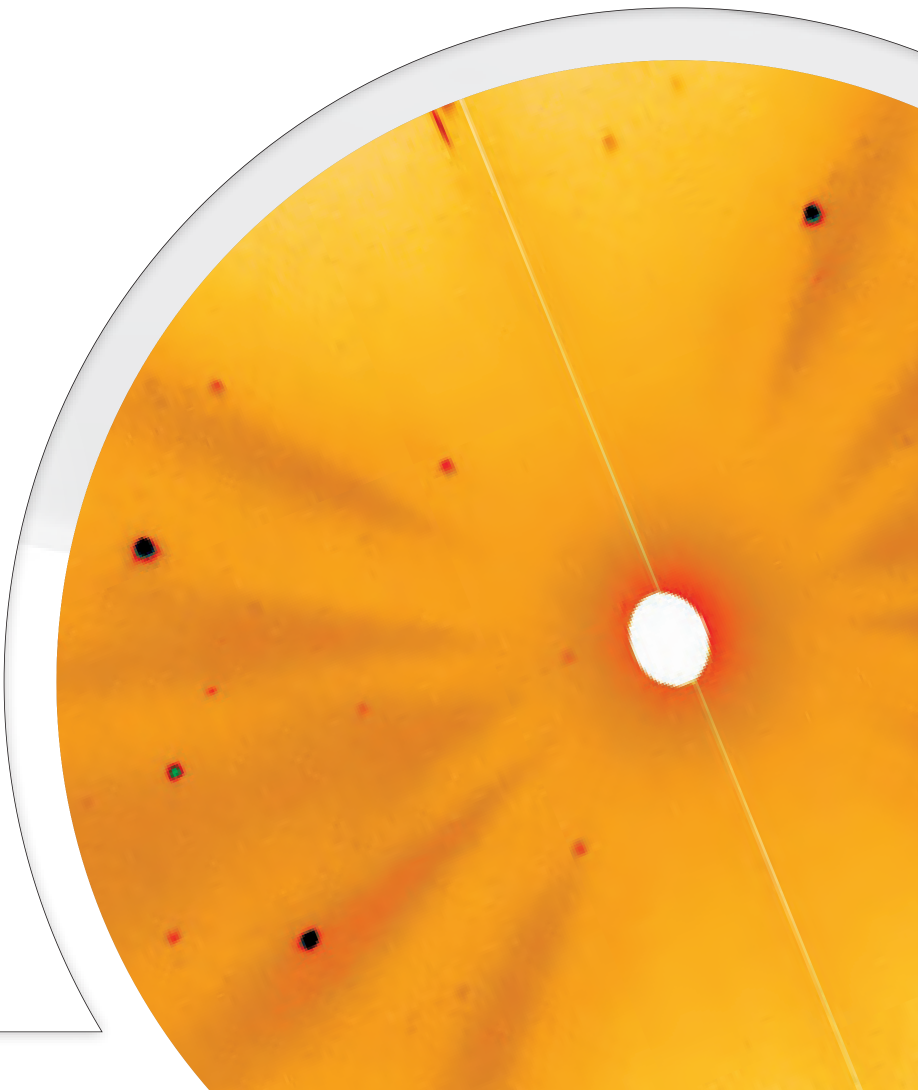
Neutrons are subatomic particles found in the nuclei of atoms. They are emitted during certain nuclear processes including the fission of uranium-235, so they can be obtained from a nuclear reactor. Like all subatomic particles, neutrons obey the laws of quantum mechanics, which means they behave like waves as well as particles. The wavelength of neutrons (tenths of nanometres) corresponds to the distances between atoms and molecules in solids and liquids. Consequently when they interact with matter, for example, a regular array of atoms or molecules in a crystal lattice, the neutron waves are reflected, or scattered – **neutron diffraction**. Waves reflected from similarly oriented planes of atoms in the crystal interfere and re-inforce each other periodically to produce a characteristic **diffraction pattern** (like water waves on a lake that meet after being reflected off two rocks). The pattern can be recorded as a series of peaks of the scattered neutron intensity, which provides information about the position of the atoms and the distance between them.

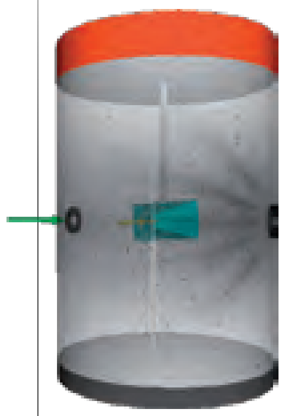
There are many variations of the scattering process, which give the technique its wide applicability to many different kinds of materials. As well as determining structure, neutron scattering can also reveal the motions of atoms and molecules via an exchange of energy between the neutrons and the sample – **inelastic scattering**. As with all radiation, the energy of neutrons is related to both their wavelength and their velocity. This relationship is fully exploited in the design of neutron-scattering instruments and experiments.

The ILL has a dedicated nuclear reactor – the most intense neutron source in the world – which provides intense beams of neutrons over a wide energy range, from **hot** (high-energy or fast), **thermal** (medium-energy), **cold** (low-energy or slow) to **ultra-cold** (very slow) neutrons – for not only neutron-scattering experiments but also nuclear and fundamental physics experiments. Ultra-cold neutrons are employed in extremely precise experiments on gravity, and on the properties of neutrons themselves.



THE BASICS OF NEUTRON SCATTERING
NEUTRONS ARE REFLECTED BY ATOMS IN A CRYSTAL LATTICE AT A CERTAIN ANGLE, WHICH DEPENDS ON THE NEUTRON WAVELENGTH (λ), THE SPACING OF THE CRYSTAL LATTICE (d) AND AND THE ANGLE AT WHICH THE BEAM IS REFLECTED (θ). THIS RELATIONSHIP IS CALLED BRAGG'S LAW – $\lambda=2d\sin\theta$. THIS MEANS THAT THE LARGER THE SPACING OF THE CRYSTAL LATTICE, THE SMALLER THE ANGLE OF REFLECTION.

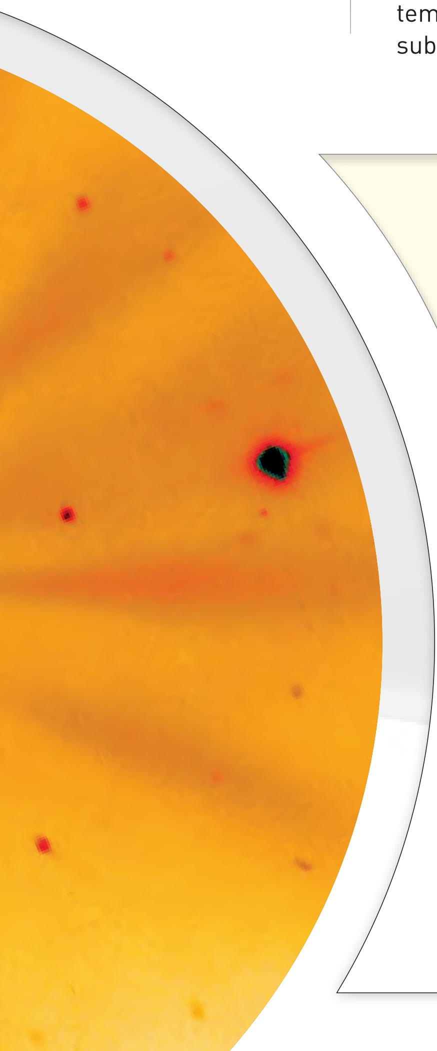


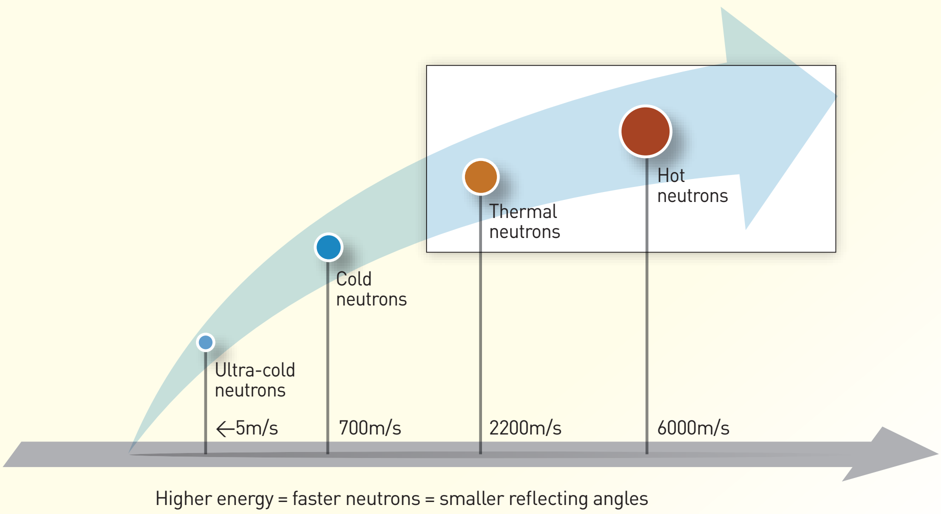


WHY NEUTRON SCATTERING IS USEFUL

Neutrons have properties that allow them to reveal the structure and behaviour of materials, at the atomic and molecular scale, which are not accessible using other types of radiation such as X-rays.

- They are **electrically neutral**, so can penetrate deep into matter, while remaining non-destructive. This makes them an ideal probe for studying – for example, biological samples or engineering components under extreme conditions of pressure, temperature and magnetic field, or within chemical-reaction vessels.
- They have **wavelengths**, varying from 0.01 to 100 nanometres, which are suitable for exploring structures ranging from materials consisting of one kind of atom, to polymers and large, complex biomolecules. They can even monitor interatomic distances in the welds of engineering components, thus acting as a 'strain gauge'.
- The associated **energies** of millielectronvolts are of the same magnitude as the diffusive motion of atoms and molecules in solids and liquids, and the coordinated mechanical or magnetic oscillations in single crystals or vibrations in molecules. The slow motions within larger assemblies (down to 1 microelectronvolt) can be detected. Very small changes in energy measured at low temperatures may also be used to measure subtle quantum processes in exotic materials.
- Neutrons also have a **magnetic moment**, called spin, which can interact with the electron spins in magnetic materials. Polarised neutron beams (in which all the spins are parallel) offer a unique tool for characterising exotic materials with complex magnetic structures and behaviour – an area of growing technological interest.
- Neutrons are particularly sensitive to **hydrogen atoms**, so can probe the locations of water molecules in a sample, or the all-important hydrogen in organic and biological molecules associated with pharmaceutical and genomic research. They are also a powerful probe of hydrogen-storage materials and fuel cells.
- The variation of scattering power from nucleus to nucleus in a sample can change dramatically even in different isotopes (variants with different masses) of the same element. For this reason, substituting atoms of one isotope with those of another can be employed to 'highlight' structural components. In particular, hydrogen scatters very differently from one of its heavier isotopes, deuterium. The degree of **isotopic substitution** can be controlled such that the scattering strength in one part of a structure is the same as in the surrounding medium, thus rendering it invisible, so that another part of the structure stands out in contrast (**contrast matching**).
- Neutrons can readily be **reflected** off thin layers and interfaces, making them suitable for studying coatings, nanomaterials and advanced multilayer composites.





RANGE OF NEUTRON ENERGIES AVAILABLE AT THE ILL

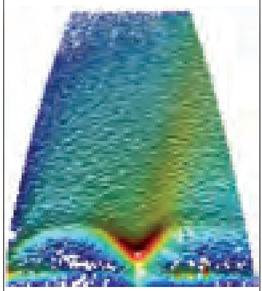
NEUTRON-SCATTERING TECHNIQUES

Techniques can be divided into two types.

- **Diffractometry** maps the distances between atoms in a material to reveal its **3-D structure** at the atomic or molecular level. The numbers (intensity) of diffracted neutrons at various scattering angles are recorded. The sample may be a single crystal, a powder, an amorphous material or a liquid, or even a large composite object.
- **Spectrometry** enables the **dynamics** (both individual and coordinated motions or oscillations) of atoms, molecules and electron spins to be studied by recording the changes in energy in a neutron beam after it has been inelastically scattered by a sample.

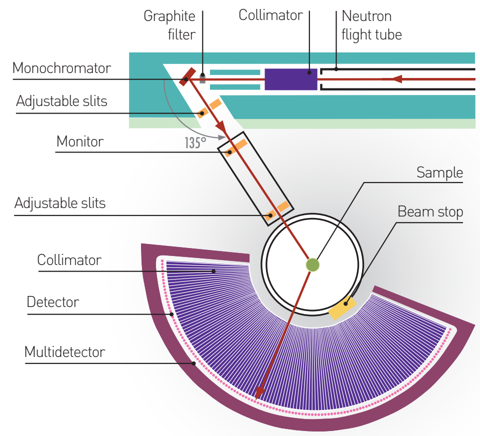
DIFFRACTOMETRY

- **Single-crystal diffractometry** uses an instrument, such as a four-circle diffractometer, that allows the sample crystal to be oriented in all directions with respect to the incident beam. The scattered neutrons, which are then detected over a range of angles, give a diffraction pattern according to Bragg's law. It is a powerful method for investigating structural details in materials at the scale of atoms or molecules.



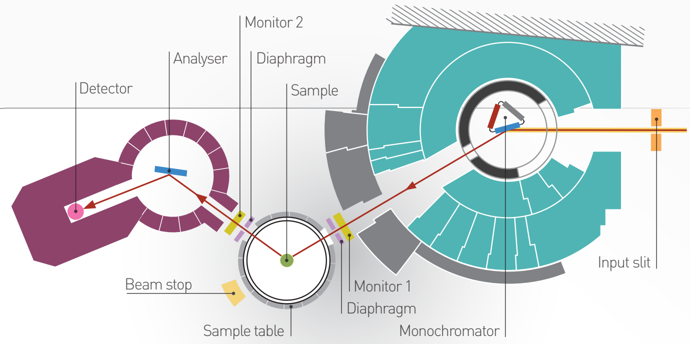
- **Powder diffractometry** records the scattered neutron beam from every possible angle simultaneously. As its name suggests, it is the method of choice for powders and solids composed of tiny crystals that are randomly oriented. The data are then processed and converted into a one-dimensional diffraction pattern from which the structure can be determined.
- **Laue diffractometry** uses a beam composed of neutrons of a broad wavelength band to record simultaneously the many reflections in an, often very small, crystalline sample with a large, complex molecular structure such as a protein. The diffraction pattern is captured using a large-area detector.
- **Small-angle neutron scattering (SANS)** measures diffracted neutrons specifically at very small angles of scattering, and it requires a special, very long instrument to resolve the small angles. SANS is used to investigate structures that are relatively large, such as polymers or biological structures, and also extended defects and heterogeneities in materials for engineering..
- **Reflectometry** measures the variation in intensity of the reflected neutron beams from surfaces and interfaces (reflectivity curves). It is used to characterise the structure of surfaces and thin layers.

TYPICAL INSTRUMENT LAYOUTS



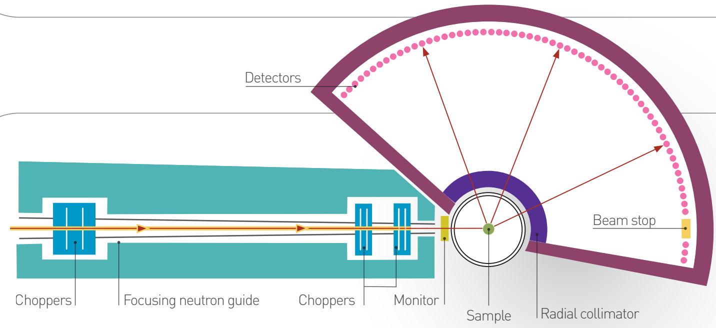
A TYPICAL NEUTRON DIFFRACTOMETER

THE INCIDENT NEUTRON BEAM PASSES THROUGH A SERIES OF COLLIMATORS, A MONOCHROMATOR, SLITS AND FILTERS BEFORE REACHING THE SAMPLE. A LARGE, CURVED POSITION-SENSITIVE DETECTOR COMPOSED OF MANY INDIVIDUAL ELEMENTS THEN MEASURES THE NEUTRON INTENSITIES AT VARIOUS ANGLES.



A TYPICAL TIME-OF-FLIGHT SPECTROMETER

A SERIES OF ROTATING CHOPPERS SELECTS NEUTRONS OF A GIVEN VELOCITY (ENERGY), WHICH ARE THEN SCATTERED BY THE SAMPLE BEFORE BEING COUNTED AT A SEGMENTED DETECTOR, AND THE ENERGY MEASURED ACCORDING TO THE TIME OF ARRIVAL.



A TYPICAL TRIPLE-AXIS SPECTROMETER

THE TRIPLE-AXIS SPECTROMETER IS SO-NAMED BECAUSE THE NEUTRON BEAM IS REFLECTED OR DIFFRACTED THREE TIMES: ONCE BY A MONOCHROMATOR, WHICH SELECTS NEUTRONS OF A PARTICULAR ENERGY (FIRST AXIS); THEN BY THE SAMPLE, WHICH SCATTERS THE NEUTRONS (SECOND AXIS); AND FINALLY BY AN ANALYSER, WHICH ANALYSES THEIR ENERGIES (THIRD AXIS), BEFORE REACHING A DETECTOR WHERE THE NEUTRONS ARE COUNTED.

SPECTROMETRY

- **Triple-axis spectrometry (TAS)** measures the difference of wavelength, and thus energy, between the incident and scattered neutrons, while simultaneously measuring the angle of scattering. An analyser is used to determine the wavelength composition of the diffracted neutron beam.
- **Time-of-flight (TOF) spectrometry** measures the time neutrons take to travel from the sample to the detector, and thus their velocity (usually kilometres per second) and energy.
- **Neutron spin-echo (NSE) spectrometry** is used when the neutron energy or wavelength variation is too weak to be detected by TAS or TOF. Spin-echo spectrometers measure the variation of the neutron spin orientation, caused by transfers in energy associated with molecular movements over relatively long timescales, such as the slow movement of polymer chains.
- **Neutron backscattering spectrometry** measures the change in energy of scattered neutrons using a set-up in which the energy of incident and scattered neutrons can be selected particularly precisely. It is used to analyse the dynamics of atoms over timescales between those measured by TOF and NSE methods.

A third technique, **polarisation analysis,** employs polarised neutrons to investigate the magnetic properties of materials. There are several ways of producing a beam of polarised neutrons: for example, by using polarising mirrors or polarising filters (usually helium-3 which lets through only neutrons polarised in one direction).

NUCLEAR AND FUNDAMENTAL PHYSICS EXPERIMENTS

In addition to neutron scattering, the ILL carries out nuclear physics experiments using neutrons to induce nuclear reactions, and also experiments investigating the fundamental properties of neutrons.

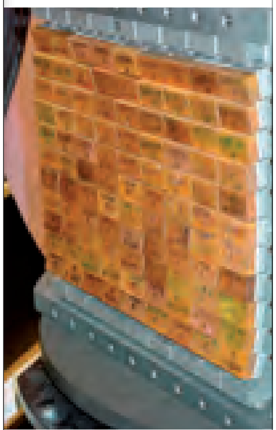
- A range of novel devices is required, such as **targets** which generate rare, unstable isotopes when neutrons impinge on them, **gamma-ray and exotic-isotope spectrometers**, and specialised instruments such as '**neutron bottles**' and '**gravitational traps**' for studying the fundamental properties of neutrons at very low energies.

THE NEUTRON FACILITY INFRASTRUCTURE AND INSTRUMENTS

After the neutrons emitted by the reactor have been moderated to lower energies, they are conducted to a suite of instruments installed in large experimental halls. Each instrument is designed to carry out a particular technique using neutrons with a selected energy range, using a series of devices, many of which were designed and developed at the ILL.

PREPARING THE INCIDENT NEUTRON BEAM

- **Neutron guides** and optical devices called **supermirrors** transport and focus the neutron beams, guiding them to the measuring instruments over distances up to 100 metres.
- **Collimators** make the beam more parallel, improving the angular resolution of the instrument.
- Various optical devices – **slits** and **diaphragms** – optimise the instruments' performance.



A COPPER CRYSTAL MONOCHROMATOR

A SUPERMIRROR FOR FOCUSING NEUTRONS

- Rotatable **monochromators** – usually crystals – select neutrons according to wavelength.
- Various **filters** select the neutrons according to direction and energy; **spin filters** polarise the neutron spin.
- **Choppers** – rotating discs with segments cut-out, or cylinders with square windows (Fermi choppers) – are used in TOF instruments to select bunches of incident neutrons with the same and known velocity.

THE SAMPLE ENVIRONMENT

- A **sample stage** can often be oriented at different angles.
- A variety of **cryostats** – both general-purpose and extremely specialised – maintain samples at down to near absolute-zero temperatures.
- **Pressure cells**, **humidity chambers** and **furnaces** allow experiments to be carried out under a wide range of environmental conditions.
- **Superconducting magnets** are used to apply very high magnetic fields to samples.

MEASURING THE SCATTERED NEUTRONS

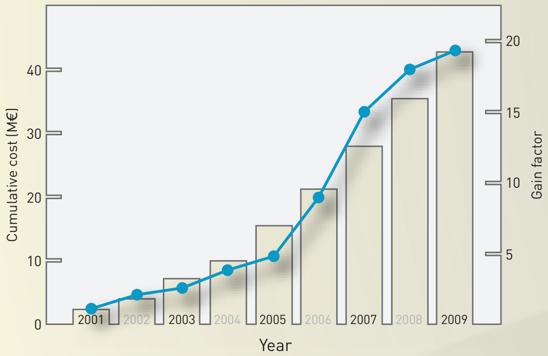
- Rotatable crystal **analysers** determine the wavelength (energy) composition of the diffracted neutron beam in inelastic scattering experiments.
- **Detectors** count the number of neutrons scattered by the sample. The ILL has many different detector types – gas and solid-state detectors – ranging from single-point detectors to large one and two-dimensional multi-detectors, the most advanced of which are developed and built in-house.
- **Automated instrument-control** and **data-collection electronics** manage individual experiments.
- Sophisticated **data-reduction** and **analysis suites** transform the raw data into the physical quantities that can be visualised and interpreted.
- All the equipment requires specialised **shielding** to prevent stray neutron radiation leaking out or to reduce the background 'noise' from unwanted scattered neutrons.

AT THE FOREFRONT OF NEUTRON SCIENCE

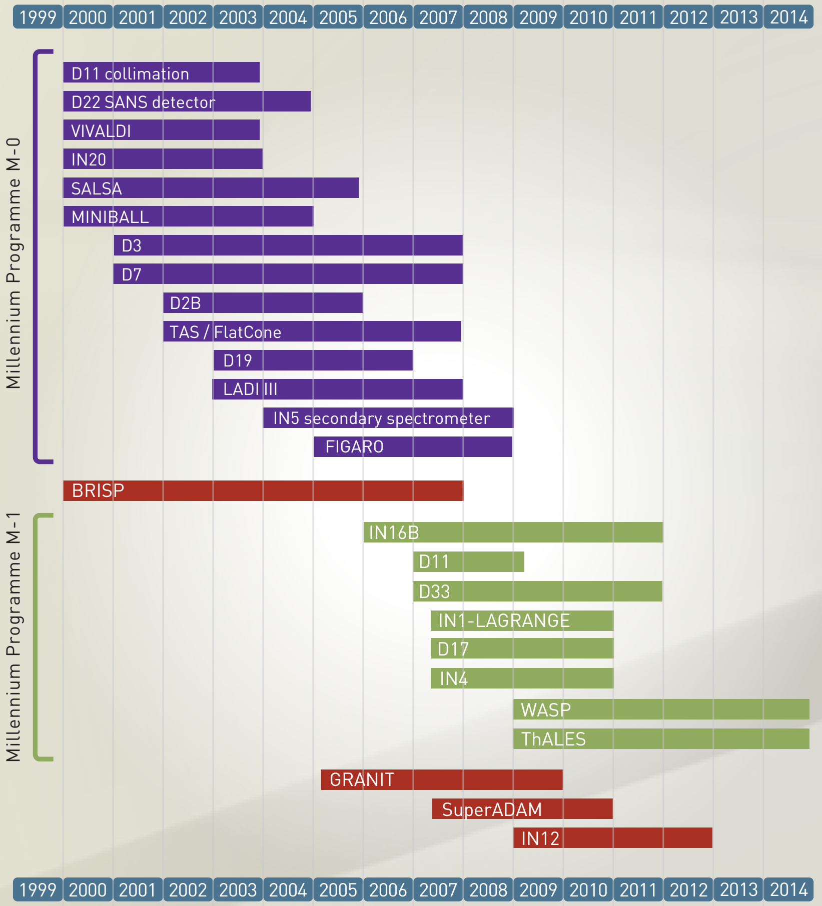
In 2000, the ILL launched the Millennium Programme designed to establish a sustainable strategy for the continual improvement of its infrastructure and instruments

By the late 1990s, it had become clear that there was an urgent need for a fresh and vigorous initiative to keep the ILL at the leading edge of neutron science, and so an ambitious roadmap for renewal was drawn up. Founded on new scientific opportunities, as well as exciting developments in instrument design – detectors, monochromators, spin polarisers and higher-performance neutron guides, this strategy attracted substantial funding from the ILL's Associate Members. Together with the skill and diligence of ILL staff, we have been able to deliver six new instruments, and extensively upgrade a further eight in the first phase (M-0) of our Millennium Programme. A new CRG instrument has been added in parallel (Collaborating Research Groups, or CRGs, from various partner countries develop their own instruments). The replacement of the neutron guides alone has led to a doubling of neutron intensity for many instruments, and has contributed to improving the average detection rate of neutrons for all instruments by 19 times. This exceeds the programme's original goal of achieving an average gain factor of five to eight.

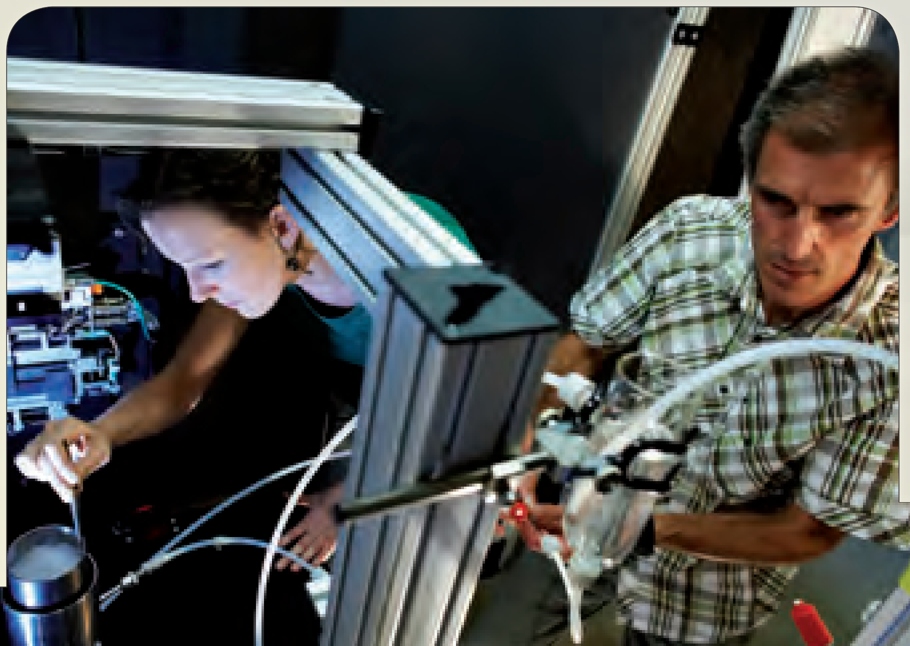
Our plans do not stop there – the next (M-1) phase of the Millennium Programme, is already underway, with funding in place to deliver a further 11 new or upgraded instruments (including both those available to all researchers from member countries and those implemented by CRGs).



THE AVERAGE INCREASE IN DETECTION RATE FOR ALL ILL INSTRUMENTS COMPARED WITH THAT IN 2001 (BLUE LINE CONNECTING POINTS), AS WELL AS THE EVOLUTION OF THE BUDGET DURING THE FIRST PHASE M-0 OF THE MILLENNIUM PROGRAMME



THE MILLENNIUM PROGRAMME TIMETABLE PAST, PRESENT AND NEAR-FUTURE, DIVIDED INTO THE COMPLETED (M-0) PHASE AND THE CURRENT (M-1) PHASE. BRISP, IN12, SUPERADAM AND GRANIT ARE DISPLAYED SLIGHTLY DIFFERENTLY BECAUSE THEY ARE LARGELY OR ENTIRELY FUNDED THROUGH EXTERNAL BODIES



THE LAUE DIFFRACTOMETER LADI-III

EXCITING DEVELOPMENTS TO DATE

The capacity of instruments for studying large-scale structures, ranging from the nano to the microscale, has greatly increased. The SANS instruments **D11** and **D22** have been recently upgraded and are now regarded as the best in the world.

A complete overhaul of **D11** (p.15) has enabled a wider range of length-scales to be probed in a single measurement, while the three-fold increase in detected neutron intensity has improved sensitivity, offering bright new insights into **soft-condensed matter** – from **liquid crystals** to **polymer processing**.

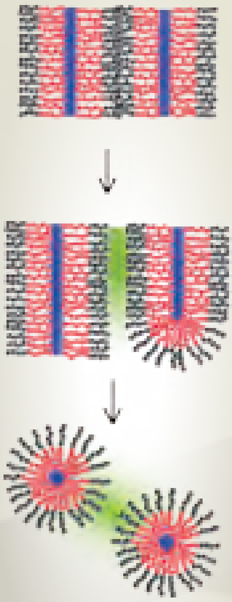
A new detector for the high-neutron intensity SANS instrument **D22** (p.17) has increased its maximum count-rate by more than 40 times, greatly enhancing its capacity for **kinetic measurements of chemical reactions**, and the very small or weakly-scattering samples typically available for **biomolecules such as proteins**.

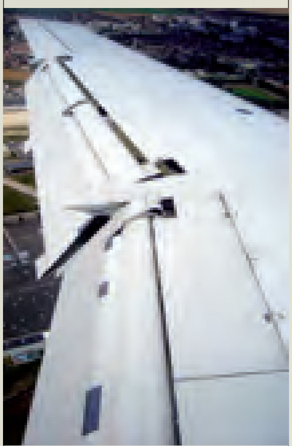
The ILL's first horizontal-surface reflectometer **FIGARO** (p.21) allows the rapid study of a wide range of fluid interfaces for the first time – opening up new fields such as **atmospheric and environmental chemistry**, and extending the scope of studies of **biomembranes** – key to developing better **drug-delivery processes**.

The newly formed **Partnership for Soft Condensed Matter (PSCM)*** will provide the facilities to prepare and validate samples or perform complementary measurements – crucial in a field where samples are often metastable or history-dependent.

LADI-III (p.23) is an upgrade to the cold-neutron Laue diffractometer (p.6), providing a two to three-fold gain in detected flux. This, coupled with selectively deuterated samples (p.6) provided by the dedicated **Deuteration Laboratory (D-Lab)***, has transformed the scope of neutrons to determine the detailed crystal structure of **small samples of biomolecules**, and, in particular, the hydrogen atoms or bound solvent molecules that are often crucial to function.

The new thermal-neutron Laue instrument **VIVALDI** (p.26) allows rapid surveys of structure across a wide range of length-scales, enabling both the preliminary investigation of **very small crystals of new materials** and the mapping of large swathes of features over a range of conditions such as **temperature, pressure and magnetic field**.





A new large solid-angle 'banana' detector has improved the detection rate of the thermal neutron single-crystal diffractometer **D19*** (p.16) by a factor of 20, making it ideal for determining medium-sized crystal structures, and a unique probe of the structures of **biological** and **industrial fibres**.

The high-resolution powder diffractometer **D2B*** (p.12) now includes a new 2-D detector, which allows it to tackle yet smaller powder samples, in more extreme sample environments. It is now producing detailed structures of materials undergoing chemical changes such as those occurring during the **discharge of a battery**.

A new strain-scanning instrument **SALSA*** (p.25) supported by the **FAME38 (Facility for Materials Engineering)*** interface laboratory can reveal the detailed structure of **engineering materials under real operating conditions**, and is attracting increasing interest from the **aerospace** and **transport industries**, as well as from companies designing the next generation of **nuclear power plants**.

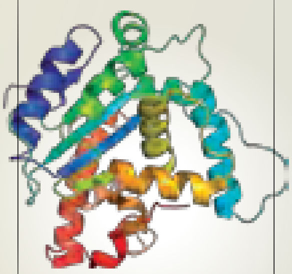
* Projects wholly or partly funded by external bodies: D2B, D19 and SALSA largely through the UK Engineering and Physical Sciences Research Council (EPSRC), together with FaME38 and the D-Lab; upgrades to CRG instruments through the partner organisations, BRISP (German-Italian), IN12 (Germany), SuperADAM (Germany-Sweden) and GRANIT (France).

The hot-neutron polarised diffractometer **D3C** (p.13) is now equipped with a device for controlling neutron spin, CRYOPAD, as well as helium-3 spin filters (p.7), needed to determine the **magnetic structures of novel materials** useful in **information processing and storage**. **D7** (p.14) is a unique cold-neutron TOF spectrometer (p.6) that analyses polarisation in three dimensions, and is designed to explore the complex behaviour of **novel functional magnetic materials**, The new array of polarising supermirrors (p.7), is one of several features that have increased the detection rate by a factor of 70.

The cold-neutron TOF instrument **IN5** (p.18) has enjoyed a complete overhaul, including a new vacuum flight chamber equipped with a large-area array of linear position-sensitive detectors. This instrument is once more world-leading – exploring fundamental molecular behaviour and low-energy dynamics, important in advanced functional compounds such as **electronic** and **biological materials**.

The new thermal-neutron Brillouin scattering CRG spectrometer **BRISP*** (p.20) is optimised to operate at small scattering angles with good energy resolution. Its applications are centred on studying the dynamics of disordered systems such as **glasses, liquid metals** and **water in biological systems**.

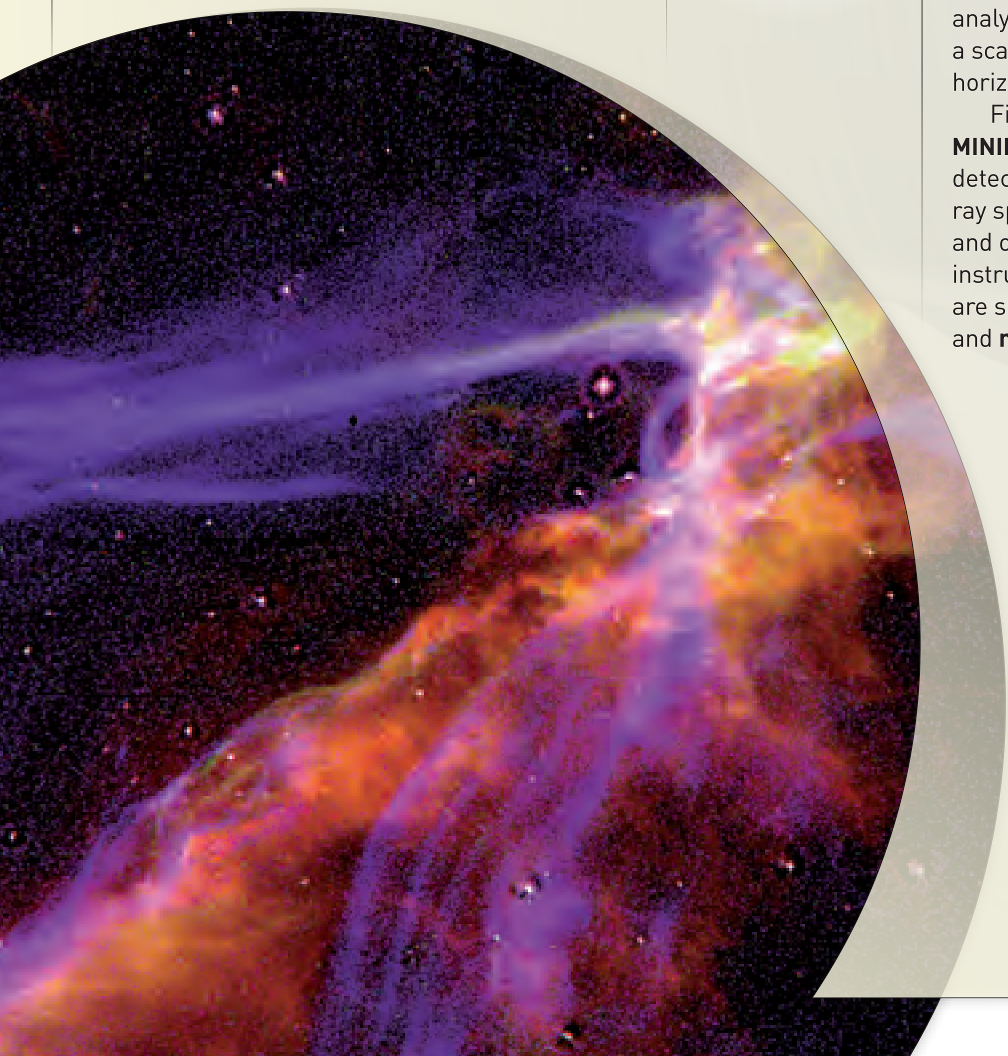
VIEW OF THE BRISP SPECTROMETER



For some years, **IN20** (p.19) has been a flagship, thermal-neutron TAS instrument for studying magnetic behaviour relevant to functional materials such as **high-temperature superconductors**. Now, equipped with a new monochromator and an upgrade to equipment for polarisation analysis, it can collect data 30 to 50 times faster in full polarisation mode.

Most of the suite of **TAS** instruments (p.6) now also benefits, in terms of speed and breadth of mapping **magnetic scattering in exotic materials**, thanks to the roving multi-analyser detector **FLATCONE** (p.22); thirty-one separate analyser/detector channels simultaneously cover a scattering angle of 75°, and can tilt out of the horizontal scattering plane.

Finally, in the domain of nuclear physics, **MINIBALL** (p.24), which is an array of germanium detectors, has considerably improved gamma-ray spectroscopy probing nuclear structure. This and other modifications allow the ILL's Lohengrin instrument to study **highly unstable nuclei** that are significant in the study of **stellar processes** and **next-generation nuclear power plants**.



THE FUTURE

The momentum of the Millennium Programme has continued, with the next phase launched in June 2007, and funding from the Member Associates of 42.9 M€. Upgrades to the existing the vertical surface reflectometer, **D17**, as well as the CRG instrument ADAM, which has reappeared as **SuperADAM***, will be complete in 2010, and will provide enhanced tools to unravel the structure of thin **films** and **magnetic multilayers** in **hard and soft-condensed matter**.

The thermal-neutron TOF instrument **IN4** is the highest-flux instrument of its class in the world, and will enjoy much increased sensitivity by the end of 2010, enabling it to characterise molecular motion, vibrations and magnetic excitations in **functional materials** that operate under **everyday conditions**.

Two further high-resolution instruments are under construction: the backscattering spectrometer **IN16B** will improve on its predecessor IN16, with up to 10 times more flux and a wide dynamic range, enabling new science in the dynamics of **macromolecules** and other systems where relatively **slow dynamics** are crucial to function.

The high-intensity spin-echo spectrometer **WASP** will be 500 times brighter than the high-flux option of the existing instrument IN11 that it will replace. It will enable the **dynamics of soft matter and biomaterials** to be explored, particularly for small or weak scatterers such as **biological membranes** and **weakly magnetic materials**.

The reconstruction of **IN14** will provide the cold-neutron TAS **ThALES** with greatly increased flux and kinematic range, opening up new areas of the fundamental physics of functional materials, particularly **quantum magnetism** and **strongly-correlated electron systems**; work in this field will be boosted further through an upgrade to our other cold neutron TAS, the CRG instrument **IN12***.

The beryllium-filter configuration of the hot-neutron spectrometer **IN1** will be radically overhauled in the **LAGRANGE** project, raising the sensitivity 100-fold, with particular benefits for real, reactive chemical systems such as **catalysts** and **materials for hydrogen storage**.

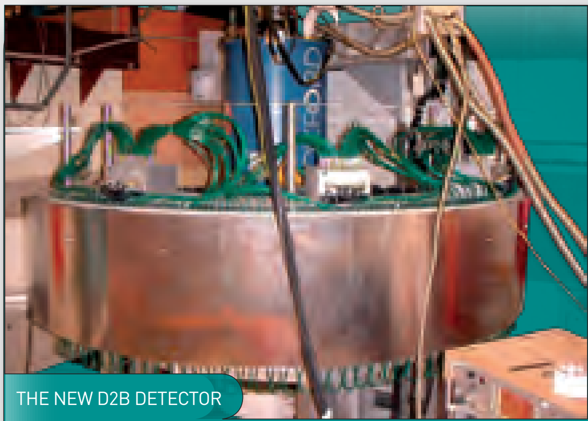
The study of large-scale structures will be even better served when **D33** comes on stream. This new TOF SANS instrument optimised for materials science, will elucidate the structure of **nano- or meso-structured systems** embracing **novel magnetic recording media** and **superconductors**. It will also take pressure off the Facility's other SANS instruments, which are among the most oversubscribed, and free them for studies in biology and soft-condensed matter.

Finally, the second-generation ultra-cold-neutron gravitational spectrometer **GRANIT*** will provide the keenest insights into the properties of **quantum-gravity states** from later in 2010.

So, after this period of extensive development, what next?

ESFRI (European Strategy Forum for Research Infrastructures) has funded us generously to explore technical innovations that may underpin several new instruments, while we will work with our user community over the next year to develop a well-rounded case for next-generation instruments and infrastructure – **the ILL2020 Vision** – to serve their science to 2025 and well beyond.

REFERENCE: ILL 20/20 *Upgrade in European Roadmap for Research Infrastructures* (European Communities, 2006), www.ill.eu/news-events/press/esfri-roadmap, and the update (European Communities, 2008), ftp.cordis.europa.eu/pub/esfri/docs/esfri_roadmap_update_2008.pdf



THE NEW D2B DETECTOR

SUPER
D2B

The upgraded D2B powder diffractometer has opened up the field of high-resolution neutron diffraction to scientists who have to work with very small samples when studying their crystallographic or magnetic structures under a variety of environmental conditions.

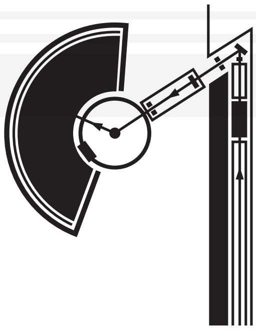
INSIDE POWDERS MORE CLEARLY

Many technologically important materials – whether newly synthesised inorganic compounds or naturally occurring minerals – come in the form of a powder or polycrystalline solid. To study their structure requires a special technique called powder diffraction (p.6). The randomly-oriented tiny crystals composing the powder scatter the neutron beam in the different directions characteristic of the material's structure. The scattered neutrons are then recorded by a detector.

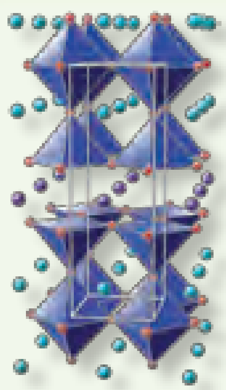
For 25 years, the ILL's high-resolution two-axis diffractometer, D2B, has been the first port of call for scientists exploring the structure of new materials in powder form. It has played a crucial role in investigating complex electronic materials and minerals. D2B is one of the ILL's most requested instruments and is extremely productive. Because samples are often very small, they require a highly sensitive detector, and a neutron beam of optimum brightness and sharpness. To meet these needs, as well as an increasing demand for access, D2B was upgraded to provide the best possible resolution, together with rapid and reliable performance.

THE UPGRADE

- **A MORE SENSITIVE DETECTOR:** a new 2-D detector was constructed, increasing the detector area and providing a six-fold increase in detection rate. A pattern can now be collected in about 30 minutes, and samples down to 200 milligrammes may now be measured routinely.
- **A SHARPER NEUTRON BEAM:** the optical devices (monochromator and collimator) were upgraded to provide more precise and faster control of the quality and flux of the primary beam.
- **IMPROVED SAMPLE HANDLING AND ENVIRONMENT:** a new rotating, multi-sample changer allows samples or sample environments (cryostats, furnaces) to be changed over automatically, thus reducing the down-time of the experiment. Up to nine samples can be installed on the sample changer and measured sequentially without human intervention. New cooling, heating and magnetic devices have been designed to simplify operations.



NEW SCIENCE



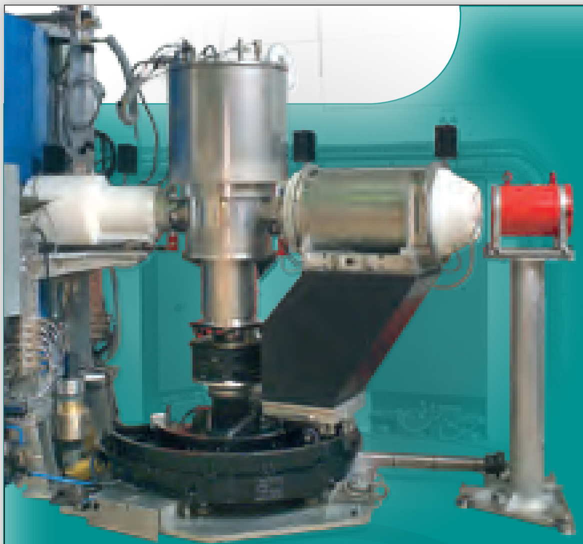
THE CRYSTAL STRUCTURE OF $YBa_2Fe_3O_8$

PUZZLING PEROVSKITES

Complex, mixed metal oxides called perovskites are of huge technological interest because some of them have extraordinary electronic and magnetic properties such as superconductivity and colossal magnetoresistance. A knowledge of the detailed arrangement of the atoms, particularly of oxygen, in the structure is the prerequisite for understanding their behaviour. The new super-D2B can be used routinely to study these samples, often available only in small amounts.

- Recently, studies of one iron-oxide compound, $YBa_2Fe_3O_8$ (related to the famous high-temperature cuprate superconductor $YBa_2Cu_3O_8$), carried out on D2B, revealed the locations of the oxygen atoms in the crystal structure.
- Barium iridium oxide, $BaIrO_3$, which also shows interesting electronic behaviour, was explored at high pressures, revealing structures never before seen for a perovskite.

PROJECT TEAM: **E. Suard**, J. Allibon, P. Cross, L. Gendrin, A. Hewat, C. Ritter, P. Thomas and P. Van Esch
COST: 0.8 M€, with additional funding from the UK Engineering and Physical Sciences Research Council (EPSRC)
PROJECT DURATION: 2002-2005

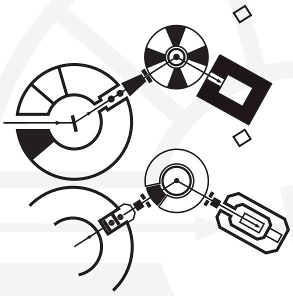


D3C WITH THE CRYOPAD POLARIMETER INSTALLED

D3C

The D3 polarised neutron diffractometer is now equipped to investigate in-depth the magnetic structure and magnetisation distribution of materials with potentially useful magnetic and electronic properties.

A NEW PROBE FOR ADVANCED MAGNETIC MATERIALS



One of the most exciting areas in electronics is the development of new materials with intricate magnetic and electric behaviour that may find use as the next generation of sensors, magnetic recording technology and other devices.

The diffractometer D3 enables users to carry out detailed measurements of a sample's magnetisation, using polarised neutrons (p.5), and has provided deep insights into magnetic behaviour. It has now been re-furbished with advanced instrumentation that will prepare and analyse the polarisation vectors of the neutron beam. With this combination, a technique called spherical neutron polarimetry (SNP) can be carried out on D3, whereby accurate polarisation measurements at all angles can be made. The D3C instrument is the most incisive instrument in the world for mapping complex magnetisation in materials.

THE UPGRADE

- **AN ADVANCED POLARISATION-ANALYSIS SET-UP:** a dedicated third-generation polarimeter, Cryopad-III, was designed to measure polarisation rotations to a precision better than one degree. The device extends technology invented at the ILL for carrying out SNP. It consists of a cryogenically cooled toroidal chamber hosting superconducting coils, soft magnetic yokes and Meissner screens used to orient the beam polarisation vector. Cryopad-III is the best ever zero-field neutron polarimeter. The polarisation is analysed with a device, also invented at the ILL, which uses polarised helium-3 gas as a neutron spin filter. The high-performance spin analyser is hosted by the new magneto-static cavity, Decpol.
- **A VERSATILE DIFFRACTOMETER:** the primary spectrometer has been upgraded to allow measurements at high-field strengths with the 10-tesla magnet. The electronics have been updated, and many of the mechanical components have been replaced to provide a modular and robust instrument.

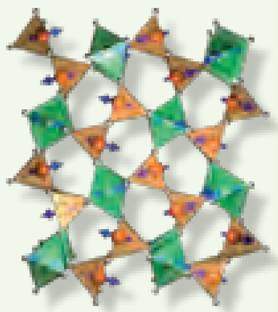
NEW SCIENCE

MAKING THE MOST OF MAGNETISM

- The magnetoelectric effect, whereby an electric field induces a magnetisation, or *vice-versa*, could be the basis of recording devices and novel 'spintronic' information processing. SNP carried out on the classic magnetoelectric material, chromium oxide (Cr_2O_3), was able to map the magnetisation distribution responsible for this property.

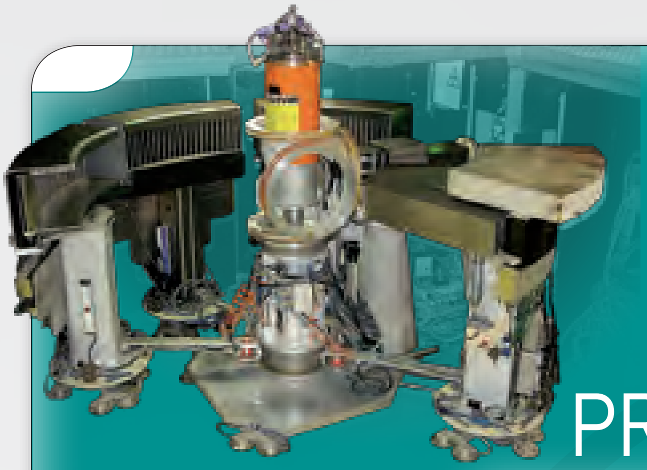
NEW MAGNETO-ELECTRONIC DEVICES

- Such compounds belong to a broader class of materials called multiferroics in which magnetic, electric and mechanical properties are coupled to produce physical effects such as ferroelectricity and various kinds of magnetism. They also have potential in multi-functional magneto-electronic devices. D3C was recently used to study how the magnetic structure of a multiferroic compound, yttrium manganese metal oxide (YMn_2O_5), responded to a magnetic field, which revealed how the magnetic and electric behaviours were coupled.



THE STRUCTURE OF THE MULTIFERROIC COMPOUND YTTRIUM MANGANESE OXIDE

PROJECT TEAM:	**E. Lelièvre-Berna**, E. Bourgeat-Lami, Y. Gibert, N. Kernavanois, J. Locatelli, T. Mary, G. Pastrello, S. Pujol, R. Rouques, F. Tasset and M. Thomas
COST:	0.8 M€, with additional funding from the UK Engineering and Physical Sciences Research Council
PROJECT DURATION:	2002-2005



D7 IN 2007 WITH TWO DETECTOR BANKS COMPLETED AND A THIRD ONE READY FOR THE INSTALLATION OF THE ANALYSER DISCS

D7

The diffuse scattering spectrometer, D7, is a unique instrument for studying magnetic and non-magnetic materials with short-range order and defect structures. It has been upgraded to increase the neutron detection rate by 70 times, and to give a five-fold improvement in energy resolution for dynamic measurements.

PROBING WEAK MAGNETS

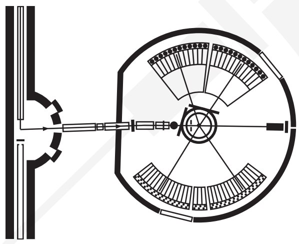
THE UPGRADE

- **IMPROVED TRANSMISSION:** supermirrors, developed and manufactured by the ILL, are used to polarise the incident beam and to analyse it after scattering. However, supermirrors have a very low neutron transmission. Improved supermirrors with much lower losses have been constructed and installed.
- **A GAIN IN INCIDENT NEUTRON FLUX:** the monochromator was upgraded, providing better focusing to increase the intensity by a factor of 1.3.
- **A NEW POLARISER:** the monochromator produces a large divergent beam that the polarising supermirror could not accept. The solution was a new wide-angle focusing supermirror that more than doubled the incident flux.
- **ANALYSER MIRRORS AND DETECTOR BANKS:** the angular coverage of detection had been poor, but with the improved supermirror analysers, and a new configuration of thinner, taller detectors, it was improved by a factor of 6.3.
- **TIME RESOLUTION:** a Fermi chopper replaced the old disc chopper to improve the energy resolution by five times.

Many materials of current interest have structures in which atoms or magnetic components are arranged in a partially disordered way; magnetic compounds, in particular, often show such weak and sometimes fluctuating disorder. Neutron scattering can give information about this disorder if the weak and broad magnetic-scattering component can be teased out from the other, stronger scattering contributions. This requires analysing the polarisation (p.5) of the scattered neutrons in all three directions. D7 is one of two such instruments in the world that can do this.

Disordered systems tend to scatter neutrons diffusely into large regions of space around the sample, and so require a wide detector array. D7's detector covers an angular range of 140°. To gain a complete understanding of fluctuating systems, the energies of scattered neutrons also need to be analysed, and this is done on D7 using the time-of-flight mode and a Fermi chopper (p.7).

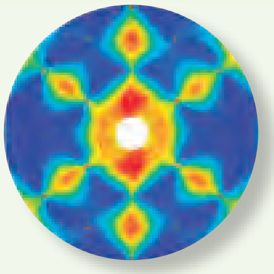
Before the upgrade, the 3-D detection system required long neutron-counting times – over several weeks – to collect enough useful data. Key components of the instrument were therefore given a radical makeover, with the aim of significantly increasing the detection rate and thus greatly extending the number and variety of samples that can be studied.



NEW SCIENCE

A SPIN ICE

In certain rare-earth mixed oxides such as holmium titanium oxide ($Ho_2Ti_2O_7$) the spins on the metal ions sit at the vertices of tetrahedra. Competing interactions with neighbouring spins result in a magnetically frustrated structure called a spin ice, which is of great theoretical interest, as defects in this state are a type of magnetic monopole. Studies on D7 have shown that the correlations in $Ho_2Ti_2O_7$ are of the type required by theories of emergent magnetic monopoles.



THE DIFFUSE SCATTERING MAP FROM HOLMIUM TITANIUM OXIDE

QUANTUM CRITICAL BEHAVIOUR REVEALED

The heavy-metal magnetic compound, cerium palladium silicide ($Ce_3Pd_{20}Si_6$) has a cubic structure with cerium atoms located in two different locations. D7 experiments showed that when a magnetic field is applied, the long-range magnetic order of the cerium spins is gradually suppressed to reveal the subtle quantum interactions between cerium spins at the two sites.

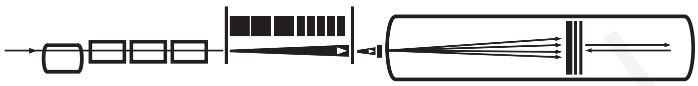
PROJECT TEAM:	**J. R. Stewart**, I. S. Anderson, K. H. Andersen, J. F. Barthelemy, P. P. Deen, P. Georg, W. Graf, P. Høghøj, F. Horst, A. M. Murani, J. M. Preston, H. Schober and M. Thomas
COST:	0.95 M€
PROJECT DURATION:	2000-2007

D11

The small-angle neutron spectrometer D11 has been completely upgraded, resulting in a significant improvement in sensitivity and resolution. This is already benefiting research into technologically important materials with a hierarchical structure over a range of length-scales.

A NEW BENCHMARK FOR SANS

D11 IN NEW COLOURS



Small-angle neutron scattering (SANS, p.6) provides unique insights into structure at the molecular and supramolecular scale, for example, in soft matter (polymer-based architectures, proteins, cell membranes) and electronic materials (complex superconductors). D11 is one of the world's most powerful and versatile SANS instruments, and has been in operation for nearly 40 years.

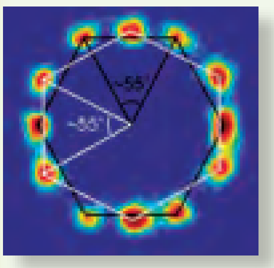
New scientific challenges are imposing ever greater demands – to explore structure on shorter timescales, often over a range of length-scales in one measurement, and on smaller samples. To meet these needs, D11 has been completely overhauled, and is now re-established – together with D22 (p.17) – as the world's leading instrument for studying soft and biological matter. A key driver is the increasing interest in 'smart materials' such as complex microgels containing nanoparticles, of interest in drug delivery and as 'nano-machines'. This requires measuring very low-angle scattering with the best possible neutron flux and resolution, and over a broad range of length-scales simultaneously.

The upgrade is part of a strategy to provide the best possible SANS facilities, which also include D22, and a new dedicated instrument D33 for studying electronic and magnetic behaviour in inorganic materials.

THE UPGRADE

- **IMPROVED INTENSITY:** new redesigned moveable guides were installed, which increased the neutron flux by a factor of three, while optimising the flexibility for different operating conditions.
- **FASTER, CLEARER RESULTS OVER A WIDER RANGE OF ANGLES:** the detector, which is mounted on a moveable trolley in an evacuated tube-shaped tank, was replaced by a larger, more efficient device with a 10-fold increase in the maximum detector count-rate and a doubling in resolution and sensitivity. The tank was widened and extended, and a new compact trolley installed.

NEW SCIENCE



THE DIFFRACTION PATTERN FROM THE MAGNETIC FLUX LINES IN A NEW IRON SUPERCONDUCTOR, KFe_2As_2

A NEW SUPERCONDUCTOR

- In 2008, a new class of superconductors was discovered, with a crystal structure containing layers of iron atoms sandwiched between layers of arsenic or similar elements. The underlying mechanism is different from that in conventional superconductors and may be magnetic in origin. D11's increased intensity could reveal the distribution of magnetic flux in KFe_2As_2, even though the scattering signal was quite weak.

HYDROGEL NANOPARTICLES

- Many polymers form minute capsules when dispersed in a solvent to form microgels with potential applications such as controlled-release vehicles for bioactive molecules, perfumes or pigments, and also as filters and nano-actuators. Understanding how they behave is of key importance in tuning their shape, size and stability. Recent experiments on the upgraded instrument showed that temperature-sensitive microgel particles swell up at room temperature but collapse at higher temperatures. The results were considerably clearer than with the old detector.

PROJECT TEAM:	**P. Lindner,** J. Aléo, C. Amrouni, D. Bowyer, S. Caunt, R. Gandelli, F. Millier, H. Ortiz, A. Perillo-Marcone, G. Pastrello, M. Platz, R. Schweins, and J.-A. Vidal-Garcia
COST:	1.534 M€
PROJECT DURATION:	2005-2009

D19

The D19 diffractometer for single-crystal and fibre diffraction has a new, more efficient 'banana' detector, combined with an increased neutron flux and flexibility to change neutron wavelength, which greatly improves the quality, scope, and throughput of experiments in structural chemistry and biological materials.

A FAST DIFFRACTOMETER FOR FUNCTIONAL MATERIALS

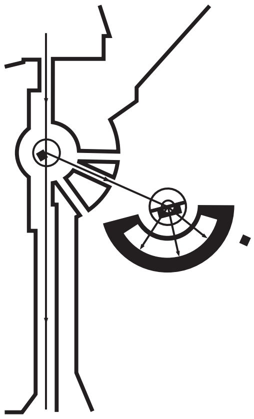
The D19 is a monochromatic diffractometer that specialises in single-crystal studies of large systems in structural chemistry. It has pioneered studies of materials with repeating crystal units over length-scales of between 1 and 10 nanometres. These include single crystals of large organic and inorganic complexes, liquid crystals, polymers, and industrial and biological fibres. An important goal is often to determine the locations of the hydrogen atoms, or interactions with surrounding water molecules through hydrogen-bonding. This information is difficult to obtain from X-ray diffraction.

Although D19 had a reliable and effective detector, its limited size meant that it could capture less than 5 per cent of the scattered neutron beam in one go, making data collection inefficient. The user community therefore supported a proposal to the UK Engineering and Physical Sciences Research Council (EPSRC) to fund an upgrade to D19, comprising primarily a state-of-the-art large-area detector, which promised to speed up data-taking by 25 times, within a set-up that would dramatically increase the instrument's capabilities for a wider range of experiments.

THE UPGRADE

The D19 upgrade had three principal components that offer improved intensity and choice of convenient wavelength, together with faster, higher resolution structure-determination for a wide range of sample structures.

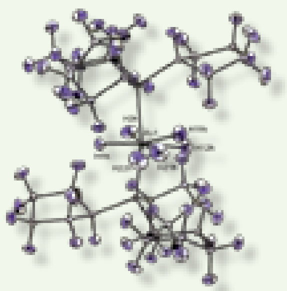
- **A POWERFUL NEW DETECTOR SYSTEM:** a new large horizontally mounted banana-shaped detector was designed and installed, which provides a very large increase in detected solid angle to speed up experiments.
- **A VERSATILE AND FLEXIBLE RANGE OF WAVELENGTHS:** a new monochromator assembly and housing for choosing the wavelength of the incident neutron beam were installed, combined with a completely refurbished optical configuration between monochromator and sample, to suit particular experiments.
- **A FLEXIBLE SAMPLE ENVIRONMENT:** this included a new cooling system, which is extremely useful for biological work and for unstable chemical systems.



NEW SCIENCE

TOWARDS NEW HYDROGEN STORAGE MATERIALS

Heavy metals form a huge variety of complex molecules with organic materials, which often mediate bonding involving hydrogen. They are being studied as possible hydrogen-storage materials. A detailed understanding of the metal-hydrogen interaction requires accurate data on the structures that form, which neutron diffraction can supply. An important compound of this type is the ruthenium complex $RuH_2(H_2)_2(PCy_3)_2$, which has been studied with D19.



THE RUTHENIUM COMPLEX STUDIED WITH D19

UNCOVERING KEVLAR

Kevlar, used for bullet-proof vests and underwater cables, is made of fibres of a highly crystalline polymer. X-ray diffraction had long shown that its remarkable strength and heat resistance result from a structure of stacked sheets held together by a regular arrangement of hydrogen bonds; however, the details of the stacking were uncertain. Now, diffraction patterns recorded on D19 have allowed the arrangement of the Kevlar sheets to be accurately determined in a way that was not possible using X-rays.

PROJECT TEAM:	**V.T. Forsyth**, J.R. Allibon, J. Archer, J.-F. Clergeau, P. Courtois, M. G. Davidson, S. Fuard, B. Guerard, J. A. K. Howard and S. A. Mason
COST:	0.50 M€ plus funding from the UK Engineering and Physical Sciences Research Council (EPSRC)
PROJECT DURATION:	2002-2006

THE D22 DETECTOR

D22

The detector for the D22 SANS (p.6) instrument has been rebuilt to a radically new design, providing up to 50 times the detection rate of the old detector, and so makes measurements much faster. The new detector now allows more efficient measurements, in particular for biological samples, making SANS at the ILL far more accessible to the biological community.

SCIENCE GETS FASTER ON D22

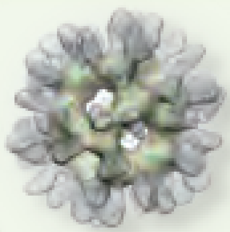
The two ILL SANS instruments D11 (p.15) and D22 are used to obtain information on the scale of larger structures, for example, biological molecules. To gain a complete view of biological form and function requires not only being able to analyse structures up to the 100-nanometre scale, at the smallest angles, but also at smaller scales (below 2 nanometres) and thus wider angles. However, biomolecular materials contain large numbers of hydrogen atoms, which scatter neutrons 'incoherently', that is similarly over all angles, which therefore adds a significant background to neutron measurements. Replacing hydrogen with deuterium mitigates this problem, but it is not always easy to substitute all the targeted hydrogens. To improve the quality of the data obtained over wider angles, a radically new, large-area detector was designed, which is much more sensitive (with a faster count rate) and can 'see' the sample's structure over a range of scales.

Such a detector also enables smaller, more dilute samples to be investigated, as well as allowing kinetic measurements to be made in real time. This is extremely interesting for studies on the processing of complex, soft materials. D22 is also used for studying hard materials such as ceramics and larger-scale structures in magnetic and superconducting materials.

THE UPGRADE

- **FASTER, MORE PRECISE DETECTION:** the new detector consists of an array of 128 individual tubes, each containing a position-sensitive detecting element. It can collect data with high precision and with count rates never previously achieved. The detector can be moved sideways and rotated slightly to measure wider scattering angles. The multi-tube design is cost-effective, more robust and easy to maintain, and is now commercially available. A later, improved design, with the tubes machined from a single block of aluminium, the so-called monoblock detector, has been deployed for the FIGARO reflectometer (p.21), and will be used on other ILL instruments.

NEW SCIENCE



THE MOLECULAR STRUCTURE OF PYRUVATE DEHYDROGENASE

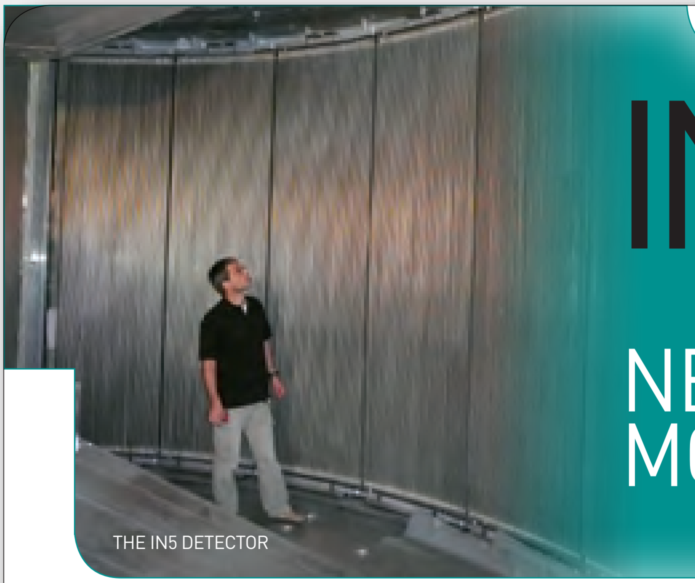
A VITAL ENZYME COMPLEX REVEALS ITS TRUE STRUCTURE

Pyruvate dehydrogenase complex (PDC) is an assembly of enzymes that plays a key role in respiration in humans and other mammals. Until recently, there were two conflicting descriptions of how it is structurally organised. Now, the first ever low-resolution structures of the central core of the complex have been determined by SANS and the X-ray equivalent – and confirmed by electron microscopy – clearly revealing which is the correct model.

UNDERSTANDING BACTERIAL DEFENCES

Restriction-modification enzymes provide bacteria with a defence mechanism against foreign DNA by breaking it up. Using selective deuterium substitution and contrast variation (p.5), SANS on D22 could resolve the structural arrangement and mode of action of these multi-component and multifunctional enzymes.

PROJECT TEAM: **R. P. May**, J. C. Buffet, P. Callow, P. Cross, C. D. Dewhurst, B. Guerard, F. Millier, L. Porcar and P. Van Esch
COST: 1.1 M€ with a contribution from ENEA (the Italian National Agency for New Technologies and Alternative Energies) of 125 k€ to optimise SANS measurements for advanced materials research. PROJECT DURATION: 2000-2004



THE IN5 DETECTOR

IN5

The IN5 spectrometer has now been completely overhauled to give a 60-fold improvement in detection rate. It will enable either the rapid mapping of molecular motion or the probing of excitations in single-crystal samples of materials ranging from exotic magnets to biological compounds.

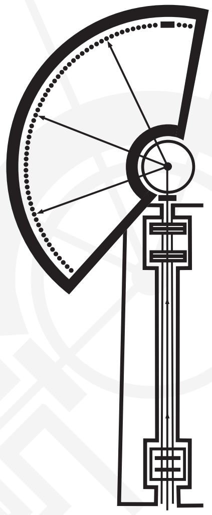
NEW INSIGHTS INTO MOLECULAR MOTION

IN5 is a general-purpose TOF spectrometer for measuring low-energy dynamics in a wide variety of materials – for example, spin fluctuations in magnets and superconductors, and the motions of molecules in soft matter such as gels, polymers and biological structures.

IN5 was one of the first instruments to be built at the ILL and has been operating for nearly 40 years, leading the way in inelastic scattering experiments. It has been continuously improved, with the neutron-delivery and chopper systems recently upgraded; this increased the flux by a factor of 10 to 15, depending on the wavelength. Now, the latest upgrades to the detector system have further improved both the efficiency of the instrument and the quality of the data that can be obtained, thus ensuring that IN5 is once more the world's leading spectrometer in this low-energy range.

THE UPGRADE

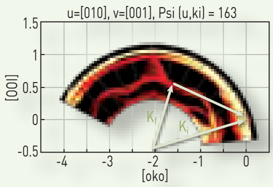
- **A FASTER DETECTOR:** a cylindrical array of pixellated detecting elements, covering 30 square metres, which measure both the energy and position of the scattered neutrons, was designed and built in-house. The aim was to create an instrument with a large seamless detecting surface covering the broadest possible scattering angle. Continuous detection over such a wide angular range opens up a new dimension in measurements on single crystals.
- **A NON-MAGNETIC VACUUM FLIGHT CHAMBER:** the new detector bank required a new 4-metre-radius vacuum flight chamber, which was constructed in aluminium so that experiments requiring high magnetic fields can be carried out.
- **IMPROVED SHIELDING:** another key objective was to reduce the background noise (mainly from stray high-energy neutrons), and this was achieved using a combination of cadmium, high-density polyethylene and elastobore cladding. The new protection has more than halved the background, and when combined with the increased detector coverage, has improved the sensitivity 15-fold.



NEW SCIENCE

MAGNETIC TRIANGLES MAPPED

Langasites are mixed metal oxides with crystal structures that have a wide range of interesting electronic, magnetic and optical properties. The first single-crystal experiment on the upgraded IN5 was carried out on an iron langasite. This compound has a complex magnetic structure resulting from arrays of triangles of weakly coupled iron (Fe^{3+}) ions. The low-temperature magnetic behaviour could be fully mapped within just two and a half days, despite the small size of the sample.



THE IN5 INELASTIC NEUTRON SCATTERING SPECTRUM OF AN IRON LANGASITE SINGLE CRYSTAL

GIANT MOLECULAR SPINS

Molecules consisting of clusters of coupled magnetic ions are of great interest because the electron spins responsible for the magnetism may combine to form a single giant spin. A spectrum was obtained of a chromium copper cluster, $Cr_{12}Cu_2$. The data were collected 50 times faster than with the 'old' IN5, and were much more detailed.

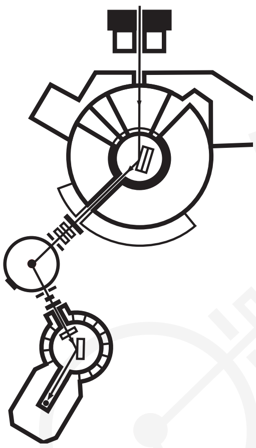
PROJECT TEAM:	**J. Ollivier**, J. C. Buffet, F. Cecillon, L. Didier, B. Grenappin, H. Mutka and J. Nucci
COST:	3.620 k€
PROJECT DURATION:	2004-2008

THE IN20 MONOCHROMATOR

IN20

The ILL's triple-axis spectrometer IN20 has been completely overhauled to increase the much-needed sensitivity by more than an order of magnitude.

UNRAVELLING COMPLEX MAGNETIC BEHAVIOUR



Advanced electronic and magnetic materials are of great technological interest for the next generation of computer memories and processors. They are often based on unusual combinations of structural and electronic or magnetic phenomena. Polarised neutrons (p.7) offer the capability to disentangle the scattering signals from the associated structural and magnetic excitations in these materials, and also to provide valuable information on their possible interactions.

IN20 is the ILL's flagship instrument for carrying out such experiments using polarised neutrons. It is a triple-axis spectrometer (p.7), and is ideally suited for studying electronic and magnetic behaviour, since it can identify even very weak magnetic signals that, in unpolarised neutron experiments, would be hidden by the stronger non-magnetic scattering. In addition to a variety of sample environments, including strong magnetic fields and very low temperatures, IN20 can accommodate the zero-field CRYOPAD device for preparing and analysing polarised neutrons independently, and the TASSE (triple-axis spin-echo) equipment for high-resolution dynamics studies using the neutron spin-echo technique (p.7) at large energy and/or scattering angles.

Experiments on emerging materials of topical interest often involve rather small samples, which also give weak signals, making ever-increasing demands on IN20 in terms of the available neutron flux and energy range, as well as on the quality of the polarisation analysis. The Millennium upgrade has responded to this demand by a complete overhaul of the instrument. This has improved the data collection rate by a factor of 30 to 50, leading to successful studies on single crystals down to 0.05 cubic centimetres in volume.

THE UPGRADE

A more intense neutron beam was required, and a monochromatic focusing geometry to give optimum energy resolution and sensitivity.

- **THE NEUTRON-DELIVERY SYSTEM:** a wider neutron beam tube and beam plug were installed to extract more neutrons from the reactor.
- **MONOCHROMATOR AND ANALYSER:** the focusing optics was completely rebuilt, with newly developed large-area multi-crystal monochromators and analysers with a variable focusing distance.
- **POLARISATION ANALYSIS:** new supports for the spin-guiding coils and for the CRYOPAD device were installed.

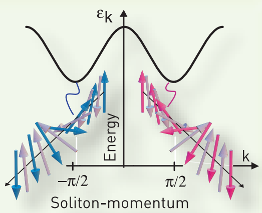
NEW SCIENCE

AN ENTANGLED ANTIFERROMAGNET

Copper formate tetradeuterate provides a textbook model of a two-dimensional quantum magnet. It contains square planar arrays of copper spins interacting antiferromagnetically (antiparallel). The renewed instrument could detect a weak continuum of fluctuations parallel to the partially ordered magnetic moment arising from the exotic 'quantum entanglement' of the copper spins.

QUANTUM MAGNETS DO THE TWIST

In the antiferromagnet caesium cobalt bromide ($CsCoBr_3$), the cobalt spins arrange themselves in a chain-like structure and are held strictly parallel to one axis. Polarised inelastic neutron scattering in a magnetic field proved that an excitation – surprisingly – does not flip the spins all over at once but rather looks like a moving spiral, propagating a single left or right-handed 'twist' (a soliton) continuously through 'forbidden' spin orientations.



THE HANDED 'TWIST' IN SPIN DIRECTION TRAVELLING ALONG ANTIFERROMAGNETIC CHAINS OF CAESIUM COBALT BROMIDE

PROJECT TEAM: **J. Kulda**, P. Courtois, L. Didier, M. Enderle, P. Flores, B. Janousova-Detlefs, J. Saroun, M. Thomas, P. Thomas and J. J. Vernier
COST: 429 k€
PROJECT DURATION: 2000-2003

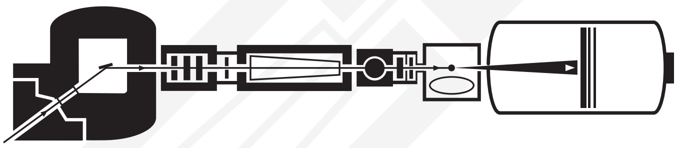
THE SAMPLE CHAMBER OF BRISP

BRISP is a new-concept neutron Brillouin scattering spectrometer that exploits the time-of-flight (TOF) technique, and is optimised to operate at a unique combination of small scattering angles and good energy resolution.

BRISP

EXCITATIONS IN DISORDERED SYSTEMS

The new TOF (p.7) spectrometer BRISP is designed to measure inelastic scattering at small angles. It thereby fills an important gap in the ILL's portfolio of instruments in providing access to the low-energy coordinated dynamics found in many kinds of disordered systems. These embrace liquids and glasses, including metals and alloys, magnetic systems and even ultra-cold systems dominated by quantum effects. It is also suitable for studying aqueous solutions, particularly of large-scale biological structures such as enzyme assemblies and membranes.



INSTRUMENT FEATURES

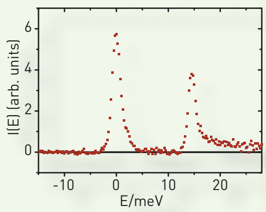
BRISP is a hybrid direct-geometry spectrometer in which the scattered neutrons are analysed by time-of-flight.

- **TWO ALTERNATIVE MULTI-CRYSTAL MONOCHROMATORS** are available to provide neutrons with three different incident energies.
- **A DISC CHOPPER** is used to reduce background neutrons and to help select the required incident wavelength. It is synchronised with the Fermi chopper (below).
- **A SERIES OF HONEYCOMB COLLIMATORS**, depending on the distance to the detector, focuses the beam before it impinges on the sample.
- **A FERMI CHOPPER** provides sharp bursts of neutrons, 10 microseconds long, to enable the TOF analysis.
- **A HIGH-VACUUM SAMPLE CHAMBER** can be equipped with a cryostat or furnace, and a magnet will soon be available for studying magnetic excitations in materials.
- **A HIGH-RESOLUTION DETECTOR** has been installed which is being followed with a movable detector bank to give access to a wider range of scattering angles.
- **A FULLY EVACUATED CONFIGURATION** is available to eliminate scattering from air, with a huge vacuum tank hosting the detector.
- **MASSIVE SHIELDING** protects the detector from background neutron signals.

NEW SCIENCE

QUANTUM FLUIDS REVEALED

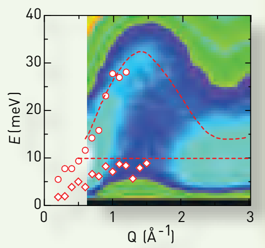
Liquid hydrogen (H_2) provides a model for studying the dynamics of quantum fluids at very low temperatures. BRISP was able to probe the quantum effects on the dynamics of liquid parahydrogen (the hydrogen molecule in which the spins of the two hydrogen nuclei are antiparallel), which is the most relevant model to be investigated both experimentally and theoretically. The observed spectrum of quantum rotational transitions contains the information required for experimental tests of recently proposed theoretical calculations.



THE SCATTERING SIGNALS FROM ORTHOHYDROGEN (NUCLEAR SPINS PARALLEL) AND PARAHYDROGEN (SPINS ANTIPARALLEL) AT 15K

EXPLORING PROTEINS AT WORK

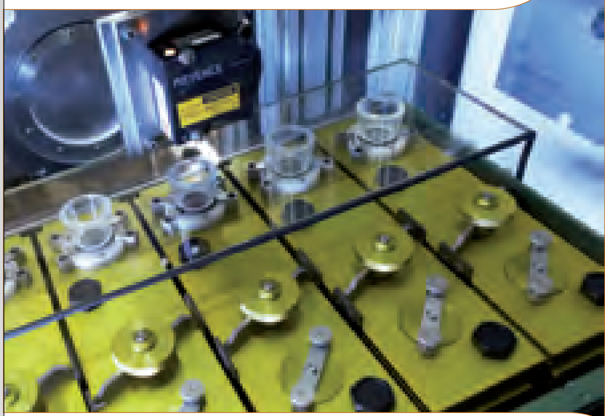
How proteins function in living cells is known to be strongly influenced by the surrounding water molecules which can behave differently from bulk water. By exploiting deuterium substitution and contrast-matching (p.5), BRISP could measure the collective motions of water molecules around the surfaces of proteins, and was used to investigate the hydration shell around ribonuclease – the enzyme that breaks up RNA.



THE COMPLEX DYNAMICS OF RNASE HYDRATION

PROJECT TEAM:	**F. Sacchetti**, S. Aisa, F. Barocchi, A. Cunsolo, A. De Francesco, F. Formisano, E. Guarini, T. Gahl, A. Laloni, A. Orecchini, C. Petrillo, W. C. Pilgrim, D. Russo, L. Sani, J. B. Suck and D. Szubrin
COST:	1.800 k€ and funded by a Italian-German CRG
PROJECT DURATION:	1999-2007

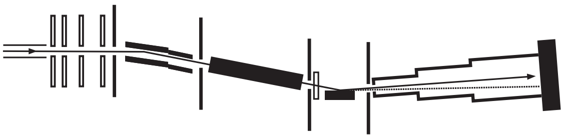
FIGARO'S ADSORPTION TROUGHS DESIGNED FOR NEUTRON REFLECTIVITY MEASUREMENTS OF FREE LIQUIDS



FIGARO

FIGARO is a brand new time-of-flight (TOF) instrument that is optimised for studying horizontal surfaces such as free liquids. It is particularly suited for applications in soft matter and biology.

A NEW HORIZONTAL REFLECTOMETER



The reflection of neutrons off surfaces provides a unique probe of the structure and composition of thin films and interfacial layers, particularly when the process is combined with selective deuteration and contrast-matching (p.5). Typical applications include the study of polymer and soap films, as well as biological membranes and molecules such as proteins that may adsorb at liquid or solid surfaces.

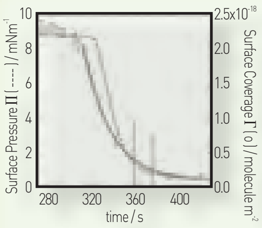
Until recently, all of the ILL's reflectometers were designed to examine only samples in which interfaces were aligned vertically – thus excluding free-liquid measurements. FIGARO (Fluid Interfaces Grazing Angles Reflectometer) works with a high-flux neutron beam that can be directed at a sample from above or below. The 'reflection-up' mode is particularly suited for studying the molecular properties of free liquids, while the 'reflection-down' configuration is useful for probing buried solid/liquid and liquid/liquid interfaces. Because FIGARO is a high-flux TOF instrument (p.7), it can be used to study fast kinetic processes concerning changes in the physical and chemical behaviour of surface materials.

INSTRUMENT FEATURES

FIGARO requires its own, tailor-made neutron guide. The neutrons then pass successively through the following elements of the instrument:

- **TWO FRAME-OVERLAP MIRRORS AND A FOUR-DISC CHOPPER ASSEMBLY**, which control the flux, resolution and wavelength distribution of neutrons transmitted. The configuration can be chosen to maximise either flux or resolution, depending on the experiment.
- **TWO DEFLECTOR MIRRORS AND A COLLIMATION GUIDE**, which provide a beam that can be reflected either up or down from horizontal samples.
- **A VIBRATION-DAMPED SAMPLE STAGE** whose orientation and position can be controlled precisely.
- **A VARIETY OF SAMPLE ENVIRONMENTS** to study different fluid interfaces. They include special troughs for studies of molecular adsorption layers or insoluble surface films, as well as dedicated cells for studies of buried interfaces.
- **A 2-D DETECTOR** drilled out of an aluminium block, designed and constructed at the ILL. It has a novel, patented design that builds on the strengths of the linear position-sensitive detectors developed for the small-angle instrument D22 (p.17).

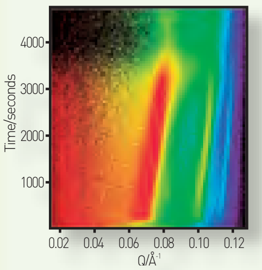
NEW SCIENCE



FAST NEUTRON REFLECTOMETRY CHARACTERISATION OF THE DESTRUCTION OF D-METHYL OLEATE MONOLAYERS BY OZONE USED TO CALCULATE THE RATE OF REACTION

MEASURING CLIMATE-CHANGE FACTORS

The atmospheric oxidation of naturally-occurring organic monolayers on airborne particles affects cloud formation and rainfall. The rate of destruction of the organic films was measured on FIGARO in a project that concerns global-warming factors. The data-acquisition rates achieved were 100 times faster than in previous experiments by the same researchers at other facilities.



THE ORDERED FILM FORMATION ON A POLYMER/ SURFACTANT SOLUTION

POLYMER STUDIES FOR SENSORS AND DRUG DELIVERY

Surfactant (detergents) and polymer films are studied extensively in relation to the slow-release and delivery of active materials in consumer products and in medical treatments. One experiment involved monitoring how a surfactant controls the structural organisation of polymer films on water. The polymers used are sensitive to glucose, and are promising materials for structurally controlled sensors and drug delivery.

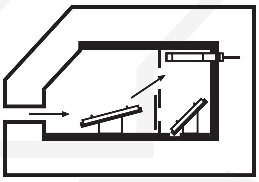
PROJECT TEAM:	**G. Fragneto**, R. Campbell, F. Cecillon, R. Cubitt, M. Jacques, G. Manzin, I. Sutton, H. Wacklin and S. Wood
COST:	€2.82M
PROJECT DURATION:	2005-2009

THE MULTIANALYSER IN THE INCLINED POSITION CORRESPONDING TO THE FLATCONE GEOMETRY

FLATCONE

The FlatCone multi-analyser for ILL's triple-axis spectrometers maps elastic and inelastic scattering over wide ranges of angles and energies, and so provides valuable insights into dynamical fluctuations and nano-scale-ordering processes in single crystals of complex, technologically important materials.

THE ILL'S NEW MULTI-ANALYSER



Traditionally, neutron inelastic scattering experiments on triple-axis spectrometers (TAS, p.7) were used to measure elementary excitations in crystalline solids. Today, these instruments (in particular, IN8, IN14 and IN20, p.19) enable researchers to study quantum fluctuations in layered and chain-like magnetic systems, and also the dynamics of ordering processes where interest is often focused on obtaining rather subtle information about the geometry and interplay between different types of ordering.

The FlatCone multi-analyser has been designed as an optional piece of equipment to be used with the TAS configuration to accelerate data collection in such experiments. It covers a scattering-angle range of 75° and offers a choice between two final neutron energies.

INSTRUMENT FEATURES

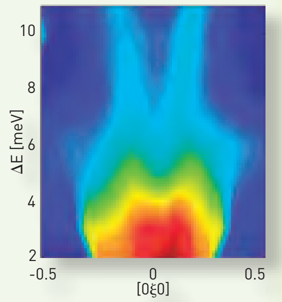
The FlatCone device consists of an array of 31 discrete analyser/detector channels.

- **EACH CHANNEL HAS TWO SILICON CRYSTAL ANALYSERS** tuned to a different final neutron energy; the actual energy is chosen by selecting the corresponding beam-path to the detector.
- **ABSORBING PARTITION WALLS** minimise the cross-talk between neighbouring channels.
- **THE TERM 'FLAT CONE'** refers to a special set-up in which the multi-analyser can be tilted vertically, so that signals can be collected successively over stacks of parallel planes. This allows static and dynamic structures to be investigated in the vertical direction as well as in layers.

NEW SCIENCE

PROBING FERROELECTRICITY

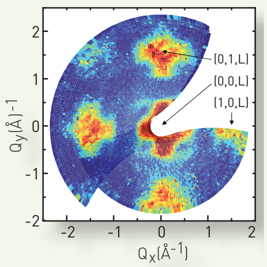
Lead magnesium niobate (PMN or $PbMg_{1/3}Nb_{2/3}O_3$) represents a class of technologically important materials that are strongly polarised by an applied electric field (ferroelectric) or become polarised, generating a large electric field, when mechanical stress is applied (piezoelectric). However, because of their chemical composition they can never attain long-range ferroelectric order. Although they are used in micro-capacitors and mechanical actuators, the exact mechanism of their remarkable properties is uncertain. However, elastic and inelastic neutron measurements at low temperatures were able to reveal the short- and long-range structural and dynamical changes accompanying the development of ferroelectric polarisation.



INELASTIC SCATTERING IN LEAD MAGNESIUM NIOBATE COLLECTED USING THE FLAT-CONE GEOMETRY ON IN20

A NEW TYPE OF SUPERCONDUCTOR

Certain ruthenates such as the strontium compound, Sr_2RuO_4, are unusual superconductors in which the pairs of electrons, known to be responsible for the superconductivity, have spins arranged parallel rather than antiparallel. The details of the complex magnetic (spin) behaviour in the ruthenate, $Ca_{1.8}Sr_{0.2}RuO_4$, were recently investigated using inelastic neutron scattering. Although the compound is not magnetic, the experiments highlighted the strongly correlated magnetic fluctuations. Following their temperature, field and energy-dependence reveals that there are both ferro- and antiferromagnetic interactions competing with each other, which result in a 'metamagnetic' transition in this material. Such experiments help in understanding the various mechanisms responsible for the superconductivity, not only in the ruthenates but also in related superconductors of technological interest.



MAGNETIC SCATTERING IN CALCIUM STRONTIUM RUTHENATE

PROJECT TEAM: **J. Kulda**, F. Descamps, P. Flores, B. Janousova, M. Kempa, F. Lapeyre, A. Perillo-Marcone, S. Roux, J. Saroun, P. Steffens and P. Thomas
COST: 373 k€
PROJECT DURATION: 2002-2007

THE OUTSIDE OF THE LADI-III DIFFRACTOMETER

LADI-III

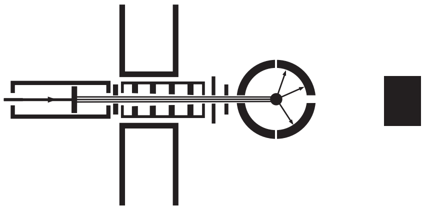
A NEW DIFFRACTOMETER FOR BIOLOGY

The ILL's upgraded neutron Laue diffractometer for biological studies is opening up the way to new insights into the life sciences. The improved detector design allows data to be collected at three times the rate of its predecessor.

INSTRUMENT FEATURES

LADI-III's design is composed of a cylindrical image-plate detector in a vertical configuration with an internal read-out system, but includes further important modifications in design.

- **THE CYLINDRICAL DRUM DETECTOR** was increased in size to improve the quality of the data.
- **THE INTERNAL ELECTRONIC READ-OUT SYSTEM** has been miniaturised to increase the usable detector area.
- **THE INSTRUMENT WAS MODIFIED** so it could be installed on an inclined beam (of up to 2°) and at any beam height from 630 to 1400 millimetres.



Large biological molecules such as proteins are often difficult to crystallise, and so only very small crystals are available for structure determination using diffraction methods. Traditionally, neutron diffraction requires large crystals; however, using the Laue method (p.6), combined with deuteration (p.5), has enabled structural biology to be tackled with neutrons, allowing hydrogen atoms to be located in both macromolecules and the surrounding solvent. Neutron diffraction is thus providing new insights into biological systems and processes, vital for developing medical therapies and solving bio-environmental issues.

In the 1990s, a Laue diffractometer LADI-I, dedicated to studying biological molecules, was built. A key component was a cylindrical image-plate detector that surrounds the sample. Its success led to the development of a second Laue diffractometer VIVALDI (p.26) for the study of smaller molecules, with a similar but superior detector. Based on the VIVALDI design, a new Laue diffractometer for biology was then constructed. Its improved performance allows much smaller crystals to be used, or faster data collection, or larger biological molecules to be studied.

LADI-III is a world-leading instrument and is extremely productive and cost-effective. Because it requires much smaller crystals, both the number and diversity of studies have increased. This trend is set to continue as the instrument will be relocated to a higher-flux beam position on a newly installed neutron guide, permitting use of even smaller crystals and faster data collection.

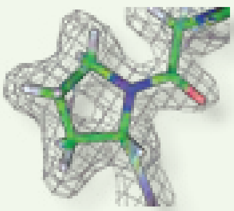
NEW SCIENCE

THE SMALLEST DNA CRYSTAL

THE DNA CRYSTAL USED FOR DATA COLLECTION ON THE LADI-III INSTRUMENT

LADI-III generated the first ever neutron crystal structure of a short section of DNA in a spatial arrangement called the A-conformation, providing details of the positions of water molecules around the structure and the chemical states of the DNA bases. The study was carried out with the smallest crystal (0.06 cubic millimetres) ever used in a neutron study of a biological molecule.

SECRET OF ANTIFREEZE PROTEINS



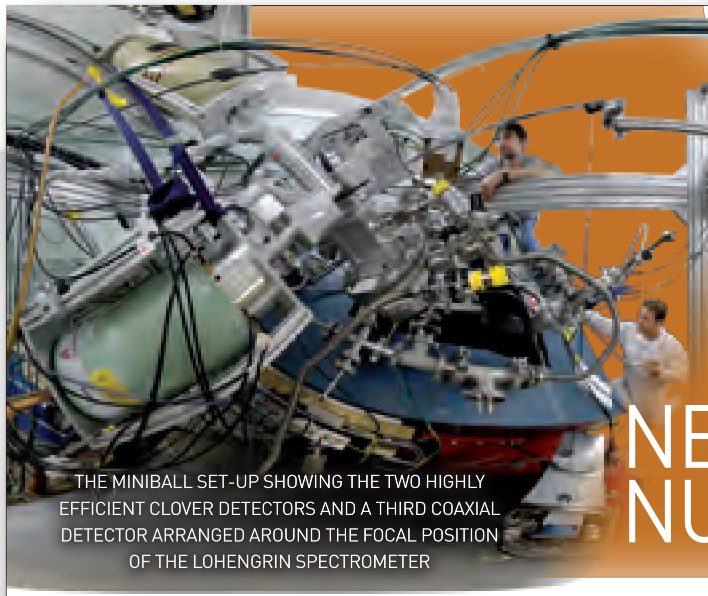
THE FIRST NEUTRON MAP OF AN ANTIFREEZE PROTEIN COMPONENT

Antifreeze proteins stop tiny ice crystals growing in cells, which would otherwise be fatal to organisms living in subzero environments. They are thought to work by binding to these ice crystallites via a flat 'ice-binding surface'. Recent data collected using LADI-III have generated the first neutron crystal structure for one type of antifreeze protein, providing details of the enzyme's structure at the ice-binding surface and the orientation of the water molecules in contact with it.

PROJECT TEAM: **M. P. Blakeley**, G. J. McIntyre, F. Meilleur, P. A. Timmins and C. Wilkinson (University of Durham), together with EMBL Grenoble Outstation partners, F. Cipriani, F. Dauvergne and D. A. A. Myles

COST: 0.793 M€ PROJECT DURATION: 2003-2007

www.ill.eu/about/movies/experiments/pn1-red-magnet

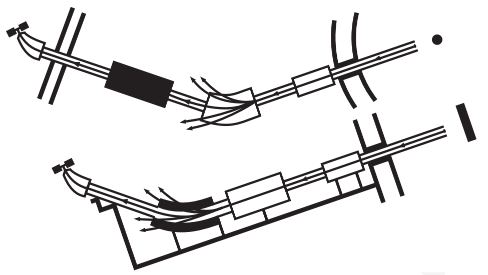


THE MINIBALL SET-UP SHOWING THE TWO HIGHLY EFFICIENT CLOVER DETECTORS AND A THIRD COAXIAL DETECTOR ARRANGED AROUND THE FOCAL POSITION OF THE LOHENGRIN SPECTROMETER

MINIBALL

A new array of germanium detectors has enhanced the potential of the ILL's Lohengrin spectrometer to study the structure of very neutron-rich nuclei.

NEW INSIGHTS INTO NUCLEAR STRUCTURE



INSTRUMENT FEATURES

The aim of the MINIBALL project was three-fold: to increase the detection efficiency, the number of nuclei created, and to measure a wide range of decay processes over time.

- **INCREASE IN DETECTION EFFICIENCY:** two large clover-shaped detectors consisting of single crystals of pure germanium weighing 8 kilograms were installed within an upgraded infrastructure, together with new data-acquisition hardware. This increased the gamma-ray detection efficiency three-fold for single gamma rays and 10-fold for pairs of gamma-rays emitted coincidentally, which happens in certain rare but significant decay modes. Dedicated ionisation chambers were developed for particle-gamma-ray coincidences, particularly useful for detecting certain short-lived nuclear species with a large excess of neutrons.
- **INCREASE OF PARTICLE FLUX:** the spectrometer was modified so that the target temperature could be measured, which enabled the thickness and area of the target to be increased, leading to a five-fold gain in intensity.
- **LONGER DECAY TIMES:** the installation of a second electric field within the magnet focusing the particle beam imposes a time-structure on the beam, ranging from 50 microseconds to many seconds, and opens up the possibility of analysing the lifetimes of longer-lived nuclei, as well as other decay processes involving electrons and neutrons.

The ILL carries out world-leading experiments on the structure of the atomic nucleus. About 7000 different nuclei composed of varying combinations of protons and neutrons could exist, although most of them are unstable. Such nuclei containing high proportions of neutrons are of interest because they can undergo drastic changes in structure and shape, which throws light on how the forces binding nuclei work. Neutron-rich nuclei are also of astrophysical interest because they are the stepping stones in the build-up of elements in stars.

Producing neutron-rich species is difficult, and the ILL is one of the few facilities where large numbers can be generated. The Lohengrin spectrometer contains a target which fissions when hit by neutrons from the ILL's nuclear reactor. The range of neutron-rich nuclei created, usually in high-energy states, are separated and analysed by a detector array that records the characteristic gamma-rays emitted as the nucleus drops to a lower energy state, or decays.

NEW SCIENCE

NEUTRON-RICH ISOTOPES TEST NUCLEAR MODELS

Systematic investigations have been carried out on the excited states and lifetimes of neutron-rich isotopes such as those of tin and zirconium. The neutron-rich tin-132 nucleus, with 50 protons and 82 neutrons, is 'doubly magic', which means its structure consists of full shells of protons and neutrons that are tightly bound. Studying its decay and that of similar isotopes such as zirconium-98 gave data that are useful in evaluating theoretical models of nuclear structure.

THE DECAY SCHEME OF ZIRCONIUM-98 AS OBTAINED ON LOHENGRIN

PROJECT TEAM:	**G. Simpson**, H. Faust, U. Koester, N. Laurens, T. Materna and S. Rowe
COST:	486 k€
PROJECT DURATION:	2000-2004

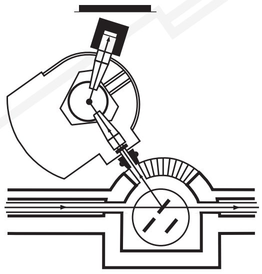
www.ill.eu/salsa

THE SALSA SET-UP

SALSA

The ILL's new dedicated strain imager is designed to determine residual stresses in 'real' engineering components.

TAKING THE STRAIN IN MATERIALS SCIENCE AND ENGINEERING



SALSA is a new diffractometer that enables scientists and engineers to measure internal strain in materials, which is important in evaluating the performance of engineering components and coatings during both their manufacture and use. Diffraction measures interatomic distances and thus non-destructively maps small structural deformations at surfaces or deep inside objects. Neutron strain imaging providing insights that help improve the processing of engineering materials, with applications as diverse as fatigue-resistant welds for aircraft, heat treatments and coatings to toughen the surfaces of aerospace components, and the development of new materials for medical implants.

Although the ILL had conducted stress measurements for many years, it was decided that a dedicated instrument was needed that would act as 'strain imager', able to scan large objects with high resolution and precision, and thus provide a standard test method for determining stress.

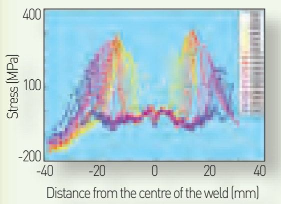
INSTRUMENT FEATURES

A strain imager needs to have a highly flexible configuration, offering a high neutron flux with good resolution, a wide choice of neutron wavelengths, flexible neutron optics to define beam-size, and precise manipulation of specimens between millimetres and metres in size. This is achieved in SALSA with the following features.

- **A BENT PERFECT CRYSTAL MONOCHROMATOR** in combination with a supermirror-coated neutron guide, to provide the intense neutron beam.
- **REMOTELY-CONTROLLED OPTICS** that can be easily altered to scan large samples or surface coatings.
- **A 2-D POSITION-SENSITIVE DETECTOR** with an angular resolution of one-fiftieth of a degree, developed at the ILL.
- **A SIX-AXIS 'HEXAPOD' SAMPLE STAGE** that can support large, heavy samples (up to 1000 kilograms in weight and up to 1.5 metres long) and perform complicated spatial scans with a precision of 10 micrometres.
- **A NEW SUPPORT LABORATORY** – the Facility for Materials Engineering (FaME38) – where the shape of complex objects can be precisely measured. The information is then used in the strain-mapping process. FaME38 also supplies rigs that allow static and dynamic loads to be applied, to simulate various forms of stress.

NEW SCIENCE

A WELDED PLATE SPECIMEN MOUNTED IN THE SALSA STRESS-RIG (ABOVE); EVOLUTION OF THE RESIDUAL STRESS WITH FATIGUE CRACK-GROWTH (BELOW)



STRESS IN AIRCRAFT WINGS

Significant residual stresses are generated in components such as aircraft wings during manufacture, which strongly influence crack growth and fatigue, and hence their safety and service life. By using a dynamic-fatigue loading rig on SALSA, it was possible to track the variation of residual stress with fatigue and correlate this with crack length.

STRONGER COATINGS

Spray coating is often used to repair aircraft components; it is transportable and applicable to nearly any surface. However, good adhesion must be guaranteed even for coatings several millimetres thick. Since residual stresses near the interface play a crucial role for adhesion, the high-resolution set-up of SALSA has been used for this investigation.

PROJECT TEAM: **T. Pirling**, J. Allibon, C. Bouton, A. Bouvier, G. Bruno, J.-C. Collet-Fenetrier, J.-M. Delpierre, R. Gandelli, W. Hutt, M. Jacques, G. Manzin, S. Million, L. Pajou, S. Rowe, P. Thomas and P. Withers

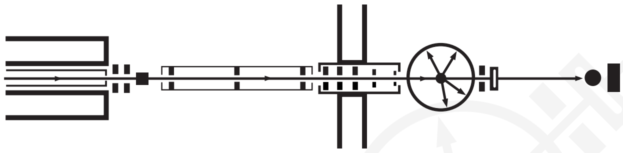
COST: 0.60 M€ plus funding from the UK Engineering and Physical Sciences Research Council (EPSRC) PROJECT DURATION: 2000-2005

www.ill.eu/vivaldi

VIVALDI

VIVALDI is a Laue diffractometer for the rapid investigation of the structure of new materials. It produces spectacular diffraction patterns from large or small molecules with complex structures, even when only very small single crystals are available.

CRYSTALLOGRAPHY IN THE FAST LANE



The Laue technique (p.6) provides a method for obtaining complete diffraction data quickly from one very small crystal, which may be a small inorganic, a metal complex, or a biological molecule. The technique requires a large-area detector to record all the scattered neutrons, and such a detector, cylindrical in shape and based on neutron-sensitive image plates, was designed and first built by the nearby European Molecular Biology Laboratory (EMBL) for the ILL/EMBL Laue diffractometer for protein crystallography, LADI.

Following the success of the LADI detector (p.23), the ILL built a new instrument, designed to analyse the detailed structure of small-molecule crystals in varying environments. VIVALDI (Very Intense Vertical Axis Laue Diffractometer) can make measurements up to 10 to 100 times faster than conventional, single-wavelength diffractometers, or on samples down to 100 times smaller. Complete data collections are possible in just an hour or so, which allows the effects of changes in temperature, magnetic or electric field, or pressure on the structure to be followed.

INSTRUMENT FEATURES

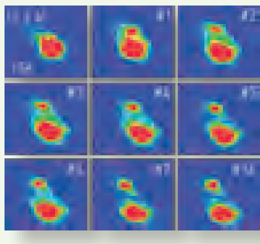
VIVALDI was the first instrument completed in the ILL Millennium Programme.

- **A CYLINDRICAL IMAGE-PLATE DETECTOR** is a key feature. It is first held stationary while the crystal, which lies on the axis of the detector, is irradiated by the neutron beam, and then spun around its axis for on-line readout. The axis of the detector is vertical to allow the use of heavy cryostats and pressure cells, and the irradiated surface of the image plates is read directly for optimum efficiency.
- **A VARIETY OF SAMPLE ENVIRONMENTS** is available. Ancillary equipment includes a dedicated cryostat that offers temperatures from millikelvin to 600K, and can also house high-pressure cells.
- **THE DATA-COLLECTION SPEED** is unparalleled. Since commissioning, the image plates have been upgraded, and some neutron-guide sections have been realigned or replaced by supermirror guides to give a three-fold gain in flux. Overall, there has been nearly a 10-fold gain in efficiency.

NEW SCIENCE

BETTER CATALYSTS

Measuring the angles and distances in a cobalt complex $[H_4Co_4(C_5Me_4Et)_4]$ revealed that the hydrogen atoms form bridges between the central tetrahedral cluster of four cobalt atoms. This result may be significant in understanding and designing better catalysts.



THE LAUE REFLECTIONS RECORDED FOR THE IRON COMPLEX $Fe(ptz)_6(BF_4)_2$ UNDER LIGHT IRRADIATION

SWITCHING STRUCTURE WITH LIGHT

Certain iron complexes have electronic structures that can undergo sudden changes when supplied with a small amount of energy. These are accompanied by signature changes in bond-length and magnetic properties. The complex, $Fe(ptz)_6(BF_4)_2$, where ptz stands for 1-propyltetrazole, undergoes such a transition when irradiated with light. An experiment on VIVALDI enabled a sample to be used that was thin enough for light to penetrate and reveal the induced changes in just a few minutes.

PROJECT TEAM: **G.J McIntyre**, J. R. Allibon, I. S. Anderson, J. A. Cowan, F. Cipriani (EMBL Grenoble), P. Decarpentrie, A. Heidemann, W. Kaiser, S. Pujol, S. Rowe, P. Thomas and C. Wilkinson (University of Durham)

COST: 610 k€ PROJECT DURATION: 1999–2001

NEUTRON GUIDES

Thanks to an innovative and ambitious programme of modernising the neutron guides, beam intensities have been increased by more than two-fold for half of the ILL's instrument suite.

TWICE AS BRIGHT!

The neutron guides allow instruments to be installed away from the nuclear core (20 to 150 metres), where the background is low and more space is available. Neutrons are emitted from the reactor via 14 beam-tubes (H1 to H14), which radiate out and are further split into a series of neutron guides to feed the instruments. The guides consist of a series of evacuated 1 metre-long sections made of optical glass, which transport the neutrons by successive total reflections on their interior, coated walls.

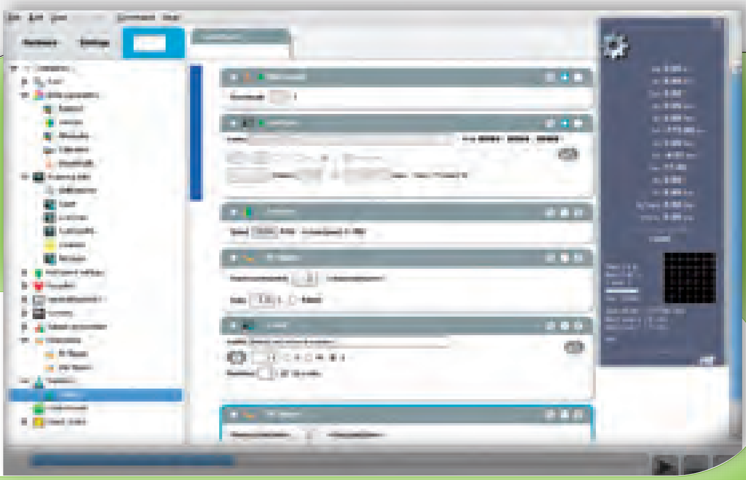
The efficiency of transport of neutron flux depends on the coating reflectivity. Traditionally, the coatings were made of nickel; however in the 1990s, a new supermirror coating was developed, composed of many layers of nickel interleaved with titanium. This has a much higher reflectivity, and can bend, focus and polarise the beam, resulting in more efficient transport. Many of the old neutron guides have now been replaced by these more efficient supermirror components.

THE UPGRADE

- **NEW H171/172 DUAL SUPER-MIRROR GUIDE:** the new horizontal-surface reflectometer FIGARO (p.21) requires a large, flat, white beam, which was made possible after the section of the H1/H2 beam-tube inside the reactor was modified. The guide is curved in both vertical and horizontal directions. The enlarged beam splits into two beams, one of which services two other new instruments, for fundamental physics – GRANIT and Cryo-EDM. The neutron fluxes are now four times higher than achieved with the old H17 guide.
- **NEW DESIGN FOR D11 COLLIMATION:** an innovative guide design was installed in 2007, with an opening 6.5-metre 'trumpet' at the collimation entrance and a focusing trumpet near to the sample position. The flux has been doubled for a wide range of collimation distances.
- **THE NEW H22 SUPERMIRROR GUIDE:** to service two of the new diffractometers, VIVALDI (p.26) and SALSA (p.25), the H22 guide was completely rebuilt using supermirror components. This more than doubled the neutron flux to these instruments and the other two H22 instruments, D1A and D1B.
- **UPGRADE OF THE UPSTREAM SECTION OF H1/H2 GUIDES:** the H1 and H2 beam tubes feed respectively seven cold-neutron guides and five thermal-neutron guides, which provide neutrons for half of the ILL instrument suite. On each line, the first 10 metres were replaced by enlarged supermirror-coated elements, and the 20 following metres were realigned.
- **THE NEW H113 BALLISTIC SUPERMIRROR GUIDE:** neutron losses for a supermirror coating are greater than for a conventional coating, so very long guides have to be shaped, allowing the neutrons to shoot down the guide with minimum reflections – a ballistic supermirror guide. In 2000, the first 72-metre-long ballistic supermirror neutron guide (H113) went into operation, delivering neutrons from the vertical cold-neutron source to the cold polarised-neutron facilitity PF1B used in fundamental-physics experiments. It was later renewed following degradation, leading to an increase of a factor of two in flux.

PROJECT TEAM: **J. Beaucour**, J. Aléo, K. Andersen, D. Bazzoli, E. Courraud, L. Didier, Y. Gibert, R. Gaehler, R. Gandelli, M. Jacques, B. Jarry, M. Kreuz, A. Lechat, P. Y. Meynent, S. Millon, C. Mounier, A. Perillo-Marcone, M. Samuel, J. Tribolet, I. Sutton, P. Suttling, P. Thomas and J. C. Tribbia

COST: 5.6M€ PROJECT DURATION: 2003-2008



THE MAIN PAGE OF THE NOMAD INTERFACE

NOMAD

The ILL has a new software control framework for its instruments that may help users to carry out experiments more efficiently, and will be easier to develop and support.

RETHINKING INSTRUMENT CONTROL

The Millennium Programme has given the ILL a unique opportunity to modernise its computing methodology for controlling instruments. Previously, most instruments were driven by a range of different FORTRAN programs, aggregated under the name MAD. One aim of the project was to provide a consistent approach, applicable to all instruments, that users would find easy to learn and use, and would be easier to support. The programming language C++ has been adopted, since this enables the code to be more modular. It allows users to design the experiment in terms of the relevant physical variables rather than the particular instrument configuration needed – which can be important for non-expert users. Our new software platform for instrument control is named NOMAD.

A DIFFERENT USER INTERFACE: many of the ILL's new users are not neutron experts and may be on site for only a few hours. They therefore require a more intuitive approach to the instruments that does not require a detailed technical knowledge of how they work. The NOMAD graphical user-interface provides a simpler way of setting up measurement processes via the familiar drag-and-drop facility. Moreover, it includes a set of tools that provide a constant report on instrument parameters and the status of the hardware, making decision-taking much easier. A web-based survey system permits the user to verify the behaviour of the experiment remotely.

HOW DOES IT WORK?: as depicted in the figure, the left column of the interface shows all the possible operations, like data-acquisition, wavelength or sample temperature settings. The experiment sequence is set up by dragging and dropping these operations in the work area. More complex structures can be included, and future sequences can be prepared and saved. The user can also test future sequences for errors, simulating all the operations foreseen for the instrument.

A NEW NERVOUS SYSTEM: as with the previous system, NOMAD provides a direct connection between the graphical interface and the instrument electronics. A click on the interface to change the measurement conditions is immediately transmitted to the hardware, providing a faster response time. The instrument's components are controlled via the input of parameters directly relevant to the experiment, such as energy or wavelength, rather than individual instrument settings such as chopper speed or axis angle. This new approach has resulted in a complete review of instrument control methods across the ILL suite, with the provision of detailed documentation, to improve the support and development of all software.

TODAY AND THE FUTURE: today, NOMAD runs reliably on one-third of the ILL's instrument suite. A complete database of drivers to connect and control all possible instrument components has been created, making it easy to incorporate a new piece of equipment for an experiment. A new set of scientific methods has been developed to facilitate the interaction between users and hardware. We have now a solid software platform that allows easy integration of the hardware and rapid exploitation on new instruments.

PROJECT TEAM:	**F. Descamps**, F. Cecillon, S. Count, A. Elaazzouzi, Y. Legoc, J. Locatelli, P. Mutti, H. Ortiz, N. Provin and J. Ratel
COST:	Cost: 506 k€
PROJECT DURATION:	2005-2008

SAMPLE ENVIRONMENT

TAKING EXPERIMENTS TO EXTREMES

The ILL is renewing all its equipment for controlling the environment – temperature, pressure, humidity, electric and magnetic fields – in which an experiment can be carried out.

THE 100-KBAR PARIS-EDINBURGH PRESS, FOR HIGH-PRESSURE STUDIES

A FULLY AUTOMATED AND MUCH SAFER POWER-RACK FOR THE 20 OR SO FURNACES

What are the best neutron instruments in the world worth if the samples they study cannot be taken to the required temperature, pressure or magnetic field? Equipment for controlling the sample environment is ageing, so the ILL has launched an ambitious project to modernise the whole equipment suite.

- **LOW TEMPERATURES:** first, we have changed the thermometry of our cryostats, renewed the old electronics, and started to introduce cryogen-free cryostats. Because users requested more beamtime at very low temperatures, we have also constructed several new dilution refrigerators with incomparable reliability.
- **HIGH TEMPERATURES:** the ILL furnaces, produced under licence by the UK company, AS-Scientific Products, are used at many facilities worldwide, but their power-bays are old and do not meet safety regulations. We have therefore designed and built new controllers, featuring automatic monitoring and control of the primary and secondary pumps, water-cooling circuit and temperature readings. It is difficult enough to take a sample up to 2000K, so can we reach 3000K as requested by our users? The container-less technique being investigated in our laboratories should soon answer that question positively.
- **HIGH PRESSURES**: our users also wish to conduct experiments at pressures up to at least 100 kbar. With a maximum available pressure of 30 kbar, we have not been able to meet this need. We have therefore purchased a 100-kbar Paris-Edinburgh press, and also designed and built a 3K cryogen-free cryostat so that high pressures can be combined with low temperatures. These devices are so popular that a second press was bought and commissioned in 2008. In the very near future, it will also be possible to ramp up the temperature or pressure automatically.
- **MAGNETIC FIELDS:** despite the acquisition of the 15-tesla cryomagnet, the demand for a wider range of magnetic fields on more instruments remains high, and two vertical-field cryomagnets will soon complete the equipment suite: a 10-tesla magnet optimised for triple-axis spectrometers, and a 7-tesla version dedicated to reflectometry and SANS (p.6). Further financial support is being sought to acquire a 12-tesla horizontal-field magnet for SANS and a high-field magnet for the IN5 TOF spectrometer (p.18).
- **MATERIALS HANDLING:** another objective of this project is to provide innovative equipment to handle samples with specific characteristics such as porous and nano-structured materials. Also available are newly-built humidity chambers and gas-injection sticks that fit into some of our cryostats – and, shortly, state-of-the-art gas-handling equipment will be installed, capable of operating with a variety of gases at pressures up to 200 bar.

NEW LIQUID-CRYOGEN LEVEL MONITORS AND COLD-VALVE CONTROLLERS FOR 80-PLUS CRYOSTATS AND CRYOMAGNETS

PROJECT TEAM:	**E. Lelièvre-Berna**, S. Baudoin, N. Belkhier, E. Bourgeat-Lami, J. Gonthier, J. P. Gonzales, J. L. Laborier, O. Losserand, F. Marchal, P. Martin, Y. Memphis, P. Mendes, C. Payre, X. Tonon and S. Turc
COST:	3.5 M€ — PROJECT DURATION: 2007-2013

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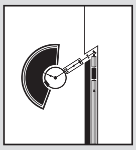
UPGRADED INSTRUMENTS



D2B 12
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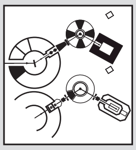

D3 13
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and Anne Stunault
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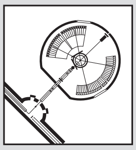

D7 14
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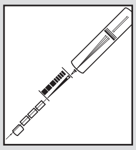

D11 15
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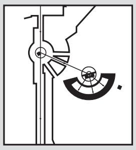

D19 16
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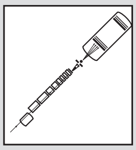

D22 17
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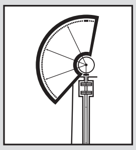

IN5 18
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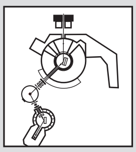

IN20 19
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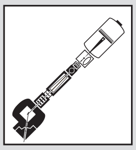
NEW INSTRUMENTS



BRISP 20
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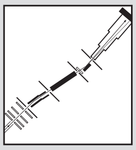

FIGARO 21
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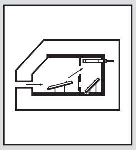

FlatCone 22
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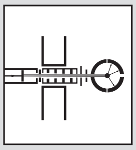

LADI-III 23
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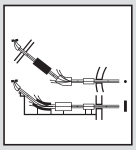

MINIBALL 24
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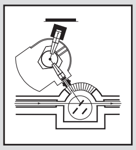

SALSA 25
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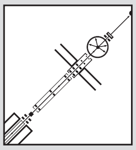

VIVALDI 26
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NEW INFRASTRUCTURE

Neutron guides 27
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NOMAD 28
Paolo Mutti
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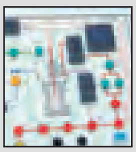

Sample environment 29
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NEUTRONS FOR SCIENCE

ABOUT THE ILL

The Institut Laue-Langevin is an international research centre at the leading edge of neutron science and technology. It is located in a setting of outstanding natural beauty in the cosmopolitan city of Grenoble in south-east France.

The Institute operates the most intense neutron source in the world, feeding neutrons to a suite of 40 high-performance instruments that are constantly upgraded.

As a service institute, the ILL makes its facilities and expertise available to visiting scientists. Every year, about 2000 researchers from more than 30 countries visit the ILL. More than 800 experiments, which have been selected by a scientific review committee, are performed annually.

The scope of the research is very broad, embracing condensed-matter physics, chemistry, biology, materials and earth sciences, engineering, and nuclear and particle physics. Much of it impacts on many of the challenges facing society today – from sustainable sources of energy, improved healthcare and a cleaner environment to new materials for information and computing technology. For example, neutron-scattering experiments have given us new insights into the structure and behaviour of biological and soft-condensed matter, important in designing better drug-delivery systems or improving polymer processing. They also provide a unique probe of the phenomena that underpin high-temperature superconductivity or the molecular magnetism that may provide the technology on which the computers of the future are based.

NEUTRONS FOR EUROPE

The ILL was founded in 1967 as a bi-national enterprise between France and Germany, with the UK joining later, in 1973. As well as these three Associate Member countries, 11 Scientific Member countries now participate in the ILL: Spain, Switzerland, Austria, Italy, the Czech Republic and more recently Sweden, Hungary, Belgium, Slovakia, Denmark and Poland.

NEUTRONS
FOR SCIENCE

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