A journey through ISIS
A behind the scenes look at using neutrons and muons for cutting-edge science
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Welcome to the journey!

ISIS at the STFC Rutherford Appleton Laboratory in Oxfordshire, UK, provides unique sources of both pulsed neutrons and muons for exploring the properties of matter by measuring the locations of atoms and the forces between them.

ISIS employs more than 300 highly-qualified scientists, technicians, engineers and administrative staff, who have unique skills. Their work ensures that the science carried out by research groups is first-class. ISIS teams looking after the accelerator, beamlines, instruments and computers work around the clock to ensure that experiments are successful.

We will follow the journey that one research group makes as they use ISIS to further their research goals – from the initial idea for an experiment, through submitting a proposal, to carrying out the experiment and analysing the results. Along this journey, we go behind the scenes to meet the people who work at ISIS and learn about how the facility operates.
Neutrons and muons for science

ISIS is a world-leading research centre serving the UK and international scientific community

Research carried out at the microscopic scale – whether into the natural world or manmade materials – has been responsible for many of the major improvements in our quality of life.

Sample objects can now be studied with various kinds of radiation to reveal the locations of atoms and the forces between them at the atomic and molecular levels. An increasingly important approach is the use of beams of electrically neutral subatomic particles – neutrons – which can scatter off materials in a way that gives just such information. Neutron scattering techniques are now applied across many scientific disciplines – physics, chemistry, engineering, materials science, environmental and geological sciences, and increasingly the life sciences.

The UK has a major facility, ISIS – based at the Rutherford Appleton Laboratory near Oxford and operated by the Science and Technology Facilities Council – which provides neutron beams for research. Academic research groups from across the UK, and further afield, can apply for ‘beamtime’ at ISIS.

ISIS experiments provide detailed insights into the arrangement and behaviour of atoms and molecules in a material, which are not obtainable by other methods.

What do neutrons tell us?

Neutrons are one of the two constituents, along with protons, of atomic nuclei. Fired into samples, they can tell us where atoms are and how they are moving deep inside materials.

This atom’s-eye view helps us to explain why substances have the properties they do – for example, how they conduct electricity, why they have particular magnetic properties, or how tough they are – and so can help us make new substances that are useful in everyday applications.

When neutrons interact with the atoms in a solid or liquid, they scatter off the nuclei in a characteristic manner that depends on the atomic positions. The angles at which the neutrons emerge from the sample tell us the distances between the atoms. Any changes in the neutron energies as they pass through the samples also reveal the motions of atoms and molecules.

Neutron techniques have distinct benefits:

• Neutron studies can range from the distances between atoms (0.1 nanometres) to those associated with the structures of large molecular arrays (over 500 nanometres).
• Neutrons are penetrating enough to reach deep inside a sample.
• They are non-destructive so can be used on delicate biological samples.
• Neutrons are very sensitive to light atoms such as hydrogen.
• Atoms such as hydrogen, in a selected sample component, can be substituted by a variant with a different number of neutrons in the nucleus – an isotope – which scatters differently and so picks out that component.
• The structure of surfaces and interfaces can be revealed by bouncing neutrons off them – the technique of neutron reflectometry.
• Because neutrons have a magnetic moment, they are sensitive to the often subtle electronic structures of magnetic and superconducting materials.
Neutrons and muons for science

Making a neutron beam

Neutrons can be released by firing bursts of accelerated protons at a metal target, knocking out, or spallating, neutrons from the target’s nuclei. This spallation neutron approach has been extremely successful. It is being taken up elsewhere in the world and will certainly be the basis of any future international neutron facility.

...and muons

As well as producing neutrons, the proton pulses are also directed at a target designed to produce muons, which are another useful probe of the properties of materials (see opposite).

The basic requirement for producing neutrons and muons is a powerful accelerator system to generate an energetic proton beam. ISIS has several accelerator stages, culminating in the circular synchrotron accelerator 50 metres across. This produces protons travelling at 84 per cent of the speed of light.

A wide range of experiments and instruments

Once the neutrons escape from the target, they are slowed down (a process called moderation) and directed along beamlines to some 20 instruments where experiments take place.

Each instrument is designed for a specific type of experiment. For example, large polymer networks are studied using an instrument which measures neutron scattering at small angles (small angle neutron scattering, SANS).

A bright future for ISIS

A second target for neutron production – Target Station 2 – with a suite of new beamlines and instruments, will increase capacity and widen research potential at ISIS. Further novel improvements are being made to increase the intensity of the neutron pulses, so ISIS users can look forward to the prospect of carrying out even more exciting science.

The ISIS pulsed muon source

Muons offer an ingenious way of exploring atomic-scale structure in solids and liquids, providing complementary information to that gleaned from neutron experiments.

Muons are produced from a thin graphite target when the energetic proton beam from the ISIS accelerator passes through. They survive for only two millionths of a second but that is long enough to carry out experiments with them. The muon beams are steered to seven experimental areas, three in the ISIS European muon facility and four in the Japanese-run RIKEN-RAL facility.

The main technique employed using muons relies on the fact that they have a spin and so behave like minute magnets. Muons readily implant themselves in samples, and the magnetic environment around them once inside affects their spins in a way that provides atomic-level information. Muons can be used to study a broad range of materials, including magnetic and superconducting samples, molecular systems and chemical reactions, semiconductors, battery materials, organic materials used in displays – to name but a few.

The RIKEN-RAL facility also carries out studies including muon-catalysed fusion – an exotic mode of nuclear fusion which happens at low temperatures, though not yet efficient enough to provide energy – and other atomic and nuclear investigations.

Neutron experiments can be used to study:

- Advanced materials such as catalysts, hydrogen-storage materials, battery materials
- Nano-materials and structures such as magnetic thin films used in computer applications
- Molecular materials such as pharmaceuticals
- Compounds with exotic magnetic and electronic properties, including high-temperature superconductors
- Engineering stresses and strains in components and welds
- Plastics, detergents, food products and paints
- Proteins, DNA and cell membranes
- Complex biomedical materials
- Disordered materials and liquids
- Rocks and minerals – terrestrial and planetary geology
- Archaeological artefacts
**Exploring drug delivery**

Understanding the basic behaviour of soft matter is one of many research areas that benefit from neutron scattering studies

A growing area of ISIS research is the study of ‘soft matter’ in which weak electrostatic interactions play a significant role in creating complex structures and behaviour. Soft matter includes polymers, surfactants and biochemical materials, and so its study is important in health and biological research.

Peter Griffiths and his research group in the Chemistry Department at Cardiff University investigate soft matter and are frequent ISIS users.

Six years ago, Peter heard a colleague Ruth Duncan give a talk on polymer therapeutics – using long-chain molecules to ferry small drug molecules into cells. Targeted delivery of pharmaceuticals and genes to particular locations in the body is a key area of medical research but is difficult to achieve.

Professor Duncan commented that there was a lack of fundamental information on the three-dimensional arrangements of the polymer chains (their conformation) and how quickly they diffused in biological issues.

Peter realised he had the experience and experimental tools to uncover the information needed.

In collaboration with Cardiff’s School of Pharmacy, Peter started looking at factors affecting how a polymer-bound therapeutic negotiates the complex delivery-pathway from initial injection, or absorption, to the targeted cellular structure. The central issue was how interactions between polymers and the local tissue environment affected their rates of diffusion. A combination of experiments which include small angle neutron scattering (SANS) could provide some answers.

**The power of neutrons**

SANS can probe structures at scales from around 1 nanometre to more than 100 nanometres, which makes it ideal for studying the conformation and behaviour of synthetic polymers, as well as biomaterials.

Neutrons are particularly effective at homing in on the positions of the hydrogen atoms. The locations of surrounding water molecules can also be established, as they have a significant role in modifying the conformation and activity of biomolecules.

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**Now you see it…**

One technique that neutron studies have to offer is that of isotopic substitution combined with ‘contrast matching’, in which specific parts of a complex molecular assembly can be highlighted. This is done by substituting selected hydrogen atoms with deuterium (D), which scatters differently. For example, in a two-component system such as a protein mixed with a polymer in water, a proportion of the aqueous solvent may be substituted with heavy water (D₂O) such that its scattering strength matches that of one of the components. This component is then rendered invisible so that only the other component is seen (see below).

“We make as much use of this approach as we can,” says Peter.

**The mucus barrier**

During the past two years, Peter has been working with one of his PhD students Paola Occhipinti on a particular drug delivery issue.

Organs such as the respiratory, gastrointestinal and reproductive tracts are coated with a layer of mucus, tens of micrometres thick, which may pose a significant barrier to drug delivery agents. In the case of diseases like cystic fibrosis, the mucus layer may be many times thicker.

Mucus is composed of mucin, a gel-like network of glycoproteins, and Peter and Paola were interested in what factors affected how various polymers diffuse through it. Does the structure of mucin change on addition of the polymer? Does the polymer bind to the gel?

SANS carried out on appropriately deuterated samples, together with measurements of viscosity, rates of diffusion, and nuclear magnetic resonance studies, can build up a picture of polymer-mucin interaction.

Recently, they carried out experiments at ISIS investigating the interaction of mucin with a typical model polymer polyethylene glycol (PEG), and also with a dendrimer – a small but highly branched polymer.

The complex structure of mucin, found in the respiratory, gastro-intestinal and reproductive tracts, researchers want to know how drug delivery agents move through it.

**Contrast matching to highlight shell and core components of surfactant particles dissolved in water**

<table>
<thead>
<tr>
<th>No contrast matching</th>
<th>Surfactant shell contrast-matched with a mixture of normal and deuterated solvent</th>
<th>Deuterated surfactant cores contrast-matched with deuterated solvent</th>
</tr>
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<td><img src="image1" alt="Surfactant shell" /></td>
<td><img src="image2" alt="Surfactant core" /></td>
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<tr>
<td><strong>Surfactant core</strong></td>
<td><img src="image1" alt="Surfactant core" /></td>
<td><img src="image3" alt="Deuterated solvent" /></td>
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<tr>
<td><strong>Mixture of normal and deuterated water</strong></td>
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<td><strong>Deuterated water</strong></td>
<td><img src="image3" alt="Deuterated water" /></td>
<td><img src="image5" alt="Deuterated solvent" /></td>
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The genetic disease cystic fibrosis results in increased production of mucin, making drug delivery difficult.
**The ISIS instrument suite**

ISIS has dedicated instruments suitable for a wide range of experiments

ISIS currently has over 30 neutron and muon instruments which provide diverse and complementary information on samples. These include diffractometers designed to analyse atomic-level structures, and reflectometers for studies of surfaces and interfaces. A suite of spectrometers measuring the energies of scattered neutrons provides rich information about atomic motions and magnetic and electronic behaviour in advanced materials.

**Small angle neutron scattering with LOQ**

About 40 different research groups carry out an average of 80 experiments a year on the SANS diffractometer LOQ – of which Peter Griffiths’ team is one.

LOQ is a relatively simple instrument, consisting of a 11-metre evacuated beamline down which the neutrons fly towards the sample. Once scattered by the sample, they hit a fixed two-dimensional detector, 4 metres away, which can detect the positions and times of arrival of the impinging neutrons. “The advantage,” says Richard Heenan, one of the scientists responsible for the instrument, “is that you can probe a large range of distances in the samples in a single measurement.”

**Continuous improvement**

ISIS instruments are continuously being upgraded. This is carried out in partnership with the user community who will always aim to stretch instrument capabilities to their limit.

At the moment, the length-scales available with LOQ do not reach those relevant to understanding the behaviour, for example, of large protein assemblies in cell membranes. The ISIS Second Target Station has been designed to overcome this limitation as well as to greatly increase the number of neutrons produced.

**Target Station 2**

Target Station 2 is ideal for the study of complex materials at longer length-scales. Neutrons are produced by the proton pulses hitting a tungsten target surrounded by novel solid-hydrogen/solid methane moderators which slow the neutrons down in just the right way. Harry Jones, head of the ISIS Target Division, has been responsible for project-managing the construction of TS-2, including all the civil engineering.

“We had to learn how to move a ‘mountain’ to put up the building, as well as designing and building the proton beamline, target station, neutron beamlines and instruments, but it’s gone to budget and schedule,” he says.

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The ISIS instrument suite

SANS2D

The Second Target Station will feed seven new instruments, with more to be added over coming years. One of the first is a new state-of-the-art SANS instrument, designed by Richard Heenan in conjunction with the user community to measure over an even wider range of distance scales. It will provide spectacular gains for researchers studying soft matter, complex materials and biological samples.

"The increased flux and resolution of TS-2 will enable us to study even more challenging systems, in particular to probe the dynamics of self-assembly processes. This is a great and much needed boost for UK science," says Peter Griffiths.

Two 1-metre square detectors can be independently moved inside a 13-metre-long vacuum tank according to experimental needs.

Project engineer, David Turner, turned the scientists’ sketches and specifications into reality, drawing up detailed engineering drawings and arranging manufacture. “One of the technical challenges was maintaining a vacuum between the moving sections of beam collimation in front of the sample. We came up with inflatable vacuum seals, based on those used in aircraft doors, that close up the gaps between the moving parts,” he comments.

Once the design and manufacture were completed, the installation team took over. ISIS engineer Jeremy Moor works on instrument installation. “The layout starts from the location of the sample and the floor is marked out from designer’s drawings”, he says. “We work with the drawing office and ‘heavy gang’ in building a whole beam-line and instrument from scratch.”

Debbie Greenfield, head of the ISIS Instrumentation Division, at the Second Target Station

David Turner from AERE Harwell during the construction of the detector frame for the SANS2d vacuum tank arriving at ISIS

Jeremy Moor did a mechanical apprenticeship at AERE Harwell and has a background, particularly in survey work. He has applied his skills in various ISIS departments, presently focusing on the Japanese RIKEN-RAL muon facility (p.5), particularly gas handling and cryogenics, together with working on the muon production target. Jeremy says there is always a lot to learn. “You can’t help getting interested in the experiments. The scientists are always very appreciative of your efforts and their enthusiasm rubs off on you.”

David Garland from Alstec during the construction of the detector frame for the Second Target Station NIMROD instrument

A journey through ISIS

People spotlight

David Turner came to ISIS four years ago, having spent 10 years at Siemens, which also sponsored him to do an engineering-design degree. Coming from industry, David says he was surprised to find how much technical information was shared with other facilities. “There’s also a lot of direct liaison with the scientists and it’s very much designing in a team,” he says. “What I like most is that everything you design is unique; it makes the job very interesting.”

Jeremy Moor

Debbie Greenfield, head of the ISIS Instrumentation Division, at the Second Target Station

Different routes to take
Submitting a proposal for ISIS beamtime

Once a research team has decided on an ISIS experiment, it is time to put in a proposal.

Access to an ISIS instrument is free to university researchers. Academic groups must first put in a proposal, outlining the scientific case for an experiment, against one of two deadlines a year — in April and October. One of seven Facilities Access Panels, composed of national and international experts, reviews the proposal, after which days may be allocated for the experiment during the next operational run of ISIS.

Key to being awarded beam-time is good preparation of the proposal. Less experienced users should always first seek advice on the experiment’s technical feasibility. Experienced users should always first make contact and find out the experimenters’ opinions. Steve King, who is also a LOQ instrument scientist, advises that they should make contact as soon as possible. “We can then start to flesh out what the researcher really wants to do,” says Steve. “For example, we can advise on decontamination of sample components. We try to help new users as much as possible and will provide feedback on the draft proposal,” he adds.

The ISIS website provides detailed instructions on proposal requirements and the online submission process. In just two sides of A4, researchers must explain why and how they want to carry out the experiment.

John Evans, who chairs the Crystallography Facility Access Panel, says that users must communicate the excitement of the science being done. He stresses the importance of providing the right amount of experimental detail, the number of days needed, as well as evidence of any supporting laboratory work carried out. “Groups who prepare excellent well-argued and clear proposals stand the highest chance of being awarded beam-time,” he notes.

Assessing proposals

The Panels consist of academics, each with expertise in representative scientific areas. Each of the 500 or so proposals submitted in each allocation period is read by members of the relevant Panel. The Panels then meet over a couple of days, during which the scientific excellence of each proposal is discussed.

Andrew Willis (University College London) discussing a crystallography proposal

Usually about 60 days are available on each instrument for each period, and so beamtime is then allotted until the time is filled. In the case of an unsuccessful proposal, the Panels always give feedback on the reasons for rejection. They also aim to achieve a balance across subject areas. Panel members enjoy the scientific interactions of panel meetings and look forward to attending.

The ISIS online proposal system

All proposals have to be submitted via a Web-based system. Guidelines are given on the ISIS website, and users are asked for the number of days required, details of the sample and sample environment, information on previous publications from ISIS work, together with a two-page description giving the science case and experimental details. This approach not only helps the review process but also allows sample-safety issues to be assessed.

This automated proposal system creates a database that will benefit users in the future. Tom Griffin, one of the software developers responsible, has also been working on taking the system further. The aim is to have a fully searchable web-based system that lies all the information regarding an experiment, from proposal stage to completion. At the moment, the system is being road-tested before being made available to the community. “The system has tremendous potential, especially if we could include experimental results,” says Tom.

Other forms of access

• Rapid access: If samples have a short shelf-life or the research is exceptionally high-priority, researchers can choose the ‘rapid access’ route, whereby a proposal can be submitted at any time and reviewed by Panel members, and then scheduled as soon as possible.

• Xpress access: Some instruments including LOQ offer a service whereby a few samples suitable for routine analysis can be sent by courier for measurement.

• Programme access: UK researchers can apply for beamtime to carry out a series of experiments that are part of a research programme funded by a grant.
Preparing for the experiment

Once a proposal has been approved, preparations for the experiment begin in earnest.

Following the recommendations of the Facility Access Panels, the instrument scientists start to plan the schedule for the next ISIS run.

“We try to be flexible and ask users when they would like to come,” says LOQ instrument scientist Steve King. “We may find that people have to delay because samples are taking a long time to make, but if a student is finishing a PhD soon, we will try to accommodate the experiment quickly.”

Sample environment

One of the instrument scientists is also assigned as the local contact for the visiting team, and will discuss the experiment in more detail. Experiments involving measurements over a range of temperatures may require furnaces or cryogenic equipment, while others might need pressure cells or an environment of a particular gas.

Chris Goodway, head of the section supplying pressure, furnace and gas-handling systems, says: “We like to know the requirements before the experiment is scheduled so that we can ensure we have the equipment available.” He and his colleagues also look after the equipment while in use, instructing the users if necessary, and remaining on-call during the experiment.

Sometimes a new piece of kit has to be specially designed and made in the ISIS machine shop. Jim Buckel, who specialises in machining small components, often has to make special sample-holders for users, for example, a narrow cell to contain a liquid. “Scientists tend to provide ‘fag-packet’ drawings of what they want,” he laughs. “One scientist wanted a crystal to be suspended in the beam without anything touching it. Using pictures, we established how to achieve what he wanted.” Jim says he’s machined a vast array of materials from plastic to gold, including preparing samples of “weird and wonderful” alloys used in aircraft wings.

Once a proposal has been approved, preparations for the experiment begin in earnest...

Vicky Kett of the University of Belfast working on protein samples for studies of the effects of freeze-drying on LOQ

Arjan Houtepen of the University of Delft mounting his semiconductor nanocrystal samples for muon studies

University of Bath researchers Adrian Hanley and Ben O’Driscoll in the ISIS sample preparation laboratory during their studies of polymer-surfactant interactions

Steve King

Steve King is one of 70 instrument scientists who help visiting teams to get the best out of their experiments. Their role is to ensure that the beamline is always in “tip-top condition” and to give visitors full support when carrying out the experiments. Like all the instrument scientists, Steve has a PhD, in his case in polymer and colloid science from the University of Bristol. He first came to ISIS as a student in 1986, later joining the ISIS staff in 1999.

Chris Goodway

Chris Goodway has been working at ISIS since 1991, having done an apprenticeship in mechanical engineering at Harwell. After working in the support workshops, he was eventually promoted to technician grade in the sample environment laboratory. “I enjoy the work enormously,” he says. “Each experiment is completely different and we can help the scientists achieve their goals.”

Jim Buckel

Jim Buckel (left) did his apprenticeship at Harwell, like many ISIS technicians, and he has worked in the machine shop for more than 20 years. Jim enjoys the fact that the work is non-repetitive. “I do like a challenge,” he says, and is most proud of a graphite-tile cleaver he designed and made for one of the beamlines.
Getting ready to go

Preparing for the experiment

Sample preparation

Scientists usually prepare their samples in advance of the experiment. For Peter Griffiths research, this might include buying deuterated water and polymers. Many standard deuterated compounds are commercially available but more particular materials need a specialist supplier or in many cases to be made to order.

“We try and do as much sample preparation beforehand as we can,” says Peter. “Usually there’s a period of frenetic activity in the week leading up to the experiment. The polymers may take a few hours to dissolve; we prepare the samples the day before so they are reasonably fresh.”

To obtain some of the mucin samples, Peter and his team had to extract material from the lining of pigs’ stomachs — “not nice material to work with”, he says. Deuterated versions of the polymers were mixed with mucin in deuterated water such that the SANS experiments would highlight the structure of the mucin in the presence and absence of the polymer so as to probe any changes in its structure. The rest of the samples were then prepared on-site using sample-preparation laboratories at ISIS.

The facility has five laboratories for preparing samples. New facilities are being built for TS-2. One lab is dedicated to the needs of biological experiments.

“We can help with biological sample preparation issues including working with users to express and prepare biomolecules in bacteria on a large scale, or just helping out with choosing the right sample cells,” says Cameron Neylon, whose role is to help biologists to carry out neutron experiments successfully.

“We are also available to help with making sure samples are appropriate for neutron scattering experiments. Many biological experiments aren’t as successful as they should be due to issues with sample concentration or purity. SANS in particular requires high concentrations and very good sample homogeneity and we can help to get that right,” Cameron comments.

Sample safety

All samples and sample environments for experiments have to be evaluated for safety, and this is carried out by Steve Roberts, support laboratories manager. “We look at the standard chemical properties such as toxicity, flammability and corrosiveness, and whether special sample-handling equipment is needed,” he says.

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The User Office

At the ‘creature-comfort’ end of preparing for an ISIS experiment are the ISIS User Office staff, who regularly check the experiment scheduling in the online proposal system and send out emails to visiting groups confirming experiment dates and reminding them to send back the COSHH form, and, if necessary, Steve Roberts, who is senior biological safety officer, will make an assessment of the sample and check to see whether special sample-handling equipment is required.

Preparation of the samples is another area that the User Office staff are involved in. The user has to fill in a COSHH (Control of Substances Hazardous to Health) form, and, if necessary, Steve advises on the correct way to handle and transport the material. Another important aspect is radiological safety. A neutron beam can induce radioactivity in samples, and the potential for this is considered.

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A journey through ISIS

People spotlight

Cameron Neylon

Cameron Neylon, who is senior scientist for biomolecular sciences, started as a biochemist at the Australian National University in Canberra studying protein-DNA and enzyme-substrate binding. He realised the potential of neutron scattering to probe these crucial interactions, so took up the challenge offered at ISIS to develop the application of neutron diffraction and spectroscopy in the life sciences. Opportunities in this area will expand as more instruments are built and commissioned on the second Target Station.

Steve Roberts

Steve Roberts trained in applied chemistry at Kingston University, working in both the paint and oil industries before coming to work at ISIS. Like many ISIS support staff, he is on a 24-hour call-out rota. “Sometimes it’s a student who just wants to know how to switch something on, but that’s part of the interest of the job. Working with academics is much more interesting than working in industry. They love talking about their work and you find out about a wide range of things from archaeology to materials for hydrogen storage and solar panels,” he says enthusiastically.
While preparations for experiments are being made, specialist teams of physicists, engineers and technicians work hard to ensure that the production of neutron beams operates reliably at optimum efficiency 24 hours a day, 7 days a week.

Producing neutron beams by the spallation method involves a complex engineering infrastructure to accelerate particles to high energies. Each section of the ISIS machine is looked after by a highly skilled and experienced team.

The process starts with an ion source to produce negative hydrogen ions (two electrons bound to a proton, H⁻) which are injected into a chain of linear accelerators. Then stripped of the electrons by a thin piece of aluminium oxide foil, the bare protons enter a circular accelerator 50 metres across called a synchrotron.

During acceleration they go round 10,000 times before being kicked out of the synchrotron towards the targets where neutrons and muons are produced.

**Ion sourcery**

Originally based on Russian technology, the ISIS ion source works by generating a discharge plasma in hot hydrogen gas fed with caesium vapour. This creates the H⁻ ions which offer the easiest way of producing negative ions for use in the ion source section. The ion sources last for between 20 and 30 days before they have to be replaced.

“Keeping the source in optimum condition is often called ‘sourcery,’” says Dan. “We have to maintain the discharge in chaotic equilibrium to produce as many H⁻ ions as possible. If you switch off the ion source and then start it up again, its behaviour changes. They are fickle beasts, which is what makes the ion-source technicians superstitious. They don’t like to talk about a source because they think it can hear you,” he jokes.

A collection of 10 sources, which are made at ISIS, is kept ready to go. “We are rebuilding a database of operating conditions and failure modes so we can improve reliability,” says Dan.

**Protons on target**

Neutron production also depends upon the target systems being kept in good condition. Although a target lasts up to five years, the surrounding liquid methane moderators (for TS1), which slow down the neutrons, degrade and have to be changed every other run cycle. Because the whole system becomes radioactive, changing the moderator requires a remote-handling cell.

“‘Sourcery’ is only one part of the riddle. Time the cell is changed to the target stations and the accelerator comes to life. ‘Special suits and breathing apparatus are required while we equip the cells with tools and cameras,’ says Dave Haynes of the Targets Section. ‘Changing the moderator is complicated but we’ve done it so many times that it is routine,’ notes Dave.

“We provide on-call 24-hour support for any operational problems,” says Dean. “We establish where the problem is and call out the relevant crew to fix it.”

**Setting up and monitoring the accelerator**

Maintaining the machine performance is essential to providing optimum experimental conditions for users. The job of Dean Adams and his team is to set up the accelerator for each user run. The whole facility works under extremely tight tolerances. The proton beam must remain very stable, which means that, for example, power supplies must remain constant to within 0.01 per cent. Any loss of beam as a result of it hitting the accelerator sides will directly affect the quality of the final experimental beams, as well as damaging the machine.

“‘We aim for at least 90-per-cent availability of the machine for over the 40 to 50 days of user time,’ he adds.

**Diagnostics Section**

The ISIS synchrotron accelerator at the point where the proton beam is extracted to the target stations.

“While preparations for experiments are being made, we are always making sure that the machine is reliable and efficient. We are constantly improving operating conditions, as well as the way we monitor the machine.”

“We are now the leading operators and developers of this type of ion source,” says Dan Faircloth, head of the ion source section. The ion source is constantly being upgraded to cope with the increasing number of experiments being run at ISIS. “I can’t imagine doing anything else. If I had to do it all over again I’d do it again, and probably better,” he says.

“I love the job,” says Tony Kershaw, the 40-year-old experimental halls group leader. “What we do is very interesting and we have a very dedicated and informed team of technicians who work very hard to ensure that we have a highly reliable and effective machine.”

“Since moving to ISIS from the UK’s largest civil nuclear reactor site at Sizewell, I’ve found that the attitude here is ‘can do.’ It is a much more positive and encouraging environment.”

**Sarah Whitehead**

Sarah Whitehead read physics at the University of Manchester and specialised in the software side of beam diagnostics, interpreting data from the monitors.

**Tony Kershaw**

Tony Kershaw (left) has been at ISIS since just after it was commissioned, having done an electronics apprenticeship at Harwell and has worked in the diagnostics section for 16 years.
Staying In control

The routine running of the accelerator is monitored from the ISIS Main Control Room by a duty officer and three assistants working in three daily shifts, 365 days a year.

Duty officer Tom Noone says to maintain the machine at maximum efficiency requires experience and a bit of a ‘black art’. “You know how the machine should run and I might give it a ‘tweak’ from time to time depending on how it’s running,” he says.

One of the team regularly tours the site carrying out routine checks on components such as the moderators. “We are all electronics engineers so if a piece of equipment goes off in the middle of the night, we will attempt a first fix before calling in a specialist,” adds Tom.

Neutrons for the chop

Once the neutron beams are generated, they must be in the right energy range for a particular experiment. This is achieved by using rotating devices called choppers which block unwanted neutrons.

ISIS uses several kinds of chopper, the simplest being a blade which sits just beyond the target and rotates at the same frequency as the neutron pulses. It eliminates high-energy, fast-moving neutrons while letting the bulk of them through. Further down the beamlines are disc choppers with slots that allow neutrons in selected energy bands to pass, whilst other choppers might be set up to allow only every other pulse through.

Tim Carter designs, develops and maintains the chopper drive-systems including new designs for TS-2. “ISIS is leading the way in chopper development. We have dramatically improved their reliability – essential for achieving good experimental results,” he comments.

Scintillating work

Each neutron instrument consists of many components, each of which is looked after and developed by groups within ISIS. For example, ISIS develops its own neutron detector arrays, which comprise mainly gas and scintillating devices. The latter consist of material that emits light when neutrons impinge on it. The light is then transmitted via thousands of optical fibres encoded in a position-sensitive way.

Brian Holland, who has been working on detector designs at ISIS for a decade, has been involved in upgrading the detectors. “Scintillators are a bit of a speciality here; they are massive and require very careful work but are great fun to build,” he enthuses.

Tim Carter

Tim Carter did a degree at the University of Durham and then went on to work on industrial robotics at Culham and Harwell. He joined ISIS five years ago and has been developing a drive that works with all in-house-designed choppers.

Brian Holland

Brian Holland (left) has worked on the site for 33 years. After a Harwell apprenticeship, he worked as an engineer supporting an endurance motor-racing team. “Here at ISIS, it’s also a team effort, which I like,” he says.
On arriving, visitors must first register at the User Office. "The students who come are often nervous and shy especially if they are from overseas. We always smile and try to make them feel at home," says Emma Roberts of the User Office.

**Safety first**

Understanding safety is an important aspect of working at ISIS, so new users have to go through safety-training. This involves viewing an online tutorial explaining, for example, the interlock system of shutters that protects users when accessing experimental areas. They then take a test — a series of short multiple-choice questions — on the use of chemicals, alarm systems, radiation hazards. "They get three chances to pass. We've never had anyone fail — although some have taken the three attempts," says Emma. Each user is given a radiation monitoring badge and a swipe card for secure access to the experiment hall.

The instrument scientist will then meet the visiting team, and take them to the experimental cabin. "If researchers are inexperienced, I usually sit down with them and discuss their samples. We try to give them the largest-diameter neutron beam we can for a sample to get the best statistics," says Steve King. "Students usually come with their supervisors, but if not, we spend time running through the experimental set-up and explaining the control software."

Instrument scientists provide all the scientific support that the visiting team needs in order to get the best from their experiment.

The SANS experiment being carried out by Peter Griffiths and Paola Occhipinti required two days, with each of the 40 samples taking about an hour to run on the LOQ instrument. An automated sample-changer allowed the researchers to plan the programme and leave it running for 12 hours. While the first set of samples was running, the next day's samples could be prepared.

"I used the ISIS laboratory facilities quite a lot," explains Paola. "It's nice because you meet lots of people and can exchange ideas."

Experiments are carried out over the full 24 hours to make full use of ISIS. The ISIS support staff for the instrument, sample environment, accelerator and beam-line are always available at the end of a telephone.

"It's quite intensive and tiring; you don't even know whether it's night or day," Paola comments. "It's a very high pressure environment for a student," agrees Peter. "It's a chapter of the PhD thesis so the experiment has got to work."

Emma Roberts has worked in the ISIS User office for eight years looking after the needs of visiting researchers. "I make sure that they know that they can come to us with any query, no matter how unusual. For example, we had someone who developed really bad toothache and we were able to direct him to a dentist."

Supriyo Ganguly and Dimitrios Bakalis (University of Manchester) using ENGIN-X for examination of residual stress around welds in an aircraft wing spar specimen.
Running the experiment

Collecting the data

Once the experiment starts, data are collected using specialist hardware and become available for the researchers to examine. The arrival of each neutron at one of the thousands of detecting elements comprising the detector area must be "time-stamped". These events are counted and this raw data is transferred to the instrument's dedicated computer.

Freddie Akeroyd is one of the computing group who maintain these computers and provide software to control the data capture. "Settings for a variety of parameters, such as sample temperature, can be specified in command files for overnight running," says Freddie.

"The software also allows you to look at the data live as it is collected, so you can quickly check if the instrument is working properly."

In a typical SANS experiment, the raw data can be reduced to meaningful results immediately using a "tried and tested" computer program that has been developed over the years. Instrument scientists such as Richard Heenan and Steve King are on hand to give advice. "If necessary I'll log on to the data at home in the evening and give the users a phone call. Sometimes I'll come back in and we will go through the data-reduction process," says Richard.

The experiment gives a two-dimensional pattern of the scattered neutron intensity which can then be manipulated in a number of ways. In the case of Peter and Paola’s experiments, a plot is derived which reveals the required information about polymer conformation.

"We obtain just enough information to make any necessary adjustments to the next set of samples," says Peter. When the final datasets are ready, they can be put onto a CD and taken back to the user's laboratory for further analysis. A copy of the data is also archived at ISIS.

Experiment control

Users need to be able to control their experiments easily, and this can be done remotely using a computer interface. This is where Kathryn Baker of the ISIS Computing Group steps in. She and her colleagues develop and monitor the software for manipulating all aspects of the experiment, including temperature, and movement of the samples and equipment. Users are presented with a visual display generated with a graphical programming language called LabVIEW. "It's all 'drag and drop' with a mouse," explains Kathryn.

Sample safety

An important practicality of neutron scattering is to check the sample for radioactivity once the experiment has finished. Neutrons can activate samples through nuclear reactions. If the dose rate is above a predetermined level, a member of the ISIS health physics section collects the sample for storage in shielded locations.

"Activated samples usually decay rapidly," says Paul Wright who is the ISIS health physicist and radiation protection officer. After a few days, the sample can normally be released for return to the user's laboratory.

People spotlight

Freddie Akeroyd

Freddie Akeroyd joined the Facility 15 years ago after completing a PhD in theoretical chemistry. He says this involved "scattering theory and a great deal of computing", so providing an ideal background for working on data acquisition and software development at ISIS.

Kathryn Baker

Kathryn Baker did a sandwich placement at ISIS while a student at the University of Glamorgan studying computer systems engineering. She says she enjoyed the academic atmosphere, where "science is more important than money", and after a few years of working in commerce, was "more than happy" to come back when a job became available.

Paul Wright

Paul Wright (left), a nuclear physicist by training, advises the Rutherford Appleton Laboratory on all aspects of radiation hazard management including safety protocols for maintenance of the facility, shielding, samples, and handling of radioactive equipment, as well as protection of users and staff. "It is extremely important that regulations are followed and the protocols are fit for purpose to minimise risks to facility staff and visitors," says Paul.

Philip Cornier (Mount Allison University, Canada) using muons on EMU to probe radiation chemistry in ionic liquids

Toby Perring (ISIS) and Andrew Walters (UCL/ISIS) reviewing their MAPS data of high-energy spin excitations in iron

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Toby Perring (ISIS) and Andrew Walters (UCL/ISIS) reviewing their MAPS data of high-energy spin excitations in iron
After the experiment

Once the experiment has been run, the task of analysing and interpreting the data can begin.

To uncover how the polymers interact with mucin, Peter and Paola must look at their data in detail. As with all other stages of an experiment, their ISIS local contact can always be called upon to help with the analysis and interpretation.

So what did the SANS experiments reveal? The PEG polymer does not show any interaction with the mucin and diffuses through quite easily, while the smaller dendrimer, which has a positive charge depending on the pH, interacts electrostatically with the negatively-charge gel, so its movement is retarded. “The scattering indicates that the mucin structure collapses in the presence of the polymer,” says Paola.

The Cardiff team has already put in a proposal for the next neutron experiment. “The results always give you a new idea,” says Peter. “We want to look at interactions with other uncharged polymers such as dextrin, and a negatively charged dendrimer, and also in the presence of agents that chemically break down mucin, such as N-acetylcysteine.”

“Small-angle neutron scattering from 5-per-cent-weight mucin solutions as a function of pH in the presence and absence of synthetic polymers, as models for polymer therapeutics.”

“Once the experiment has been run, the task of analysing and interpreting the data can begin.”

Peter and Paola are now preparing their results for publication. Experiments performed at ISIS result in around 500 publications each year, as well as conference papers, posters and seminars. Users also have to send in a report giving experimental details and its outcome – the Facility Access Panels use these reports to assess previous experiments when users apply for further beam-time.

“The scattering indicates that the mucin structure collapses in the presence of the polymer,” says Paola.

“The results always give you a new idea,” says Peter.
Training for ISIS users and staff

ISIS runs various courses and schemes to ensure that users get the most from a facility supported by staff of the highest quality.

Learning how to use neutron and muon methods

ISIS runs regular training courses for postgraduate and postdoctoral researchers from the UK and EU in both neutron scattering and muon techniques. The aim is to give them the necessary grounding to carry out experiments at ISIS successfully.

The five-day courses include lectures on theory, safety and radiation protection, the ISIS accelerator and targets, the instruments and the techniques used, combined with hands-on activities. The ISIS website also has extensive information – manuals, lectures and hand-outs – which supplements the courses.

Employment

As part of the Rutherford Appleton Laboratory, ISIS participates in several training schemes supported by the STFC. These offer a unique opportunity to young people at all stages of education to build up engineering, technical and computing skills to the highest level.

• Graduate training scheme

Graduates with good degrees in mechanical, electrical and electronic engineering are eligible for the Graduate Mechanical Engineer and Graduate Electrical and Electronics Engineer Training Schemes. These are four-year programmes involving a mix of formal and tailored training, placements and project work.

• One-year sandwich student placement

Students can apply for specific one-year placements at ISIS as part of their university courses. These offer an excellent opportunity to build skills and experience in areas such as software or detector development and to get a inside view of what it is like to work in a facility relying on cutting-edge technology.

• Vacation placement

Students can also apply to work at the Laboratory during vacations for between 6 and 12 weeks.

• Engineering Advanced Apprenticeship Scheme

School-leavers and others with five grade-C GCSE passes including English, mathematics and science, can apply for an apprenticeship in electrical, electronic or mechanical engineering, which provides an ideal start to career in the advanced technology sector. The apprenticeship, which lasts four years, is considered one of the best in the country. Once qualified, an apprentice may stay on at the Laboratory as a technician or may embark on a full or part-time university degree course.

• Ongoing Learning

ISIS provides numerous opportunities for the research community to discuss results, learn about new techniques and shape future ISIS instrument developments. These include regular user meetings, science-based workshops and conferences run by facility staff alongside community scientists.

School trips are an important way to encourage the next generation of scientists. Here we see students from Blue Coat School in Reading touring the ISIS experimental hall.

PhD students from the Max Planck Institute, Germany, during their visit to ISIS.

ISIS scientists Sean Giblin and Iain McKenzie loading a cryostat with students on the ISIS muon training course.

Helping others to travel
ISIS Scientists

ISIS scientists provide a huge range of expertise in the application of neutron scattering and muon spectroscopy to condensed matter and molecular science. They can be contacted to discuss how these techniques might be useful in research programmes, and to give advice on facility capabilities and proposal writing. They are listed here by science area, to give an indication of particular research interests.

All ISIS scientists can be contacted by email using first_name.second_name@stfc.ac.uk

Materials

- Hydrogen storage, hydride materials: Timmy Ramirez-Cuesta, Bill David, Steve Bennington
- Proton mobility in fuel cell materials: Felix Fernandez-Alonso
- Ionic conductors using muons: James Loiz, Steve Cotterill
- Carbon for hydrogen storage and other applications: Steve Bennington
- Hydrogen behaviour in semiconductors using muons: Philip King
- Nano-structured materials: Felix Fernandez-Alonso, Steve King, Sean Langridge
- Superionics, piezoelectrics, ferroelectrics, particularly order-disorder: Steve Hull, Dave Keen
- Multiferroics: Laurent Chapon
- Dielectric ceramics: Richard Ibbonson
- Structure of phase separated alloys and porous materials: Richard Heenan
- Structure of glassy materials and disordered crystals: Alex Hannon, Emma Barney
- Dynamics in disordered materials: Steve Bennington, Jon Taylor
- Powder diffraction and structure solution with X-rays and neutrons: Bill David
- Single crystal diffraction: Matthias Gutmann
- Atomic momentum distributions, particularly applied to helium: Jerry Mayers, Mark Adams
- Hydrogen bonding: John Tomkinson
- Vibrational spectroscopy of small molecules and oligomers: John Tomkinson, Stewart Parker
- Environmental chemistry: Steve King
- Electrochemistry: Rob Dalgliesh
- Fibres: Steve King

Chemistry

- Catalysis: Stewart Parker
- Radical chemistry applications of muon spectroscopy: Iain McKenzie
- Microemulsions and micelles: Richard Heenan, Sarah Rogers
- Structure and dynamics of liquids and solutions, including complex liquids and confined fluids: Alan Soper, Daniel Bowron, Silvia Imberti, Felix Fernandez-Alonso, Franz Demmel
- Structure refinement in liquids, complex fluids, confined fluids, and disordered solids: Alan Soper, Daniel Bowron, Silvia Imberti, Rowan Hargreaves
- Hydrogen bonding: John Tomkinson
- Vibrational spectroscopy of small molecules and oligomers: John Tomkinson, Stewart Parker
- Environmental chemistry: Steve King
- Electrochemistry: Rob Dalgliesh
- Fibres: Steve King

Geology, Engineering, Archaeometry

- Mineral physics with relevance to terrestrial and planetary geology: Kevin Knight
- Residual stresses, welding and structural integrity: Anna Paradouwska
- Archaeometry: Winfried Kockelmann
- Radiations effects in electronics: Chris Frost

Magnetism and superconductivity

- Magnetic structure determination: Laurent Chapon, Aziz Daoud-Aladine
- Lattice dynamics, phase changes: Chris Stock
- The role of magnetism in high temperature superconductivity: Toby Perring
- Superconductor properties using neutrons and muons: Adrian Hillier
- Heavy Fermions and quantum phase transitions: Devashibhai Adroja, Chris Stock
- Molecular magnetism and superconductivity studied by muons: Francis Pratt, Peter Baker
- Colossal magneto-resistance: Toby Perring
- Nanoscale electronic phenomena (with both neutrons and X-rays): Christian Kinane, Timothy Chariton, Sean Langridge
- Frustrated magnetism: Laurent Chapon, Ross Stewart
- Magnetic and electronic properties studied with muons and neutrons: Sean Giblin
- Relaxation in localized and itinerant moment systems: Mark Telling
- Magnetic and electronic properties studied with neutrons and X-rays: Jon Taylor

Theory and techniques

- Theory of neutron scattering, density functional theory: Nikitas Gidopoulos
- Theory of strongly correlated quantum matter: Jorge Quintanilla
- Density functional theory: Keith Refson
- Polarised gasses: Chris Frost, Ross Stewart
- Monte Carlo neutron transport calculations: Stuart Arseil