

# Neutrons for Energy Materials at the ISIS Facility

Victoria García Sakai

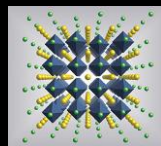
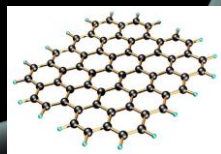
[victoria.garcia-sakai@stfc.ac.uk](mailto:victoria.garcia-sakai@stfc.ac.uk)

[www.isis.stfc.ac.uk](http://www.isis.stfc.ac.uk)

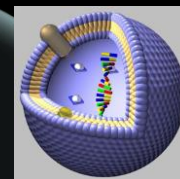


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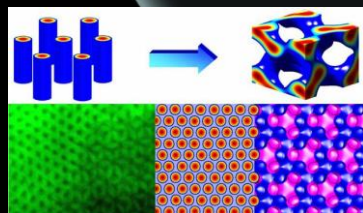
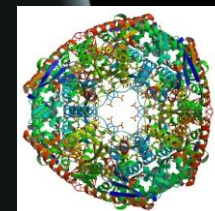


Energy

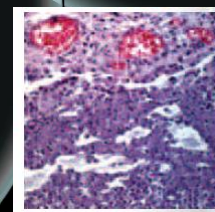


Digital  
Economy

Health



Curiosity Driven  
Research



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# The Nobel Prize in Physics 1994



Clifford G. Shull, MIT, Cambridge, Massachusetts, USA, winner one half of the 1994 Nobel Prize in Physics for development of the neutron diffraction technique.



**S** Shull made use of elastic scattering, i.e. of neutrons which change direction without losing energy when they collide with atoms. Because of the wave nature of neutrons, a diffraction pattern can be recorded which indicates where in the sample the atoms are located. Even the placing of light electrons such as hydrogen in molecular hydroides, or hydrogen, carbon and nitrogen in organic substances can be determined. The pattern also shows how atomic dipoles are oriented in magnetic materials, since neutrons are affected by magnetic forces. Shull also made use of this phenomenon in his neutron diffraction technique.



Diagram illustrating the principle of neutron diffraction.

## Neutrons see more than X-rays

It may be argued that neutrons are not as penetrating as X-rays. Well, it may be so, but neutrons are also not as penetrating as X-rays. But this is not a disadvantage, as it is not in case of X-ray beams, all kinds of atoms are visible. In a neutron diffraction experiment, the neutrons penetrate deep into the sample and scatter from the nuclei of the atoms. This means that neutrons can see through light elements such as hydrogen, which is often invisible to X-rays. Neutrons also interact with magnetic moments, allowing the study of magnetic structures.

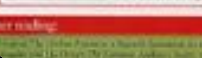


Diagram illustrating the principle of neutron diffraction.

## Neutrons reveal inner stresses

It calls for imagination to see a neutron as a tiny ball. But it is not a ball, it is a wave. And it is a wave that can be diffracted. When a neutron beam passes through a crystal, the neutrons are scattered by the nuclei of the atoms. This scattering is affected by the internal stresses in the crystal, which can be measured by analyzing the diffraction pattern. Neutrons are particularly useful for studying stresses in materials because they can penetrate deep into the sample and interact with the nuclei of the atoms.



Diagram illustrating the principle of neutron diffraction.

## Neutrons show what atoms remember

At first sight, it seems that neutrons are just a tool for studying the structure of materials. But they are much more. Neutrons can also be used to study the dynamics of atoms. By measuring the inelastic scattering of neutrons, researchers can determine the energy levels of atoms and the way they change over time. This is particularly useful for studying the behavior of atoms in liquids and solids, where they are constantly moving and interacting with each other.



Diagram illustrating the principle of neutron diffraction.

# Neutrons reveal structure and dynamics

Neutrons behave as particles and as waves

Neutrons show where atoms are



Neutrons bounce against atomic nuclei. They also react to the magnetism of the atoms.

Neutrons show what atoms do



**B**



Bertil H. Brattstrom, McGill University, Montreal, Canada, winner one half of the 1994 Nobel Prize in Physics for the development of neutron spectroscopy.

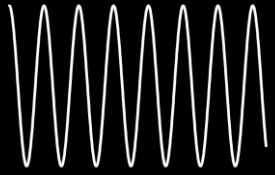
Brattstrom made use of inelastic scattering, i.e. of neutrons, which change both direction and energy when they collide with atoms. They then start on circular orbits in crystals and record neutrons in liquids and solids. Neutrons can also interact with spin waves in magnets. With his 3-axis spectrometer Brattstrom measured energies of phonons (atomic vibrations) and magnons (magnetic waves). He also studied how atomic vibrations in liquids change with time.

Changes in the energy of the neutrons are first analysed in an analyser in front of the detector.



# Neutrons tell us where atoms are and what atoms do



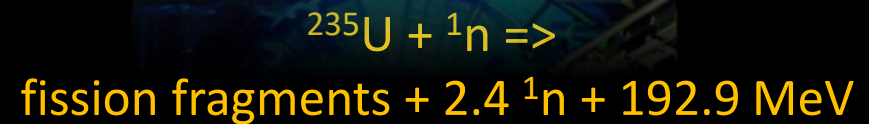
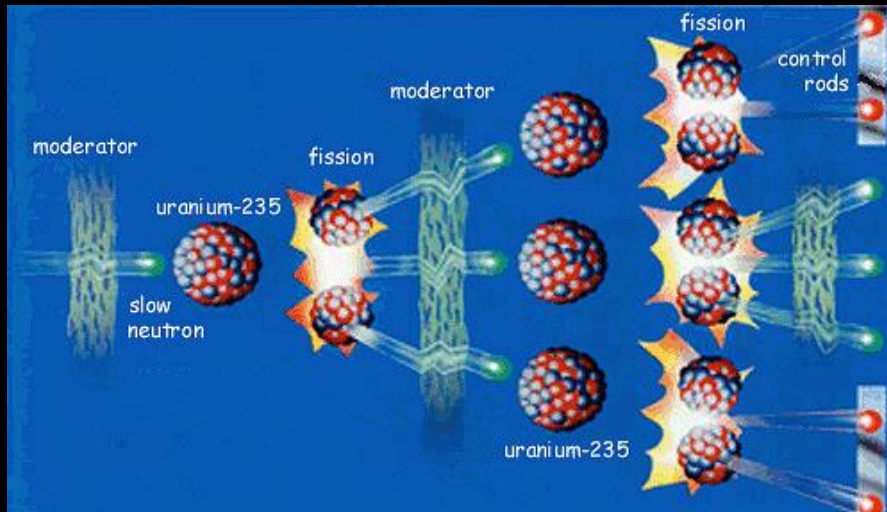


# Neutrons



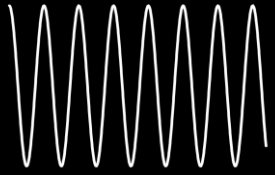
- Uncharged, subatomic particles found in atomic nuclei
- Approx. mass of proton,  $v = 2.2$  km/s at RT
- Wave-particle duality,  $\lambda = 0.18$  nm at RT

## Fission in a nuclear reactor (ILL)



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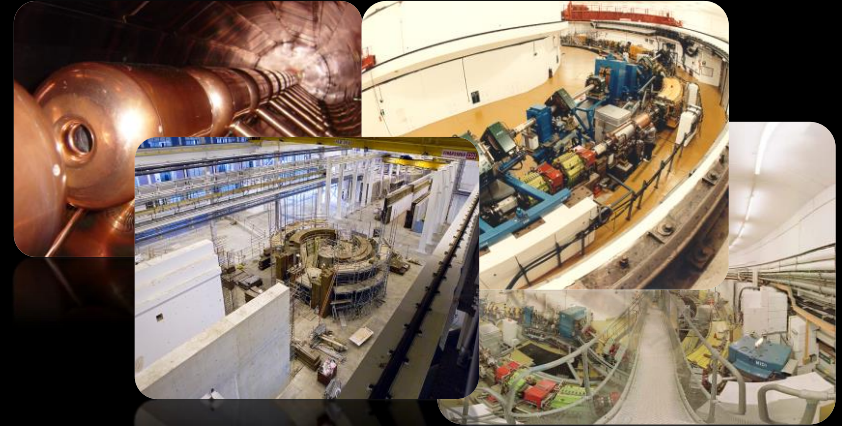
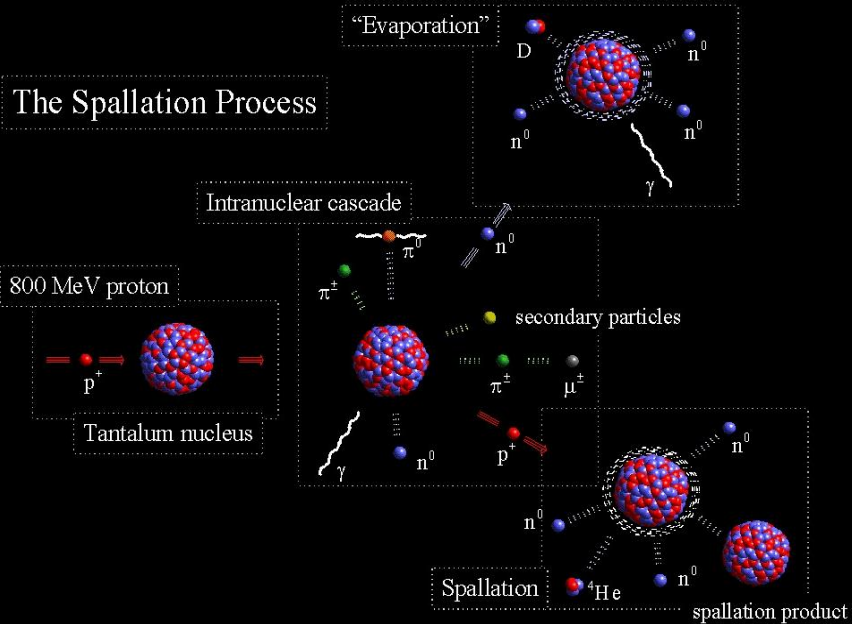
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# Neutrons



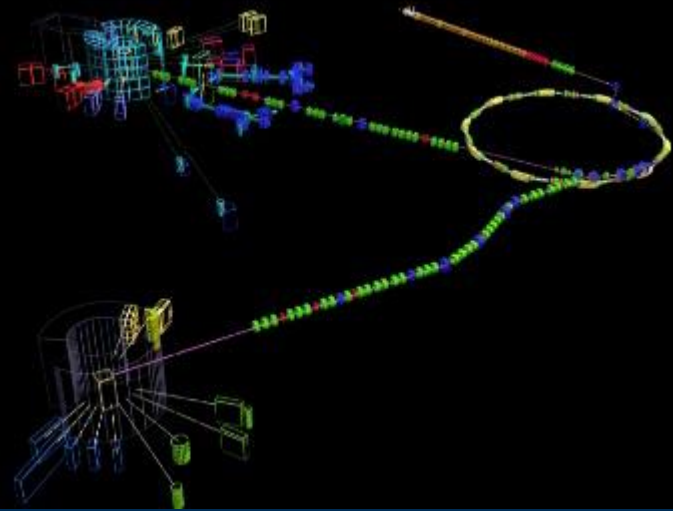
## Spallation (ISIS)



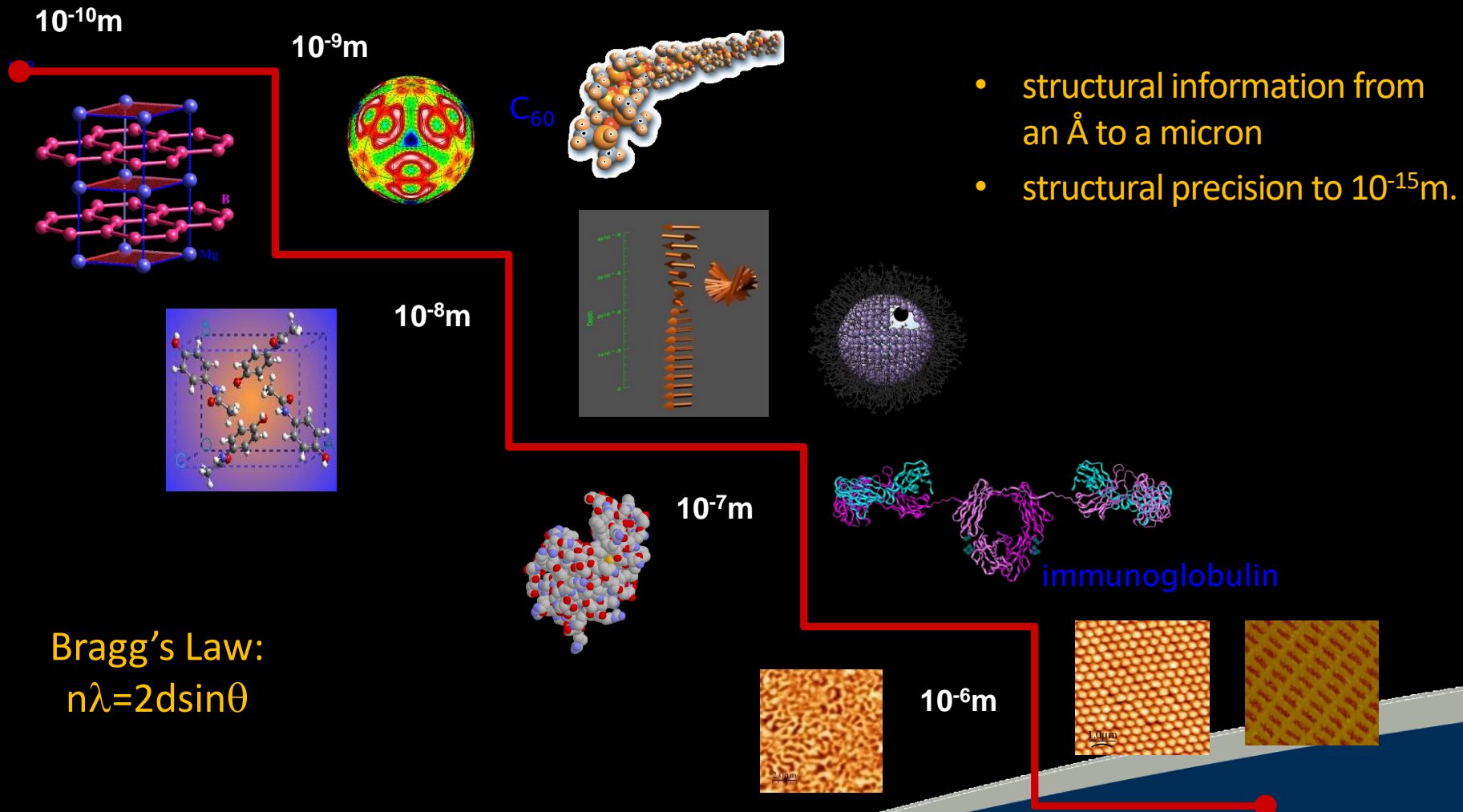
~ 16n/collision + ~20 MeV/n



EUROPEAN  
SPALLATION  
SOURCE



# Neutron wavelengths are of the same order as atomic spacings



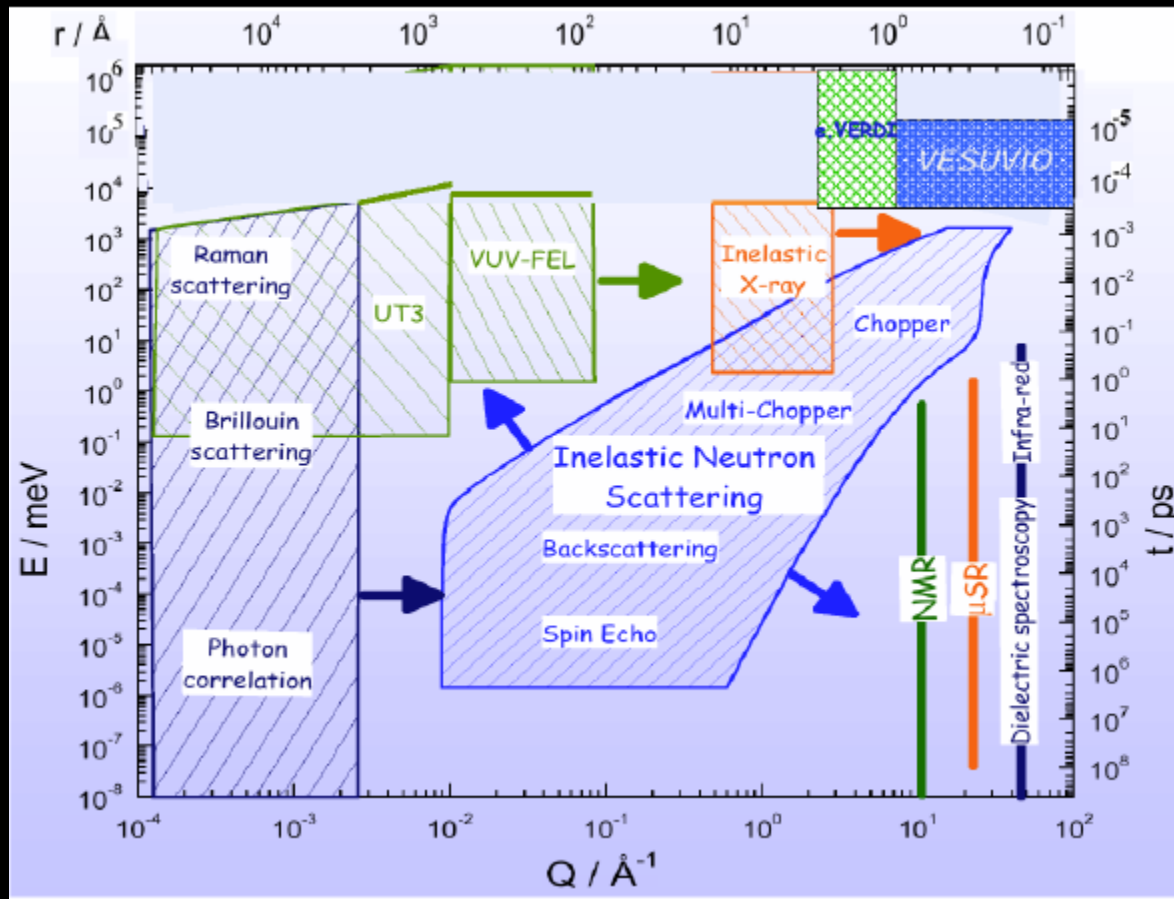
Property 1



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# Neutron energies are of the order of elementary excitations and molecular modes



- Vibrations
- Lattice modes
- Diffusion
- Rotations
- Bending and breathing modes

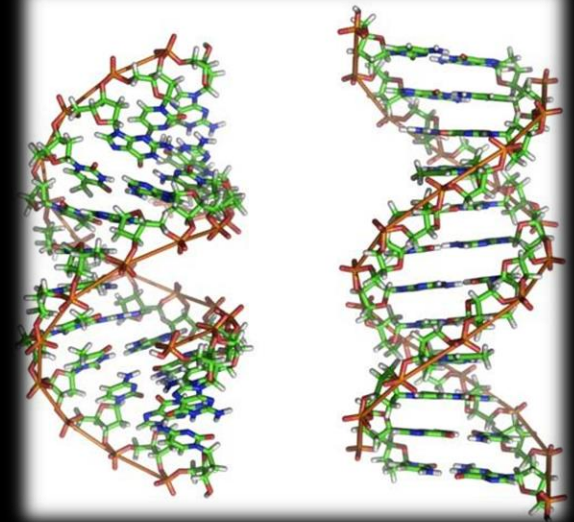
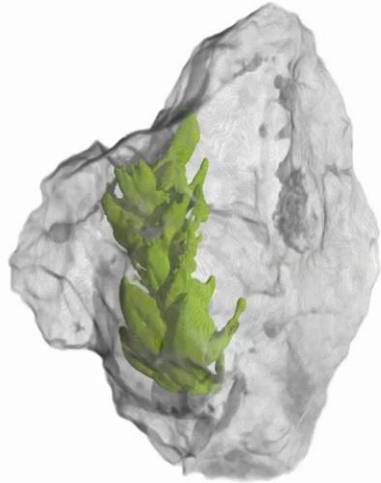
...from femtoseconds

.... to milliseconds



# Neutrons are neutral

- are highly penetrating
- can be used as non-destructive probes,
- can be used to study samples in extreme environments



Property 3



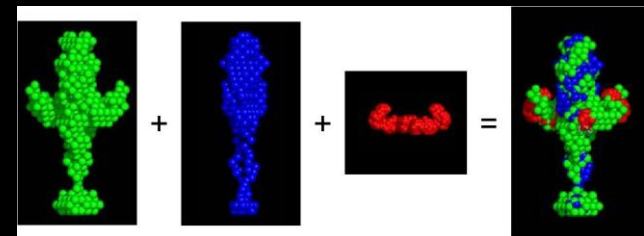
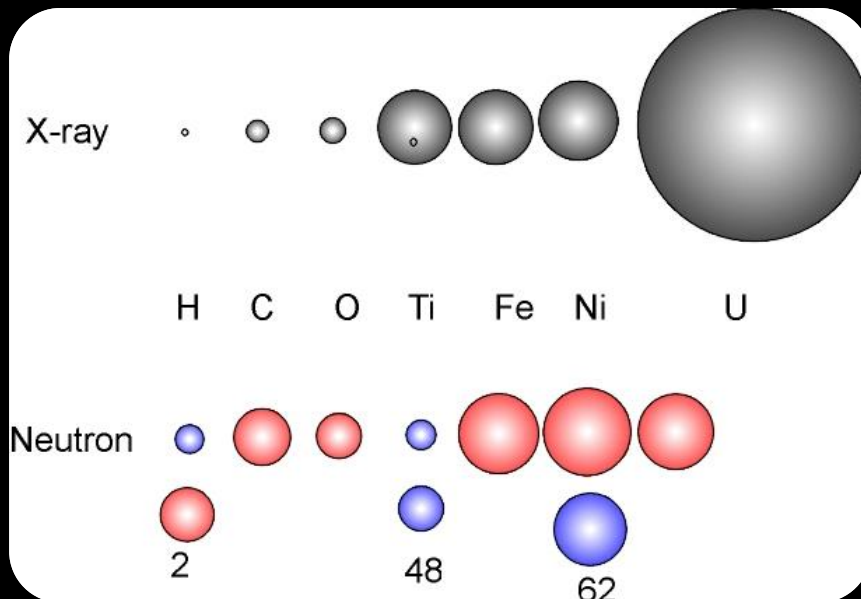
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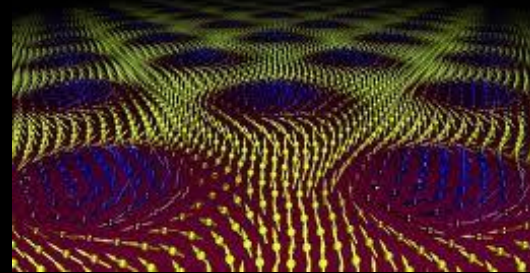
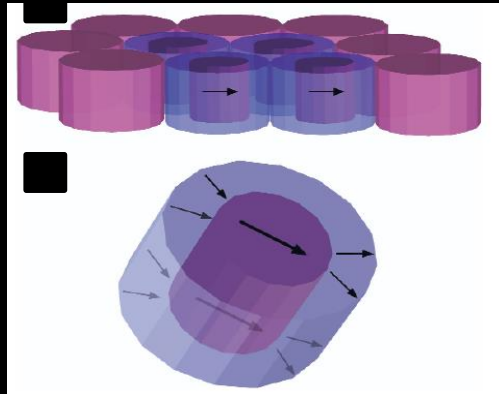
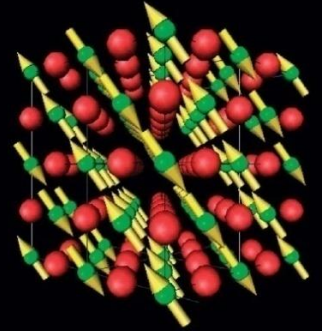
# Neutrons interact with Nuclei

- are sensitive to light atoms, particularly hydrogen
- can exploit isotopic substitution, especially H/D
- 'see' materials differently to X-rays, complementary

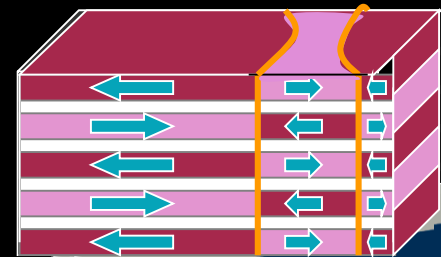


# Neutrons have magnetic moment and spin

- study microscopic magnetic structures
- study magnetic fluctuations , and
- develop magnetic materials



- formed into polarised beams,
- study nuclear (atomic) orientation, and
- separate coherent from incoherent scattering



Properties 5,6



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# Neutrons as a probe

Are **complementary** to other techniques

- X-rays and muons
- Raman, Infrared...
- Nuclear magnetic resonance...
- Dielectric spectroscopy...
- Dynamic light scattering ...
- DFT calculations and MD simulations

Unfortunately there are some **impracticalities**:

- neutrons are scarce and not available in your lab
- sample mass in some cases is large
- deuteration is not always straight forward



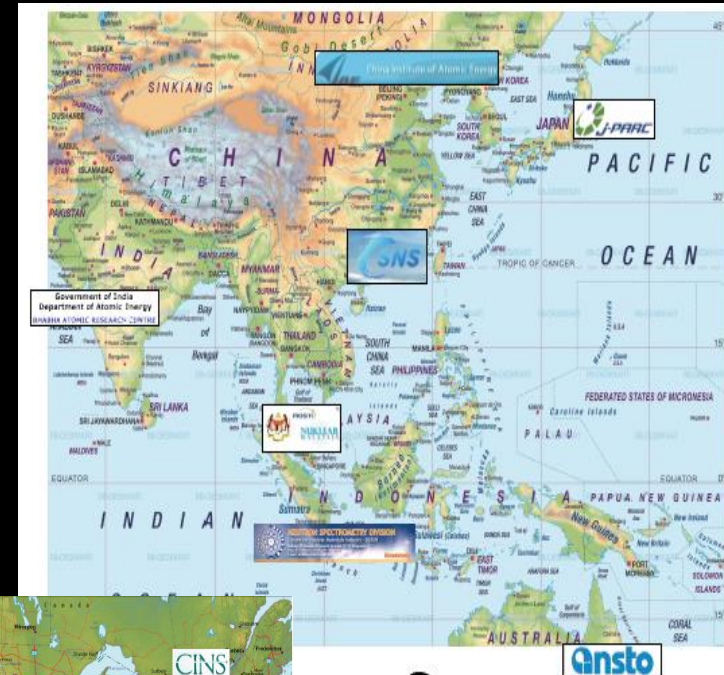
# Neutrons at Facilities

ISIS, Didcot, England

ESS, Lund, Sweden



ILL, Grenoble, France



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# Harwell Science & Innovation Campus



**Diamond –X Rays**

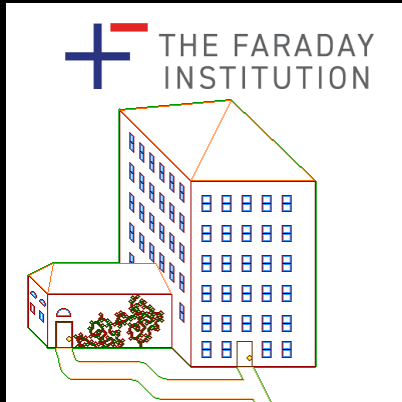
**Central LASER  
Facility**

**TS1**

**TS2**

**ISIS – Neutrons and Muons**

# Harwell Science & Innovation Campus



- ISCF Funding for battery research
- £246m over 4yrs
- Headquarters @ RAL
- Initial projects decided



- Hosts UK groups use Harwell facilities
- Has varied equipment available
- Includes UK Catalysis Hub
- 5-year renewal



- Data intensive centre for supporting facility research
- £16m over 4yrs
- Includes UK Catalysis Hub
- ISIS investment in AI and ML



- Funding for life sciences
- £100m
- Headquarters @ RAL



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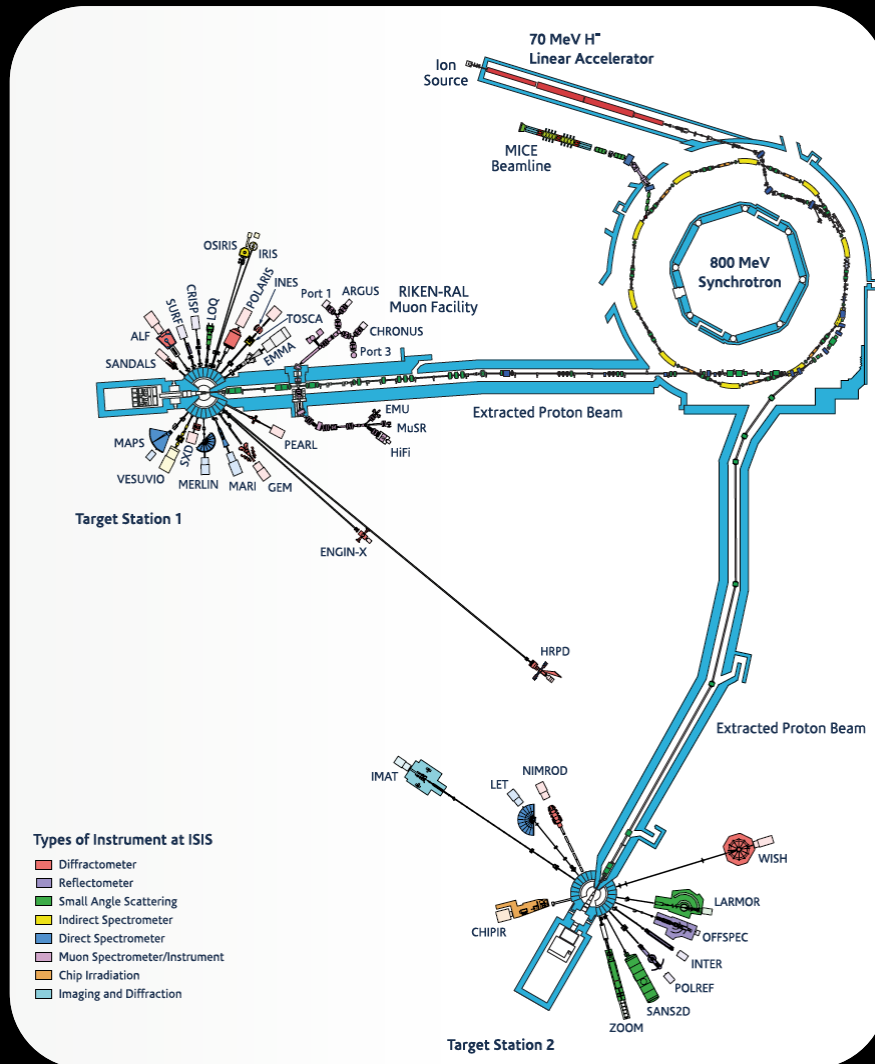
# ISIS Pulsed Neutron and Muon Source

TS1 @ 40Hz

TS2 @ 10Hz

27 Neutron instruments +  
5 Muon instruments +  
Support sample environment +  
Characterisation facilities +  
Irradiation facility +  
Deuteration laboratory +  
Catalysis Hub @ RCAH +

Structure & morphology  
Dynamics



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# ISIS Community



**588**  
PhD students  
visited as users  
**2512**  
user visits



Journal and  
conference  
papers



**142**  
remote users



including 1907 school  
pupils and public

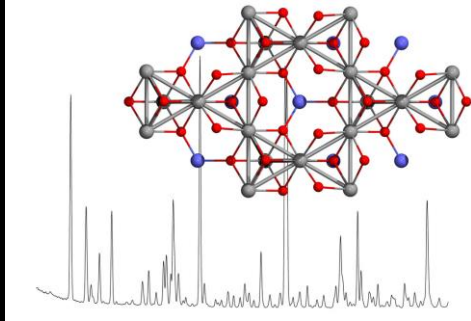
**31%**  
new users



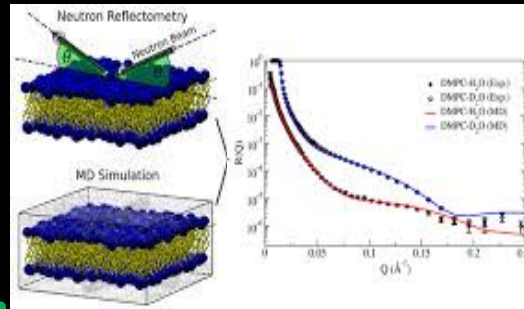


# Neutron Techniques

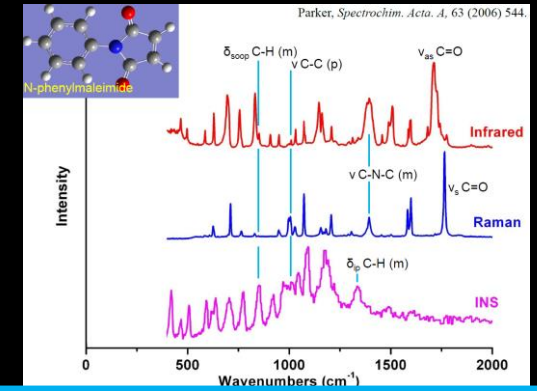
## Diffraction



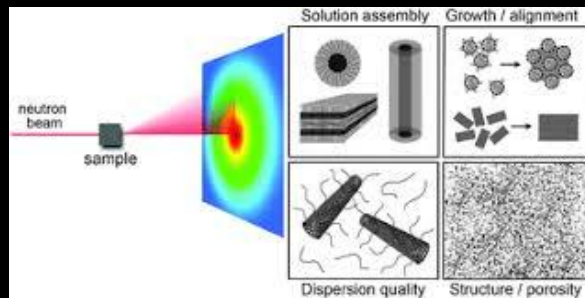
## Reflection



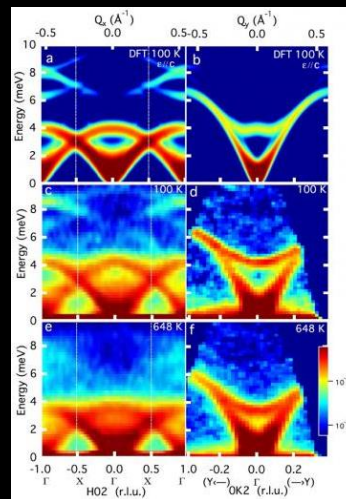
## Vibrational Spectroscopy



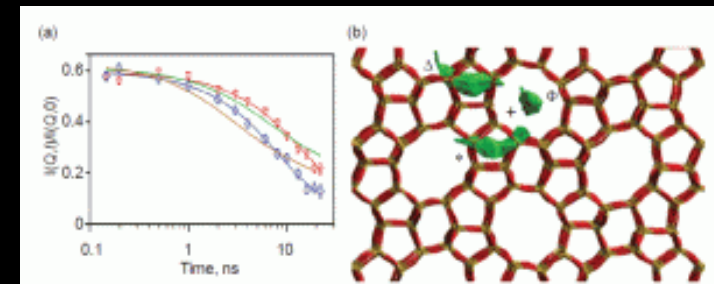
## Small Angle Scattering



## Inelastic Scattering



## Quasi-elastic scattering



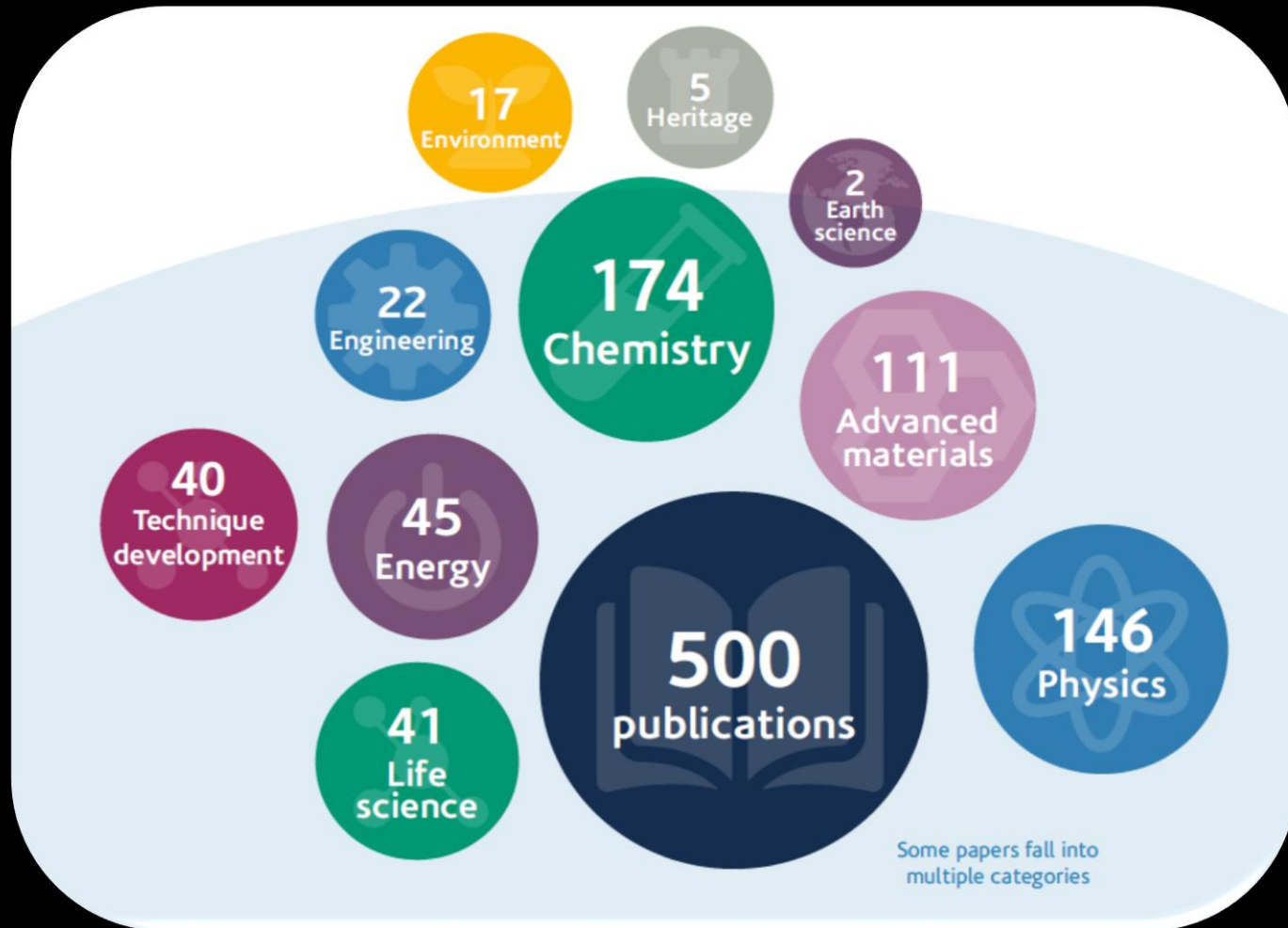
## Imaging



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# Science @ ISIS



# Science at ISIS

## Improving our understanding of metal–organic–frameworks

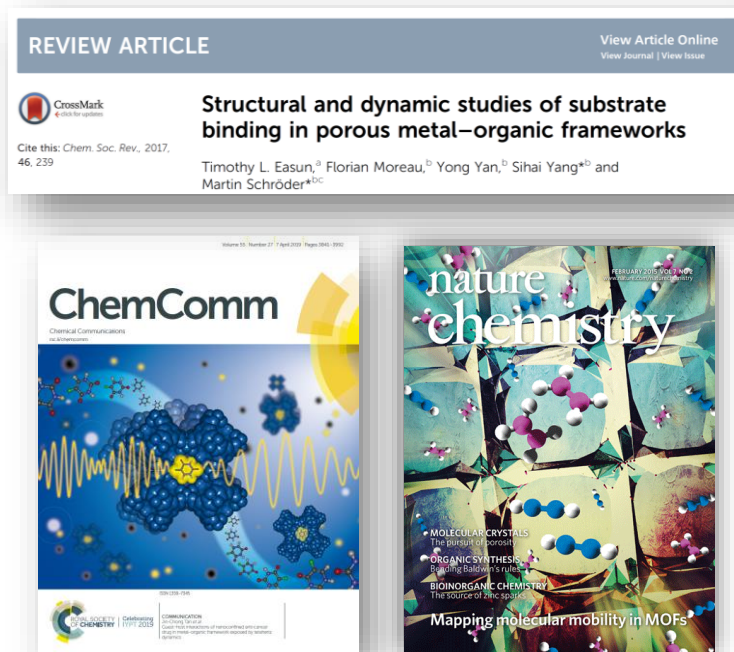
Work carried out on a number of ISIS instruments in combination with DFT calculations have been included in a review of recent progress in the field of porous metal organic frameworks. With their high porosity and capability of binding small molecules MOF's underpin a wide range of materials functions including gas adsorption, separations, drug delivery, and catalysis.

Using neutron diffraction, dynamics and kinetics give a plethora of information on how these materials function at a molecular level, which in turn will lead to the design and development of new functional materials.

Easun TL *et al*, Chemical Society Reviews 46 (2018).  
Souza BE *et al*, Chem. Commun., 55, 3868-3871 (2019)

+++

Instruments  
TOSCA + OSIRIS + WISH



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**ISIS**

# Science at ISIS

## Understanding diffusion in industrial catalysts

QENS and INS have been used to uncover crucial differences in substrate diffusion and active-site interactions in catalysts (in 1998 estimate contribution of catalysts was 15 trillion USD to world economy).

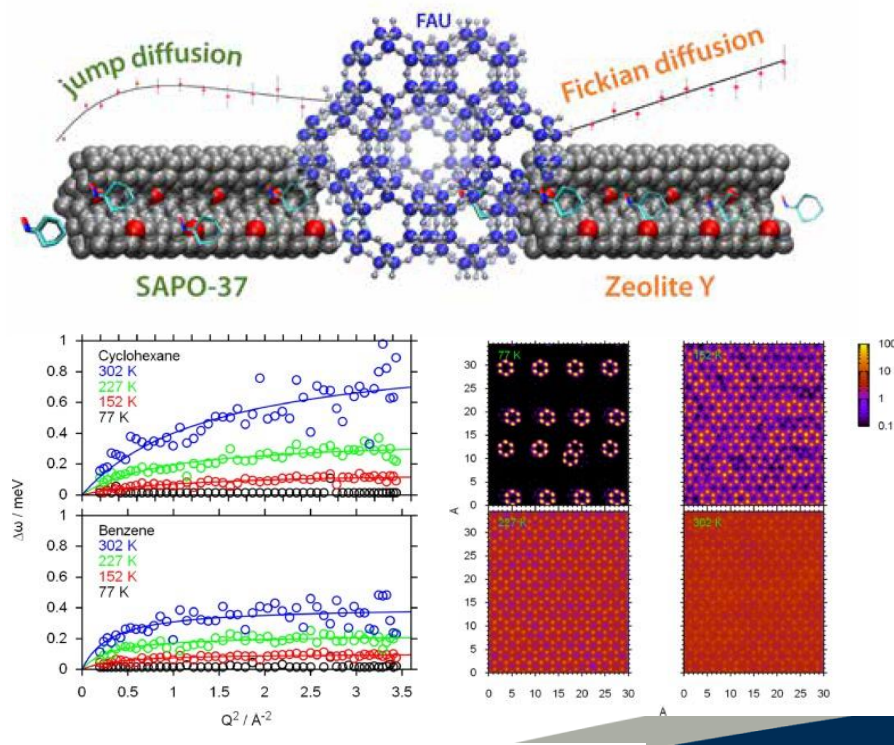
With complementary solid-state NMR, probe-based FTIR, neutron data and MD simulations, users can get insights into the role of the active site in the mechanistic and reaction pathways, and hence rationalise the activity of industrial catalysts.

Results play vital role in designing future catalysis for sustainable industrial processes.

IP Silverwood et al, Surface Science 674, 16-17 (2018)  
G Malta *et al*, ACS Catalysis 8, 8493-8505 (2018)  
ME Potter et al., ACS Catal 7 (2017) ++++



Instruments:  
IRIS, OSIRIS, TOSCA, MAPS, MERLIN



PCCP

PAPER



Methan  
quasile:  
dynam



Alexander J.  
Nikolaos Dir

Cite this: Phys. Chem. Chem. Phys.,  
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# Science at ISIS

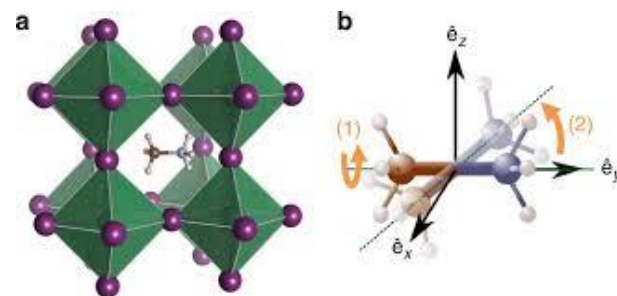
## Exploring next generation of solar cell materials

Lead-halide organic-inorganic perovskites consist of an organic molecule inside an inorganic host framework. Studies on these materials have exploded due to their exceptional photovoltaic properties, which offer the potential of improved solar panels. The rotation of the organic molecule is thought to be key to understanding the overall material properties.

Various teams are looking at different variants using a combination of INS, Raman spectroscopy, diffraction, DFT and QENS to study the structural and dynamics of, in particular of the rotations of the organic molecule. Their results confirm the strong role of hydrogen bonding, a coupling between molecular and framework dynamics and some of the observed electronic properties.

KL Brown et al. "Molecular orientational melting within a lead-halide octahedron framework: The order-disorder transition in  $\text{CH}_3\text{NH}_3\text{PbBr}_3$ " *Phys. Rev. B* 96 (2017)  
AMA Leguy et al. "The dynamics of methylammonium ions in hybrid organic-inorganic perovskite solar cells", *Nat Comms* (2015)

Instruments  
OSIRIS, IRIS and MAPS



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**ISIS**

# Science at ISIS

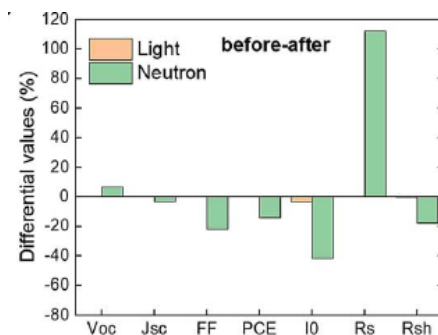
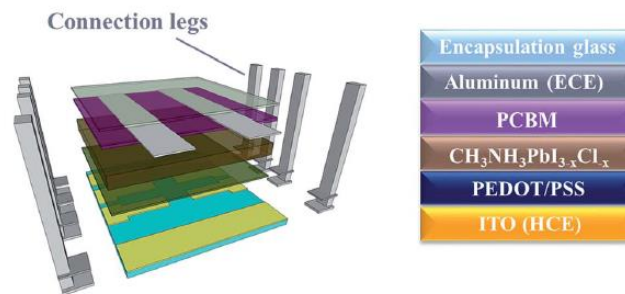
## Perovskite solar cell resistance to fast neutrons *in operando*

High power per weight ratio of metal halide perovskite solar cells is key advantage for low payload applications. Unknown effect of outer space radiation.

First *in-operando* study under light soaked and light + neutron irradiated devices.

Results show incredible resilience of devices to harsh radiation environment. Light degradation is fully reversible, neutron causes permanent damage. However, formation of neutron-induced shallow traps that act as dopants and increase open circuit voltage aid the resilience.

Instrument:  
VESUVIO (ChipIR)



# Science at ISIS

## Investigating the origin of excellent thermoelectric behaviour in $\text{Cu}_2\text{Se}$

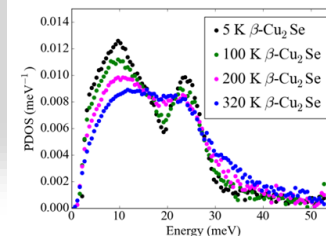
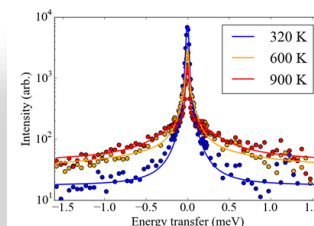
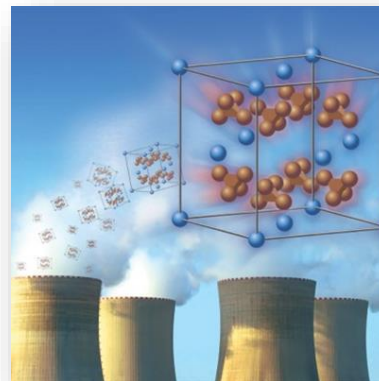
Researchers are keen to find ways to exploit the thermoelectric effect, i.e. turning heat into electricity.

Higher efficiencies could open up numerous applications like harvesting waste heat in car exhausts/power stations, or power up wearable electronics.

Best thermoelectrics minimise heat carried through vibrations of the crystal lattice without affecting electronic conductivity. Experiments tested the Phonon-Liquid-Electron-Crystal approach, suggesting that fast diffusion in thermal terms would act as a liquid while remaining a solid. It was disproved, low thermal conductivity was not due to diffusion but to extreme anharmonicity arising from crystal structure.

D Voneshen et al. *Phys Rev Lett* 118, 145901 (2017).

Instrument:  
LET & MERLIN



# Science at ISIS

## Structural and electrochemical changes in doped cathode materials for Na<sup>+</sup> ion batteries

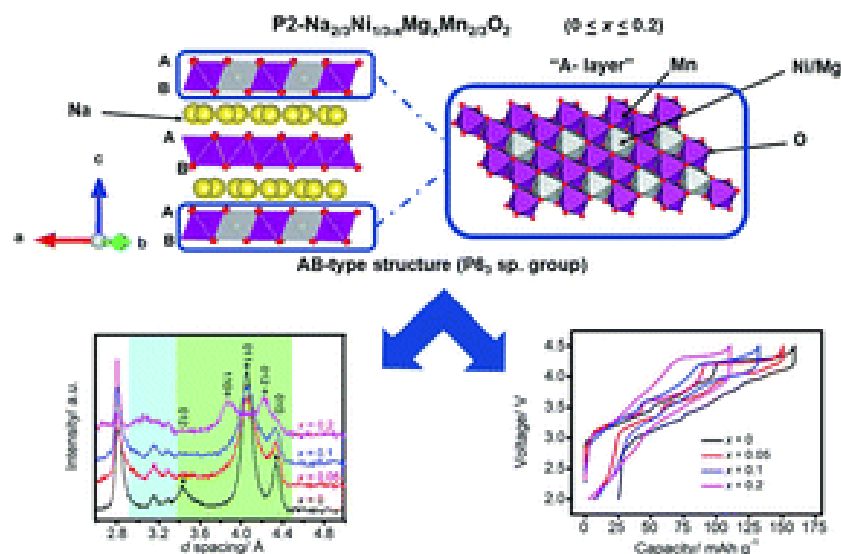
Li is essential for current modern life batteries, powering our electronics and electric vehicles, but it is limited and expensive. Sodium is a cheaper and more abundant alternative for large battery arrays.

Na<sup>+</sup> batteries based on layered oxide compounds with the general formula Na<sub>x</sub>TMO<sub>2</sub> (TM = transition metal), adopting P2-type and O3-type structures, show better electrochemical performance.

Neutron diffraction in combination with electrochemical and *ab initio* studies offers a way to detail the structure of new tailor-made new generation of battery materials with higher energy density, cheaper, safer and long lasting.

N Tap-Ruiz et al. Energy & Env. Sci 6, (2018).

Instrument:  
GEM



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# Science at ISIS

## Improving the efficiency of engine fuel delivery

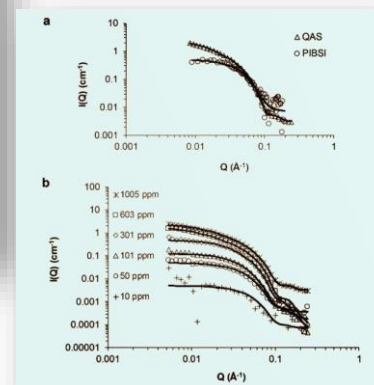
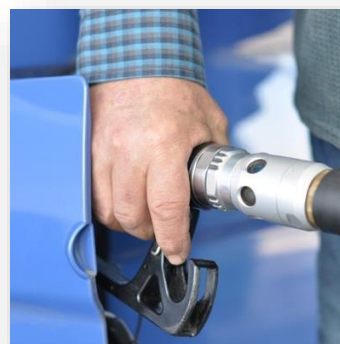
Scientists from London and Lubrizol Ltd. have used SANS to test the role that modern additives have in increasing the efficiency of fuel injectors using diesel or biodiesel, specifically in reducing flow losses.

The team used SANS to investigate variations in surfactant aggregation in additives. Deposit control additives containing a type of surfactant known as quaternary ammonium salt have the ability to remove deposit and increase the volumetric efficiency of the clean injectors by up to 5%.

Experiments revealed significant differences between the micellar structure of the traditional additive, which has been used for over 30 years, and a newly developed one.

H Naseri et al. Scientific Reports 8, 7636 (2018).

Instrument:  
SANS-2D



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# Science at ISIS

## New insights into nanoceria synthesis using Sandals

Cerium oxide (ceria) is an important component in catalytic converters and solid oxide fuel cells.

Nanostructured ceria can be used to greatly enhance catalytic activities for a number of important reactions, but many current preparations require intensive conditions, such as high temperatures, long reaction times, or corrosive or toxic additives.

Scientists from the Universities of Bath and Cambridge have developed a new, green, synthetic route for cerium oxide using deep eutectic solvents— using liquid neutron diffraction to determine the mechanism of reaction.

OS Hammond et al., *Deep eutectic-solvothermal synthesis of nanostructured ceria*, Nature Comms (2018).

Instrument:  
**SANDALS**



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# Science at ISIS

## Neutrons – a breath of fresh air for the offshore wind turbine industry

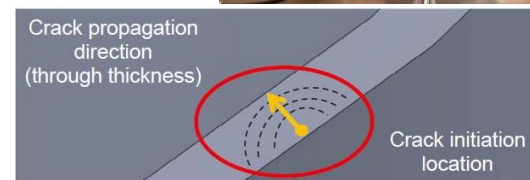
In 2010, offshore wind farms supplied 3% of Britain's electricity, but by 2030 it is envisioned to be 30%. One of the most important issues facing the energy industry today is being able to accurately assess the structural integrity of wind turbine foundations.

Monopiles are installed into the seabed, are 70m long and 3-10m diameter, consisting of hot-rolled steel plates subjected to cold-rolling and bending followed by welding to form cans.

Certain conditions can lead to accelerated fatigue crack initiation and propagation – unexpected catastrophic failure.

A Jacob et al. Theoretical and Applied Fracture Mechanics 96 (2018).

Instrument:  
ENGINE-X



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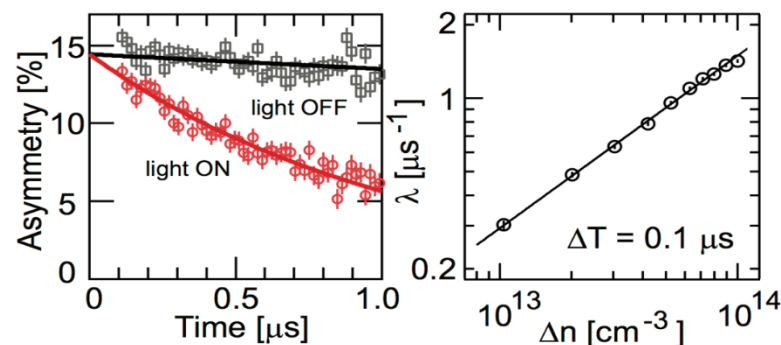
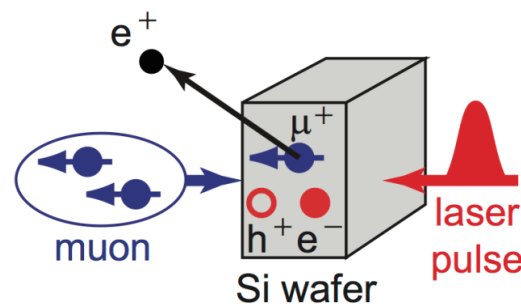
## Muons to measure excess carrier lifetime in bulk silicon

Silicon wafers are workhorses of the electronic age, used in integrated circuits and many photosensitive devices. Key to their success is understanding the behaviour of electrons and holes, and controlling their dynamics.

Novel technique that uses a laser to generate and control carrier density  $\Delta n$  uniformly distributed in a wafer, and measuring the muon spin relaxation rate (since muonium interacts with the carriers). Could have application to other semiconductors, eg. solar cell materials or even go further and do depth-dependent lifetime spectroscopy?

K. Yokoyama et al. *Photoexcited Muon Spin Spectroscopy: A New Method for Measuring Excess Carrier Lifetime in Bulk Silicon*, Phys Rev Lett 119, no. 22 (2017): 226601.

Instrument:  
HIFI



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# Summary & Outlook

- The **neutron** probe possesses a range of characteristics well matched to answer a plethora of questions in both **energy materials research**
- **Uniquely** Neutrons
  - Solve chemical and magnetic structures
  - Characterise short range and disordered systems
  - Observe the excitations of systems
  - Sensitive to surfaces and interfaces: nanoscience
  - Realistic sample environment conditions: H,T,P,E, pH, hazardous environments *etc.*
  - **Complement** other scattering and bulk characterisation tools
- *Beyond the traditional...*
  - Investigating next generation materials
  - Simplicity from complexity
  - Integrated HPC modelling
  - Many unexplored areas – new opportunities...



# Training Opportunities

## General schools

### 15<sup>TH</sup> OXFORD SCHOOL OF NEUTRON SCATTERING

3<sup>rd</sup> - 15<sup>th</sup> September 2017 // St Anne's College, Oxford University



**ISIS Neutron Training Course 2018**  
February 27 - March 8

**Physics**  
Nuclear and Magnetic diffraction, excitations & optical reflectometry

**Chemistry & Materials**  
diffraction, disordered materials, molecular spectroscopy

**Basic Principles**  
time-of-flight, instrument components, neutron materials, elementary theory

**Soft Matter**  
polymers and bio-molecular materials, small angle scattering, reflectometry, air-liquid interfaces

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Registration open 1 Oct - 30 Nov  
For further details contact: Helen C. Walker  
helen.c.walker@stfc.ac.uk +44(0)1223 343720  
www.stfc.ac.uk/pages/isis-neutron-training-course.aspx

### Muon Spectroscopy Training School

ISIS Facility, STFC Rutherford Appleton Laboratory  
19<sup>th</sup> - 23<sup>rd</sup> March, 2018



An introduction to muon techniques for PhD students & post-doctoral researchers  
Hands-on experience of muon experiments, data analysis, and interpretation

Lectures & experiments cover applications of muons in:

- Condensed matter physics
- Materials science
- Chemistry

Further details & application form are available from:

<https://www.isis.stfc.ac.uk/Pages/Muon-Spectroscopy-Training-School.aspx>



## Specialised schools

### MDANSE 2018

Simulation of Inelastic Neutron Scattering using McStas and material dynamics models

Sept. 24<sup>th</sup> - 28<sup>th</sup> 2018

Puerto de la Cruz - Tenerife

## Joint PhD studentships



## On the beamline!



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# ISIS Beamtime Access Routes - Academic

<https://www.isis.stfc.ac.uk/Pages/Apply-for-beamtime.aspx>

## Direct Access:

Two calls per year – deadlines April and October (next 16<sup>th</sup> April)

Requires standard 2-page self-contained science case – online application

Peer-reviewed proposals by Facility Access Panels (FAP) meet in June and December

Allocation 3-6mths after round

## Rapid Access:

Anytime – for ‘hot science’, extra time for PhD completion....

Standard 2-page self-contained science case sent to FAP member

Allocation decision in 2 wks, allocation ASAP

Essential to discuss with instrument scientist (IS)

## Xpress Access:

Anytime – for short ‘easy’ measurements performed by IS, eg. test signals, finish off, one-off structures

Decision and time allocation by IS

Simple form

*All UK user expenses are paid for*

*New users welcome*

*Talk to IS first*



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# ISIS Beamtime Access Routes - Industrial

<https://www.isis.stfc.ac.uk/Pages/Industry.aspx>

## ISIS Collaborative R & D programme:

Fast-track route for companies with a UK manufacturing or research base to use ISIS

Provides a low-risk way in for industry

Industry can decide after the experiment if results should:

- 1) remain confidential in which case they pay commercial rate
- 2) can be published in which case no fee

Way to increase the economic benefit that ISIS contributes to the UK

Apply at any time once a company joins the programme

Single, groups or join partnerships with academia welcome to join

## How to join the programme:

Initial discussion with Industrial Liaison Manager, Dr. Chris Frost ([Christopher.frost@stfc.ac.uk](mailto:Christopher.frost@stfc.ac.uk))

Submit proposal which includes why the need for neutrons, company description, approx.

economic benefit to company and UK, in-kind contributions & milestones

Costs to each partner, IP details, Safety.



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# ISIS Long Shutdown

ISIS will have an extended shutdown from late summer 2020 - autumn 2021 for significant refurbishment work on the TS1 and installation of a new LINAC tank, in order to extend the life of the facility for many years to come.

**TS1 will be off from Sept 2020 until Nov 2021 (for 14mths)**

**TS2 will be off from Sept 2020 until May 2021 (for 8mths)**

October 2019 proposal round will be for all instruments, April 2020 proposal round will most likely be cancelled, and the October 2020 proposal round is likely to be for TS2 instruments only.



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# Thank you for your attention

*Let us help you use neutrons (and muons)  
to view your materials in a unique way*

Victoria García Sakai

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[www.isis.stfc.ac.uk](http://www.isis.stfc.ac.uk)



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