Neutrons for Energy Materials at the ISIS Facility

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Science & Technology Facilities Council ISIS


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CALM
AND
LOG
ON
Digital
Economy
Health
Curiosity Driven
Research
goal-oriented
curiosity-driven
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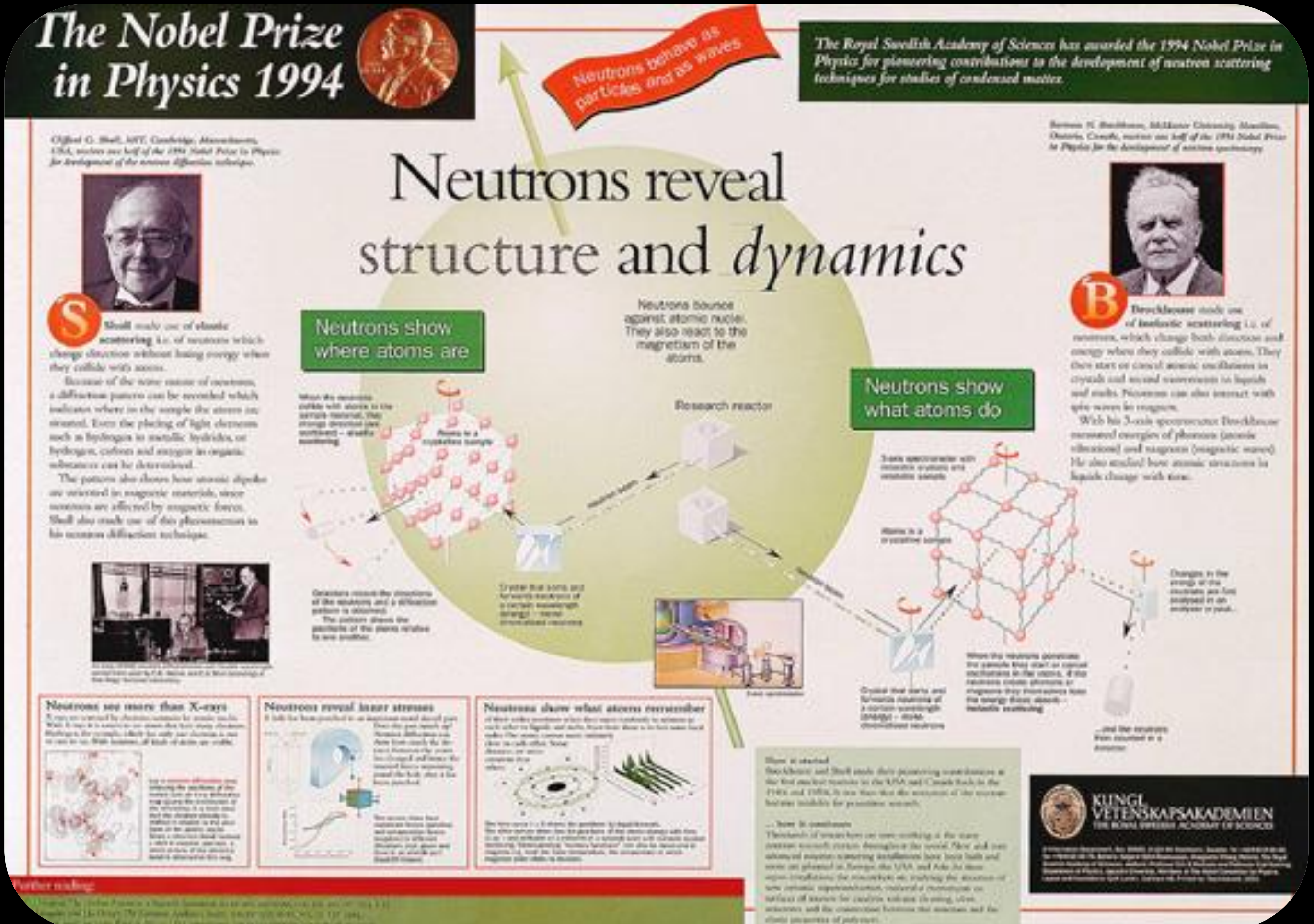
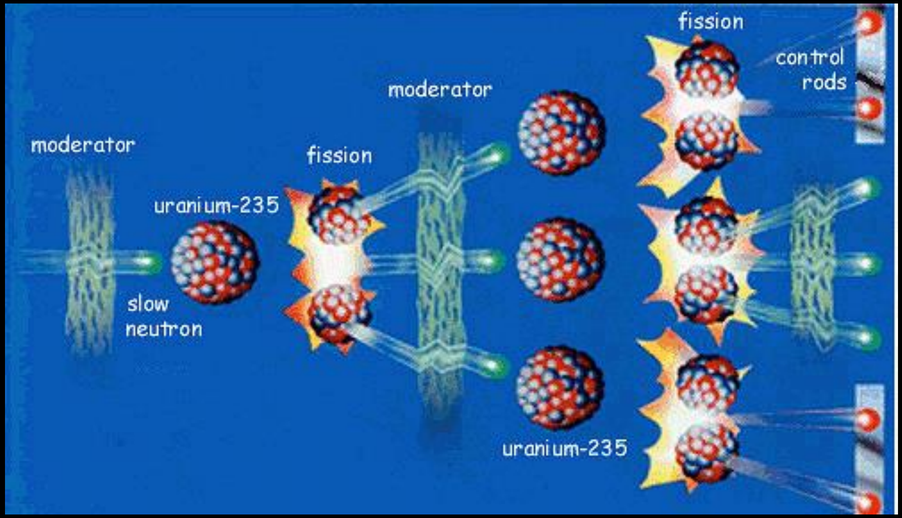


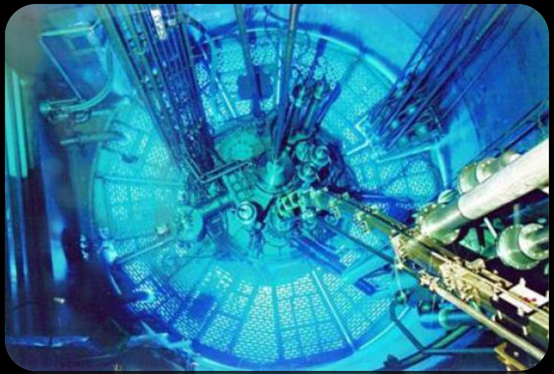
Neutrons tell us where atoms are and what atoms do

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)



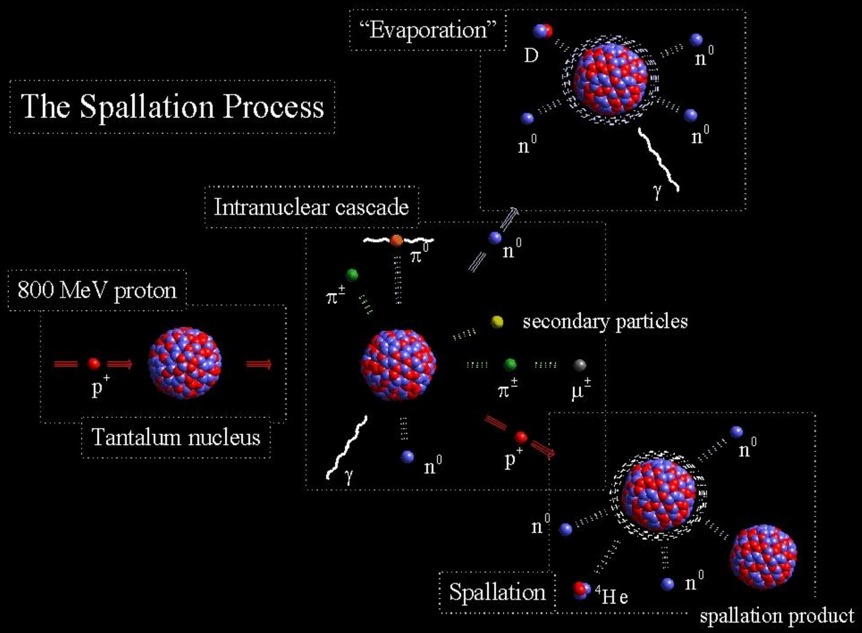


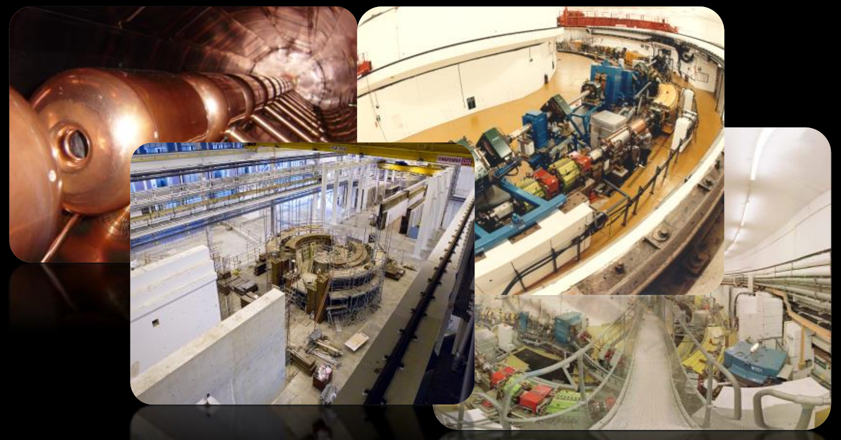
$^{235}U + {}^{1}n \Rightarrow$

fission fragments + 2.4 ^{1}n + 192.9 MeV

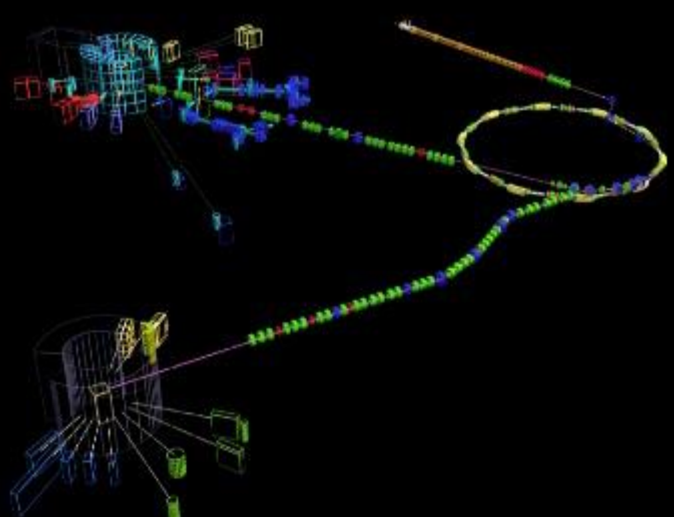
Neutrons

Spallation (ISIS)



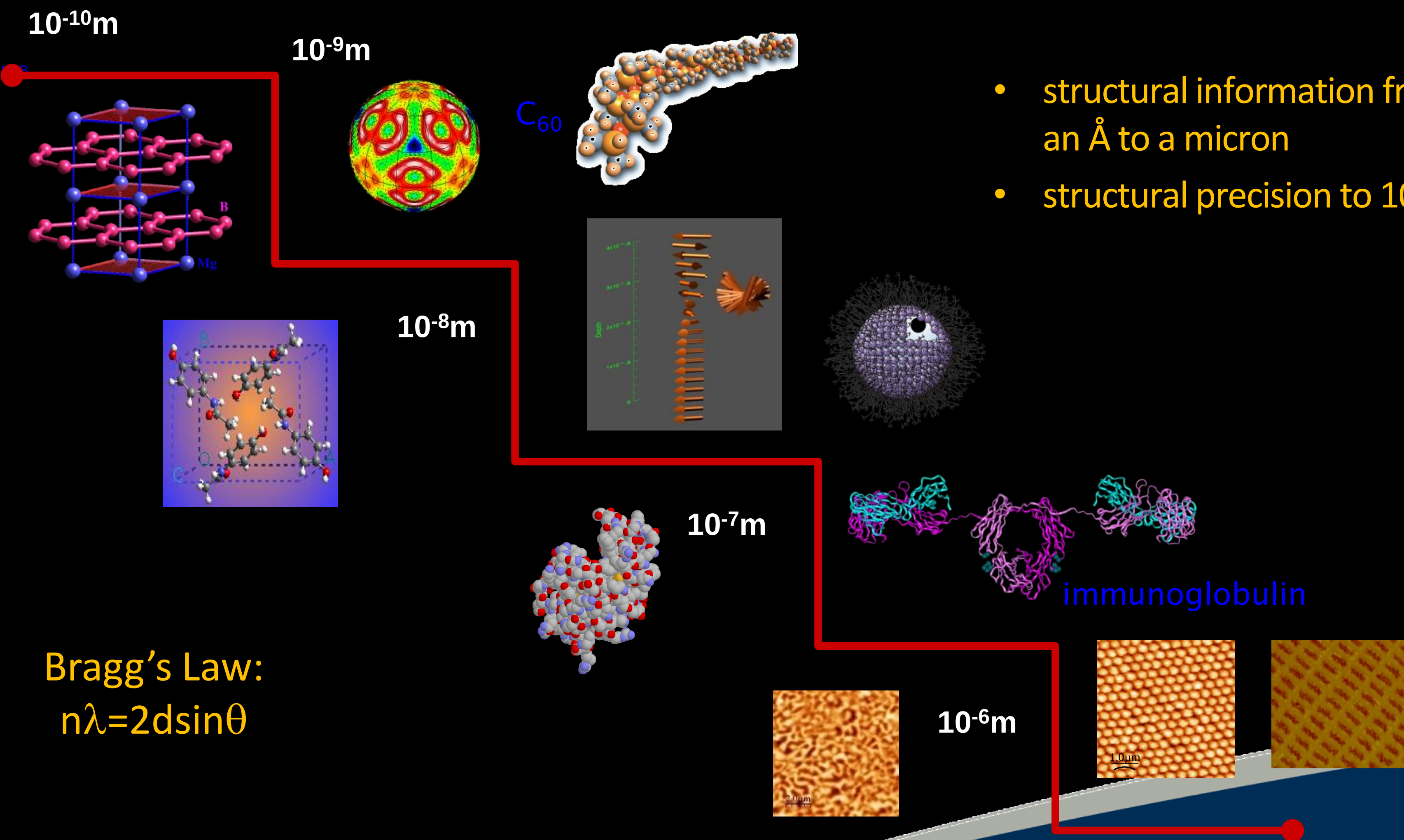


~ 16n/collision + ~20 MeV/n



EUROPEAN
SPALLATION
SOURCE

Neutron wavelengths are of the same order as atomic spacings



- structural information from an Å to a micron
- structural precision to 10^{-15}m.

Bragg's Law:
$n\lambda=2d\sin\theta$

Property 1

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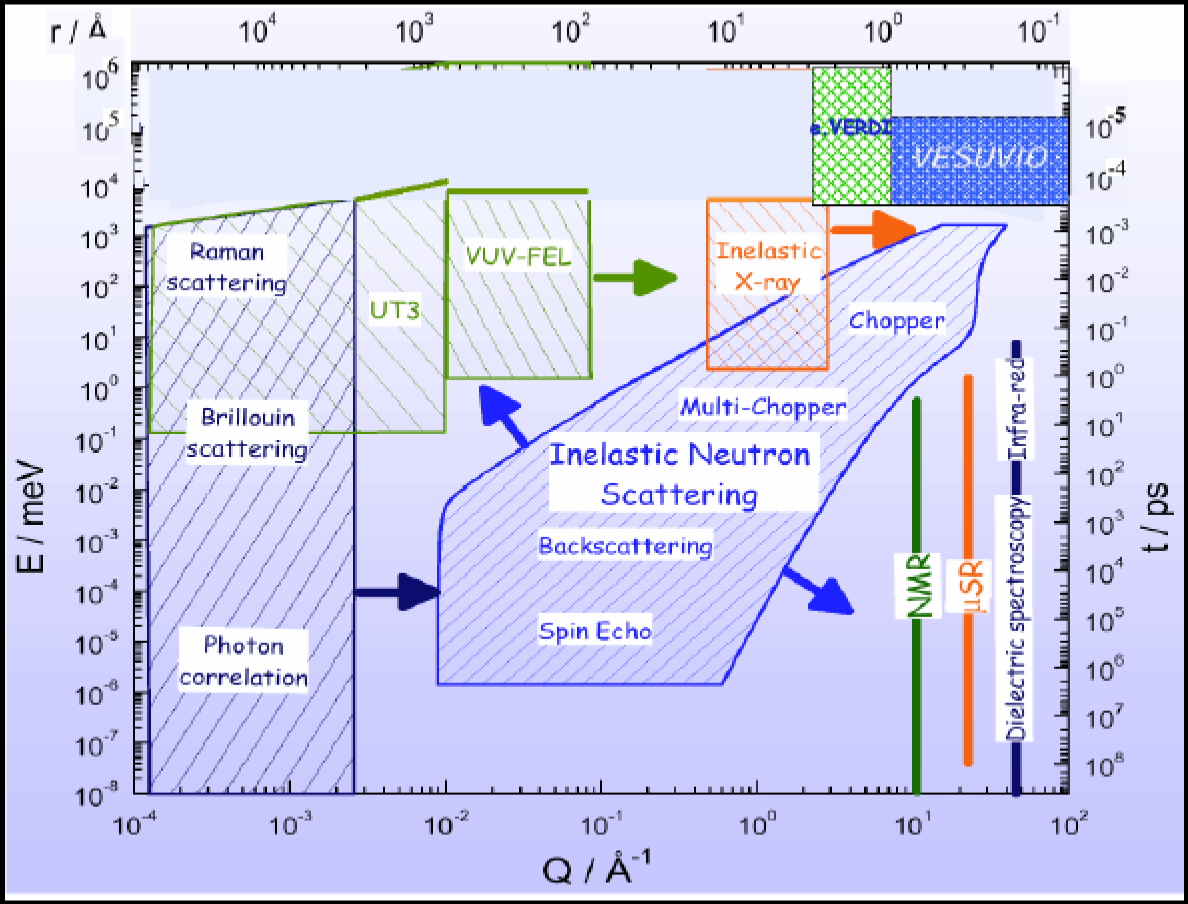
Neutron energies are of the order of elementary excitations and molecular modes



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

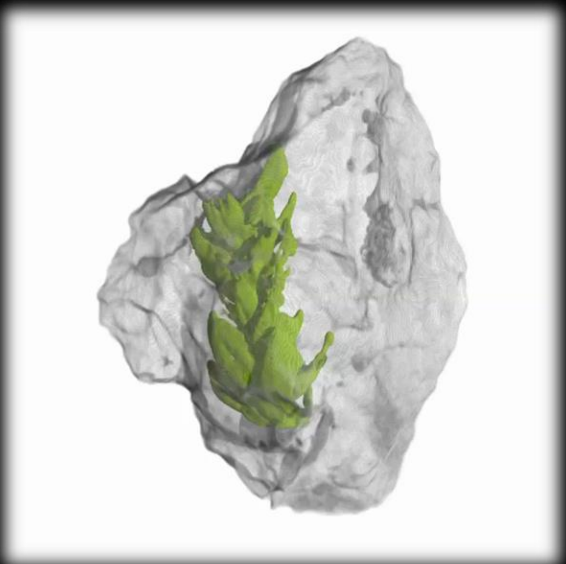
...from femtoseconds

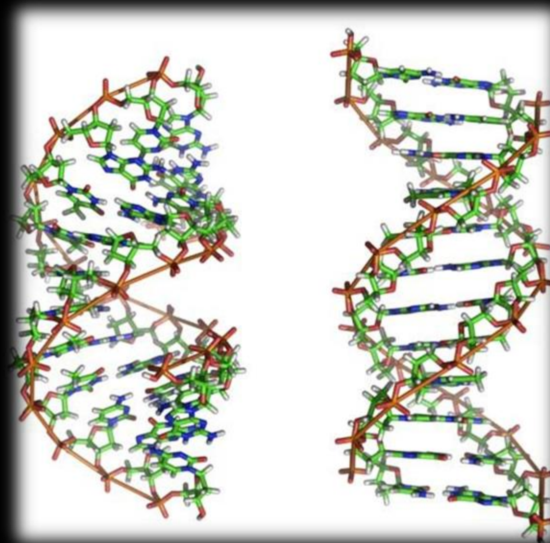
.... to milliseconds

Property 2

Neutrons are neutral

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

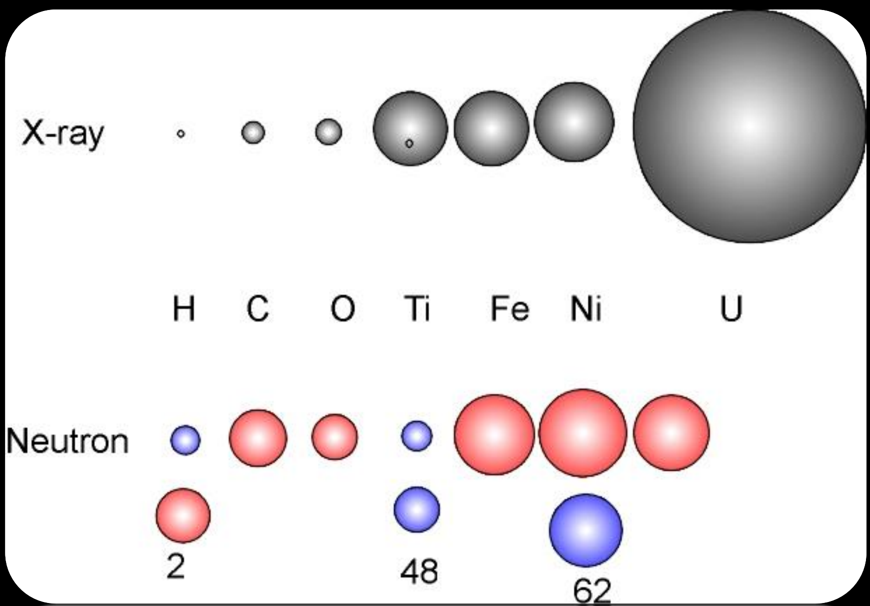


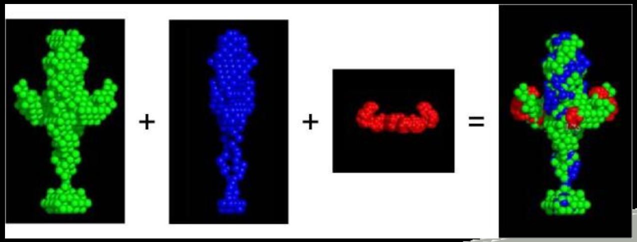


Property 3

Neutrons interact with Nuclei

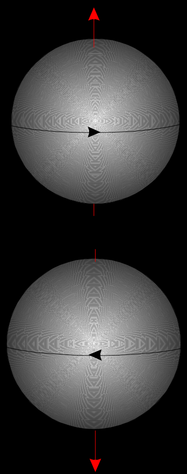
- are sensitive to light atoms, particularly hydrogen
- can exploit isotopic substitution, especially H/D
- ‘see‘ materials differently to X-rays, complementary



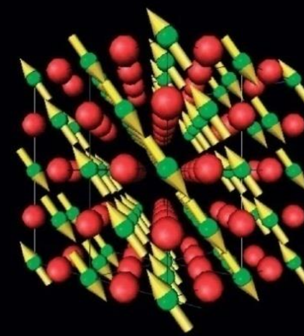


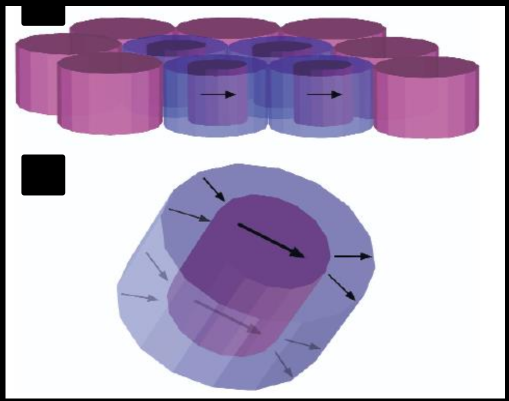
Property 4

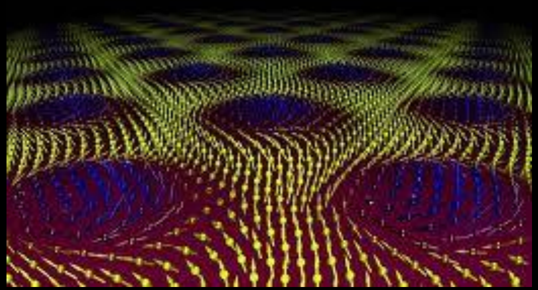
Neutrons have magnetic moment and spin



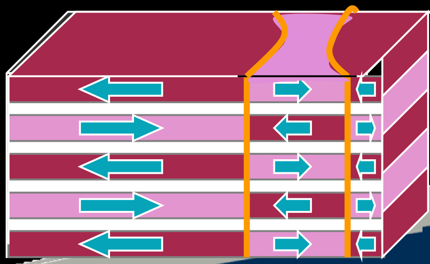
- study microscopic magnetic structures
- study magnetic fluctuations , and
- develop magnetic materials







- 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**:

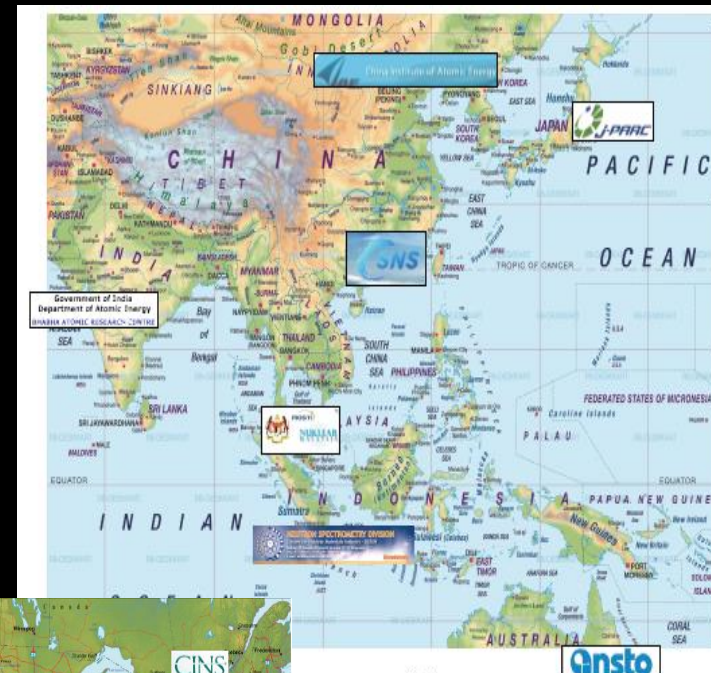
- neutrons are scarce and not available in your lab
- sample mass in some cases is large
- deuteration is not always straight forward

Neutrons at Facilities

ISIS, Didcot, England

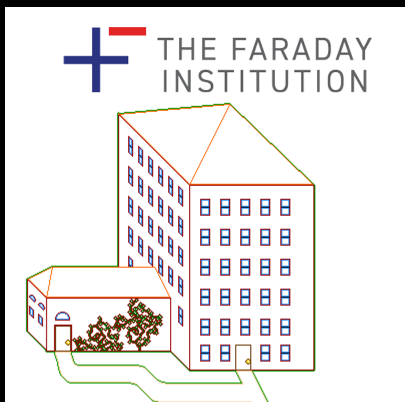
ESS, Lund, Sweden

ILL, Grenoble, France



Harwell Science & Innovation Campus

Harwell Science & Innovation Campus



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

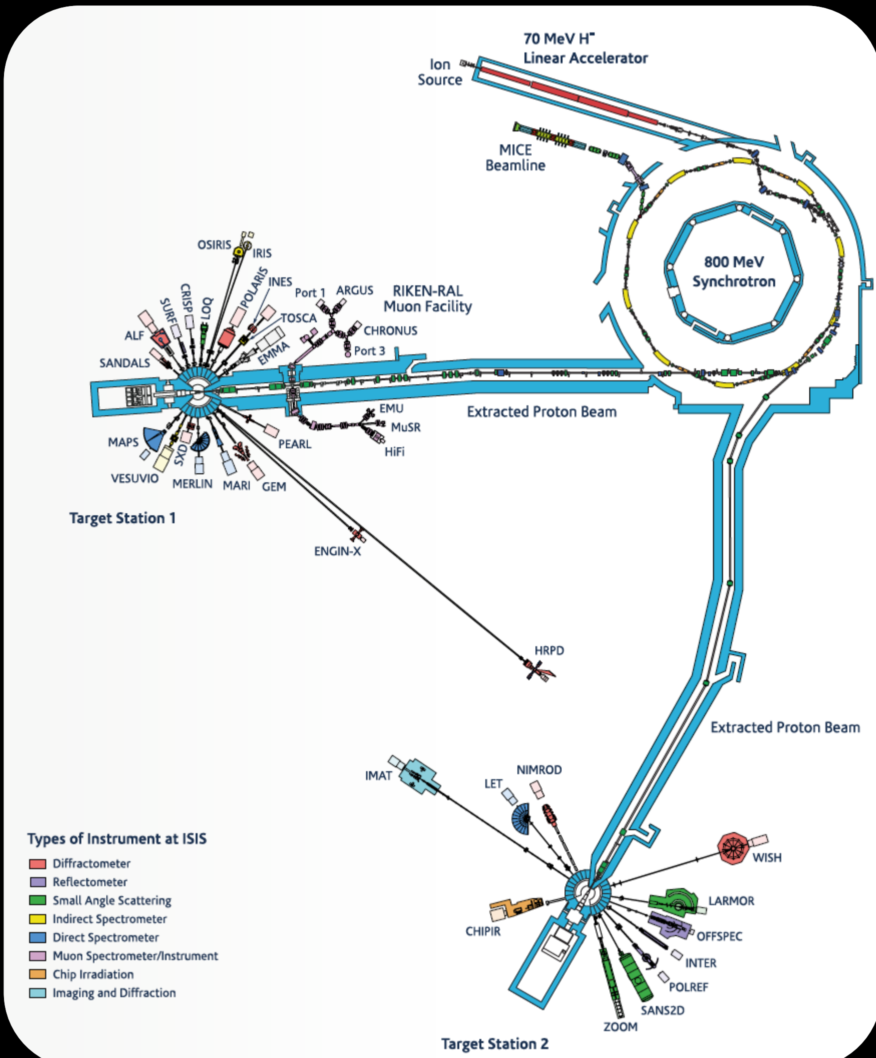
- Hosts UK groups use Harwell facilities
- Has varied equipment available
- Includes UK Catalysis Hub
- 5-year renewal

alc
ada lovelace centre

- 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

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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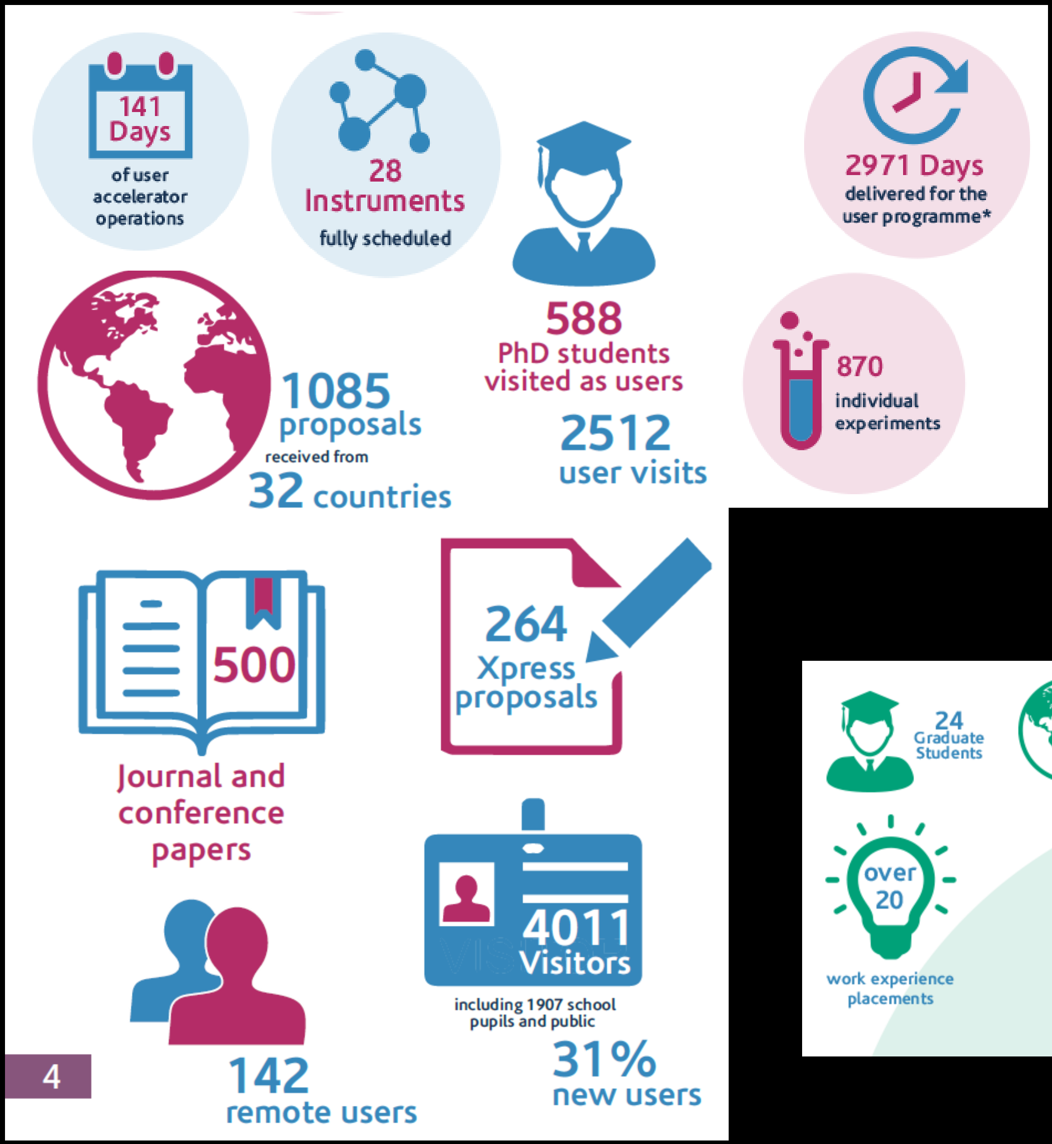
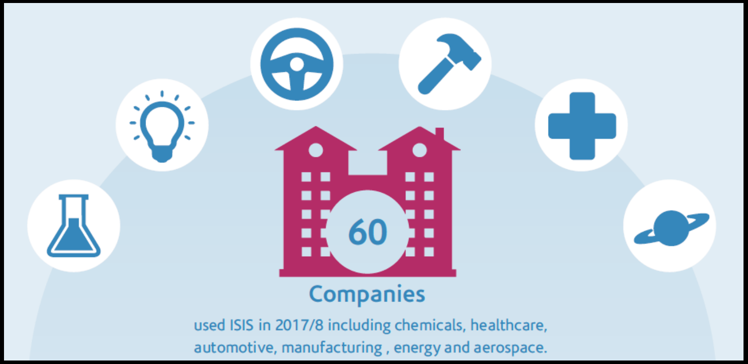
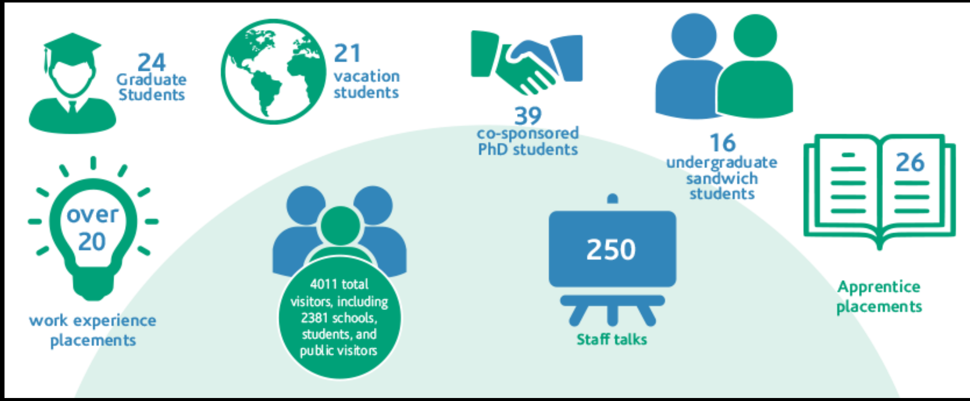
ISIS Community

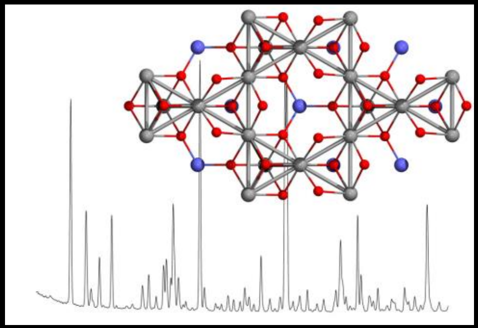




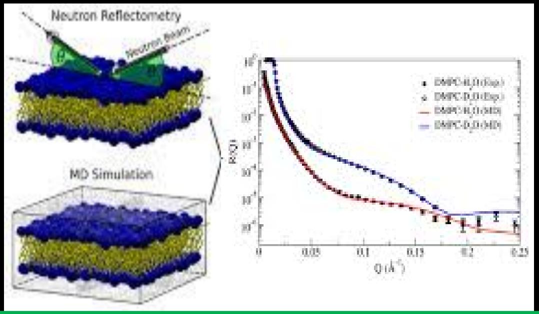


Neutron Techniques

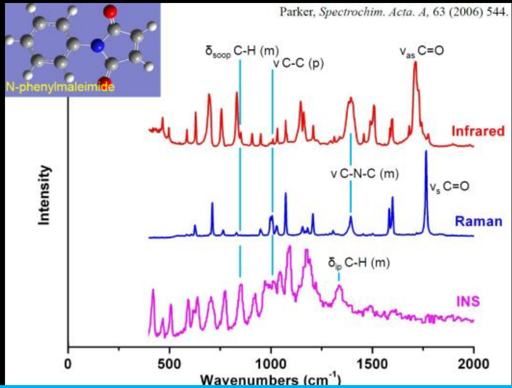
Diffraction



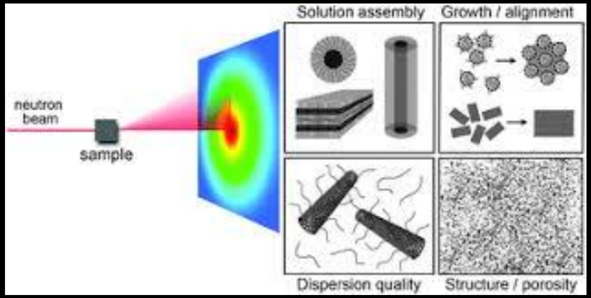
Reflection



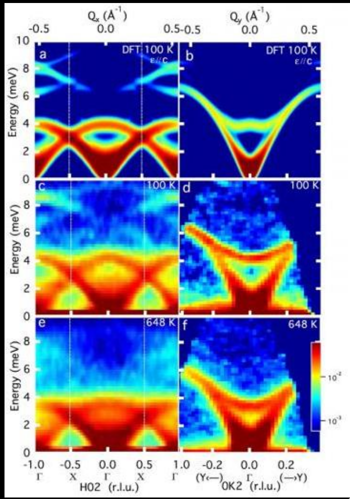
Vibrational Spectroscopy



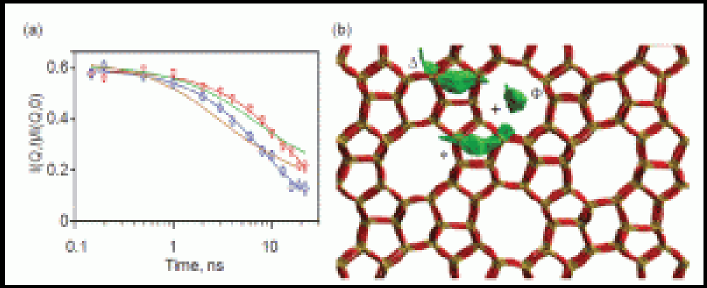
Small Angle Scattering



Inelastic Scattering



Quasi-elastic scattering



Imaging

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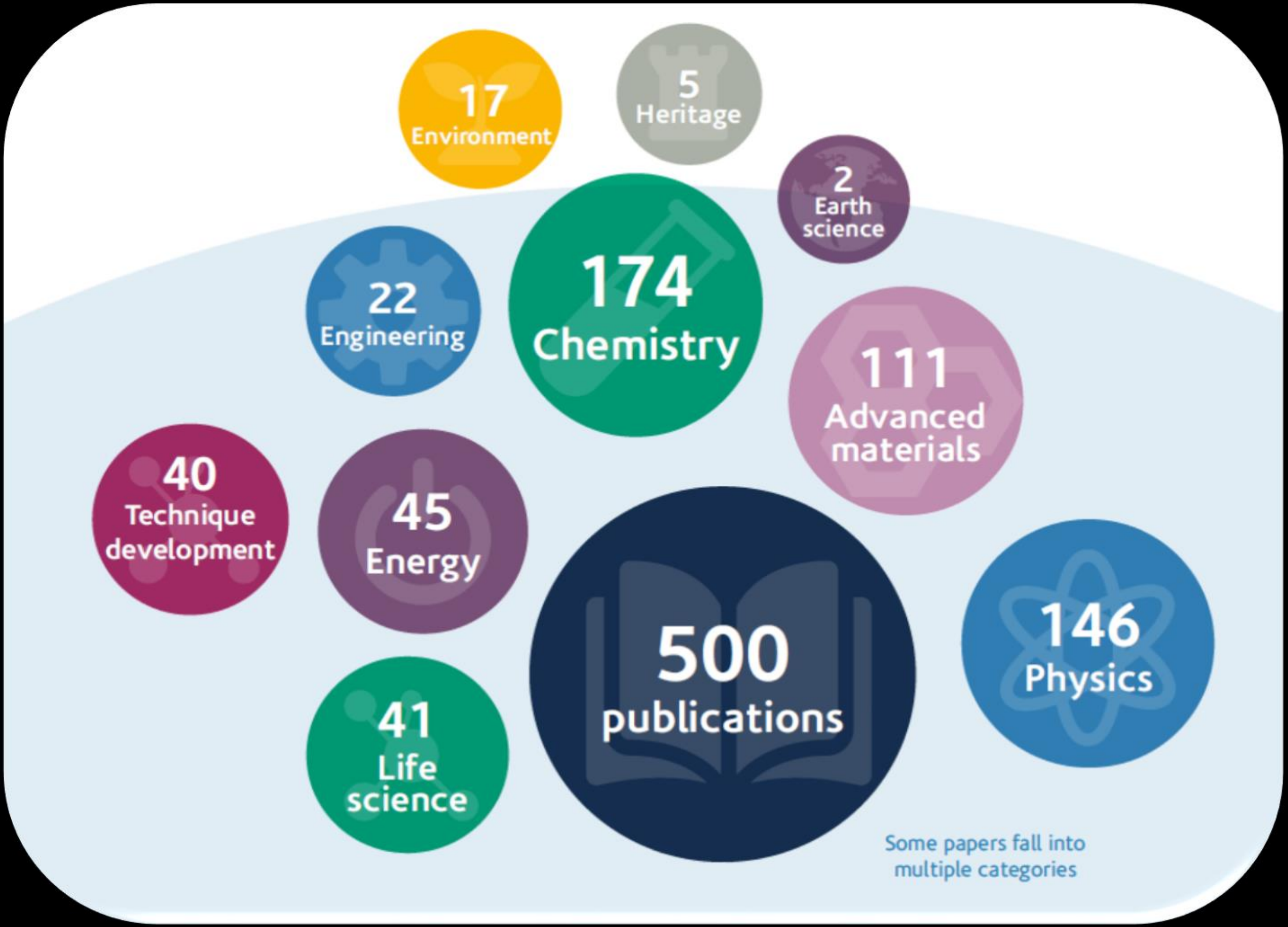

Science at ISIS

Improving our understanding of metal–organic–frameworks

Instruments
TOSCA + OSIRIS + WISH

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.


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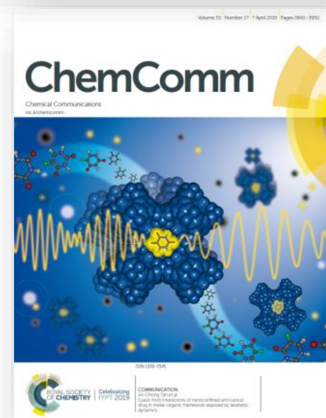
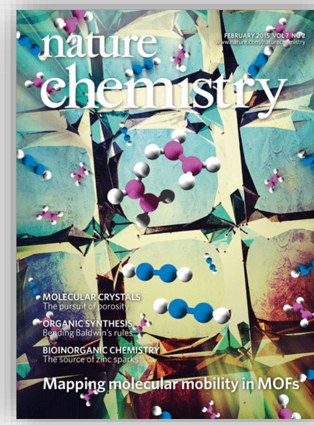
Cite this: *Chem. Soc. Rev.*, 2017, 46, 239

Structural and dynamic studies of substrate binding in porous metal–organic frameworks

Timothy L. Easun,[a] Florian Moreau,[b] Yong Yan,[b] Sihai Yang*[b] and Martin Schröder*[bc]





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

Science at ISIS

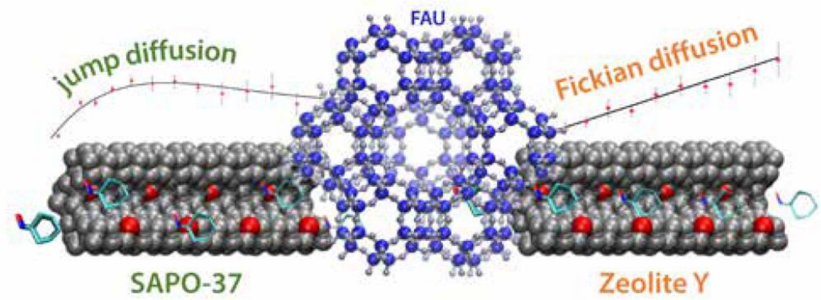
Instruments:
IRIS, OSIRIS, TOSCA, MAPS, MERLIN

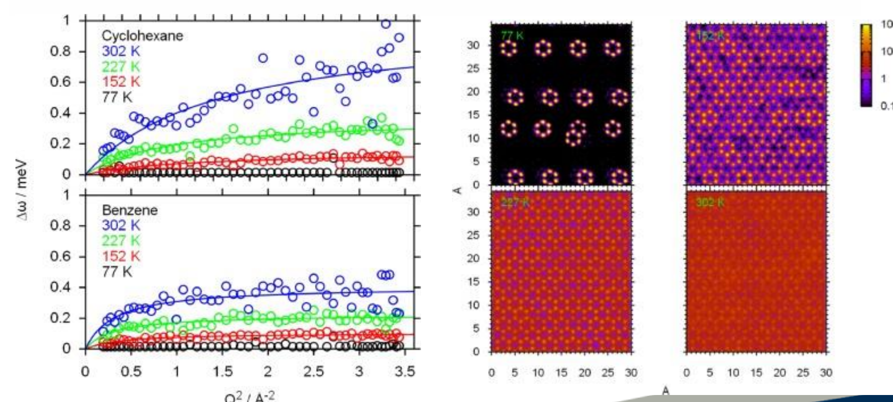
Understanding diffusion in industrial catalysts

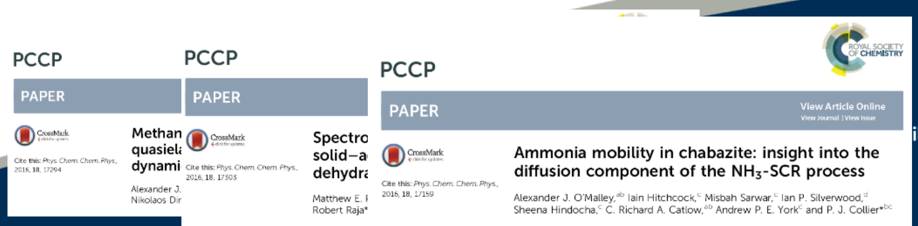
QENS and INS have been use to uncover crucial differences in substrate diffusion and active-site interactions in catalysts (in 1998 estimate contribution of catalysts was 15 trillion USD to world economy).

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.





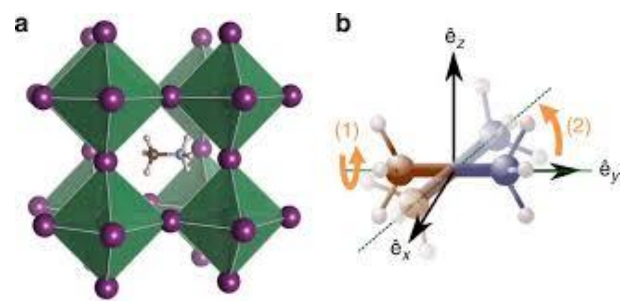


IP Silverwood et al, Surface Science 674, 16-17 (2018)
G Malta *et al*, ACS Catalysis 8, 8493-8505 (2018)
ME Potter et al., ACS Catal 7 (2017) ++++

Science at ISIS

Exploring next generation of solar cell materials

Instruments
OSIRIS, IRIS and MAPS



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 CH3NH3PbBr3" Phys. Rev. B 96 (2017)
AMA Leguy et al. "The dynamics of methylammonium ions in hybrid organic-inorganic perovskite solar cells", Nat Comms (2015)

Science at ISIS

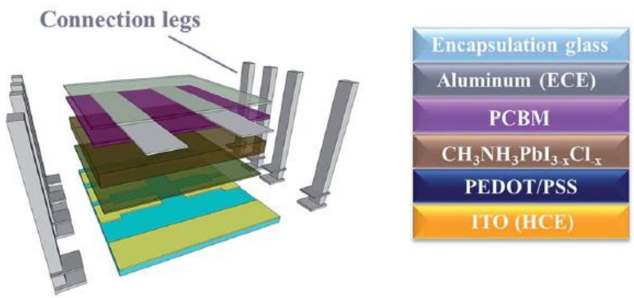
Perovskite solar cell resistance to fast neutrons *in operando*

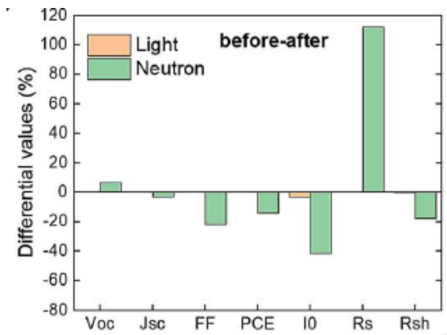
Instrument:
VESUVIO (ChipIR)

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.





Sustainable Energy & Fuels

DOI: 10.1039/c9se00102f

ROYAL SOCIETY OF CHEMISTRY

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Cite this: DOI: 10.1039/c9se00102f

Perovskite solar cell resilience to fast neutrons

G. M. Paternò,[†a] V. Robbiano,[a] L. Santarelli,[a] A. Zampetti,[a] C. Cazzaniga,[b] V. Garcia Sakai[*b] and F. Cacialli[*a]

Science at ISIS

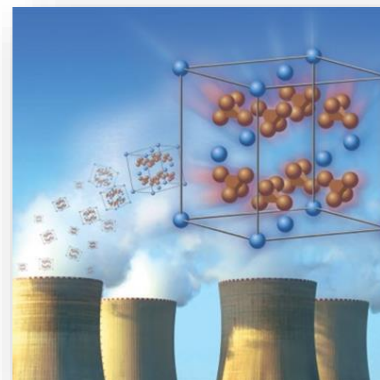
Investigating the origin of excellent thermoelectric behaviour in Cu_2Se

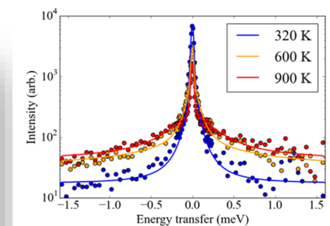
Instrument:
LET & MERLIN

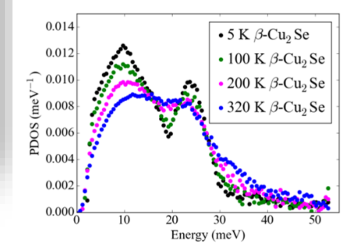
Researchers are keen to find ways to exploit the thermoelectric effect, i.e. turning heat into electricity.

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).

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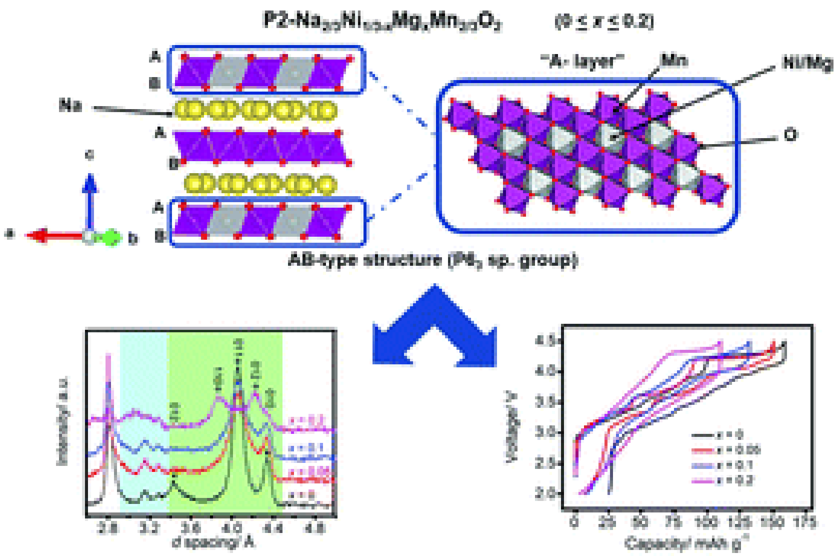
Science at ISIS

Structural and electrochemical changes in doped cathode materials for Na+ ion batteries

Instrument:
GEM

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+ batteries based on layered oxide compounds with the general formula NaxTMO2 (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).

Science at ISIS

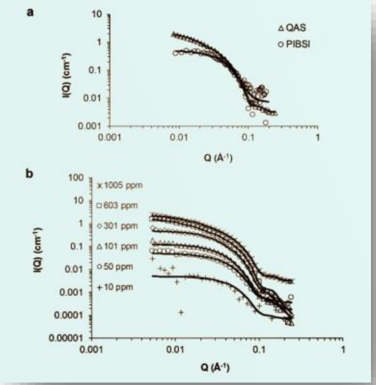
Improving the efficiency of engine fuel delivery

Instrument:
SANS-2D

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 of 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).

Science at ISIS

New insights into nanoceria synthesis using Sandals

Instrument:
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).

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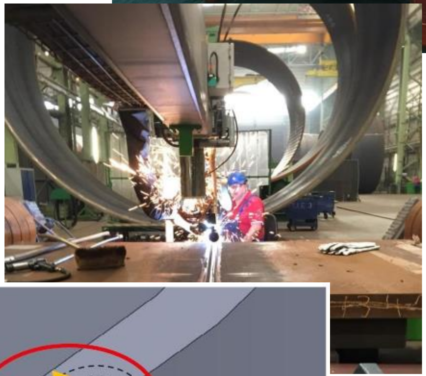
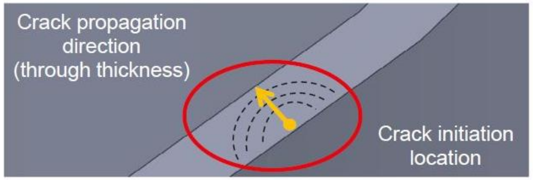
Neutrons – a breath of fresh air for the offshore wind turbine industry

Instrument:
ENGIN-X



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 asses 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.



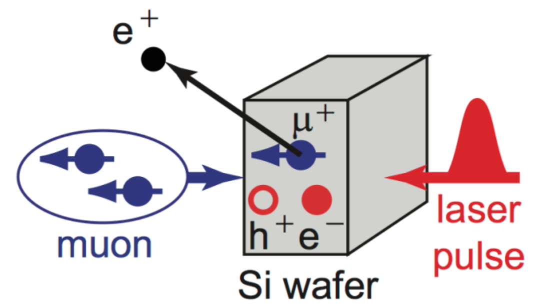
Certain conditions can lead to accelerated fatigue crack initiation and propagation – unexpected catastrophic failure.

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

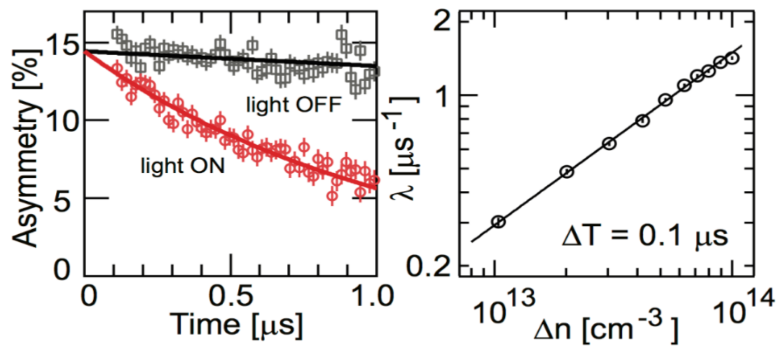
Science at ISIS

Muons to measure excess carrier lifetime in bulk silicon

Instrument:
HIFI



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 Δn 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.

Summary & Outlook

- The neutron probe possesses a range of characteristics well matched to answer a plethora pf 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

Specialised schools

Joint PhD studentships

On the beamline!

Science & Technology Facilities Council
ISIS

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 16th 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

Science & Technology Facilities Council ISIS

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)

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, Saefty.

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.

Thank you for your attention

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

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Science & Technology Facilities Council
ISIS