Rutherford Appleton Laboratory Campus

Martin Owen Jones – *Energy Materials Coordinator, ISIS facility*

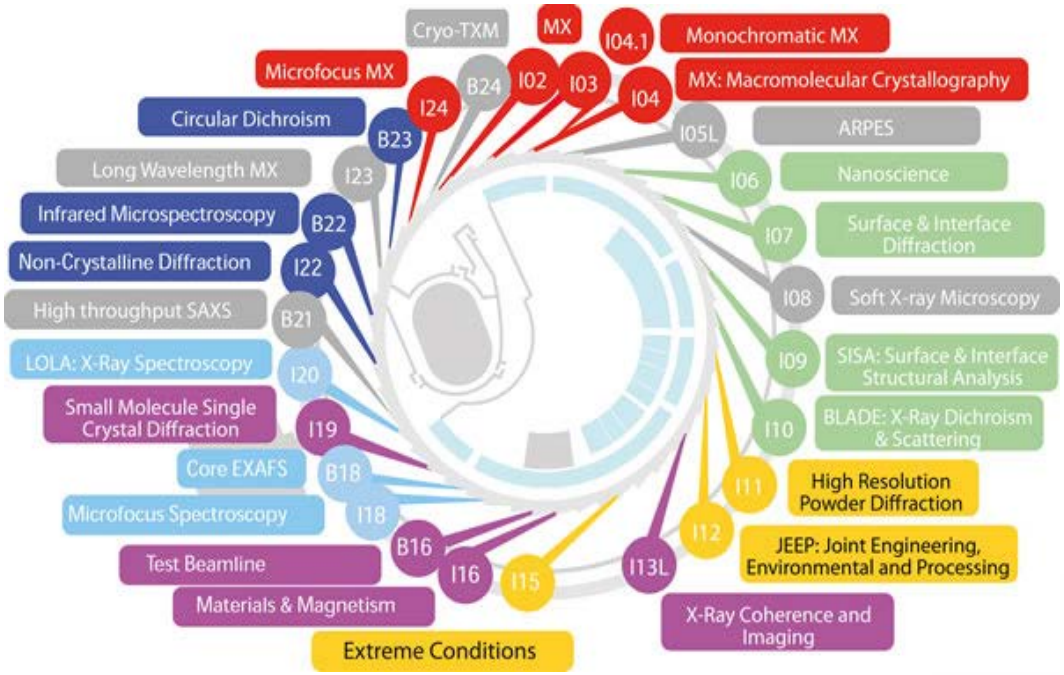
Chiu Tang - *Principal Scientist for Beam line I11, Diamond Light Source*

ISIS

diamond

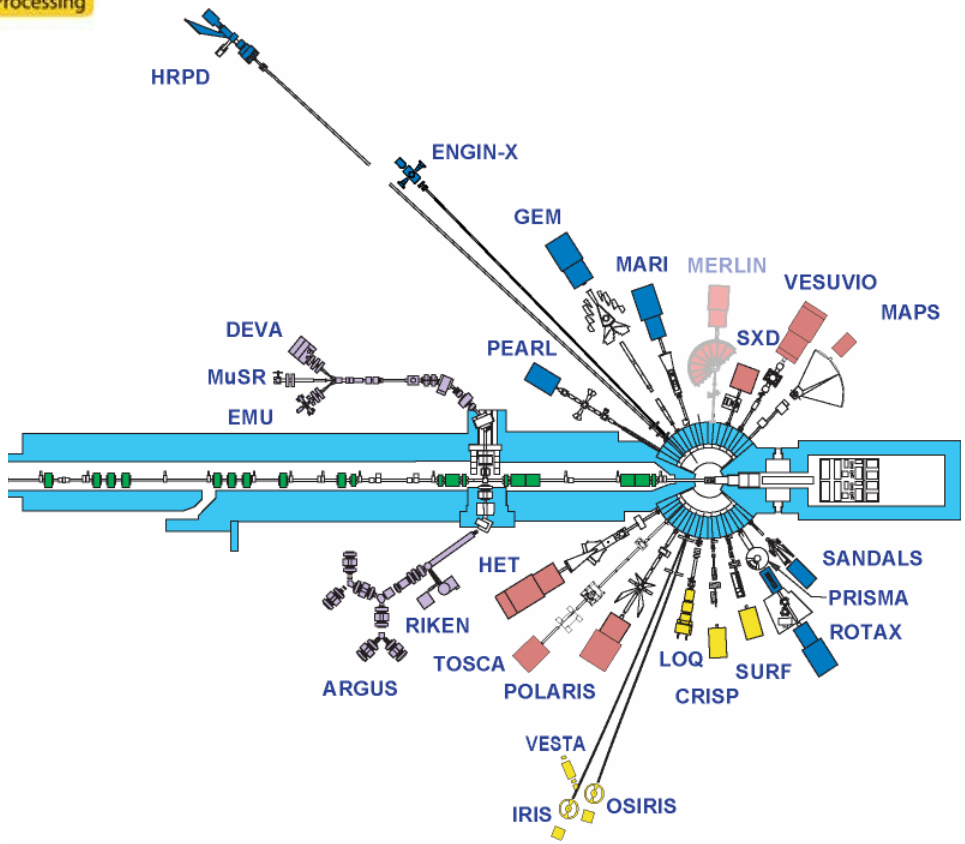
Science & Technology Facilities Council

Harwell Science & Innovation Campus

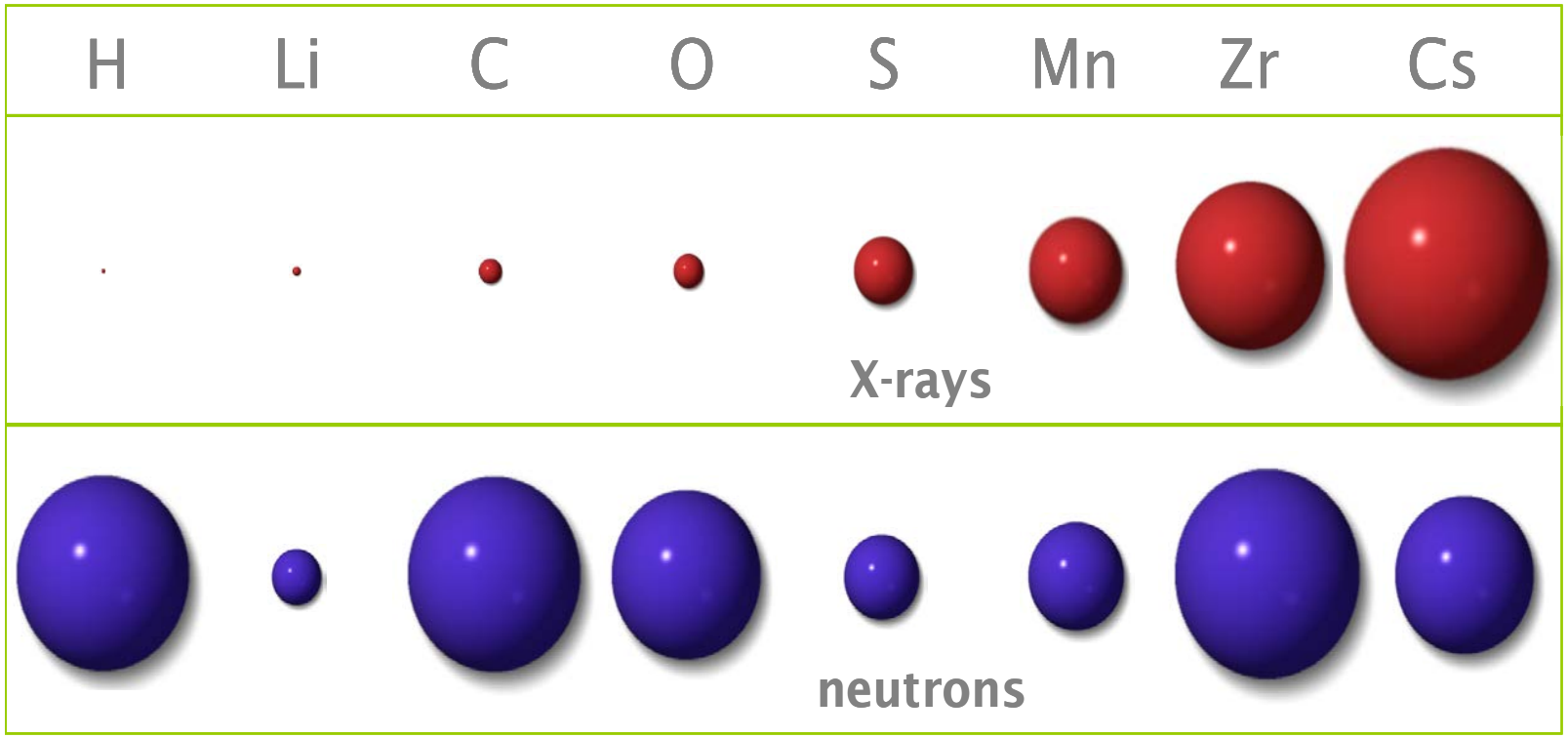


Diamond Beam lines

ISIS Beam lines



Neutron cross-section - *isotopic dependence*



Diamond and **ISIS** beam lines

Chemical Information

What's happening at a molecular level?
How do waxes/biofuels pack and what do additives do?

How is that affecting ordering?
When they aren't crystalline, do waxes/biofuels have more descript order?

Physical Information

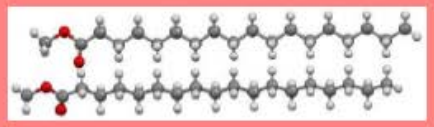
What is the impact on shape and size of particles?

How do additives effect how crystals grow?

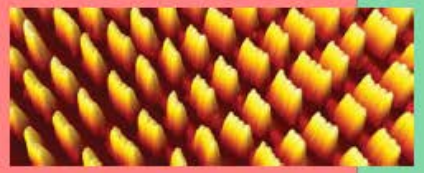
How do these particles come together to affect the microstructure of the system?

How do these crystal shapes come together to block filters?

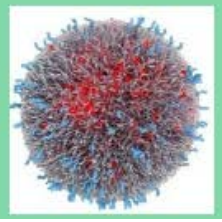
WAXS/Diffraction
Chemical bonds
Packing of molecules
Defects



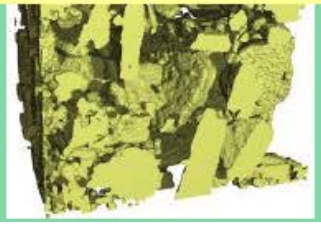
PDF
Chemical bonds
Long range ordering
Coordination



SAXS
Particle shape
Particle distribution

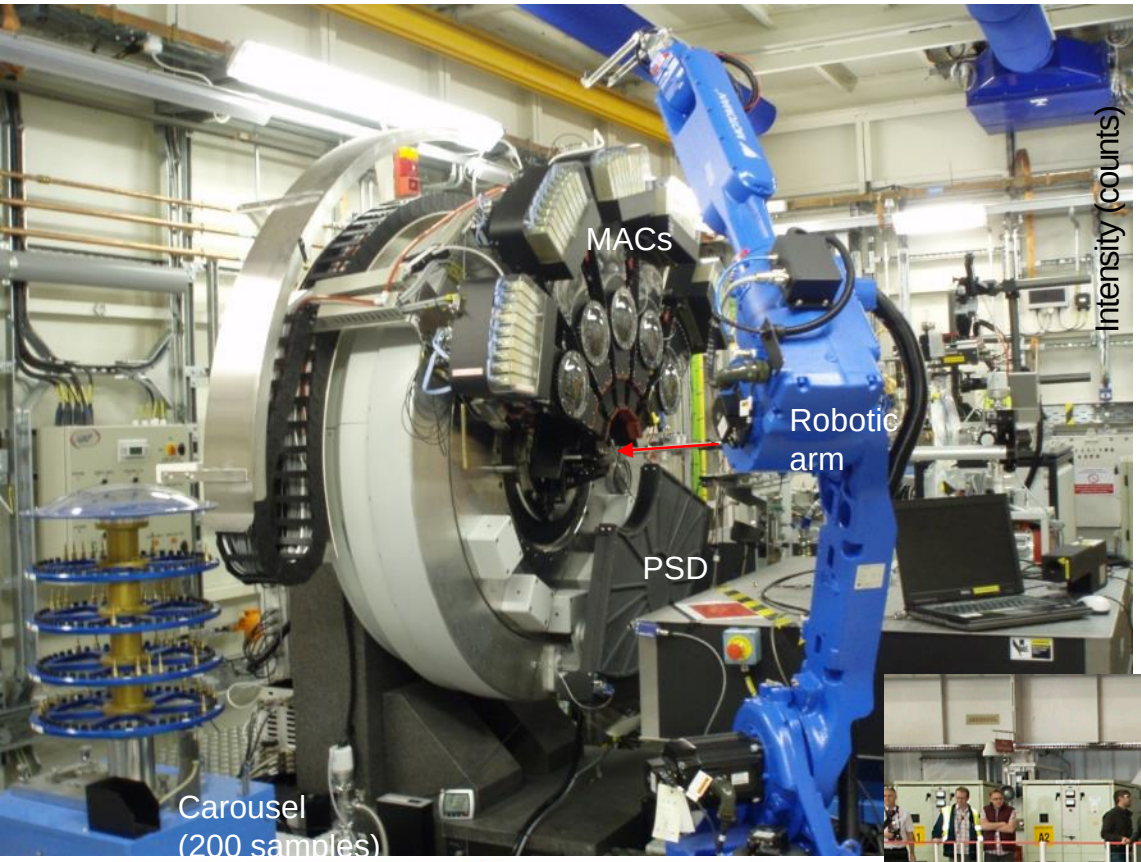


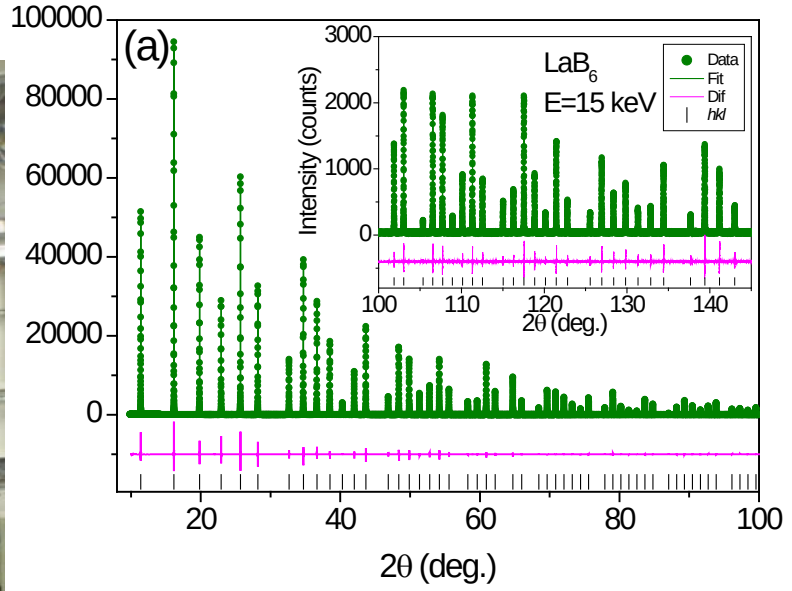
Tomography
Microstructure
Porosity
Strain

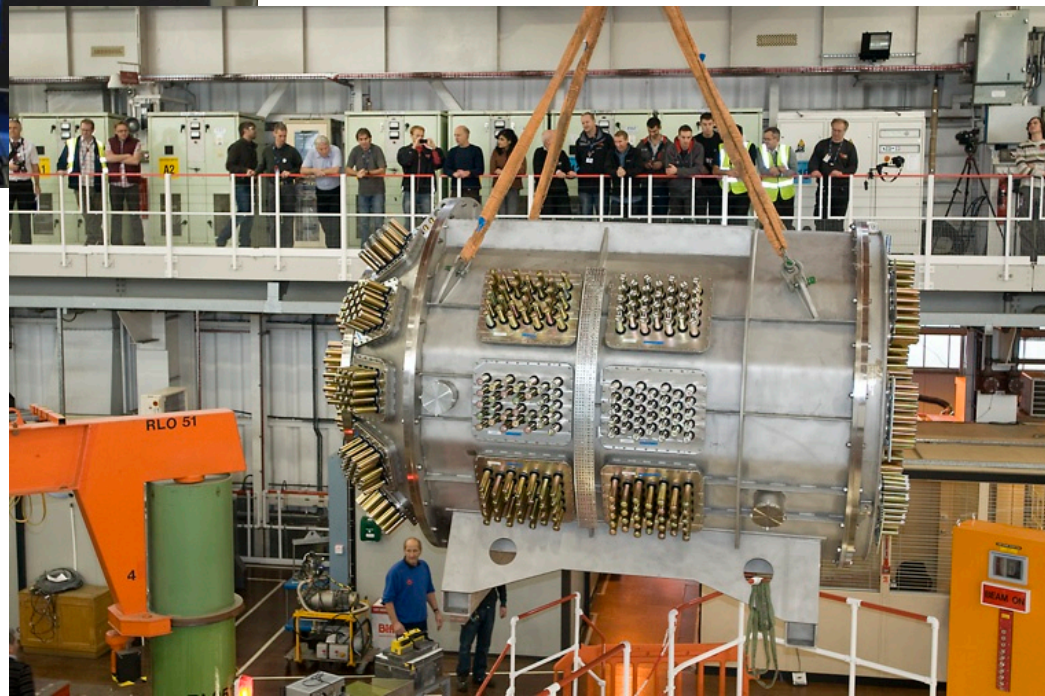


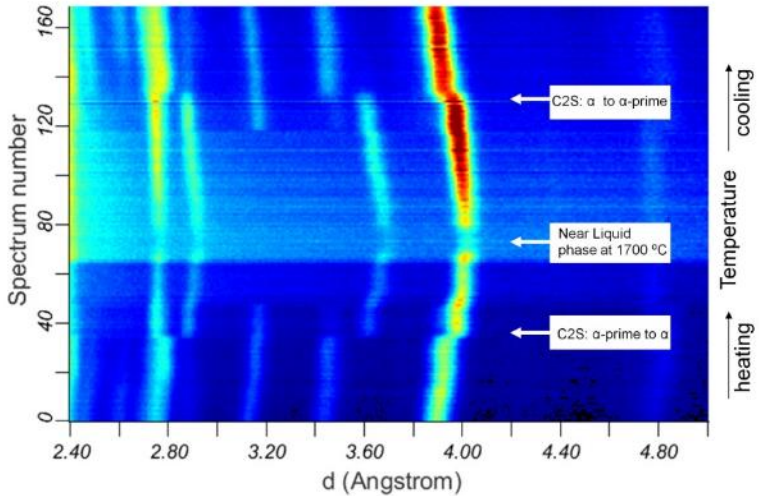
Size: < Å | Å and nm | nm | μm

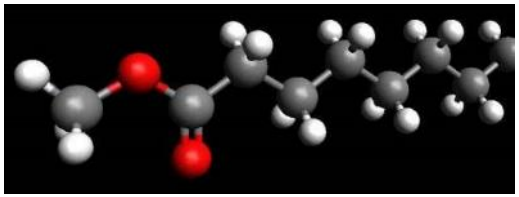
High Resolution Powder Diffraction

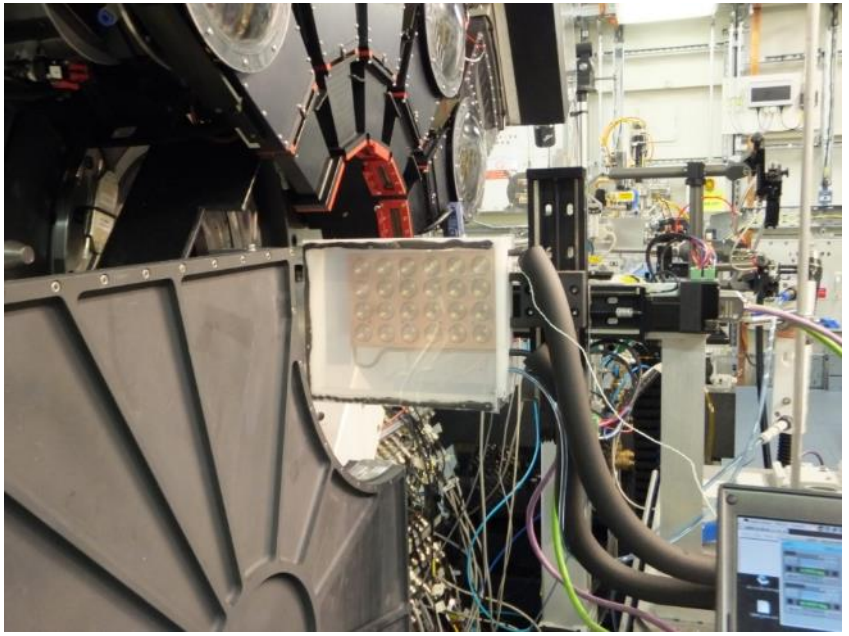










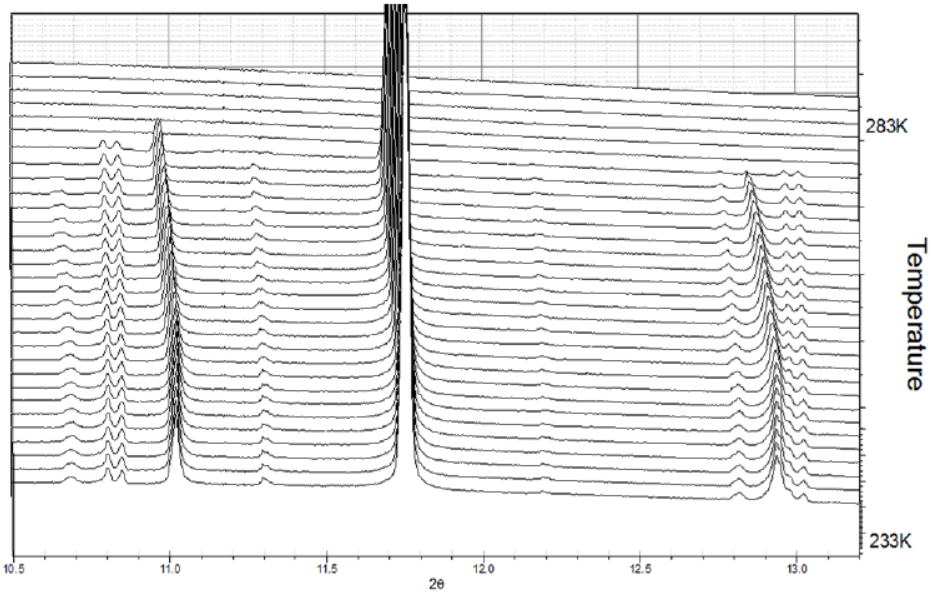


Infineum cell with multi-sample holders (-30°C) on I11

- Introduction of biofuels brings unintended consequences – especially crystallization problems in engines during winter temperatures. It is important to understand the problem so we can cure it.

- Crystal morphologies/unit cells and habit are crucial to crystallisation process.

- Work underway on I11 to study crystal structures and crystallisation, particularly from solution data, and the effect of additives

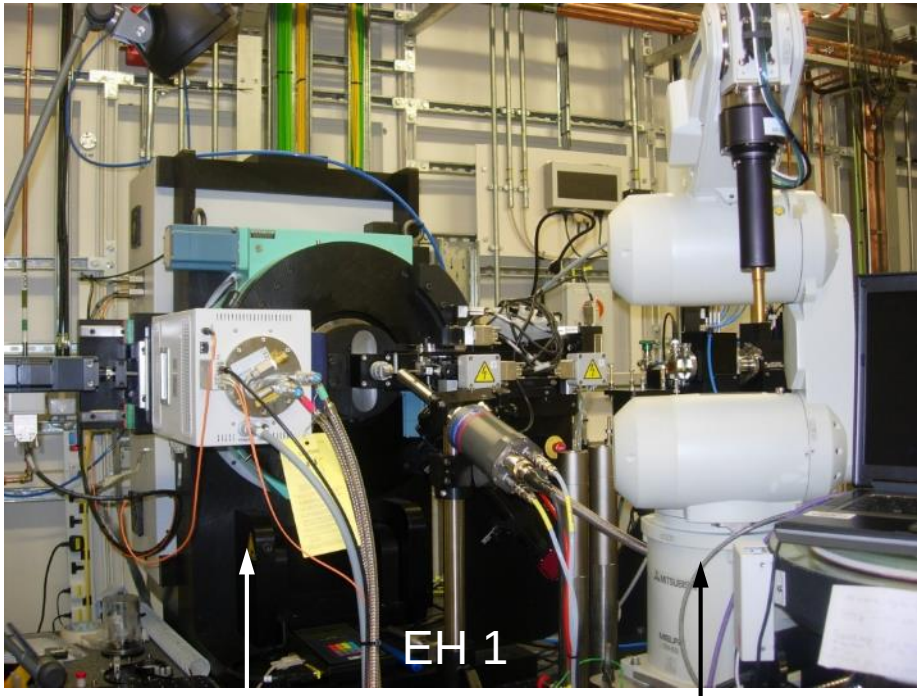


Crystallisation data as a function of temperature

I19 Small Molecule Single-Crystal Diffraction

Mono beam size at sample = 50 - 100 μm^2
Single crystal size ~ 20 – 100 μm

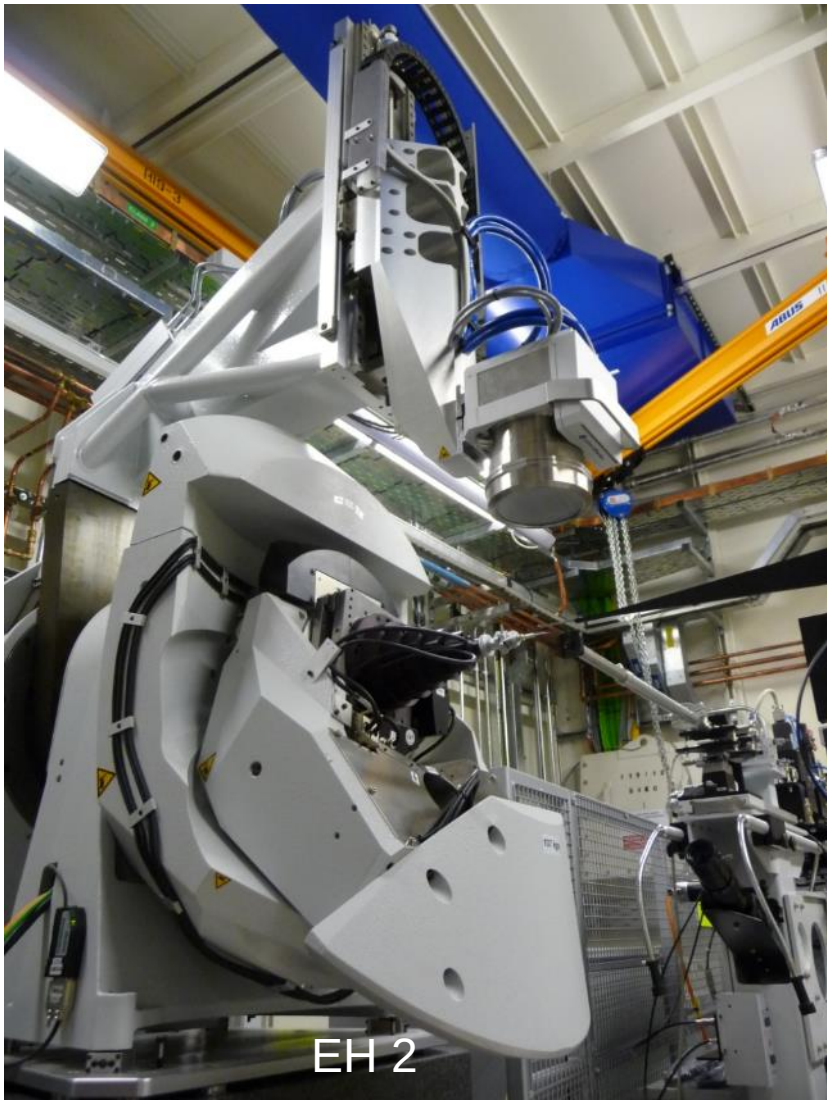
Experimental hutch 1 (EH1) – for relatively “conventional” structural chemistry – albeit on extremely challenging systems (small weakly diffracting samples with poor crystallinity).



4-circle diffractometer

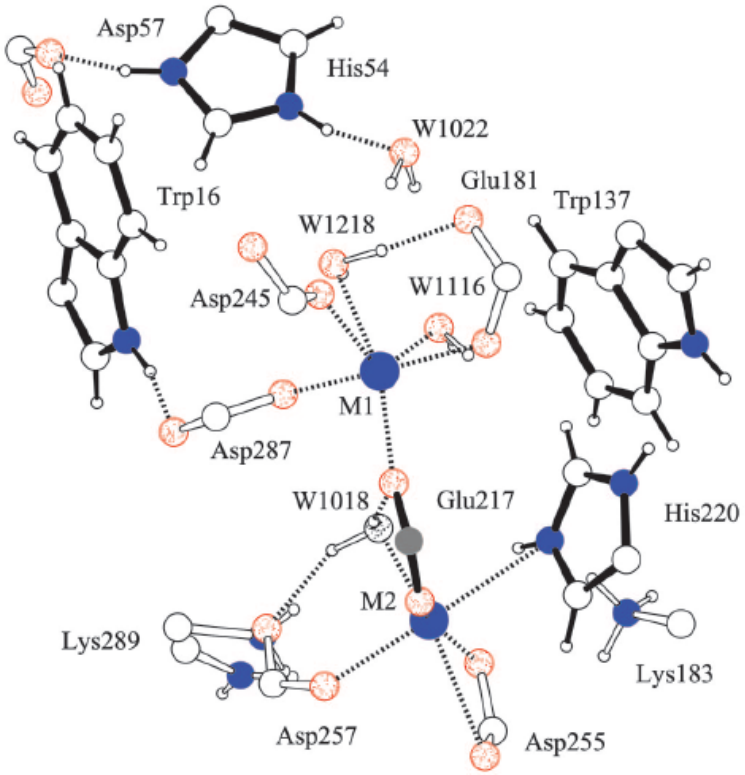
Robotic sample changer

Experimental hutch 2 (EH2) – large heavy duty 4-circle diffractometer for large, bulky, sample environment cells (cryostat, pressure or chemical cell, others)

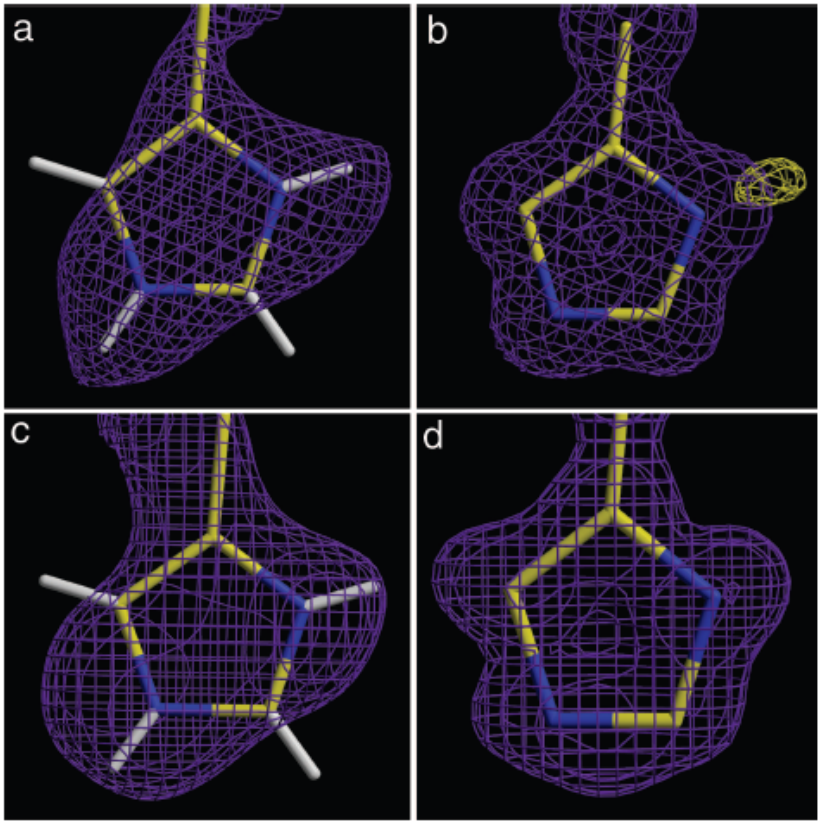


Locating active-site hydrogen atoms in D-xylose isomerase: Time-of-flight neutron diffraction

Amy K. Katz[†‡], Xinmin Li[‡], H. L. Carrell[†], B. Leif Hanson[§], Paul Langan[¶], Leighton Coates[¶], Benno P. Schoenborn[¶], Jenny P. Glusker[†], and Gerard J. Bunick[‖††‡‡]



Crystal structure, active-site and hydrogen position determination

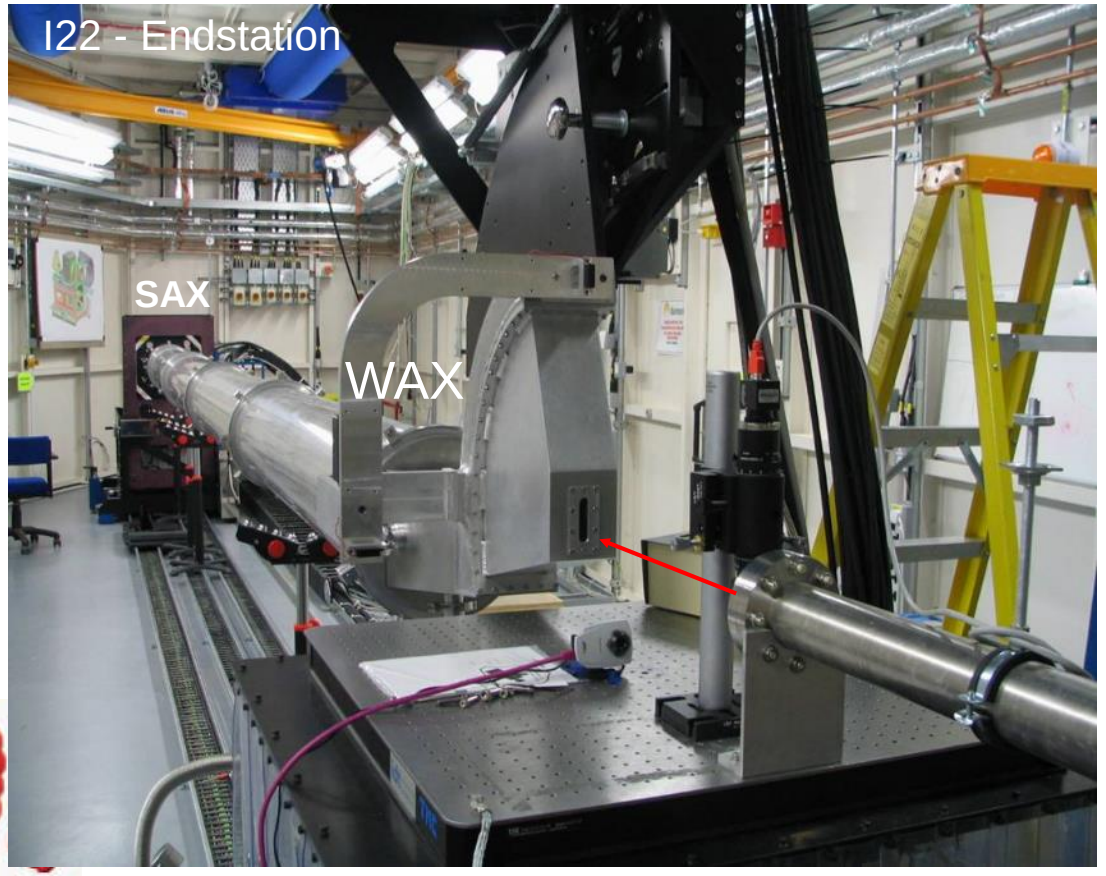


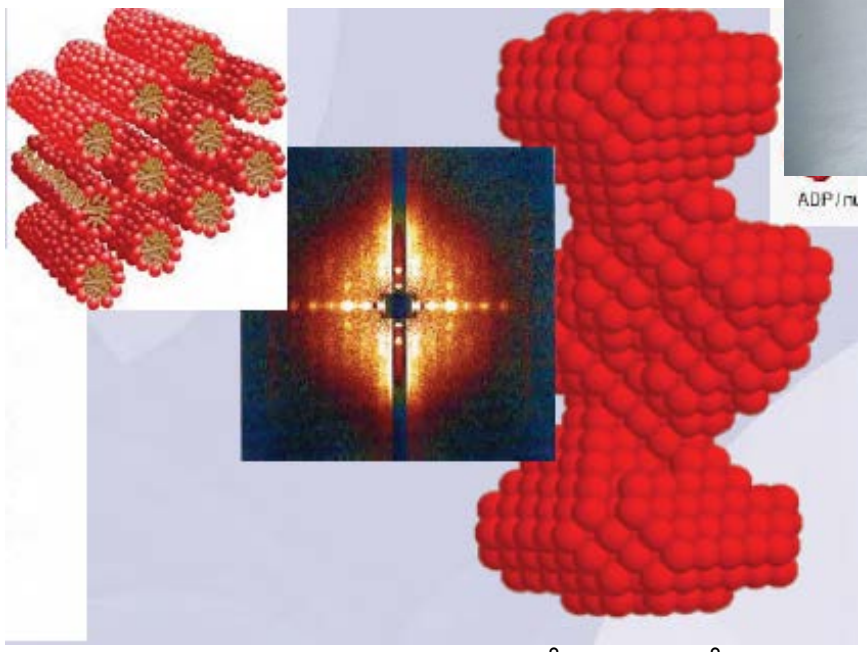
Nuclear density maps (a,c) and electron density maps (b, d) unambiguously identify deuterium atoms (white)

Beamline I22 - Non-Crystalline Diffraction (NCD)

What information?

NCD provides information on the structure and dynamics of large molecular assemblies in low ordered environments.





Length scale 10Å - 10000Å (1µm)

These are characteristic of many complex materials such as biofuels, polymers and colloids….

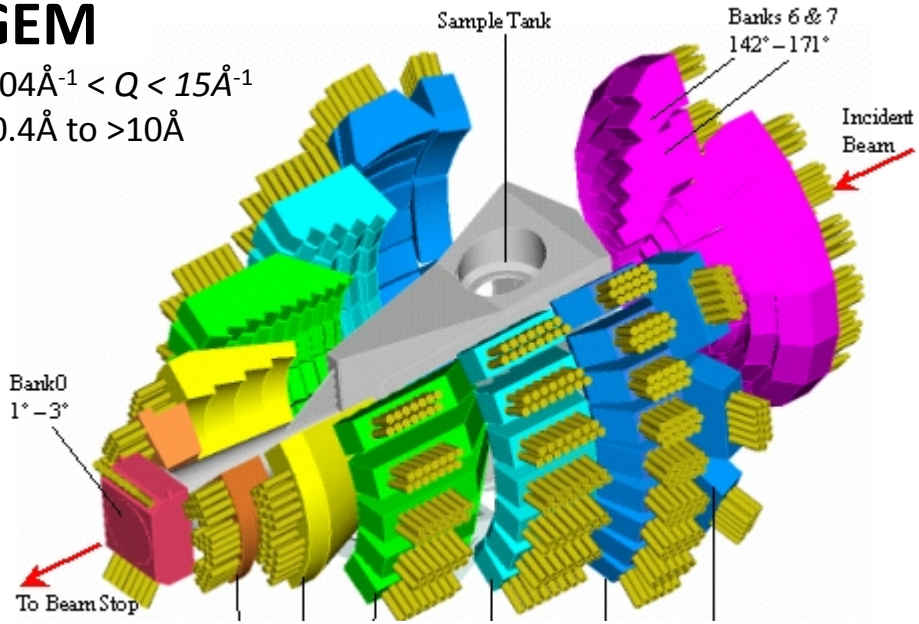
Techniques: Small and Wide Angle Scattering (SAXS & WAXS)

Total Scattering Instruments

GEM

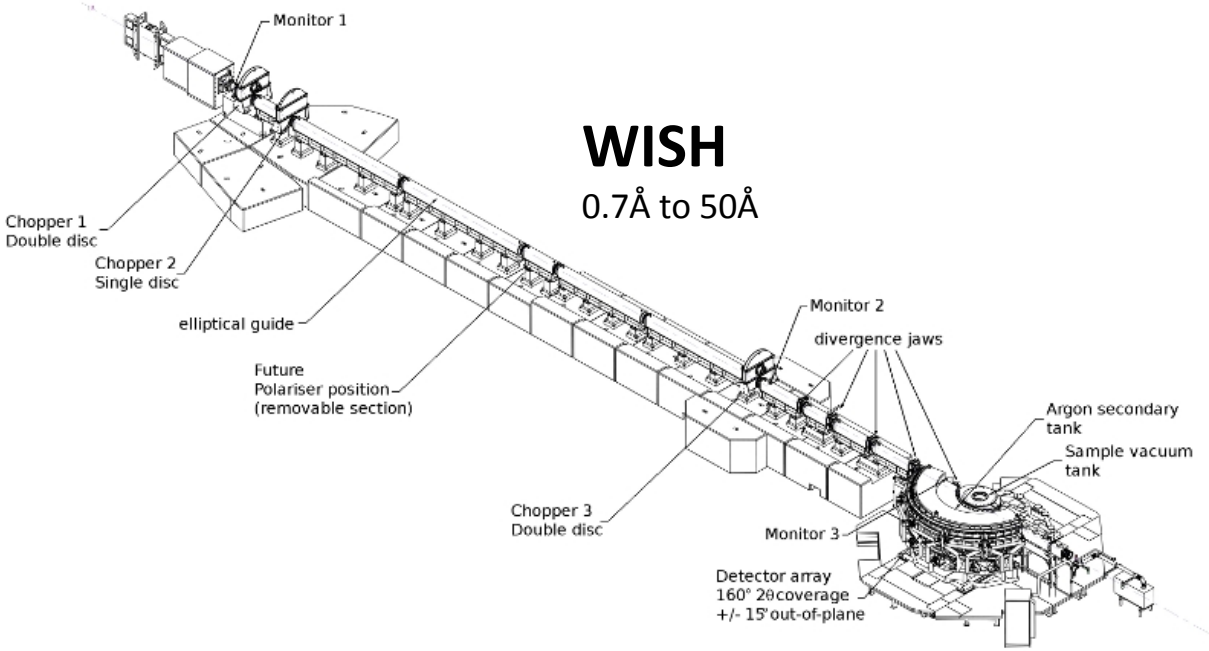
$0.04Å^{-1} < Q < 15Å^{-1}$

~0.4Å to >10Å



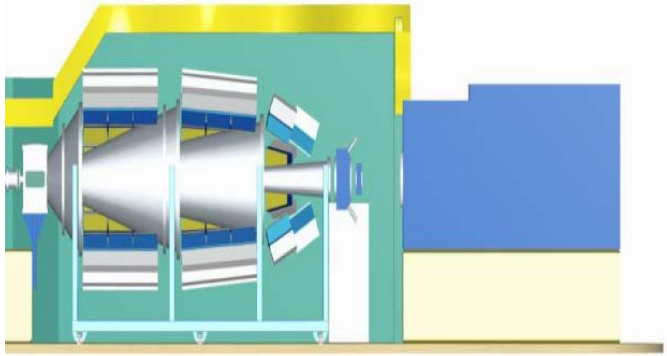
POLARIS

0.2Å to 21Å



WISH

0.7Å to 50Å



NIMROD

$0.01Å^{-1} < Q < 50Å^{-1}$

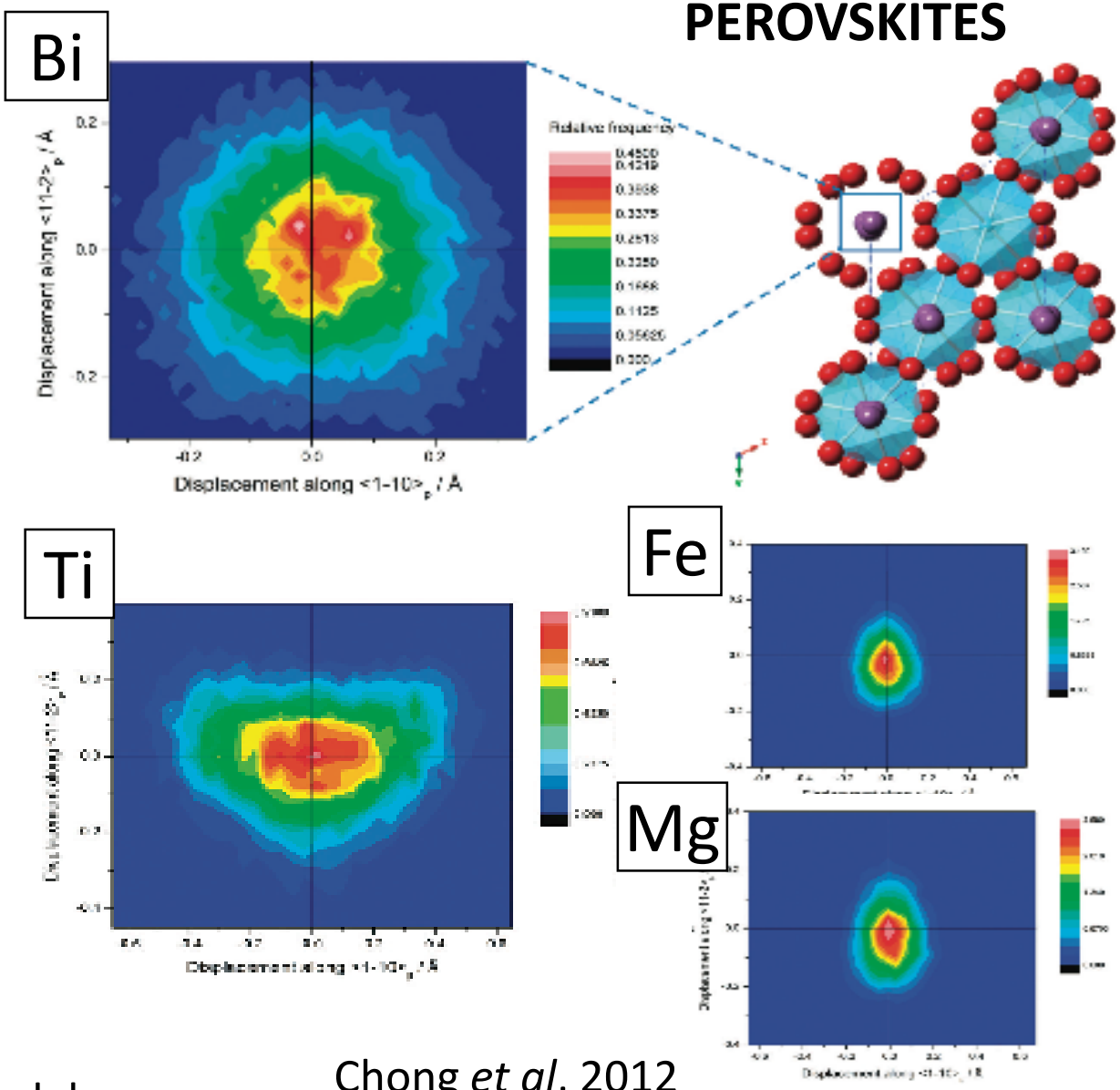
<1Å to >300Å

GEM : LOCAL STRUCTURES IN PEROVSKITES

$Bi(Ti_{3/8}Fe_{2/8}Mg_{3/8})O_3$ possible lead-free replacement for PZT piezoelectric material.

RMC modelling of provide insights into several unexplained aspects of earlier crystallographic studies.

Locally monoclinic clusters accounts for micro-strain broadening required for Rietveld refinement using the rhombohedral structural model.

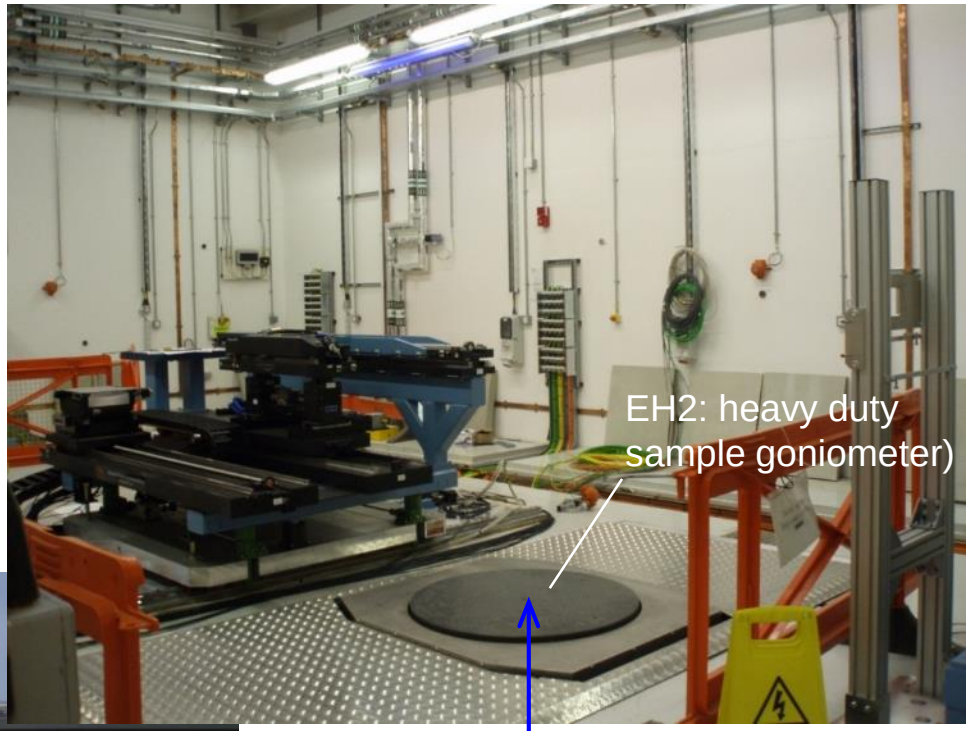


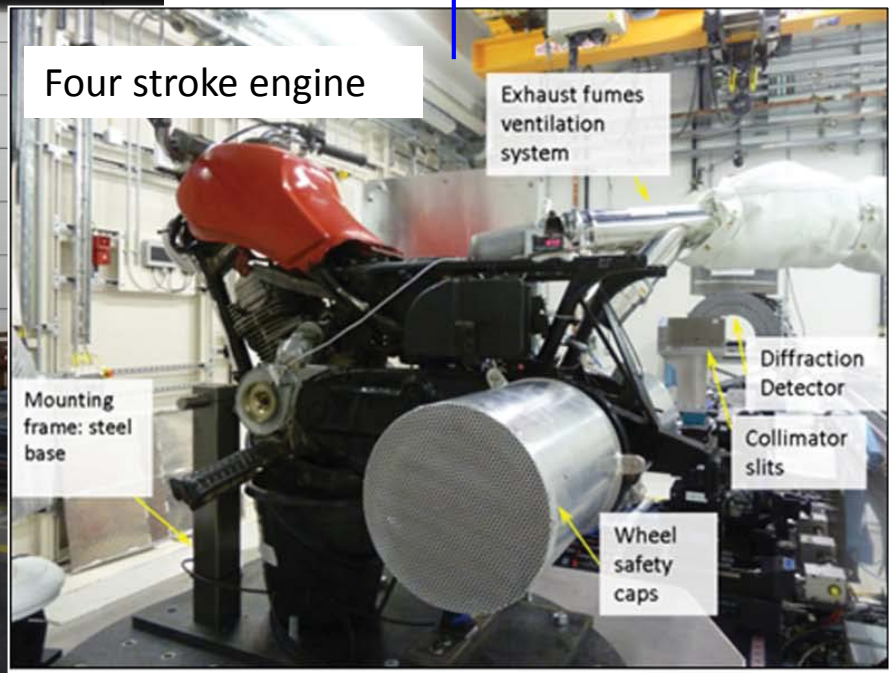
Chong *et al*, 2012

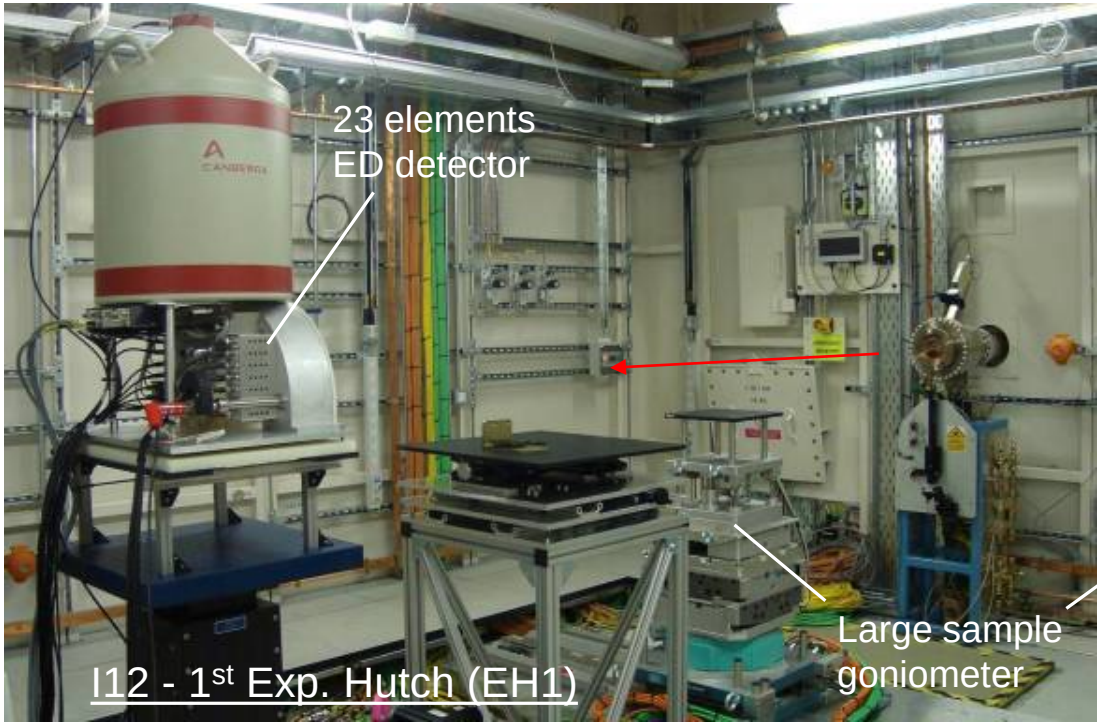
I12 Joint Engineering Environment and Processing (JEEP)

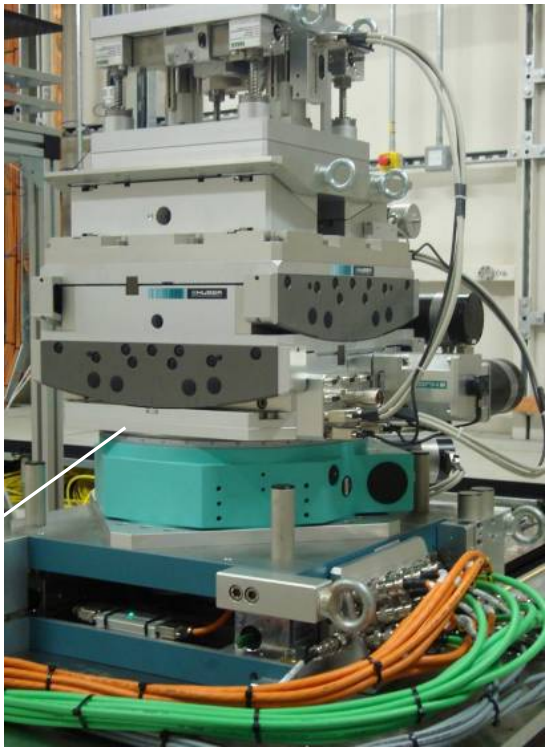
High energy X-rays - 50 keV – 150 keV (0.28 Å - 0.08 Å)

X-ray tomography, difffraction and scattering (white or monochromatic beam)









Furnace Installed (100-1000ºC)

Heraeus “Nobleight” IR lamp heating elements.

Split furnace fits around load chain and sample.

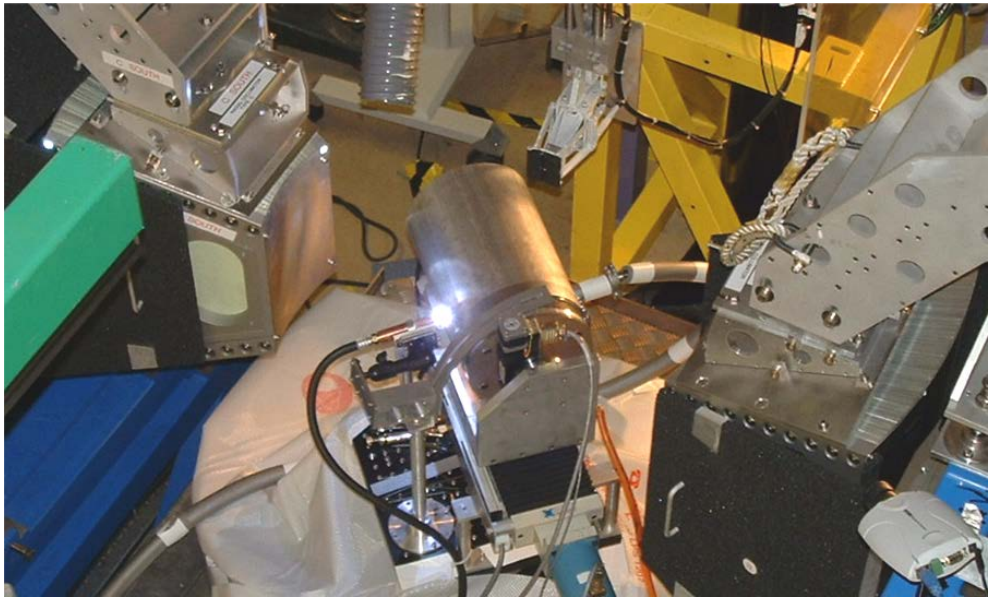
Temperature monitoring with thermocouples

Cold Stage Installed

Two thermo-electric coolers, at top and bottom of sample cup.

temperature range of +20 to -20ºC.

Neutron tomography



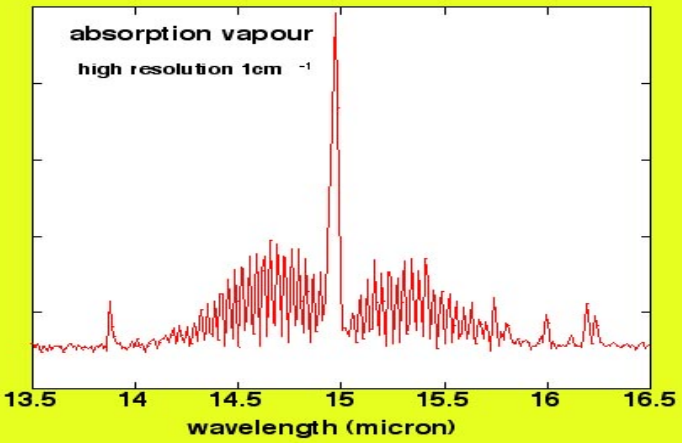
Turski *et al* (2010).

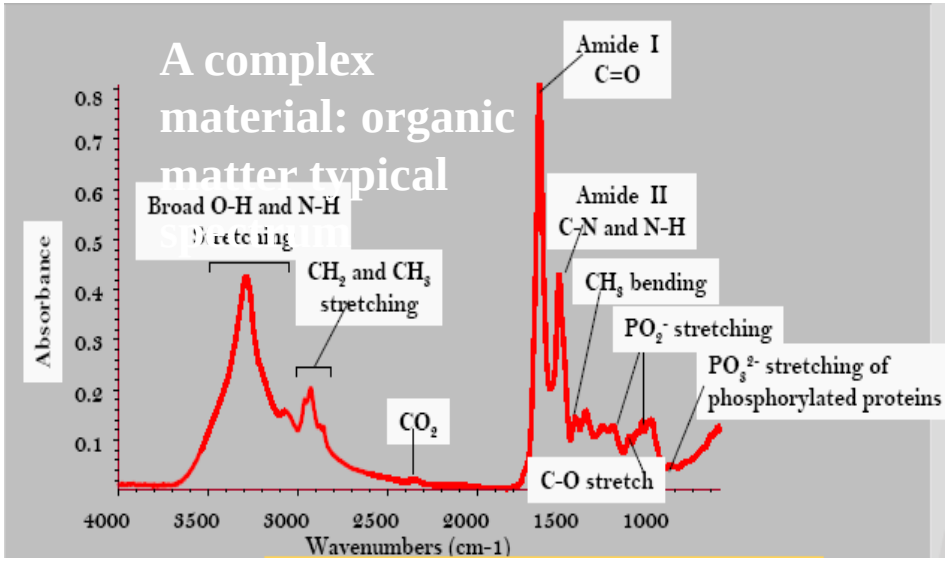
Beamline B22 - Infrared Microspectrocopy

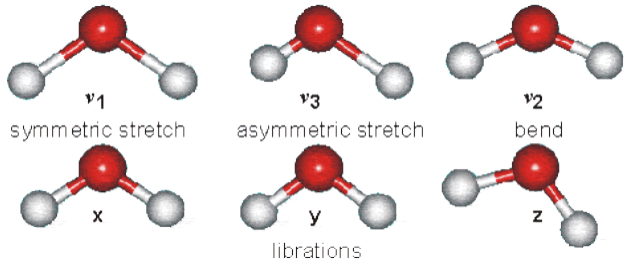
To study molecular structures and their physical, chemical and boilogical properties.

Applications: life sciences, solid state physics & chemistry, forensic, polymer, ….

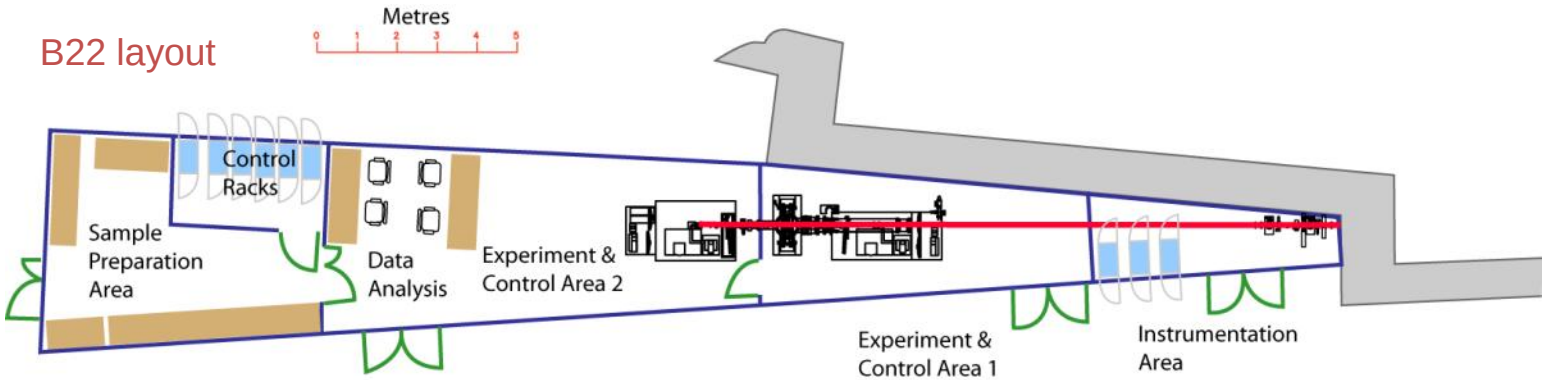
A simple gas: water vapor



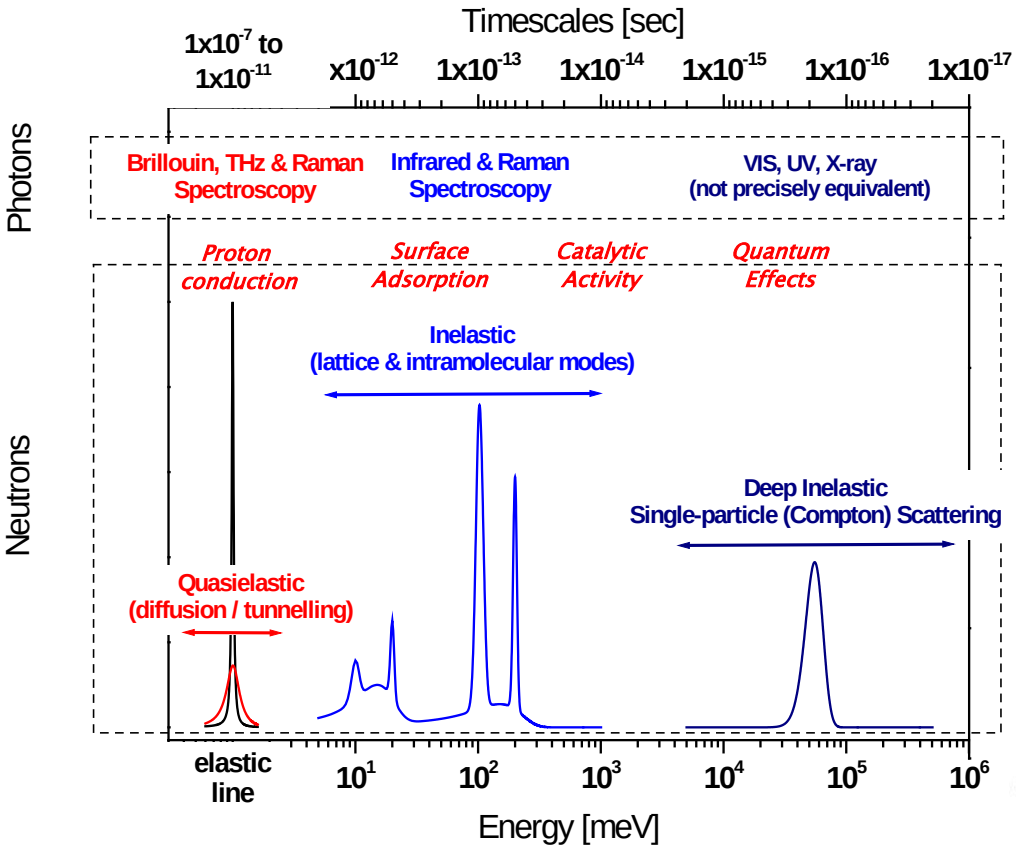


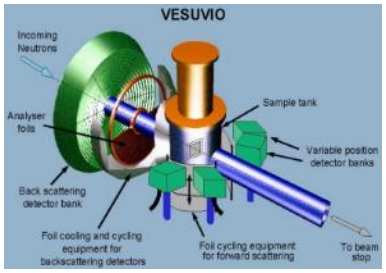


B22 layout

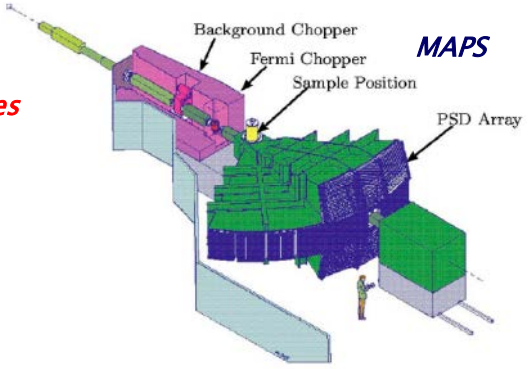


Chemical Spectroscopy at ISIS





Simultaneous high-resolution diffraction

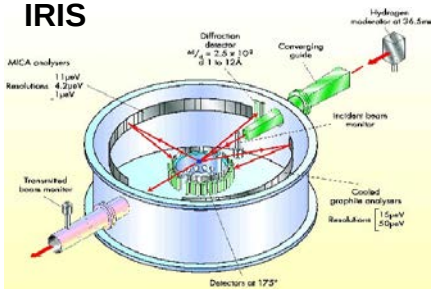


MAPS

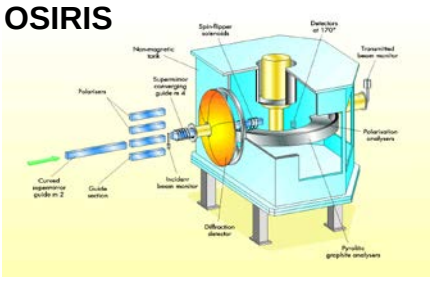
High energies

Widest spectral range in the world

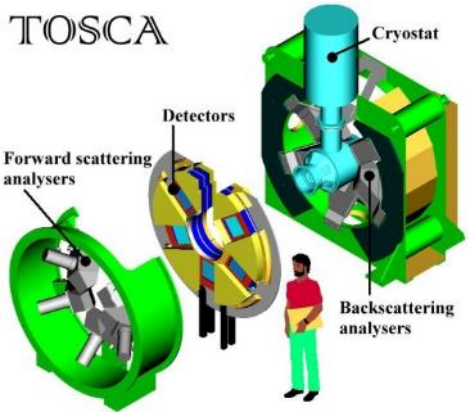
IRIS



OSIRIS



Low energy (Brillouin, THz)



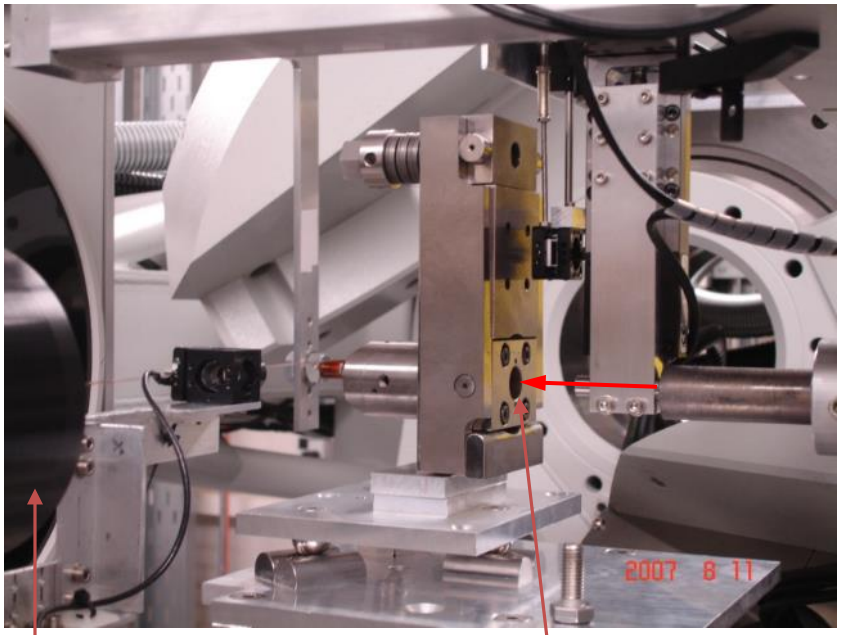
Intermediate energies (infrared, Raman)

I15: Extreme conditions

White and monochromatic

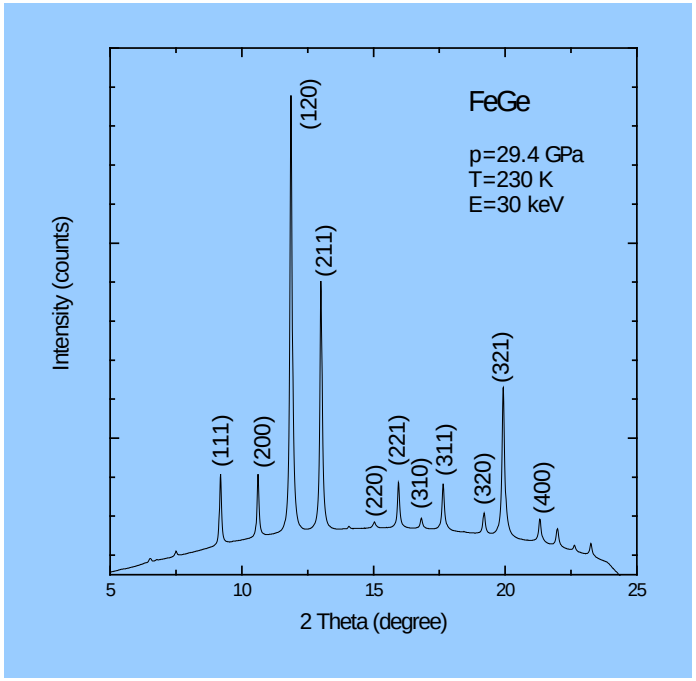
E = 20 -80 keV

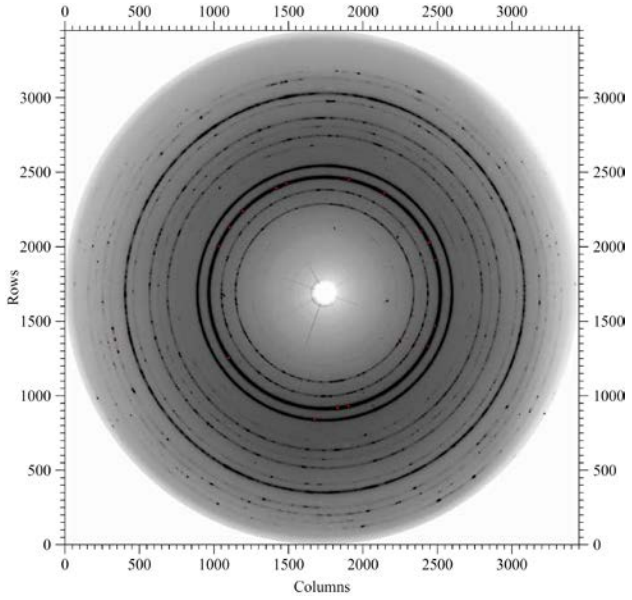
Beam size at sample: 30 – 80 μm^2

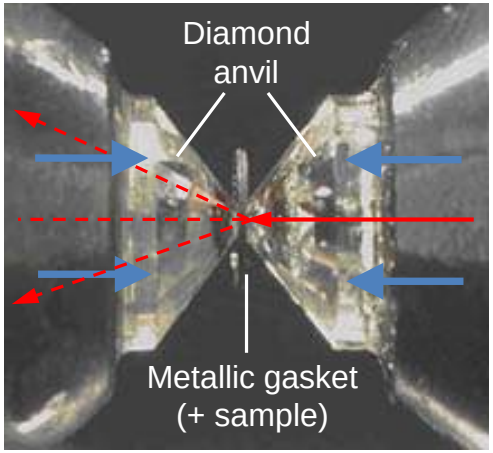


2D detector

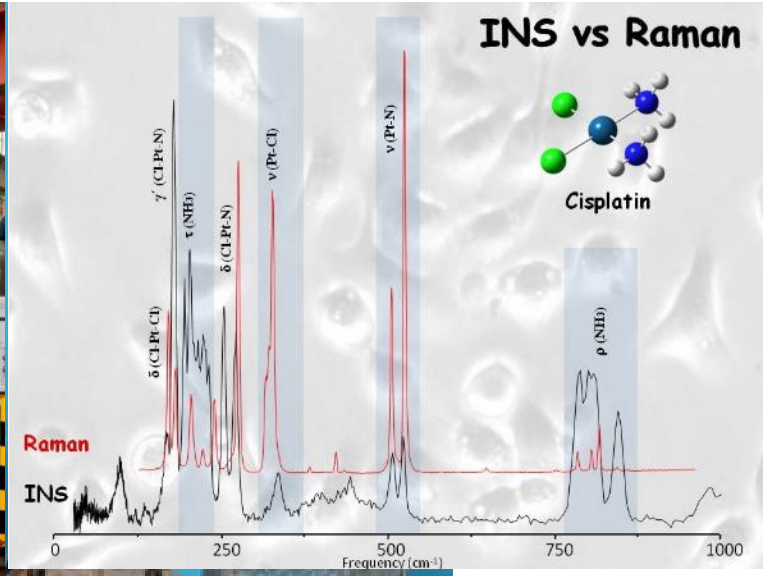
Diamond anvil cell (DAC)

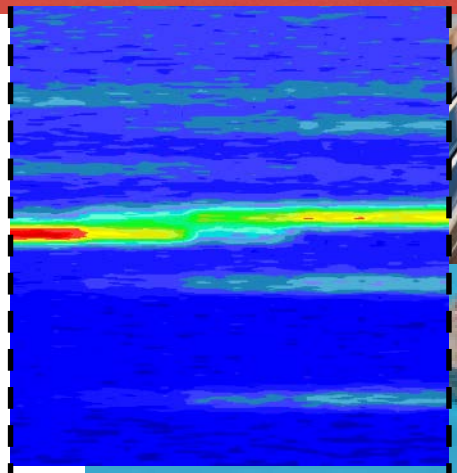




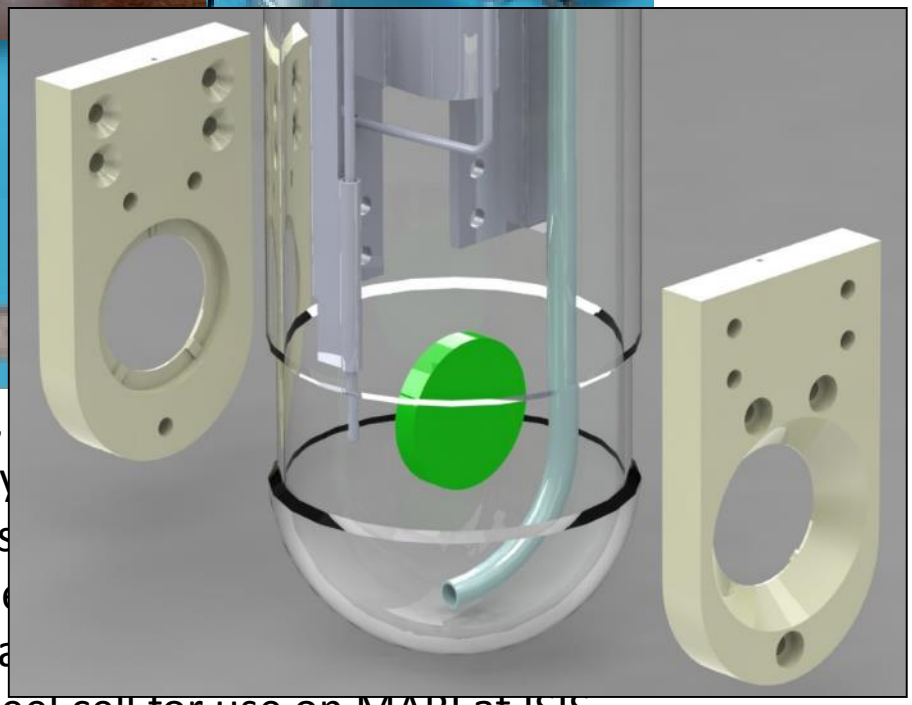


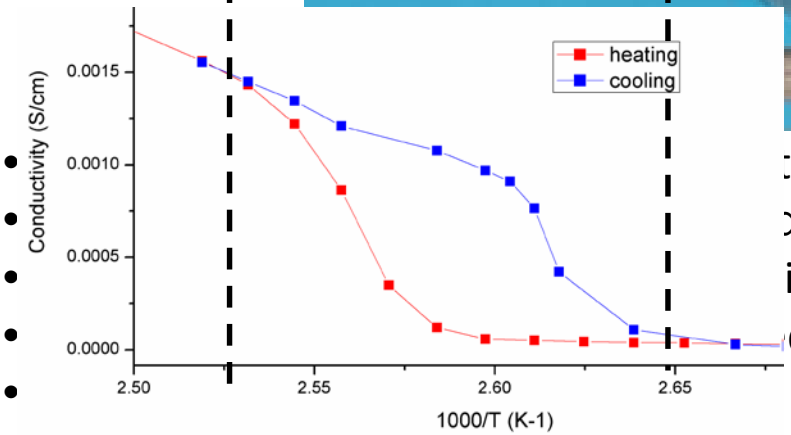
In-situ





d-spacing





- hrough,
- d Zircaloy
- ium cells
- d sample
- wire sea
- (h) flow-through, Conflat™ sealed stainless steel cell for use on MARI at ISIS.

I11 Long Duration Experiments (LDE)

- Slow dynamics in natural systems, engineering processes and energy materials
  - Important information on the development of phases over time cannot be obtained via *ex-situ* methods
- Use LDE to periodically monitor the experiment *in-situ* over longer timescales (weeks, months, years...)

Areas of Scientific Interest

- Energy
- Catalysis
- Environmental
- Radiation exposure
- Pharmaceuticals
- Metallurgy and alloys
- Gas storage materials
- Corrosion

Project key milestones

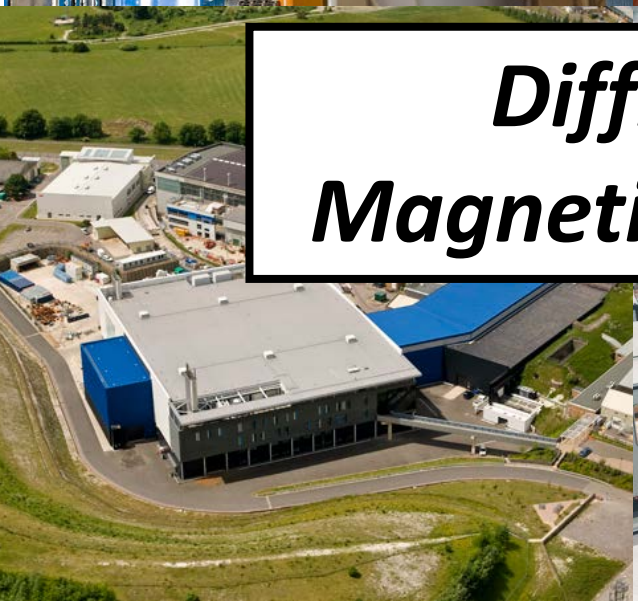
- Start of project (Oct 12) **√**
- LDE Hutch built (Jul 13) **√**
- Cabin and Service (Sep13) **√**
- Diagnostic & Shutter (Jan 14) **√**
- PSS system (Mar 14) **√**
- 1st beam (Apr 14) **√**
- Test samples with beam (Jul 14) **√**
- 1st Users and LDE expt (Oct 14) **√**

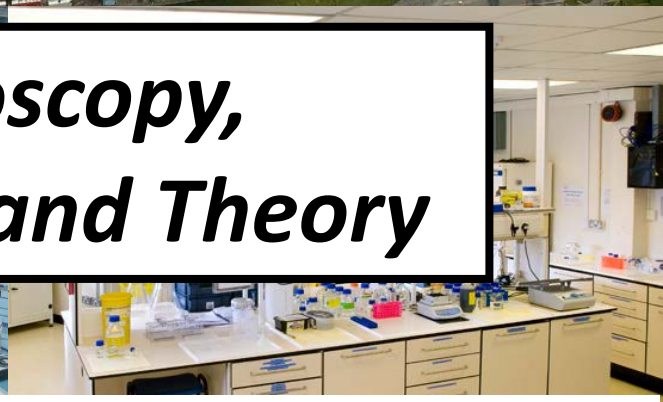
Joint ISIS –Diamond CDT Training School

9th – 15th March 2015

Neutrons
X-rays
Muons

Diffraction, Spectroscopy, Magnetism, Modelling and Theory





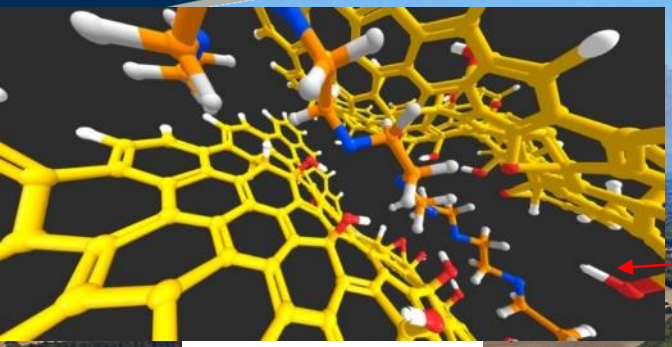
Science & Technology Facilities Council
Central Laser Facility

Science & Technology Facilities Council
ISIS

diamond

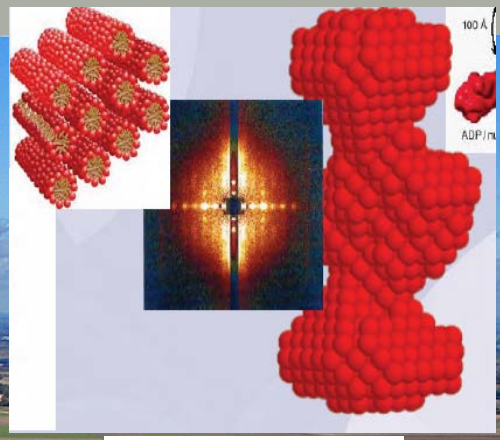
Science & Technology Facilities Council

Harwell Science & Innovation Campus

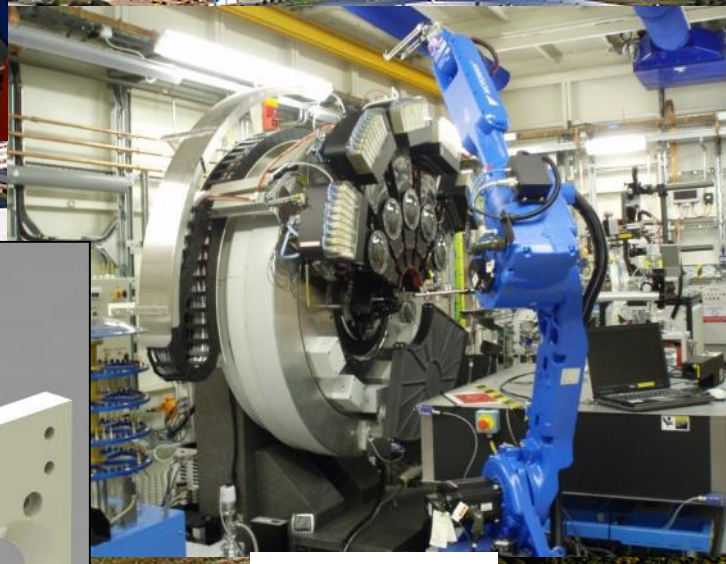


Spectroscopy

Tomography

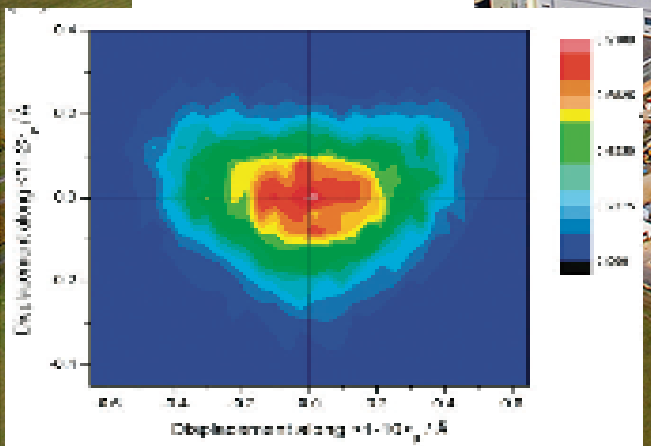


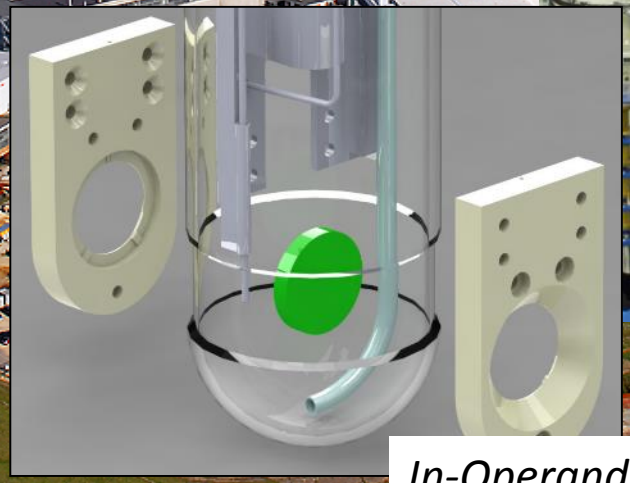
Single Crystal



Diffraction

Total Scattering





In-Operando