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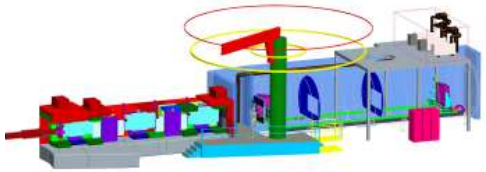
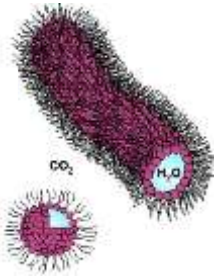
S_{mall}-A_{ngle} N_{eutron} $S_{cattering}$ at ISIS: Applications to Food Science

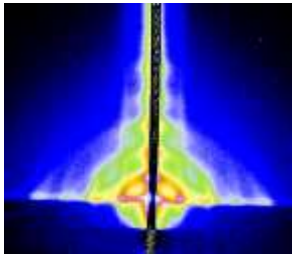
Sarah Rogers

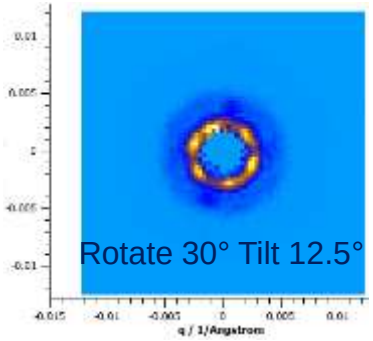
Overview:

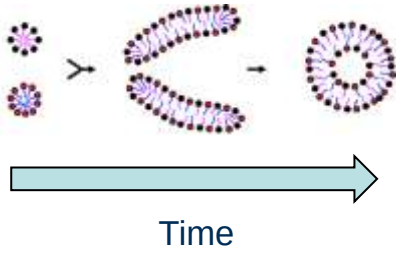
- What's is SANS
- The Instruments
- Sample Environment
- Science examples:
  - Viscosity modifiers
  - Colloidal Crystal
  - Time-resolved measurements
  - Pin-a
- Summary
- Acknowledgements











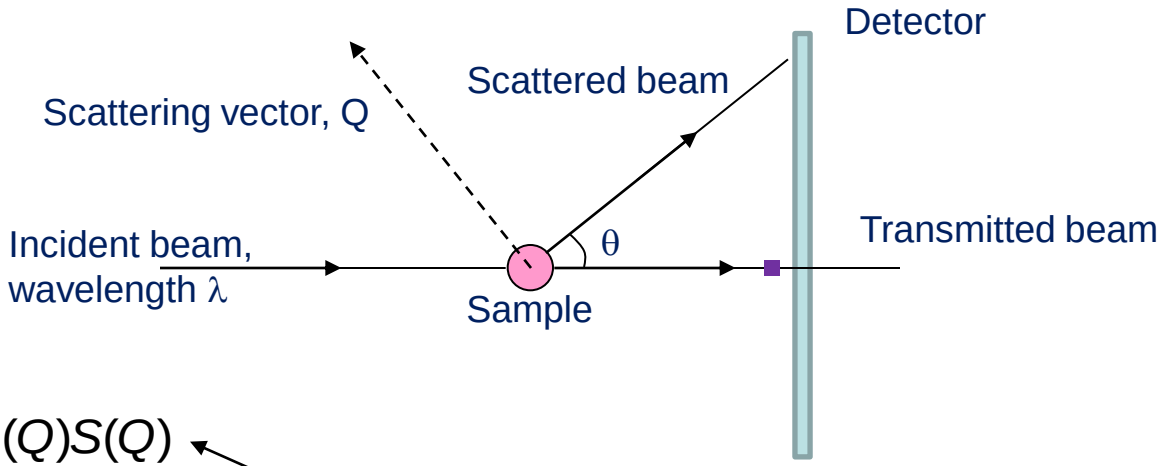
What is S$_{mall}$-A$_{ngle}$ S$_{cattering}$?

Can determine the size, polydispersity and structure of a wide range of disordered materials

Covers a size range of ~ 1 – 200 nm

$$Q = \frac{4\pi \sin(\theta/2)}{\lambda}$$

$$Q = \frac{2\pi}{D}$$



$$I(Q) = (\rho_P - \rho_M)^2 N_P V_P^2 P(Q) S(Q)$$

Form factor: intra-particle information - size and shape of particle

Structure factor: inter-particle information. Depends on the type of interactions in the system. S(Q) = 1 for dilute dispersions

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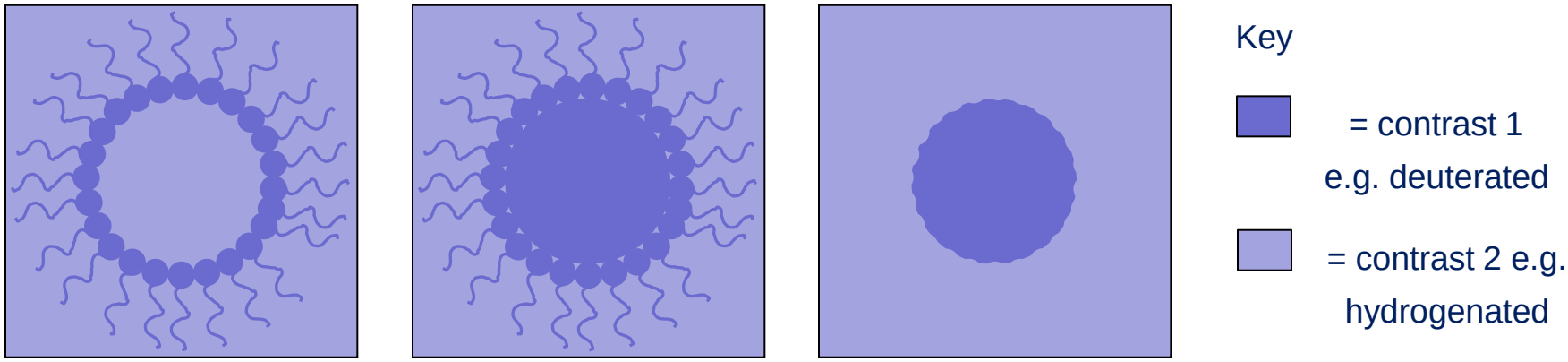
Bonuses of Neutron Scattering and SANS at ISIS!

Unique insights from Contrast Variation



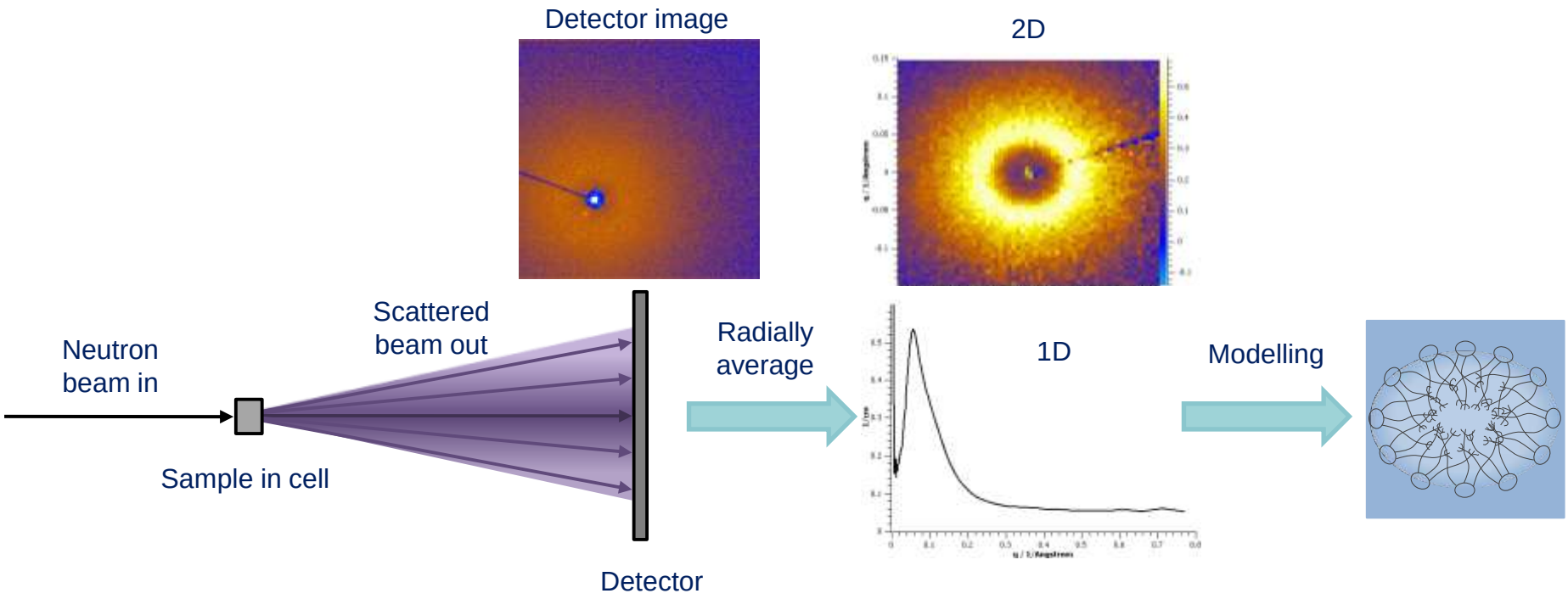
Shell contrast Drop contrast Core contrast

Contrast Variation *via* selective deuteration allows core, shell and drop of aggregates to be studied

Time of Flight SANS

Q is varied by changing either the wavelength of the neutrons or the angle. A large simultaneous Q-range is achievable (without moving detectors i.e. angle) on a spallation source as the white beam delivers a broad wavelength range of neutrons. This is very useful for time-resolved measurements

‘Typical’ Experiment



Sample held in neutron transparent cell – quartz, sapphire are common for SANS

Typical detector image and 1D scattering pattern of a micelle

Radially average, background subtraction and normalisation to give Intensity vs. Q plot

Known approximations and mathematical modelling used to fit such data – size, polydispersity and volume fraction – www.small-angle.ac.uk/

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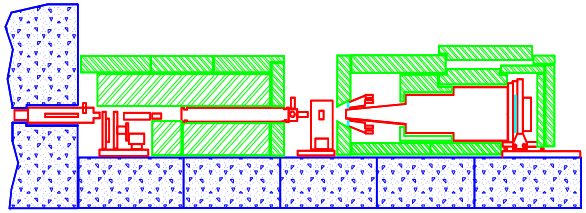
The Instruments

Loq

1st SANS beamline at ISIS. Situated on the 1st target station TS-1.

Fixed L_2 = 4m. Q-range of 0.007 – 0.3 Å^{-1} on main detector - this can be extended to 1.4 Å^{-1} by employing the wide-angle detector bank



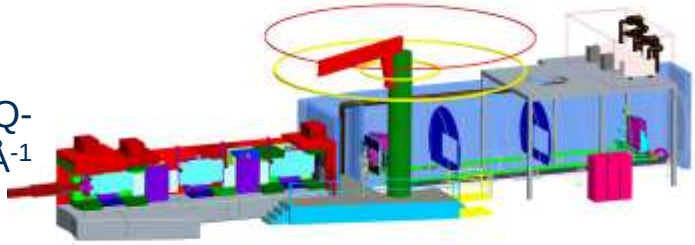
Contact: stephen.king@stfc.ac.uk

Sans2d

1st SANS beamline on the 2nd target station TS-2.

Movable detectors and neutron guides match the Q-range to the science. Q-range of 0.001 – 3 Å^{-1} achieved using 2 x 1m^2 detectors

Greatly improved flux and S/N compared to Loq

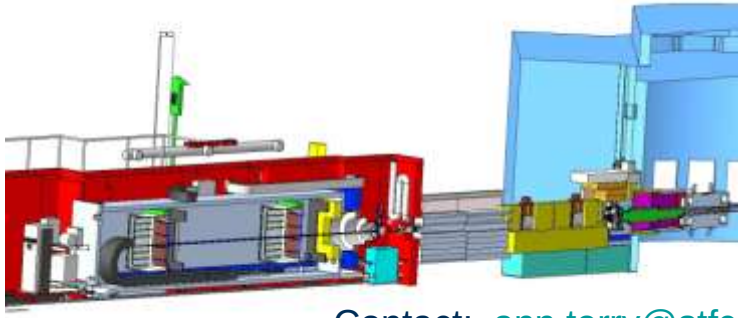


Contact: sarah.rogers@stfc.ac.uk

ZOOM

Phase 2 beamline on TS-2. Building!

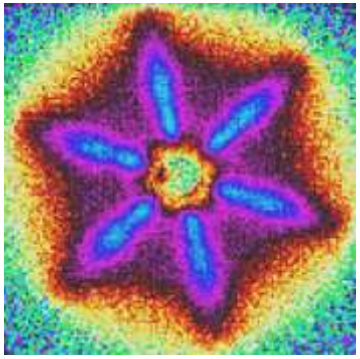
Focussing will allow ultra low Q (0.0003 Å^{-1} ~ 2 μm). Will also have polarisation option.



Contact: ann.terry@stfc.ac.uk

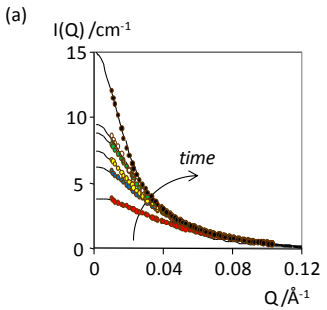
The Sample Environment

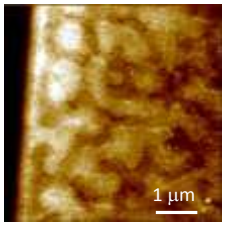
Extensive available sample environments allow a broad range of science to be studied via SANS at ISIS. The Sans2d sample area has been designed with industrial partners so large kit can be accommodated

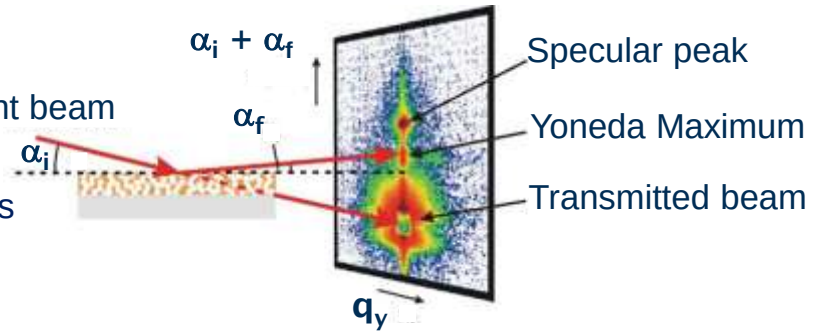


Sample environment includes:

- Standard ISIS cryostats, furnaces and magnets
- Sample changer with temperature control
- Rheometer and shear cells
- Pressure cell – 600 bar with stirring. Predominantly used with CO_2
- T-jump cell – study non-equilibrium phases
- *In-situ* DLS and UV-vis
- Grazing Incidence SANS (GISANS)
  - Nanoscale density correlation and/or shape of
  - Nanosized objects at surfaces, buried interfaces or in thin films
- Stopped-flow – mixing kinetics
- Well equipped offline labs allow for further characterization – X-ray sets, AFM, BAM, spectrometers







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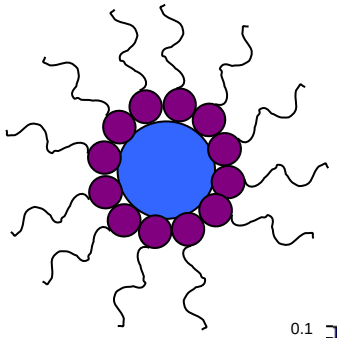
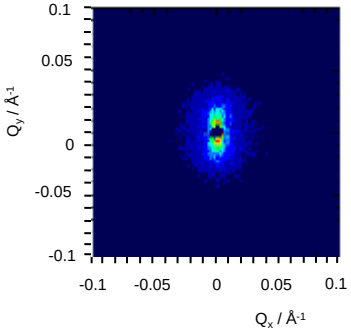
The Science

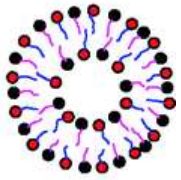
For the next two cycle alone Sans2d has the following:



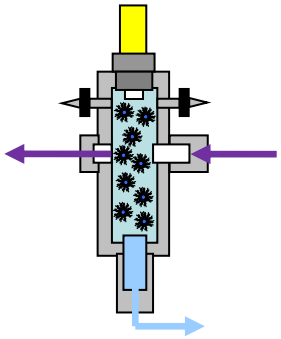
- **Surfactant chemists**
  - Interaction of perfumes with micelles
  - Aggregation in unusual solvents – sc-CO_2 and ILs
  - Thickness of polymer and protein layers on nanoparticles
- **Chemists**
  - The structure of foams
  - Orientation of peptide fibrils and hydrogels aligned with magnetic fields and fibre diffraction



- **Biologists**
  - Solution scattering
  - Growth of fibrils



- **Pharmacists**
  - Movement of drugs through and into vesicle bilayers
- **Polymer scientists**
  - Interfacial structure of polymers at solid-liquid interfaced via GISANS
  - Polymer structure in solution for templating



- **Physicists**
  - Structure of Sr_3HoCrO_6 above and below Neel Temperature
- **Food Scientists?**

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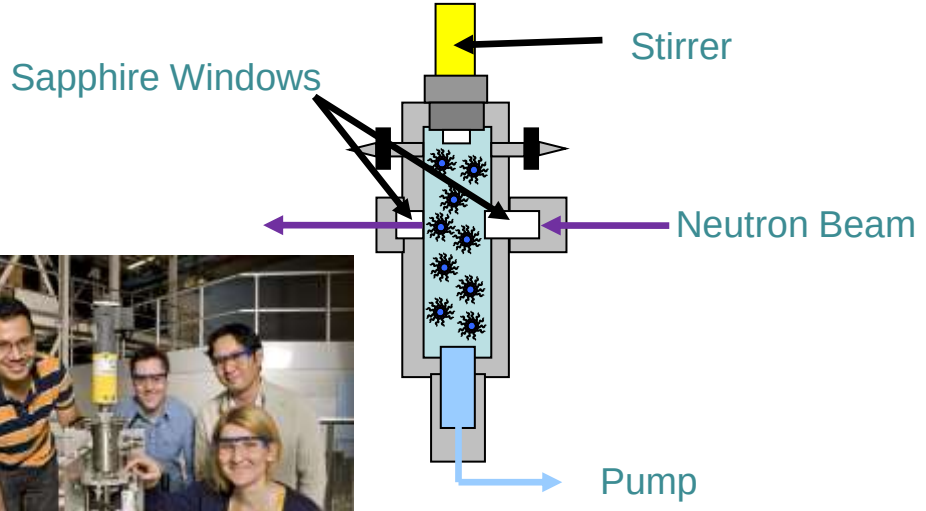
Viscosity Modifiers

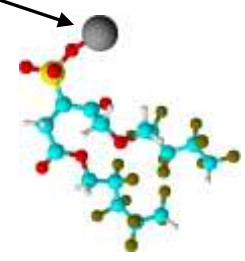
J. Eastoe (University of Bristol) & R. Enick (University of Pittsburgh)

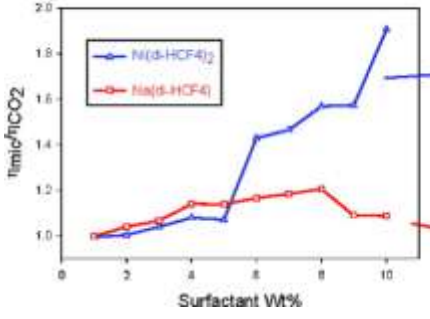
Langmuir **2010**, *26(1)*, 83-88

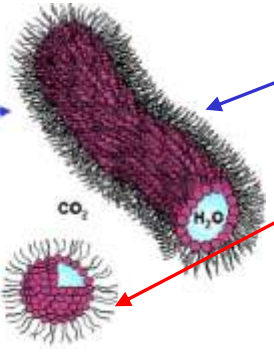
- Studying the use of high pressure CO_2 for enhanced oil recovery using dedicated 600 bar pressure cell
- Low viscosity of CO_2 promotes fingering through porous media rather than a uniform sweep
- Modifiers commonly used in oily solvents are incompatible with CO_2. Can self assembled custom-made surfactants be used?
- Yes! Altering the counterion of the surfactant from Na to Ni or Co causes a viscosity enhancement of up to 90% compared to pure CO2.
- Why? Neutrons have the answer! Use D_2O for contrast

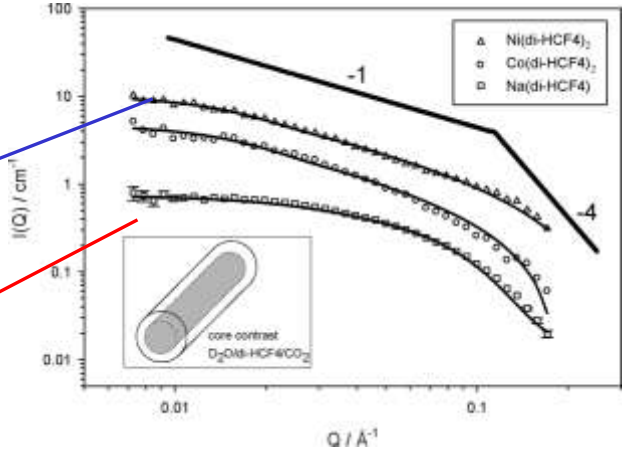


Na^+ or $Co^{2+}n(D2O)$









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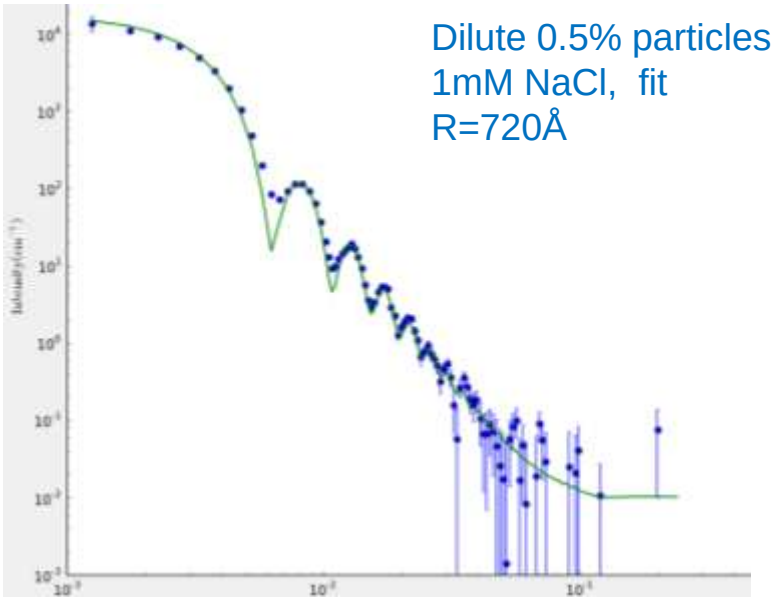
Crystallography with 0.14 micron “atoms”

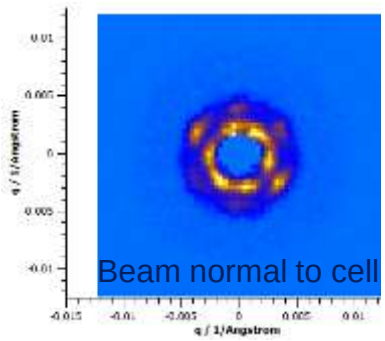
A.Rennie & M.Hellsing (Uppsala)

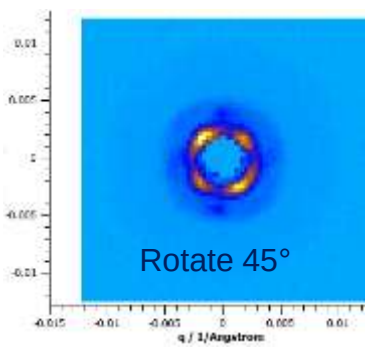
- Understanding the physics of flowing colloidal particles is important for many industrial processes
- Here near monodisperse 0.14 micron diameter polystyrene particles at 8% in water crystallise into domains a few mm in size.
- By rotating and rocking the colloidal crystal in the neutron beam the nature of the packing (fcc, hcp, bcc) and stacking faults can be revealed.
- Data was collected to very small Q at 12m sample to detector on Sans2d with neutrons of wavelength 1.75 to 12.5Å.

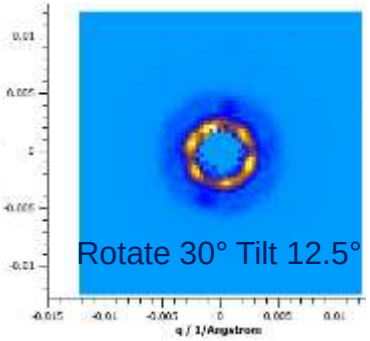


Dilute 0.5% particles, 1mM NaCl, fit R=720Å

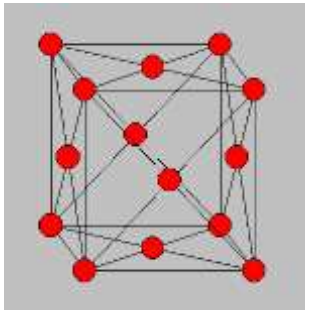
SANS from 8% particles







FCC



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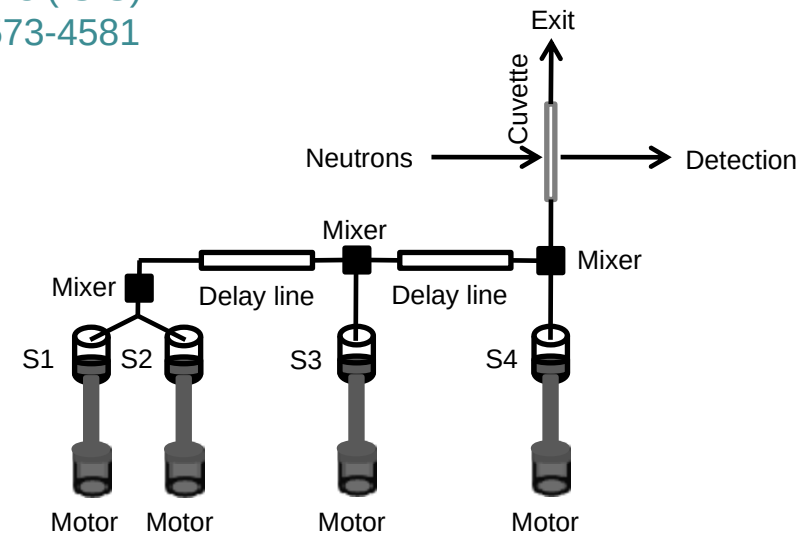
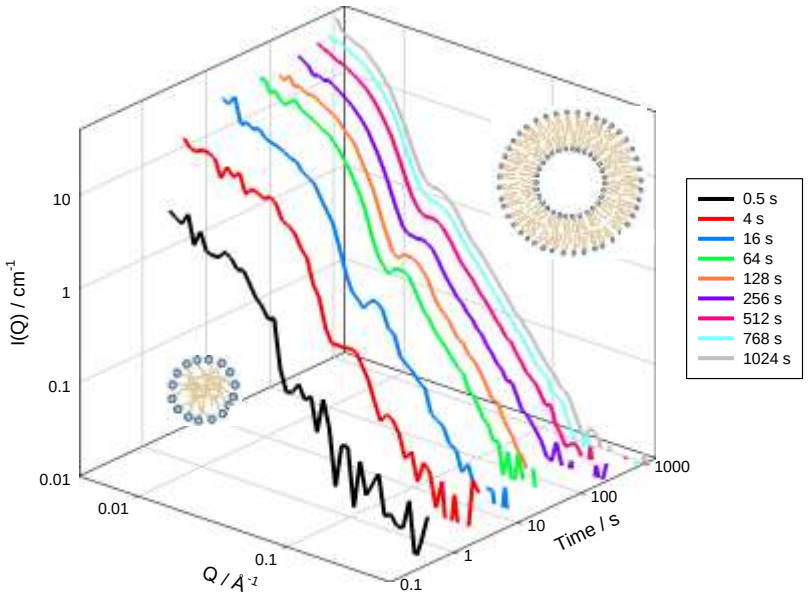
Time Resolved Measurements

I. Grilo (ILL) & S. Rogers (ISIS)

Langmuir **2003**, *19*, 4573-4581

- Stability and mixing mechanisms are important in many processes
- Stopped-flow + SANS allows us to probe nanoscale structural changes on the timescale of ms to hours
- T-O-F SANS is ideal for these measurements as a large lengthscale range can be studied in one shot
- Data collection is synchronized with the kit and automatic cycling is used to improve statistics
- Event mode has now been commissioned on Sans2d – each neutron has it's own time stamp and so data can be 'time sliced' after the measurement has been taken – first time slice here is 0.5s
- Test system studied: AOT + NaCl and shows a micelle to vesicle transition





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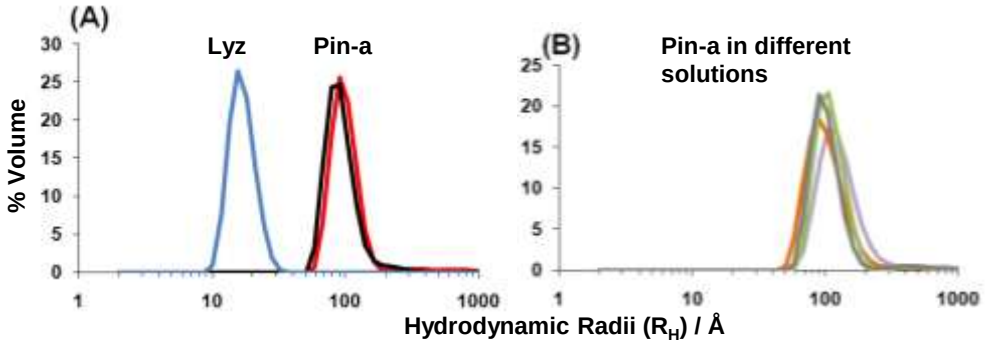
Structure of Pin-a in Solution

L. Clifton (ISIS-STFC) & R. Frazier et al (University of Reading)

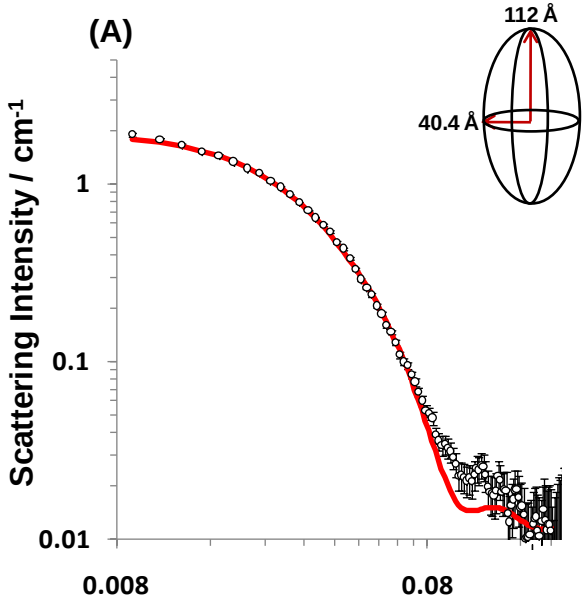
PCCP **2011**, *26*, 8881-8888

- Puroindolines (Pins): small amphiphilic proteins from wheat and barley – role in determining endosperm texture of wheat and possibly in seed defence
- Toxic to a broad range of bacterial and fungal species – perturbs membrane function *via* ion channel formation
- *In vitro* studies with lipid monolayers suggest Pin-a aggregates within lipid monolayers rather than discrete assemblies needed for channel formation
- Use SANS to study Pin-a aggregation in D_2O – ellipsoidal aggregates formed
- Data taken using Xpress scheme

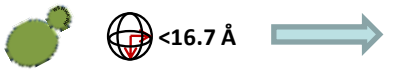
DLS



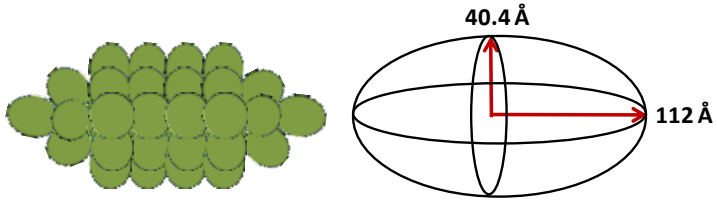
SANS Pin-a in D_2O



Monomer



Micelle



Modes of Access

There are various modes of access for time at ISIS: http://www.isis.stfc.ac.uk/apply-for-beamtime/apply-for-beamtime2117.html

For academics:

Direct access – standard route to apply. Two calls for proposals each year with deadlines 16 April and 16 October. All direct access proposals are peer reviewed by the ISIS Facility Access Panels (FAPs)

Xpress access - no advance peer review for this service, and your beamtime proposal does not require a science case. Samples are sent by courier to ISIS for measurement and fully reduced and corrected high-quality data, ready for analysis, will be provided in return. You are expected to carry out data analysis with minimal assistance from ISIS – excellent for test samples!

For industrial users:

ISIS Collaborative R&D Programme: Contact Christopher Frost (Christopher.Frost@stfc.ac.uk)

- Criteria: Impact + technical feasibility
- Rapid allocation mechanism
- 50/50 STFC/industry funding
- Industry partner supplies impact measures
- Emphasis on Long-Term partnerships

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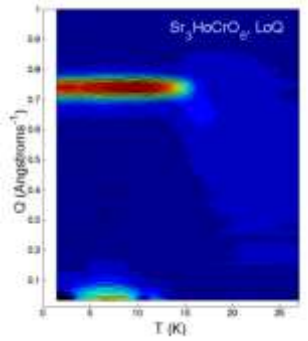
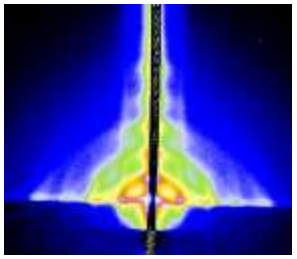
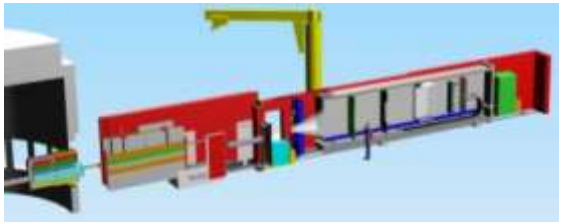


Summary:

- SANS is a very useful technique for determining the size, polydispersity and structure of a wide range of disordered materials
- When combined with the broad sample environment available the science studied using SANS is incredibly varied
- Well equipped labs for further characterization and onsite sample preparation
- The Sans2d sample area was designed with industrial partners and so very large sample environment can be accommodated
- *In-situ* and real time measurements possible
- The increased flux and improved signal to noise of Sans2d on TS-2 allows weakly scattering samples to be studied more efficiently and also allows faster kinetics to be measured
- Zoom and Larmor are under construction and will eventually allow even smaller Q values (larger objects) to be achieved and polarised SANS to be performed
- Various routes of access available
- We need to get food scientists involved!





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THANKS to…..

University of BRISTOL

UPPSALA UNIVERSITET

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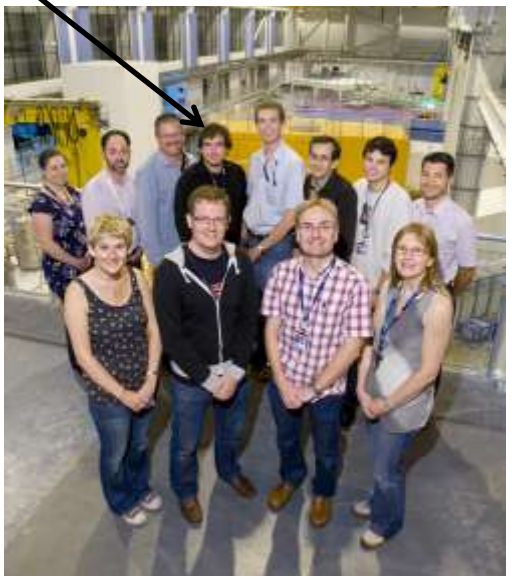
Luke



NEUTRONS FOR SCIENCE

University of Reading

And you for listening!