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SANS and SESANS: what can they do for you?

Sarah Rogers

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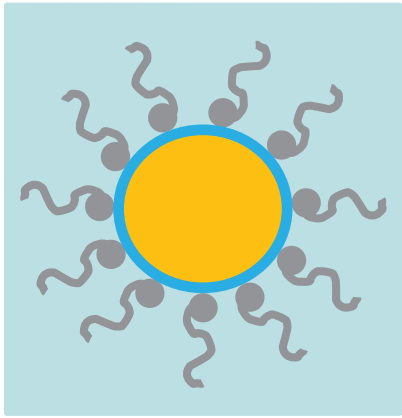
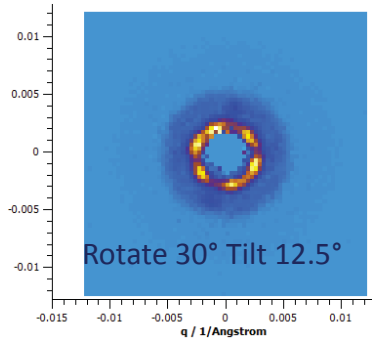
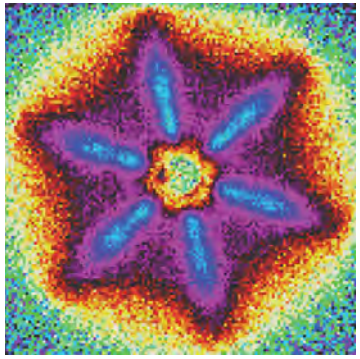
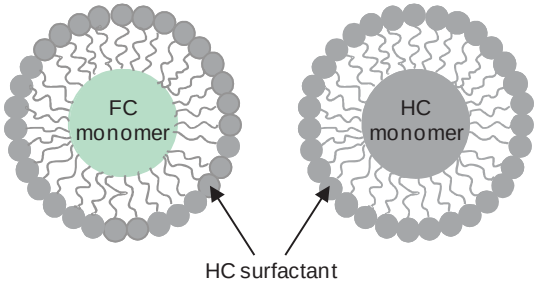


Overview:

- What are SANS and SESANS
- Why use neutrons and ISIS?
- The Instruments
  - Current: Loq, Sans2d and Offspec
  - Future: Larmor
- Sample environment
- Science examples
- Summary
- Acknowledgements







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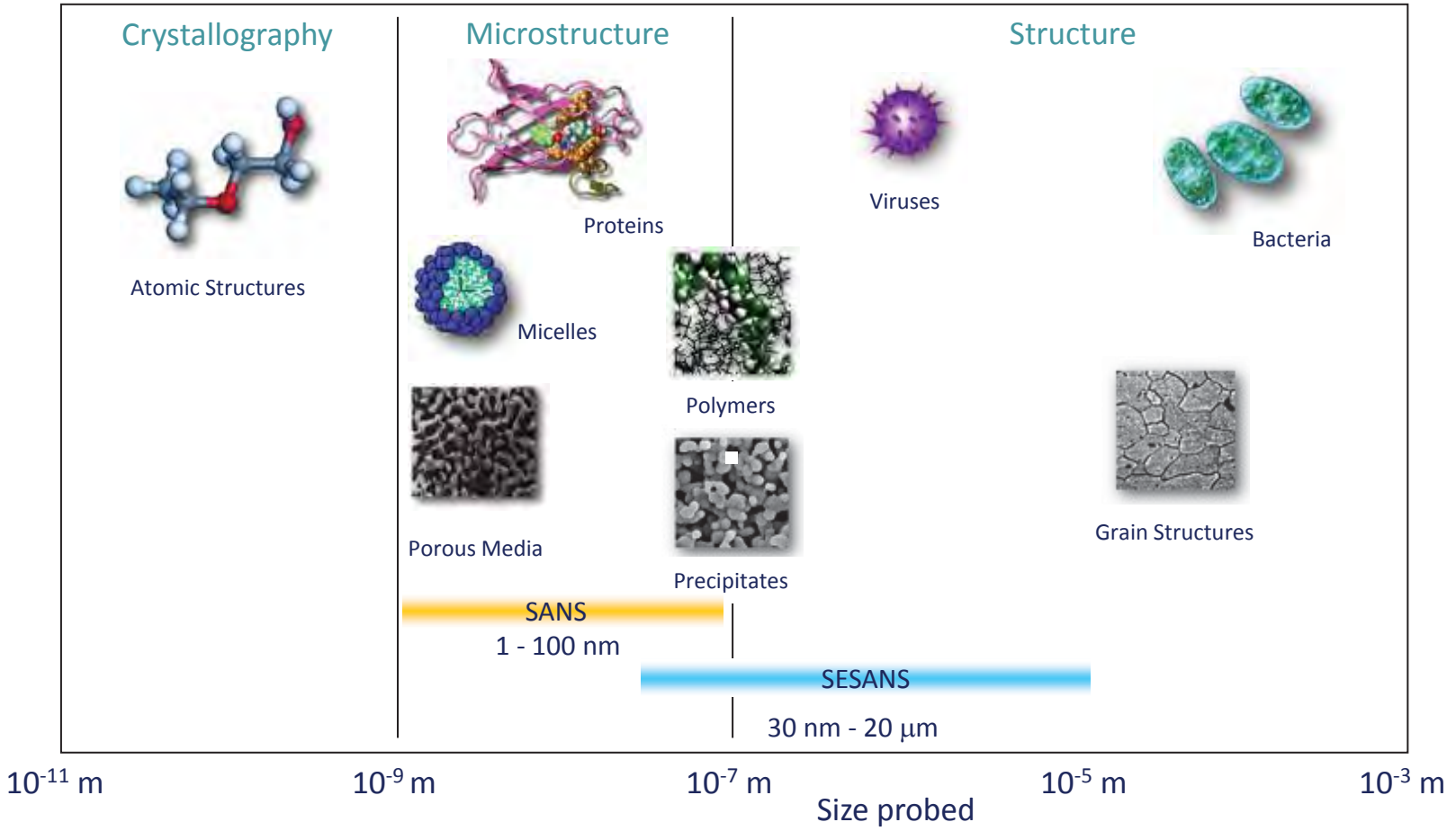
What are S_{mall}-A_{ngle} N_{eutron} $S_{cattering}$ and S_{pin}-E_{cho} SANS?

- Can determine the size, polydispersity , structure and interactions within a wide range of disordered materials
- Materials studied include surfactants, polymers, liquid crystals, nanoparticles, lipids and fibres
- Lengthscales probed range from 1s to 100s nm for SANS and 10s nm to microns for SESANS



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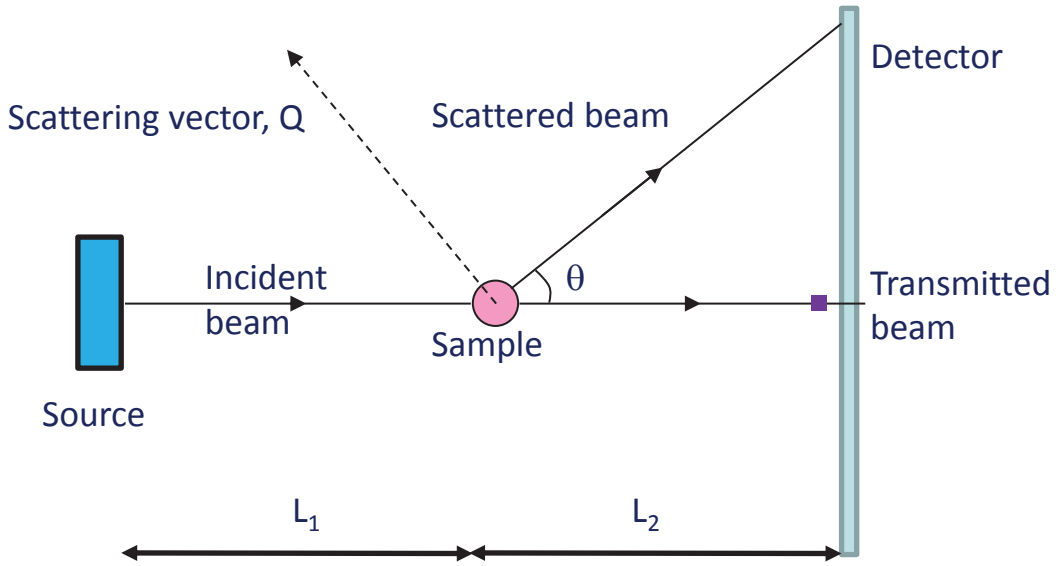
SANS Transmission Geometry

Lengthscales are explored in reciprocal space by detecting the number of scattered neutrons as a function of the scattering vector, Q. Q is inversely proportional to distance, D, by the approximation:



$L_1 = L_2$ for optimal Q resolution

To reach the smallest Q values the incident flux is always lower in conventional 'pinhole collimation' SANS as a long incident collimation is needed

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

Units are either $Å^{-1}$ or nm^{-1} i.e. the smaller the value of Q the bigger the object

Q is also related to wavelength and the scattering angle by:

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

Q (size) range is varied by altering θ or λ

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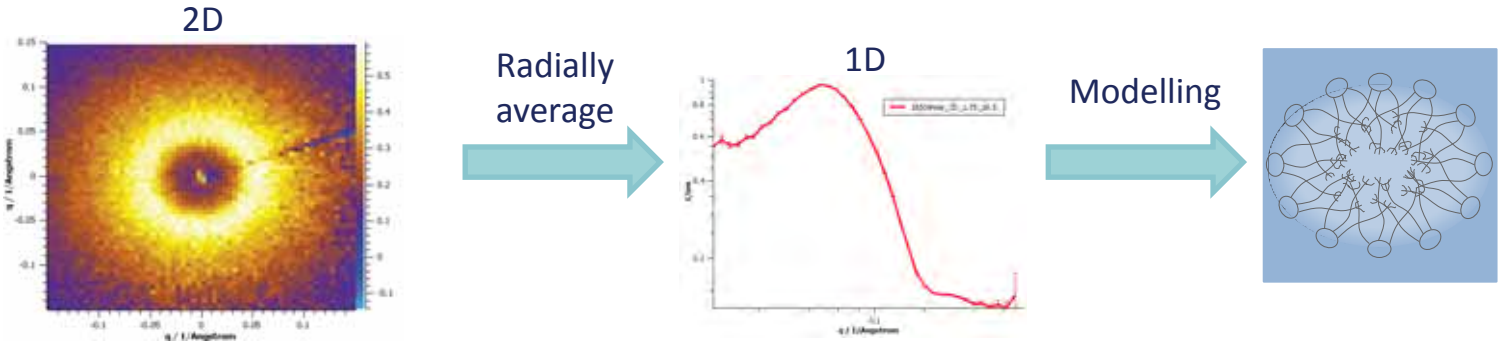
'Typical' Experiment

The 2D SANS patterns obtained are often radially averaged to given an intensity, I(Q), vs. Q plot



I(Q) contains the information on size, shape and interactions between the scattering centres in the sample. For monodisperse spheres I(Q) can be defined as:

$$I(Q) = (\rho_p - \rho_m)^2 N_p V_p^2 P(Q) S(Q) + \mathrm{B}$$

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

'Flat background'. Generally regarded as due to 'incoherent' scattering, often due to hydrogen

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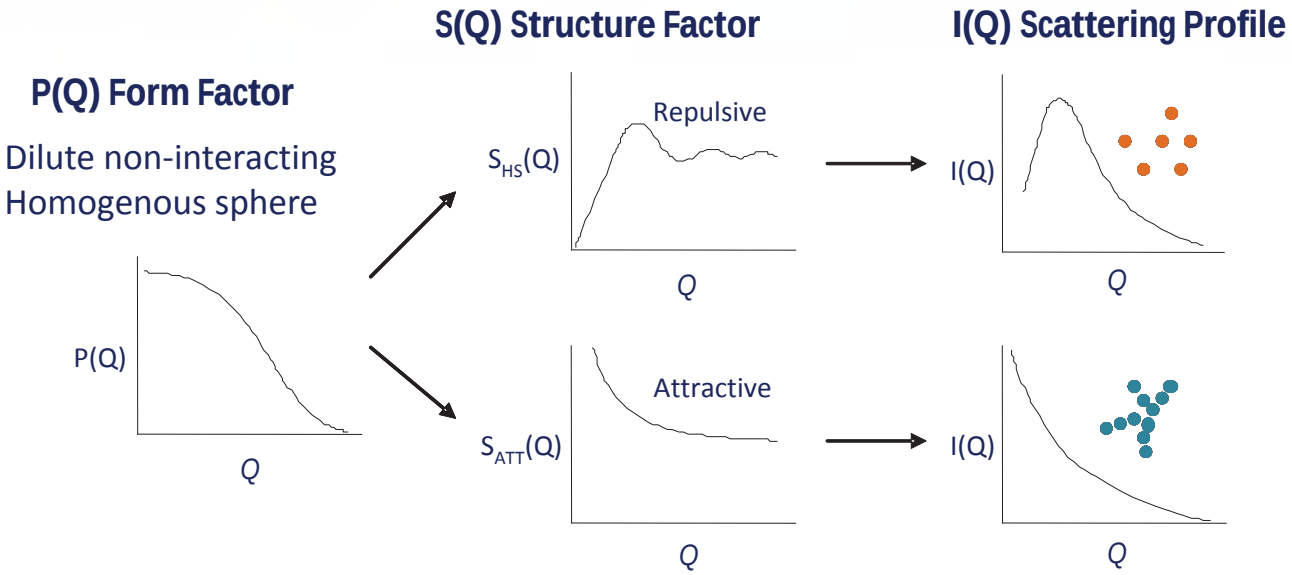
Effect of Form and Structure Factor



Form factor, P(Q):

- the larger the particle the more steeply P(Q) falls off with Q

Structure factor, S(Q)

- non-interacting (hard spheres) and repulsive interaction push down I(Q) at small Q as concentration increases -> ordered structure with a 'Bragg Peak'
- attractive interactions tend to increase scatter at small Q, as transient large particles or actual aggregates form

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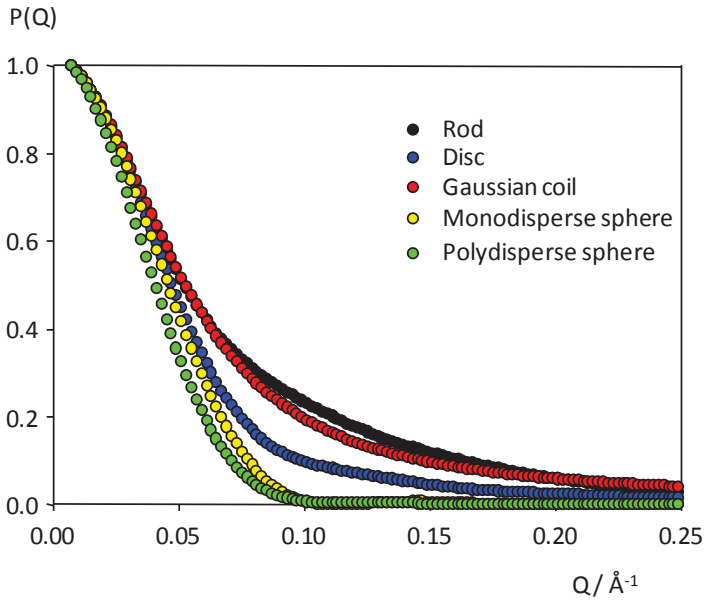
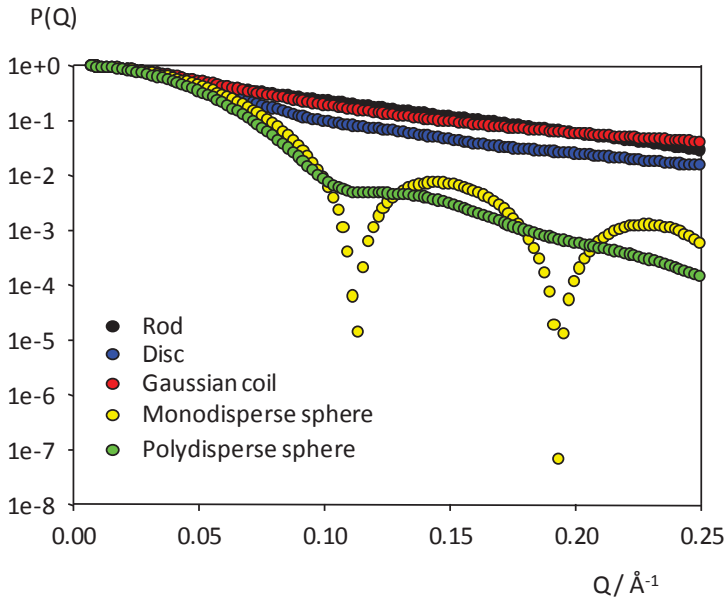
Retrieving Information From Your Data: Graphical Plots

Guinier Approximation

- Relates low Q part ($QR_g < 1$) of scattering profile to the R_g of a particle
- In this regime the form factor is insensitive to shape – all the plots below are particles with R_g = 31 Å

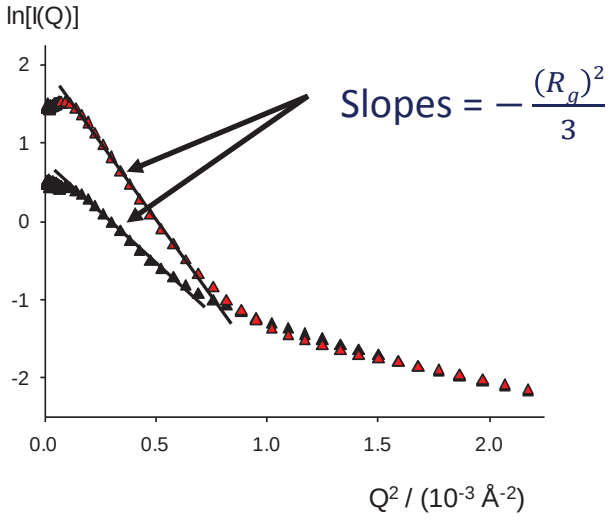




Guinier Approximation continued

- In the Guinier regime P(Q) for a dilute system is: $P(Q) = 1 - \frac{(QR_g)^2}{3}$
- Assume S(Q)=1 and $1 - X^2 = \exp(-X^2)$ then: $I(Q) \approx \phi_p \Delta\rho^2 V_p exp\left(-\frac{(QR_g)^2}{3}\right)$
- A plot of ln(Q) versus Q^2, the Guinier plot, should include a linear section up to $QR_g < 1$.

This Guinier plot is for two different size spherical particles. ▲ = particle radius of ~ 40 Å and ▲ = particle radius of ~ 25 Å



- Slope is $-\frac{(R_g)^2}{3}$ i.e. R_g can be found from the slope!
- Often used for proteins in solution, micelles, polymer coils etc

Retrieving Information From Your Data: Graphical Plots

Porod Approximation

- SANS intensity plots are sensitive to scattering from local interfaces at high Q

- For a smooth, sharp interface I(Q) is related to the total interfacial area, S, by:

$$I(Q) = 2\pi\Delta\rho^2\left(\frac{S}{V}\right)Q^{-4}$$

Total interfacial area per unit of volume of solution

- The average area per surfactant head group, a_s, can be estimated (assuming all molecules are located at the interface):

$$a_s = \frac{S/V}{N_s}$$

Number density of molecules: concentration x N_A

- Can also be used to estimate the particle radius. For monodisperse spheres of radius R, plotting $[I(Q).Q^4]$ versus Q gives a first maximum at Q = 2.7/R and a minimum at Q = 4.5/R

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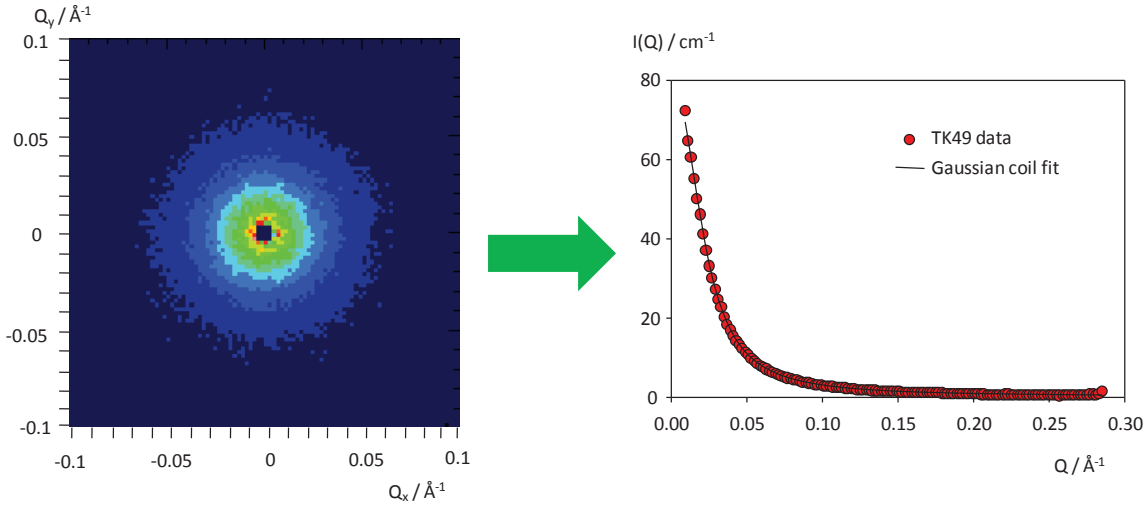
Retrieving Information From Your Data: Mathematical Modelling

- Information of size, shape, polydispersity, interaction etc. can be found
- Models not selected at random! Known or calculated physical parameters such as P_c, volume fraction, estimated particle size, charge, Q-dependence etc. are used to make informed choices
- Run a series of samples at different concentration, temp., pH etc. to give a more complete picture
- Scale factors are also used to verify models after fitting – why it is important to work in absolute units for SANS

$$I(Q=0) = \phi_p V \Delta\rho^2$$



Partially deuterated solid polymer (polystyrene). Fit using Gaussian coil model. I(Q=0) was found to be 80cm^{-1} and R_g = 74 Å

- SASVIEW (http://www.sasview.org/) is commonly used by ISIS/ILL/NIST users

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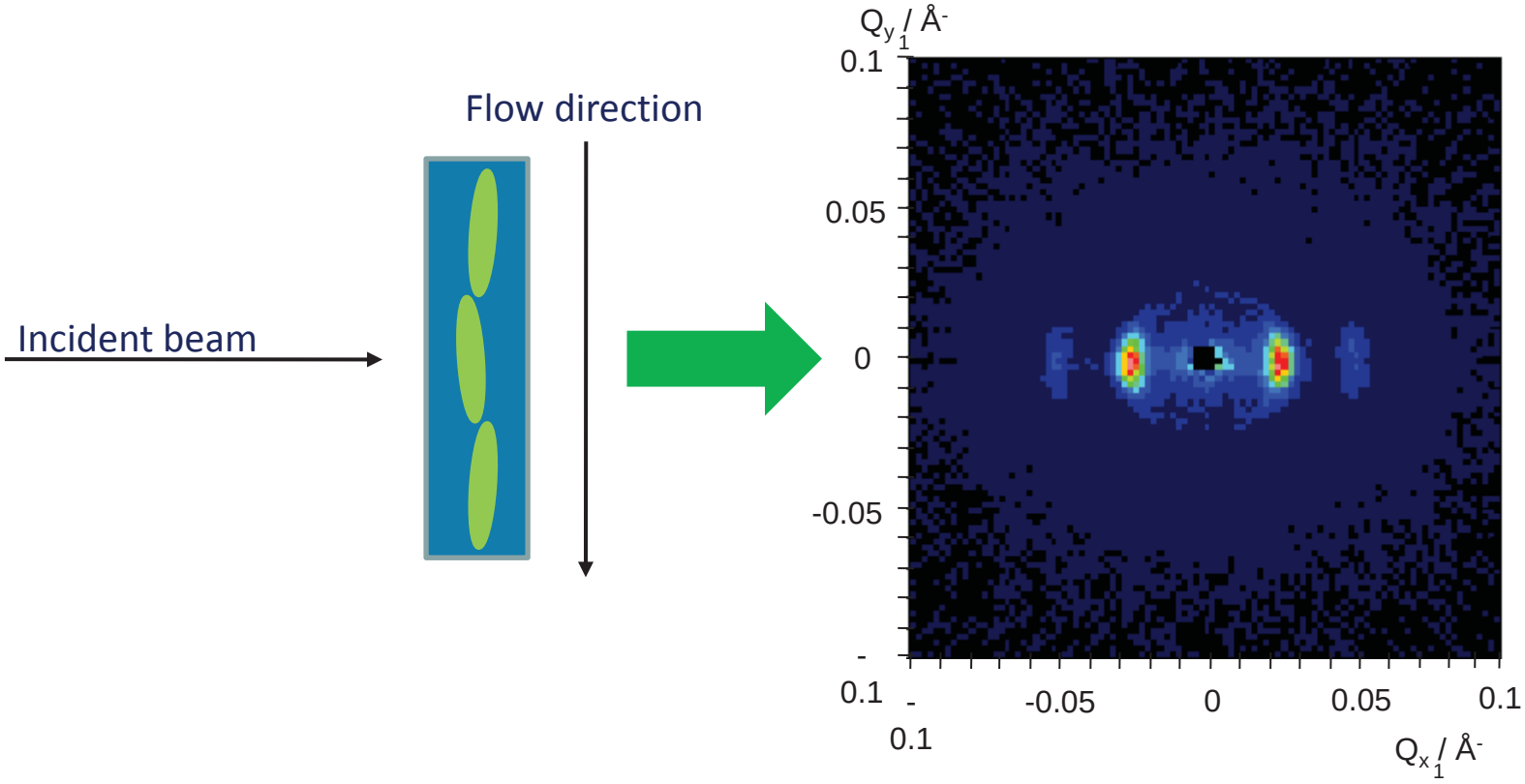
Anisotropic Scattering

- Not all systems give an isotropic scattering pattern
- Sample can be orientated by shear, magnetic fields etc
- Information about the orientation within the sample can be uncovered using SANS



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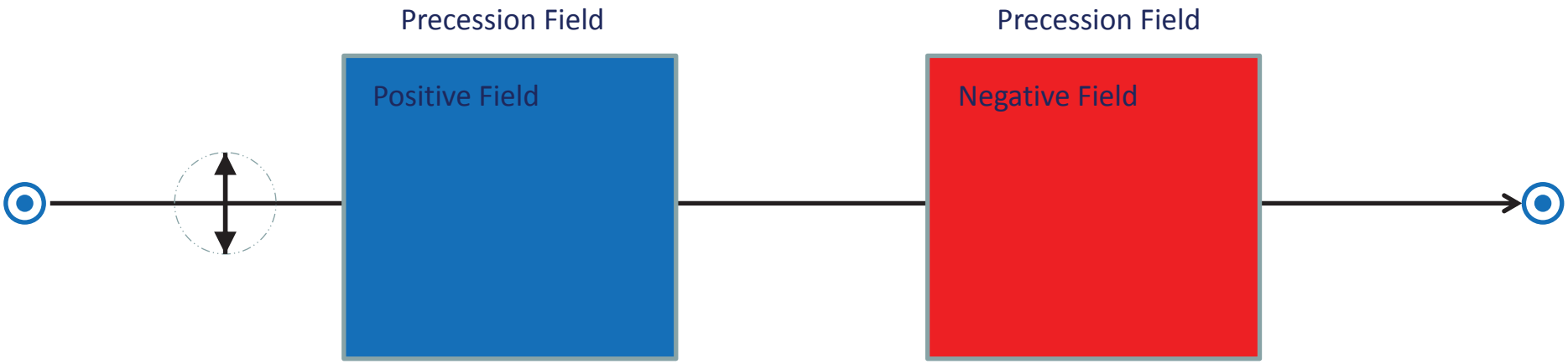
SESANS – the Neutron

- The neutron has a magnetic moment with spin 1/2
- Can be thought of as a bar magnet
- When placed in a magnetic field the neutron starts to precess



Precession proportional to magnetic field line integral:

$$\phi \propto \int B dL$$

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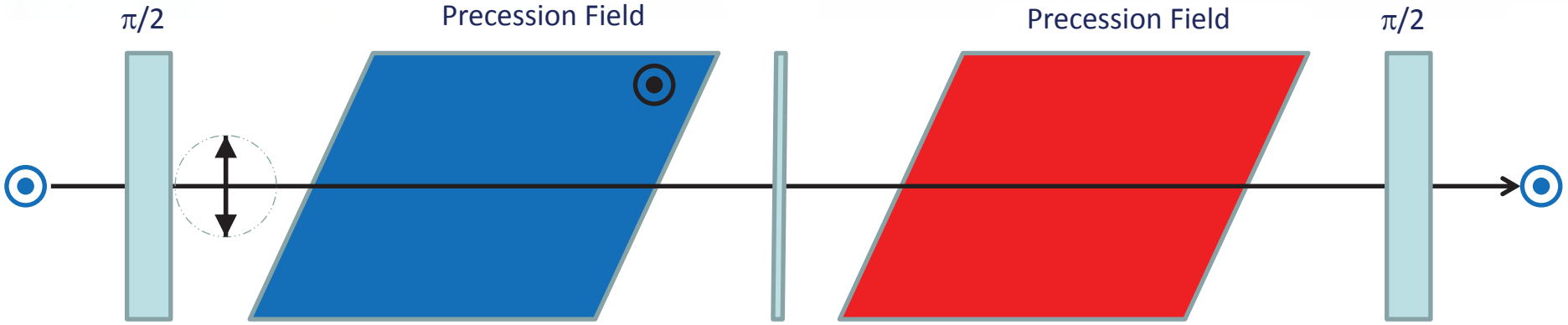
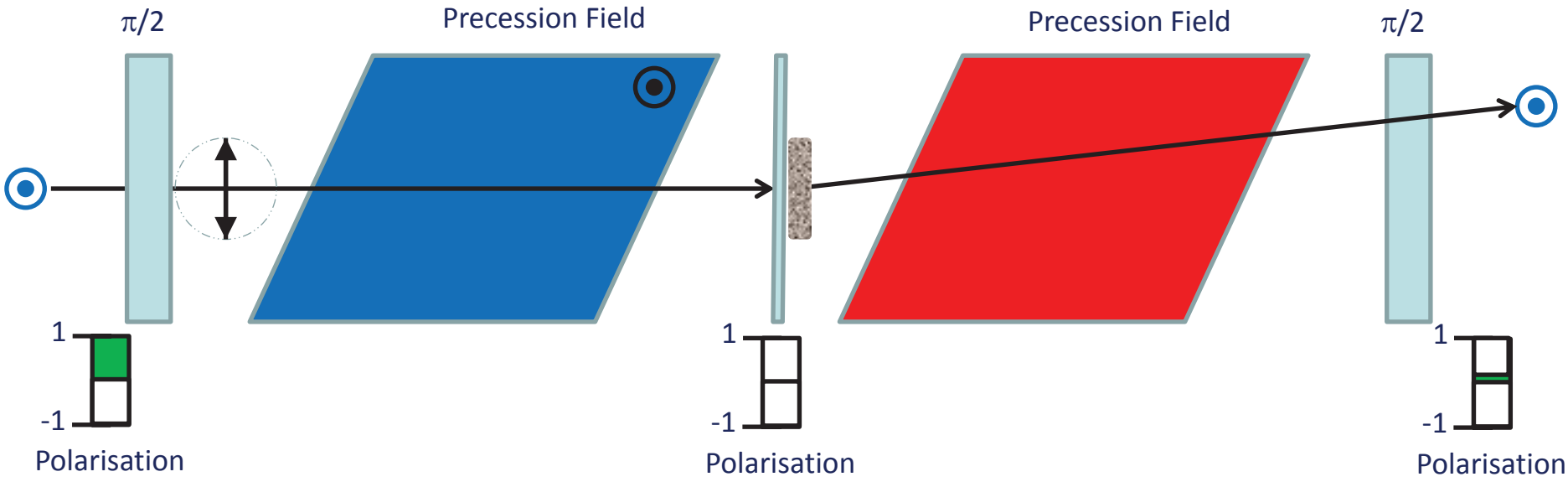
SESANS - Larmor Labelling

Without sample: unscattered beam gives spin echo $\phi = 0$ independent of height and angle:



With sample: no complete spin echo and net precession angle



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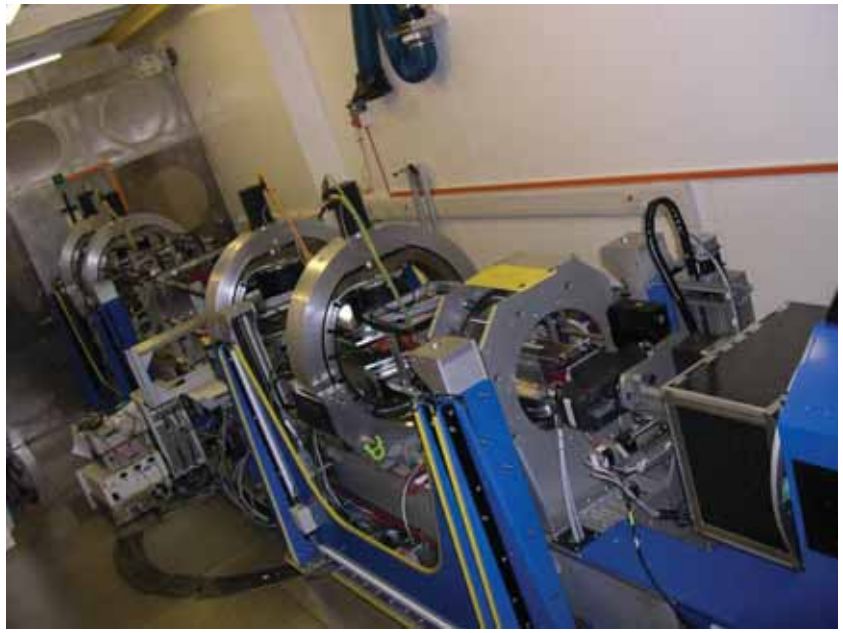
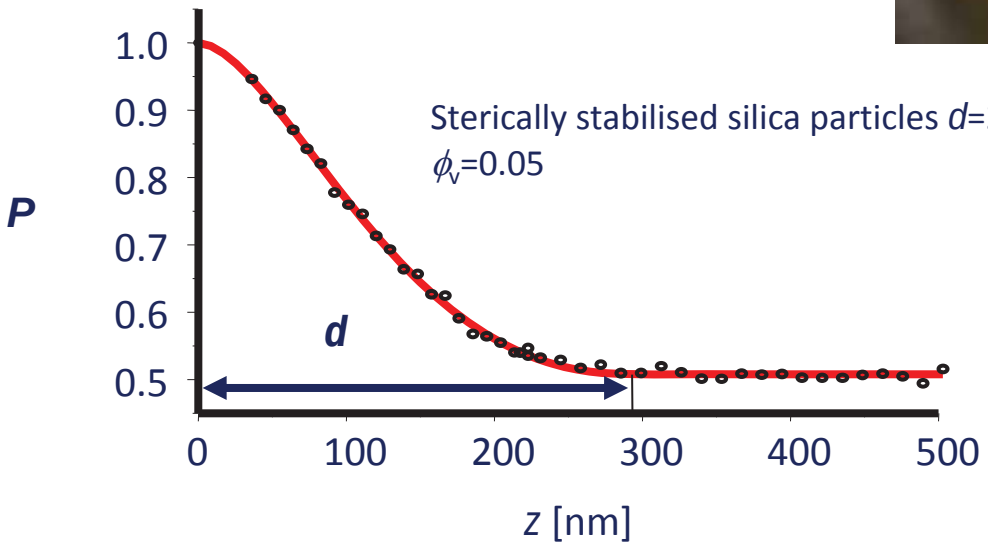
'Typical' Experiment

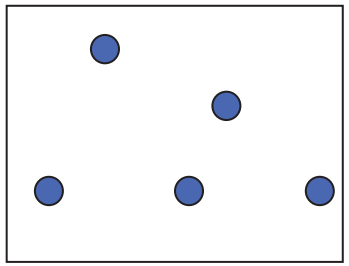
- Measure the direct beam polarisation (P_0)
- Measure the polarisation through the sample
- Divide and calculate the normalised polarisation
- Result is a real space correlation function integrated over the measurable Q range.

$$\frac{P}{P_0} \propto G(r) - 1$$





Sterically stabilised silica particles *d*=298 nm in deuterated cyclohexane
ϕ_v=0.05



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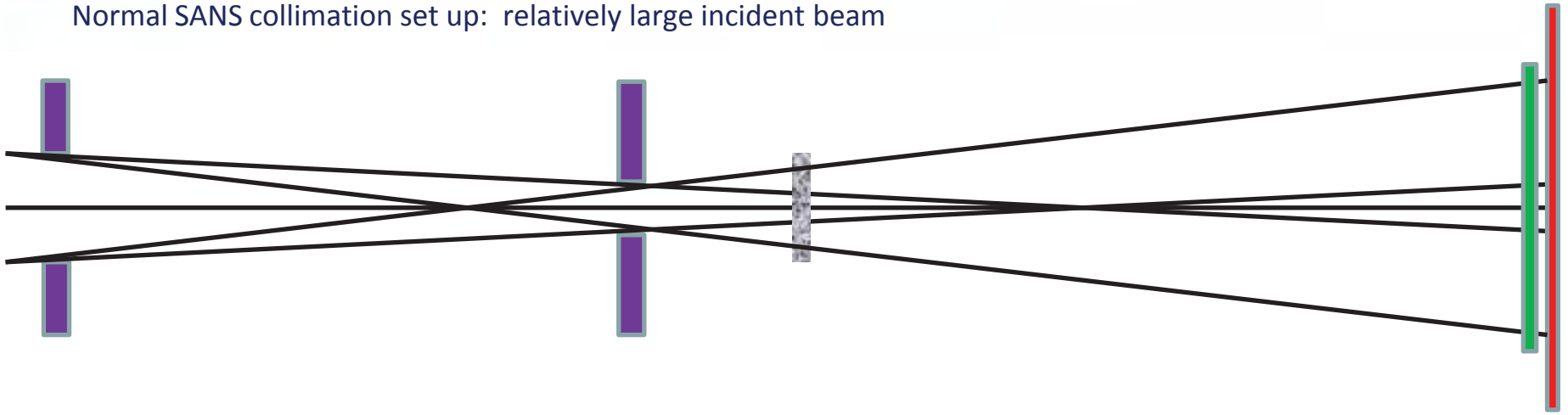
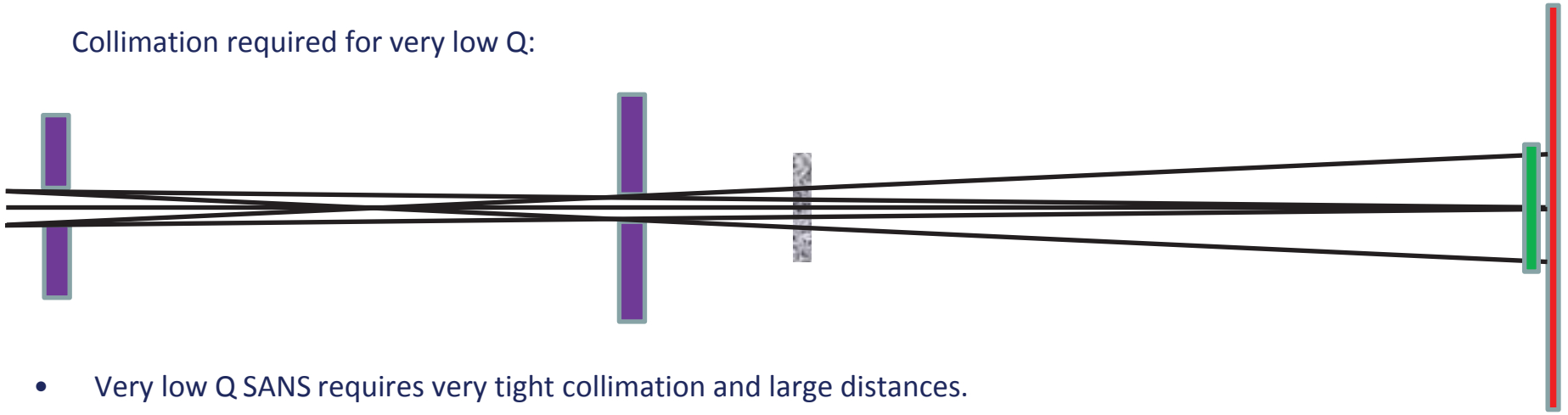
What is the Advantage?

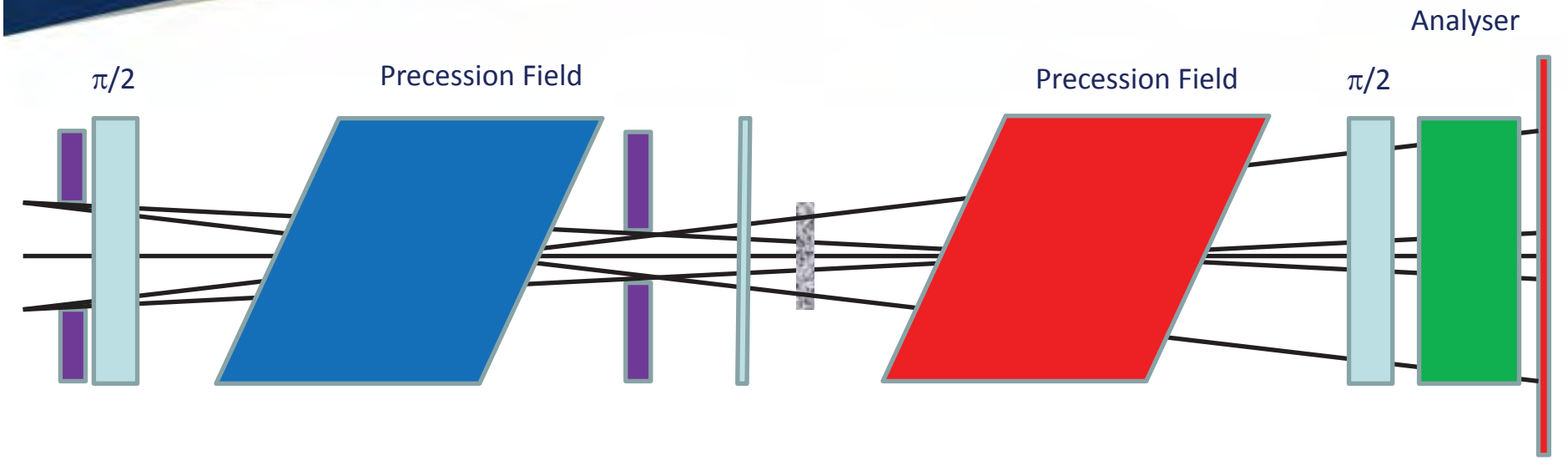
Normal SANS collimation set up: relatively large incident beam



Collimation required for very low Q:



- Very low Q SANS requires very tight collimation and large distances.
- No Neutrons Left!



- Encode beam with relaxed beam divergence.
  - High count rates
  - Length scales ~ 50 nm to 50 μm can be probed
- Spin-Echo length is proportional to the field strength, length and angle between the inclined face and the average beam direction as well as wavelength^2

$$z = \frac{m\gamma_L BL\lambda^2 cot\theta_0}{2\pi h}$$

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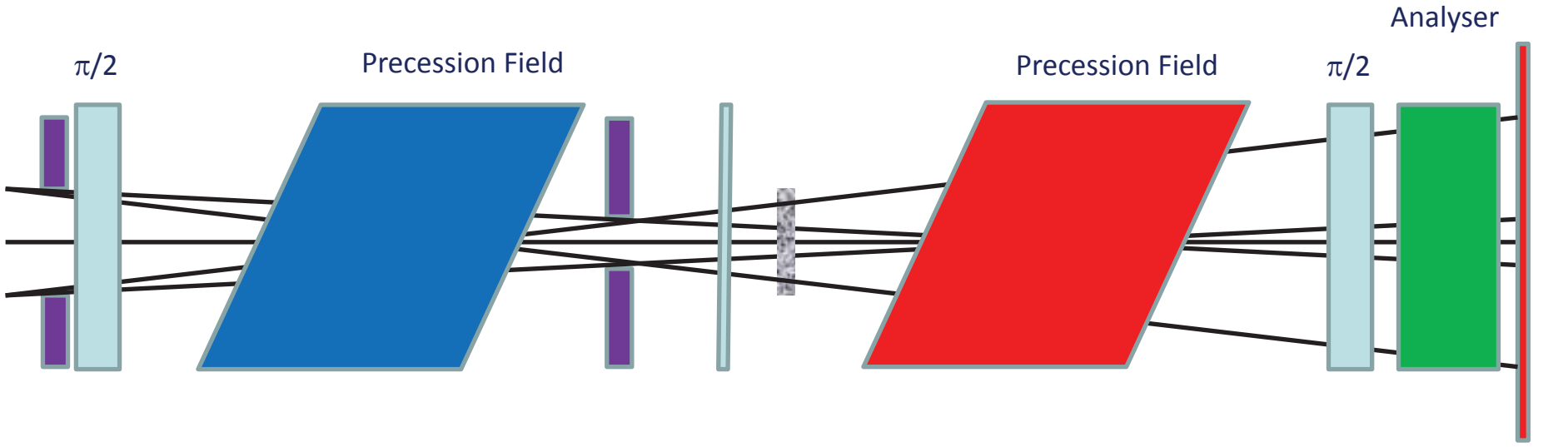
What are the Disadvantages?



- Signal is superimposed on the direct beam
  - Can only measure strong scatters
- The analyser limits the detector angular coverage
  - SANS and SESANS cannot be done at the same time
- Encoding is in 1 direction only

  Non-isotropic scatters?

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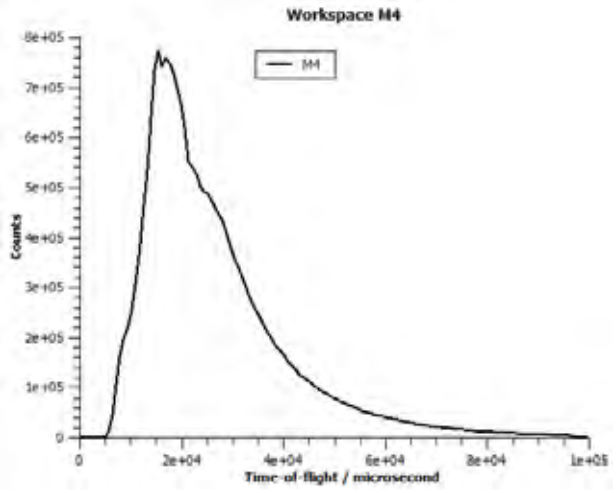
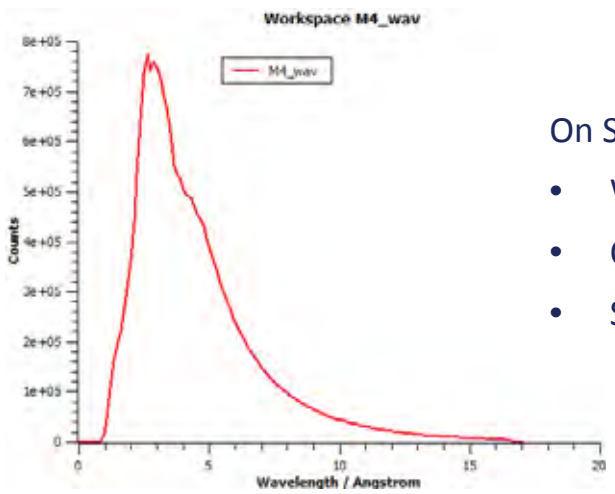
Bonuses!

- High penetrating power that allows the structure in the bulk of a material to be probed
- They are non-destructive and therefore delicate samples can be studied
- They do not significantly perturb the system under study and so the results from experiments can be clearly interpreted
- The neutron has a magnetic moment which allows the study of magnetic structures
- Neutrons scatter from materials by interacting with the nucleus of an atom (rather than the electron as with X-rays…this is very useful!
- ISIS is a spallation source and operates in time-of-flight mode. This means that a large simultaneous Q-range is achievable (without moving detectors) as white beam of delivers a broad wavelength range of neutrons. This is very useful for time-resolved measurements





On Sans2d:

- Wavelength range = 1.75 – 17.5 Å
- Q-range = 0.001 – 3 $Å^{-1}$
- Size range ≈ 3 – 6000 Å = 0.3 – 600 nm

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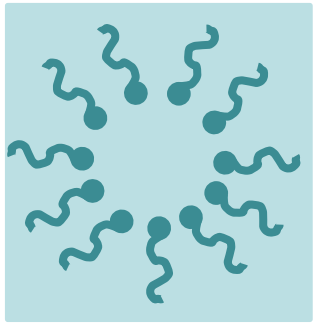
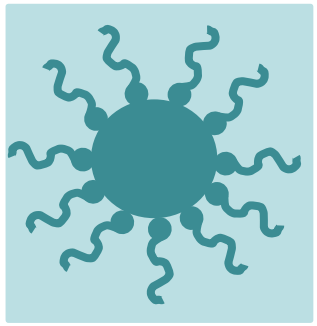
Unique insights from Contrast Variation

- The neutron scattering power of atoms varies, randomly, from element to element and isotope to isotope
- Light atoms e.g. hydrogen, can be distinguished from heavier atoms, such as metals
- Also isotopes of the same element can have substantially different scattering powers: by altering the isotopic content of a sample (switching the solvent H_2O to D_2O, for example) or by using isotopic substitution within a molecule specific area of interest within the system under investigation can have their scattering power enhanced without changing the chemical properties of the sample

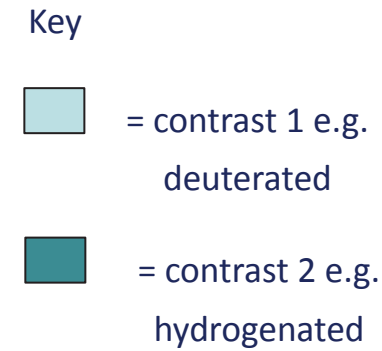


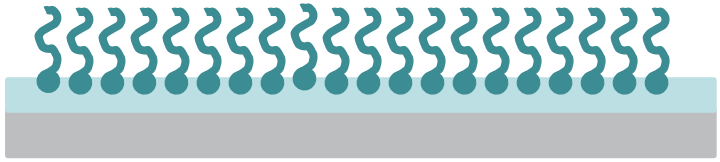
Shell contrast



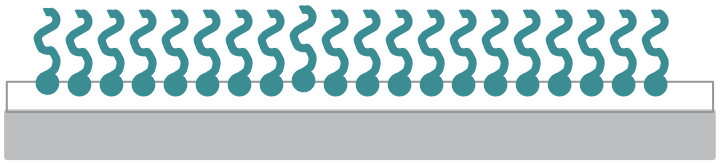
Drop contrast

Core contrast





Surfactants at the air/water interface



Surfactants on ACMW

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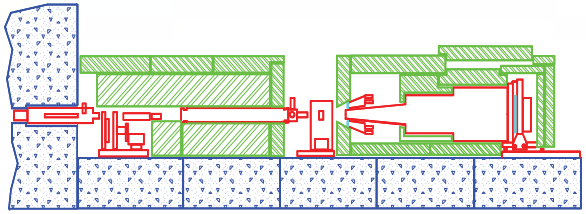
Loq and Sans2d

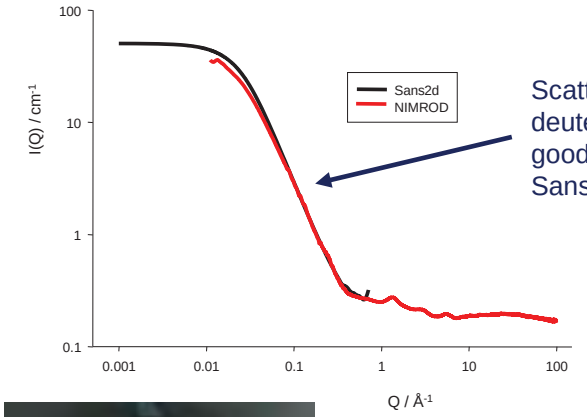
Loq

- Using a fixed sample - main detector distance of 4 m and neutron wavelengths of 2 to 10 Å at 25 Hz a Q-range of 0.007 – 0.3 $Å^{-1}$ is accessible. This Q-range can be extended to 1.4 $Å^{-1}$ by employing the wide-angle detector bank
- Currently having chopper upgraded



Sans2d

- 1st SANS instrument on optimized TS-2
- 2 x $1m^2$ detectors provide a uniquely wide simultaneous TOF Q range at good resolution: Q = 0.001 to 3 $Å^{-1}$, λ = 1.75 to 17 Å.
- Movable detectors will match the overall Q range to the science
- Currently having front end vacuum box upgrade
- **Project underway to replace Ordela detectors with 2 x 1m x 1m ^{3}He Tube Array – these will allow us to count even faster!**



Scattering from partially deuterated polymer shows good overlap between Sans2d and Nimrod

For further information contact Sarah Rogers: sarah.rogers@stfc.ac.uk

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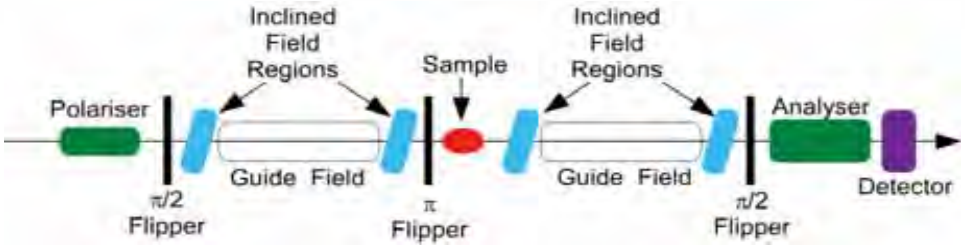
Offspec

- This is the first instrument to be built on the optimized ISIS second target station, TS-2, which is capable of performing SESANS
- An RF flipper, spin-echo system, designed and constructed at TU-Delft, provides a total potential length scale range from 20nm-20μm
- Two Stage polariser to achieve >95% polarisation over the complete wavelength band (1.5-14.5Å)
- Measurement time for strongly scattering, high contrast systems are similar to SANS.
- Large simultaneous spin-echo range accessible using the ISIS white beam.
- Continuous improvements to polarisation and effort to reduce parasitic air scatter are ongoing



For further information contact Rob Dalgliesh: robert.dalgliesh@stfc.ac.uk

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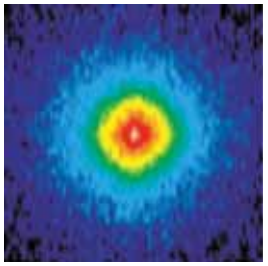
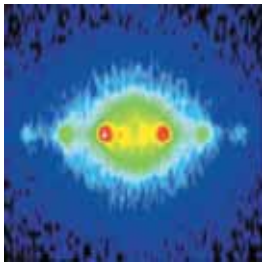
Larmor: Flexible Spin-Echo for Larmor Precession

Day One Science

SANS:

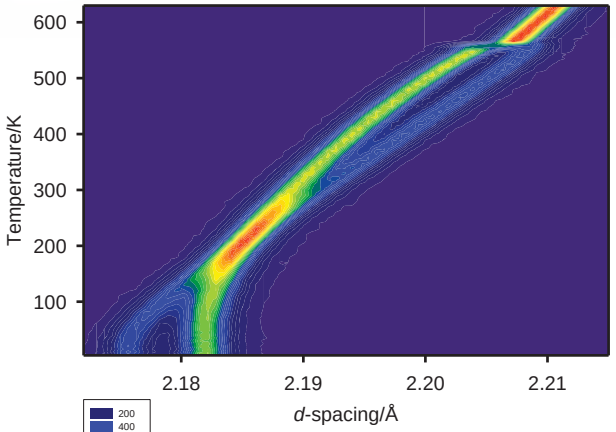
- Polarised Beam with Analysis
- 0.5 – 13 Å
- Fixed ~4m L1 & L2
- 600 x 600mm ^{3}He Tube Array
- 8 x 8mm pixel size.
- Q range with offset detector: TBC 0.004 - 1.6 Å^{-1}
- Similar basic parameters to LOQ
  - Lower background
  - Higher Peak flux

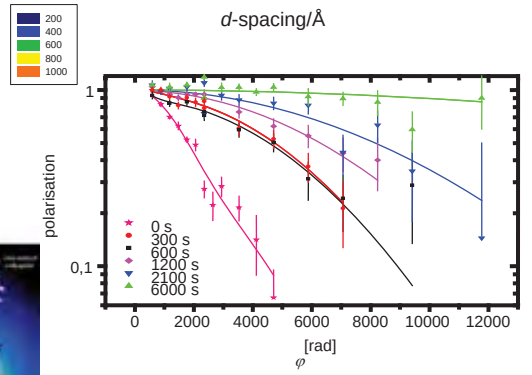




Larmor Diffraction:

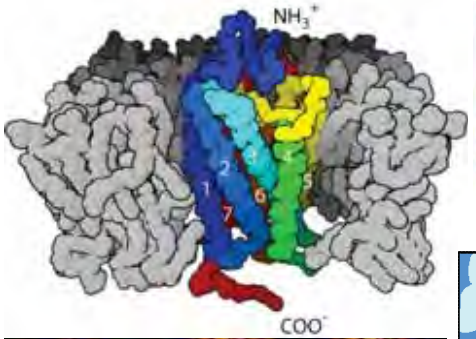
- Small structural change in crystalline materials
- Pressure, temperature, field induced transitions
- $\Delta d/d < 10^{-5}$

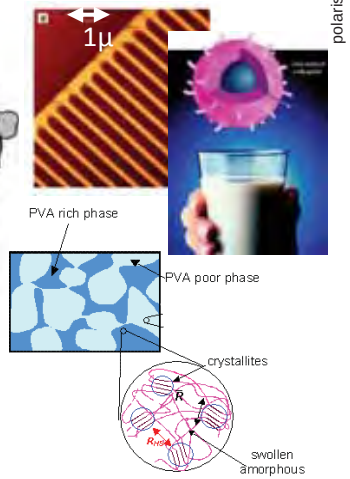




SESANS:

- Length scales from ~20 nm to 20 μm
- Aggregation
  - Colloids
  - Food Science
  - Advanced Materials
  - Bio-Materials
  - Granular Materials
- Significantly more flux than OffSpec because of the beam optics





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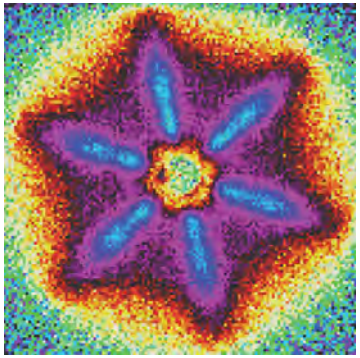
The Sample Environment

Extensive available sample environments allow a broad range of science to be studied via SANS/SESANS at ISIS.

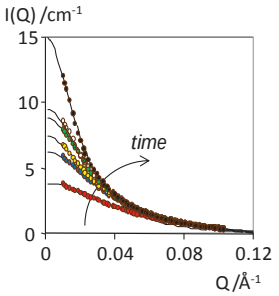
Sample environment includes:

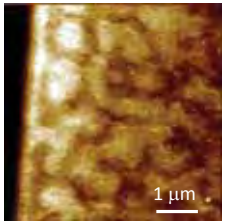
- Standard ISIS cryostats, furnaces and magnets
- Sample changer with temperature control
- Linkham stages for advanced temperature control
- Rheometer and shear cells
- Pressure cell – 600 bar with stirring. Predominantly used with CO_2
- T-jump cell – study non-equilibrium phases
- *In-situ* DLS and UV-vis
- Grazing Incidence SANS (GISANS)
  - ➢ Study of in-plane structure on the nm lengthscale
- Stopped-flow – mixing kinetics
- Well equipped offline labs allow for further characterization – X-ray sets, AFM, BAM, spectrometers

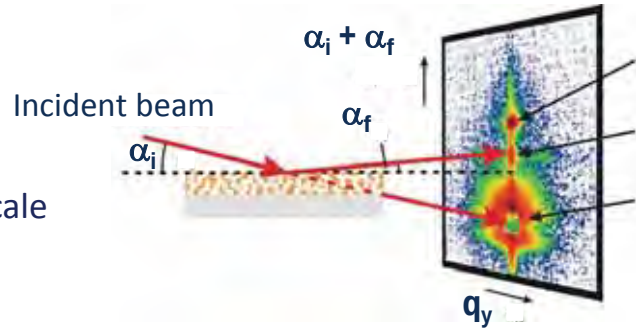


(a)

(b)







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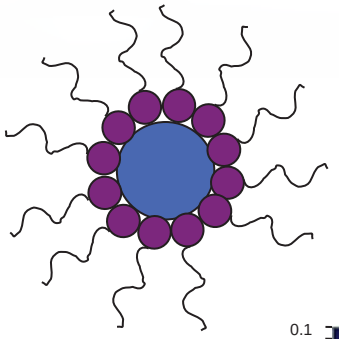
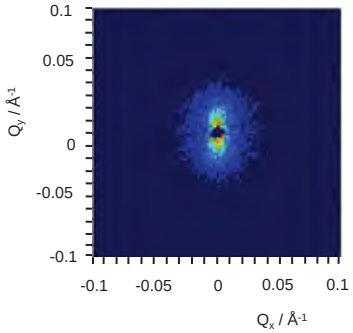
The Science

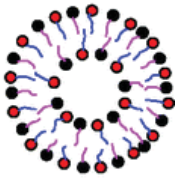
For the next two cycle alone Sans2d has the following:



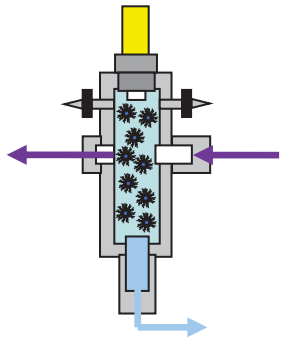
- **Surfactant chemists**
  - Interaction of perfumes with micelles
  - Aggregation in unusual solvents – sc-CO_2 and ILs
  - Thickness of polymer and protein layers on nanoparticles
- **Chemists**
  - The structure of foams
  - Orientation of peptide fibrils and hydrogels aligned with magnetic fields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

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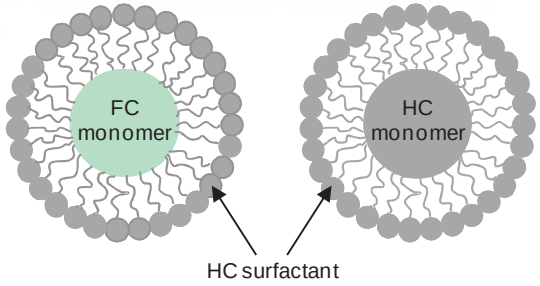
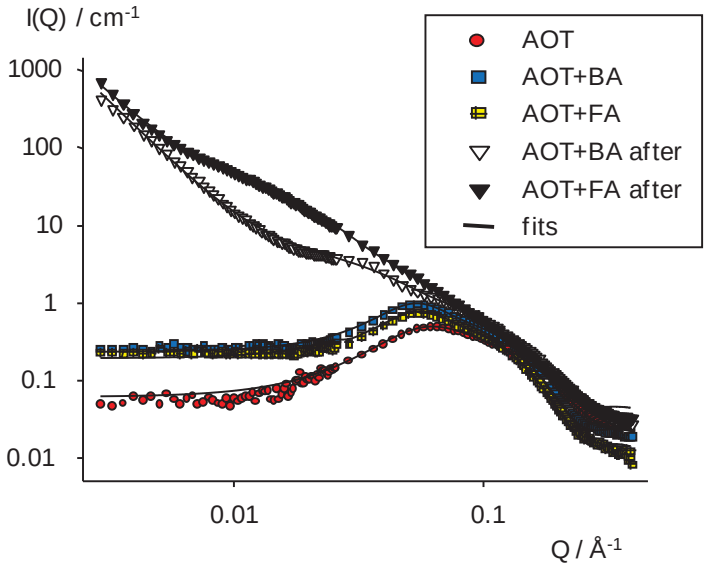
Microemulsion Polymerization



The natural incompatibility of the fluorocarbon (FC) and hydrocarbon (HC) moieties employed within the system (as shown left) is exploited to drive a local phase segregation inside the microemulsion droplet and hence influence the size and shape of the final polymer particle produced.

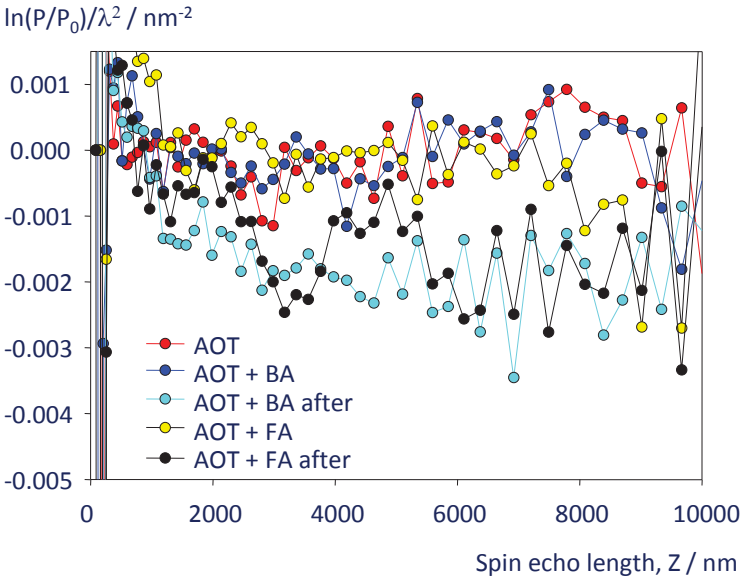
Conventional SANS has allowed the smaller particles produced to be characterised and also given the volume fractions of each particle distribution present. It was found that the AOT + FA system had a larger volume fraction of the smaller particles present when compared to the AOT + BA system which indicates than the H-F immiscibility gave a positive result. Can SESANS give us information on the larger particles within the polymerised system?



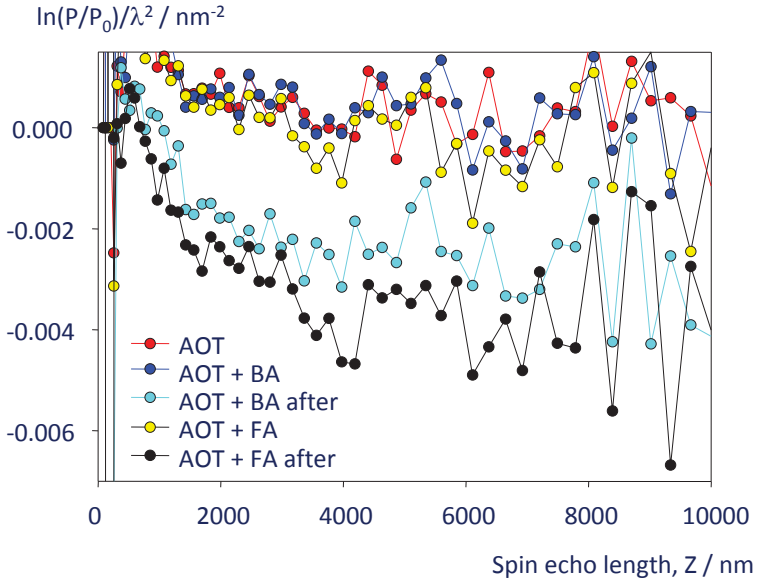
Oil used	Results of SANS analyses demonstrating nanolatex formation (excluding swollen micelle scattering)		
	Sphere		Q dependence at low Q
	R_{bar} / Å	polydispersity	
Butyl acrylate	34.9	0.28	-3.1
Fluoro acrylate	32.6	0.33	-2.9

There is a clear difference in scattering between the unpolymerised and polymerised systems. The flat scatter of the unpolymerised systems shows that there are no particles in this size regime present, as expected.

5mm pathlength:



10mm pathlength:



The decay of the signal to an approximately flat line at a spin echo length of around 4000nm indicates that there are significant correlations in the system with this length scale. The apparent peaks present in the AOT + FA system could, potentially, indicate additional ordering or clustering of these large structures. Further analysis and measurement is required on these systems in order to obtain more detailed information on the size and shape of the larger polymerised particles.

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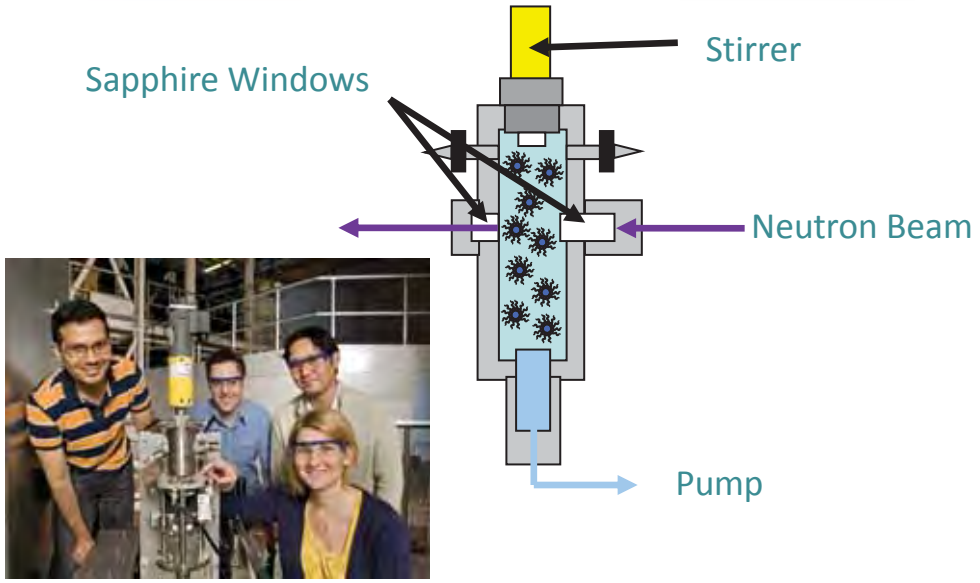
Viscosity Modifiers

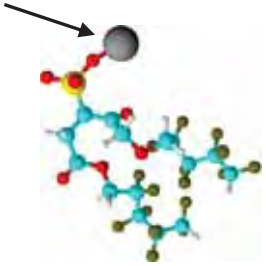
J. Eastoe (University of Bristol)

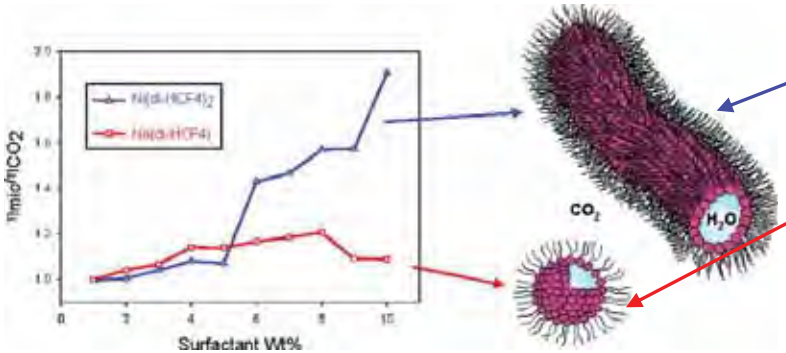
Langmuir **2010**, *26(1)*, 83-88

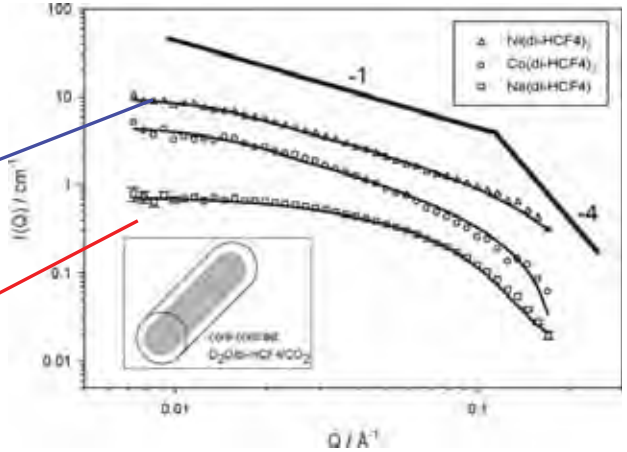
- Studying the use of high pressure CO_2 for enhanced oil recovery using dedicated 600 bar pressure cell
- Low viscosity of CO_2 promotes fingering through porous media rather than a uniform sweep
- Modifiers commonly used in oily solvents are incompatible with CO_2. Can self assembled custom-made surfactants 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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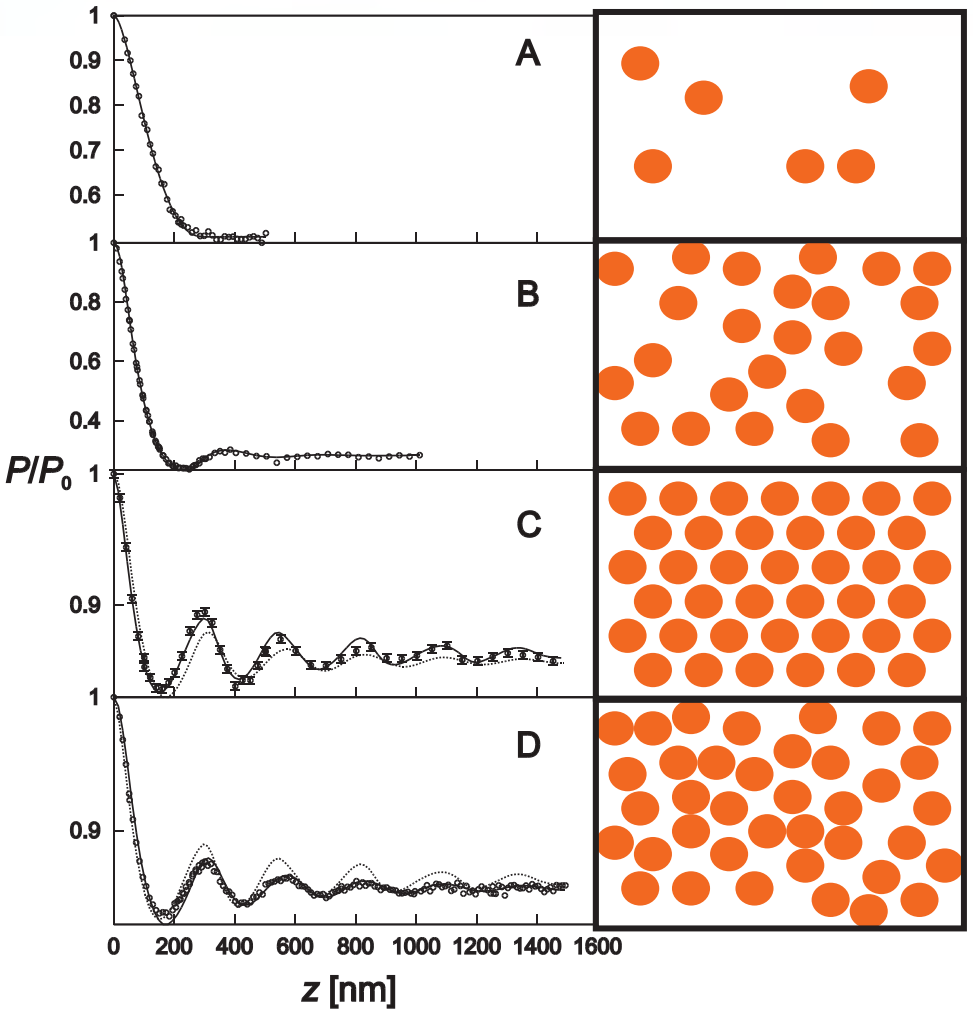
Colloidal phase transitions as function of concentration



gas

liquid

crystal

glass

Krouglov et al. J. Appl. Cryst. **36**, 1417-1423 (2003)

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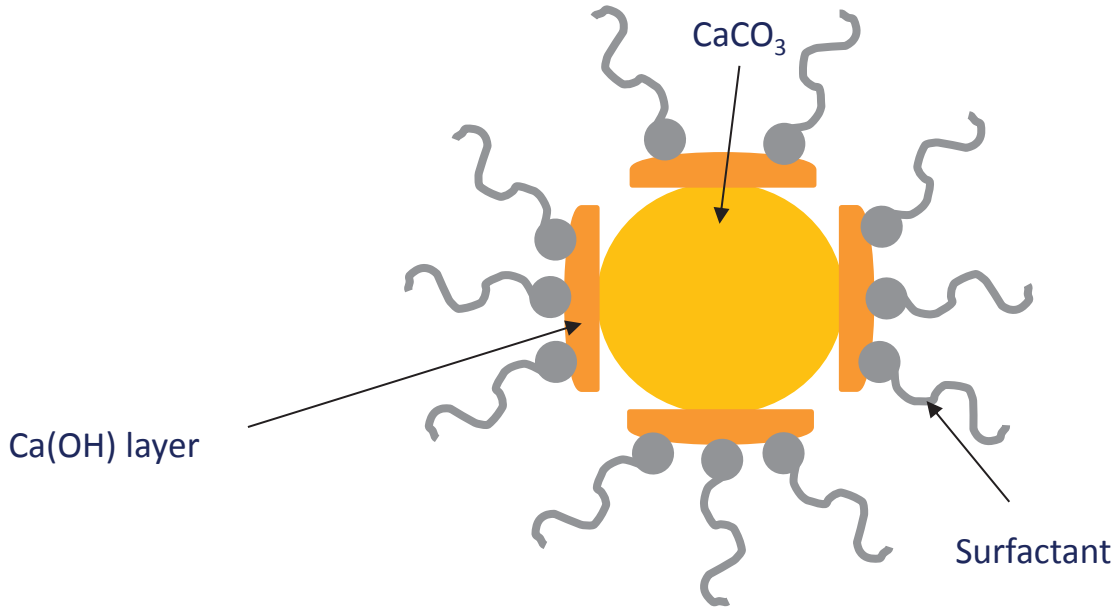
Nanoparticles

Collaboration between Dr. P. Dowding (Infineum) and Dr. A. Routh (University of Cambridge) studying overbased sulfonate engine oil additives (OBSAs). These OBSAs consist of calcium carbonate nanoparticles – $CaCO_3$ - stabilized by a sulfonate surfactant. The stability of these particles is crucial for their correct performance. The combustion process can produce a considerable amount of water: how does the presence of water effect these particles?



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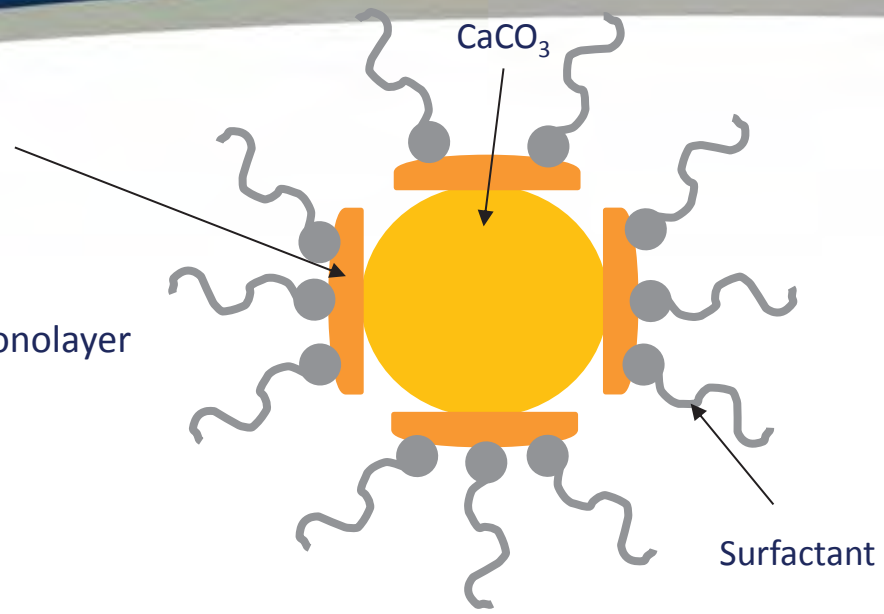
Proposed Structure

Amorphous $CaCO_3$ core (< 10 dia.nm) surrounded by a monolayer of alkyl aryl sulfonate surfactant



Three possible locations of water molecules:

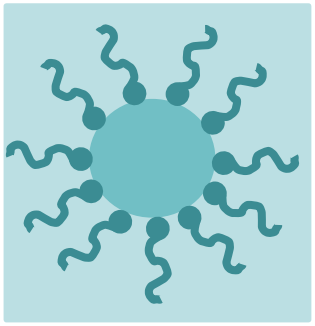
- Water-in-oil microemsulsions
- Throughout the CaCO3 particles
- Surface of the particles

The Experiment

Used contrast variation SANS to see core, shells and drop:

1. H-surf + $CaCO_3$ in D-cyclohexane → Drop – shell and core
2. H-surf + $CaCO_3$ in H-cyclohexane → $CaCO_3$ core
3. H-surf + D_2O + $CaCO_3$ in H-cyclohexane → $CaCO_3$ core + D_2O ‘shell’

1.



2.

3.

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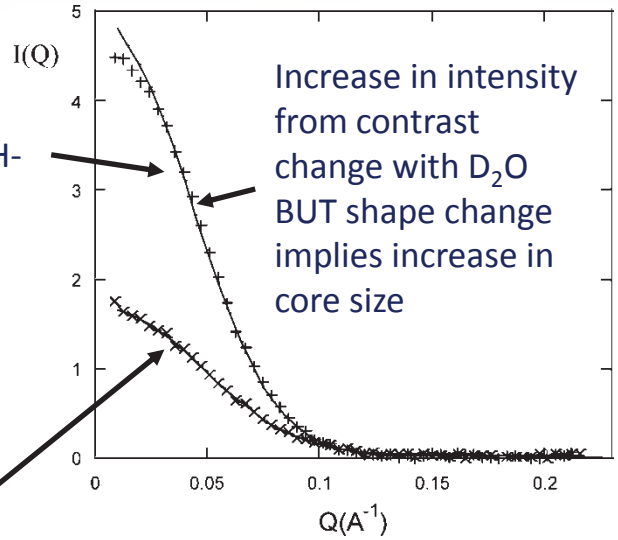
Results

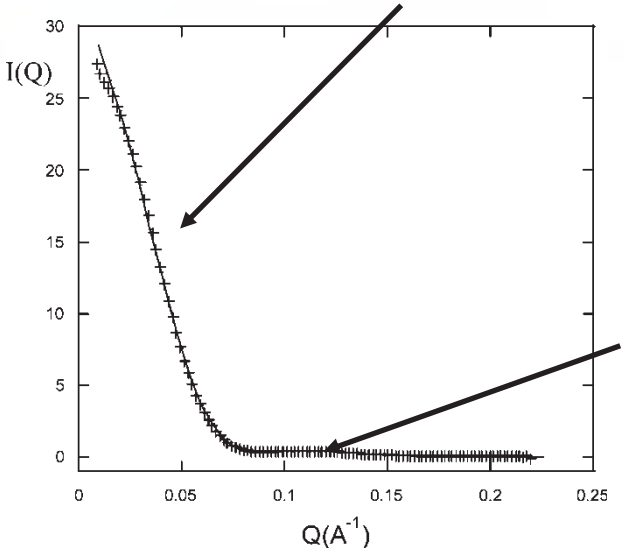
$CaCO_3$ + surfactant doped with D_2O in H-cyclohexane

Undoped $CaCO_3$ + surfactant in H-cyclohexane



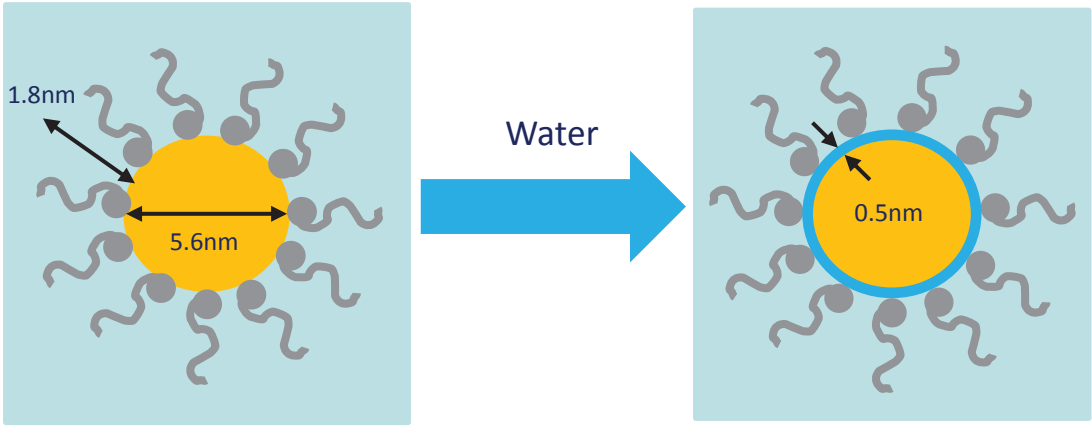
Increase in intensity from contrast change with D_2O BUT shape change implies increase in core size

Undoped $CaCO_3$ + surfactant in D-cyclohexane



'Wiggle' from surfactant shell

- $CaCO_3$ particles are spherical with dia. ~5.6nm
- Surfactant monolayer is of thickness ~1.8nm
- Water layer inserts between the calcium cation at the surface of the particle and the sufonate anion



Ref: Langmuir 2008, 24, 3807 - 3813

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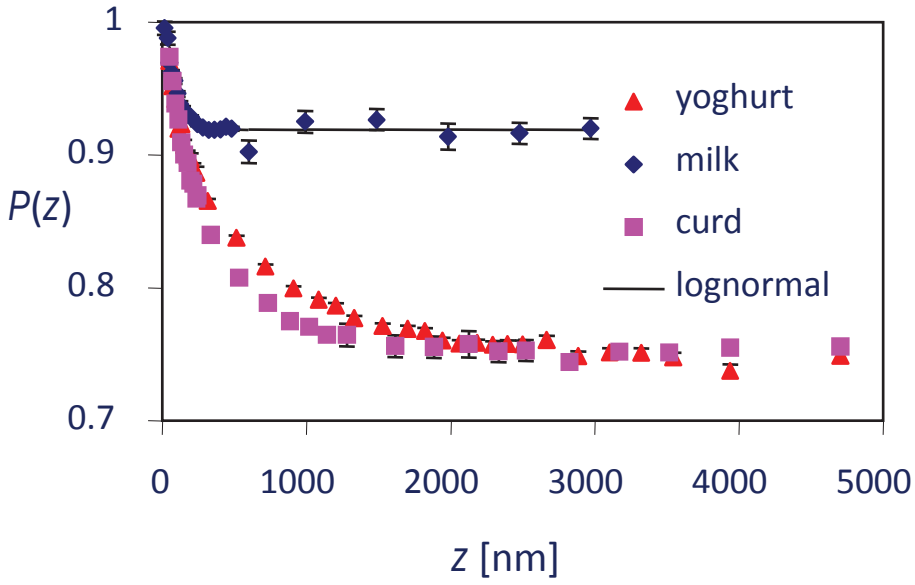
Dairy Products



- Stability of dairy products (for transport and increased shelf life) is very important
- Lengthscales of interest often fall well within SESANS range
- Samples can be studied *in-situ* whilst changing the conditions – temperature, pH etc.
- Here it can be seen how the particles in yoghurt and curd are much larger than those found in milk

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Engineering

H. Stone et al (Cambridge University)
Met Trans A **2011** *42A (1)*, 49-59

The System

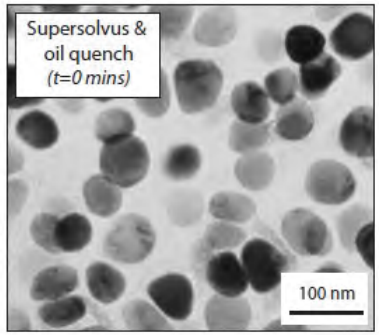
- Nickel-base superalloys as used in aero-engine turbine discs
- Exhibit both exceptional mechanical properties and superior corrosion resistance at high temperatures.

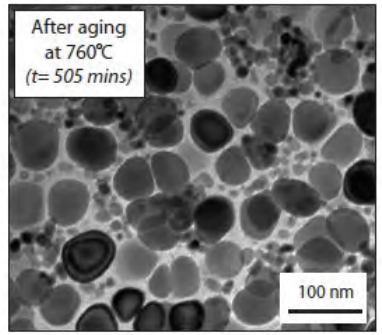
Why SANS?

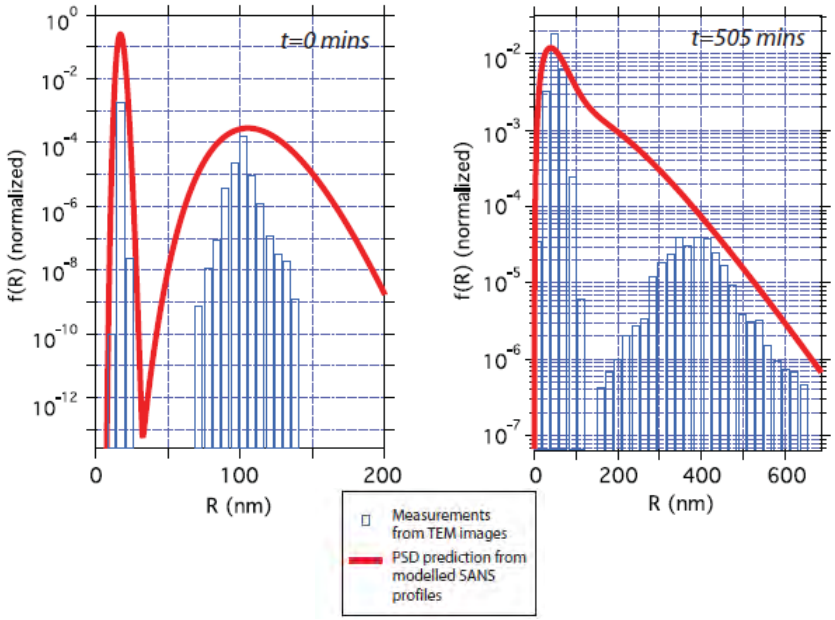
- Processes must be studied *in-situ*
- Large penetrating power of neutron allows structures buried deep in materials to be studied
- Lengthscales being probes are ideal for SANS

The Experiment and Results

- Ni alloy composed of γ' $Ni_3(Al, Ti, Ta)$ precipitates
- *In-situ* heating of sample using a furnace is used to mimic heat treatments during manufacturing process
- SANS results validate computer models of precipitates
- Understanding of these important alloys is improved







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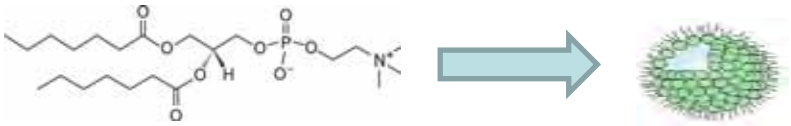
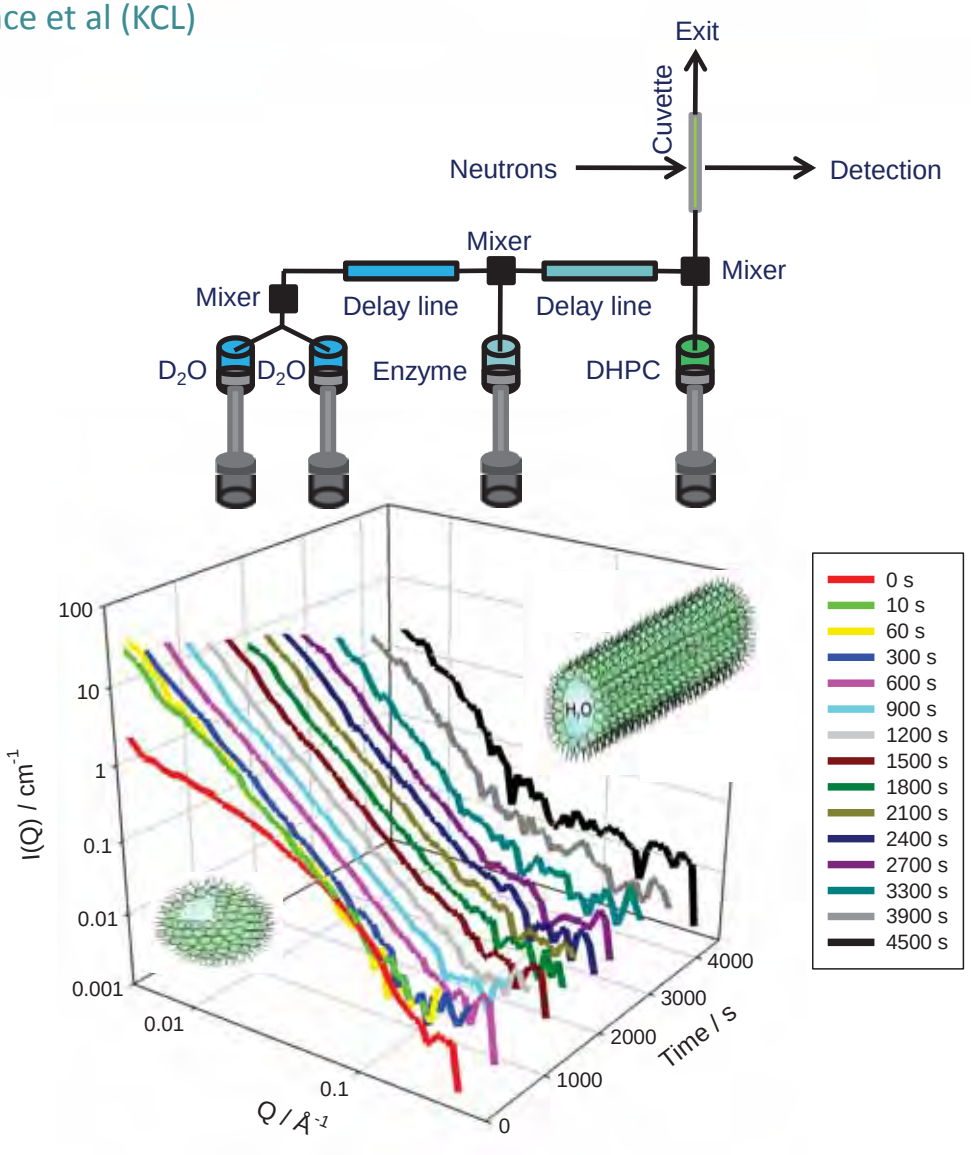
Time Resolved Measurements

J. Lawrence et al (KCL)

- T-O-F SANS is ideal for these measurements as a large lengthscale range can be studied in one shot
- Model system for cells in the body: the molecule is DHPC and forms ellipsoidal aggregates in solution



- This is a kinetic measurement – studying how the 'cell' structure is changed (damaged) when it is attacked by an enzyme – aggregates become more asymmetric with time
- Data collection is synchronized with the kit and automatic cycling is used to improve statistics
- Improved data acquisition electronics allows the data to be 'time sliced' after the measurement has been taken – now driven by science rather than electronics
- Happy Users!



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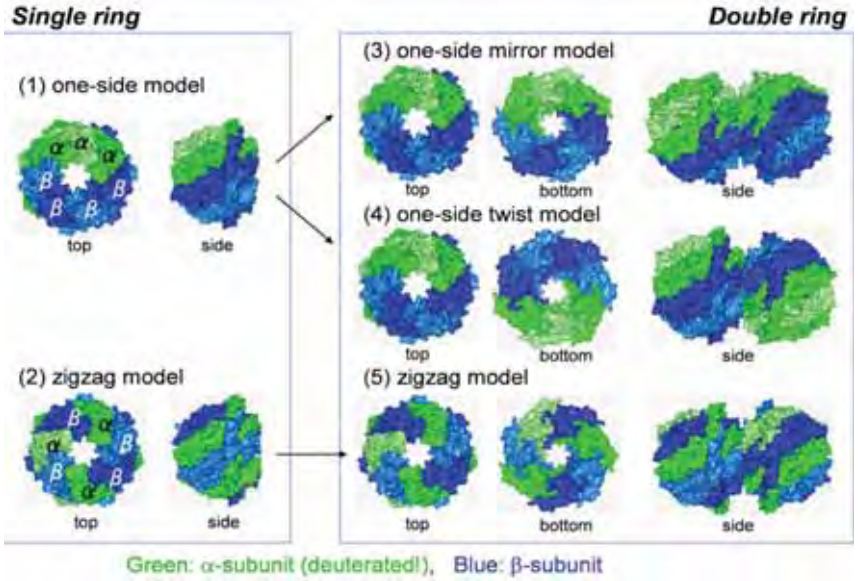
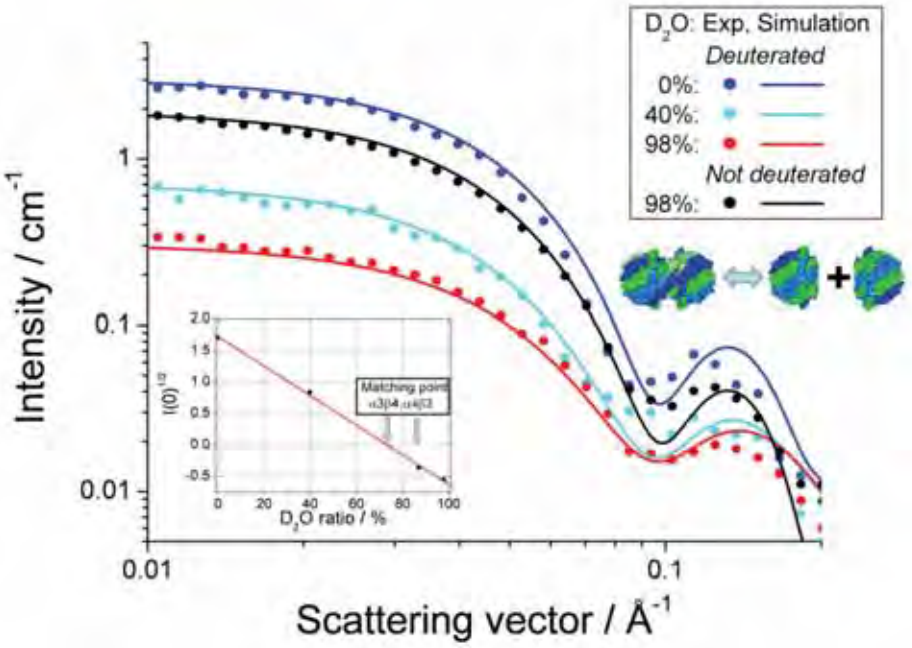
Solution structure of proteasome activator 28

M. Sugiyama et al (Kyoto University)

- In cells, 20S proteasome breaks down damaged or unneeded proteins for recycling into new ones
- Proteasome Activators (PA) regulate this function
- PA28 binds to proteasomes and the resulting complex promotes the production of immune-response peptides
- PA28 is comprised of a seven-membered ring containing two very similar subunits, named α and β

Possible models:





- Contrast variation shows that the PA28 heptamer rings are made up of three α and four β subunits in an alternating zig-zag
- SANS intensities also reveal that there is a well defined solution equilibrium between heptamer and its double-ring dimer

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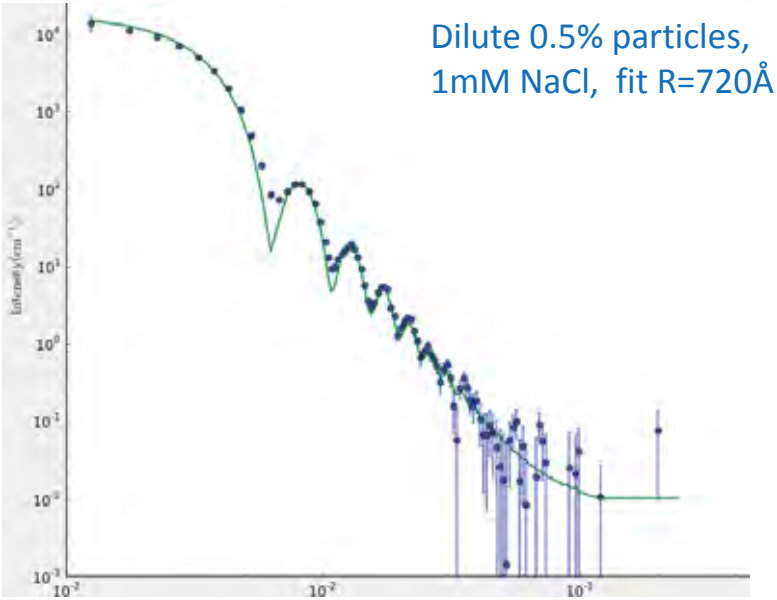
Crystallography with 0.14 micron “atoms”

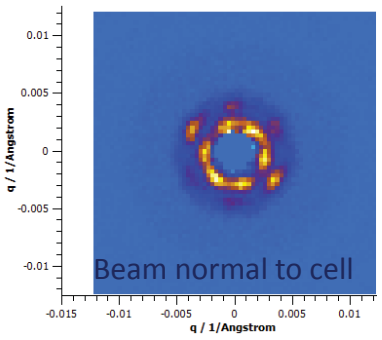
A.Rennie & M.Hellsing (Uppsala)

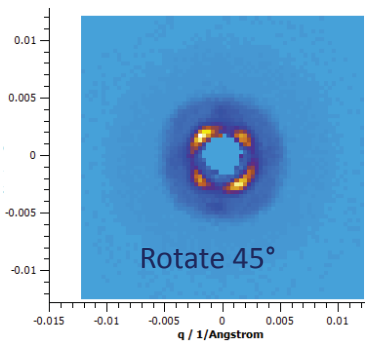
- Understanding the physics of flowing colloidal particles is important for many industrial processes
- Here near monodisperse 0.14 micron diameter polystyrene particles at 8% in water crystallise into domains a few mm in size.
- By rotating and rocking the colloidal crystal in the neutron beam the nature 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.

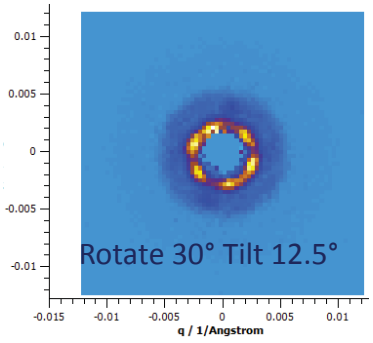


Dilute 0.5% particles,
1mM NaCl, fit R=720Å

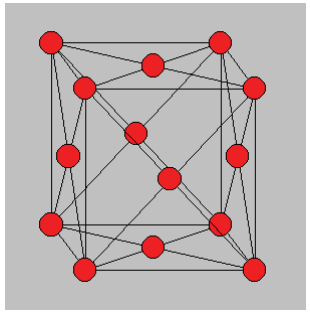
SANS from 8% particles







FCC



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GISANS – theory and system

Performed TOF GISANS on Sans2d

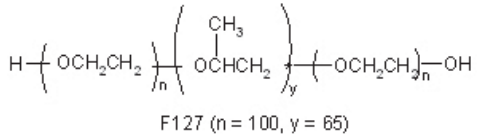
Nanoscale density correlation and/or shape of nanosized objects at surfaces, at buried interfaces or in thin films

α chosen between about half α_c and several α_c of the film material:

$\alpha < \alpha_c$ surface >> internal

$\alpha \geq \alpha_c$ surface and internal

$\alpha > \alpha_c$ surface << internal

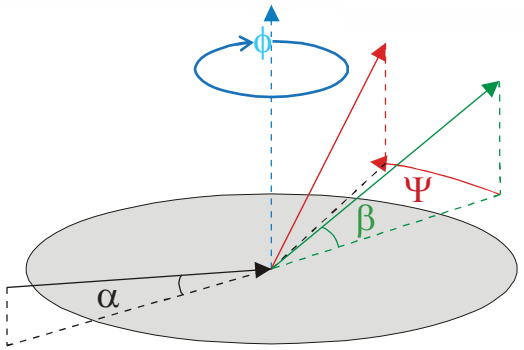


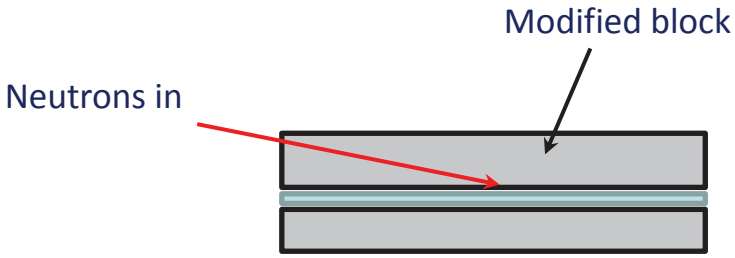
F127 (n = 100, y = 65)

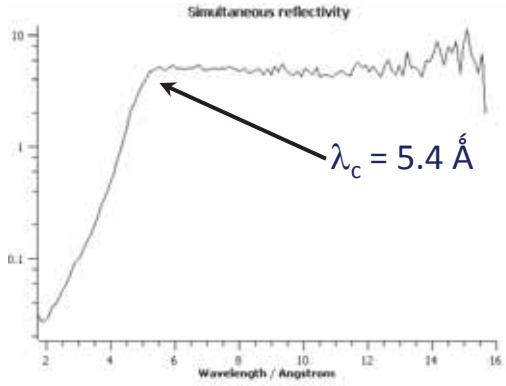
With TOF we capture GISANS simultaneously with bulk and surface scatter. “Near surface” SANS happens close to “critical wavelength” λ_c.

Sample is 20% F127 solution between modified Si blocks

Beam stop detector records simultaneous NR profile







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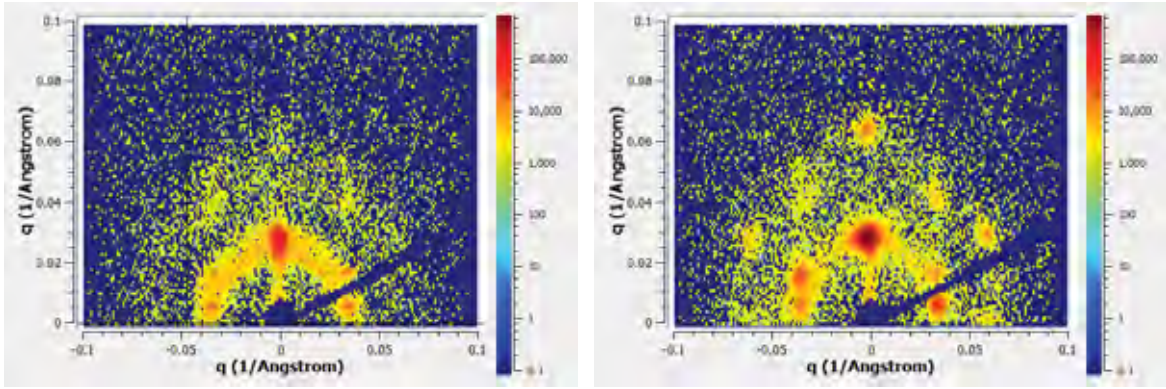
Crystallization of 20wt% F127 in D_2O via GISANS, M Wolff (Uppsala)

GISANS allows in-plane structure on the nm lengthscale to be studied

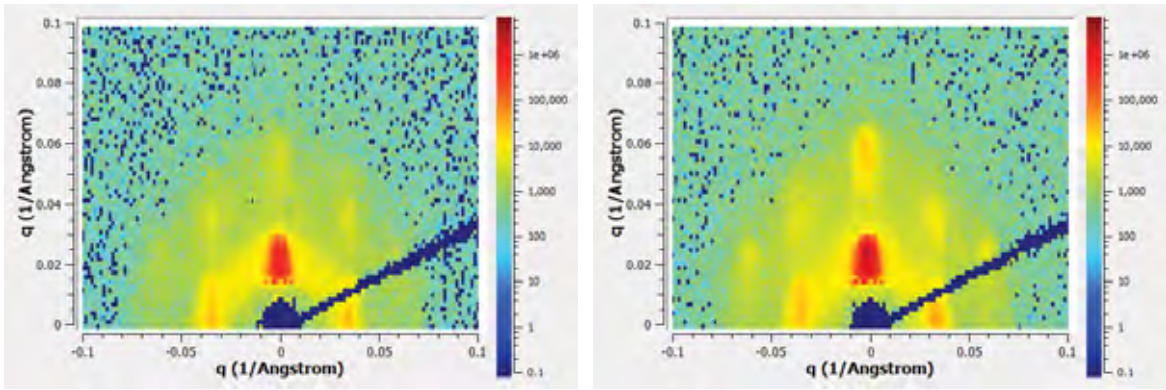
- Above and below critical temperature (T_c)
  - ➢ below T_c = micelles
  - ➢ above T_c = crystallization
- Studying the D_2O/Si interface. Two different Si surfaces
  - ➢ one hydrophobic (OTS)
  - ➢ one hydrophilic (piranha cleaned)
- Critical wavelength = 5.4 Å
  - ➢ Anything below 5.4 Å = bulk > surface
  - ➢ Anything above 5.4 Å = surface > bulk
  - ➢ **TOF gives you all the above conditions simultaneously**

Hydrophobic Hydrophilic

At λ_c GISANS shows different crystallization at surface



Below λ_c bulk structure is dominant and structures are the same



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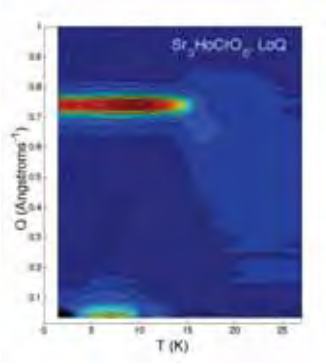
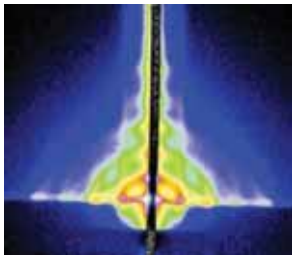
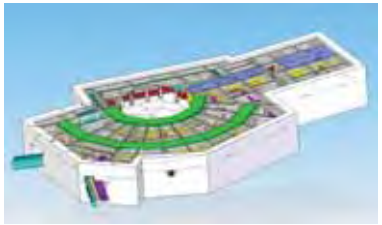


Summary:

- SANS and SESANS are very useful techniques 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 and SESANS is incredibly varied
- Well equipped offline labs allow for last minute sample preparation and alternative characterization
- *In-situ* and real time measurements possible
- The increased flux and improved signal to noise on TS-2 when compared to TS-1 allows weakly scattering samples to be studied more efficiently and also allows faster kinetics to be measured when compared to Loq
- Larmor is under construction and will allow polarised SANS to be performed and higher flux SESANS





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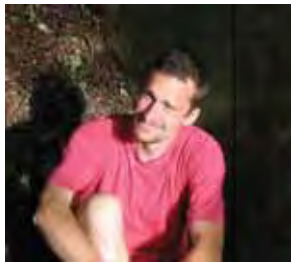
THANKS to…..

KING'S College LONDON



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And you for listening!