Overview

L8.1 – Introduction to Small Angle Neutron Scattering

L8.2 – SANS Instrumentation

EX8 – Virtual SANS Experiment

L9.1 – How to do a SANS Experiment

L9.2 – Small Angle Scattering Data Analysis

F9.3 – Applications of SANS

EX9 – Analysing Small Angle Scattering Data

How to Do a SANS Experiment

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NNSP-SwedNess Neutron School 2017, Tartu

Lecture L9.1

Planning an Experiment

- What is the question?
- Choosing samples
- Choosing an instrument
- Sample characterisation

As with any experiment, the question being asked must be carefully chosen.

SANS provides information about structure on the 1 to 100's of nm length scale

Is there contrast in the sample?

Do you need to use a deuteration scheme?

Can your system be studied as is, or does a model system need to be developed?

Planning an Experiment

- What is the question?
- Choosing samples
- Choosing an instrument
- Sample characterisation

Having identified the question, what samples are needed to answer that question?

This includes choices of concentration, deuteration, sample conditions (pH, temperature, pressure etc) and available sample amount.

Sample volumes for SANS are in the 0.1 to 1 ml range

Planning an Experiment

- What is the question?
- Choosing samples
- Choosing an instrument
- Sample characterisation

The choice of instrument is determined by:

- Required Q range
- Required beam flux
- Availability of access
- Availability of sample environment

To determine the requirements of Q range and flux, the scattering should be simulated.

Counting times are between minutes and hours per sample.

This requires some knowledge or expectation of what the sample structure will be.

The simulation can often be performed using the tools that will be used for data analysis.

Planning an Experiment

- What is the question?
- Choosing samples
- Choosing an instrument
- Sample characterisation

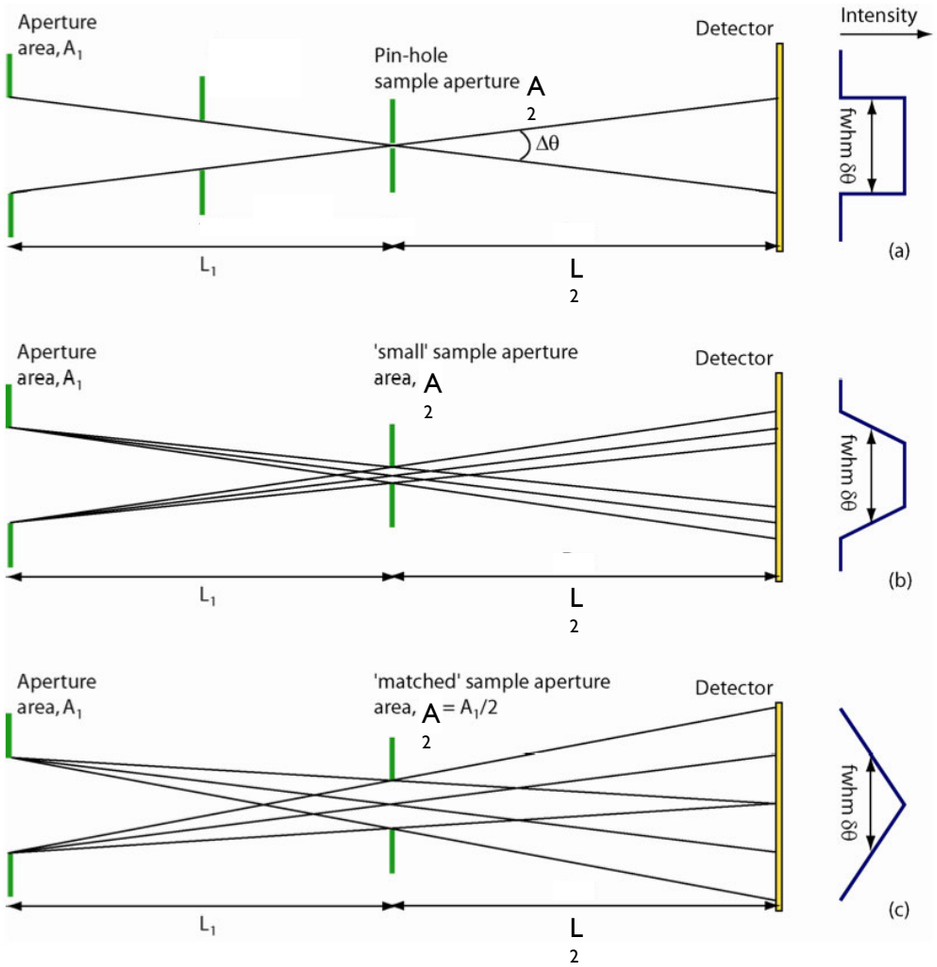
SANS is a relatively expensive technique that is uniquely capable for answering specific questions about nanoscale structure.

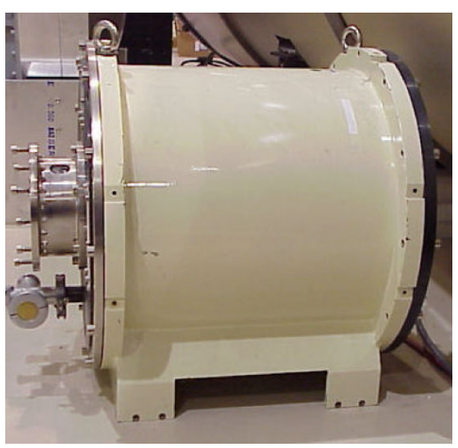
In order to make best use of SANS, the samples should be characterised with other techniques before planning and executing the SANS experiment.

Thus, for example, if light scattering or lab SAXS are available, these should be measured. Perhaps microscopy (light or electron) would be appropriate.

Bear in mind that these other techniques measure different aspects of the sample from SANS, but are all valuable information in being able to understand the SANS data.

Setting up the Instrument

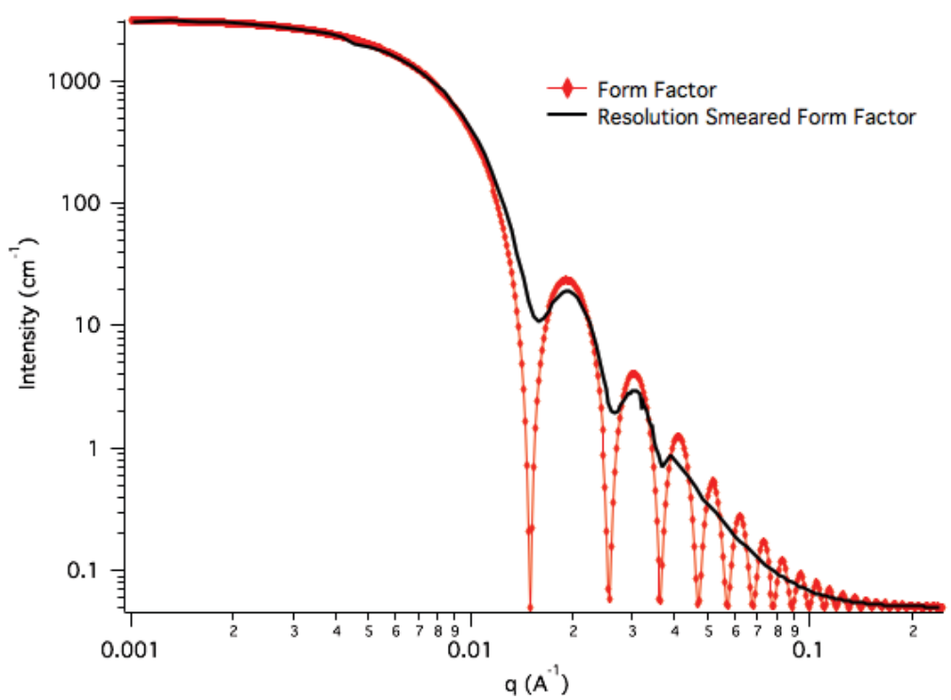




The instrument scientist who is your contact at the scattering facility ("local contact") will help you to determine the best instrument settings for your experiment.

You need to choose:

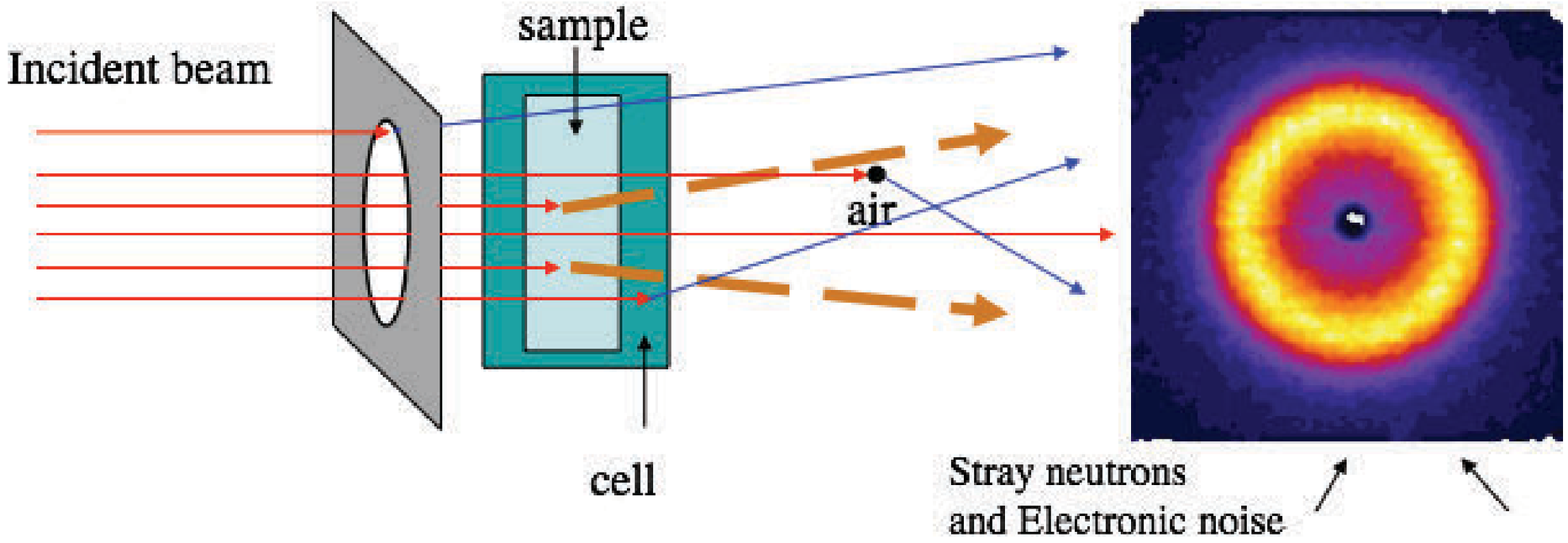
Collimation length
Aperture sizes
Wavelength or wavelength range
Detector position



Making a measurement

Contributions to counts on the detector:

1. Scattering from sample (what we want!)
2. Scattering from other than the sample (neutrons still go through sample)
3. Stray neutrons and electronic noise (neutrons don't go through sample)



$$I_{meas}(i) = \Phi\, t\, A\, \varepsilon(i)\, \Delta\Omega\, T_{c+s}[(d\Sigma/d\Omega)_s(i)\, d_s + (d\Sigma/d\Omega)_c(i)\, d_c] + I_{bgd}\, t$$

Making a measurement

$$\frac{d\Sigma}{d\Omega} \propto I(Q)_{measured}$$

$$\mathbf{I_{meas}(i) = \Phi\ t\ A\ \varepsilon(i)\ \Delta\Omega\ T_{c+s}[(d\Sigma/d\Omega)_s(i)\ d_s + (d\Sigma/d\Omega)_c(i)\ d_c] + I_{bgd}\ t}$$

$\mathbf{\Phi}$ = neutron flux on sample
t = counting time for measurement
A = sample area
$\mathbf{\varepsilon(i)}$ = detector element efficiency
$\mathbf{\Delta\Omega}$ = detector element solid angle

T_{c+s} = measured transmission of sample and holder
d_s = thickness of sample
d_c = thickness of cell
I_{bgd} = stray neutrons and noise

We must make the necessary measurements:

A. Scattering with sample in the neutron beam
B. Scattering with an empty sample holder in the neutron beam
C. Scattering with the sample position blocked by a neutron absorber
D. The direct beam intensity with nothing in the neutron beam
E. The direct beam intensity with the sample in the neutron beam
F. The direct beam intensity with the sample holder in the neutron beam
G. A measurement of the detector response variation (usually done by the facility before your experiment)

Your local contact for your experiment will make sure that these things are measured and the facility will provide the software necessary for you to leave with "reduced data" on "absolute scale" which is what you need to be able to perform an analysis and answer your scientific question.

What does it look like?

Image from ORNL

Two SANS Instruments @ HFIR reactor at Oak Ridge National Lab

What does it look like?

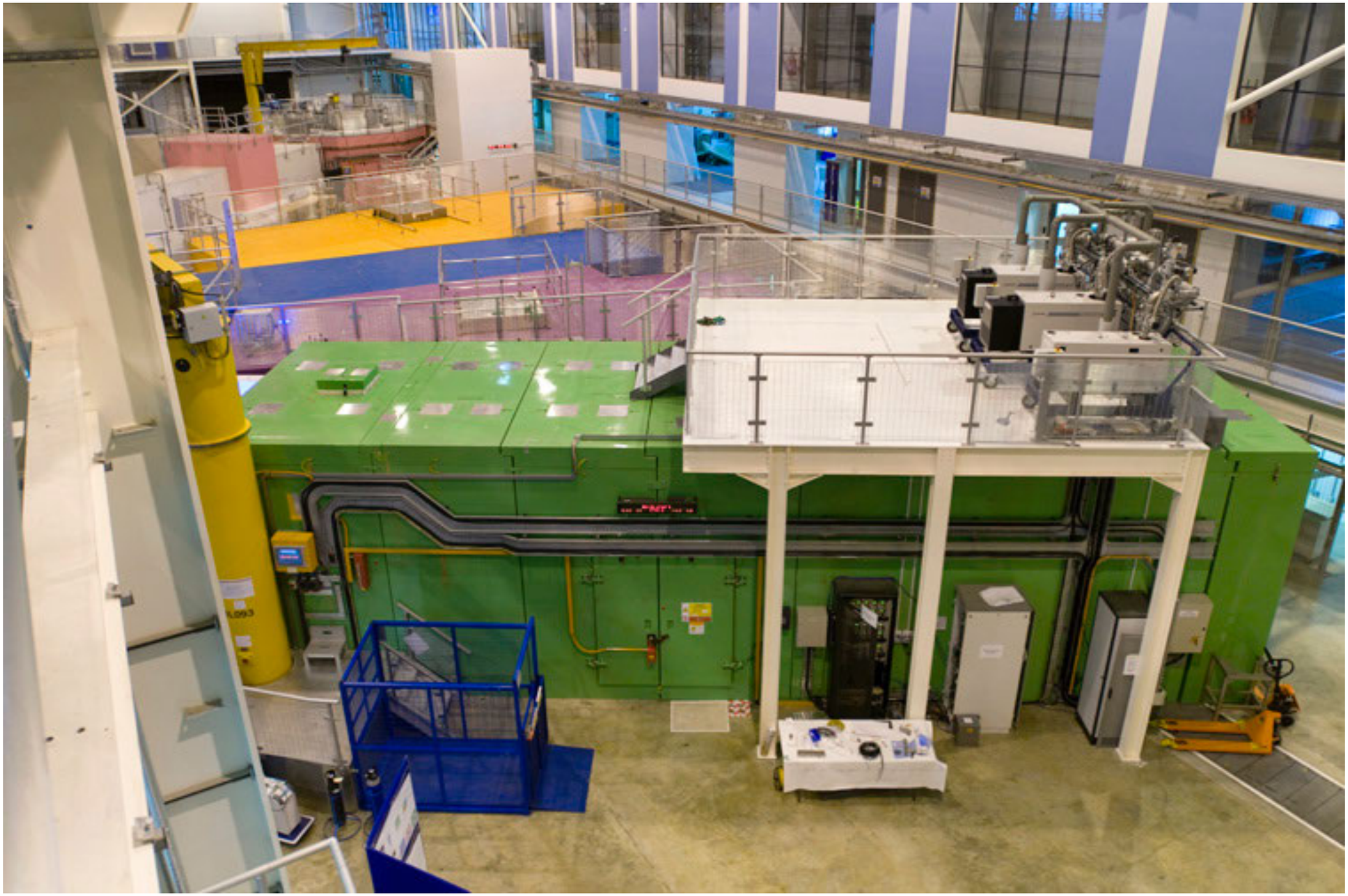
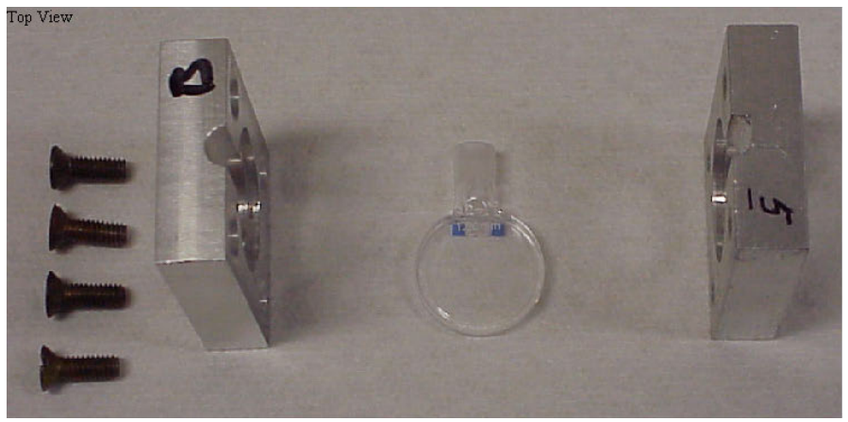


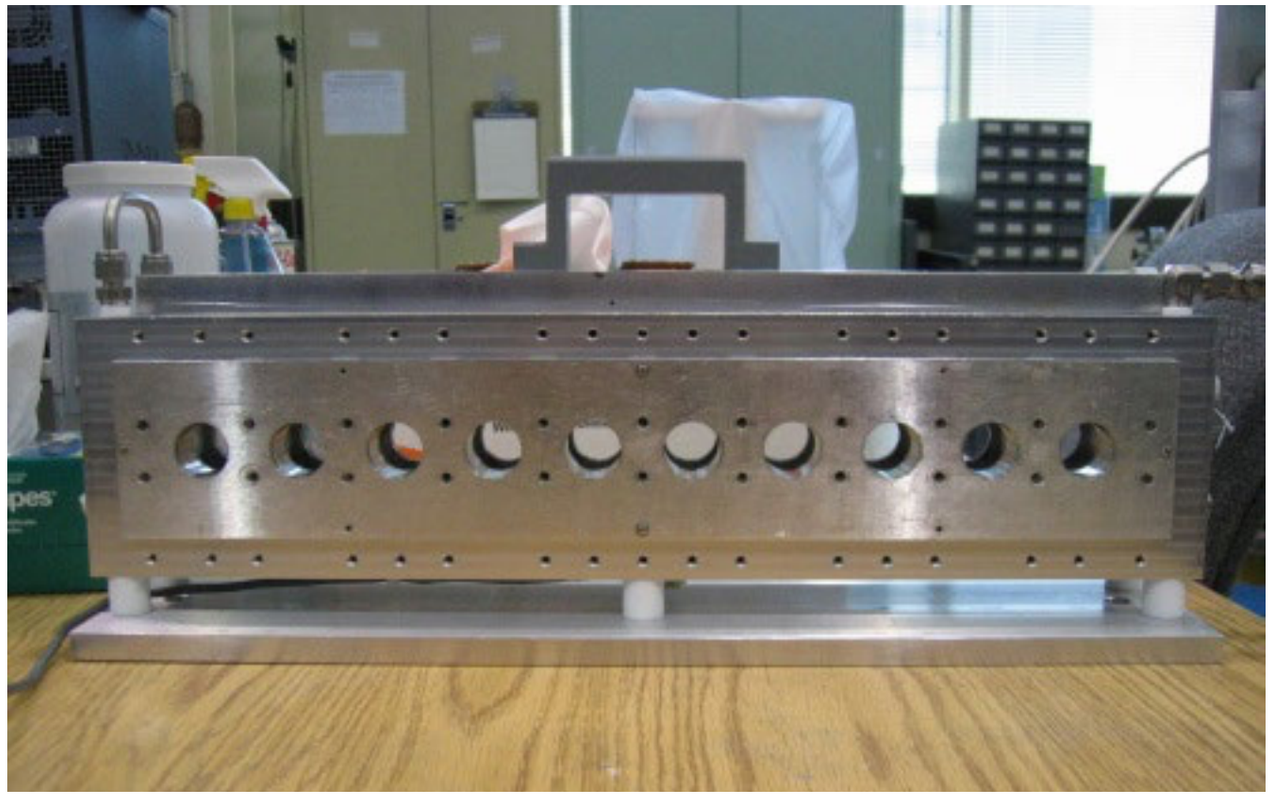
Image from ISIS/STFC

SANS instrument @ ISIS spallation neutron facility

What does it look like?

Sample Cells





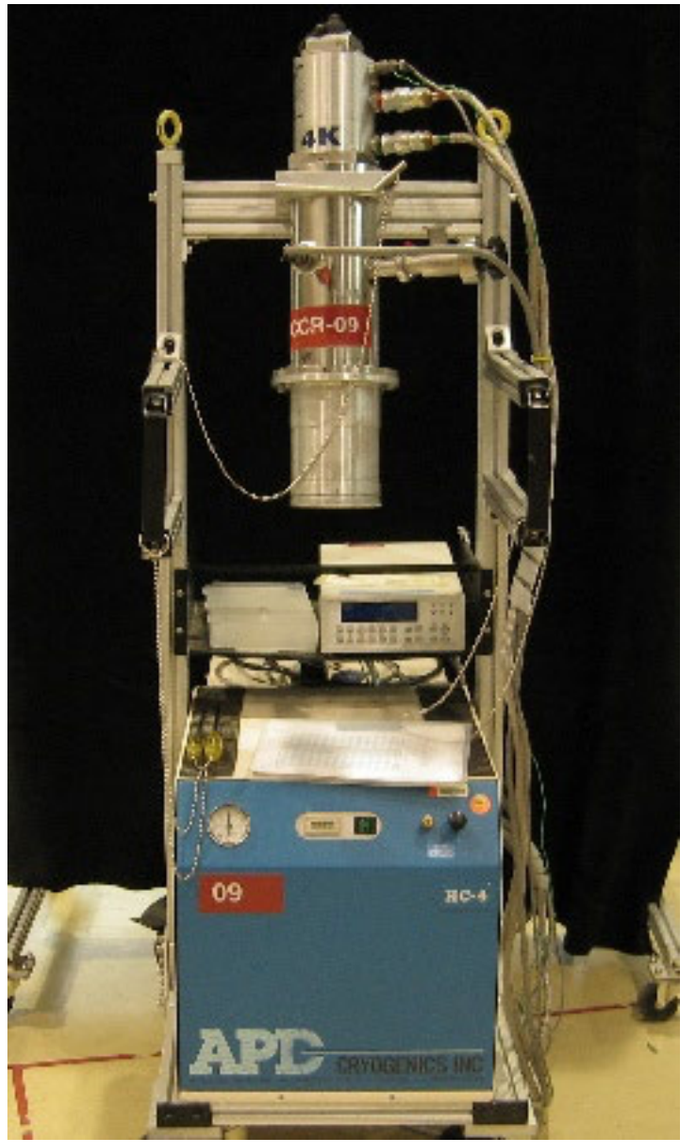
Temperature Controlled Sample Changer

Images from NIST Center for Neutron Research

Sample environment is the various equipment that the sample is placed in - usually to apply a stimulus to the sample

What does it look like?

Rheometer



Closed Cycle Refrigerator

Humidity Chamber

Images from NIST Center for Neutron Research

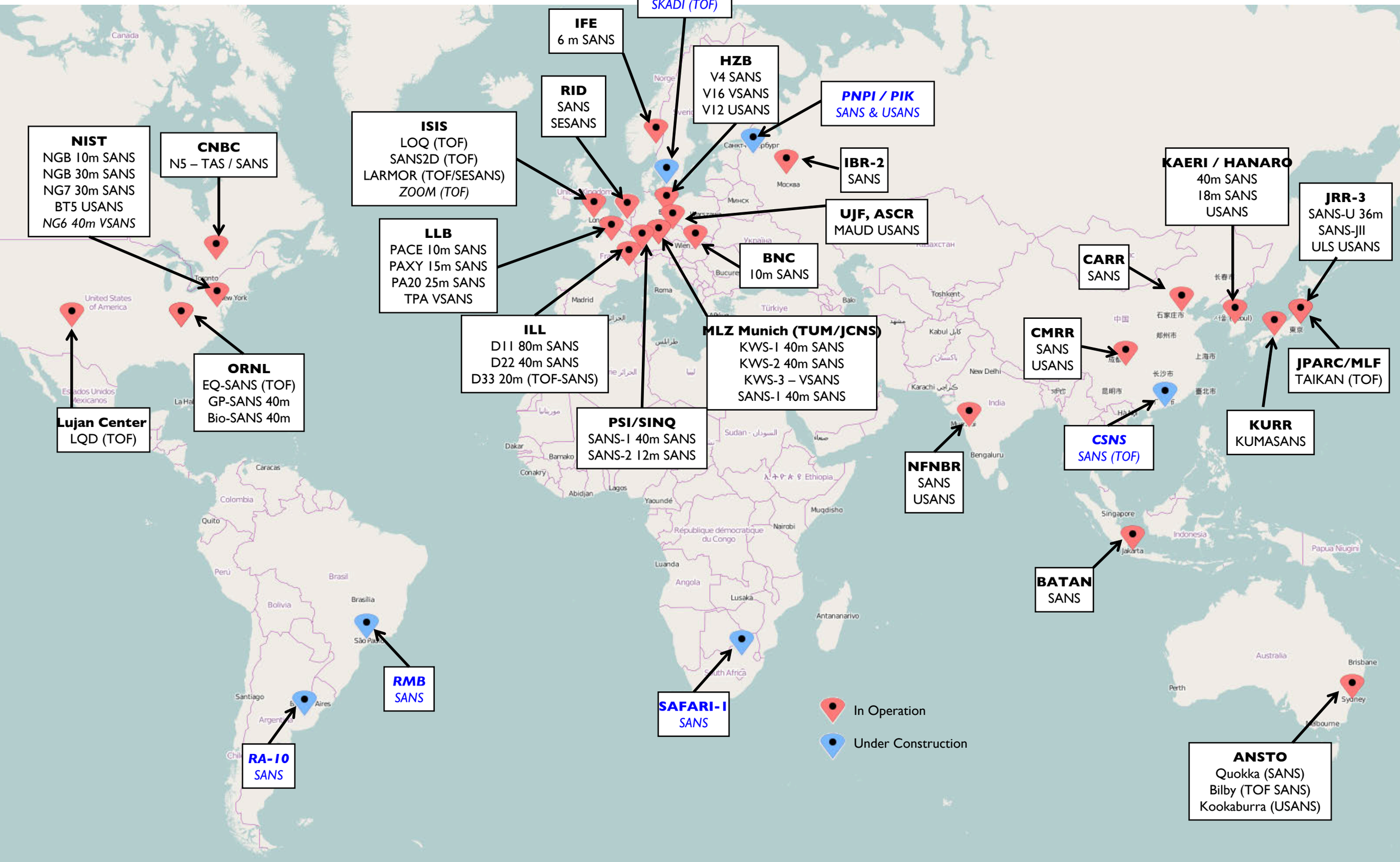
Sample environment is the various equipment that the sample is placed in - usually to apply a stimulus to the sample

What does it look like?

Image from ISIS/STFC

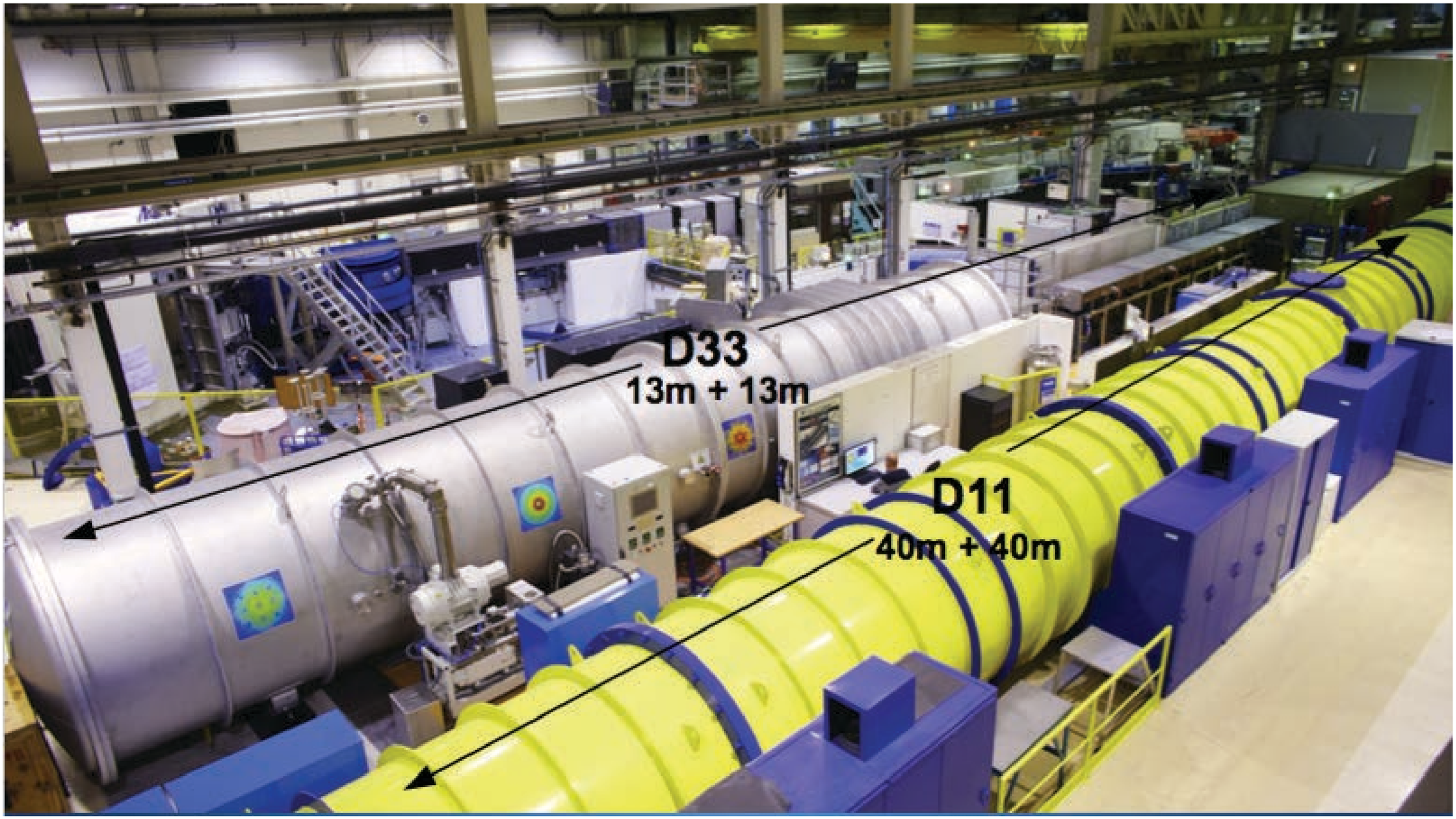
SANS sample position at SANS2D @ ISIS with 17T superconducting cryomagnet in place

SANS Instruments Around the World



SANS Instruments Around the World

ILL



SANS Instruments Around the World

ILL

D11

Monochromator	
velocity selector	
Eleanore/ Felicia (ASTRIUM standard selectors)	$\Delta\lambda/\lambda$ = 9% (FWHM)
incident wavelength	variable, 4.5 ≤ λ [Å] ≤ 40
max. wavelength at max. detector dist. (39 m)	λ = 22 Å
selector rotation normal to n-beam possible minimum wavelength at rotation -7 deg	-7 deg to + 7 deg λ = 3.2 Å
Collimation	
11 guide sections (computer controlled) uncoated glass guides (SwissNeutronics) diverging "trumpet" + straight guide + focussing guide near sample position	cross section (height x width) : 50 x 30 mm^2 -> 50 x 45 mm^2 over 6.5 m straight guide 50 x 45 mm^2 over 28 m 50 x 45 mm^2 -> 35 x 31.5 mm^2 over 4 m
guide-to-sample distances	1.5, 2.5, 4, 5.5, 8, 10.5, 13.5, 16.5, 20.5, 28, 34, 40.5 m (12 discrete distances)
Attenuators	
choice between 3 cadmium sheets of different transmission (computer controlled) T_a=3.461 10^{-3} (att 1), T_a=1.089 10^{-3} (att 2), T_a=3.524 10^{-4} (att 3)	
Sample area	
flux at specimen at lowest resolution	$1 \cdot 10^8$ $ncm^{-2}s^{-1}$
typical sample size	10 x 10 mm^2
Detector	
sample-to-detector distances *L*	variable between 1.2 m and 39 m
momentum transfer range	$3\ 10^{-4} \le Q\ [Å^{-1}] \le 1$
detector type	^{3}He gas detector (CERCA)
area	96 x 96 cm^2
pixel size	7.5 x 7.5 mm^2
background	1 Hz on whole multidetector
detector deadtime	420 ns

D22

Monochromator	
velocity selector Anatole	$\Delta\lambda/\lambda$ = 10 % (standard)
wavelength	4.5 < λ/Å < 40 (for $\Delta\lambda/\lambda$ = 10 %)
Collimation	
8 guide sections	55 x 40 mm
source-to-sample distances / m	1.4, 2.0, 2.8, 4.0, 5.6, 8.0, 11.2, 14.4, 17.6, variable apertures at 19.1
Sample area	
maximum flux at sample (for $\Delta\lambda/\lambda$ = 10%)	$1.2x10^8$ n cm^{-2} s^{-1}
typical sample size	10 to 300 mm^2
Detector	
distances	1.1 ... 17.6 m
rotation	-2° < 2Θ <22°
horizontal offset	-5 ... 50 cm
area	102.4 x 98 cm^2
pixel size	8 x 8 mm^2
maximum counting rate	5 MHz
electronic noise	2 Hz for the whole multidetector

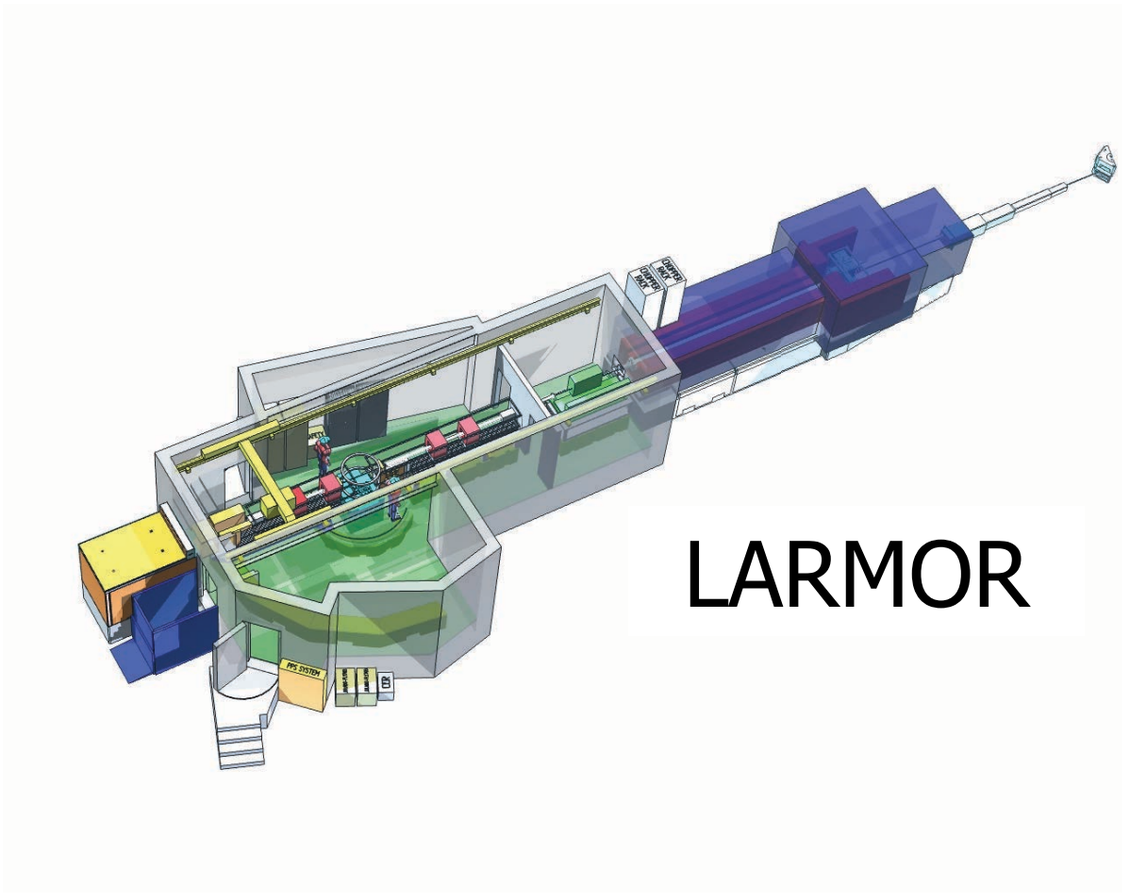
D33

Wavelength Definition	
Monochromatic Mode:	
Velocity selector (Astrium)	$\Delta\lambda/\lambda$ = 10 % (standard)
Wavelength range	4.5 < λ/Å < 40 (for $\Delta\lambda/\lambda$ = 10 %)
Time-of-Flight (TOF) Mode:	
4-chopper system (Astrium)	
Wavelength cut-offs	14 Å and 20 Å
Wavelenth resolutions	$\Delta\lambda/\lambda$ = 2 % to 26 % (depending on chopper pair & detector distance)
Collimation	
4 movable guide sections	2.5 m
Beam nose	2.8 m
Guide cross-section	30 x 30 mm
Source-to-sample distances (m)	2.8, 5.3, 7.8, 10.3, 12.8
Source apertures	30x50 mm (off-centre), 30x30 mm diameters: 5, 10, 20, 30 mm
Sample area	
Maximum flux at sample (for $\Delta\lambda/\lambda$ = 10%)	$4.1x10^7$ n cm^{-2} s^{-1}
Brightness (flux / unit solid angle)	$3.57x10^{11}$ n cm^{-2} s^{-1} $strd^{-1}$
Maximum sample dimensions	15 mm x 15 mm
Sample environments	Sample changer, Electromagnet, Cryostat, Cryomagnet, Furnace, Stopped-flow, Shear cell
Detectors	
Sample - Detector distances	1.2 ... 12.8 m
Detector 1 (rear):	
Single panel monoblock	640 x 640 mm
Pixel size	5 x 5 mm^2 (128 x 128 pixels)
Maximum count rate	4 MHz (global) ; 3 kHz/pixel (local)
Detector 2 (front):	
4-panel monoblock	160 x 640 mm each panel
Pixel size	5 x 5 mm^2 (32 x 128 pixels)
Maximum count rate	4 MHz (global) ; 3 kHz/pixel (local)

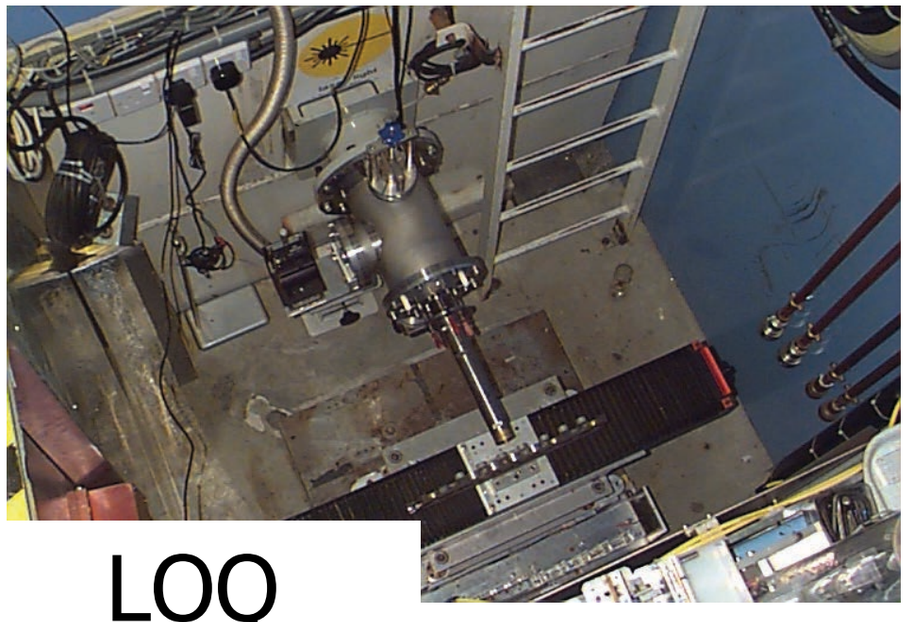
SANS Instruments Around the World

SANS2D

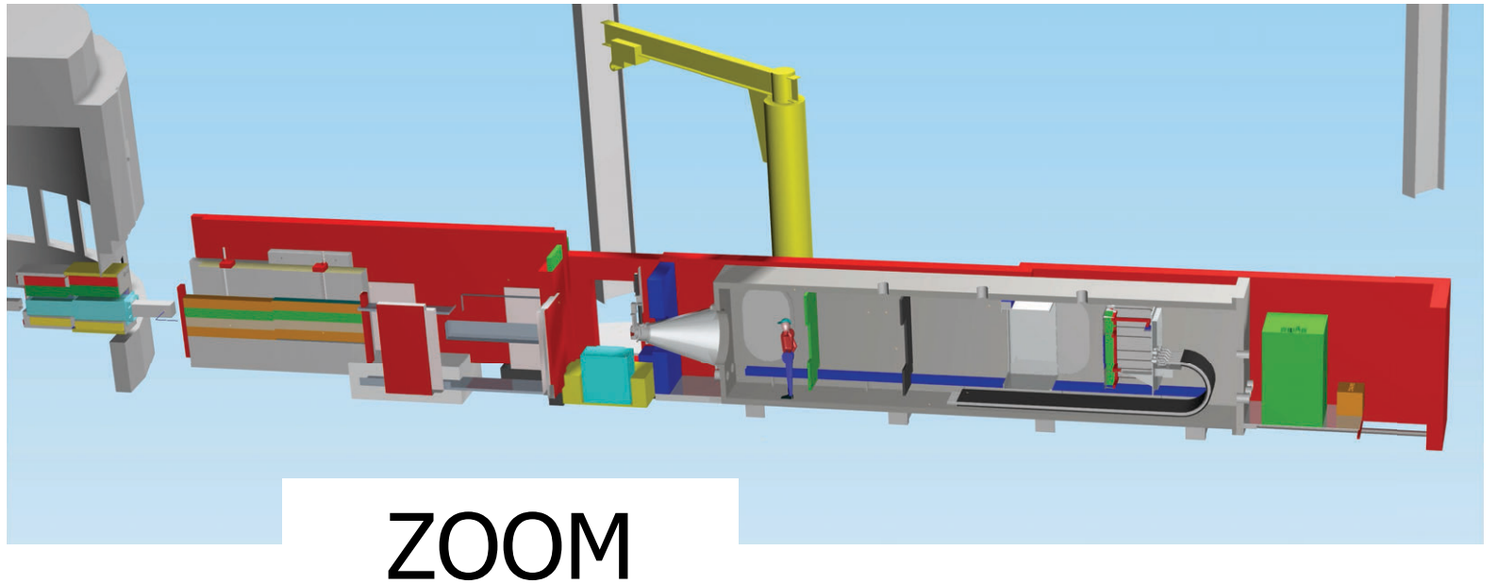
ISIS



LARMOR



LOQ



ZOOM

SANS Instruments Around the World

SANS2D

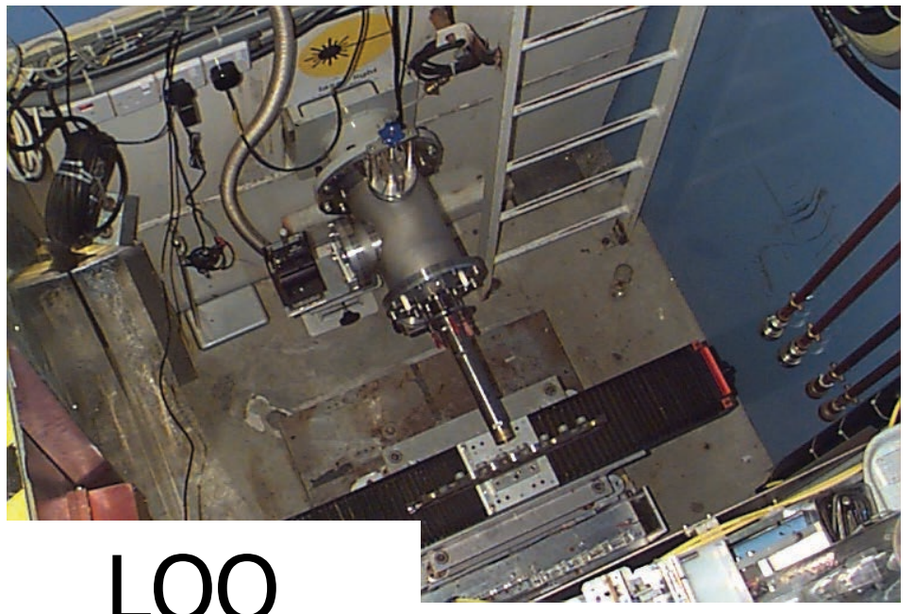
Incident wavelengths	2.0 - 14.0 Å at 10 Hz
Momentum transfer, Q	Depends on sample-detector distances and detector offsets: Q_{min}~0.002 $Å^{-1}$, Q_{max}~3 $Å^{-1}$

ISIS

Isis Beamline	E2, viewing the coupled cold moderator
Primary flight path	$3\Theta_c$Ni super-mirror bender (to remove neutrons with wavelengths less than 1.5 Å), upstream scintillator monitor, overcount protection fast shutter, variable-opening counter-rotating disc chopper, 5 x 2 m moveable evacuated collimation sections (each with choice of Ni guide sections or plain pipe) for variable incident collimation, 5 moveable aperture strips (each with different size apertures), sample position scintillator beam monitor, sample aperture strip, final collimation tube.
Sample position	Around 19 m from moderator. Side access. No height restriction. Beam is approximately 1 m above base plate. Crane access possible (SWL <5000 Kg). Sample transmission scintillator monitor on motorised rack. Provided with water, helium and electrical services.
Beam size at sample	Defined by sample aperture strip and final collimation. Up to 15 mm diameter is possible, typically 8 mm diameter.
Neutron flux at sample	Dependent on collimation, accelerator performance and target type. Typical time-averaged flux is *currently estimated* to be >10^6 cm^{-2} s^{-1} (ISIS TS2 at 10Hz, 40 uA 800 MeV proton beam, tantalum target).
Secondary flight path	Evacuated tank containing detectors.
Detectors	*Two* ^{3}He-CF_4 filled ORDELA "area" detectors. Active area of each is 96.5 cm x 96.5 cm with 5 mm resolution. The detectors can be moved in the vacuum tank both along the beam (to vary sample-detector distance between 2 and 12 m) and to give a sideways offset (to extend the Q range at a given detector distance) of up to 1200 mm. The front detector can also be rotated to face the sample. Detector mapping under software control.

SANS Instruments Around the World

Incident wavelengths	2.2 - 10.0 Å at 25 Hz, 2.2 - 6.7 Å or 6.3 - 10.0 Å at 50 Hz
Momentum transfer, Q	0.006 - 0.24 Å-1 (main detector) 0.15 - 1.4 Å-1 (high-angle bank)
Dynamic range in Q	40 (on main detector), 230 (simultaneous use of all detectors)



LOQ

ISIS

Isis Beamline	N5, viewing the 25 K liquid hydrogen (lower) moderator.
Primary flight path	Soller supermirror bender (24 mrad, to remove neutrons with wavelengths less than 2 Å), upstream scintillator monitor, aperture dial No 1, variable-opening (2 - 126 degrees) disc chopper, frame overlap mirror (removes neutrons with wavelengths greater than 12 Å), 3 m evacuated flight tube, sample position scintillator beam monitor, aperture dial No 2, final collimation tube.
Sample position	Around 11.1 m from moderator. Approximate size is 0.4 m (parallel to beam) by 1.5 m. No height restriction. Beam is approximately 0.63 m above base plate. Crane access possible (SWL 1000 Kg). Sample transmission scintillator monitor on motorised rack. Provided with water, helium and electrical services. Secondary, top-loading, in-vacuum sample position with limited access and services around 12.5 m from moderator (giving an approximate Q range of 0.01 - 0.34 Å-1).
Beam size at sample	Defined by aperture No 2 and final collimation. Between 2 - 20 mm diameter. Typically 8 mm diameter.
Neutron flux at sample	Dependent on collimation, ISIS accelerator performance and target type. Typical time-averaged flux is 2x10^5 cm-2 s-1 (ISIS TS1 at 40Hz, 160 uA 800 MeV proton beam, tantalum target).
Secondary flight path	Evacuated tank to main detector.
Detector	3He-CF4 filled ORDELA "area" detector 15.15 m from moderator. Active area is 64 cm x 64 cm with 5 mm resolution. Detector mapping under software control. External, annular, high-angle, scintillator "area" detector bank 11.6 m from moderator.

Summary

Careful planning is needed to get the most information from a SANS experiment

Processing the data requires knowledge of some instrument specific values and calibrations – these will be provided by the facility.

So, choice of SANS instrument is driven by the needs of the experiment in terms of **Q-range**, **resolution** and **sample environment**

Questions?

Small Angle Scattering Data Analysis

Andrew Jackson

NNSP-SwedNess Neutron School 2017, Tartu

Lecture L9.2

What is SANS Data Analysis?

$$\frac{d\Sigma}{d\Omega} = \frac{N}{V}\frac{d\sigma}{d\Omega} = \frac{1}{V}\left|\int_V \rho(\vec{r})e^{i\vec{q}\cdot\vec{r}}d\vec{r}\right|^2$$

"Rayleigh-Gans Equation"

Thus, inhomogeneities in $\rho(\vec{\mathbf{r}})$ give rise to small angle scattering

Aim of data analysis is (usually) to extract information about the structure of the system from the scattering data.

This means recovering information about ρ(r) from I(Q)

SANS Data Analysis

Model Independent

We can use an approximation from Guinier

$$I(Q) = I(0)e^{-\frac{(QR_g)^2}{3}}$$

$$ln(I(Q)) = ln(I(0)) - \frac{R_g^2}{3}Q^2$$

to obtain the radius of gyration of the scattering objects assuming particulate scatterers and S(q) = 1.

Similar approximations can be made to get the cross section of cylinders or the thickness of disks. Various other model independent approaches exist to extract information from the data without a scattering model.

Indirect Fourier Transform

Since we are missing the phase information as a result of the differential cross section being related to the square of the amplitude of the fourier transform, we cannot simply take the fourier transform of our data to get back to ρ(r). Thus we must use an indirect method.

A popular implementation of this method is found in the *ATSAS suite* of software from Prof. Svergun's group. *SasView* also has an implementation of this method.

Model Dependent

We calculate the form and structure factors for a given scattering system and compare that with the measured scattering data. The model is fitted to the data to obtain the parameters that describe the scattering. We can simultaneously fit multiple contrasts to be able to study complex structures.

The software we will be using for this course is called *SasView* (http://www.sasview.org) and is being jointly developed by NIST, ILL, ISIS, SNS, ANSTO and ESS. Other software packages for this kind of analysis include the *NIST Igor Macros* developed at the NCNR and *SasFit* developed at the Paul Scherrer Institute.

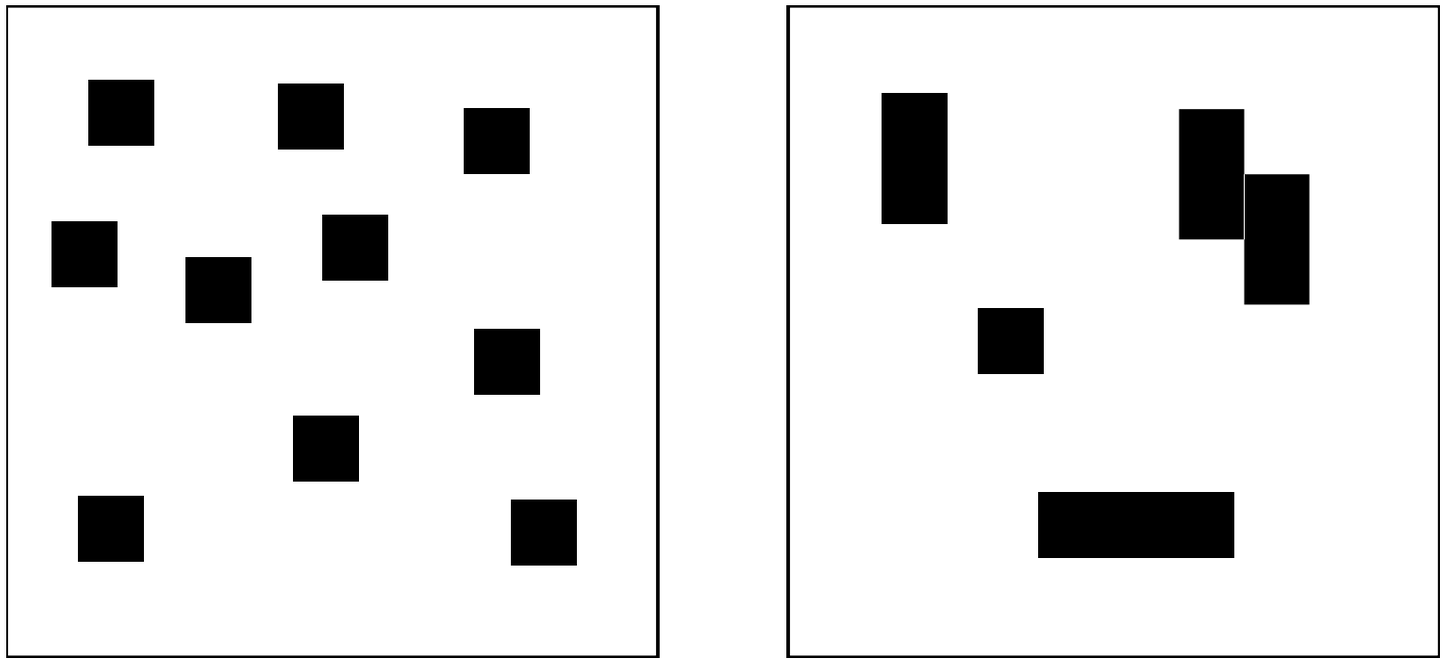
Ab-inito Structure Generation

An approach that is popular for bio-macromolecules in solution is to generate a structure from many sub-resolution spheres and calculate what the scattering would be. That is then compared with the data and the spheres redistributed. This is repeated until agreement is found.
The *ATSAS suite* is the primary example of software using this method

Model Independent

Scattering Invariant

Porod showed that the total small angle scattering is invariant, irrespective of how the matter is distributed.



Two systems where the contrast and volume fraction are the same, but the distribution of matter is different. Both are 10% black and 90% white.

$$Q = \int \frac{d\Sigma}{d\Omega}(\mathbf{q})d\mathbf{q}$$
$$= (2\pi)^3(\rho(\mathbf{r}) - \overline{\rho})^2$$

$$\frac{Q}{4\pi} = Q^* = 2\pi^2\phi_1(1-\phi_1)(\rho_2 - \rho_1)^2$$

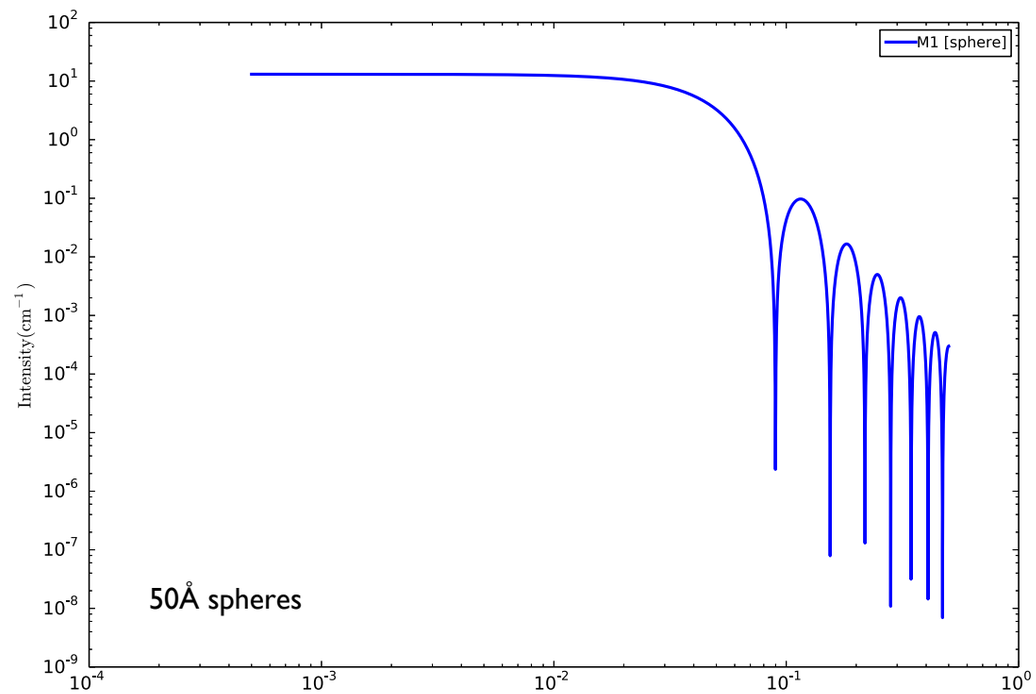
Model Independent

Guinier Plot

We can use an approximation from Guinier[1] for spherical/globular objects

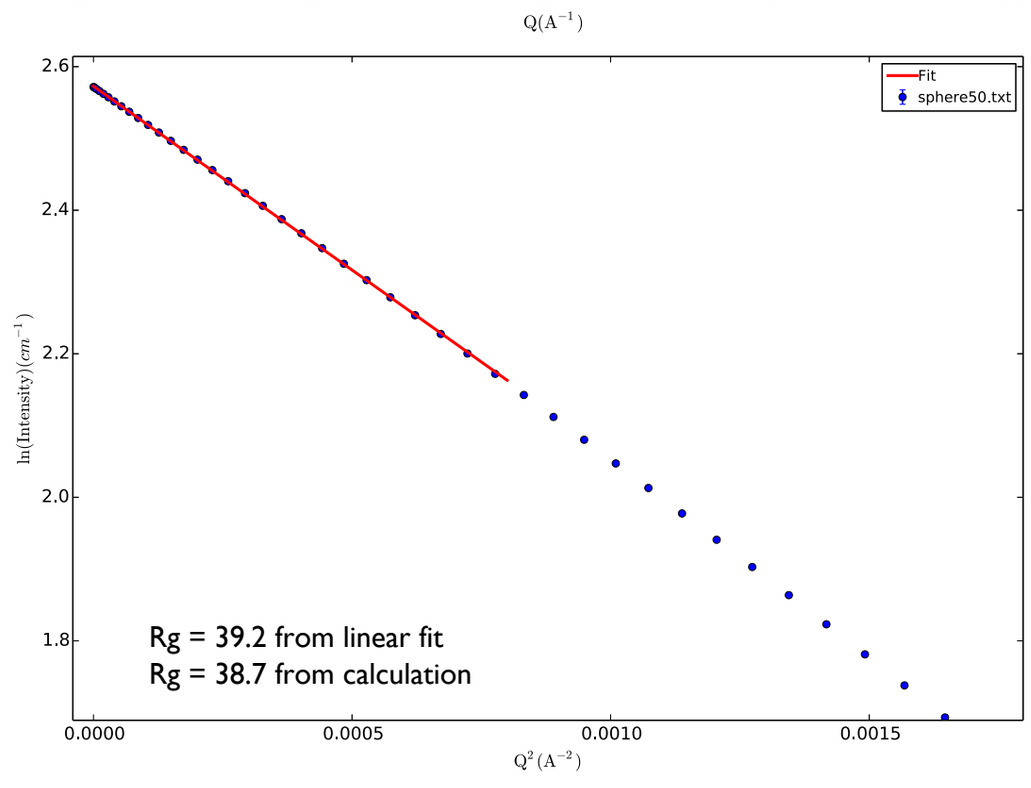
$$I(Q) = I(0)e^{-\frac{(QR_g)^2}{3}}$$

$$ln(I(Q)) = ln(I(0)) - \frac{R_g^2}{3}Q^2$$



Perform fit for y(x) = ax + b

Parameter a	-512	+/-	7.37e-15
Parameter b	2.57	+/-	2.25e-18
Chi2/dof	5.78e+26		
	Min		Max
Maximum range (linear scale)	0		0.0283
Fit range of x^(2)	0		0.0008
I(q=0)	13.1	+/-	2.95e-17
Rg [A]	39.2	+/-	2.82e-16
Rg*Qmin	0		
Rg*Qmax	1.11		



[1] Guinier A. (1939) Ann. Phys. 12, 161

Model Independent

Guinier Plot

For non spherical objects, similar results exist.

In the case of rod-like and disk-like or lamellar objects, the overall radius of gyration is given similarly to spherical objects from a plot of Ln[I(Q)] vs Q^2:

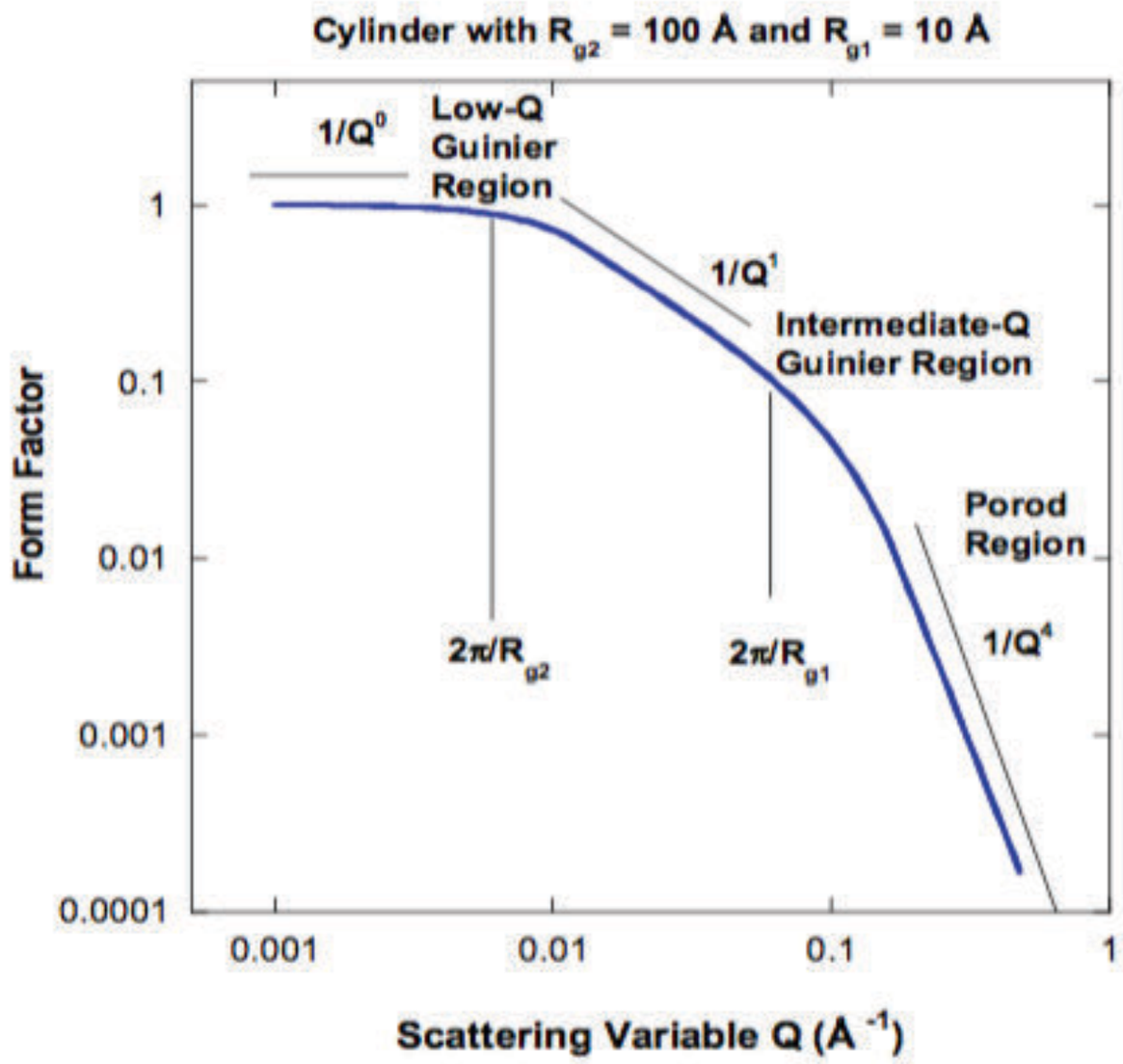
$$I(Q) = I(0)\exp\left(-\frac{Q^2 R_g^{\,2}}{3}\right) \quad \text{where } R_g^{\,2} = \frac{L^2}{12} + \frac{R^2}{2}$$

There is also the intermediate region that gives the "cross sectional Rg" from a plot of Ln[QI(Q)] vs Q^2:

$$I(Q) = \frac{I(0)}{Q}\exp\left(-\frac{Q^2 R_g^{\,2}}{2}\right) \quad \text{where } R_g^{\,2} = \frac{R^2}{2}$$

In the case of disk-like or lamellar objects, the intermediate region gives the "thickness Rg":

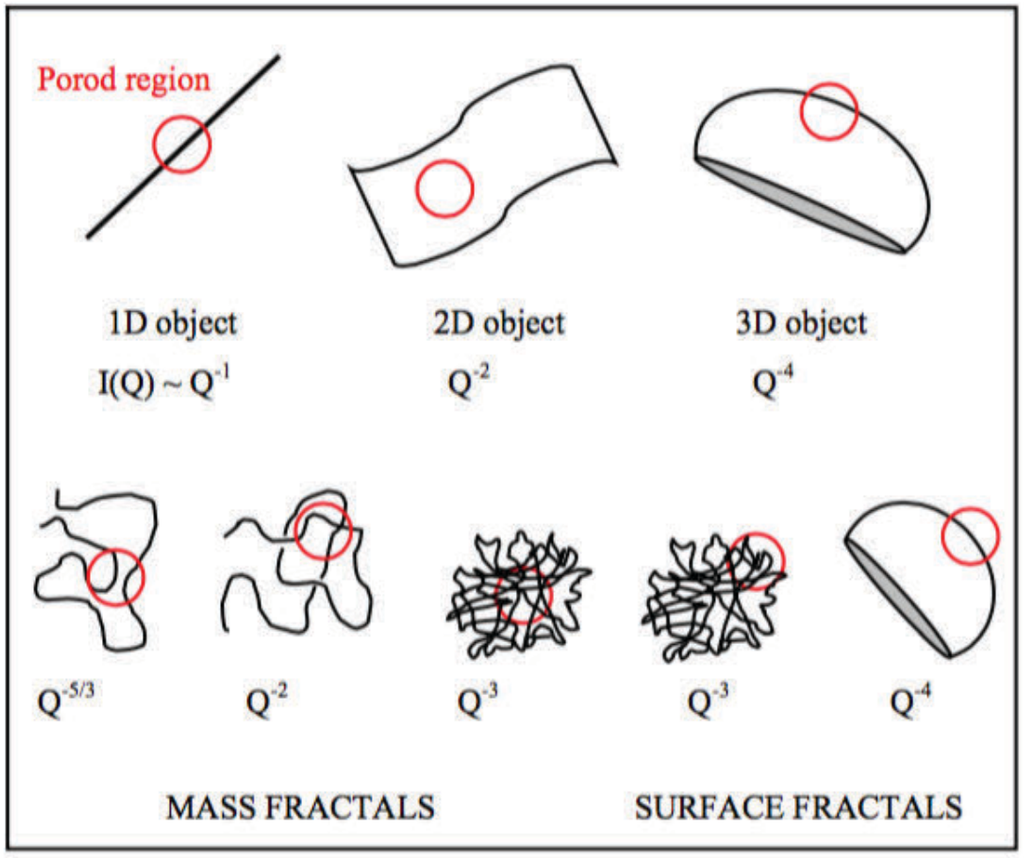
$$I(Q) = \frac{I(0)}{Q^2}\exp\left(-\frac{Q^2 R_g^{\,2}}{1}\right) \quad \text{where } R_g^{\,2} = \frac{T^2}{12}$$



From "The SANS Toolbox" by B. Hammouda

Model Independent

Porod Region and Porod's Law

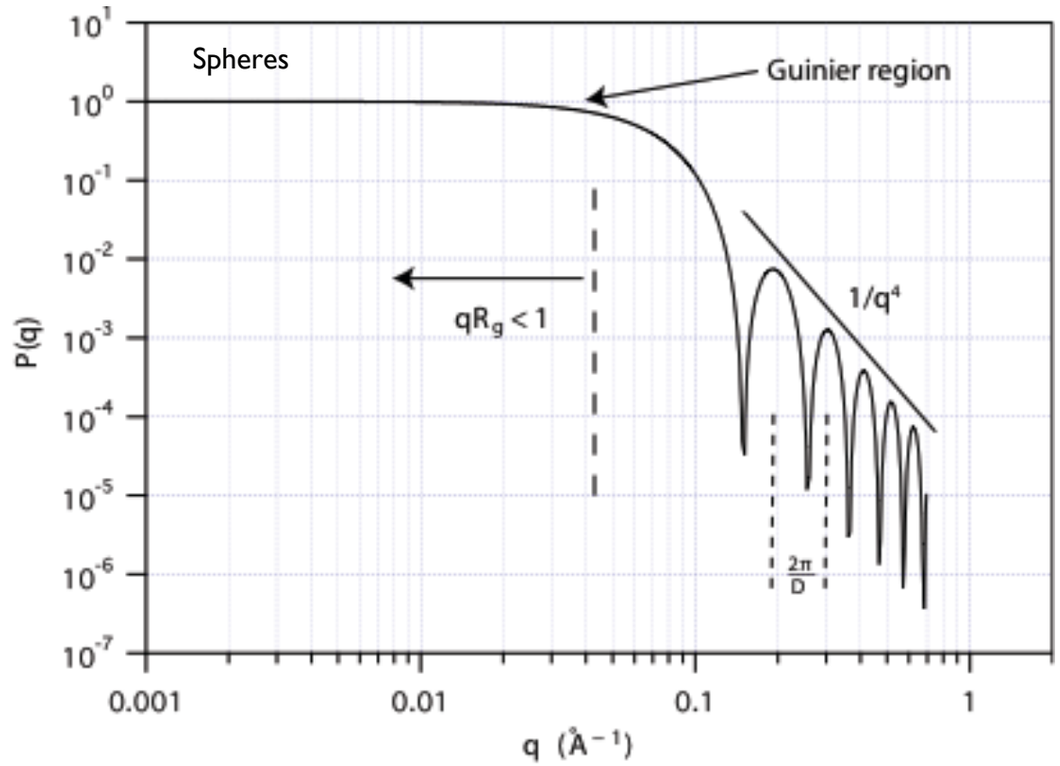


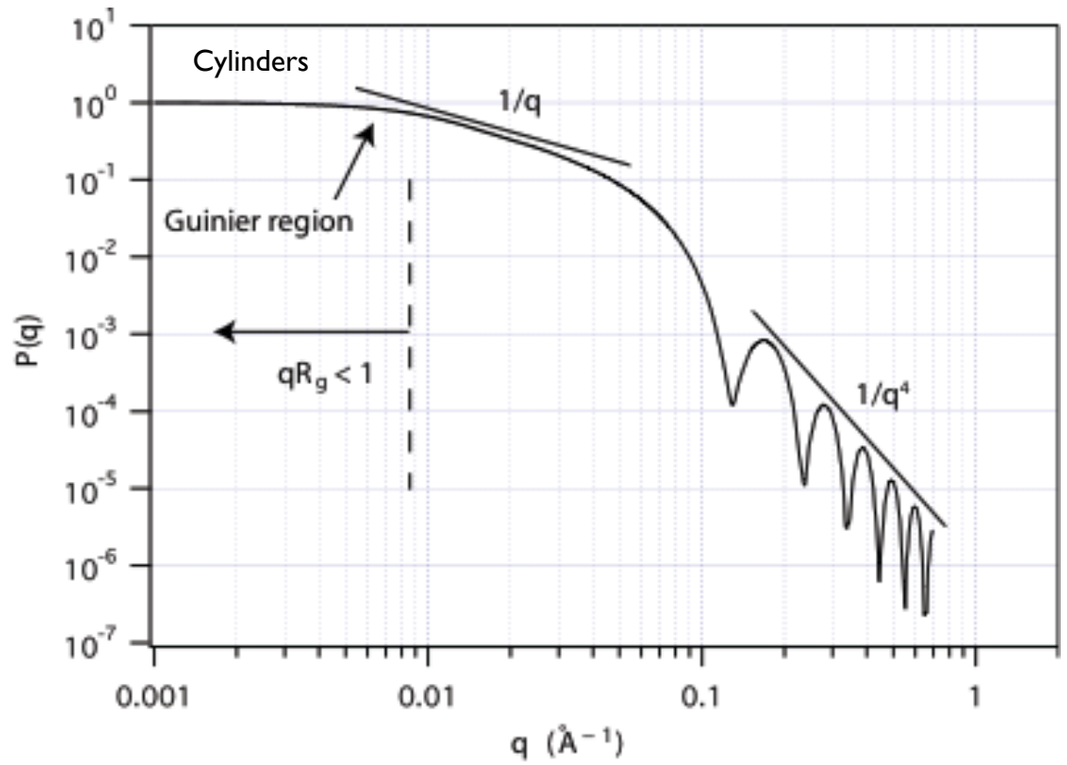
From "The SANS Toolbox" by B. Hammouda

The slope of a log-log plot in a region where the size being examined is smaller than the scattering object is called the "Porod region" and gives information about the local structure.

Porod's Law gives us the surface to volume ratio from the scattering invariant for systems with sharp interfaces:

$$\frac{\pi}{Q^*} \cdot \lim_{q->\infty}(I(q) \cdot q^4) = \frac{S}{V}$$





Form and Structure Factors

Lots of form and structure factors have already been calculated ....

ELSEVIER

Advances in Colloid and Interface Science
70 (1997) 171–210

ADVANCES IN COLLOID AND INTERFACE SCIENCE

Analysis of small-angle scattering data from colloids and polymer solutions: modeling and least-squares fitting[1]

Jan Skov Pedersen

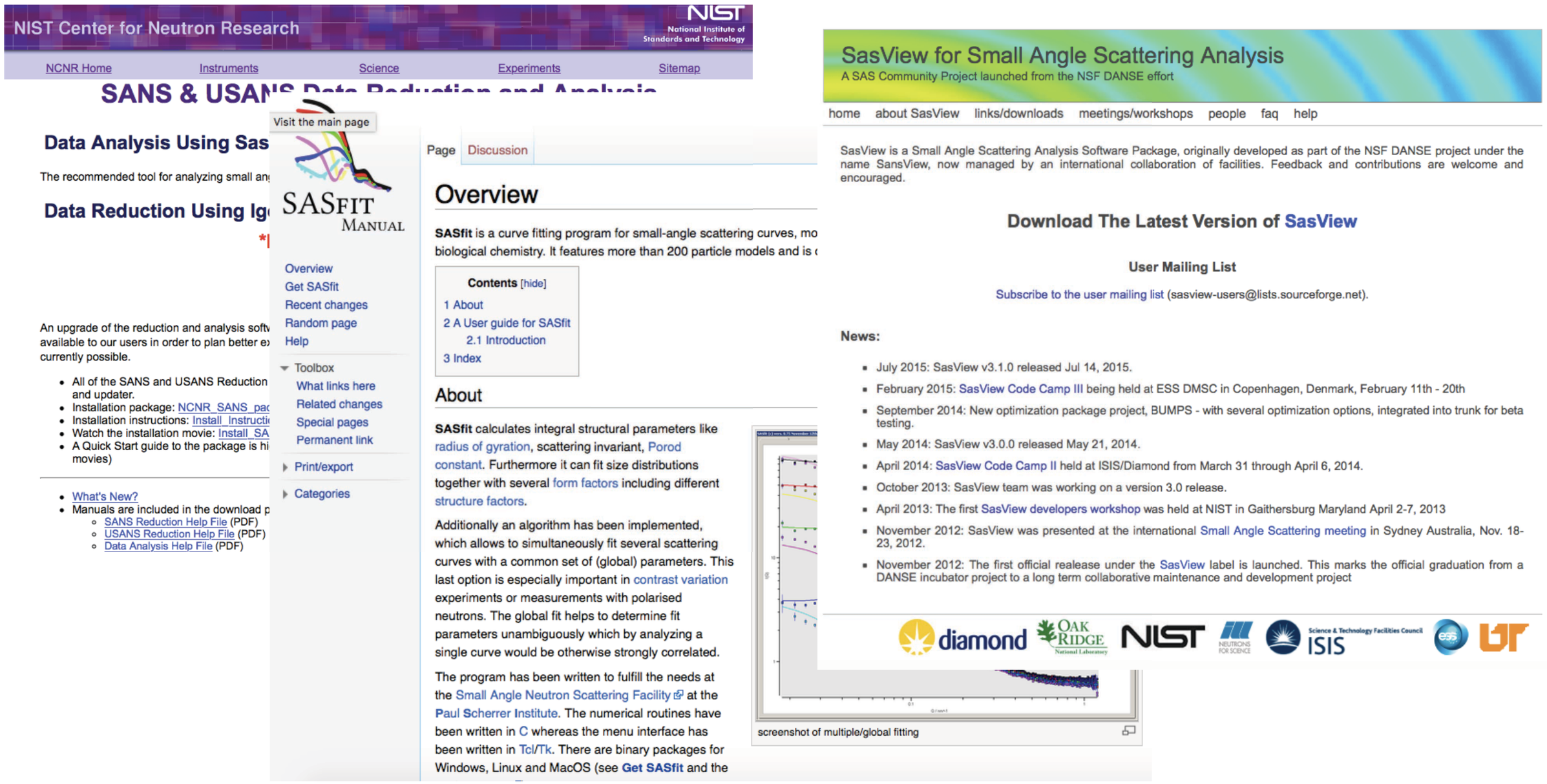
Department of Solid State Physics, Risø National Laboratory, DK-4000 Roskilde, Denmark

Abstract

Analysis and modeling of small-angle scattering data from systems consisting of colloidal particles or polymers in solution are discussed. The analysis requires application of least-squares methods, and the basic principles of linear and non-linear least-squares methods are summarized with emphasis on applications in the analysis of small-angle scattering data. These include indirect Fourier transformation, square-root

Form and Structure Factors

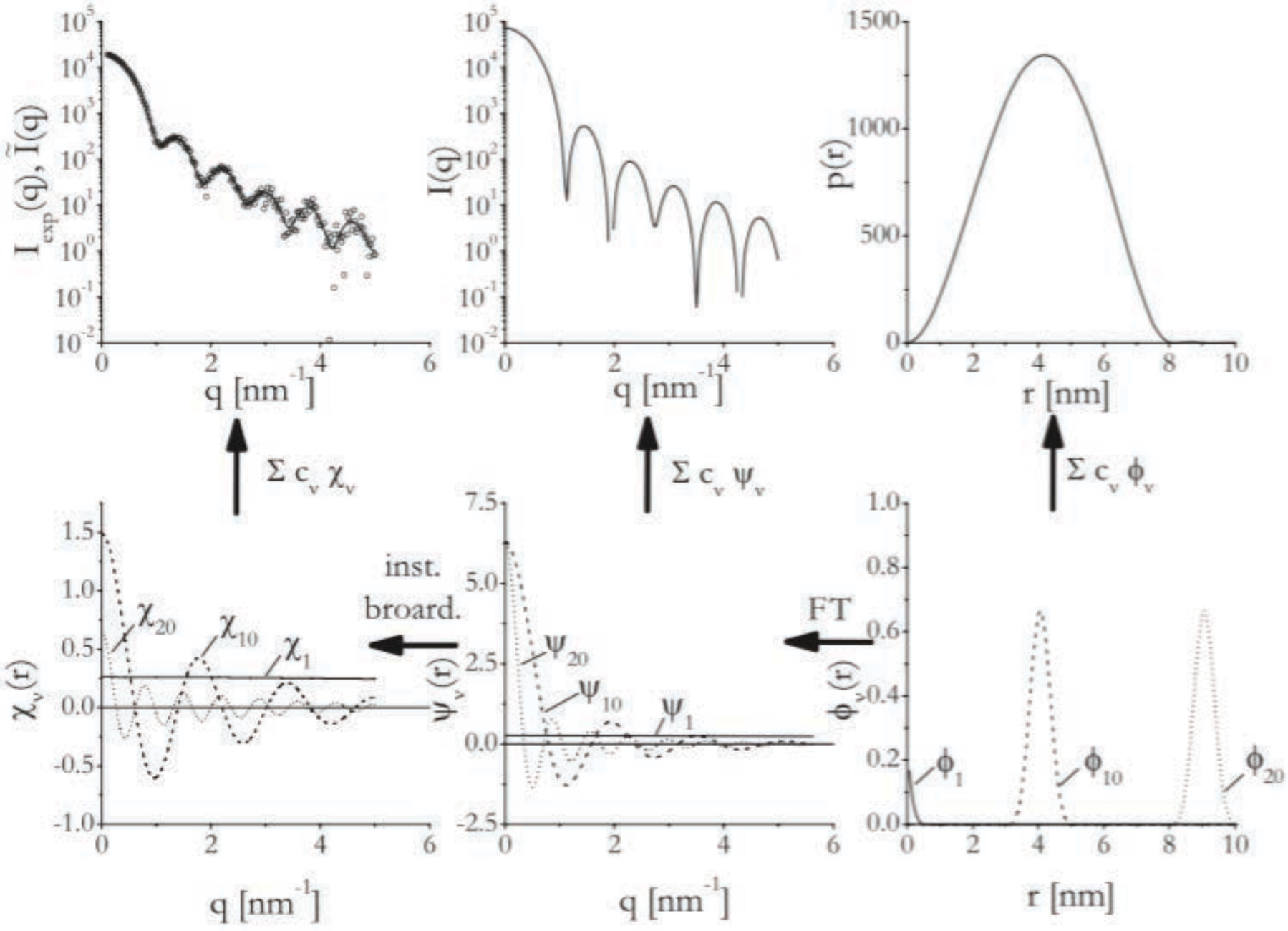
Lots of form and structure factors have already been calculated ....



SasView : http://www.sasview.org
SASFit : https://www.psi.ch/sinq/sansii/sasfit
NIST Igor : http://ncnr.nist.gov/programs/sans/data/red_anal.html

... and coded into software.

Indirect Fourier Transform



Glatter, O. (1977) New Method for Evaluation of Small Angle Scattering Data, *Journal of Applied Crystallography*, *10*, 415–421.

Fritz, G., & Glatter, O. (2006) Structure and interaction in dense colloidal systems: evaluation of scattering data by the generalized indirect Fourier transformation method *New Journal of Physics*, *18*, S2403–S2419

Ab-Initio Structure Generation

Data analysis software ATSAS 2.7.1

A program suite for small-angle scattering data analysis from biological macromolecule

Data processing
PRIMUS - manipulations with experimental 1D SAS data
GNOM - indirect transform program that evaluates the particle distance distribution function p(r)
Data manipulation and analysis tools - AUTORG, ALMERGE, DATGNOM, DATPOROD etc.

***Ab initio* methods**
DAMMIN - *ab initio* shape determination using a dummy atom model
DAMMIF - rapid shape determination
GASBOR - reconstruction of a protein structure by a chain-like ensemble of dummy residues
MONSA - shape determination using a multiphase dummy atom model

Rigid body modelling
SASREF - modelling of multisubunit complexes
BUNCH - modelling of multidomain proteins against multiple data sets
CORAL - modelling of multidomain protein complexes against multiple data sets
MASSHA - interactive modelling of atomic structures and shape analysis
GLOBSYMM - rigid body modelling of symmetric oligomers

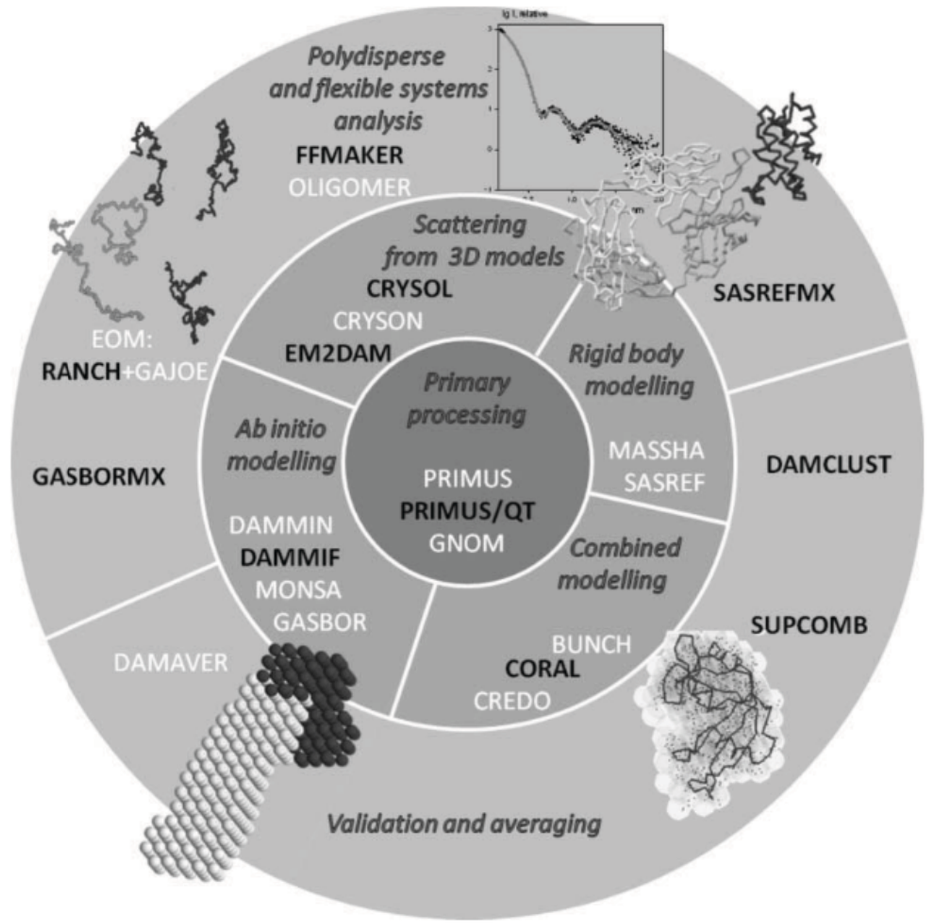
Mixtures and flexible systems
OLIGOMER - volume fractions of mixtures with known scattering intensities from the components
MIXTURE - modelling of multicomponent systems
EOM - Ensemble Optimization Method for flexible proteins
SREFLEX - flexible refinement of high-resolution models combining SAXS and NMA

PDB oriented tools
CRYSOL - X-ray scattering patterns from known hi-res structures
CRYSON - neutron scattering patterns from known hi-res structures
SUPCOMB - superimposes one 3D structure onto another
DAMAVER - align *ab initio* models, select the most typical one

Manuals

If you use ATSAS please cite:

Petoukhov, M.V., Franke, D., Shkumatov, A.V., Tria, G., Kikhney, A.G., Gajda, M., Gorba, C., Mertens, H.D.T., Konarev, P.V. and Svergun, D.I. (2012)
New developments in the ATSAS program package for small-angle scattering data analysis
J. Appl. Cryst. 45, 342-350 © International Union of Crystallography DOI



http://www.embl-hamburg.de/biosaxs/software.html

Philosophy of Data Analysis

1) Look at the data …
   - Trends
   - Shape
     ... do they match your expectation?
     ... if not, why not?

2) Extract model free information ...
   - Rg (Guinier)
   - Slopes (Porod)
   - I(0)

3) Consider model fitting ...
   - Which model?
   - If model doesn't fit, don't just add more parameters!
   - How much information is in my data?

4) Consider Indirect Fourier Transform ...
   - Can give hints as to why model isn't fitting
   - If possible use deconvolution to SLD profile
   - Use as a basis for devloping parameterised model fit

5) Consider Ab Initio methods ...
   - Do you have a known structure to compare to?

Applications of SANS

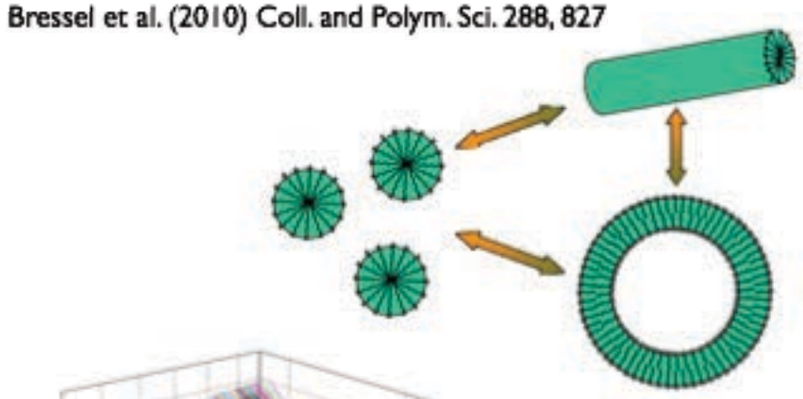
Andrew Jackson

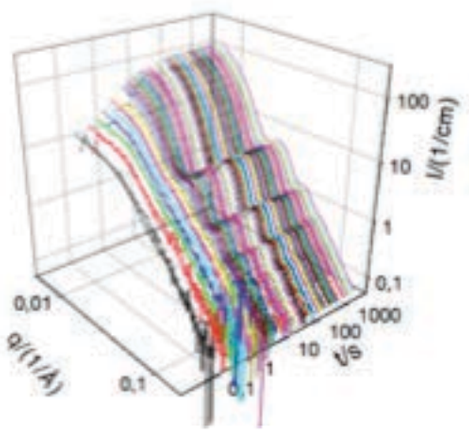
NNSP-SwedNess Neutron School 2017, Tartu

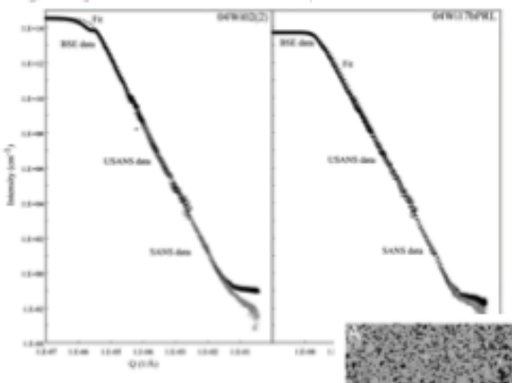
Lecture L9.3

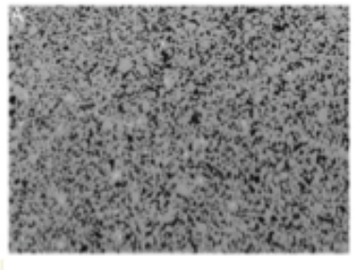
SANS is Versatile

Bressel et al. (2010) Coll. and Polym. Sci. 288, 827

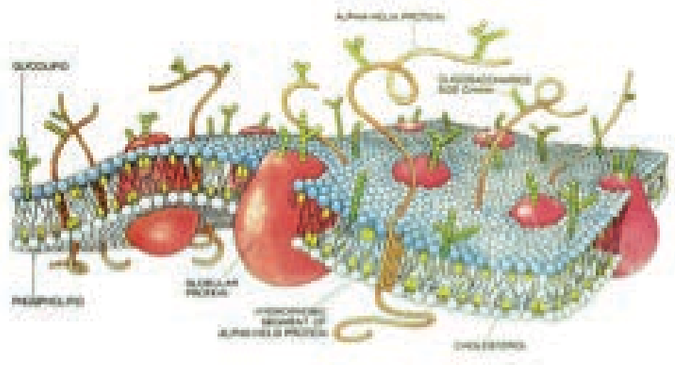


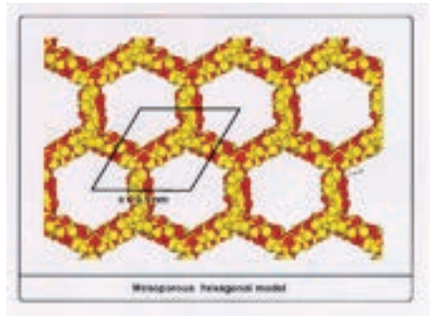




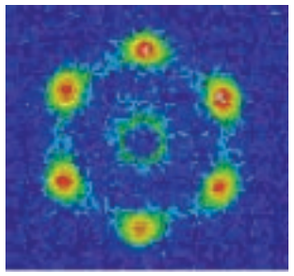


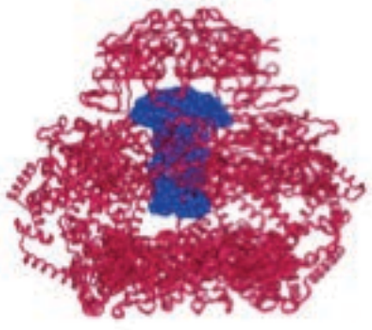
Anovitz et al. (2012) In Press.

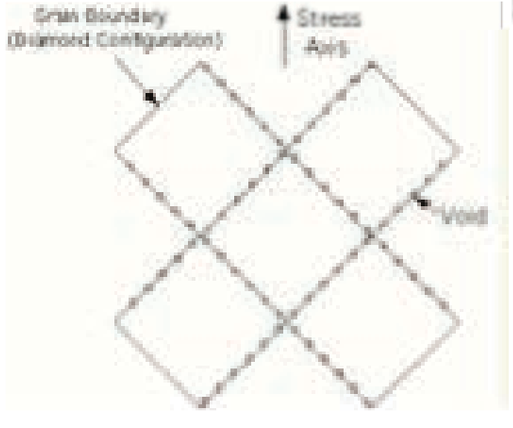


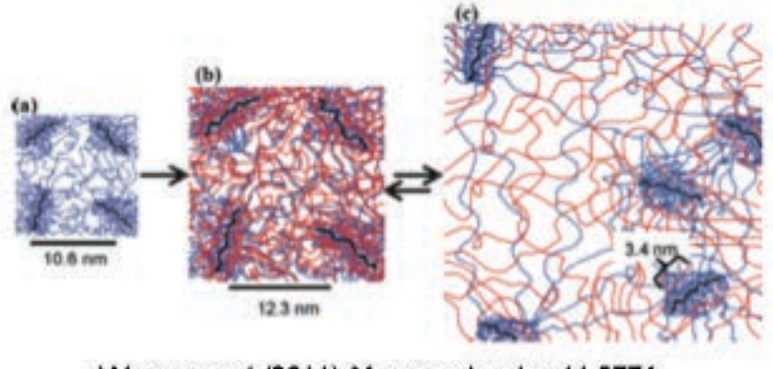


Mesoporous structures
Biological structures (membranes, vesicles, proteins in solution)
Polymers
Colloids and surfactants
Magnetic films and nanoparticles
Superconductors
Voids and Precipitates
Geology

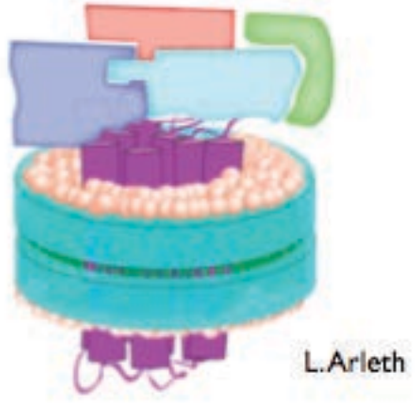








Waters et. al (2011) Macromolecules 44 5776



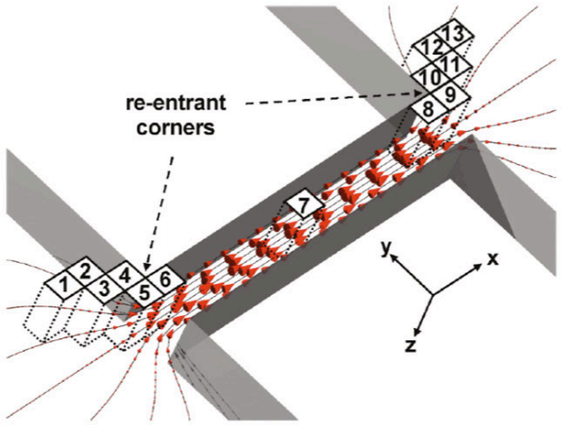
L.Arleth

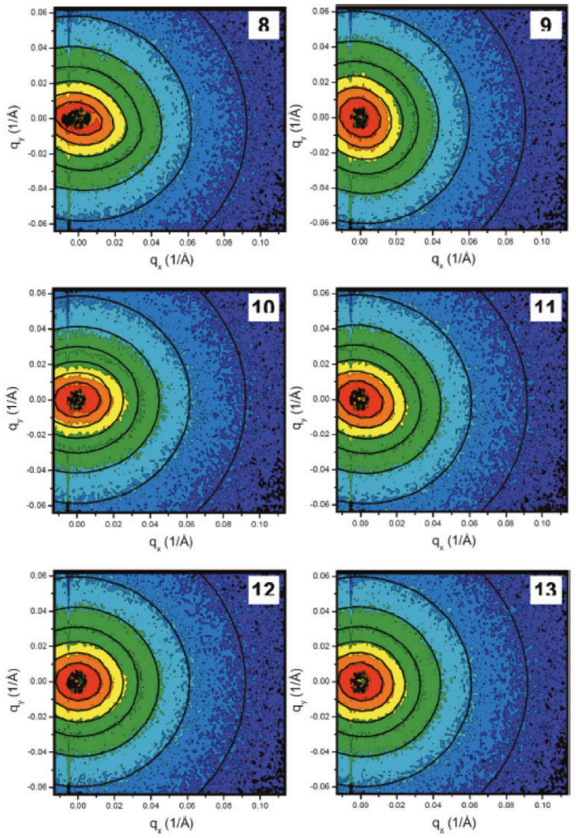
Fluid Flow

The flow of **complex fluids** through **complex geometries** is relevant to many industrical processes including polymer processing and oil recovery.

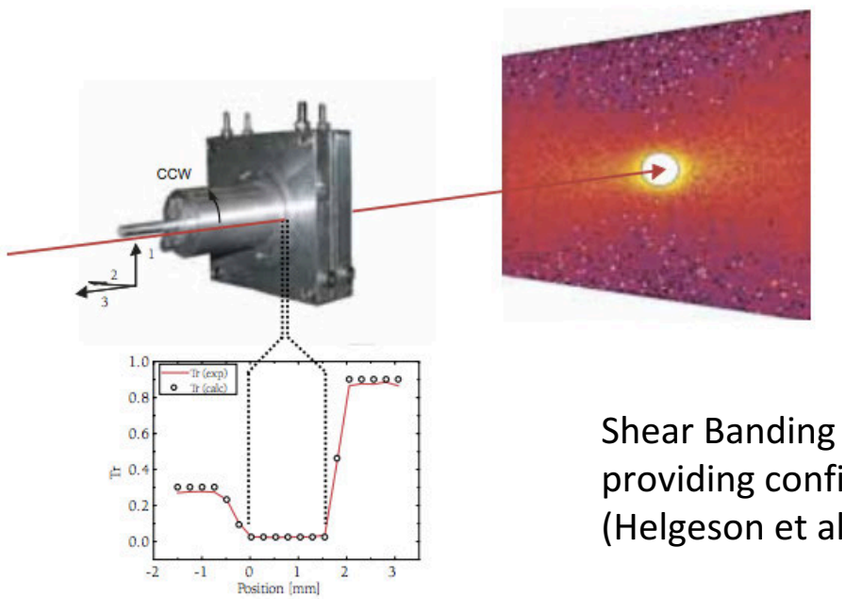
Microfluidic devices are increasingly used for chemical and pharmaceutical discovery, production and processing.

There is a need to understand **structural effects of flow** both for practical purposes and to compare with fluid flow models.

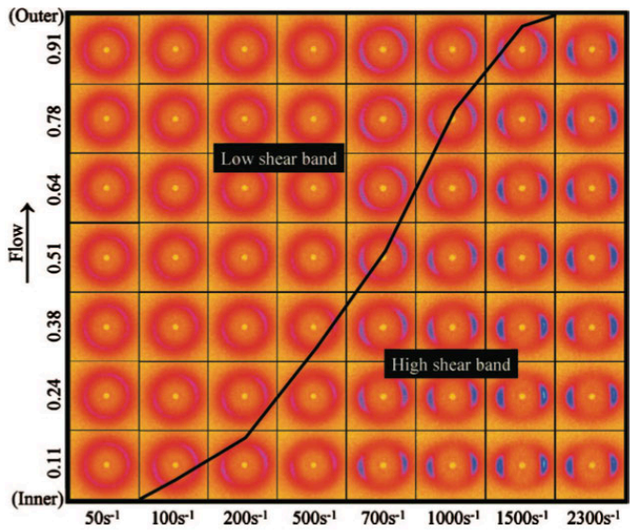




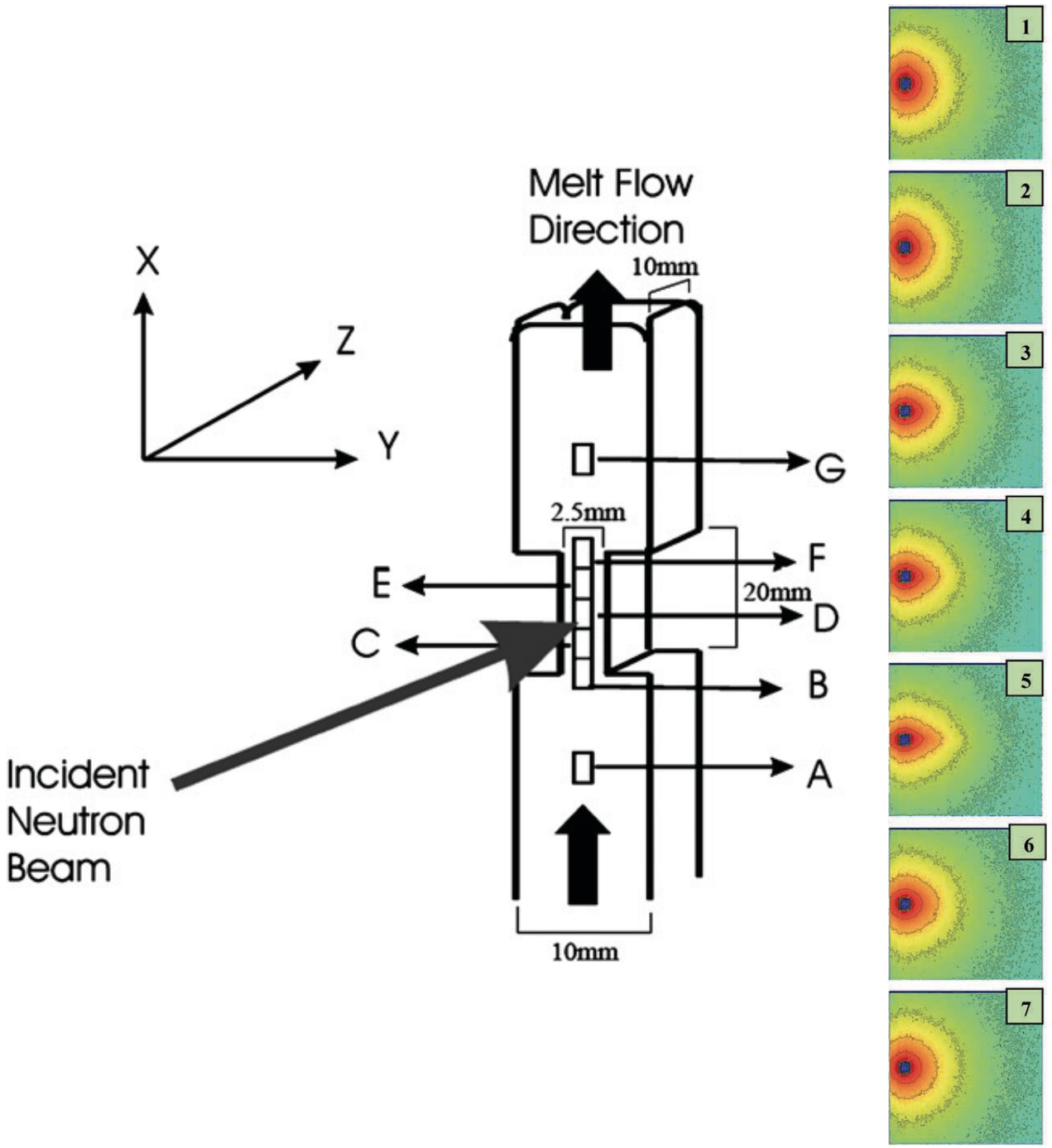
Measuring the deformation of polymer chains allowing development of new models of polymer flow (Clarke et al. (2010) Macromolecules 43, 1539)

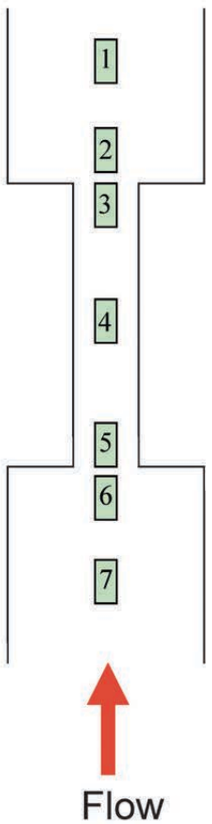


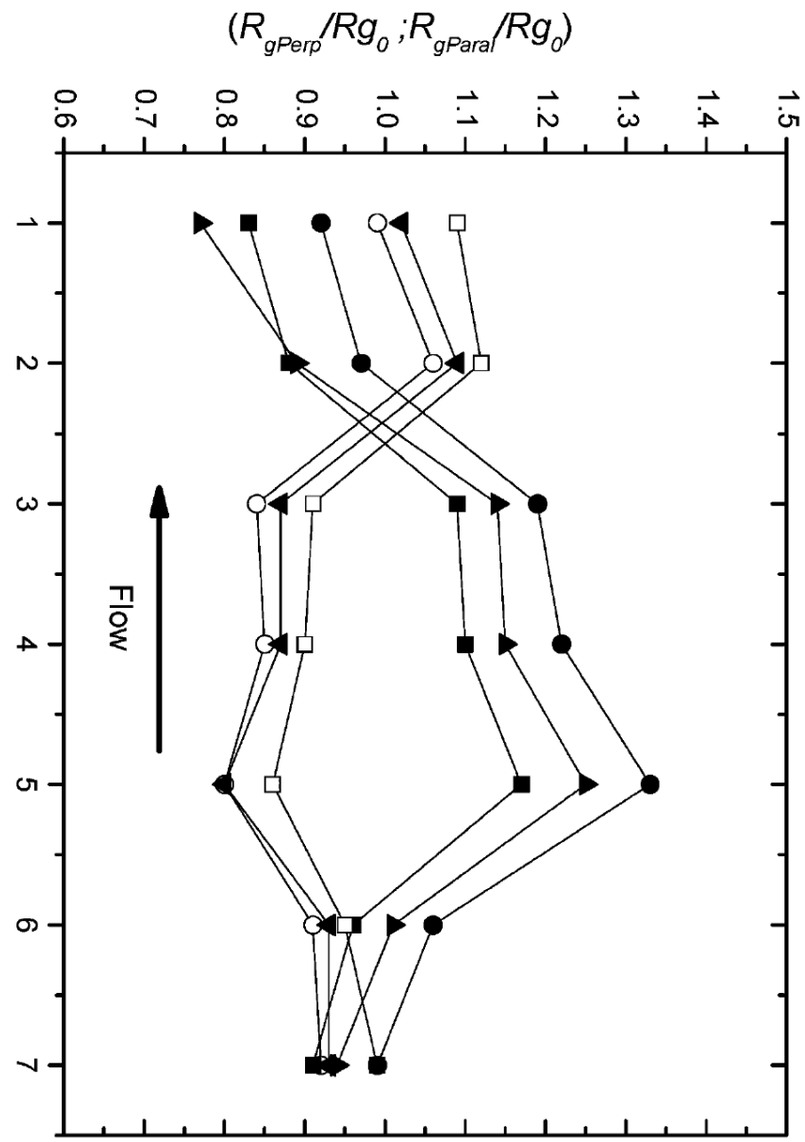
Shear Banding in CTAB wormlike micelles providing confirmation of rheological model. (Helgeson et al. (2009) J. Rheol 53, 727)



Flow mapping in polymer melts

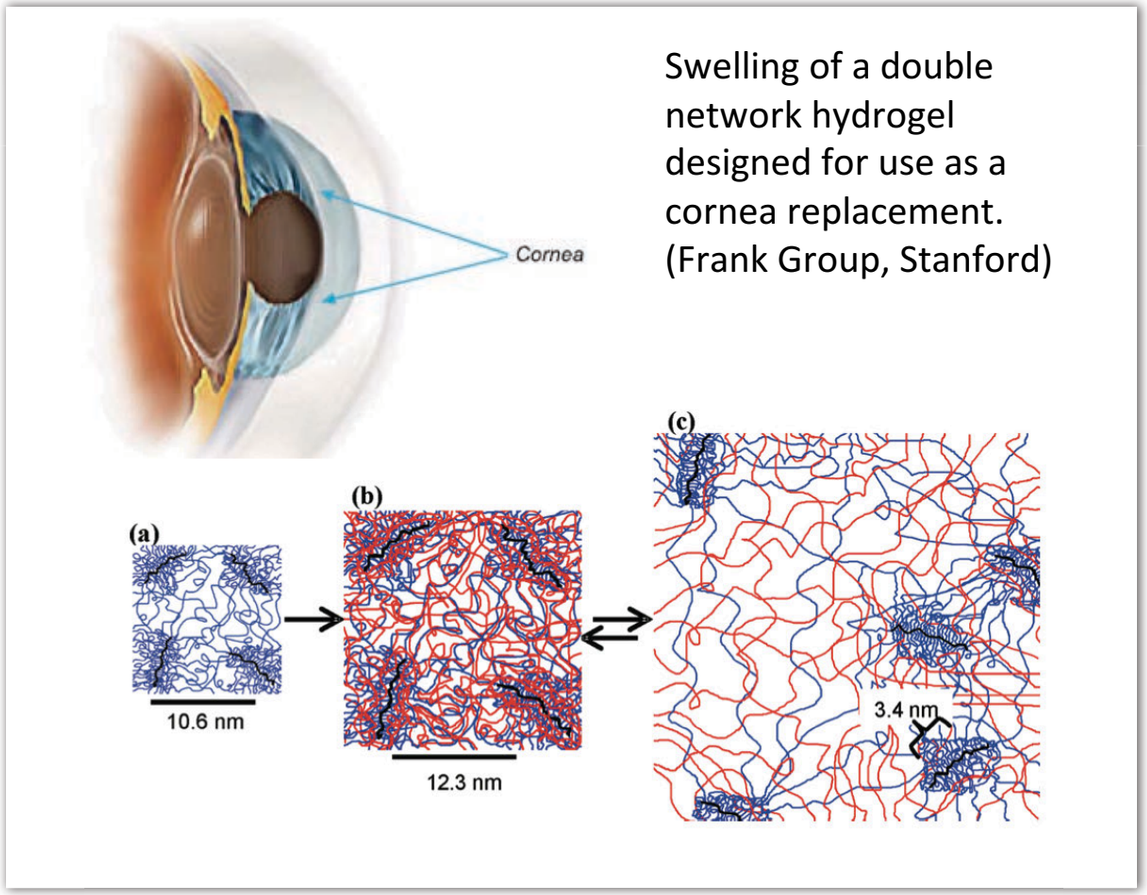




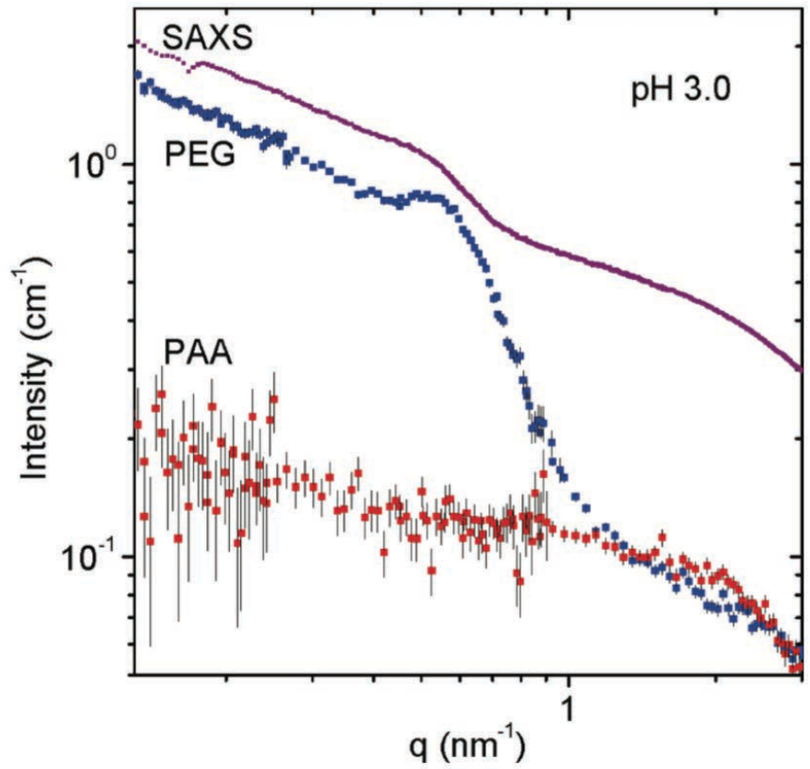


McLeish et al., *Soft Matter*, 2009, **5**, 4426

Double Network Hydrogels

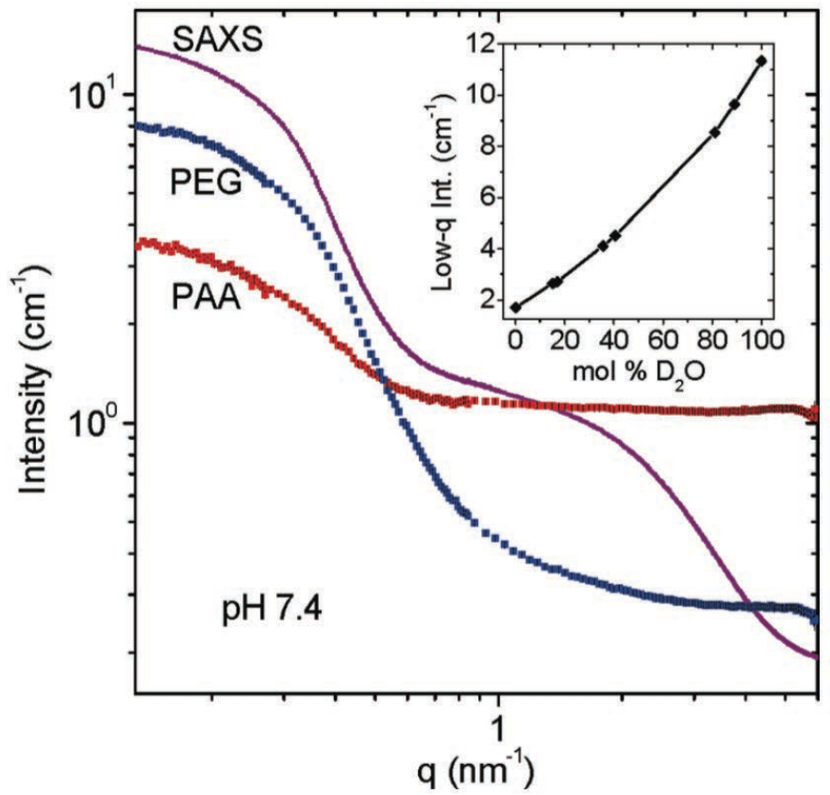


Swelling of a double network hydrogel designed for use as a cornea replacement. (Frank Group, Stanford)



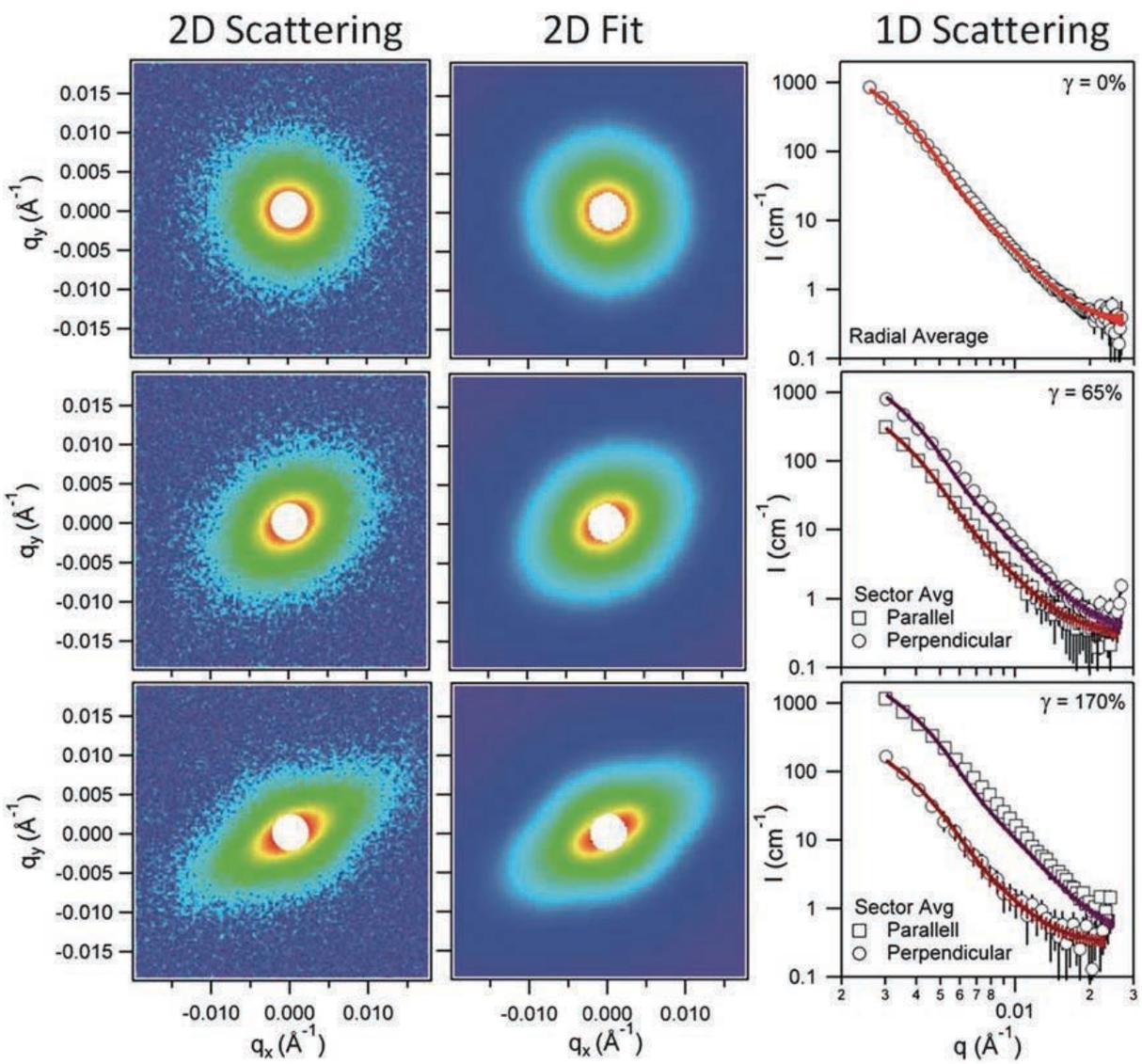
Double network hydrogels provide strength and resilience together with high water content.

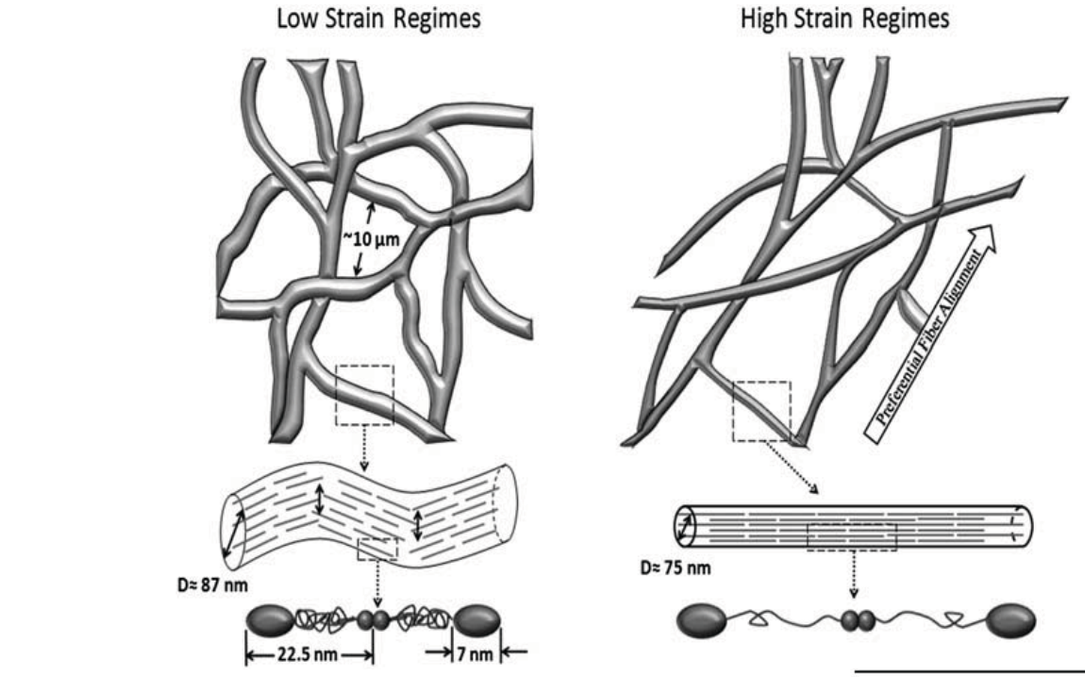
Neutrons provide the structure of each component in the presence of the other. But contrast matching can lead to weak scattering signals and incoherent scattering can be a problem.

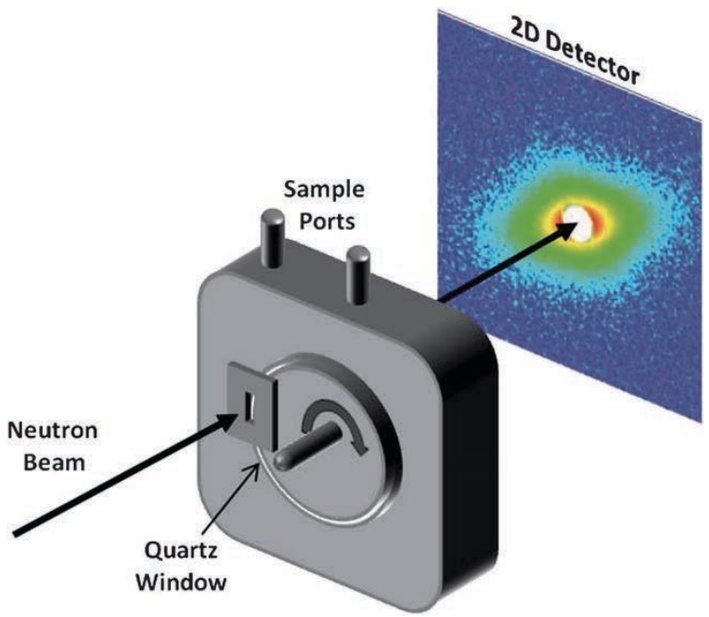


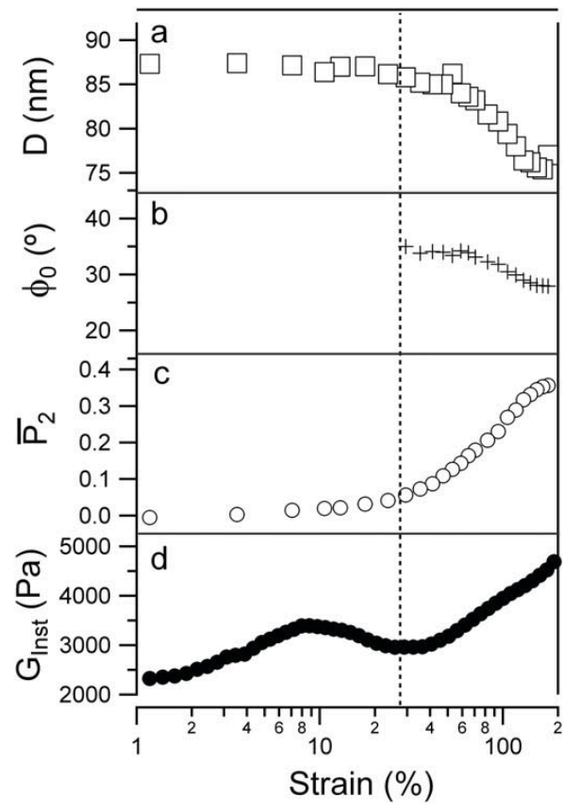
Waters *et al* (2011) Structure and Mechanism of Strength Enhancement in Interpenetrating Polymer Network Hydrogels. *Macromolecules*, *44*(14), 5776–5787. http://doi.org/10.1021/ma200693e

Structure of gel networks under shear



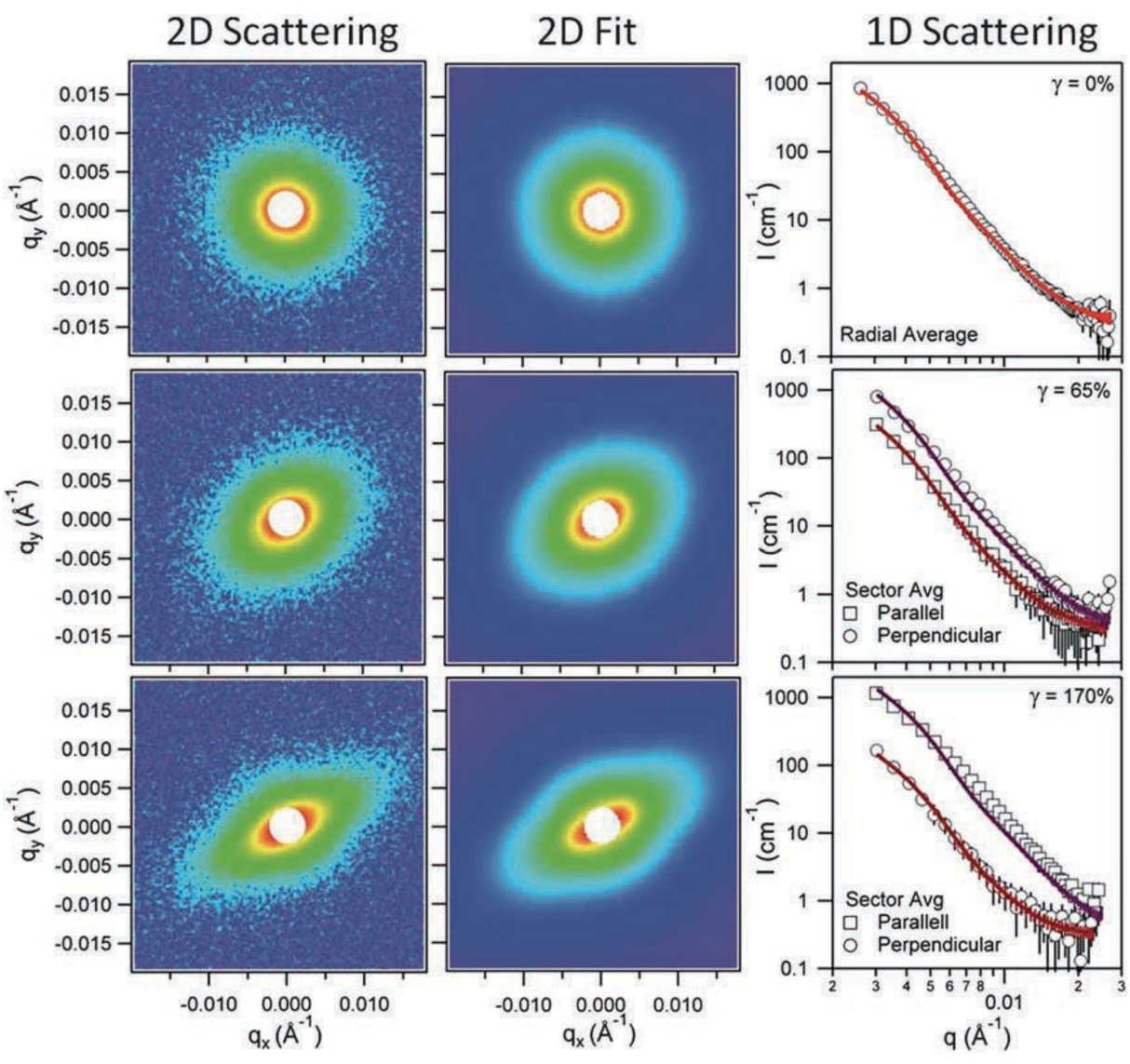


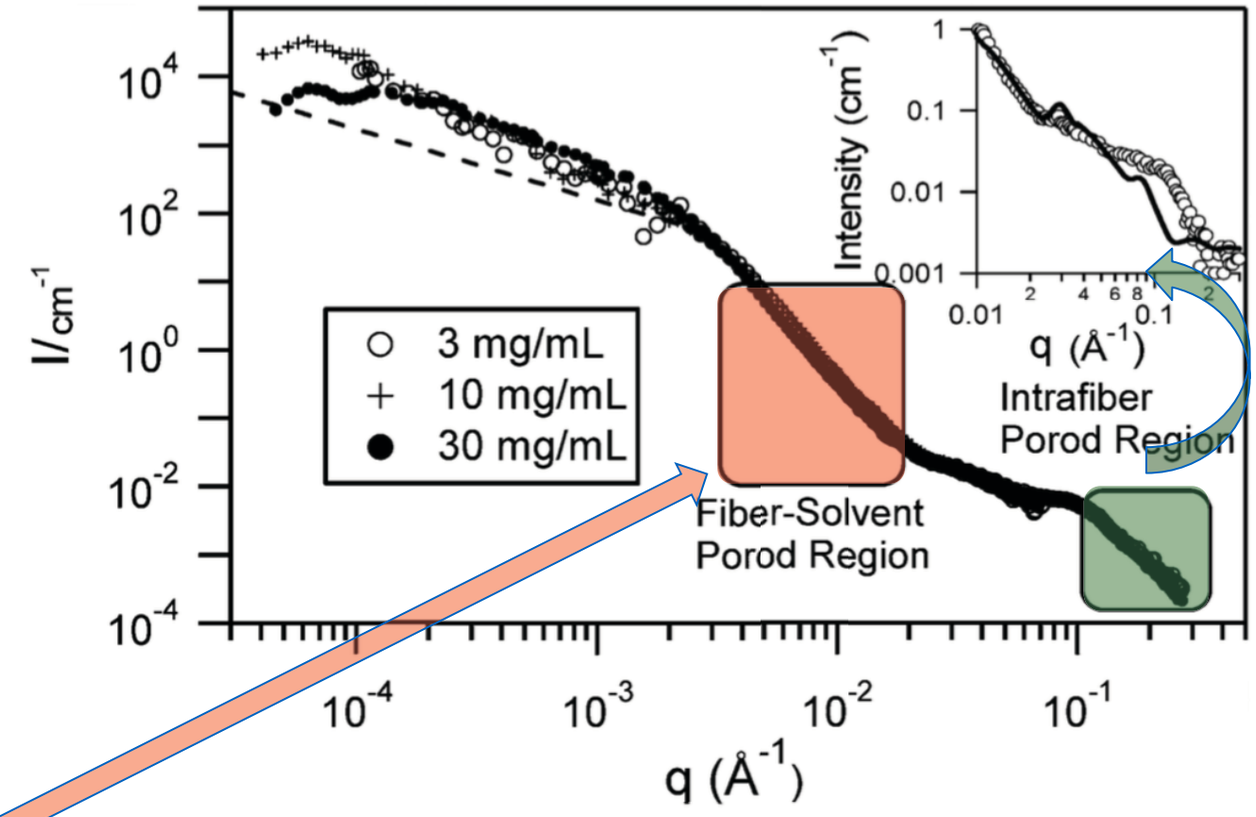


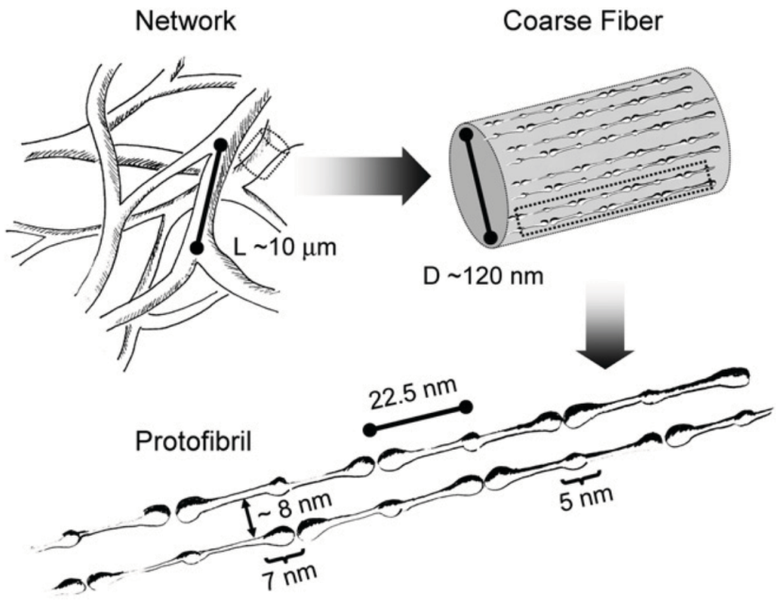


Weigandt, Porcar and Pozzo, *Soft Matter*, 2011, **7**, 9992

Structure of gel networks under shear







Weigandt, Porcar and Pozzo, *Soft Matter*, 2011, **7**, 9992
Weigandt, Pozzo and Porcar, *Soft Matter*, 2009, **5**, 4321

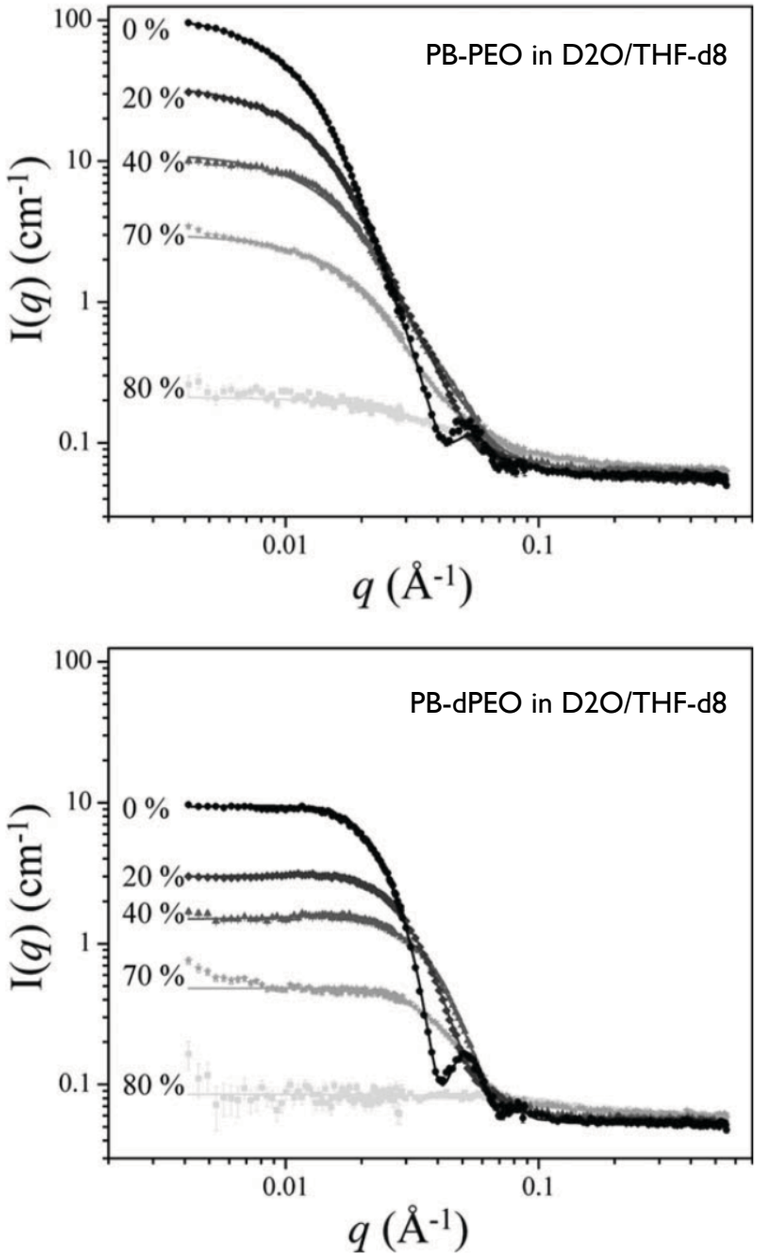
Block Copolymers

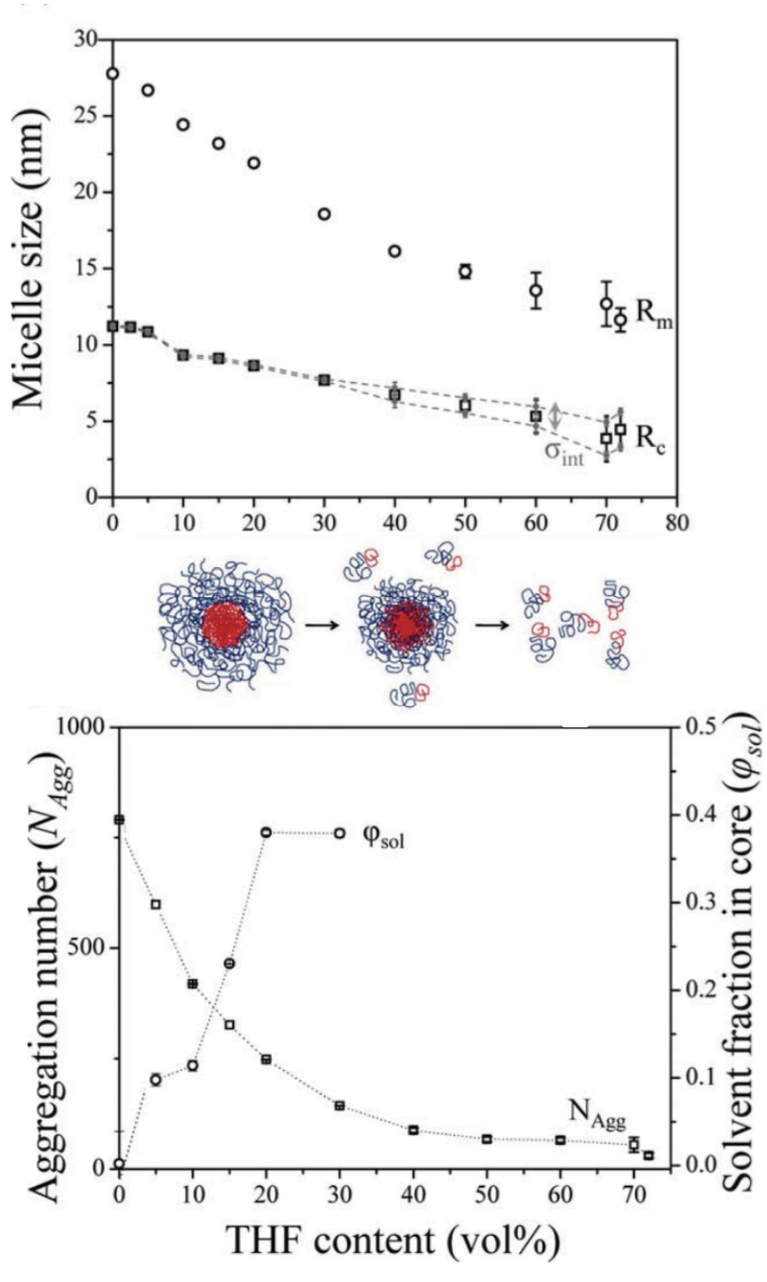
Amphiphilic block copolymer assemblies are good candidates for targeted drug delivery applications due to their synthetic versatility, stability, and ability to encapsulate therapeutic molecules

Solvent interactions can be used to tune the structure of micelles formed by block copolymers – here poly(1,2- butadiene-b-ethylene oxide) [PB-PEO].

Contrast variation (solvent and polymer) SANS revealed the structural variation seen as the water-THF ratio was varied.

This control of structure allows for micelles to be designed to hold specific drugs through functionalization of the butadiene block.





Kelley *et al* (2011). Structural changes in block copolymer micelles induced by cosolvent mixtures. *Soft Matter*, *7*(15), 7094–7102. http://doi.org/10.1039/c1sm05506b

Stopped-flow SANS - in-situ vesicle formation

Disc to vesicle transition: 50-100ms shots repeated 10-25 times (1ms mixing time)

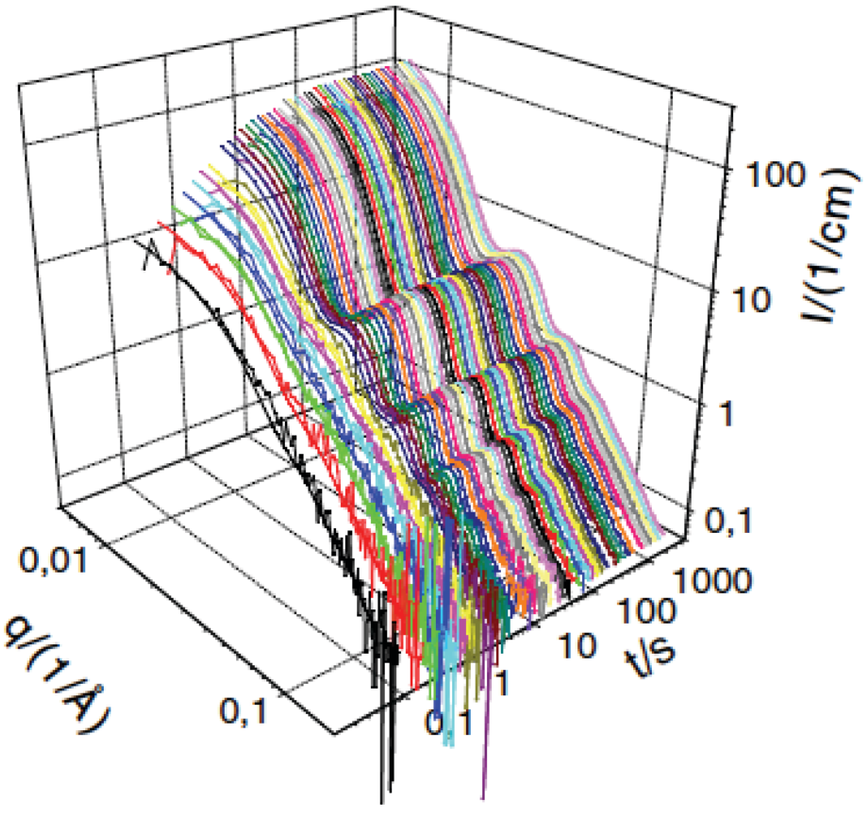
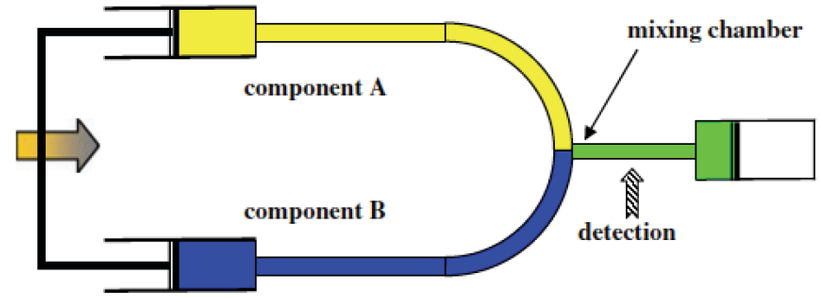


Fig. 6 Time-resolved SANS spectra for the case of mixing 50 mM TDMAO with 50 mM LiPFOS to yield a final composition with x(TDMAO)=0.525 mixing ratio at 25 °C (fits are included as *solid lines*)



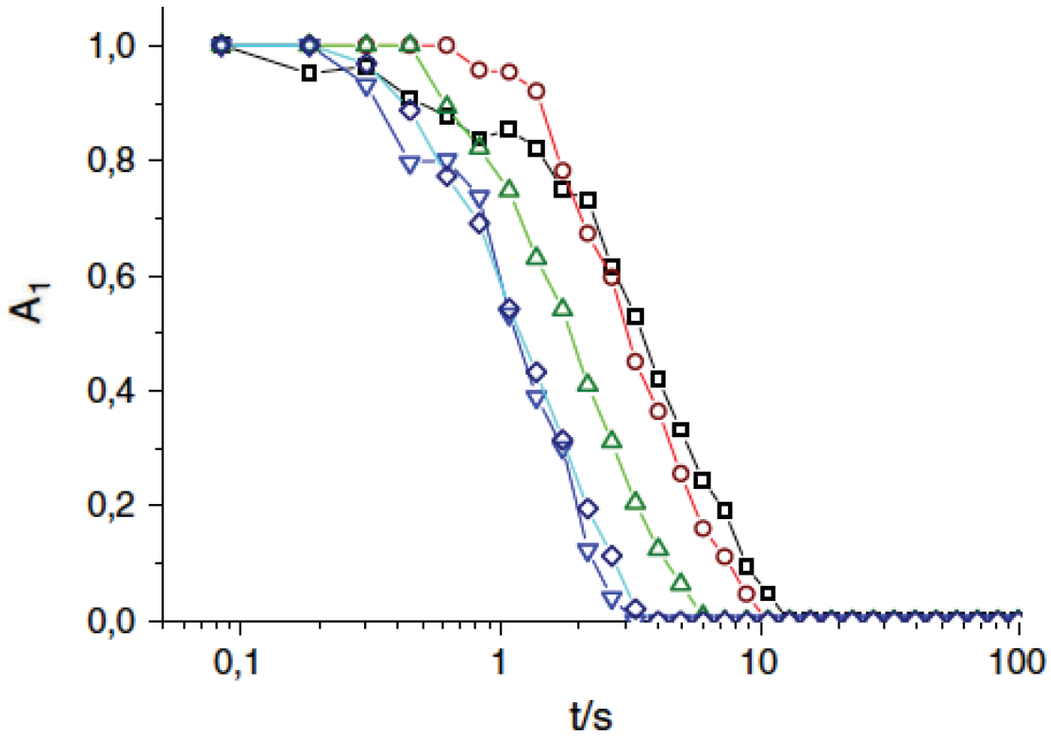
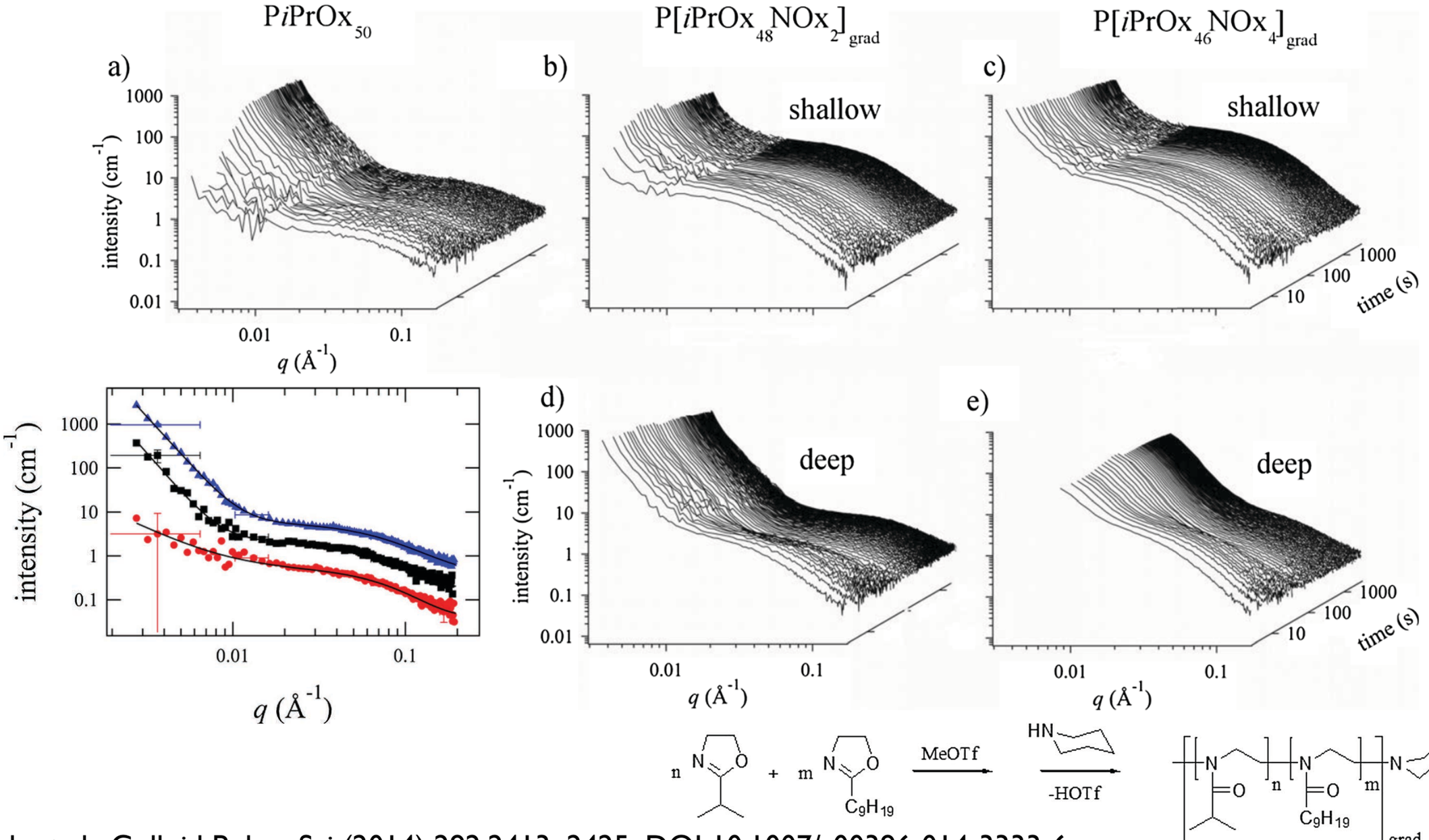


Fig. 9 The relative content A_1 of disks as a function of time in the mixtures with vesicles for various mixing ratios of TDMAO/LiPFOS at 25 °C; x(TDMAO) in the mixtures was *open square*, 0.5; *open circle*, 0.525; *open up-pointing triangle*, 0.55; *open down-pointing triangle*, 0.6; *open diamond*, 0.65

Bressel et al., Colloid Polym Sci (2010) 288:827–840 DOI 10.1007/s00396-010-2212-z

T-jump TR-SANS on poly(2-oxazoline) gradient copolymers

Sample injection into pre-heated cuvette.



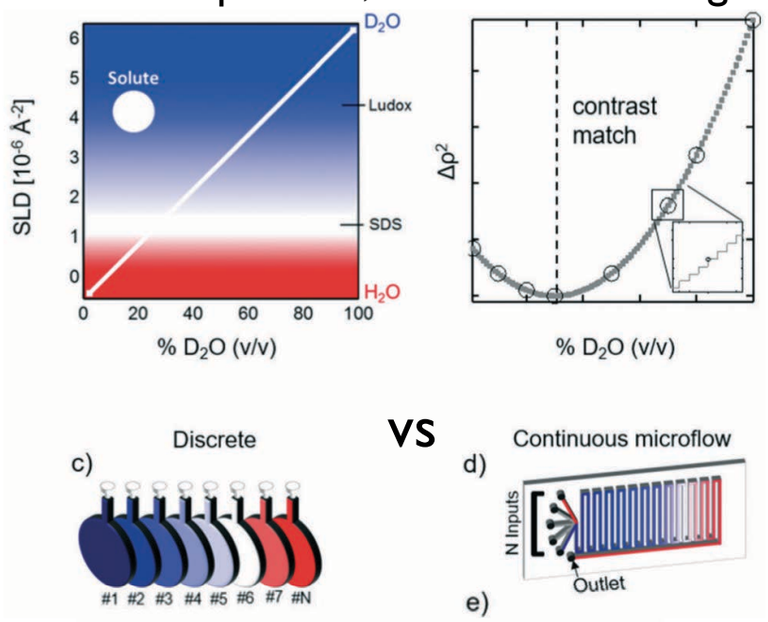
Jaksch et al., Colloid Polym Sci (2014) 292:2413–2425, DOI 10.1007/s00396-014-3333-6

Microfluidic SANS

High Throughput Mixing & Tailored Flow Geometry

400 um channels, 12 mm beam avg. over 20 channels

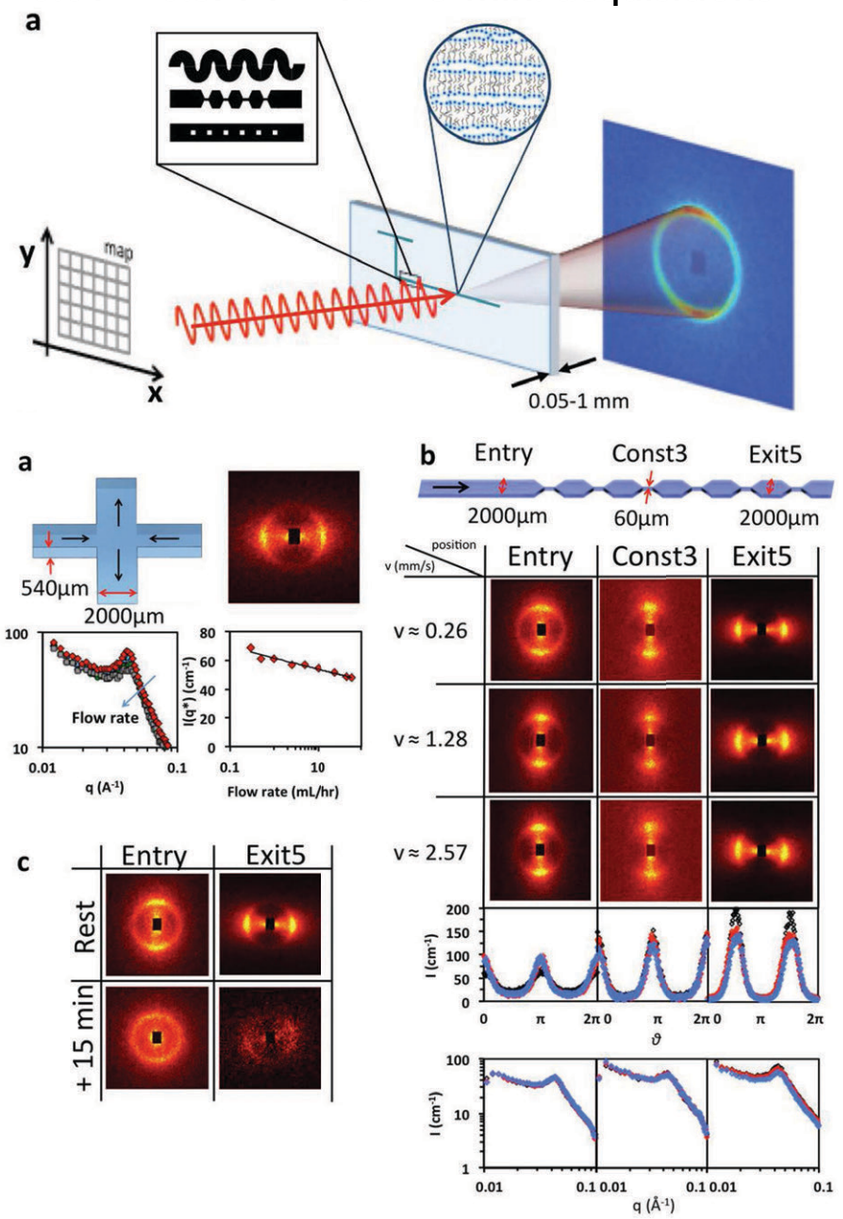
5s acquisition, continuous mixing



Adamo, M., Poulos, A. S., Miller, R. M., Lopez, C. G., Martel, A., Porcar, L., & Cabral, J. T. (2017). *Lab Chip*, *17*(9), 1559–1569.

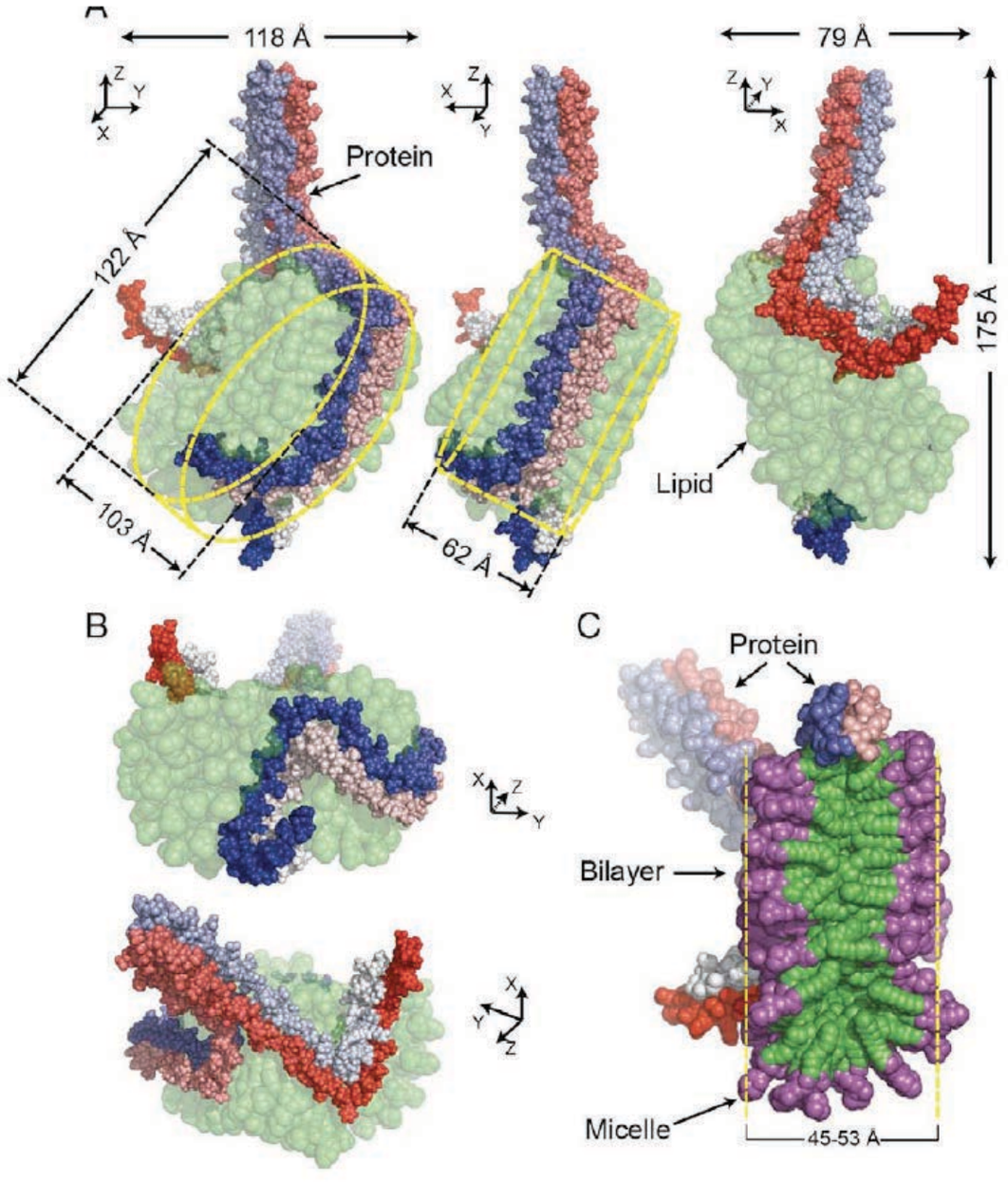
60um channels, 500um beam

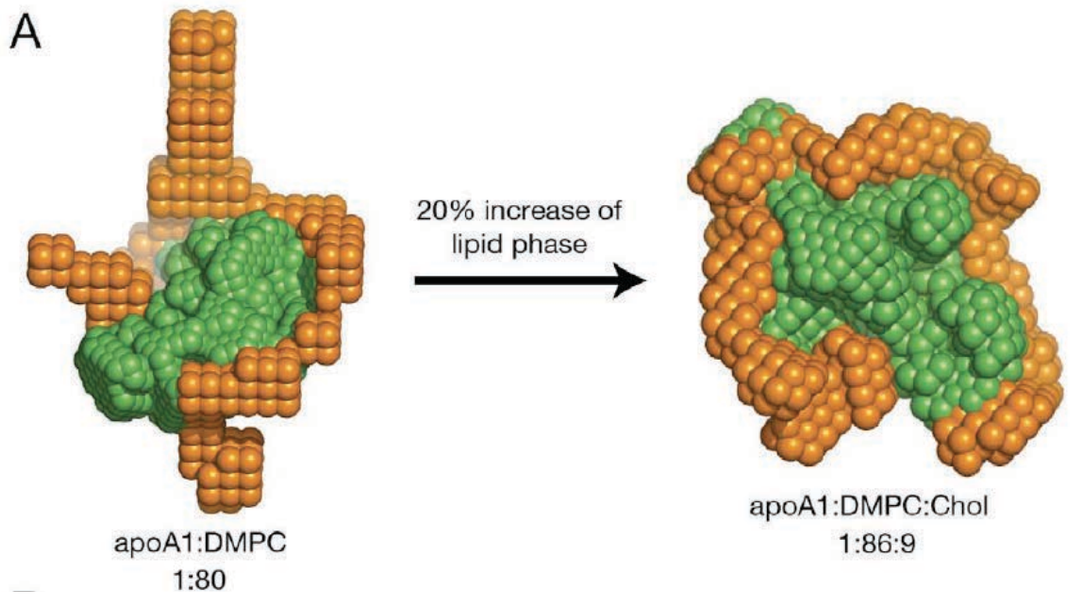
0.01 – 0.3Å-1 – 1s - 5 min. acquisitions



C.G. Lopez, T. Watanabe, A. Martel, L. Porcar, J.T. Cabral, Scientific Reports, 5 (2015) 7727.

Small Angle Neutron Scattering of High Density Lipoprotein in solution



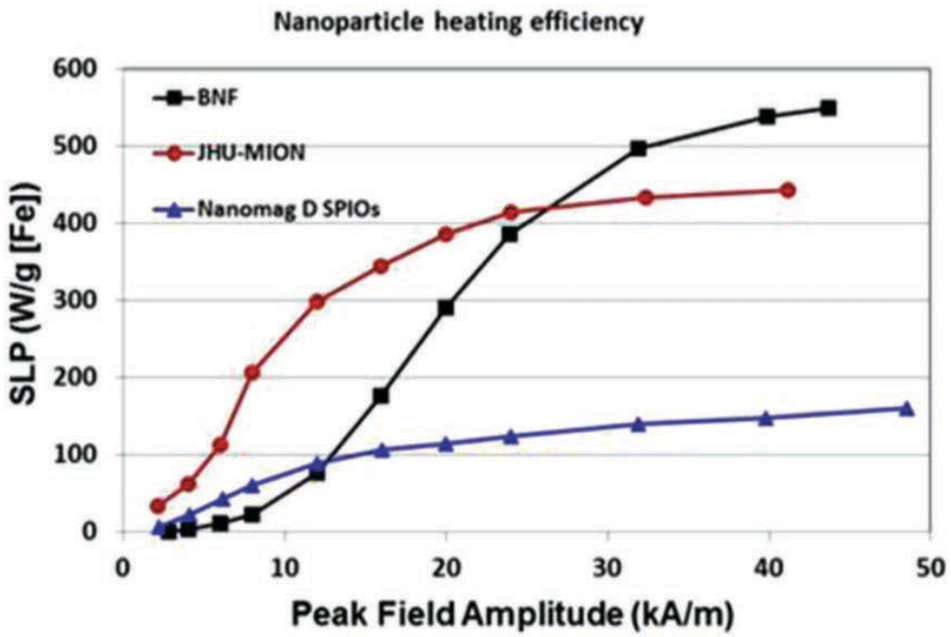


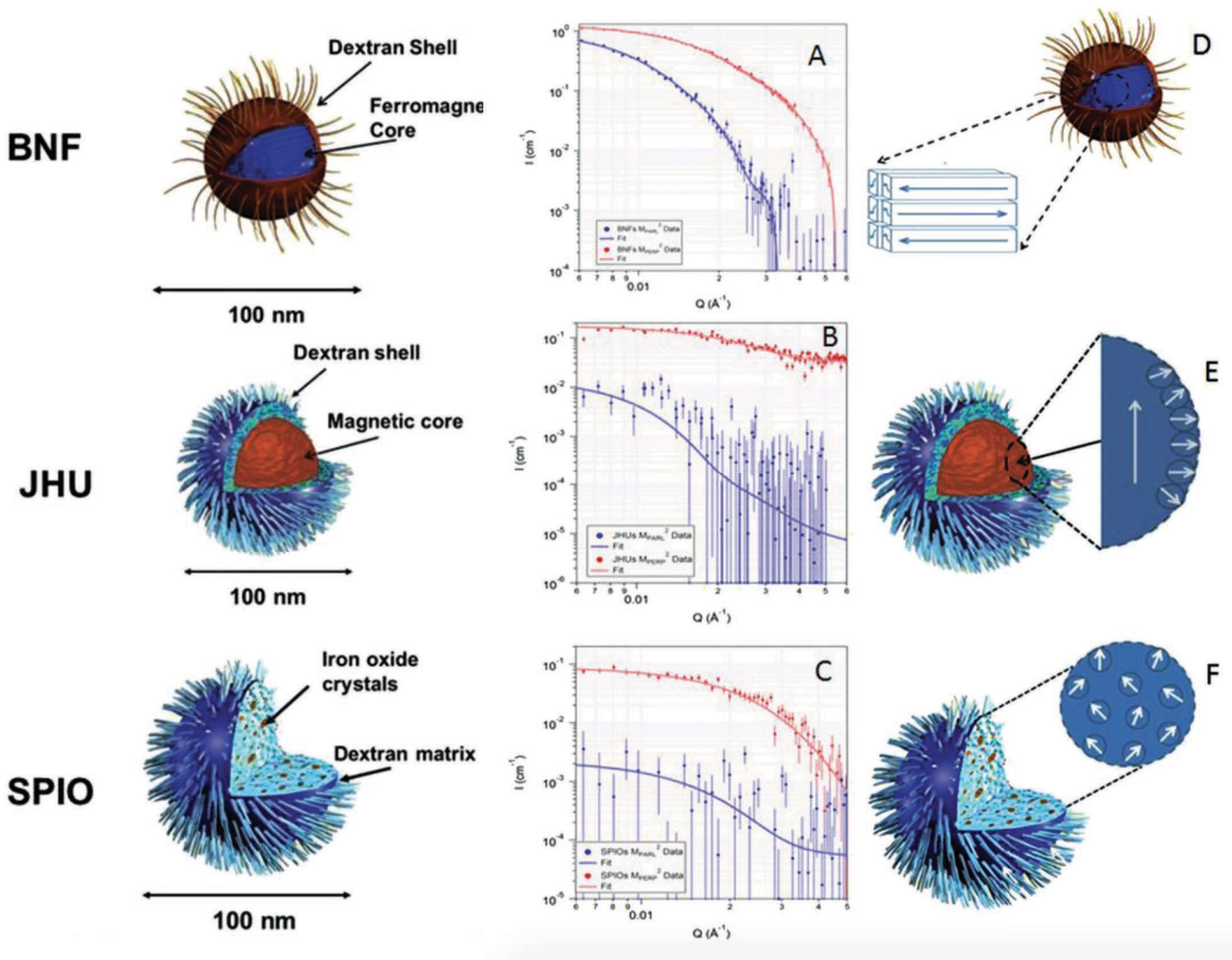
The low resolution structure of nascent high density lipoprotein with and without cholesterol reveals a mechanism for particle expansion

V. Gogonea et al. Journal of Lipid Research, (2013).

Hyperthermia Treatment of Cancer

- Use magnetic nanoparticles with alternating magnetic field to locally heat tumour
- Structure of the nanoparticles has strong impact on heating response
- Use polarized neutron scattering to understand these differences





Dennis et al. *Advanced Functional Materials*, (2015), **25**, 4300

Summary

SANS is a versatile method for studying structure on the nano- to micro- scale

A wide range of scientific areas can be studied

The use of contrast variation methods is key to maximising the information from SANS

Sample environment equipment for SANS is varied and may need to be designed or modified for your experiment

Questions?