



UNIVERSITY OF  
BATH

# Small Angle Scattering (SAS)

- what is SAS & what can it measure?
- how is it measured?
- sample considerations
- data analysis

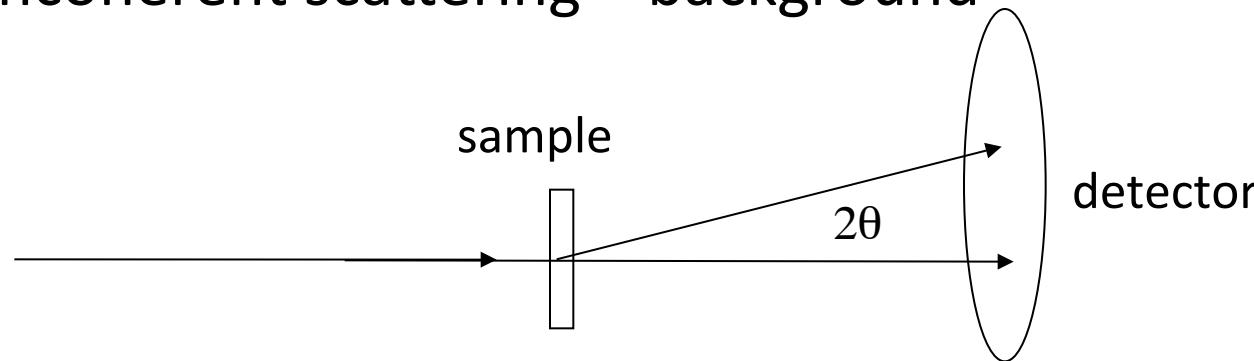


# Reference Texts

- The SANS Toolbox, B. Hammouda, NIST (available as pdf:  
[http://www.ncnr.nist.gov/staff/hammouda/the SANS toolbox.pdf](http://www.ncnr.nist.gov/staff/hammouda/the_SANS_toolbox.pdf))
- Structure Analysis by SAXS & SANS, L.A. Fegin & D.I. Svergun (1987) (available as pdf:  
[http://www.embl-hamburg.de/biosaxs/reprints/feigin svergun 1987.pdf](http://www.embl-hamburg.de/biosaxs/reprints/feigin_svergun_1987.pdf))
- Small Angle X-ray Scattering, eds O. Glatter & O. Kratky (1982) (available as pdf:  
<http://physchem.kfunigraz.ac.at/sm/Software.htm>)

# What is SAS?

- Coherent, elastic scattering of radiation at small angles – close to the straight-through beam
- Incoherent scattering = background

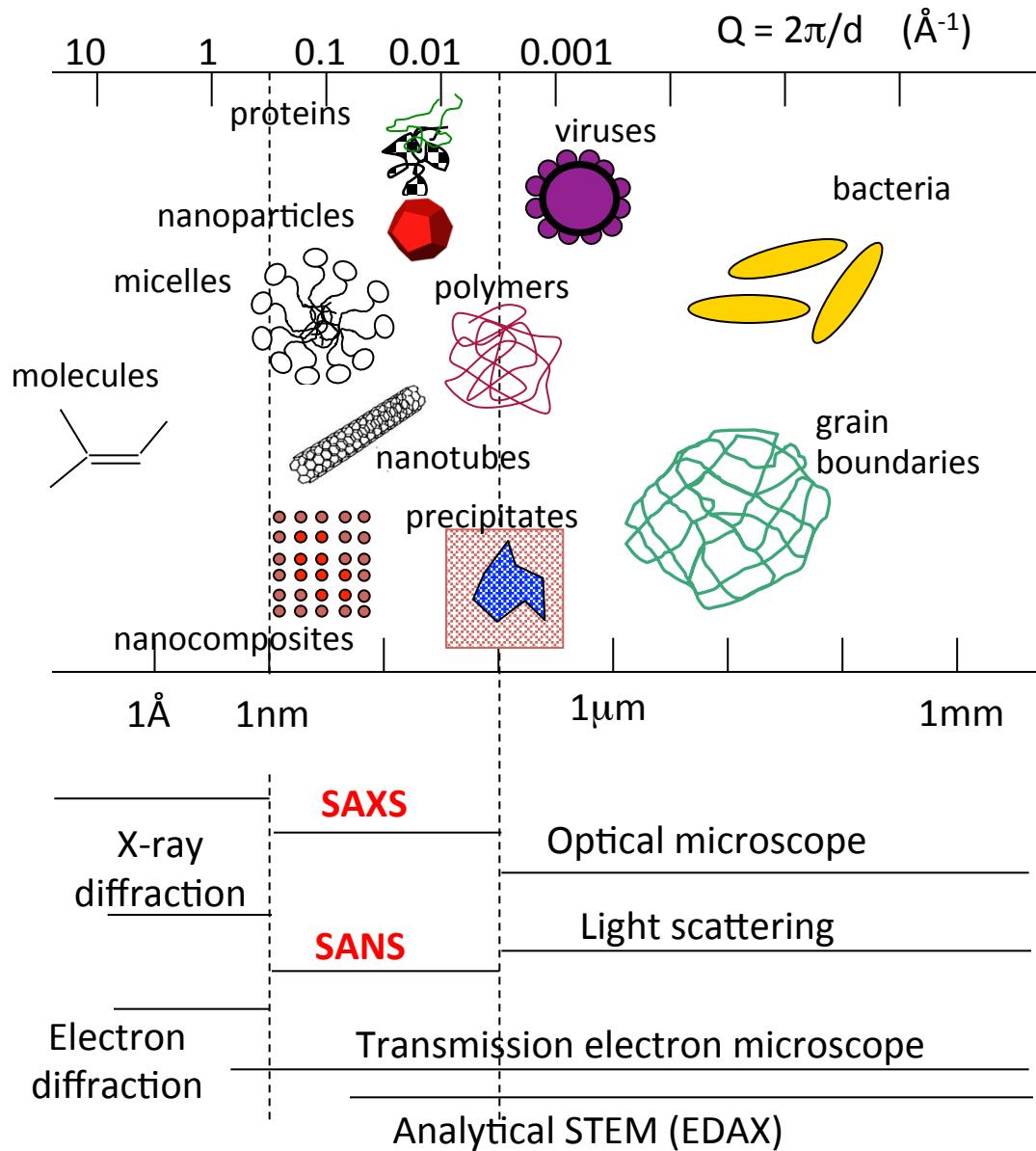


- Typically  $0.3 < 2\theta < 5^\circ$  or  $Q < 0.5 \text{ \AA}^{-1}$

$$Q = \frac{4\pi}{\lambda} \sin \theta \quad \text{units: \AA}^{-1}$$

$$Q = \frac{2\pi}{d} \quad \Rightarrow \text{bigger distance, smaller } Q$$

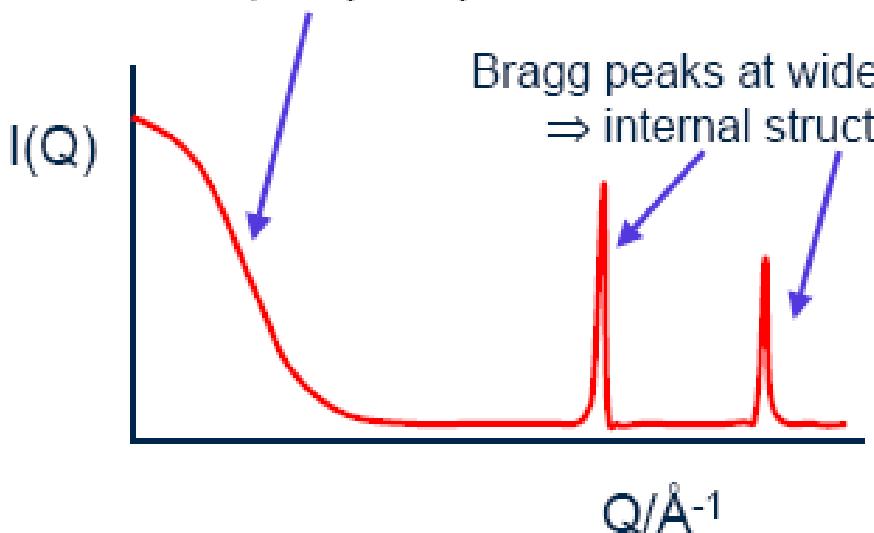
# Size Range Comparisons



# Scattering from Large Structures

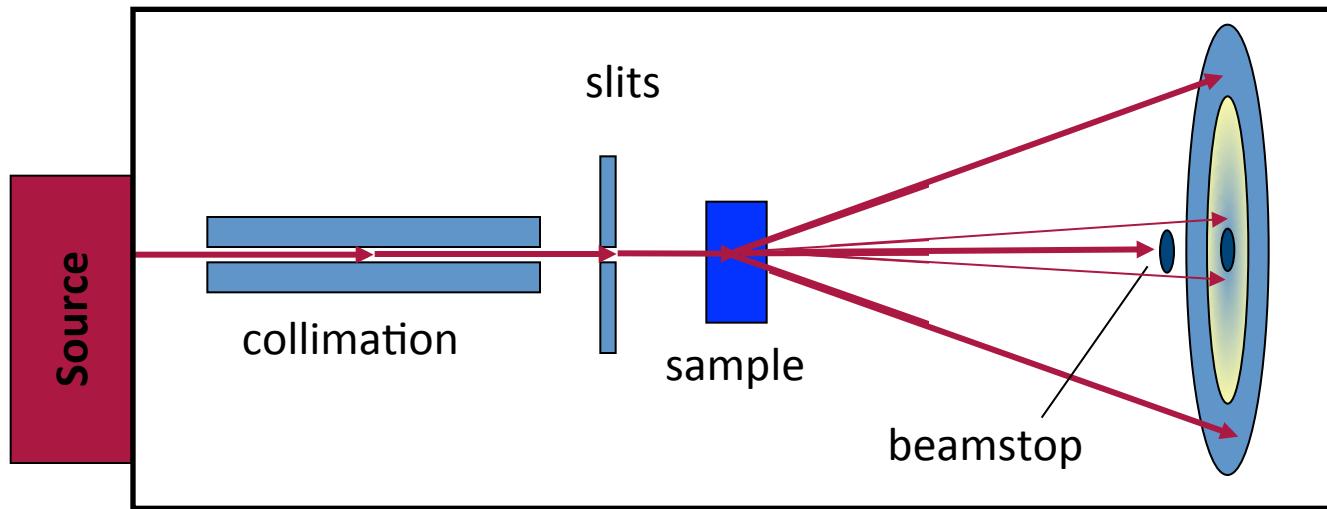
- Neutron/X-ray wavelength  $\lambda \approx$  space between atoms in crystal  
     $\Rightarrow$  bounce off layers of atoms like light off a mirror  
     $\Rightarrow$  see diffraction peaks at high angles (correspond to atomic positions)
- BUT for larger objects, sees average structure

Small angle scattering  
 $\Rightarrow$  size , shape of particles



- large structures scatter at small angles  
 $\Rightarrow$  for techniques using small angles use material properties rather than atomic properties

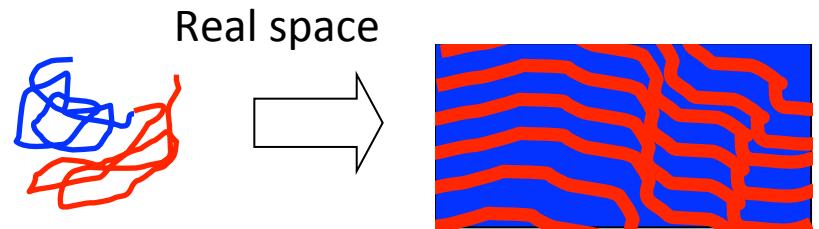
# SAS Instruments



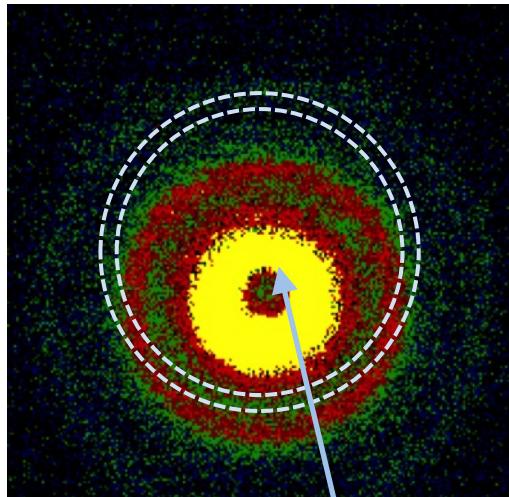
- Neutrons/X-rays must be parallel to each other; “collimated”
- Slit defines shape of beam (circle, square, slit)
- Distance from sample to detector & wavelength determines size range measured
  - Tof – wide simultaneous Q range, lower flux
  - Reactor – smaller Q range, higher flux at short sample-detector distances

# Scattering Patterns: From detector to 1D

- eg diblock copolymers
- Circular 1D average
  - take average over ring
  - each ring corresponds to one data point in reduced 1D SAXS data

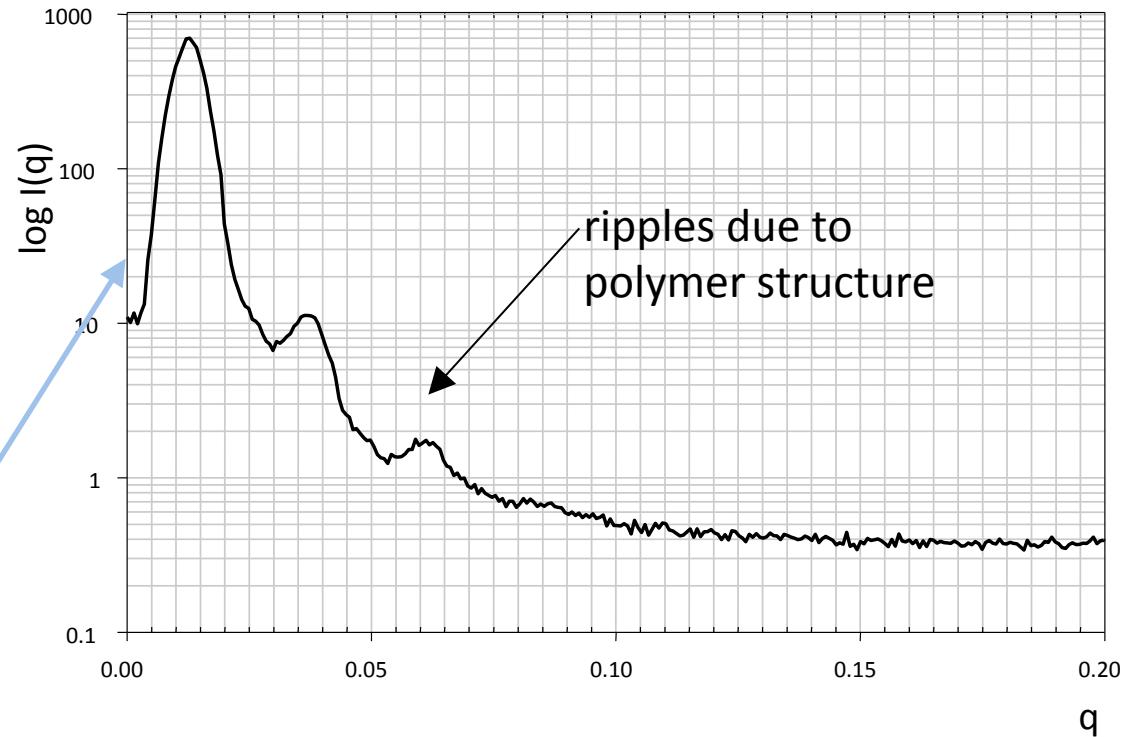


detector



shadow of beamstop

radial average:



# SAS Measurements

- Observed intensity:

$$J(\lambda, \theta) = J_0(\lambda) \Delta\Omega \eta(\lambda) T V I(Q)$$

where:

$J_0(\lambda)$  = flux incident on sample

$\Delta\Omega$  = angle covered by detector

$\eta$  = detector efficiency

T = sample transmission

V = volume of sample in beam

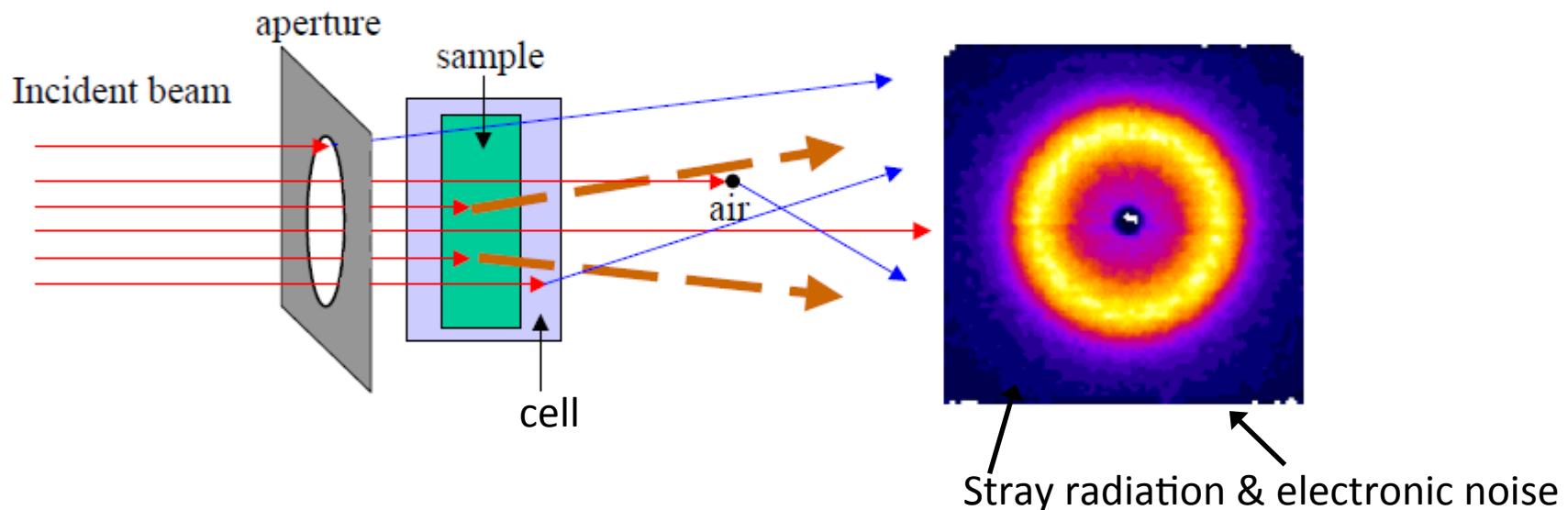
$I(Q)$  = differential cross section

⇒ contains information about sample

Can measure all of these  
⇒ Used to correct data during data reduction

# Sample Scattering

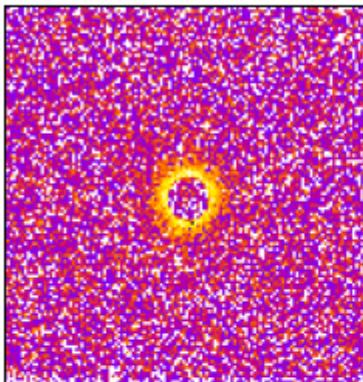
- Measured intensity due to:
  - Scattering from sample
  - Scattering from container, slits, air etc
  - Stray X-rays and electronic noise



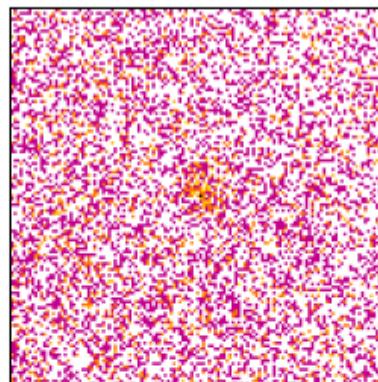
Need to measure more than just sample scattering...

# Extra measurements

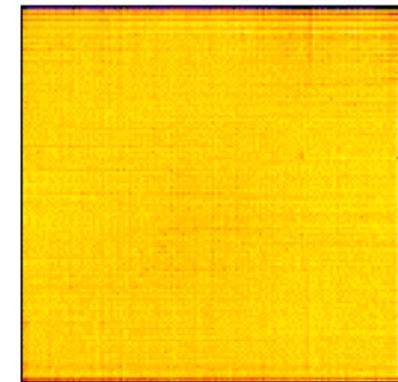
Empty cell



Blocked beam



Detector efficiency



## Source

Scattering from:

- 1) empty cell
- 2) windows & collimation slits
- 3) air scattering

- Minimize air in beam path

- Carefully choose cell & window materials

- Measure an empty cell

## Source

- 1) Detector dark current
- 2) Stray radiation
- 3) Cosmic radiation

- Measure a blocked beam

## Why ?

Sensitivity of each pixel  
is slightly different  
(~ 1%)

- Use isotropic  
scattering material  
(Plexiglass or water)  
or “flood” source

# Standards - Intensity

- Y-axis in “counts”
  - Need to convert to absolute intensity
- Intensity standards:
  - water
  - glassy carbon
  - direct beam + attenuator (if flux is known)
  - standard polymer sample
- Scattering cross section (intensity) is known
- Measure intensity of standard under same conditions as sample
- Compare measured and known intensities
- Calculate “scale factor” to multiply data

# Scattered Intensity

- observed scattered intensity is Fourier Transform of real-space shapes

$$I(Q) = N_p V_p^2 (\rho_p - \rho_s)^2 F(Q) S(Q) + B$$

where:  $N_p$  = number of particles

$V_p$  = volume of particle

$\rho$  = scattering length density (of particle/solvent)

B = background

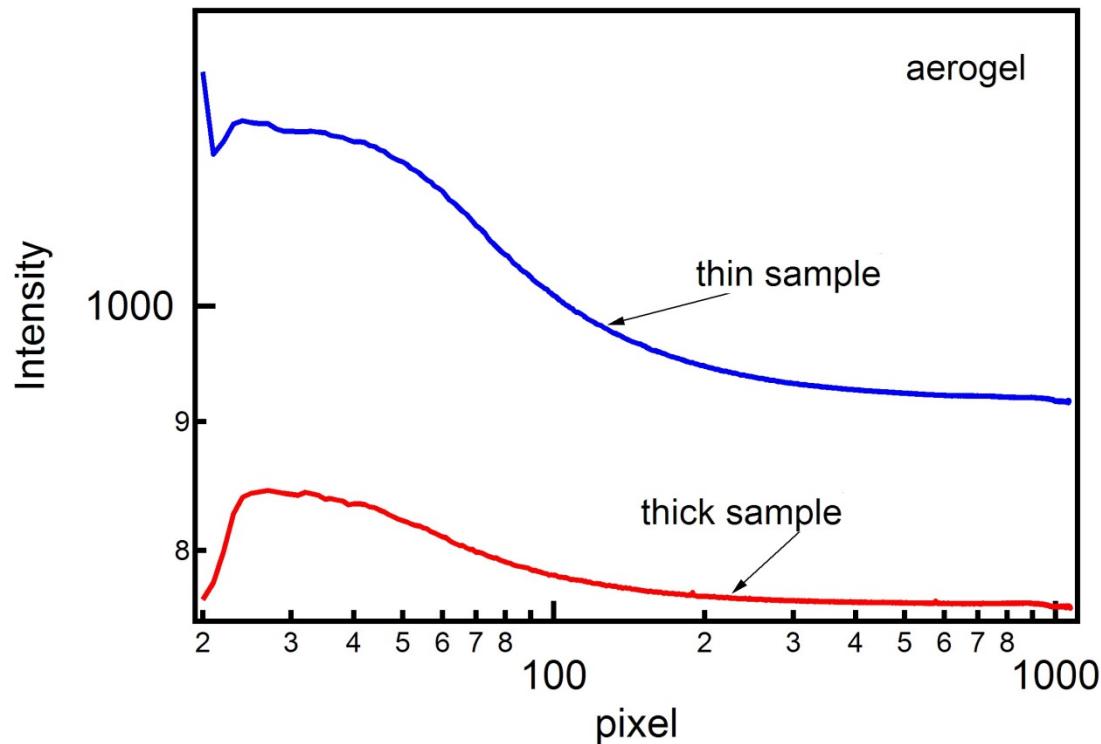
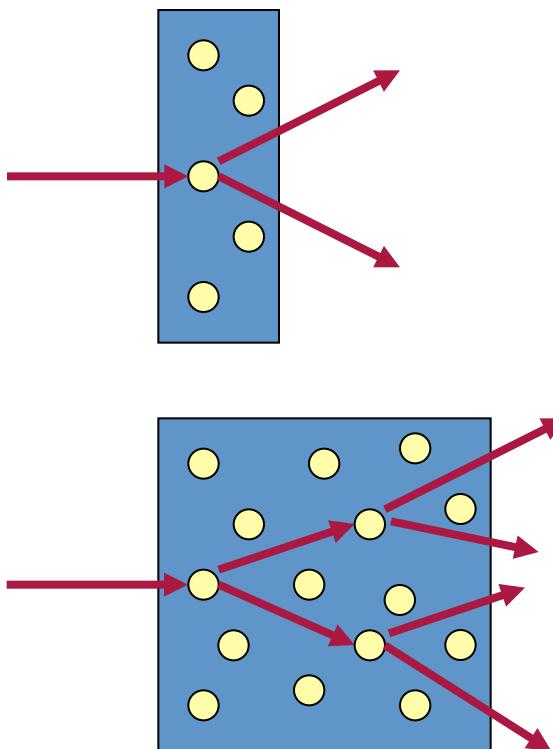
F(Q) = form factor

S(Q) = structure factor

- Sample considerations... (discussed last week)

# Sample thickness

- Affects transmission (total intensity)
- Also affects shape of curve  $\Rightarrow$  hard to analyse
- More problematic for high flux, strongly scattering samples
- Aim for  $\sim 70\%$  transmission (X-rays/ $D_2O$  solutions,  $>\sim 50\%$   $H_2O$  solutions)



# Concentration

$$I(Q) \propto N_p V_p$$

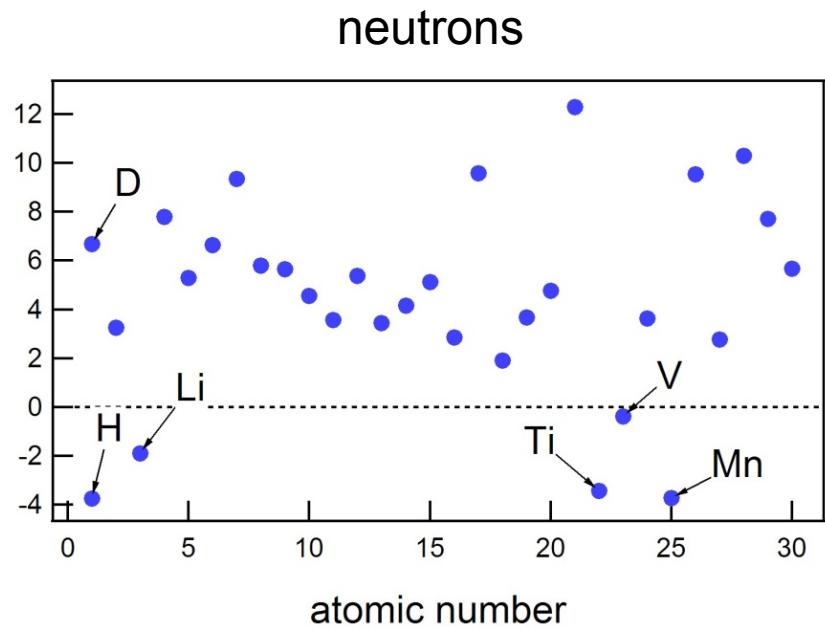
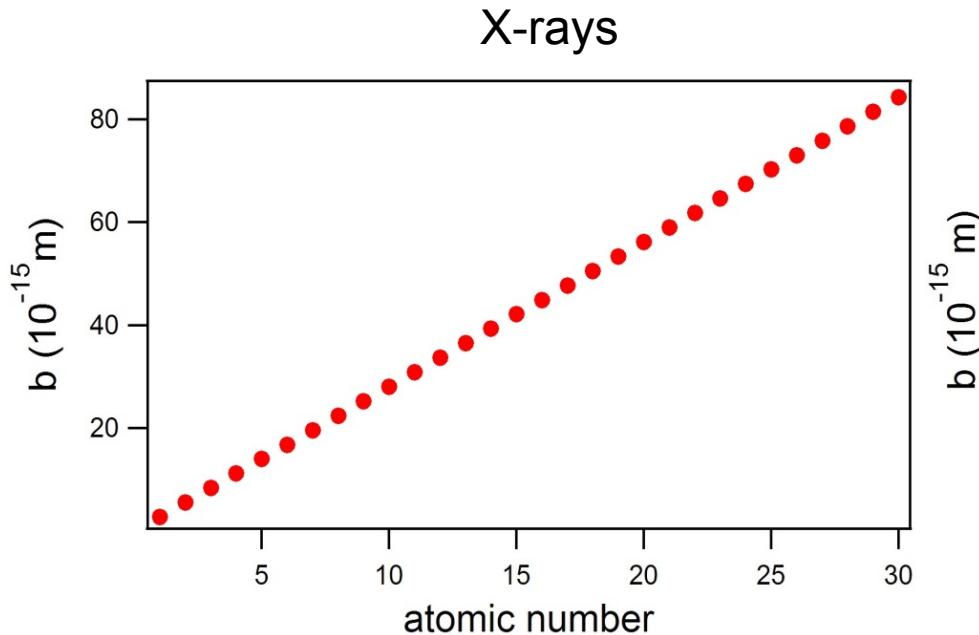
- Big particles scatter more (can hide small ones)
- Higher concentration = more signal

BUT

- **Consider detector limits!**
  - Don't burn out your detector...
- High concentration can complicate analysis
  - especially for charged particles (see later)
- Minimum concentration for neutrons/lab X-ray source: ~10mg/ml
  - watch out for highly coloured solutions eg nanoparticles

# Neutrons/X-rays & “Contrast”

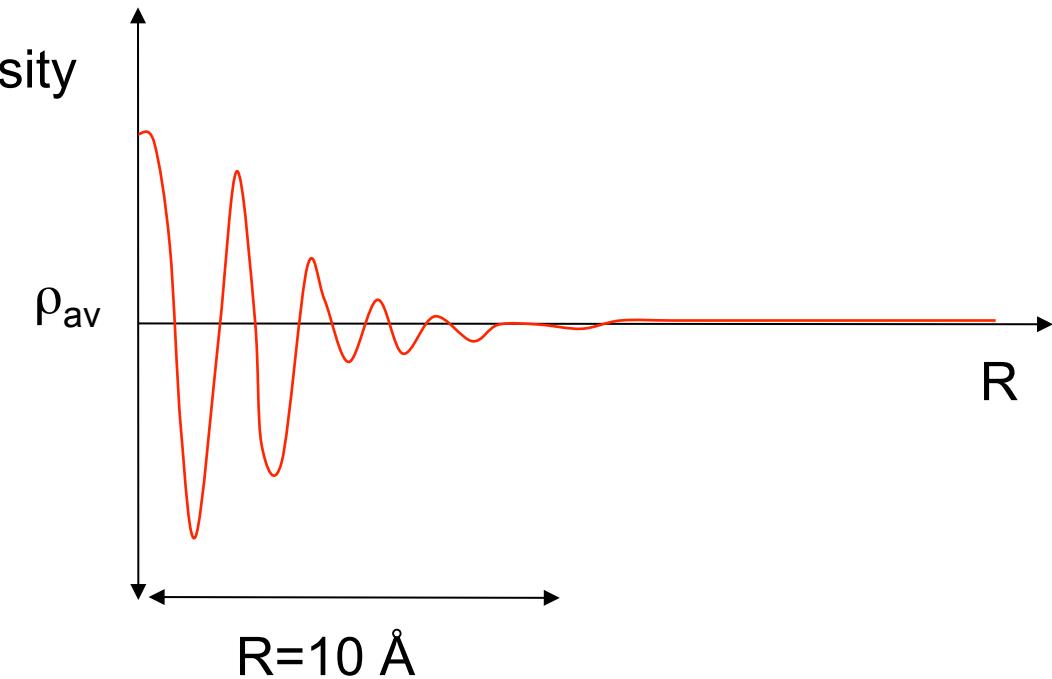
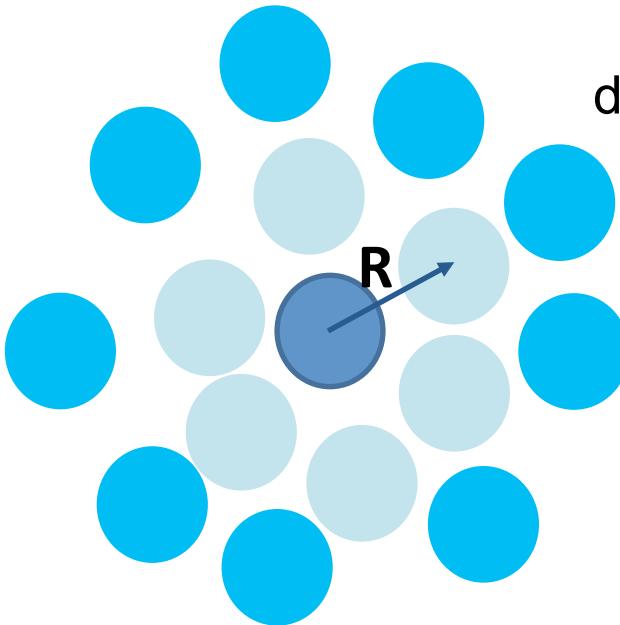
- Neutrons more penetrating than X-rays (interact less with matter)
- Interaction of neutrons with nuclei depends on isotope
- Interaction of X-rays just depends on number of electrons
- $b$  = scattering length (units Å or cm, normally)
- Scattered intensity measured depends on which isotopes are in sample for neutrons, only on elements for X-rays



# Scattering from Large Structures

- Consider H<sub>2</sub>O: volume of one molecule =  $30\text{\AA}^2$

radius of one molecule =  $2\text{\AA}$



⇒ for distances  $> \sim 5$  molecules, see only average density

$$Q = 2\pi/d$$

- so can use material properties for  $Q < \sim 0.6\text{\AA}^{-1}$

# Scattering Length Density

- scattering from an object/material depends on how many electrons or nuclei there are in a unit volume
- use *scattering length density*,  $Nb$ , to calculate scattering from molecules:

$$Nb = \frac{N_A \cdot \rho}{MW} \sum_i b_i \quad \text{Units of } Nb: \text{ cm}^{-2}$$
$$= N \sum_i b_i$$

where:  $b_i$  = scattering length for element, cm

(for X-rays  $b = 2.81 \times 10^{-13} \times \text{no. of e}^- \text{ in atom}$ )

$\rho$  = density of compound, g cm<sup>-3</sup>

$N_A$  = Avogadro's number, mol<sup>-1</sup>

MW = molecular weight, g mol<sup>-1</sup>

$N$  = number density of atoms in material, cm<sup>-3</sup>

NB/ if feeling lazy see: [www.ncnr.nist.gov/resources/sldcalc.html](http://www.ncnr.nist.gov/resources/sldcalc.html)

# Important Scattering Length Densities

$$\text{H}_2\text{O} \quad b_{\text{H}} = -3.742 \times 10^{-13} \text{ cm}$$

$$b_{\text{O}} = 5.805 \times 10^{-13} \text{ cm}$$

$$\text{MW}_{\text{H}_2\text{O}} = 2\text{H} + \text{O} = 20 \text{ g mol}^{-1}$$

$$\rho_{\text{H}_2\text{O}} = 1.0 \text{ g cm}^{-3}$$

$$\text{D}_2\text{O} \quad b_{\text{D}} = 6.674 \times 10^{-13} \text{ cm}$$

$$b_{\text{O}} = 5.805 \times 10^{-13} \text{ cm}$$

$$\text{MW}_{\text{D}_2\text{O}} = 2\text{D} + \text{O} = 18 \text{ g mol}^{-1}$$

$$\rho_{\text{D}_2\text{O}} = 1.1 \text{ g cm}^{-3}$$

$$Nb = N \sum_i b_i$$

These have similar number densities of atoms ie number of atoms in 1 cm<sup>3</sup>:

$$N_{\text{H}_2\text{O}} = \frac{6.022 \times 10^{23} \text{ atoms mol}^{-1} \times 1.0 \text{ g cm}^{-3}}{18 \text{ g mol}^{-1}}$$

$$N_{\text{H}_2\text{O}} = 3.35 \times 10^{22} \text{ atoms cm}^{-3}$$

$$N_{\text{D}_2\text{O}} = 3.31 \times 10^{22} \text{ atoms cm}^{-3}$$

BUT very different scattering length densities!

$$Nb_{\text{H}_2\text{O}} = (2b_{\text{H}} + b_{\text{O}}) \times N_{\text{H}_2\text{O}}$$

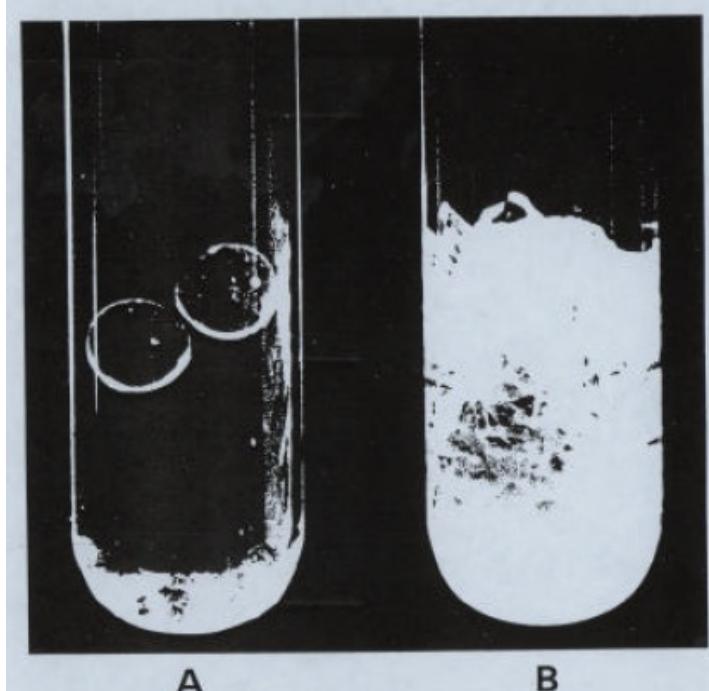
$$Nb_{\text{H}_2\text{O}} = -0.562 \times 10^{10} \text{ cm}^{-2}$$

$$Nb_{\text{D}_2\text{O}} = (2b_{\text{D}} + b_{\text{O}}) \times N_{\text{D}_2\text{O}}$$

$$= 6.34 \times 10^{10} \text{ cm}^{-2}$$

# Contrast & Contrast Matching

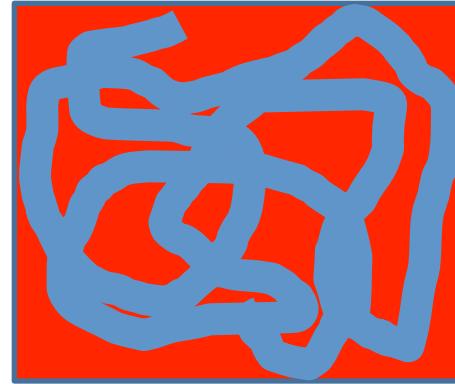
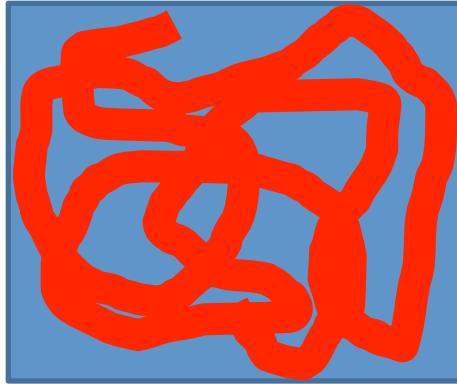
- Both tubes contain pyrex fibers + borosilicate beads + solvent.
  - (A) solvent refractive index matched to pyrex fibres
  - (B) solvent index different from both beads & fibers – scattering from fibers dominates



Similarly, there must be a difference between object and surrounding to measure scattering

$$I(Q) \propto (\rho_p - \rho_s)^2$$

# Babinet's Principle



- These two structures give the same scattering

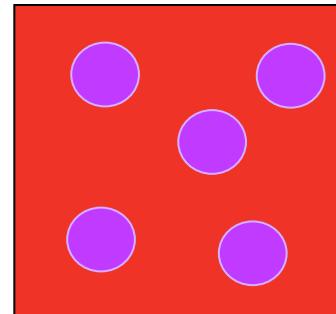
$$I(Q) \propto (\rho_p - \rho_s)^2$$

- Contrast is relative
- Loss of phase information i.e.: is  $\rho_1 > \rho_2$ ?
- Very important in multi-phase systems
  - Solve by use of multiple contrasts using SANS!  
(for X-rays = anomalous scattering)

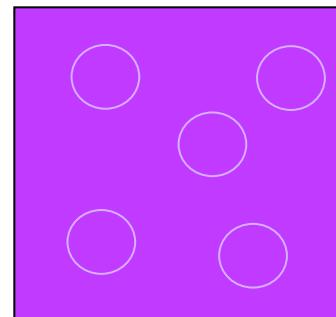
# Scattering $\propto$ “Contrast”

- objects and solvent have different **scattering length densities** (SLD)
- Intensity  $\propto$  SLD **difference** between solvent & particle
- in water for neutrons can manipulate solvent  $\rho$  by using mixture of H<sub>2</sub>O and D<sub>2</sub>O
- When solvent and object have same SLD they are said to be “contrast matched”

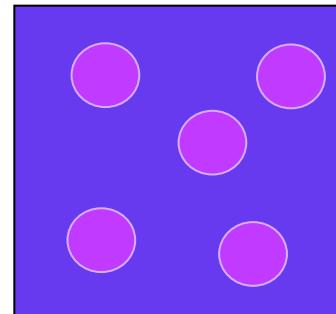
Example: silica spheres in water



30% D<sub>2</sub>O in H<sub>2</sub>O



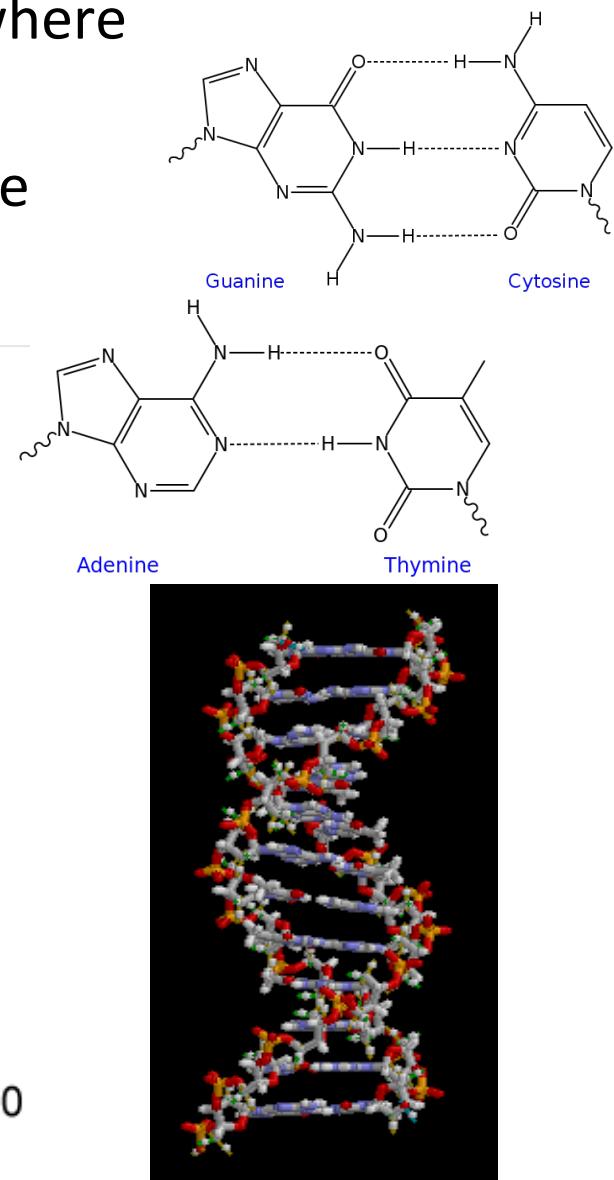
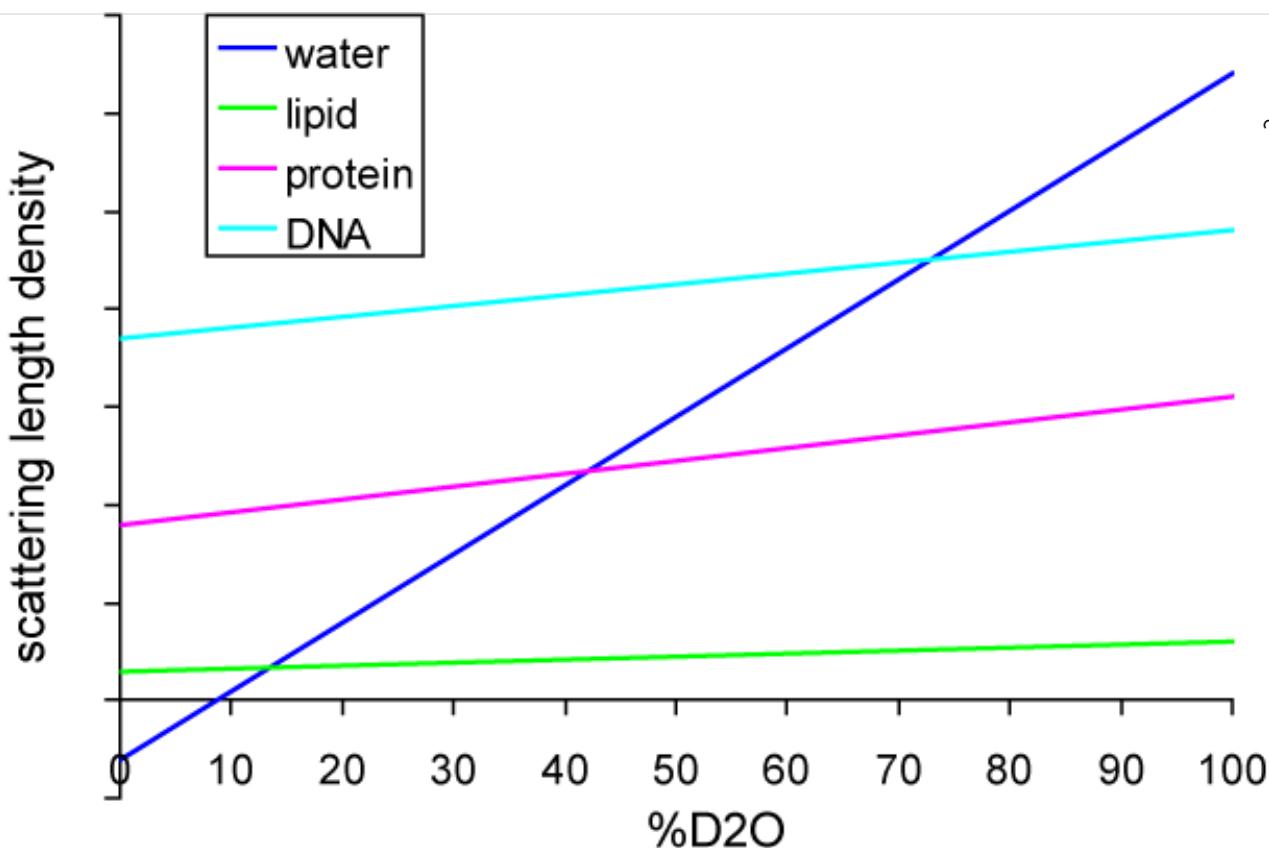
59% D<sub>2</sub>O in H<sub>2</sub>O



95% D<sub>2</sub>O in H<sub>2</sub>O

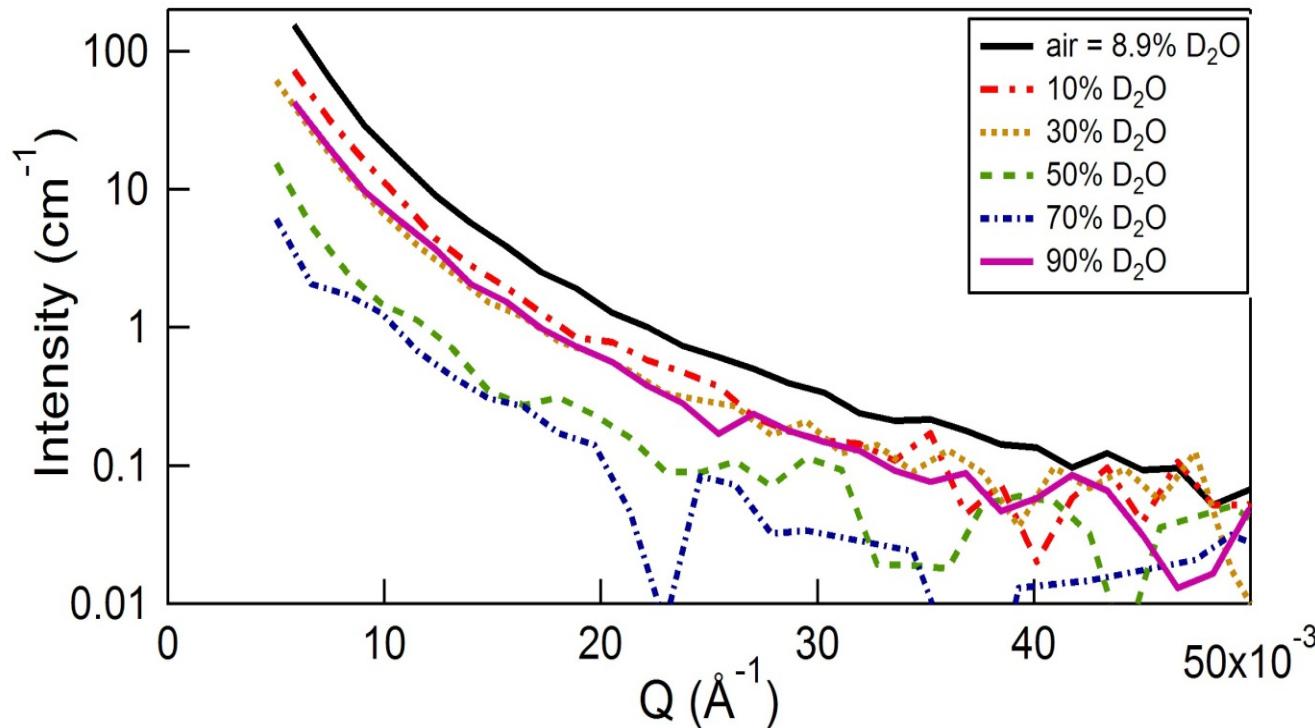
# Predicting Contrast Match Point

- By calculating the SLD can predict  $\%D_2O$  where the scattering signal will be zero
- BUT if have exchangeable hydrogens in the structure the SLD will vary with  $\%D_2O$



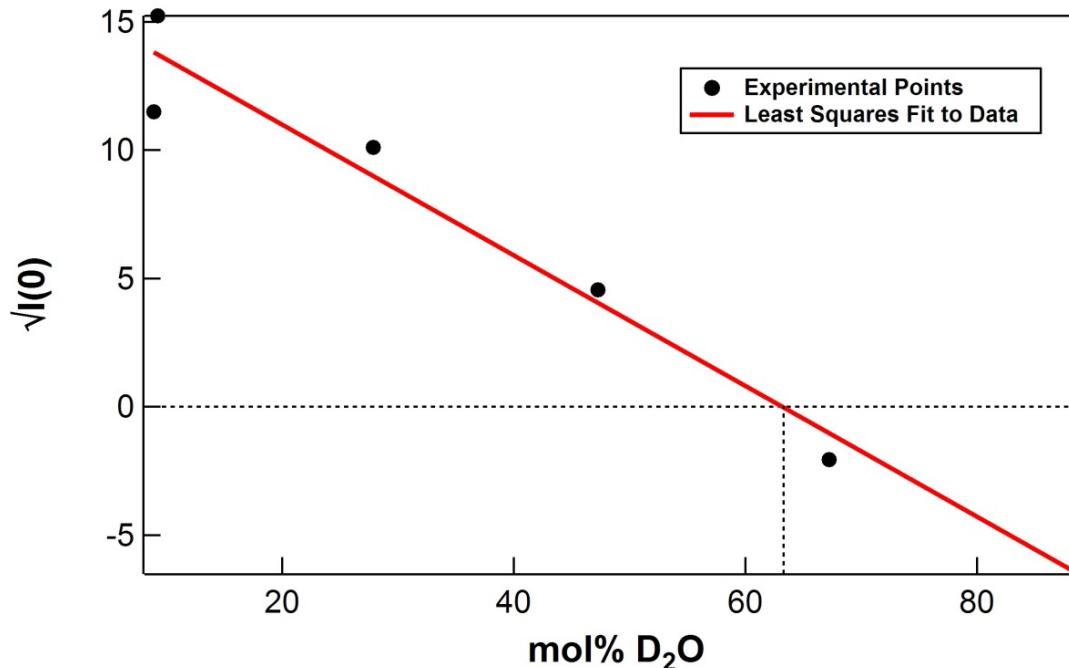
# Neutron “Contrast” Series

- intensity of scattering depends on difference between particle and solution.
$$I \propto (Nb_{\text{particle}} - Nb_{\text{solution}})^2$$
- measure scattering at a series of solution contrasts
- extrapolate scattering to  $Q = 0$  and measure  $I_0$



# Contrast Match Point

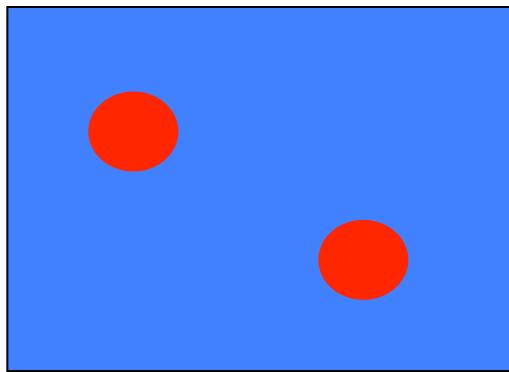
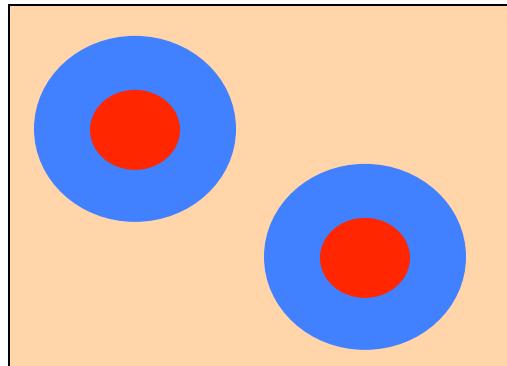
- Plot as  $\sqrt{I_0}$  vs  $[D_2O]$



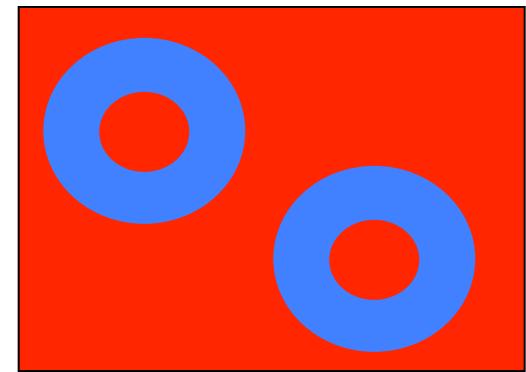
- Place where line cuts zero is where the solution has the same scattering length density as the particle  
⇒ *contrast matched*
- Can use this to find the density of the particle

# Neutron “Contrast” for Complex Objects

- contrast matching allows us to “remove” scattering from parts of an object



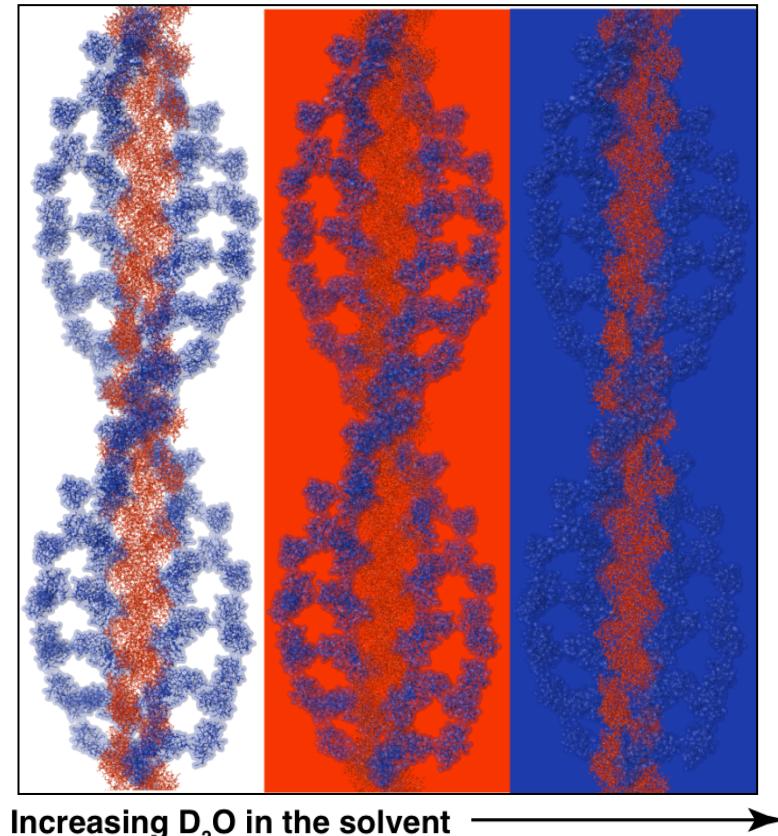
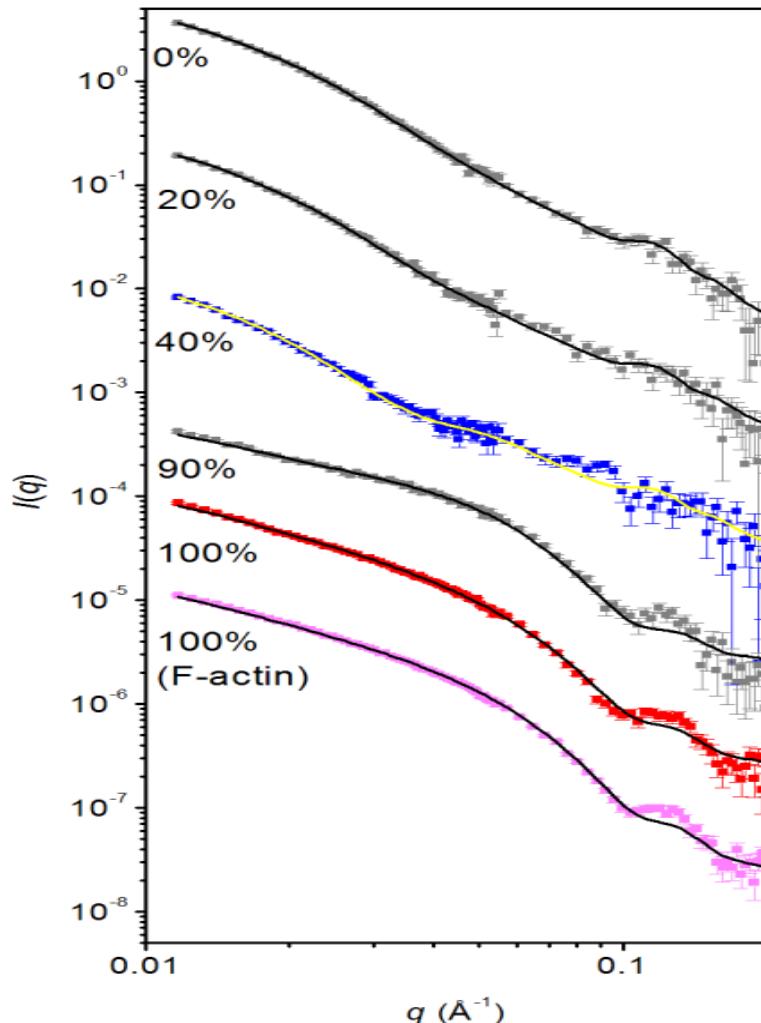
“shell-contrast”  
⇒ see only core



“core-contrast”  
⇒ see only shell

# Solvent matching for C0C2-actin assembly

- cardiac myosin binding protein C (C0C2) has extended modular structure
- Mixing C0C2 with G- actin solutions results in a dramatic increase in scattering signal due to formation of a large, rod-shaped assembly



Whitten, Jeffries, Harris, Trehewella (2008)  
Proc Natl Acad Sci USA 105, 18360-18365

# Scattered Intensity

- For concentrated solutions:

$$I(Q) = N_p V_p^2 (\rho_p - \rho_s)^2 F(Q) S(Q) + B$$

where:  $N_p$  = number of particles

$V_p$  = volume of particle

$\rho$  = scattering length density (of particle/solvent)

B = background

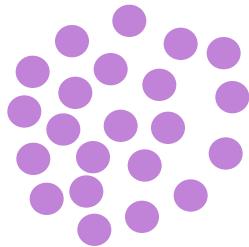
F(Q) = form factor

S(Q) = structure factor

**Form Factor** = scattering from within same particle  
⇒ depends on particle shape

**Structure Factor** = scattering from different particles  
⇒ depends on interactions between particles

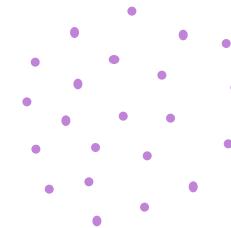
# Solution of particles



=



\*



Solution

$$I(c, Q)$$

Motif

(protein, micelle, nanoparticle)

$$F(0, Q)$$

\*

Lattice

$$S(c, Q)$$

Form factor  
of the **particle**

Structure factor  
of the **particle**

c = concentration

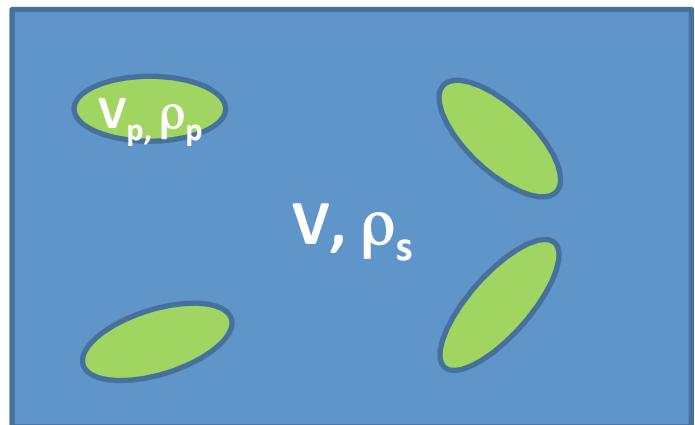
# SAS Data Analysis

- Simple but not very accurate:
  - Porod slopes
  - Guinier analysis
- More helpful, but more complex:
  - fitting models to data
- Most complex (need more data):
  - fitting protein structures using crystal structures
  - monte carlo/simulated annealing methods

# Scattering from Independent Particles

- Scattered intensity per unit volume of sample
  - arises from spatial distribution of regions with different scattering length density

$$I(q) = d\sigma/d\Omega = 1/V \int V \rho(\mathbf{r}) e^{i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r}/12$$



- For identical particles:

$$I(q) = N/V (\rho_p - \rho_s) / 12 V / p / 12 \langle 1/V \downarrow p \int_{\text{particle}} e^{i\mathbf{q} \cdot \mathbf{r}} d\mathbf{r} / 12 \rangle$$

↗ Particle form factor, F(Q)

# Dilute Randomly Ordered Uniform Particles

- scattering from independent particles:

$$I(q) = N/V (\rho \downarrow p - \rho \downarrow s) \frac{1}{2} V \downarrow p \frac{1}{2} \langle 1/V \downarrow p | \int_{\text{particle}} e^{\frac{i}{\hbar} \mathbf{q} \cdot \mathbf{r}} dr | \frac{1}{2} \rangle$$

- Assume:
  - i) system is isotropic, then  $\langle e^{\frac{i}{\hbar} \mathbf{q} \cdot \mathbf{r}} \rangle = \sin(qr)/qr$

ii) no long range order, so no correlations

between two widely separated particles

$$I(q) = I \downarrow e(q) (\rho \downarrow p - \rho \downarrow s) \frac{1}{2} V \downarrow p \frac{1}{2} \int_0^\infty \gamma(r) \sin(qr) / qr 4\pi r \frac{1}{2} dr$$

$\gamma(r)$  = correlation function within particle

$P(r) = 4\pi r^2 \gamma(r)$  is the probability of finding two points in the particle separated by  $r$

# Porod's Law

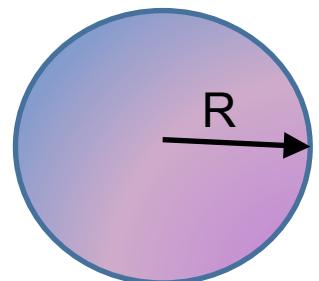
- Start with form factor:

$$F(q) = \frac{1}{V_p} \int_0^\infty \gamma(r) \frac{\sin(qr)}{qr} 4\pi r^2 dr$$

- Now consider radial pair correlation function for sphere, with sharp edges, radius R:

$$\gamma(r) = 1 - \frac{3}{4} \left(\frac{r}{R}\right) + \frac{1}{16} \left(\frac{r}{R}\right)^3$$

$$F(qR) = \frac{1}{V_p} \int_0^\infty \left[ 1 - \frac{3}{4} \left(\frac{r}{R}\right) + \frac{1}{16} \left(\frac{r}{R}\right)^3 \right] \frac{\sin(qr)}{qr} 4\pi r^2 dr$$



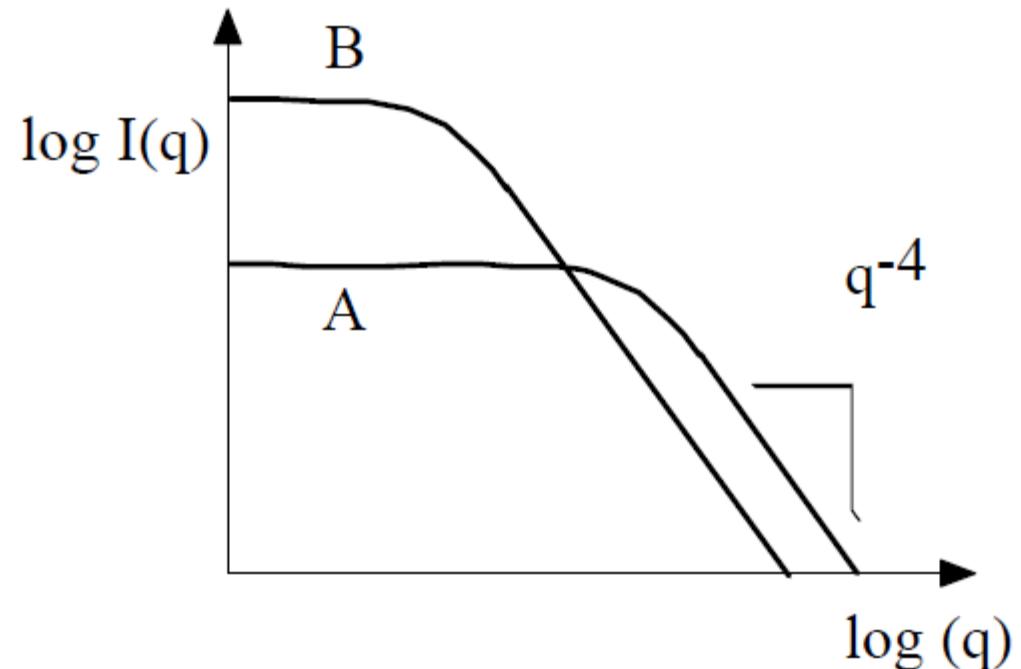
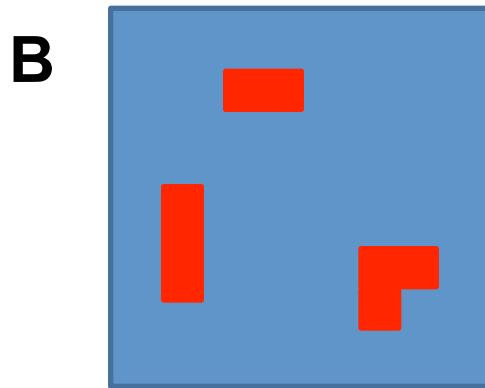
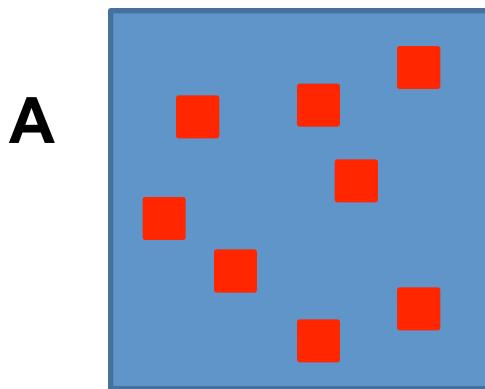
- Integrate by parts three times:

$$F(qR) \approx \frac{3}{2R^3} \frac{S_p}{V_p} \frac{1}{q^4}$$

At high scattering angles, for any system with sharp, smooth surfaces:  $I(Q) \propto \frac{1}{q^4}$

# Porod Scattering

- Slope at high  $q$  the same
- But point where slope changes depends on particle dimensions

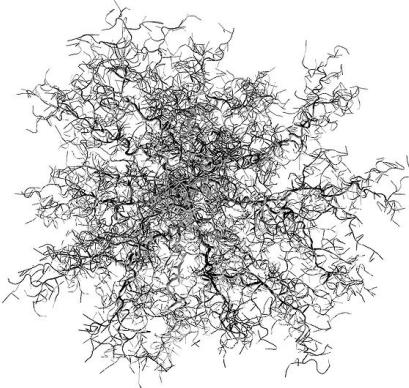


10% red / 90% blue in each square

\*Glatter & Kratky pp. 30-1.

# Fractal Systems

- Fractals are systems that are self-similar as you change scale



Diffusion-limited aggregation in 3 dimensions  
(Paul Bourke, [http://local.wasp.uwa.edu.au/~pbourke/  
fractals/dla3d/](http://local.wasp.uwa.edu.au/~pbourke/fractals/dla3d/))

- For a **Mass Fractal** the number of particles within a sphere radius  $R$  is proportional to  $R^D$  where  $D$  = fractal dimension
- Thus:

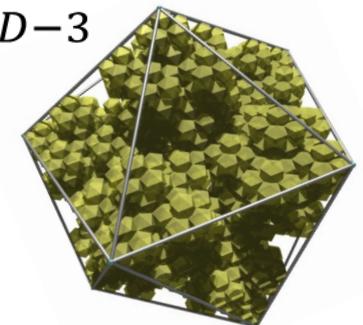
$$\begin{aligned} 4\pi R^2 \gamma(R) dR &= \text{number of particles between distance } R \text{ and } R+dR \\ &= c R^{D-1} dR \end{aligned}$$

# Fractal Systems Continued...

- So for a **Mass Fractal**:

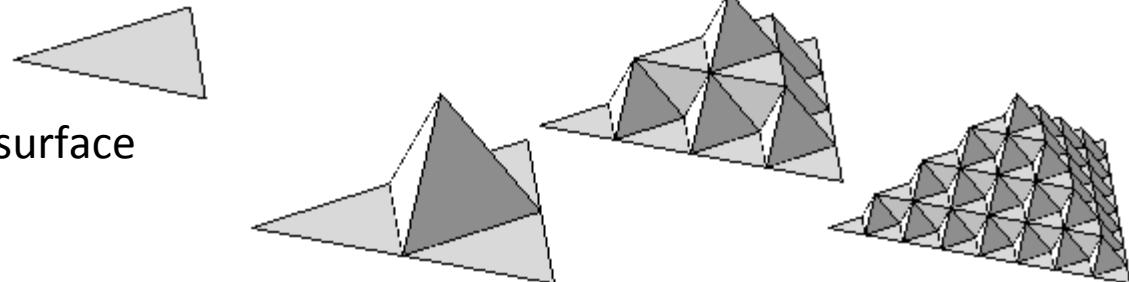
$$F(Q) = \int dR e^{iQR} \gamma(R) = \frac{2\pi}{Q} \int dR . R \sin(QR) . \left(\frac{c}{4\pi}\right) R^{D-3}$$

$$= \frac{c}{2} \frac{1}{Q^D} \int dx . x^{D-2} . \sin x = \frac{\text{constant}}{Q^D}$$



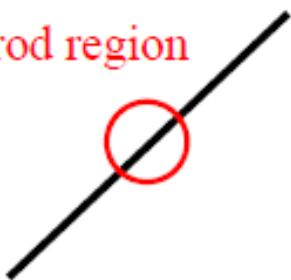
Paul Bourke

- For a **fractal surface** can show that  $F(Q) = \frac{\text{constant}}{Q^{6-D}}$  (this reduces to the Porod Law for smooth surfaces of dimension 2)



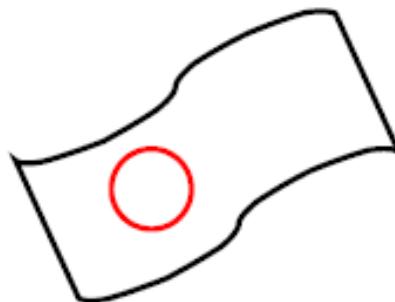
First stages of Koch (triangle) surface  
(Robert Dickau)

Porod region



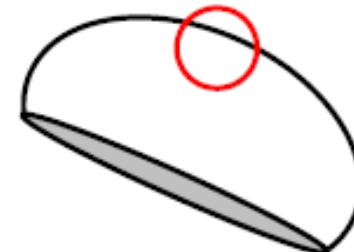
1D object

$$I(Q) \sim Q^{-1}$$



2D object

$$Q^{-2}$$



3D object

$$Q^{-4}$$



$$Q^{-5/3}$$



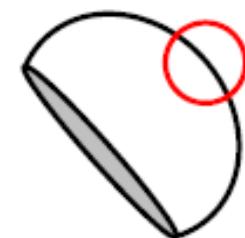
$$Q^{-2}$$



$$Q^{-3}$$



$$Q^{-3}$$

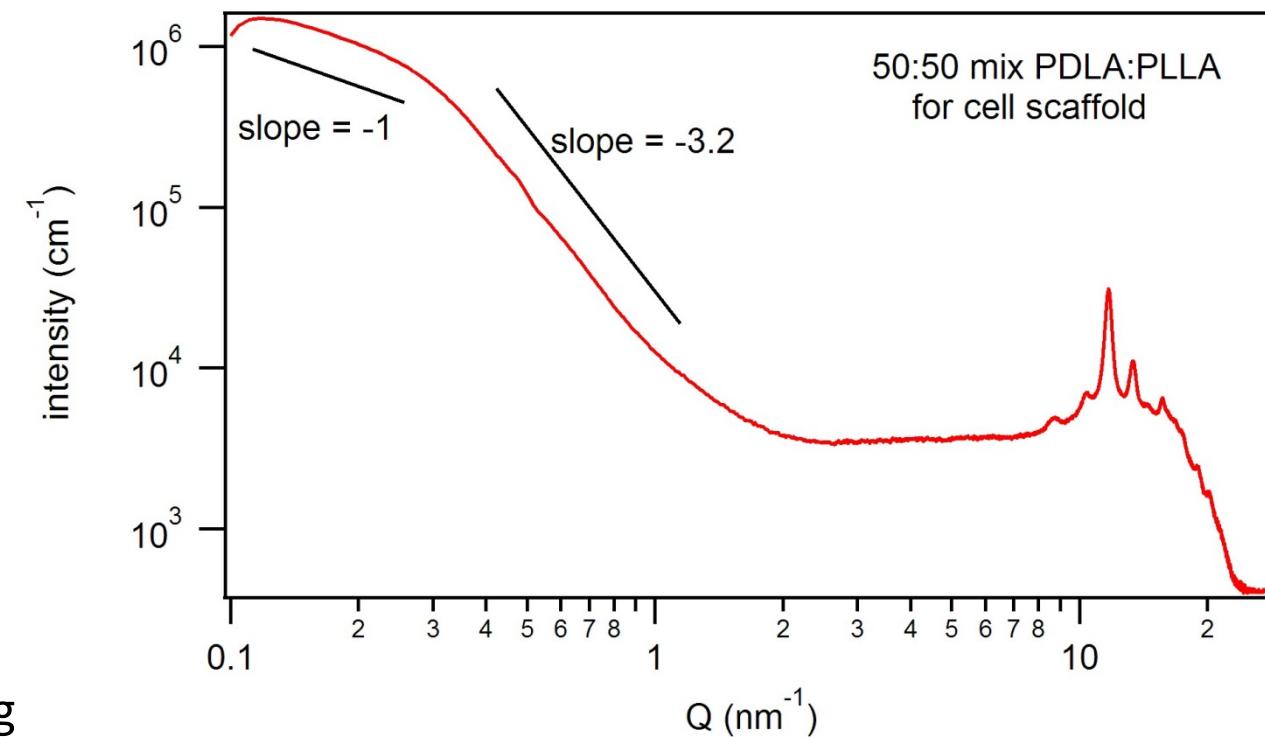


$$Q^{-4}$$

MASS FRACTALS

SURFACE FRACTALS

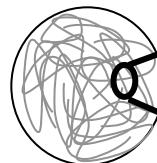
# Porod Slopes & Structures



NB/ SAXS data,  
seldom measure such  
a wide Q range in  
SANS

eg

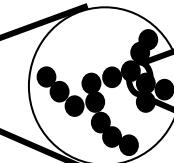
continuum



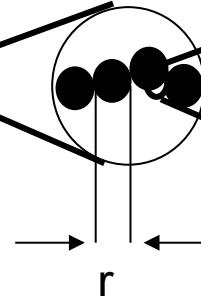
cluster



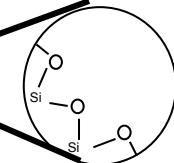
network



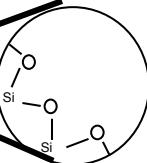
particle



surface



atoms

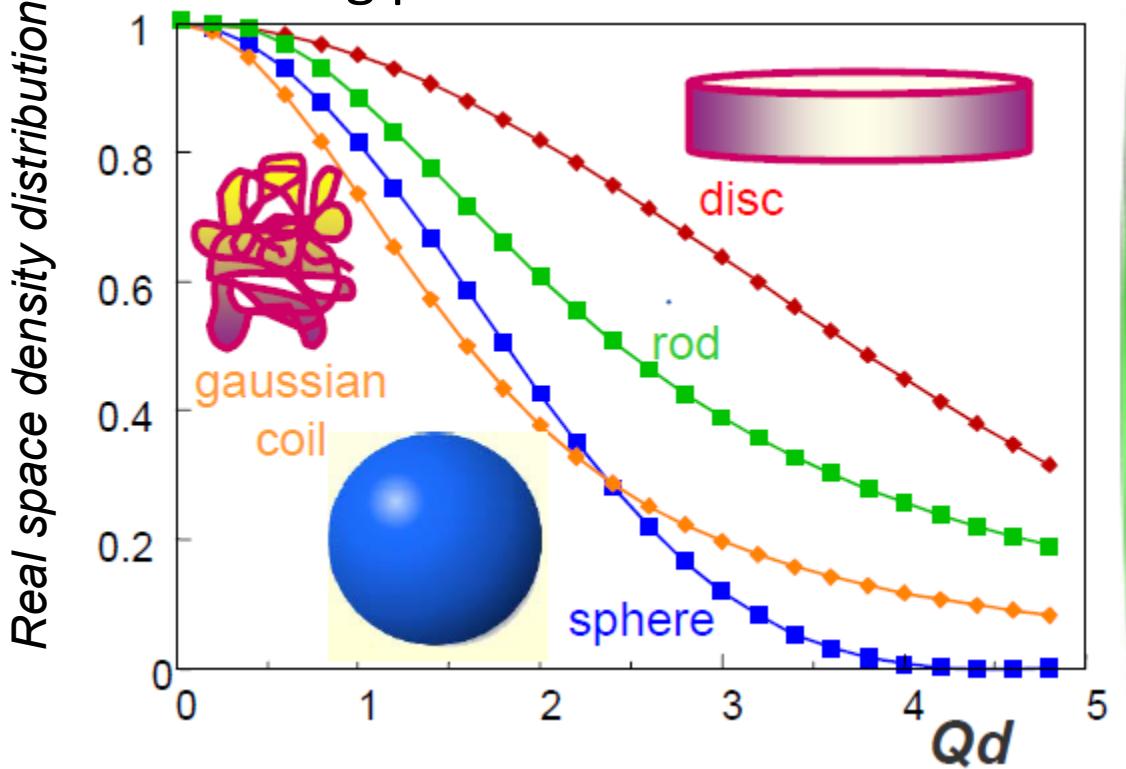


# Form Factors

- Form factors are the sum of scattering from every point inside a particle

$$F(Q) = \sum_I \sum_J \left\langle \frac{\sin(Qr_{IJ})}{Qr_{IJ}} \right\rangle_{\text{orientations}}$$

- Simplify to the integral
- Scattering pattern calculated from the Fourier transform of the real-space density distribution



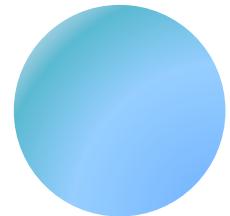
- Pattern for most shapes must be solved analytically
- Some simple shapes can be solved directly

# Simple Analysis - Guinier Approximation

- Assume particle is a sphere in dilute solution

$$F(Q) = \left[ \frac{3(\sin(QR_p) - QR_p \cos(QR_p))}{(QR_p)^3} \right]^2$$

$R_p$  = radius of sphere



- Measure scattering at very low angles so

$$R \downarrow g \quad Q \lesssim 1$$

- Use mathematical expansion of  $F(Q)$

$$F(Q) = 1 - \left( \frac{Q^2 R_g^2}{3} \right) + O()^5 \dots$$

- Write in logarithmic form  $\Rightarrow$

# Guinier Plots

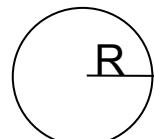
- at **low** concentrations and **small** values of  $Q$ , can write intensity as:

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

- so plot of  $\ln(I)$  against  $Q^2$  will have slope =  $\frac{-R_g^2}{3}$
- only valid for  $R_g Q \leq 1$

*Radius of Gyration* – depends on particle shape

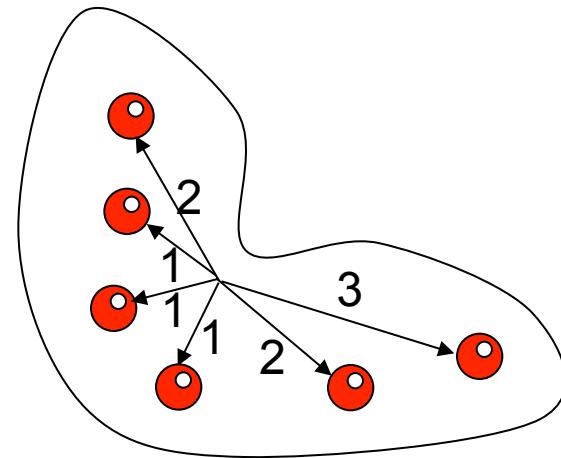
- Sphere  $R_g^2 = \frac{3}{5} R^2$



# What do we mean by “R<sub>g</sub>”?

Radius of gyration:

R<sub>g</sub><sup>2</sup> is the average squared distance of the scatterers from the centre of the object



$$R_g^2 = (1^2 + 1^2 + 1^2 + 2^2 + 2^2 + 3^2)/6 = 20/6$$

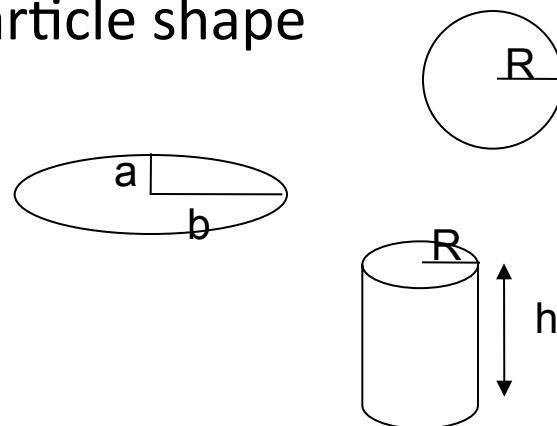
$$R_g = \sqrt{3.333} = 1.82$$

*Radius of Gyration* – depends on particle shape

- Sphere  $R_g^2 = \frac{3}{5} R^2$

- Ellipse  $R_g^2 = \frac{a^2 + b^2}{4}$

- Cylinder  $R_g^2 = \frac{R^2}{2} + \frac{h}{12}$

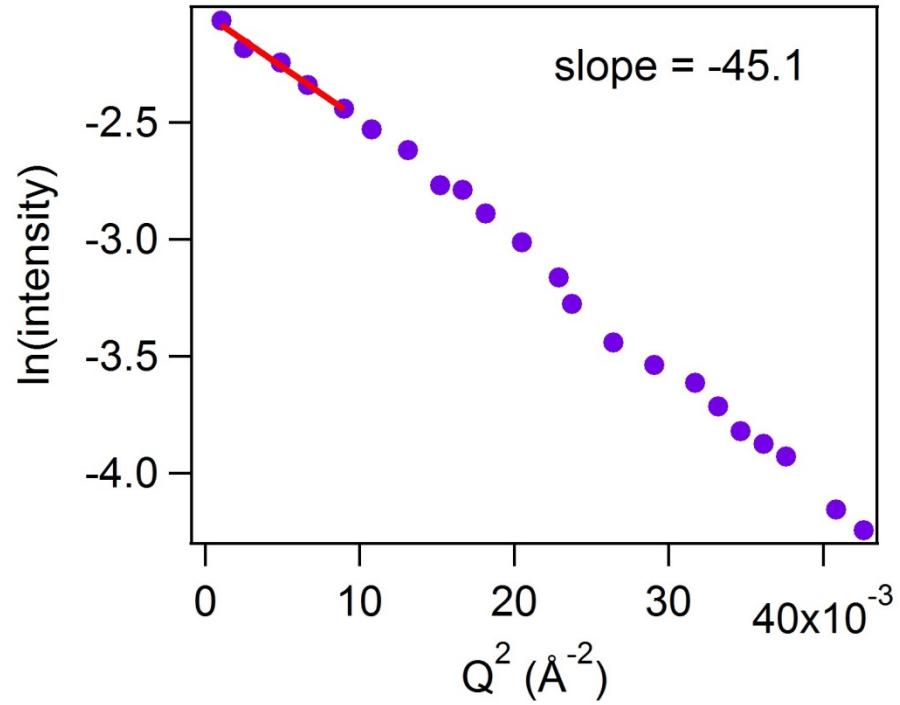


# Guinier Plot Example

- Polymerised surfactant micelles

*Large Scale Structures, ISIS Annual Report, 1999-2000* <http://www.isis.rl.ac.uk/isis2000/science/largescale.htm>

Q ( $\text{\AA}^{-1}$ )	$Q^2 (\text{\AA}^{-2}) \times 10^{-3}$	Intensity ( $\text{cm}^{-1}$ )	ln(intensity)
0.032	1.03	0.127	-2.064
0.050	2.51	0.113	-2.183
0.070	4.87	0.106	-2.245
0.081	6.56	0.096	-2.341
0.095	9.03	0.087	-2.441
0.104	10.81	0.080	-2.528
0.115	13.23	0.073	-2.618
0.123	15.13	0.063	-2.769
0.129	16.64	0.062	-2.789



$$\text{Slope} = \frac{-R_g^2}{3} = -45.1 \text{ \AA}$$

$$\text{so: } R_g = 11.6 \text{ \AA}$$

Check validity:  $R_g \times Q_{\max} = 11.6 \times 0.095 = 1.1 \quad \text{OK}$

# More Complex: Fitting Scattering

- observed scattered intensity is Fourier Transform of real-space shapes

$$I(Q) = N_p V_p^2 (\rho_p - \rho_s)^2 F(Q) S(Q) + B$$

where:  $N_p$  = number of particles

$V_p$  = volume of particle

$\rho$  = scattering length density (of particle/solvent)

B = background

F(Q) = form factor

S(Q) = structure factor

**Form Factor** = scattering from within same particle  
⇒ depends on particle shape

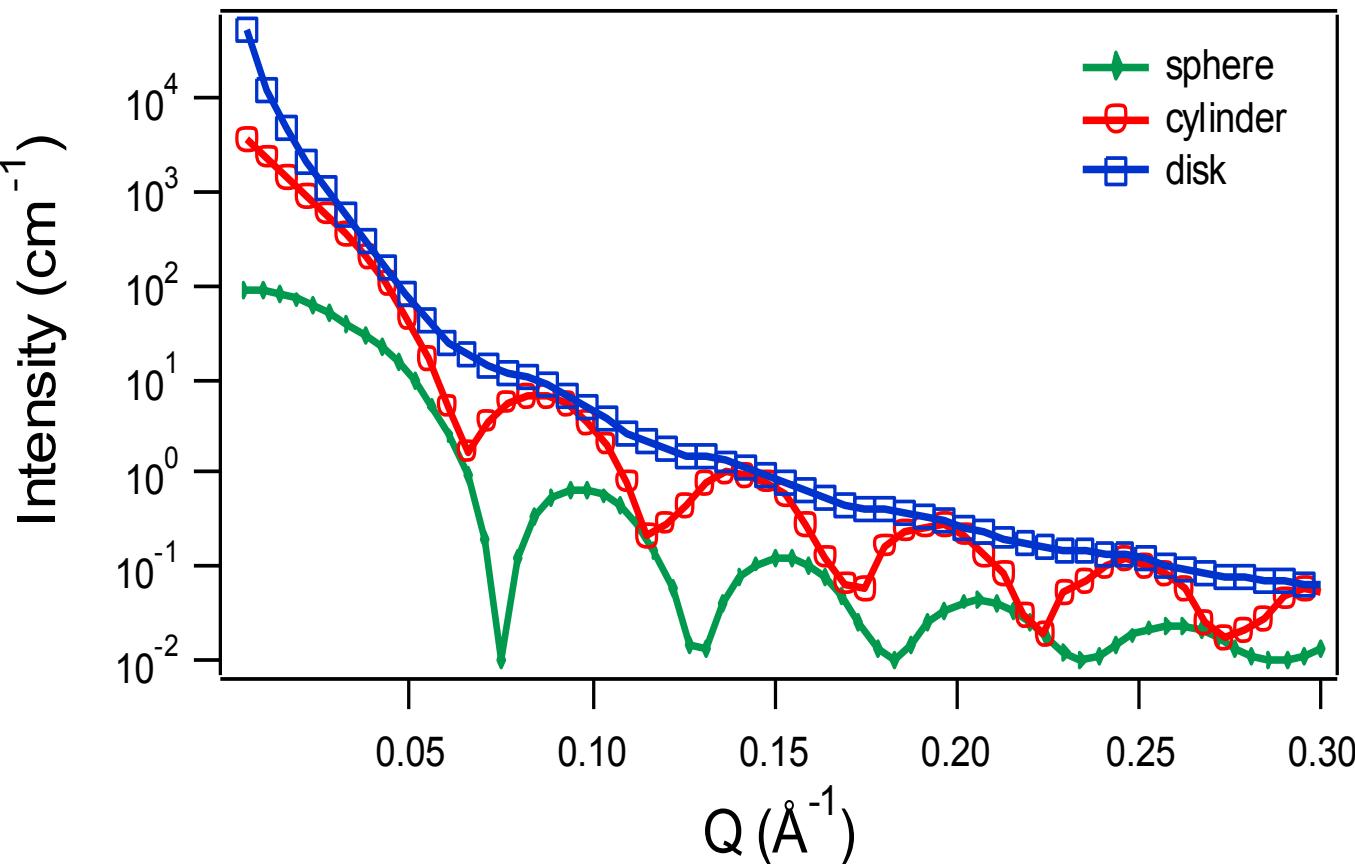
**Structure Factor** = scattering from different particles  
⇒ depends on interactions between particles

# Form Factors

- depend on shape of particle
- for dilute solutions  $S(Q) = 1$  and so  $I(Q) \propto F(Q)$

- General form of  $F(Q)$ :

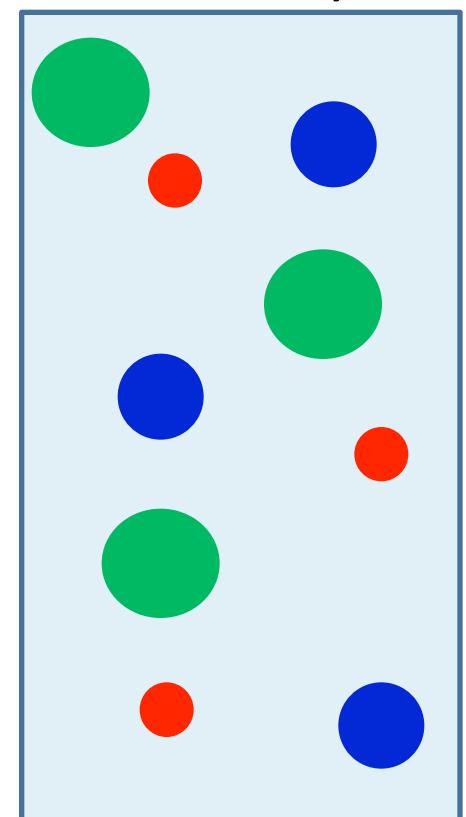
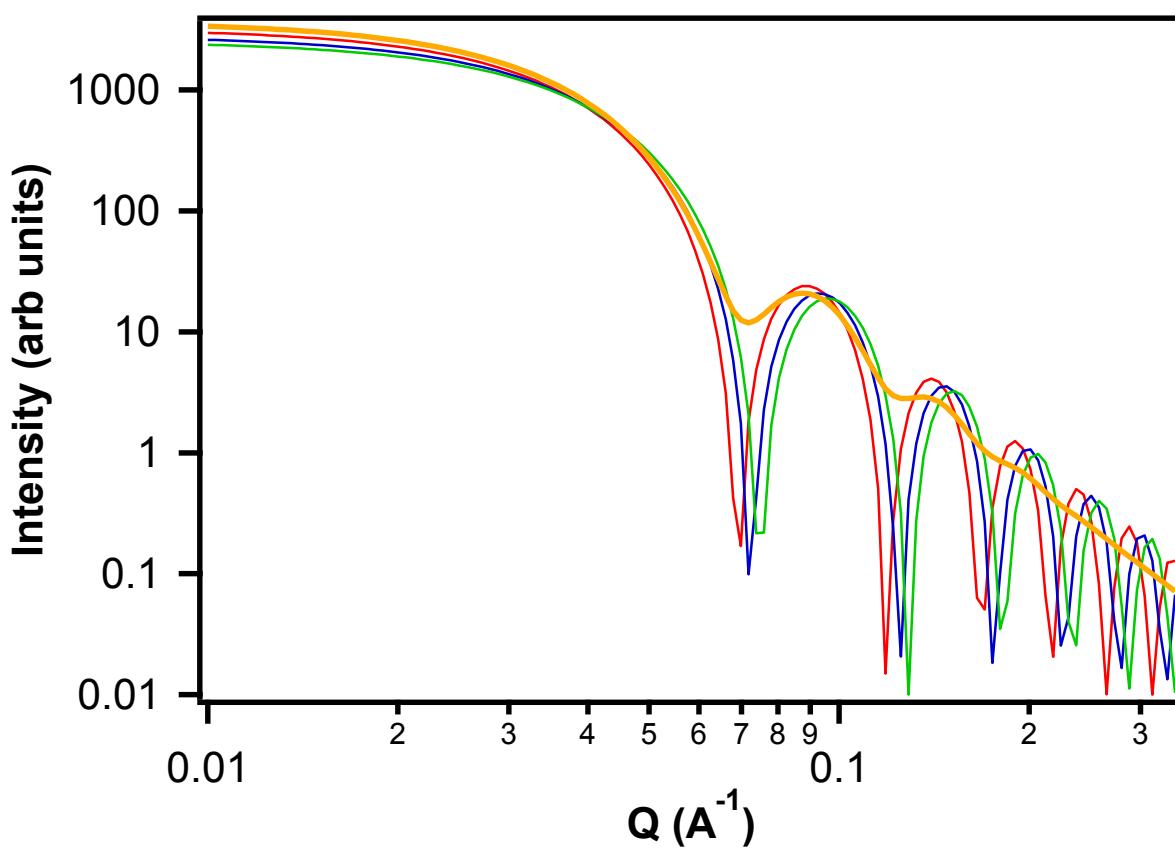
$$F(q) = \frac{1}{V_p} \int_0^{\infty} \gamma(r) \frac{\sin(qr)}{qr} 4\pi r^2 dr$$



where  
r = shape parameter  
eg radius of gyration

# Polydispersity

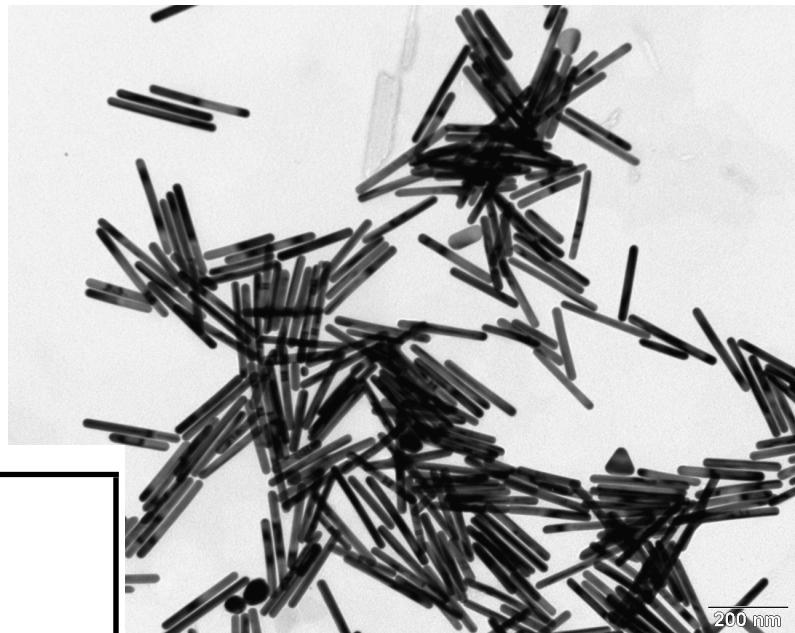
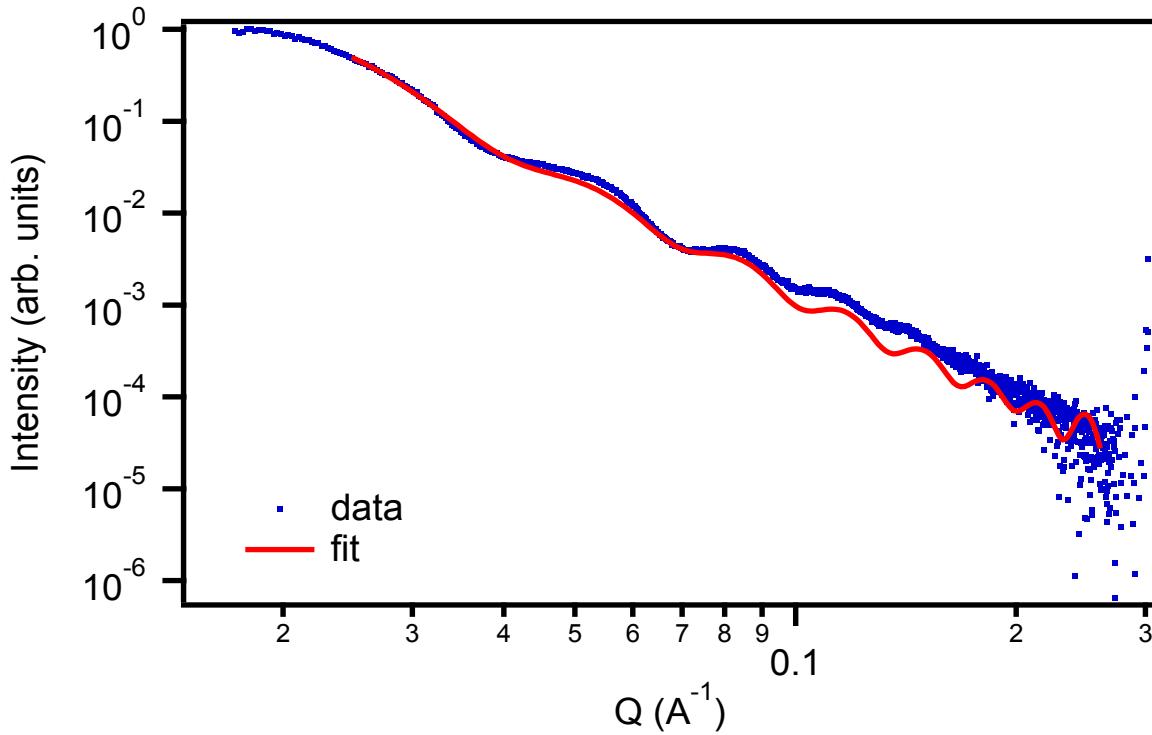
- “smears out” sharp features in pattern
- “smearing” can also be due to poor Q resolution or beam shape (correct for this during data reduction)



# Au Nanorods

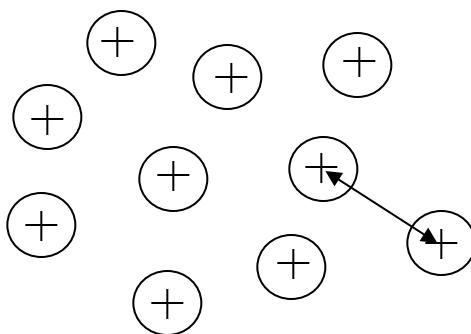
Fitted to charged cylinders

- Radius 80Å
- Length 190Å
- Polydispersity 0.29



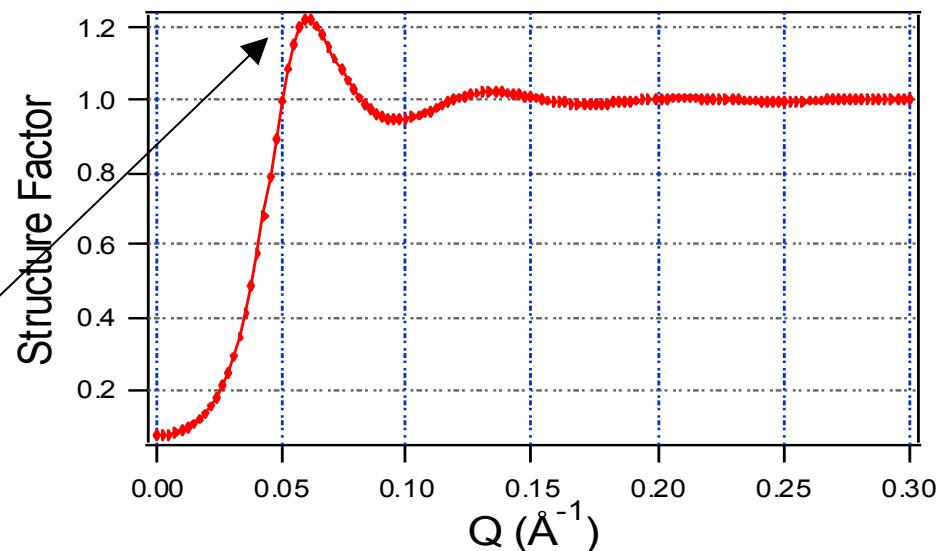
# Structure Factors

- for dilute solutions  $S(Q) = 1$
- particle interactions will affect the way they are distributed in space  $\Rightarrow$  changes scattering
- for charged spheres:



Average distance between nearest neighbours relatively constant  
= “correlation distance”

Position of first maximum related to correlation distance



# Concentration effects

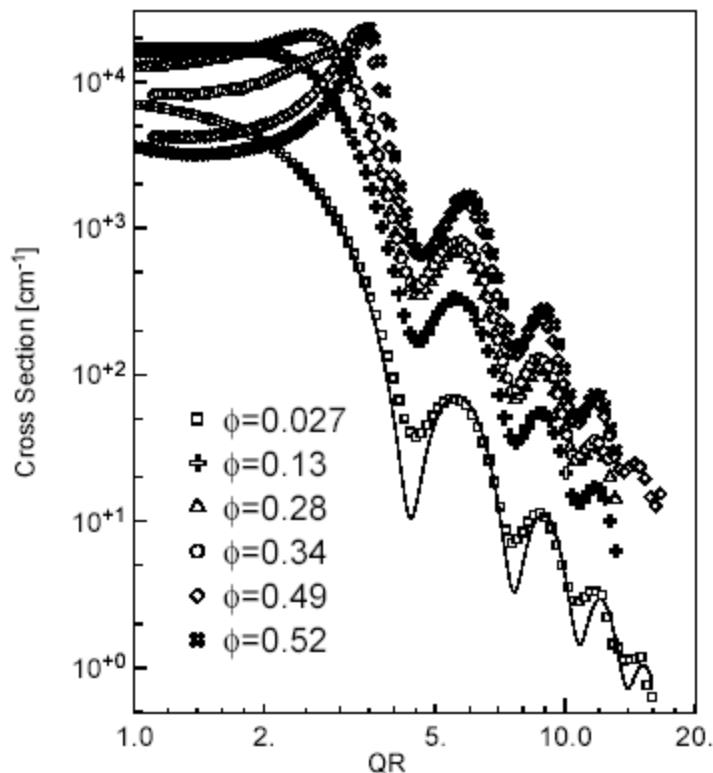


Figure 1: Cross-section for several different volume fractions of PS spheres in glycerol vs.  $QR$ .

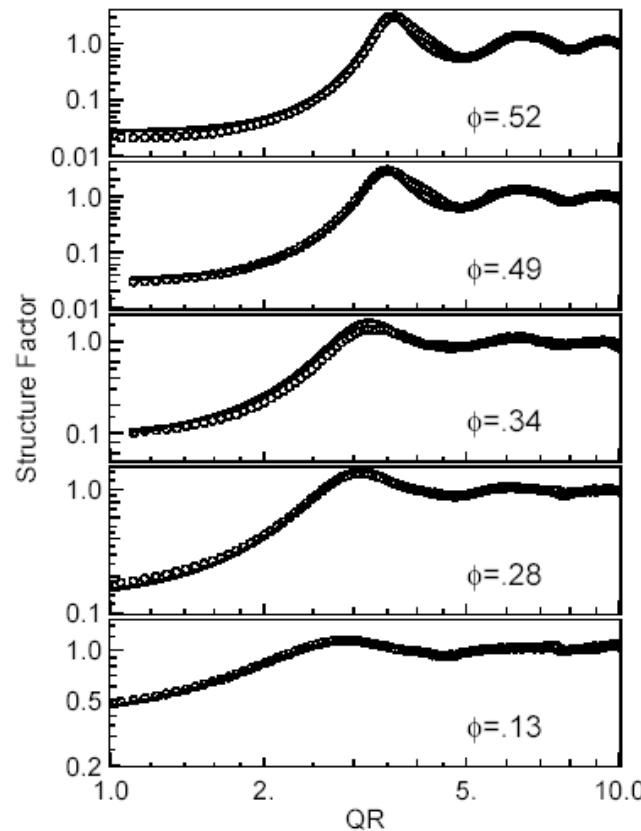


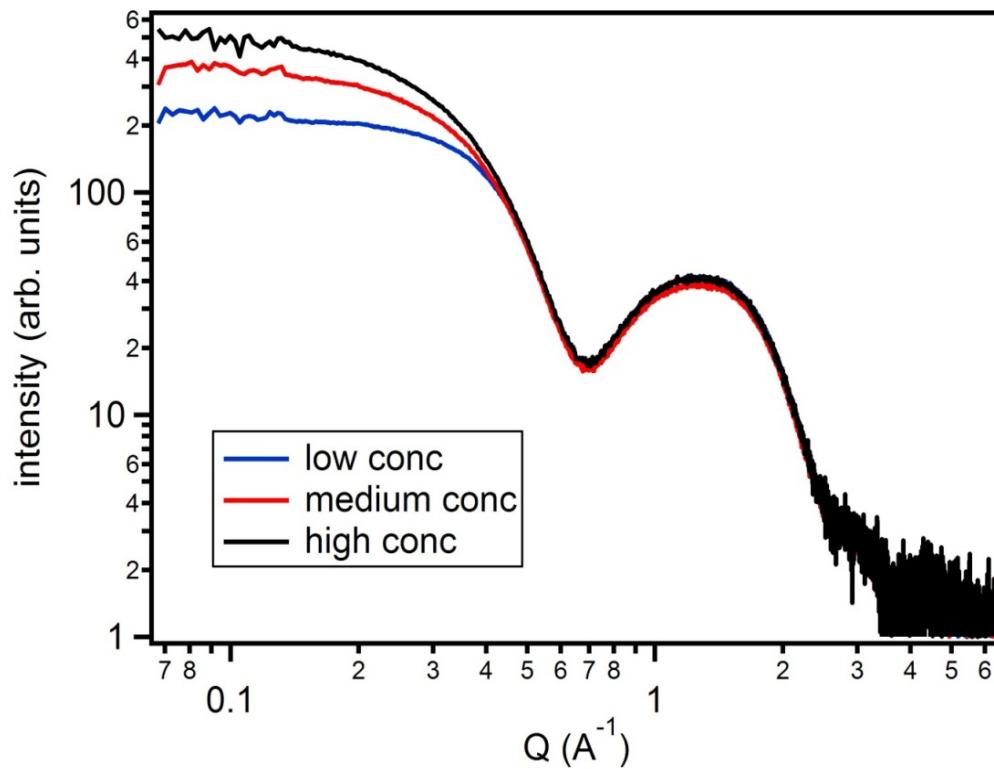
Figure 2: Measured and model structure factors,  $S(Q)$ , (circles and dashed lines, respectively) vs.  $QR$  for PS spheres in glycerol.

## Small Angle X-ray Scattering Study of a Hard-Sphere Suspension: Concentrated Polystyrene Latex Spheres in Glycerol

L. B. Lurio<sup>1</sup>, D. Lumma<sup>1</sup>, A. R. Sandy<sup>1</sup>, M. A. Borthwick<sup>1</sup>, P. Falus<sup>1</sup>, S. G. J. Mochrie<sup>1</sup>,  
J. F. Pelletier<sup>2</sup>, M. Sutton<sup>2</sup>, Lynne Regan<sup>3</sup>, A. Malik<sup>4</sup> and G. B. Stephenson<sup>4</sup>

# Combining F(Q) & S(Q)

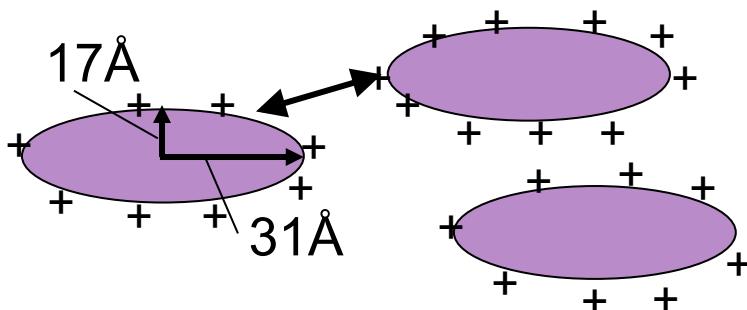
- In most cases when fitting will need to include both form and structure factor
- Can tell by taking concentration series
  - if shape of scattering doesn't change when sample is diluted then  $S(Q) = 1$



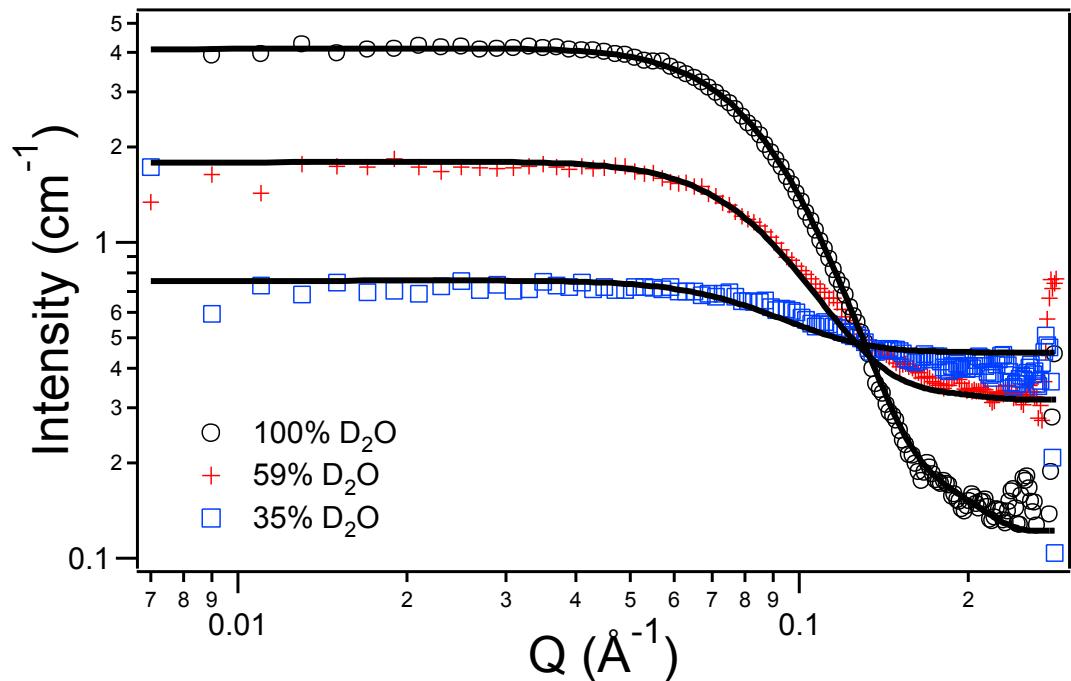
- Polymer-lipid discs
- Normalised for concentration

# Combining F(Q) & S(Q)

- Use computer programs to combine form factor and structure factor:



Brennan, Roser, Mann, Edler,  
*Chem. Mater.* **2002**, *14*, 4292



- Fit using ellipse + structure factor for charged objects which repel each other  $\Rightarrow$  many parameters!
- Use three contrasts to help pin down shape and size accurately

# Fourier Inversion Techniques

- Scattering from dilute, uniform, independent particles
- Assuming i) system is isotropic, then  $\langle e^{\uparrow - iQr} \rangle = \sin(Qr)/Qr$ 
  - ii) no long range order, so no correlations between two widely separated particles

$$I(Q) = \langle \downarrow e(Q) (\rho \downarrow p - \rho \downarrow s) \rangle / V \downarrow p \uparrow \int_0^\infty \gamma(r) \sin(Qr) / Qr 4\pi r dr$$

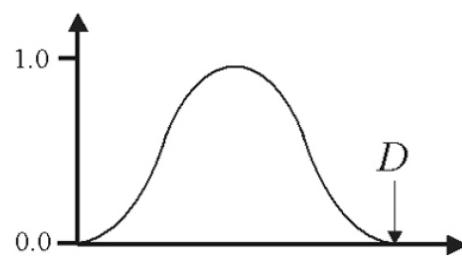
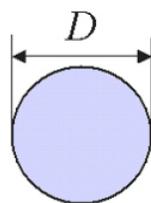
$\gamma(r)$  = correlation function

$P(r) = 4\pi r^2 \gamma(r)$  is the probability of finding two points in the particle separated by  $r$

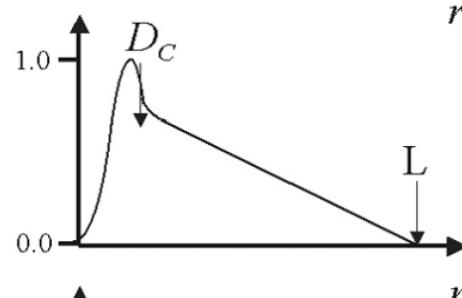
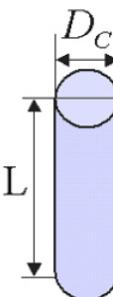
- If can measure  $I(Q)$  over big enough range can take inverse Fourier transform to find  $P(r)$ :  
$$P(r) = 4\pi r^2 \gamma(r) = \frac{2}{\pi} \int_0^\infty Q I(Q) \sin(Qr) dQ$$

# $P(r)$ for Simple Shapes

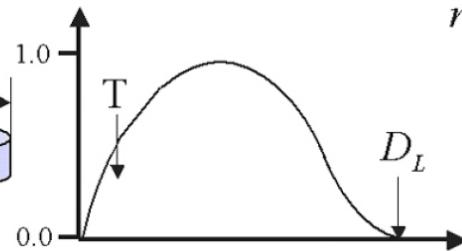
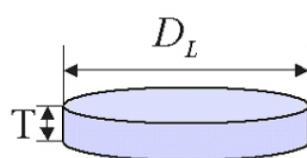
Globular:



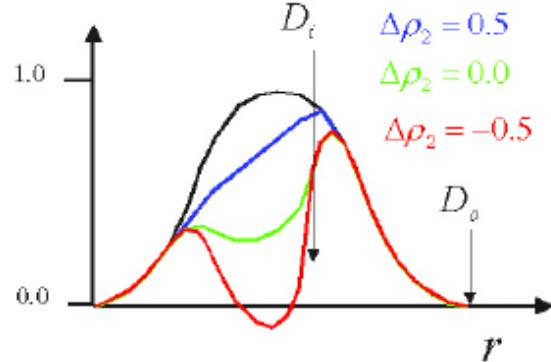
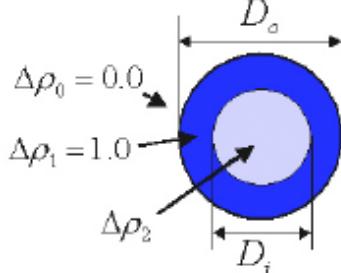
Cylindrical:



Lamellar:

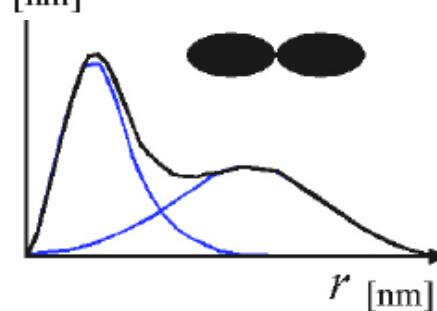
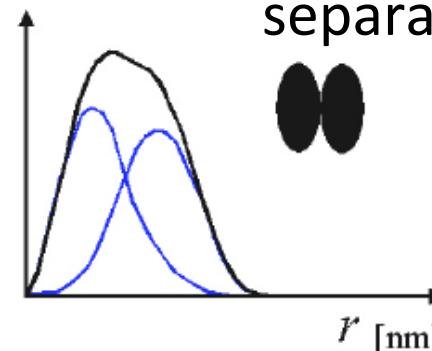


Core-Shell Sphere:



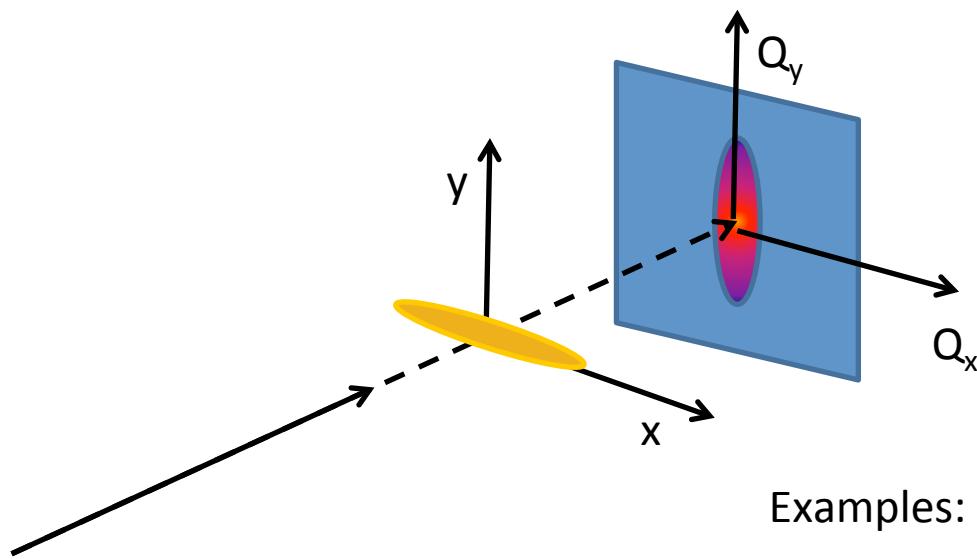
- Note:  $P(r)$  can be ambiguous if have polydisperse samples

Aggregates = sum of separate shapes



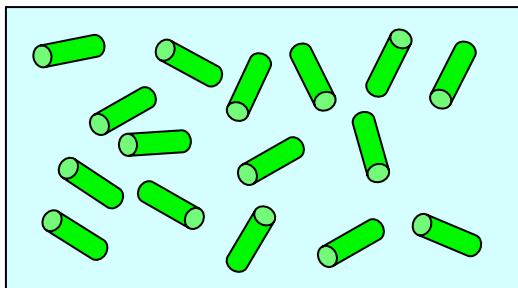
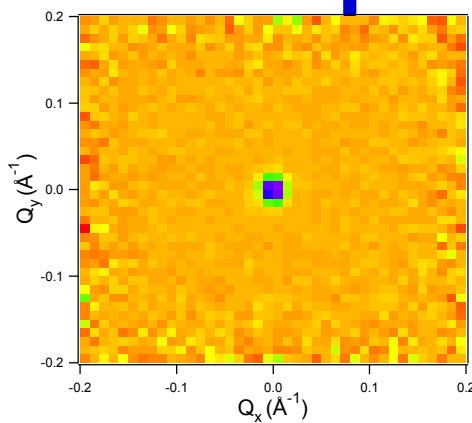
# Effects of Sample Alignment

- Scattering no longer circular
- Form areas of high intensity perpendicular to direction of alignment

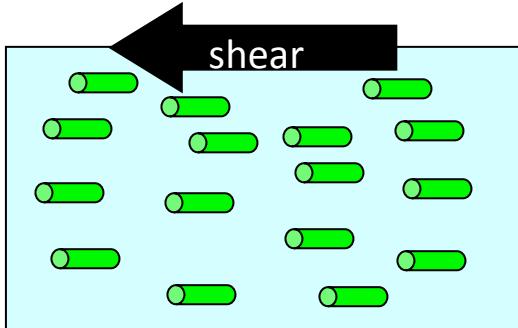
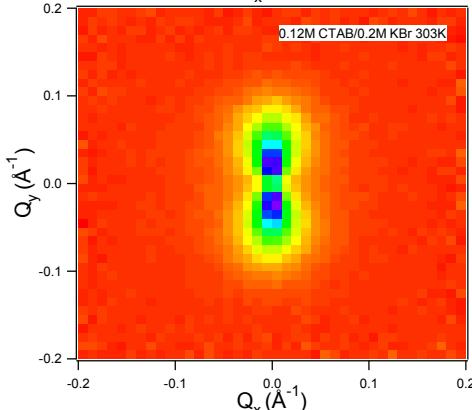


Examples: shear, flow  
magnetic alignment

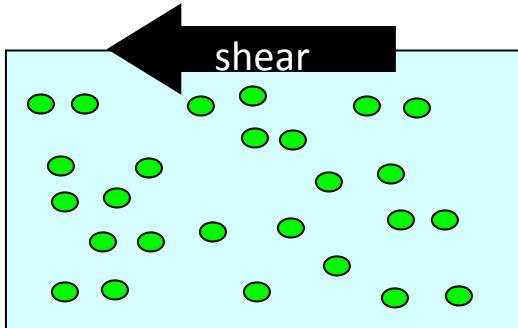
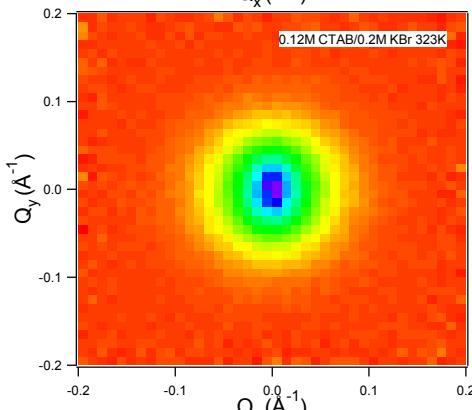
# Isotropic vs Nonisotropic Structures



No shear  
⇒ Isotropic solution



Shear  
⇒ aligned micelles



Shear + higher T  
⇒ isotropic again

# Free SANS Fitting Software

DANSE SANSView software

- Designed for fitting neutron data but can also be used (with care) for X-ray data
- Includes reflectivity analysis
- Available from: <http://danse.chem.utk.edu/sansview.html>

OR library of other available software at:

<http://www.small-angle.ac.uk/small-angle/Software.html>