Small Angle Neutron Scattering for Beginners

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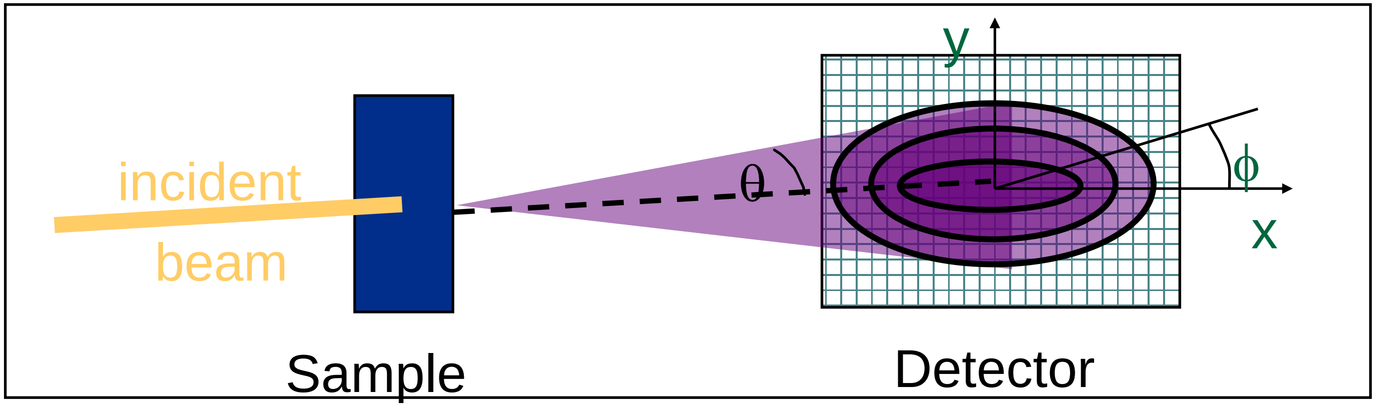
Outline

- What is Small Angle Scattering
- Applications for SAS
- Why Neutrons?
- SANS instrumentation
- SANS Data Reduction
- Today's System

What Is Small Angle Scattering?



I(Q) = A $\Delta\rho^2$ n V^2 P(Q) S(Q)

Calibration

Isotope labeling (6.34E10 cm^{-2} for D_2O and -0.56E10 cm^{-2} for H_2O)

$$R = \frac{2\pi}{Q} \quad ; \quad Q = \frac{4\pi}{\lambda} \cdot \sin\frac{\theta}{2}$$

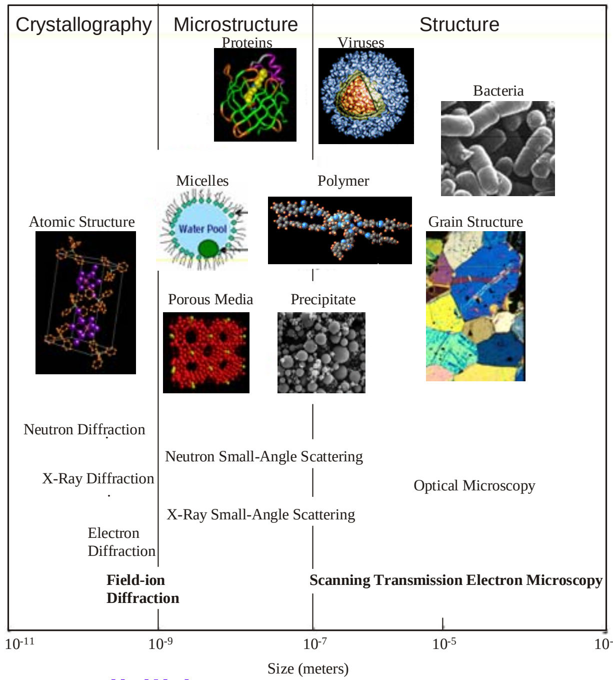
λ is neutron or x-ray wavelength

Probes length scales of ~ 0.5 - 100 nm

Using angles of θ = 0.5 to 5°

A powerful tool for studying disordered bulk materials (solid and solution phases)

Small Angle Scattering and Other Techniques



Contrast Variation Possibilities

$I(Q) = N V^2 (\rho_p - \rho_m)^2 e^{-Q^2 Rg^2/3}$

When $\rho_p = \rho_m$ Contrast match Situation
No Coherent Scattering

SANS: Vary by changing the Deuterium level

SAXS: Sensitivity increases with Z

Applications for SAS

- Typical Systems
  – Polymer Science
  – Metallic Alloys
  – Ceramics
  – Porous Materials
  – Structural Biology
  – Nanocomposites
  – Magnetic Materials
  – Colloidal Science
  – Fuel Science
- Typical Samples
  – Solutions,
  – Solids,
  – Powders,
  – Thin Films
- Typical Control Variables
  – Temperature
  – Concentration
  – Pressure
  – Magnetic field
  – Time
  – Shear

Examples of Self-Assembling Systems

- Detergent Molecules to Micelles- Aqueous Media
- Detergent Molecules to Reverse Micelles-Organic solvents
- Amphiphilic phosphotidylcholines into biomembranes
- Supramolecular Assembly of smaller proteins
- Protein and RNA folding to their native conformations
- Chemically engineered systems into supramolecular structures
- Amphiphilic block copolymers in Solvents
- Amyloids to fibrous structures

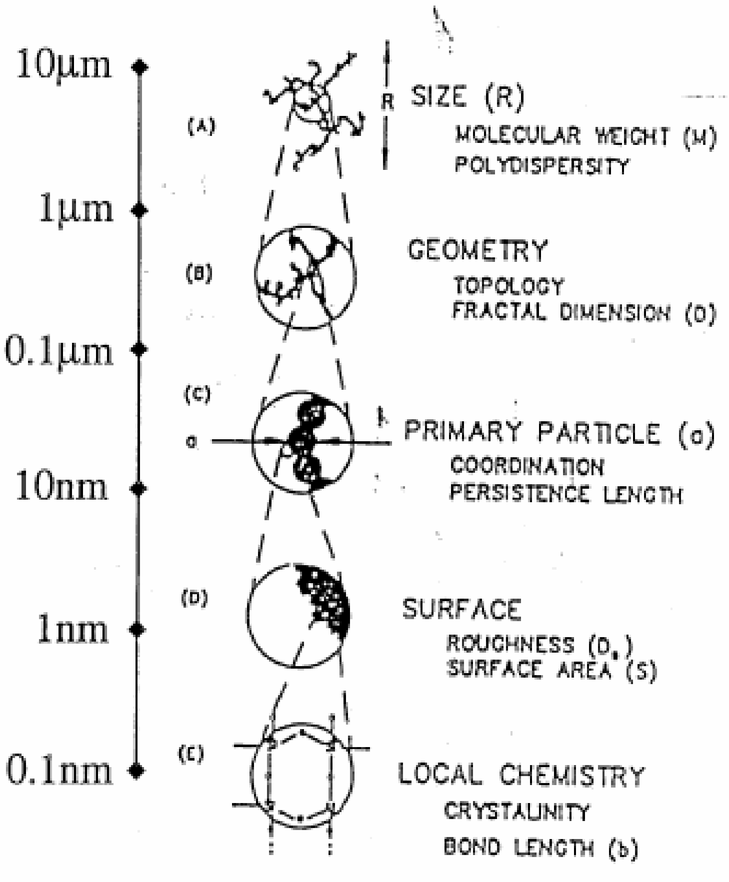
Information from SAS

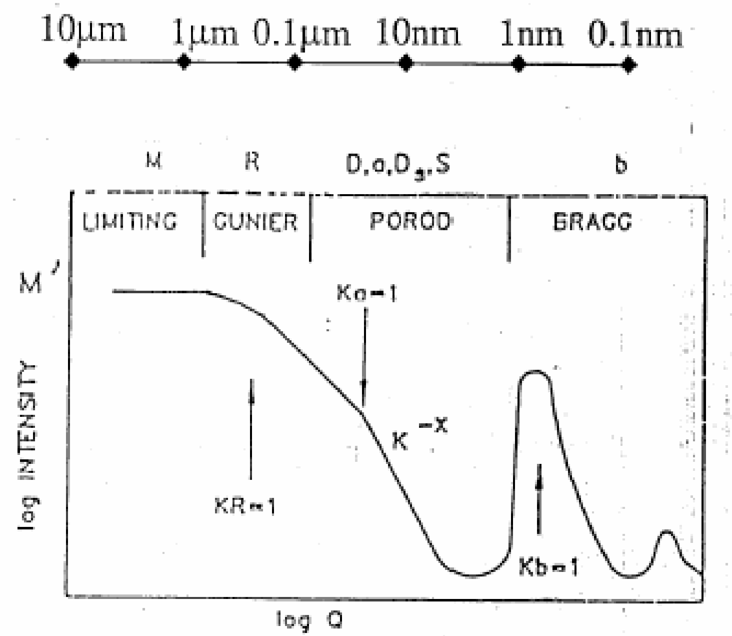
- Size
- Shape
- Molecular Weight
- Interaction distances
- Self Assembly
  - Phase transition
  - Thermodynamics
  - Reaction kinetics
- Crystallization
- Fractal Dimension

Why Neutrons?

- Low Energy (E=80meV for 1 Å neutrons vs. 12.42 keV for x-rays. No radiation damage.)
- High Penetration of cold neutrons – bulk samples
- Large difference in the scattering cross-section for the hydrogen and deuterium- contrast variation capability.
  - Solute or solvent can be deuterated to vary the contrast ($\rho H_2O=-0.56\times10^{10} cm^{-2}$, $\rho D_2O=6.334\times10^{10} cm^{-2}$)
  - Study of multicomponent systems through selective deuteration and contrast matching with H/D mixtures.

SANS from heirarchical structures

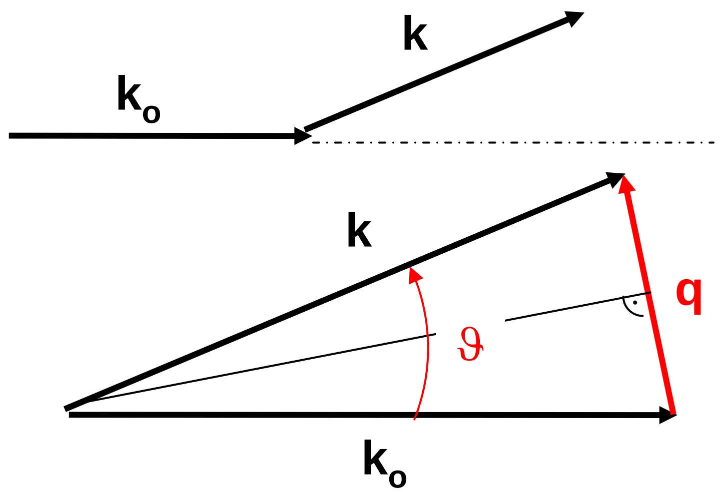




SANS is a structural technique that probes a wide array of length scales

Small-angle Scattering Basics

Wave vector **k**: $|\mathbf{k}| = k = 2\pi/\lambda$



$$\frac{1}{d} = \frac{2}{\lambda}\sin\left(\frac{\vartheta}{2}\right)$$

$$d = \frac{2\pi}{q}$$

$$q = 2k\sin\left(\frac{\vartheta}{2}\right) = \frac{4\pi}{\lambda}\sin\left(\frac{\vartheta}{2}\right)$$

q in nm^{-1} or $Å^{-1}$

Physics of scattering is no different than physics of diffraction

What SANS Measures

SANS probes differences in scattering length density within a sample

The measurement probes the time and ensemble average

Interference of wavelets from distribution of nuclei (= structure) adds up to “net scattering” amplitude (Fourier transform of structure).

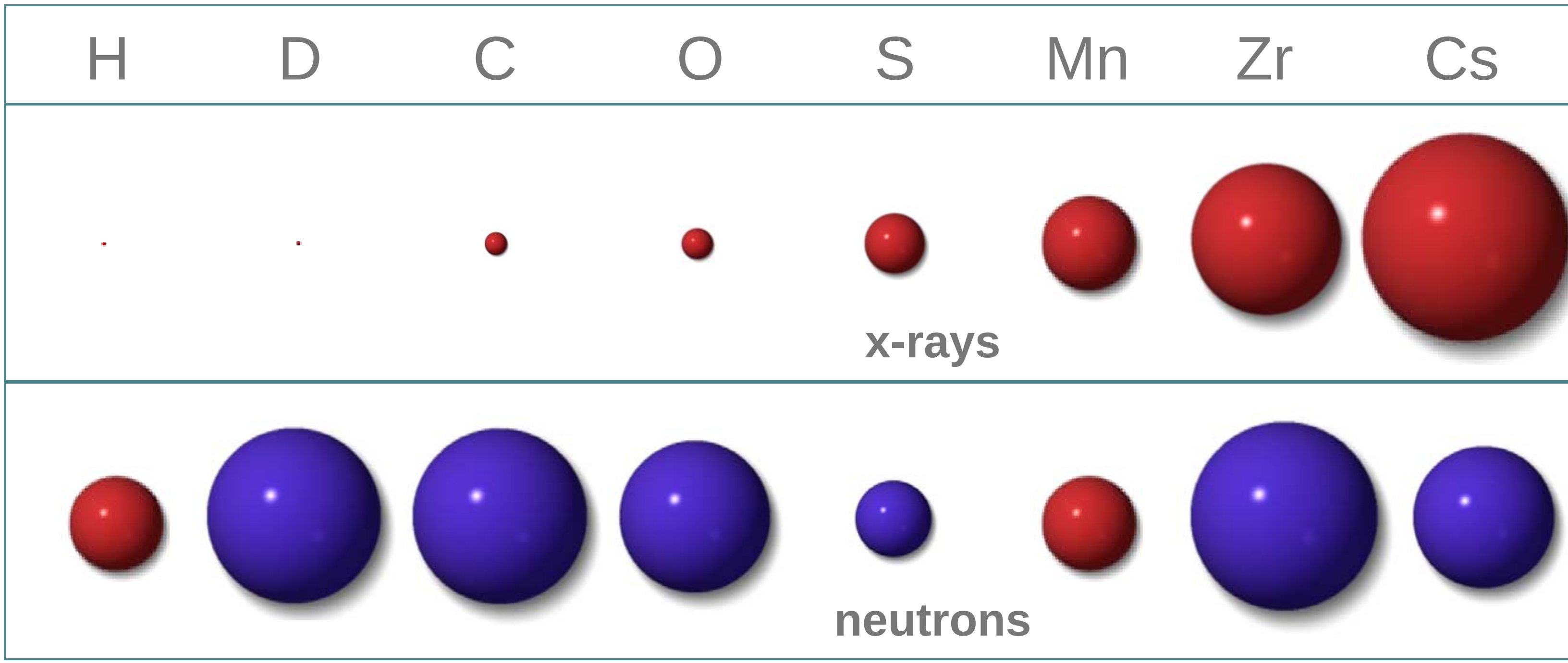
$$I(q) = \left| \left\langle \int_V (\rho(\vec{r}) - \rho_s) e^{-i\vec{q}\bullet\vec{r}} d^3 r \right\rangle \right|^2$$

$$|\vec{q}| = (4\pi \sin\theta) / \lambda$$

Neutron and x-ray scattering cross-sections

X-ray and neutron scattering are essentially the same, except...

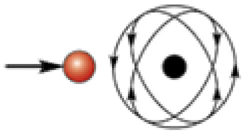
- X-rays scatter from electrons
- Neutrons scatter from nuclei



SANS Differences

Neutrons see nuclei and distinguish between isotopes

	C	N	O	H	D
b_{coh} (fm)	+6.65	+9.36	+5.81	-3.74	+6.67
σ_{coh} (barns)	5.56	11.03	4.23	1.76	5.59
σ_{inc} (barns)	0	0.49	0	80.27	2.05



Selective deuterium labeling in a sample is a powerful tool for looking at specific features within an intact system because it enables **contrast variation**.

It is the main reason why SANS is so powerful!

Scattering Length Densities

Neutron Scattering Length Densities

System	ρ ($10^{10}cm^{-2}$)	Eq. D_2O (%)
H_2O	-0.56	0
D_2O	6.338	100
C_6H_{12}	-0.277	
C_6D_{12}	6.678	
AOT	0.62	17
SiO_2	3.5	59
TiO_2	2.37	42
ZnS	2.09	38
ZnSe	3.1	53
Gold Coll.	3.59	66
PbS	2.30	41.5

Electron Densities (X-rays)

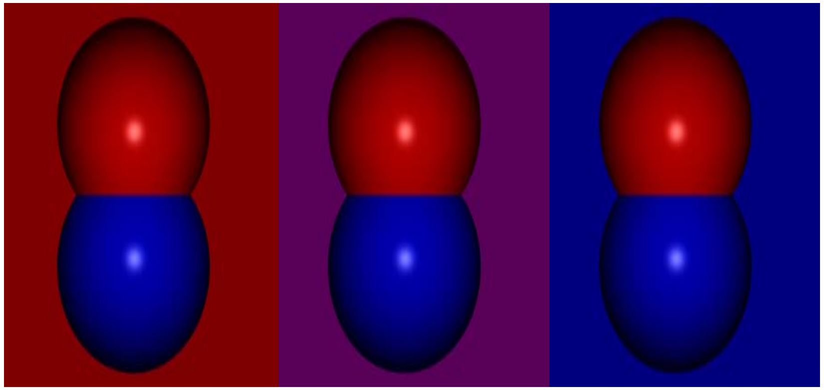
System	($10^{10}cm^{-2}$)
H_2O	9.36
C_6H_{12}	7.48
AOT	10.0
SiO_2	19.02
TiO_2	30.8
ZnS	31.68
ZnSe	40.42
CdS	36
CdSe	41.98

Contrast variation in SANS

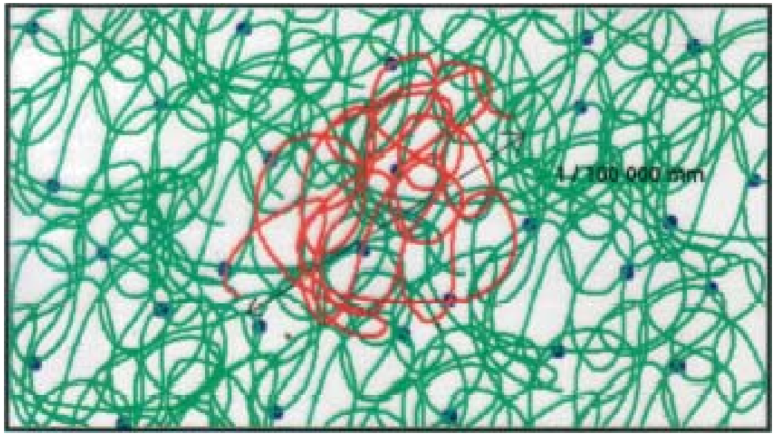
One way to think about contrast variation of selectively labeled complexes is that you are changing the "color" of isotopically-labeled material

Selective labeling to provide contrast has a smaller impact on the material than labeling used in other experimental methods.

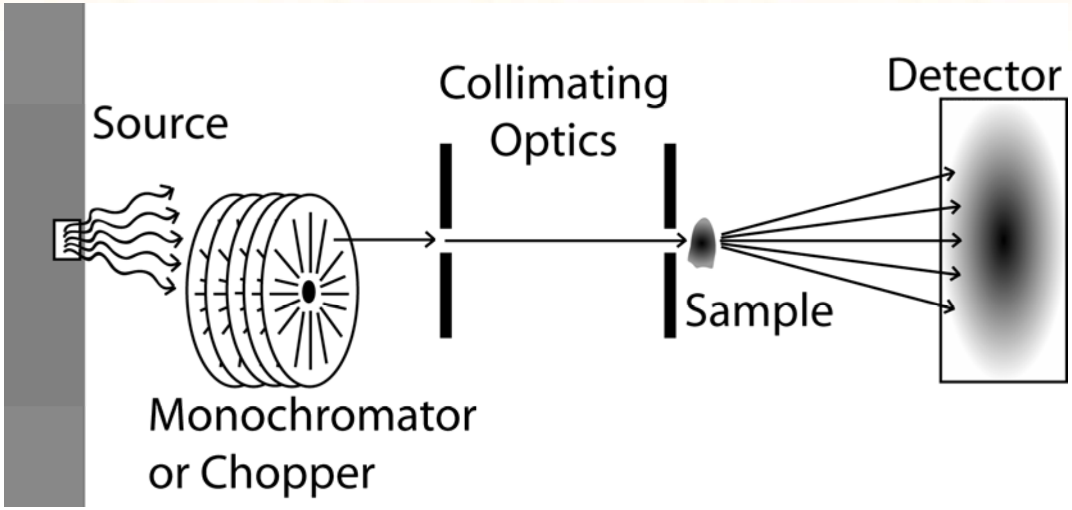
Highlight components of a multi-component system relative to the solvent and the other subunits



Visualize individual elements within a dense bulk material



SANS Instruments



Source: A spallation source or a reactor

Monochromator/Chopper: Defines wavelength(s)

Collimating Optics: Defines the angular divergence of the beam

Determines the maximum size probed

Detector: Collects the neutrons scattered by the sample

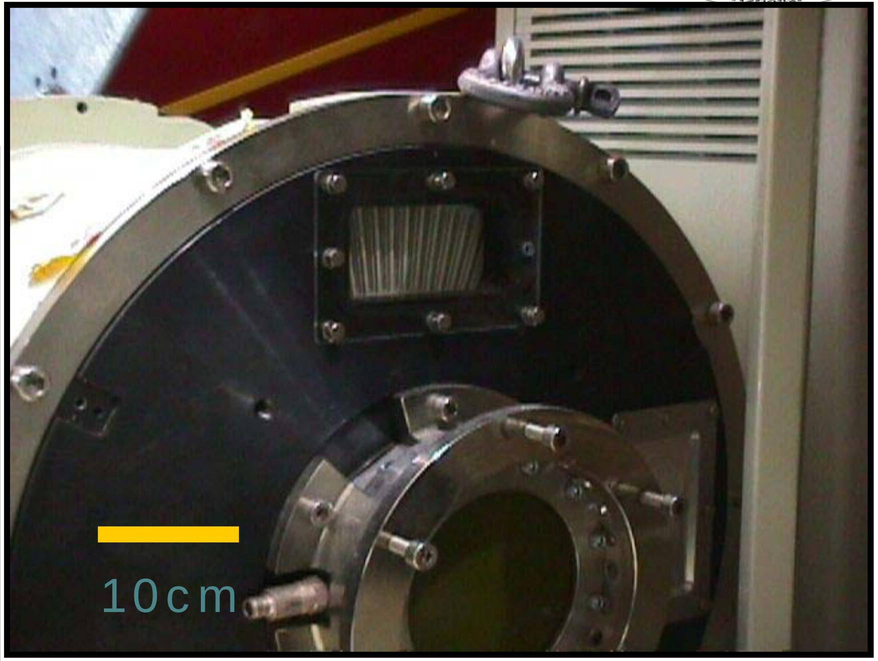
SNS and HFIR have large area detectors

SANS Instruments—steady-state vs. TOF

- **Steady-state**
  - **Higher flux**
  - **Larger Δλ at each wavelength**
  - **Only one wavelength per measurement**
    - **Smaller Q range**
    - **Multiple measurements required for wide Q range**
    - **Better resolution at low Q**
- **Pulsed-source TOF**
  - **Lower flux (as of now)**
  - **Smaller Δλ at each wavelength**
  - **Multiple wavelengths simultaneously**
    - **Larger Q range**
    - **Longer measurements required for signal-to-noise**
    - **Better at resolution at high Q**

Anatomy of a SANS

The velocity selector



	Cold	Thermal
T (K)	20	300
v (m/s)	574	2224
E (meV)	1.7	25.9
λ (Å)	6.89	1.78

De Broglie: $$\lambda = \frac{h}{p} = \frac{h}{mv}$$

SANS guide hall (HFIR)

SANS guide hall (HFIR)

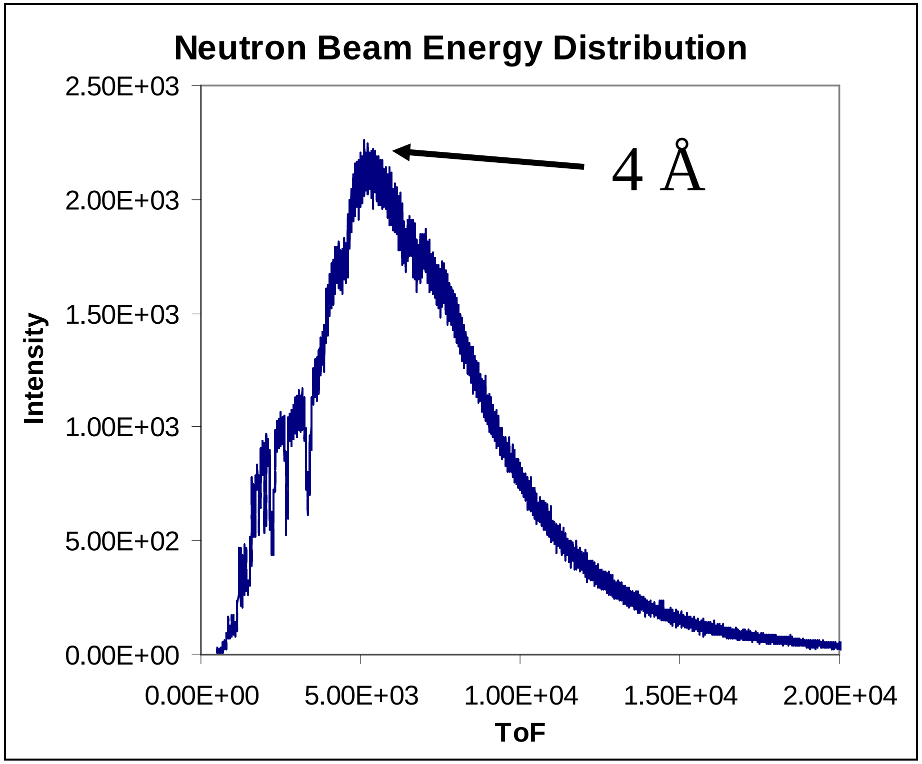
GG2 General-Purpose SANS Instrument Details

- **$1m^2$ Ordela 21000N area detector with 5.1 mm edge length square pixels**
- **Wavelength range from 4.2–18 Å (or longer)**
- **Initial flight path 1.8–17.4 m, Final flight path 1.1–19.4 m**
- **Full Q range <0.001–1 $Å^{-1}$, single-setting Q_{max}/Q_{min} 10–20 (detector offset)**
- **Flux on sample with 1.8 m initial flight path at 4.2 Å: 4×10^7 n/s/cm^2**
- **Flux on sample with 17.4 m initial flight path at 18 Å: 0.9×10^4 n/s/cm^2**
- **Sample environments: -5-100+°C sample changer, 5-300K cryostat, horizontal and vertical translation, Magnetic fields, more…**

Moderator

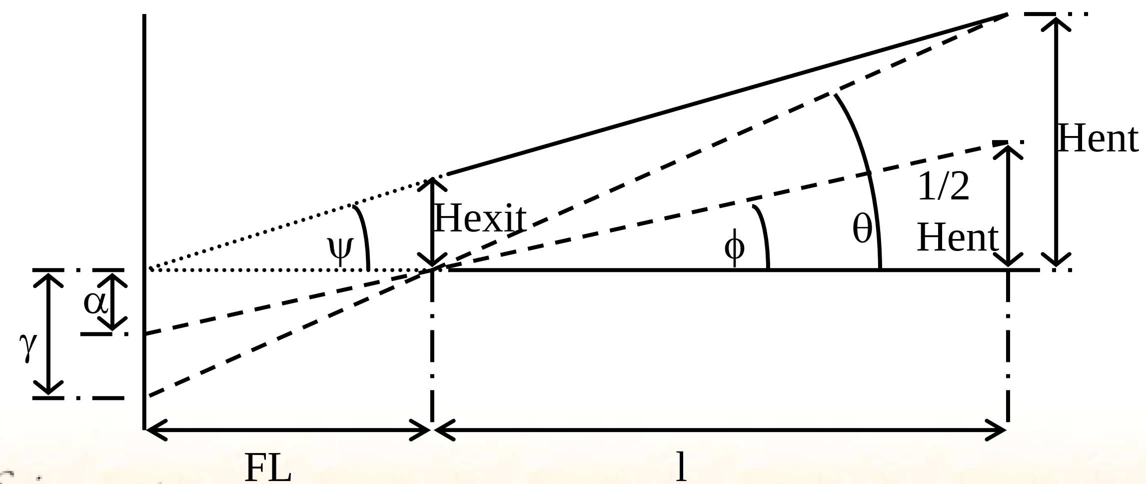
- **Thermalizes neutrons**
  - **Inelastic collisions**
  - **Highly protonated**
  - **~30K**

- **Creates energy spectrum**



Collimator

- **Focuses Neutrons**
- **Defines center**
- **Critical component of resolution**
- **Balance resolution with flux**



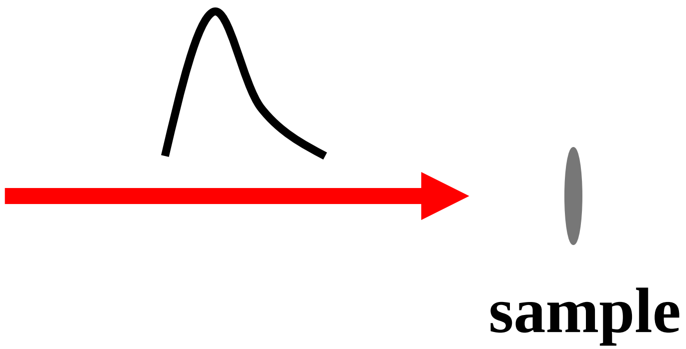
Detector

- He^3 detector
  - Triton
  - Proton
- Xe/CO_2
  - Multiplication
  - Quench
- Two wires
  - Start stop time-position
- Highly efficient
  - Neutron efficient
  - Gamma insensitve
- Position Sensitive
- Time encoding (for ToF)

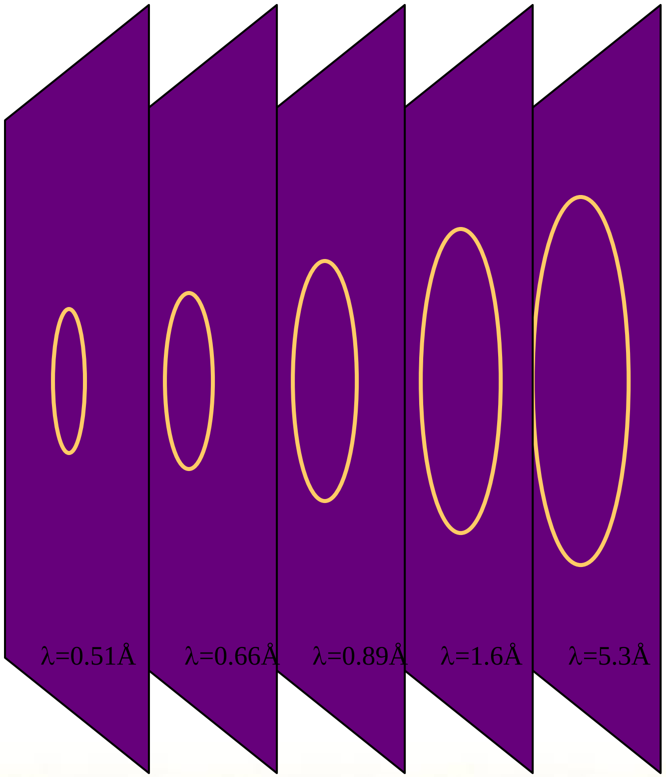
Combining data from different settings

Bragg diffraction: nλ=2dsinθ

Fastest n, shortest λ



Ring radius increases with both wavelength and distance



θ Range, Q_{MIN} and Q_{MAX}, and Binning

$$Q_{MIN,MAX} = \frac{4\pi}{\lambda_{MAX,MIN}} \sin\theta_{MIN.MAX}$$

$$0.15^{\circ} \leq 2\theta \leq 60^{\circ}$$

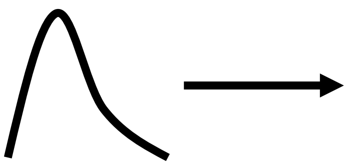
$$0.001\mathring{A}^{-1} \leq Q \leq 1.3\mathring{A}^{-1}$$

λ large

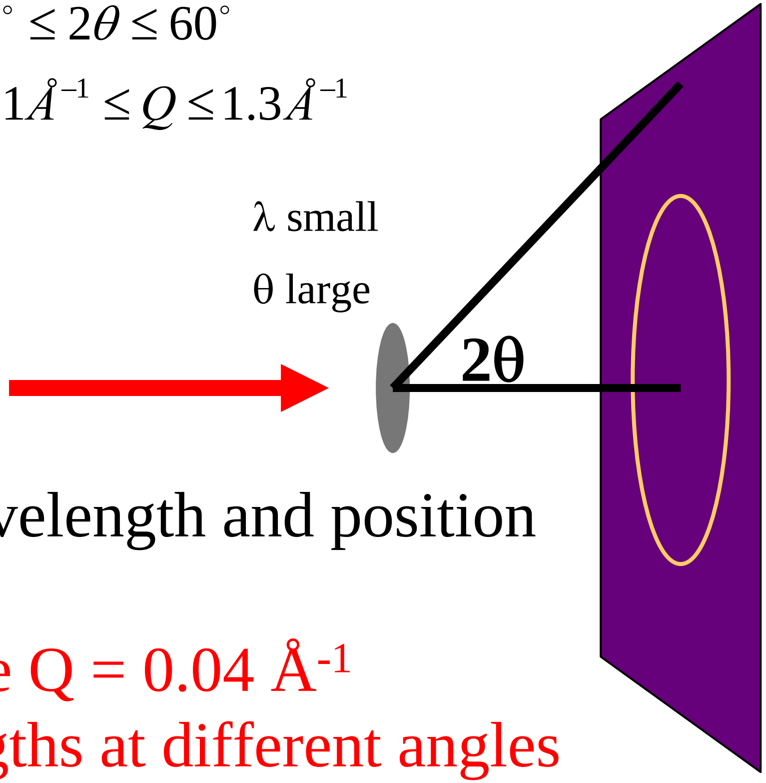
θ small

λ small

θ large



λ,θ



Each sample, BIN data according to wavelength and position

λ = 2 Å scattered at 2θ = 0.73º

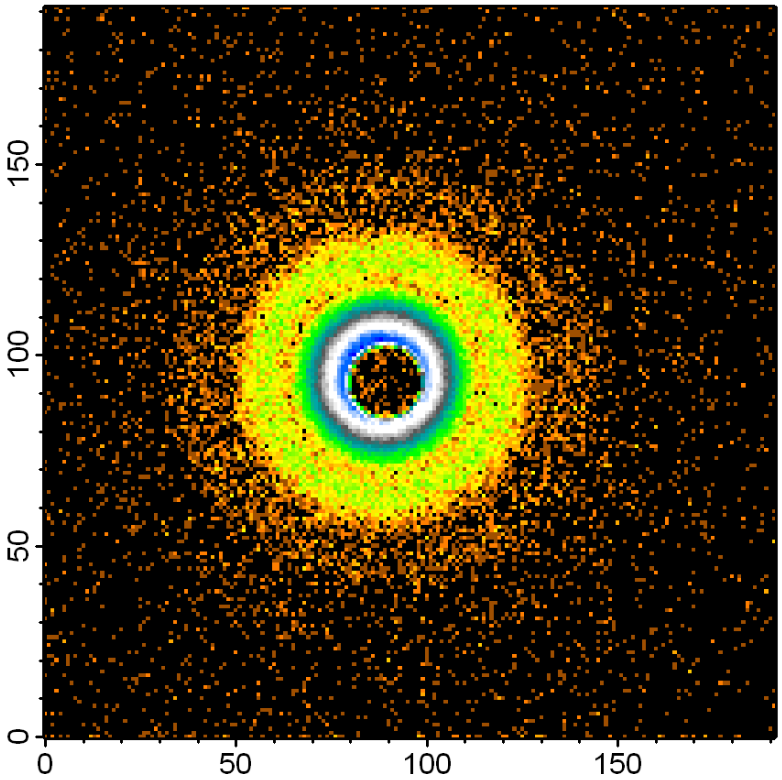
λ = 8 Å scattered at 2θ = 2.92º both give Q = 0.04 $\mathring{A}^{-1}$

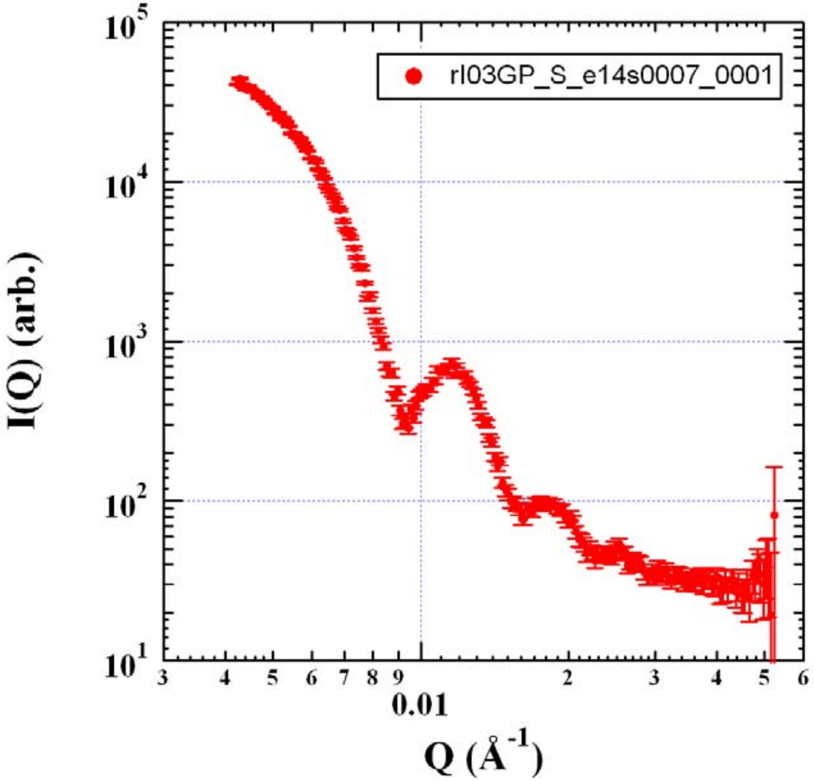
Neutrons scattered at different wavelengths at different angles can contribute to same Q.

Number of neutron S(x,y,t) to Absolute Intensity I(Q)

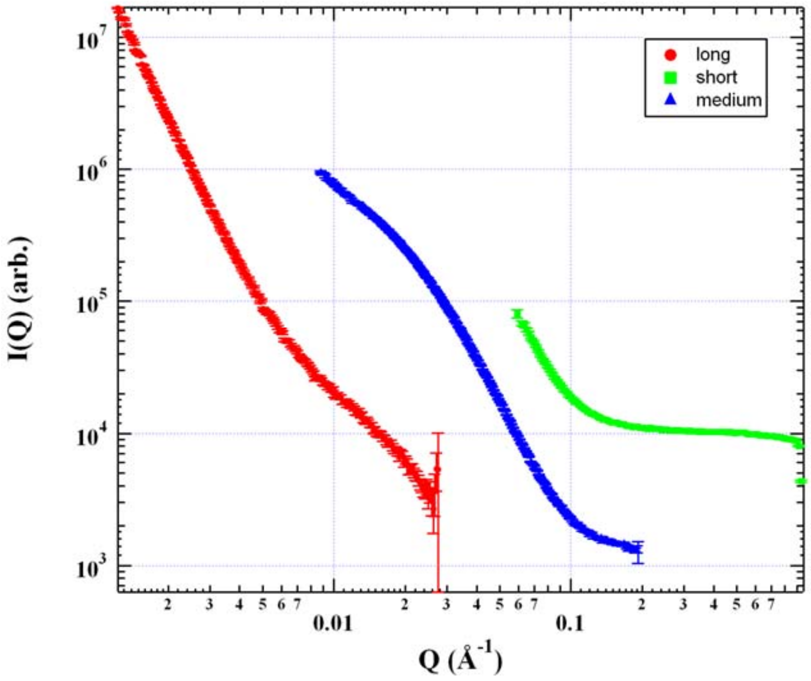
- **Detector efficiency and sensitivity**
- **Background (sample and non-sample)**
- **Convert intensity to absolute scale (units = cm-1)**
- **For each sample you will need**
  - **Transmission data for each wavelength (attenuated direct beam or beam spreader)**
  - **Scattering data for each wavelength and instrument geometry**
- **For each wavelength and geometry you will need**
  - **Empty beam transmission data**
  - **Data mask**
- **For the whole experiment you will need**
  - **Dark current data**
  - **Detector pixel efficiency**

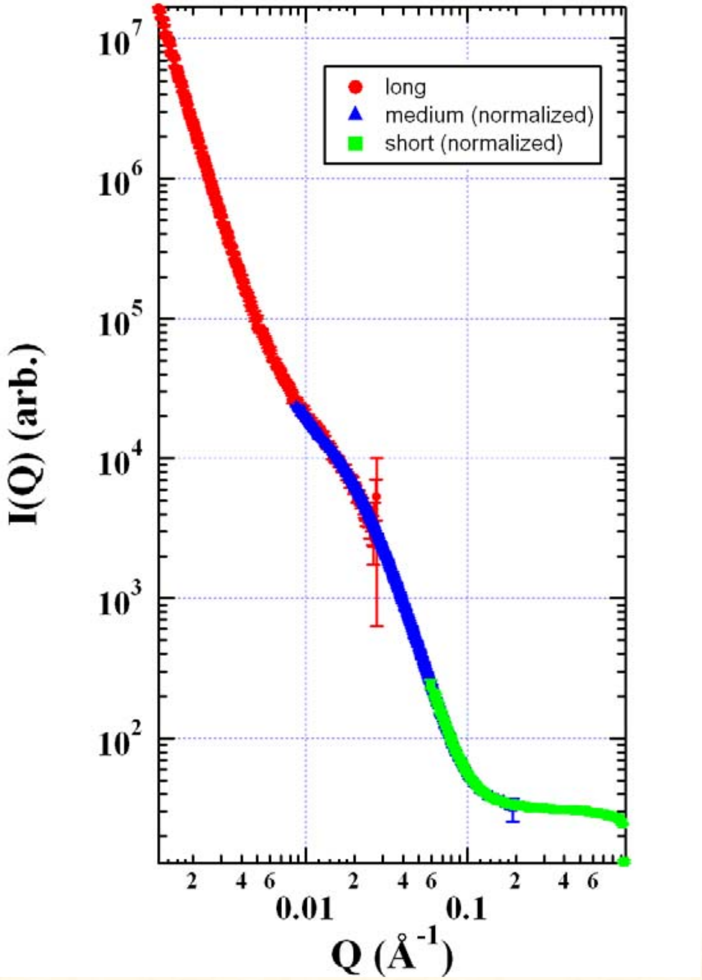
Detector Counts To I(Q) Function





Combined data from different settings





Absolute Scattering Intensity (as a function of Q)

Bates Polymer: $I(0)_{std} = 2800(30)\mathrm{cm}^{-1}$

$$I(Q)_{sample} = S(Q)_{sample} \frac{d_{standard}}{d_{sample}} \frac{I(0)_{standard}}{S(0)_{standard}}$$

RECAP

$$\mathbf{S(x,y,t)} \longrightarrow \mathbf{S(\theta,\lambda)} \xrightarrow[\text{2D-1D}]{} \mathbf{S(Q)} \xrightarrow[\text{Standard sample}]{} \mathbf{I(Q)}$$

Conclusions

- **In practice, much of the data reduction is in the form of a black box**
- **However, you should understand how it works**
- **It usually does.**