

SANS Experimental Methods

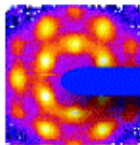
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NCNR Summer School

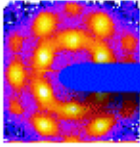
Neutron Small Angle Scattering and
Reflectometry from Submicron Structures

June 5 - 9 2000

Outline

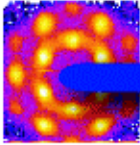


- Procedure of SANS Measurements
 - From the initial planning
to SANS data in absolute scale
 - Sample preparation
 - What to measure
- Further consideration
 - Effects of Q-resolution
 - Multiple Scattering
- Summary



Procedure of SANS measurements

- 1) Initial planning
- 2) Sample preparation
- 3) Setup proper SANS configurations
- 4) Sample scattering
- 5) Additional measurements for correction
- 6) Absolute scaling

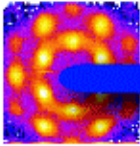


Initial Planning

- What information do I want to measure ?

$$\left(\frac{d\Sigma(Q)}{d\Omega} \right)_{sample}$$

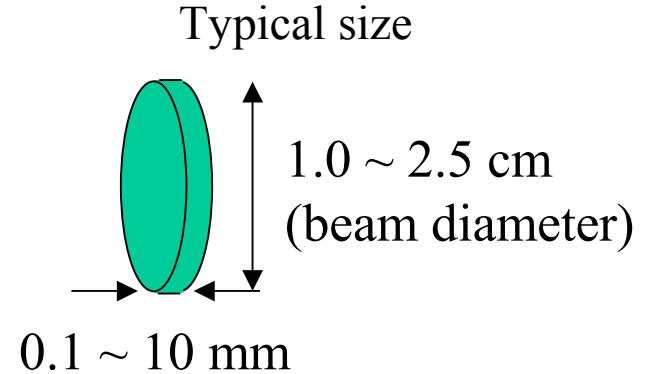
- Is it accessible with SANS ?
 - Length scale of interest ($\sim 10 \text{ \AA} - \sim 5000 \text{ \AA}$)
 - Sample size
 - Neutron scattering contrast
 - Sample environment
(Temperature, Pressure, Magnetic field and etc.)
 - How long would it take ?
- Use NCNR Web based tools
- Consultation with SANS staff members



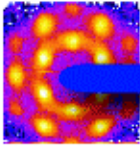
Sample Preparation

- SANS can handle various forms of samples.
Liquid, Gel, and Solid

- How much sample do I need ?
 - Depends on sample
 - Neutron transmission

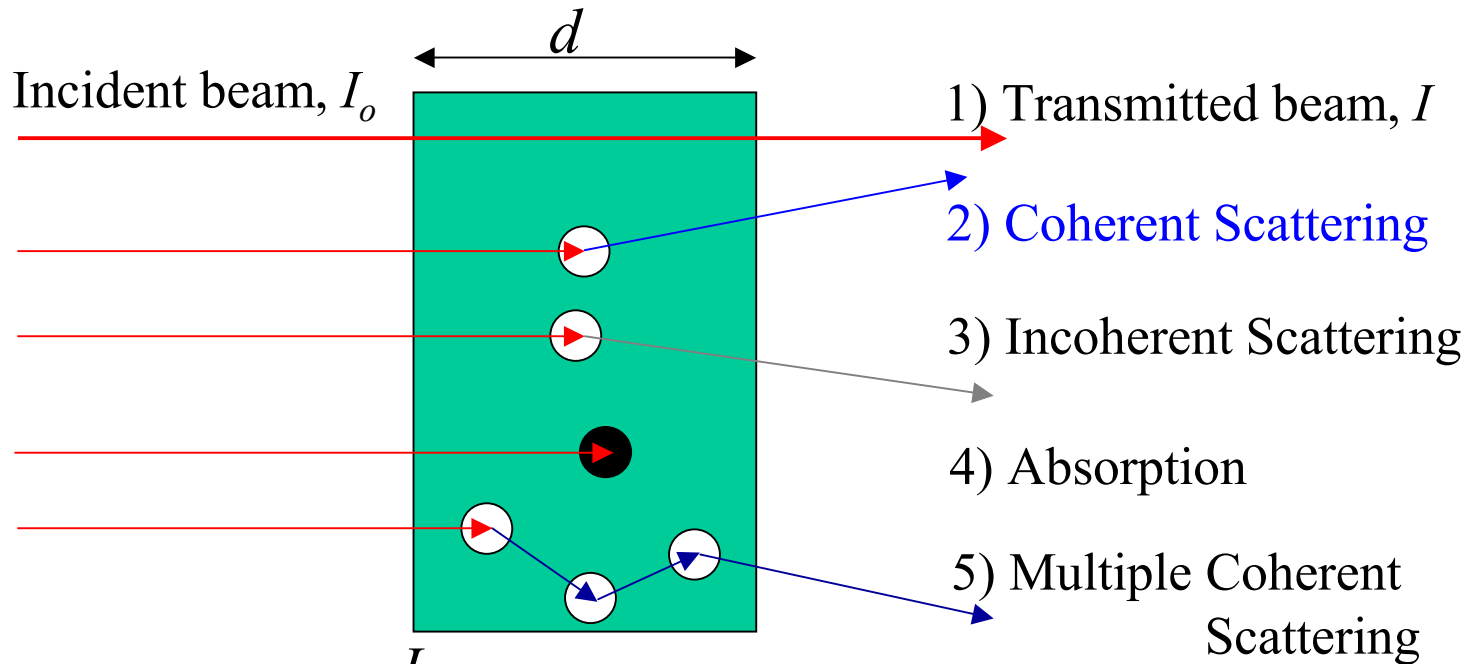


- Prepare proper neutron contrast
- Standard SANS sample cell
- Custom made sample cell



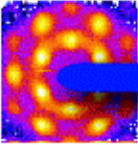
What Should Sample Thickness Be?

- To decide the sample thickness, we need to understand what is happening in the sample

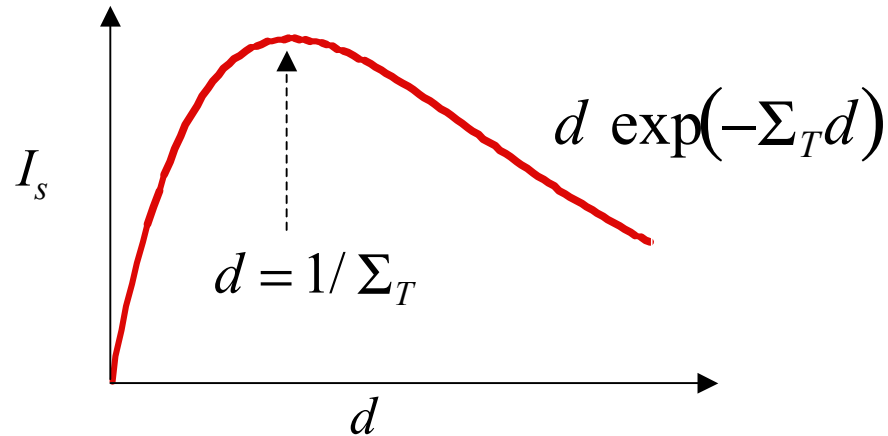


- Transmission $T = \frac{I}{I_o} = \exp(-\Sigma_T d)$ where $\Sigma_T = \Sigma_{coh} + \Sigma_{inc} + \Sigma_{abs}$

- Scattered Intensity $I_s \propto d T \left(\frac{d\Sigma_{coh}}{d\Omega} \right) \propto d \exp(-\Sigma_T d)$



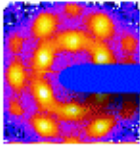
Optimal Sample Thickness



- I_s is maximum when $d = 1 / \Sigma_T$

$$\begin{aligned} \longrightarrow T &= \exp(-\Sigma_T d) \\ &= 1/e = 37\% \end{aligned}$$

- When $\Sigma_T \approx \Sigma_{coh}$ $\longrightarrow d = 1 / \Sigma_T$ is too large.
will have multiple scattering problem
 \longrightarrow want $T \geq 90\%$
- When $\Sigma_{coh} \ll \Sigma_T \approx \Sigma_{inc} + \Sigma_{abs}$ $\longrightarrow d = 1 / \Sigma_T$, $T = 37\%$



Examples of Sample Thickness

- When $\lambda = 5\text{\AA}$

$$\Sigma_T \approx \Sigma_{coh}$$

(want $T > 90\%$)

$$\Sigma_{coh} \ll \Sigma_T \approx \Sigma_{abs} + \Sigma_{inc}$$

($T = 1/e = 37\%$ is optimal)

Silica 0.5 mm, $T = 96\%$

1 mm, $T = 92\%*$

3 mm, $T = 78\%$

$$\sigma_{coh} = \mathbf{10.62 \text{ barns}}$$

$$\sigma_{inc} = 0.005 \text{ barns}$$

$$\sigma_{abs} = 0.17 \text{ barns}$$

H₂O 1 mm, $T = 52\%$

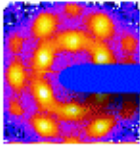
1.5 mm, $T = 38\%*$

3 mm, $T = 14\%$

$$\sigma_{coh} = 7.75 \text{ barns}$$

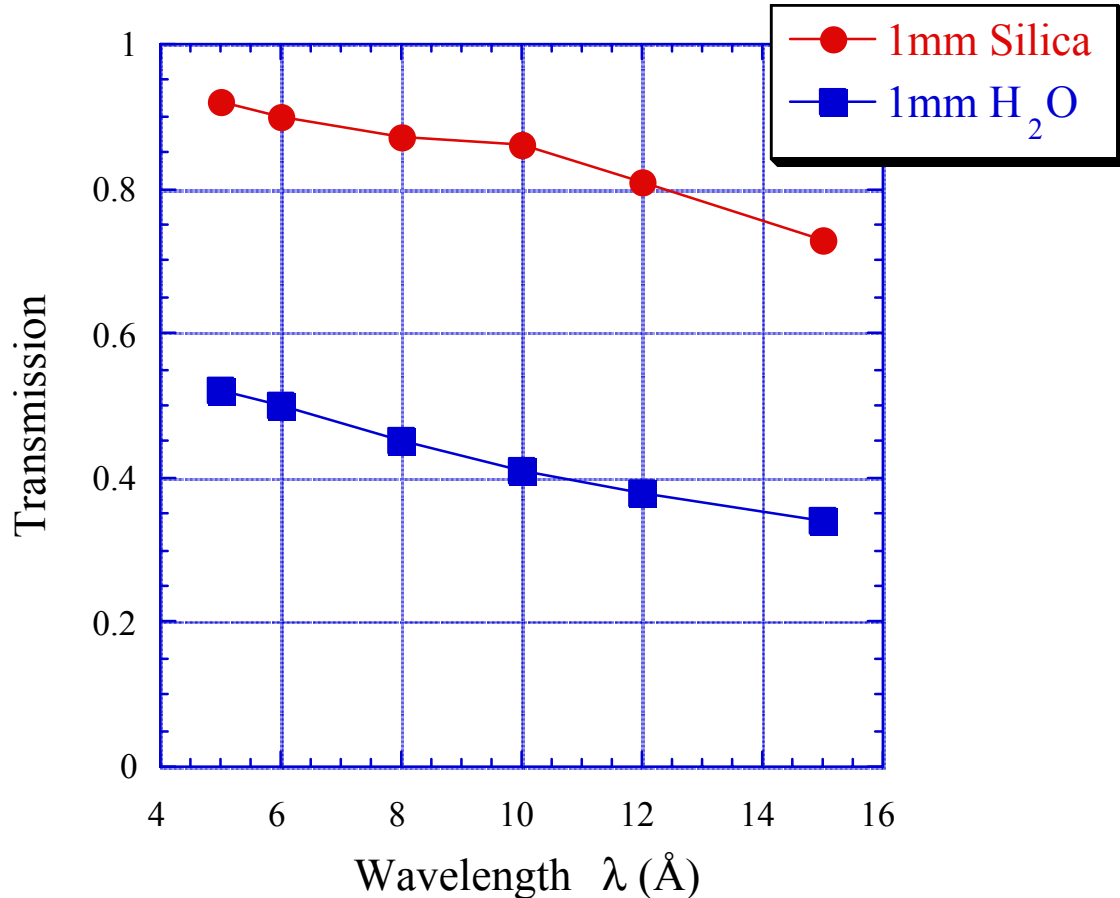
$$\sigma_{inc} = \mathbf{164 \text{ barns}}$$

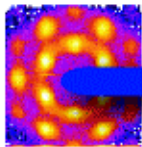
$$\sigma_{abs} = 0.66 \text{ barns}$$



Wavelength Dependence of Transmission

- Neutron cross-section depends on neutron wavelength λ .
- Total cross-section increases as the neutron wavelength increases.





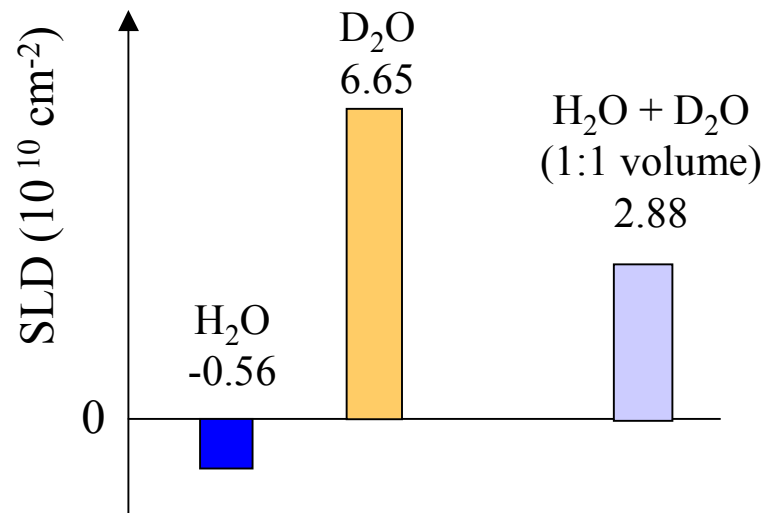
Calculation of Scattering Length Density

$$\rho_{SLD} = \frac{\sum_i^n b_i}{\bar{V}} = N_A \left(\frac{\rho_{mass}}{M_w} \right) \left(\sum_i b_i \right)_{molecule}$$

N_A = Avogadro's number = 6×10^{23}

M_w = molecular weight

b_i = bound coherent scattering length of atom i



• H₂O

$$\begin{aligned} \rho_{SLD, H_2O} &= (6 \times 10^{23} / \text{mol}) \left(\frac{1.0 \text{ g} / \text{cm}^3}{18 \text{ g} / \text{mol}} \right) (2 \times b_H + b_O) \\ &= \underline{-0.56 \times 10^{10} \text{ cm}^{-2}} \end{aligned}$$

• D₂O

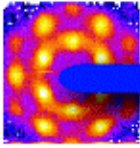
$$\begin{aligned} \rho_{SLD, D_2O} &= (6 \times 10^{23} / \text{mol}) \left(\frac{1.1 \text{ g} / \text{cm}^3}{20 \text{ g} / \text{mol}} \right) (2 \times b_D + b_O) \\ &= \underline{6.32 \times 10^{10} \text{ cm}^{-2}} \end{aligned}$$

• H₂O + D₂O mixture (1:1 volume)

$$\begin{aligned} \rho_{SLD, Mixture} &= x_{H_2O} \times \rho_{SLD, H_2O} + (1 - x_{H_2O}) \times \rho_{SLD, D_2O} \\ &= 0.5 \times (-0.56 \times 10^{10} \text{ cm}^{-2}) + 0.5 \times (6.32 \times 10^{10} \text{ cm}^{-2}) \\ &= \underline{2.88 \times 10^{10} \text{ cm}^{-2}} \end{aligned}$$

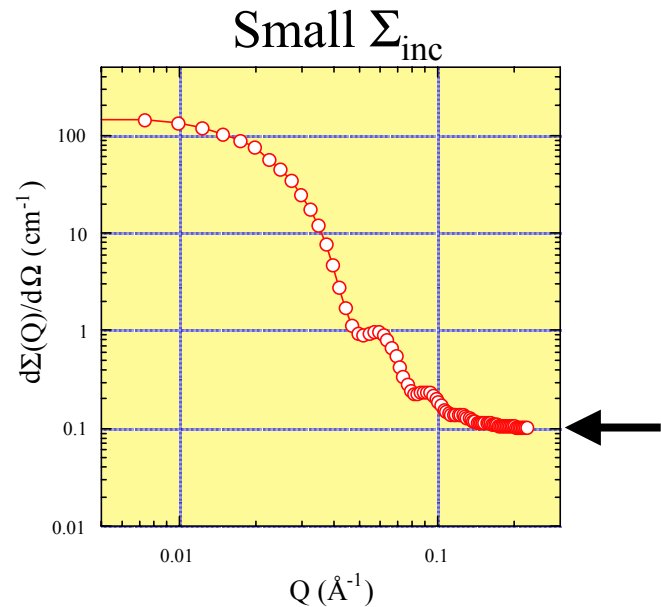
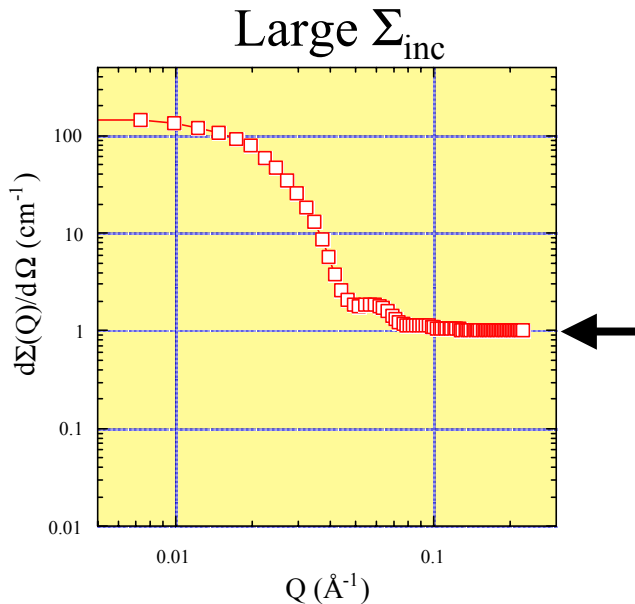
- bound coherent scattering length (10^{-13} cm^{-1})

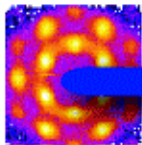
$$b_H = -3.74 \quad b_D = 6.67 \quad b_O = 5.80$$



Coherent and Incoherent Scattering

- Coherent scattering contains the structural information of sample
- Incoherent scattering is flat background.
- Examples
 - H_2O or Hydrocarbons $\sim 1 \text{ cm}^{-1} \text{ ster}^{-1}$
 - D_2O or Deuterated sample $\sim 0.1 \text{ cm}^{-1} \text{ ster}^{-1}$
 - SiO_2 (amorphous) $\sim 0.02 \text{ cm}^{-1} \text{ ster}^{-1}$
- Large incoherent scattering reduce the dynamic range of measurement.
- **Use deuterated solvent whenever it is possible.**



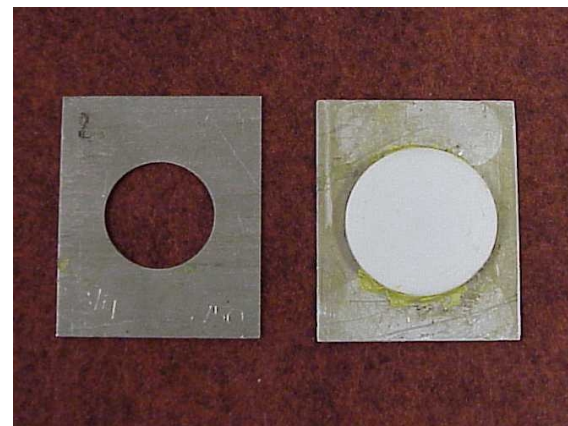
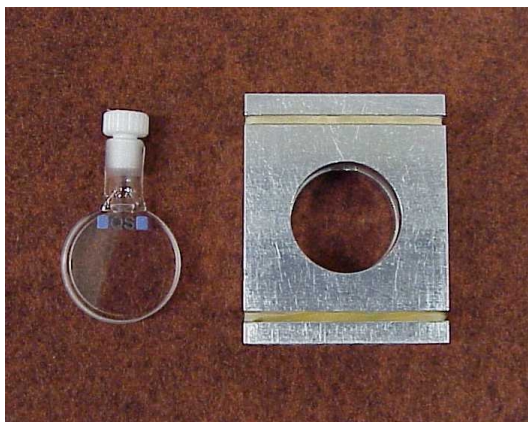


SANS Sample Holders

Liquid

Gel or Polymer Melt

Solid



Path length (Volume)

1 mm (0.3 ml)

2 mm (0.6 ml)

5 mm (1.5 ml)

Diameter = 2.0 cm

Path length (Volume)

1 mm (0.3 ml)

2 mm (0.6 ml)

4 mm (1.2 ml)

Diameter = 2.0 cm

Glue on a Cd mask

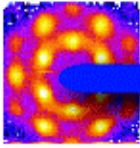
Size mountable on a standard

Sample changer

width < 3.5 cm

height < 5 cm

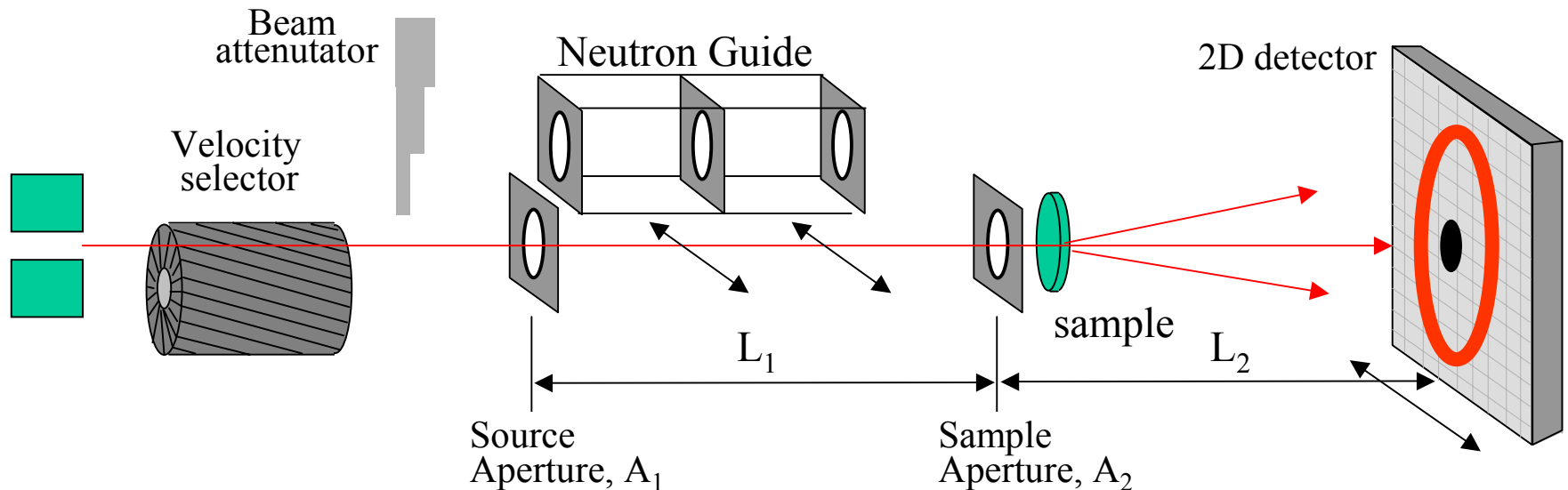
thickness < 2cm

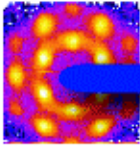


Set up SANS Configuration

- Q-range cover : Q_{\min} to Q_{\max} (1, 2, or 3 configuration)
- Q-resolution needed
- Beam intensity available, $I \propto Q_{\min}^4$
 - neutron wavelength (5 - 20Å)
 - wavelength spread ($\Delta\lambda/\lambda=10-30\%$)
 - source to sample distance (L_1 , #of guide, 4-15m)
 - sample to detector distance (L_2 , 1-15m)
 - detector offset (0 - 25 cm)
 - sample (1- 2.5cm) and source(1.5-5cm) aperture sizes
 - position of beam attenuator during transmission measurement (0-8)

Use SASCAL or
Web Based Tools

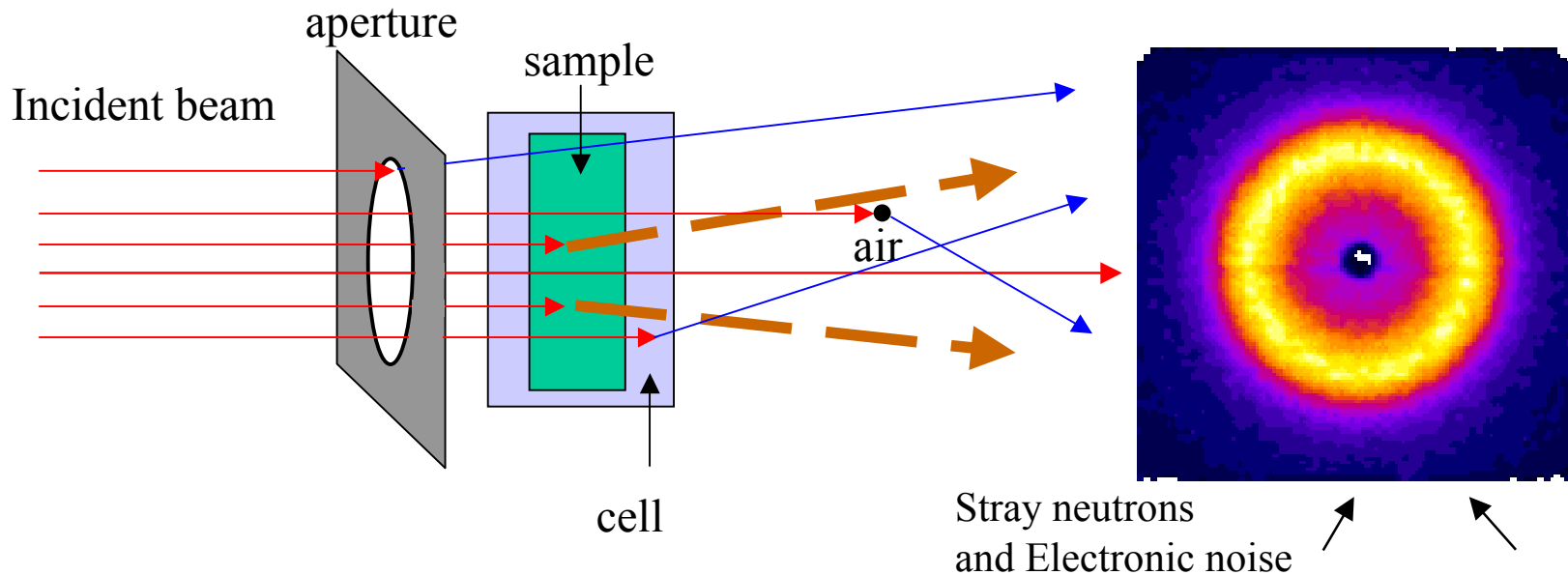




Sample Scattering

- Contribution to detector counts

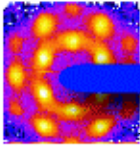
- 1) Scattering from sample
- 2) Scattering from other than sample (neutrons still go through sample)
- 3) Stray neutrons and electronic noise (neutrons don't go through sample)



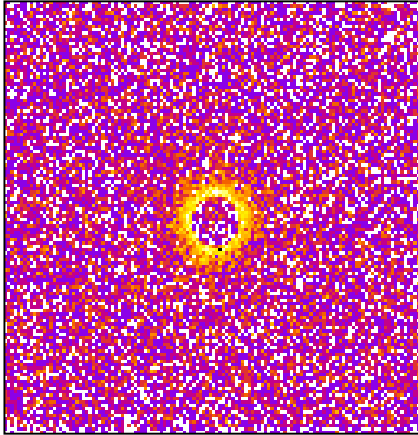
• We need MORE measurements

Counting time
$I_{SAM} = (\text{Count Rate})_{\text{sample}} t$
$\sigma_{I_{SAM}} = \sqrt{I_{SAM}} \quad \sigma_{I_{SAM}} / I_{SAM}$

Additional Measurements



- Empty Beam

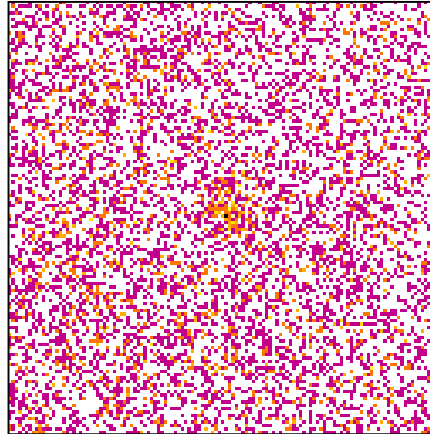


Source

- 1) Scattering from empty cell
- 2) Scattering from windows and collimation slits
- 3) Air scattering

- Minimize air in beam path
- Carefully choose cell and window materials
- **Measure an empty cell**

- Blocked Beam

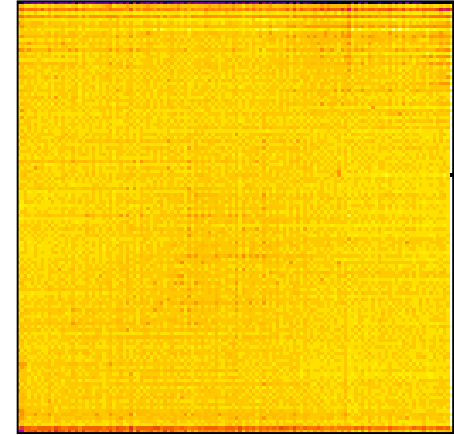


Source

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

- **Measure a blocked beam**
(⁶Li or Boronated material)

- Detector sensitivity



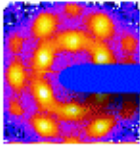
Why ?

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

- Use isotropic scattering material (Plexiglass or water)
- **We calibrate each reactor cycle**

- Counting time

$$\frac{t_{background}}{t_{sample}} = \sqrt{\frac{\text{Count Rate}_{background}}{\text{Count Rate}_{sample}}}$$



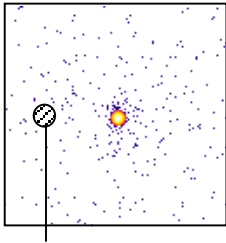
Absolute Scaling

- This is what we have

$$I(Q)_{CAL} = \phi A d T_{sample+cell} \left(\frac{d\Sigma(Q)}{d\Omega} \right)_{sample} \Delta\Omega \varepsilon t$$

- **Direct Beam Flux Method**

- Measure a direct beam with nothing in the beam except an attenuator.



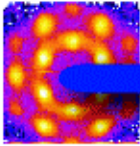
$$I_{Direct} = \phi A T_{atten.} \Delta\Omega \varepsilon t \quad \left(\frac{d\Sigma(Q)}{d\Omega} \right)_{sample} = \left(\frac{I(Q)_{CAL}}{I_{Direct}} \right) \left(\frac{1}{d} \right) \left(\frac{T_{atten.}}{T_{sample+cell}} \right)$$

- **Standard Sample Calibration**

- Use a sample with known absolute scattering cross-section at $Q=0$.
- Measure the standard sample with the exactly same configuration

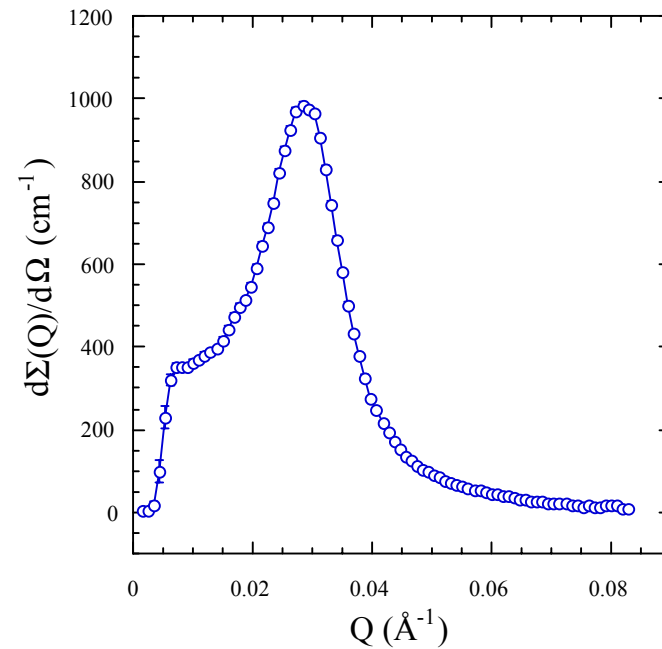
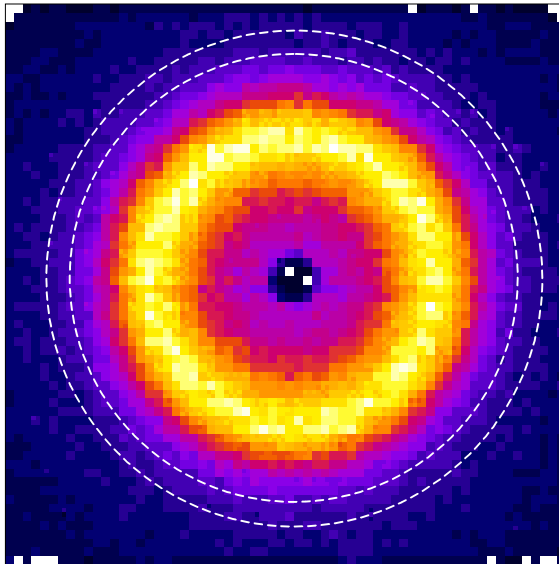
$$I(Q=0)_{STD} = \phi A d_{STD} T_{STD+cell} \left(\frac{d\Sigma(Q=0)}{d\Omega} \right)_{STD} \Delta\Omega \varepsilon t$$

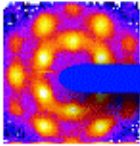
$$\left(\frac{d\Sigma(Q)}{d\Omega} \right)_{sample} = \left(\frac{I(Q)_{CAL}}{I(Q=0)_{STD}} \right) \left(\frac{d_{STD}}{d} \right) \left(\frac{T_{STD+cell}}{T_{sample+cell}} \right) \left(\frac{d\Sigma(Q=0)}{d\Omega} \right)_{STD}$$



Circular 1D Average

- Take average over annulus
- Each annulus corresponds to one data point in reduced 1D SANS data





Q-Resolution Function

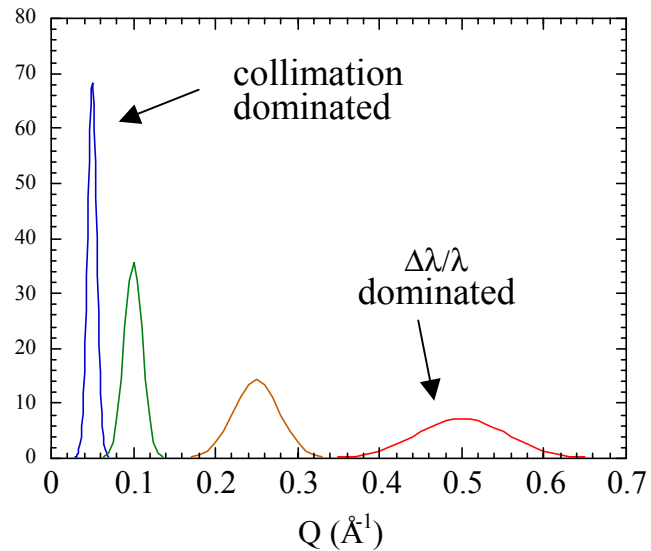
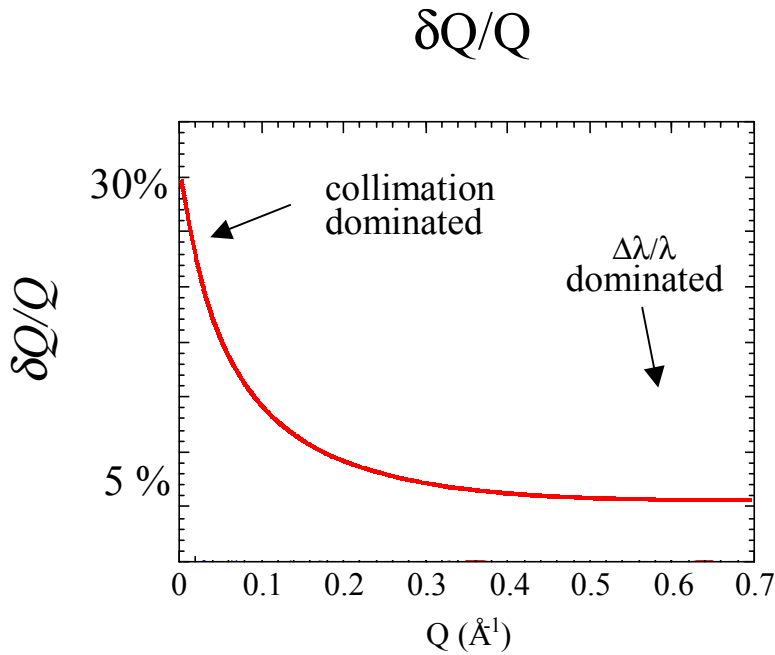
$$Q = \frac{4\pi}{\lambda} \sin\left(\frac{\theta}{2}\right) \approx \frac{2\pi}{\lambda} \theta$$

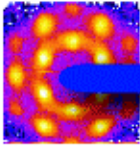
$$\left(\frac{\delta Q}{Q}\right)^2 = \left(\frac{\delta\theta}{\theta}\right)^2 + \left(\frac{\delta\lambda}{\lambda}\right)^2$$

↑ collimation ↑ wavelength spread

- Collimation : $L_1, L_2, A_1, A_2, \delta D$ (detector resolution)

Q-Resolution Function
in Gaussian Approximation
 $R(Q, Q_0) = A \exp(-Q^2 - Q_0^2 + 2QQ_0) / \delta Q^2$





Smearing Effect

- The magnitude of smearing effect is proportional to the curvature of scattering function

$$I_{\text{smearred}}(Q_o) \cong I(Q_o) + A\sigma_Q^2 \left. \frac{d^2 I(Q)}{dQ^2} \right|_{Q=Q_o} + \dots$$

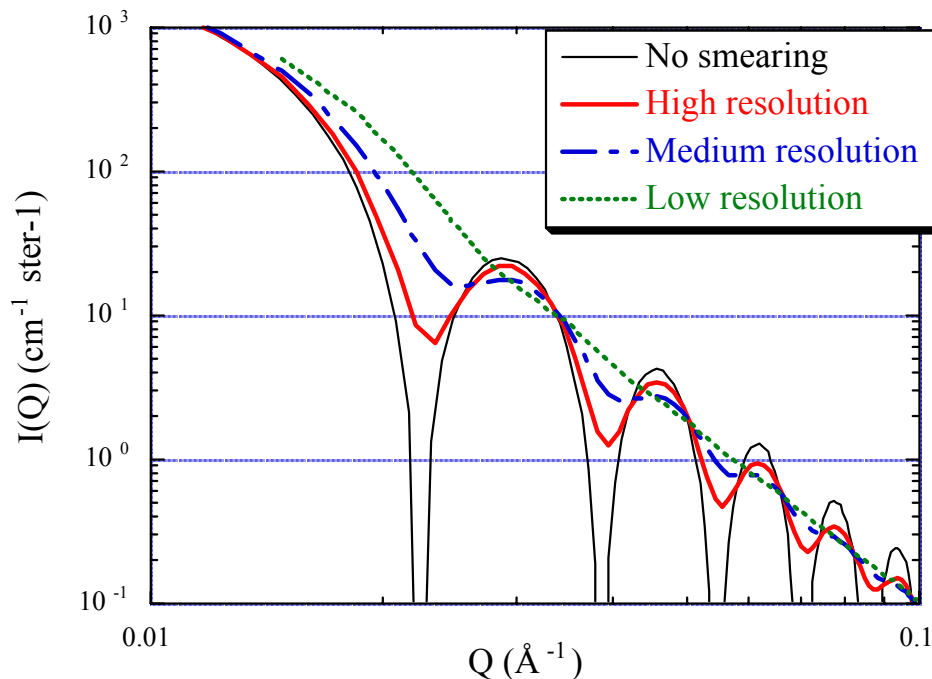
→ Sharp features are smeared the most.

High resolution

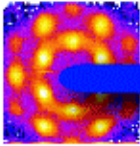
Small A_1, A_2 ,
Long L_1, L_2
Small $\Delta\lambda/\lambda$
Large λ

Low Resolution

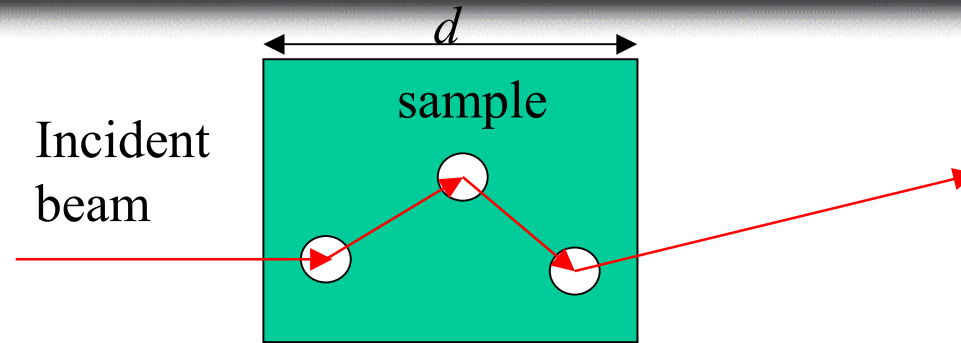
Large A_1, A_2 ,
Short L_1, L_2
Large $\Delta\lambda/\lambda$
Small λ



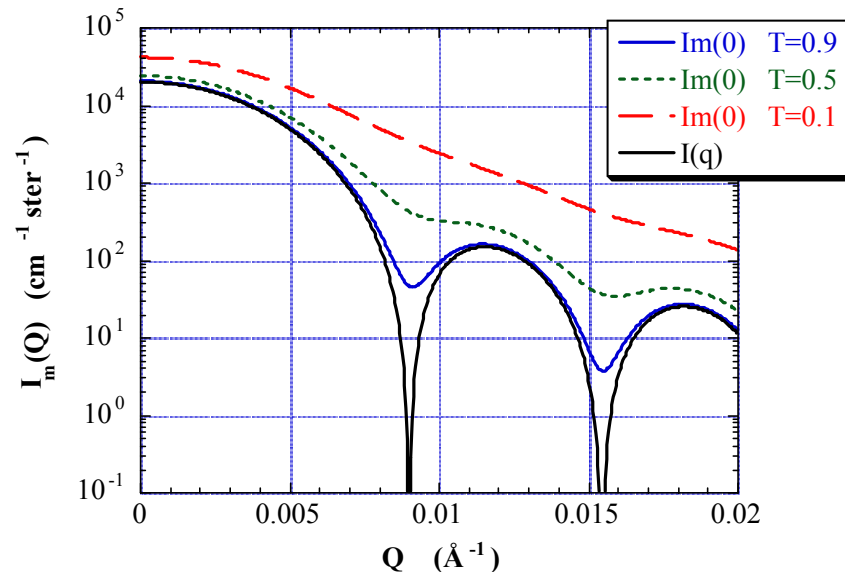
Form factor
of a monodisperse sphere
($R=200\text{\AA}$)



Multiple Scattering



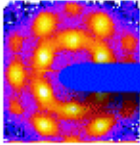
- When sample has a strong coherent scattering X-section and thick.
- Final scattering angle is added incoherently
- To reduce the multiple scattering, we need to reduce d .



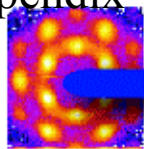
When attenuation is only due to coherent scattering

(J.G. Barker)

Summary



- Now We have a whole picture of SANS Experiment.
 - Sample preparation
 - Optimization of configuration.
 - What to measure.
- To get good quality of data, Initial Planning is VERY Important.
- Use Beamtime Efficiently

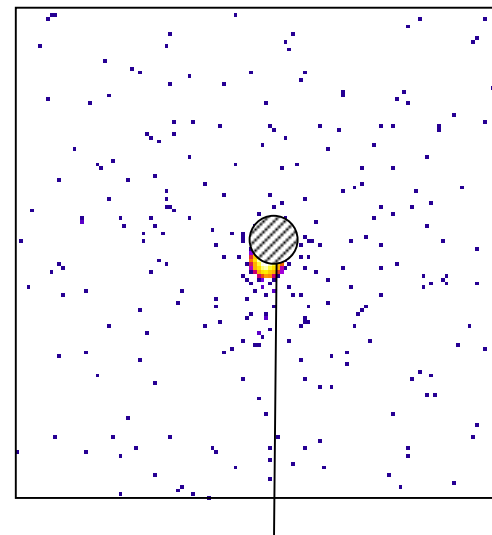
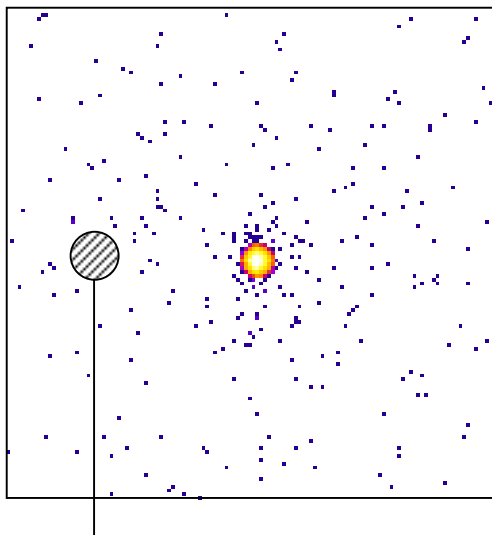


Beam Alignment and Initial Measurements

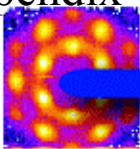
- Align the center of sample with neutron beam
 - laser beam and neutron camera
- Measure a beam center
 - $Q = 0$ position
 - Use a proper beam attenuator
- Align beamstop
 - 1, 2, 3, 4 inch diameter
 - NO attenuator

128 x 128
pixels

Beam center
(65.94, 63.87)



(Beamstop needs to move down and left)

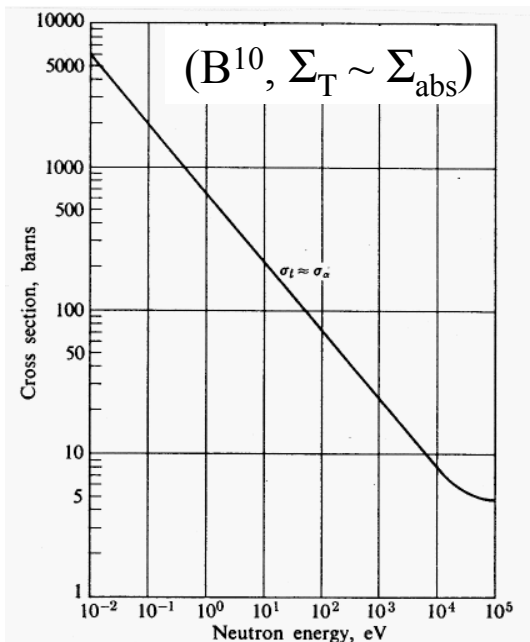


Wavelength Dependence of Cross-Section

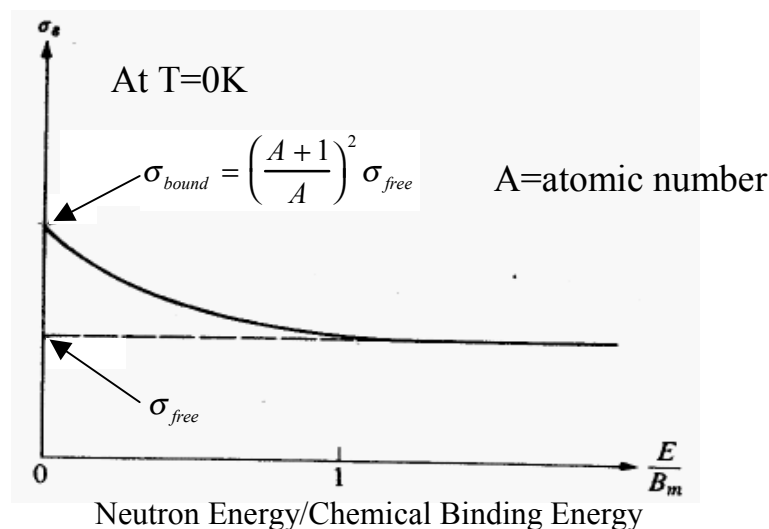
- Neutron cross-section depends on neutron wavelength λ .
- Absorption Cross-Section
- Scattering Cross-Section

$$\Sigma_{abs} \propto 1/v_n \propto \lambda$$

where v_n = neutron velocity

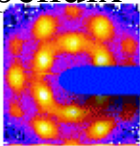


- Scattering lengths, b , listed in table are **bound scattering length**.



For light element $\sigma_{bound} > \sigma_{free}$
 (Hydrogen, $\sigma_{bound} = 4 \sigma_{free}$)

For heavy element $\sigma_{bound} \approx \sigma_{free}$



Q-Resolution Function

- The measured scattering intensity is $I(Q)$ of sample convoluted with a resolution function $R(Q, Q_0)$.

$$I_{\text{smearred}}(Q_0) = \int R(Q, Q_0) I(Q) dQ$$

- Q-resolution Function $R(Q, Q_0)$ is determined by :
 - 1) Beam collimation
 - 2) Detector resolution
 - 3) Wavelength
 - 4) Wavelength spread

$$\sigma_Q^2 = \underbrace{\left(\frac{2\pi}{\lambda}\right)^2 \sigma_\theta^2}_{1) \ 2) \ 3)} + \underbrace{Q^2 \left(\frac{\Delta\lambda}{\lambda}\right)^2}_{4)}$$

$$\sigma_\theta^2 = \frac{1}{16} \left(\frac{A_1}{L_1}\right)^2 + \frac{A_2^2}{16} \left(\frac{1}{L_1} + \frac{1}{L_2}\right)^2 + \left(\frac{\sigma_d}{L_2}\right)^2$$

