SANS Experimental Methods

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NCNR Summer School

Neutron Small Angle Scattering and
Reflectometry from Submicron Structures
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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 ?

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

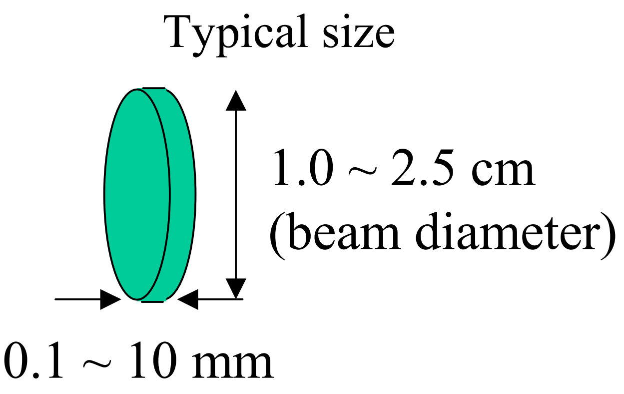
- Is it accessible with SANS ?
  - Length scale of interest (~10 Å - ~5000 Å)
  - Sample size
  - Neutron scattering contrast
  - Sample environment
    (Temperature, Pressure, Magnetic field and etc.)
  - How long would it take ?

- 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

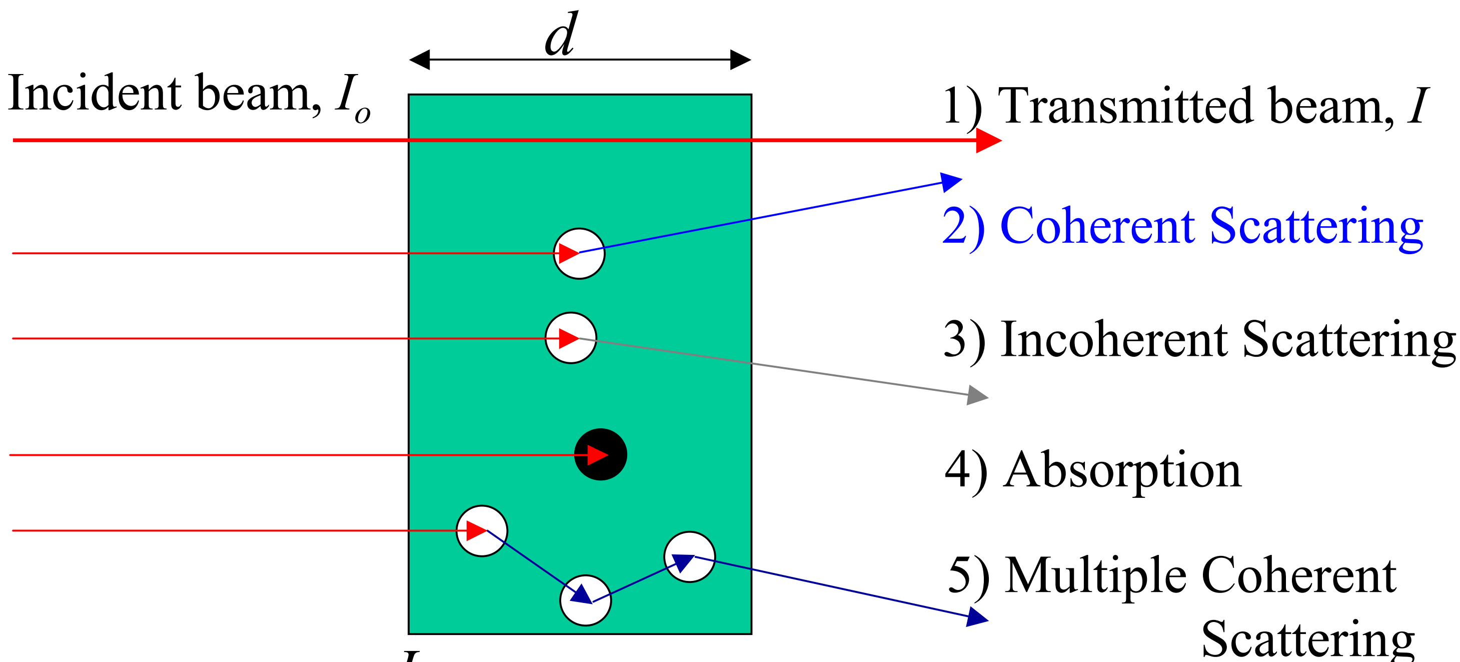
- Prepare proper neutron contrast

- Standard SANS sample cell

- Custom made sample cell

What Should Sample Thickness Be?

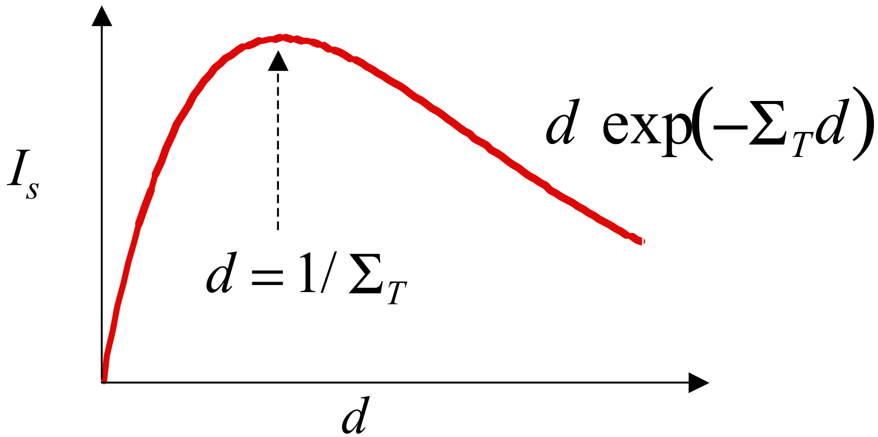
- To decide the sample thickness, we need to understand what is happening in the sample



- 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 $d = 1/\Sigma_T$

$\longrightarrow T = \exp(-\Sigma_T d)$

$= 1/e = 37\%$

- 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 $\Sigma_{coh} << \Sigma_T \approx \Sigma_{inc} + \Sigma_{abs}$ $\longrightarrow$ $d = 1/\Sigma_T$, $T = 37\%$

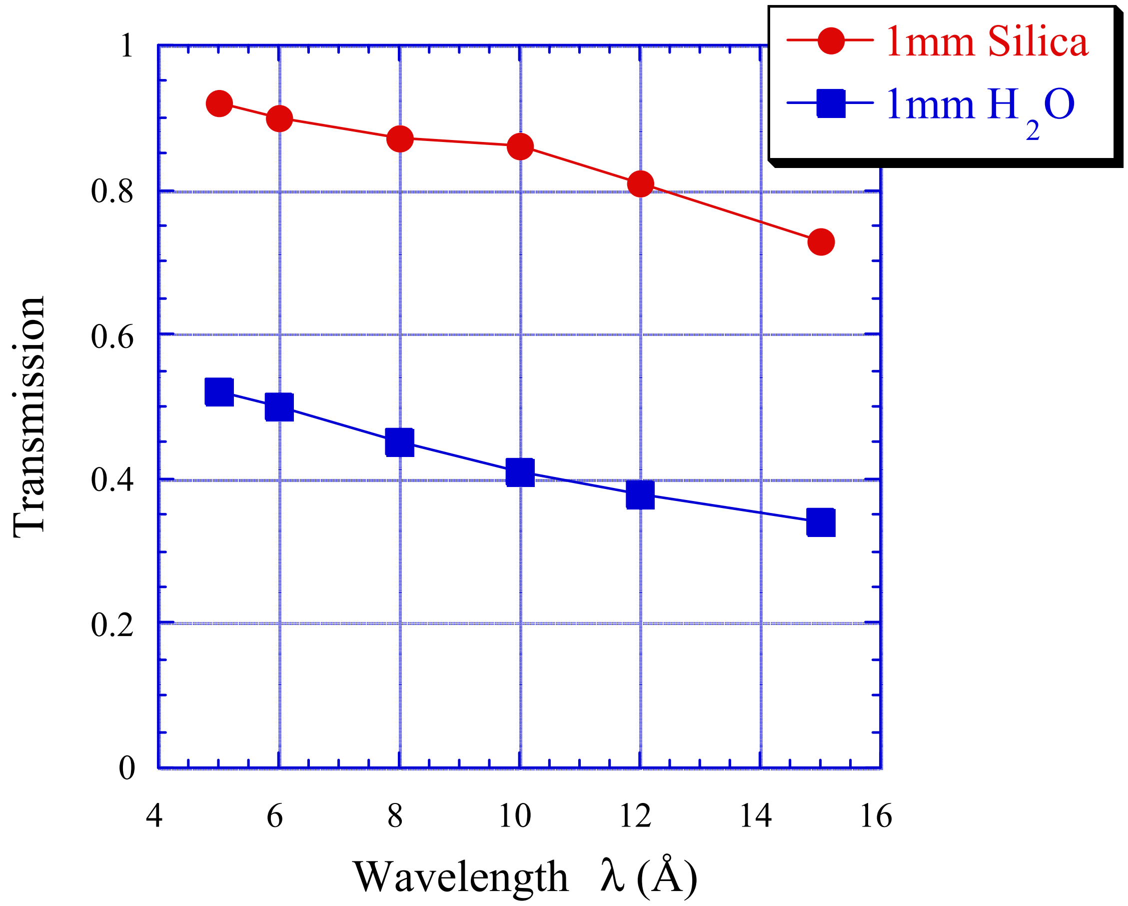
Examples of Sample Thickness

- When $\lambda = 5Å$

$\Sigma_T \approx \Sigma_{coh}$ (want T > 90%)	$\Sigma_{coh} << \Sigma_T \approx \Sigma_{abs} + \Sigma_{inc}$ (T = 1/e = 37 % is optimal)
Silica 0.5 mm, T = 96 %	H_2O 1 mm, T = 52 %
1 mm, T = 92 %*	1.5 mm, T = 38 % *
3 mm, T = 78 %	3 mm, T = 14 %
$\boldsymbol{\sigma_{coh}}$ **= 10.62 barns**	σ_{coh} = 7.75 barns
σ_{inc} = 0.005 barns	$\boldsymbol{\sigma_{inc}}$ **= 164 barns**
σ_{abs} = 0.17 barns	σ_{abs} = 0.66 barns

Wavelength Dependence of Transmission

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



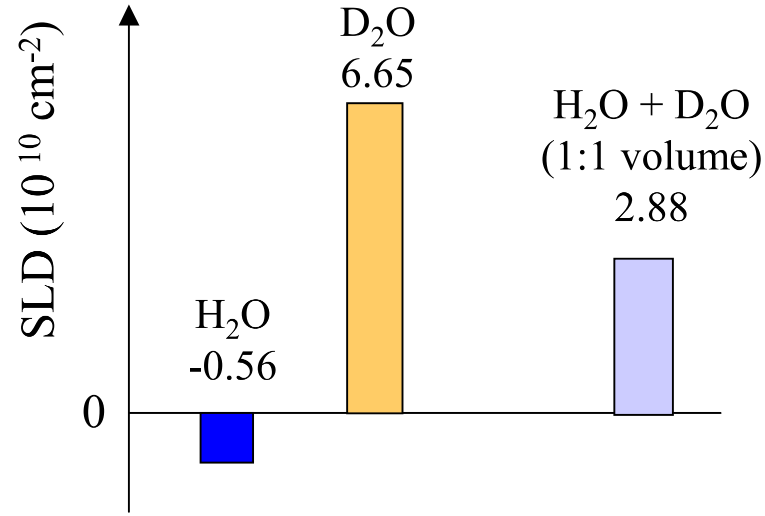
Calculation of Scattering Length Density

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

N_A = Avogadro's number $= 6 \times 10^{23}$

M_w = molecular weight

b_i = bound coherent scattering length of atom i



- H_2O

$$\rho_{SLD,H_2O} = (6\times10^{23}/mol)\left(\frac{1.0\,g/cm^3}{18\,g/mol}\right)(2\times b_H + b_O)$$
$$= \underline{-0.56\times10^{10}\,cm^{-2}}$$

- D_2O

$$\rho_{SLD,D_2O} = (6\times10^{23}/mol)\left(\frac{1.1\,g/cm^3}{20\,g/mol}\right)(2\times b_D + b_O)$$
$$= \underline{6.32\times10^{10}\,cm^{-2}}$$

- $H_2O + D_2O$ mixture (1:1 volume)

$$\rho_{SLD,\,Mixture} = x_{H_2O}\times\rho_{SLD,H_2O} + (1-x_{H_2O})\times\rho_{SLD,D_2O}$$
$$= 0.5\times(-0.56\times10^{10}\,cm^{-2}) + 0.5\times(6.32\times10^{10}\,cm^{-2}$$
$$= \underline{2.88\times10^{10}\,cm^{-2}}$$

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

b_H = -3.74 b_D = 6.67 b_O = 5.80

Coherent and Incoherent Scattering

- Coherent scattering contains the structural information of sample
- Incoherent scattering is flat background.
- Examples

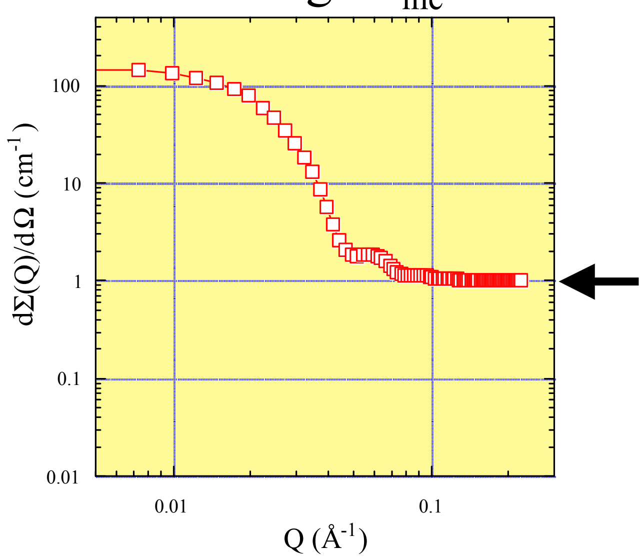
  H_2O or Hydrocarbons ~ 1 cm^{-1} $ster^{-1}$

  D_2O or Deuterated sample ~ 0.1 cm^{-1} $ster^{-1}$

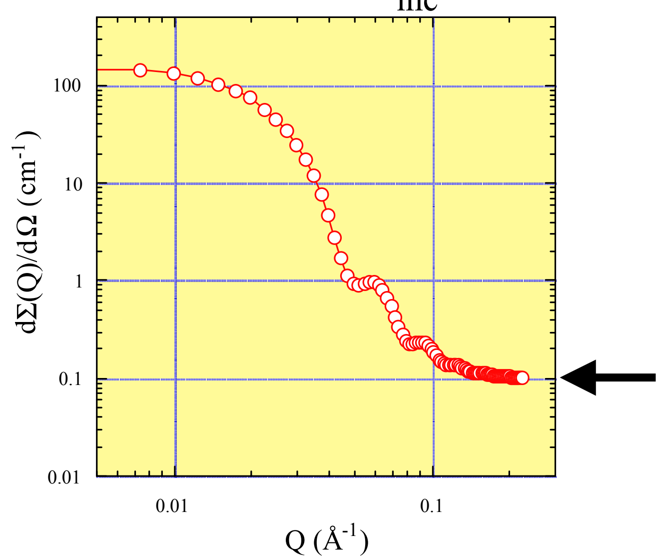
  SiO_2 (amorphous) ~ 0.02 cm^{-1} $ster^{-1}$

- Large incoherent scattering reduce the dynamic range of measurement.
- **Use deuterated solvent whenever it is possible.**

Large Σ_{inc}

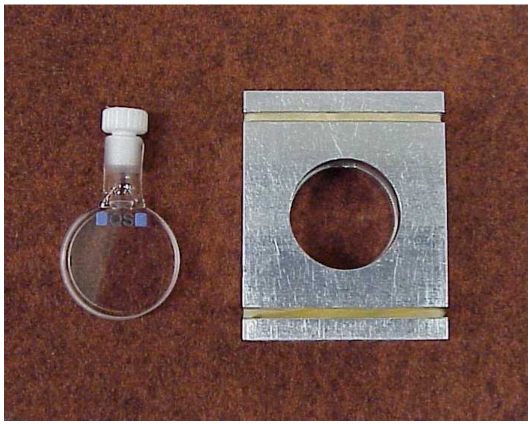


Small Σ_{inc}



SANS Sample Holders

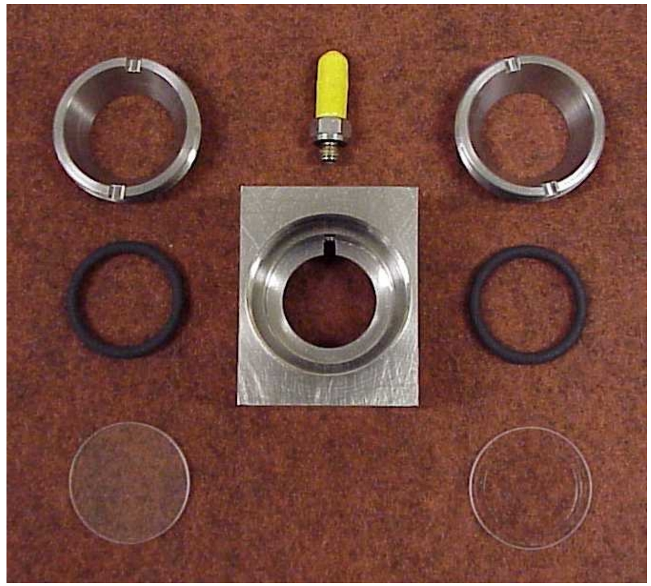
Liquid



Path length (Volume)
1 mm (0.3 ml)
2 mm (0.6 ml)
5 mm (1.5 ml)

Diameter = 2.0 cm

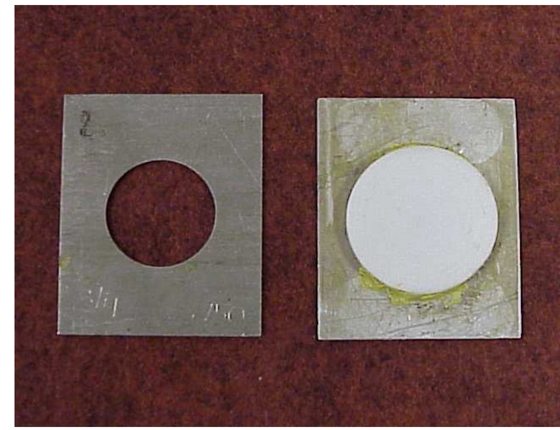
Gel or Polymer Melt



Path length (Volume)
1 mm (0.3 ml)
2 mm (0.6 ml)
4 mm (1.2 ml)

Diameter = 2.0 cm

Solid

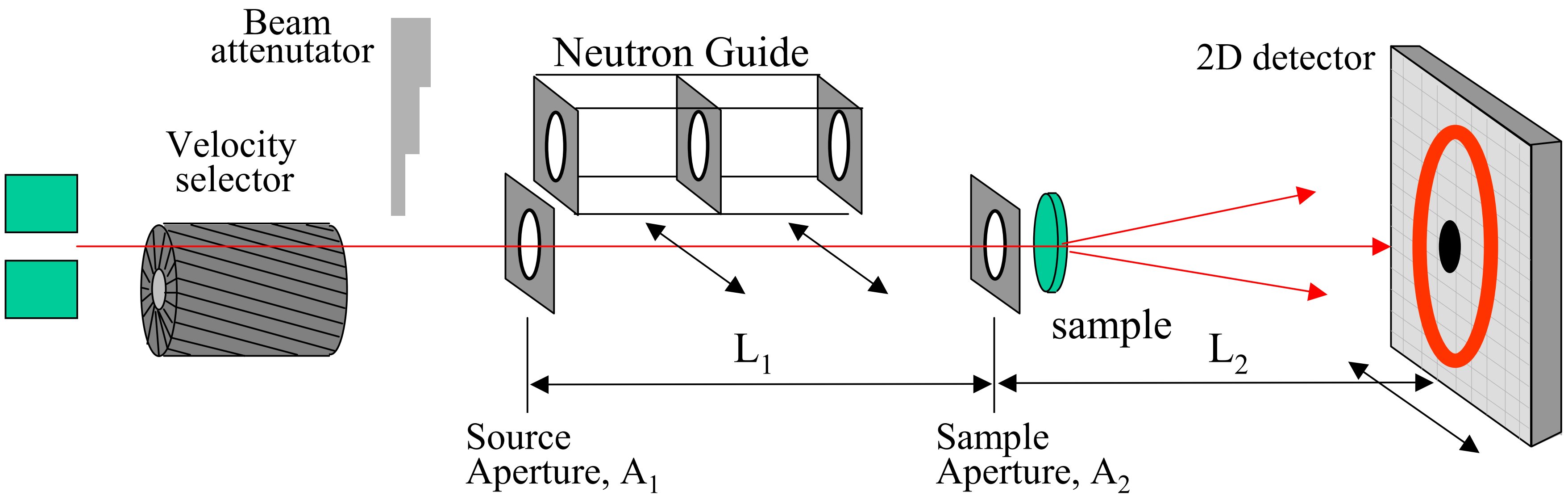


Glue on a Cd mask
Size mountable on a standard
Sample changer
width < 3.5 cm
height < 5 cm
thickness < 2cm

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 (Δλ/λ=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)

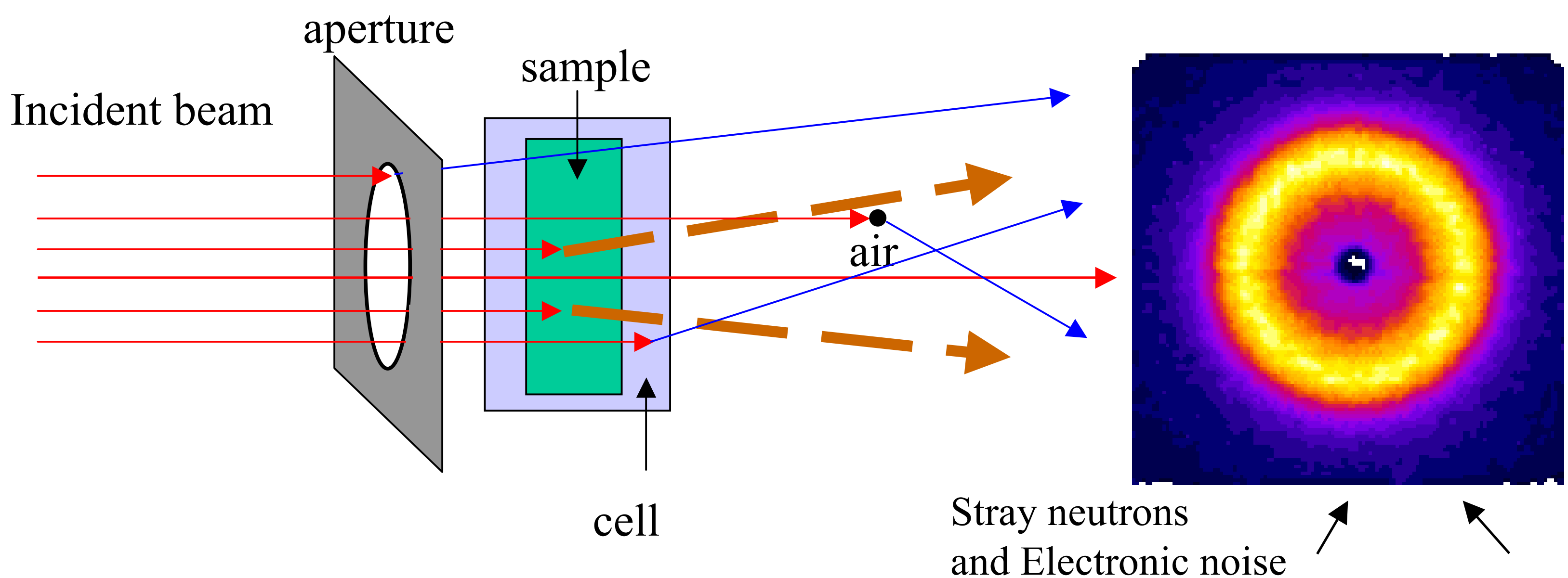
Use SASCAL or Web Based Tools



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

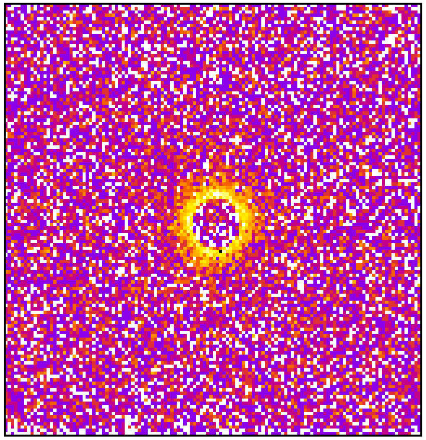
Counting time

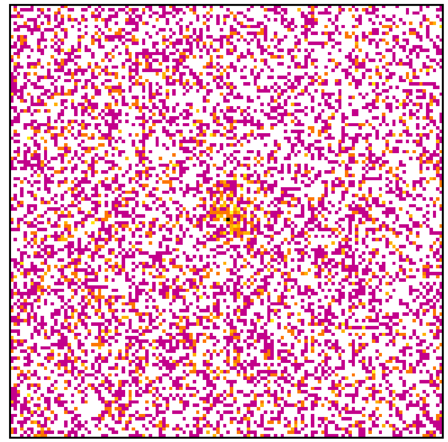
$$I_{SAM} = (\text{Count Rate})_{\text{sample}}\ t$$

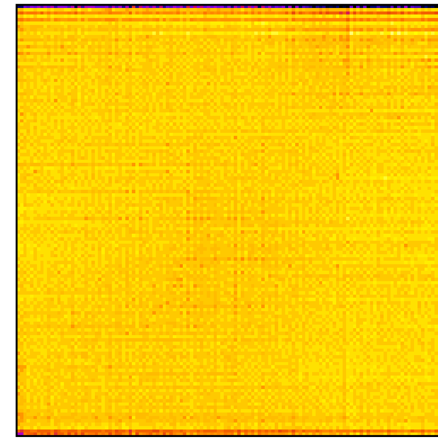
$$\sigma_{I_{SAM}} = \sqrt{I_{SAM}} \qquad \sigma_{I_{SAM}} / I_{SAM}$$

Additional Measurements

- Empty Beam
- Blocked Beam
- Detector sensitivity







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**

Source

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

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

Why ?

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

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

- Counting time

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

Data Correction

Measured Raw Data

$$I_{SAM} = C_O\, T_{sample+cell} \left(\left(\frac{d\Sigma(Q)}{d\Omega} \right)_{sample} + \left(\frac{d\Sigma(Q)}{d\Omega} \right)_{EMP} \right) + I_{Blocked\ Beam}$$

$$I_{EMP} = C_O\, T_{cell} \left(\frac{d\Sigma(Q)}{d\Omega} \right)_{EMP} + I_{Blocked\ Beam}$$

$$I_{BGD} = I_{Blocked\ \ Beam}$$

$T_{sample+cell}$ and T_{cell}

$$C_O = \phi\, A\, d\Delta\Omega\, \varepsilon\, t$$

ϕ = incident neutron flux

A = sample area

d = sample thickness

$\Delta\Omega$ = solid angle of each pixel

ε = detector efficiency

t = counting time

Corrected SANS data

$$I_{COR} = (I_{SAM} - I_{BGD}) - \left(\frac{T_{sample+cell}}{T_{cell}} \right) (I_{EMP} - I_{BGD})$$

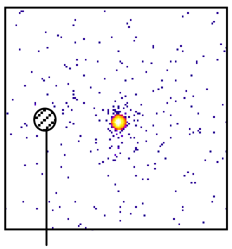
- The corrected SANS data is then calibrated with detector sensitivity.

$$I_{CAL} = I_{COR} / (\text{Normalized Detector Sensitivity})$$

Absolute Scaling

- This is what we have

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



- **Direct Beam Flux Method**
  - Measure a direct beam with nothing in the beam except an attenuator.

$$I_{Direct} = \phi\, A\, T_{atten.} \Delta\Omega\, \varepsilon\, t$$

$$\boxed{\left(\frac{d\Sigma(Q)}{d\Omega}\right)_{sample} = \left(\frac{I(Q)_{CAL}}{I_{Direct}}\right)\left(\frac{1}{d}\right)\left(\frac{T_{atten.}}{T_{sample+cell}}\right)}$$

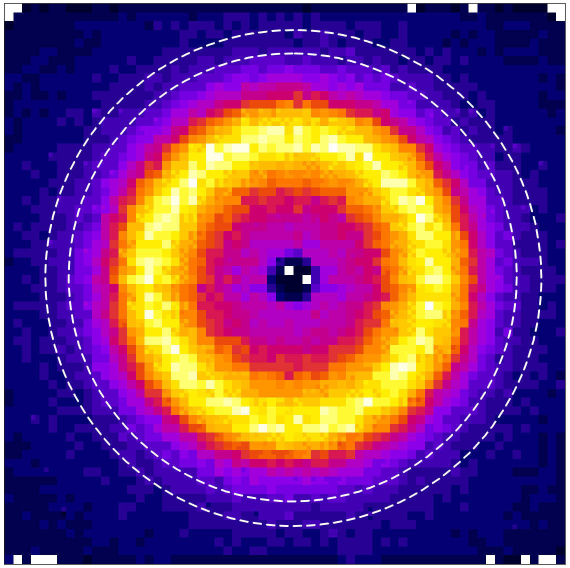
- **Standard Sample Calibration**
  - Use a sample with known absolute scattering cross-section at Q=0.
  - Measure the standard sample with the exactly same configuration

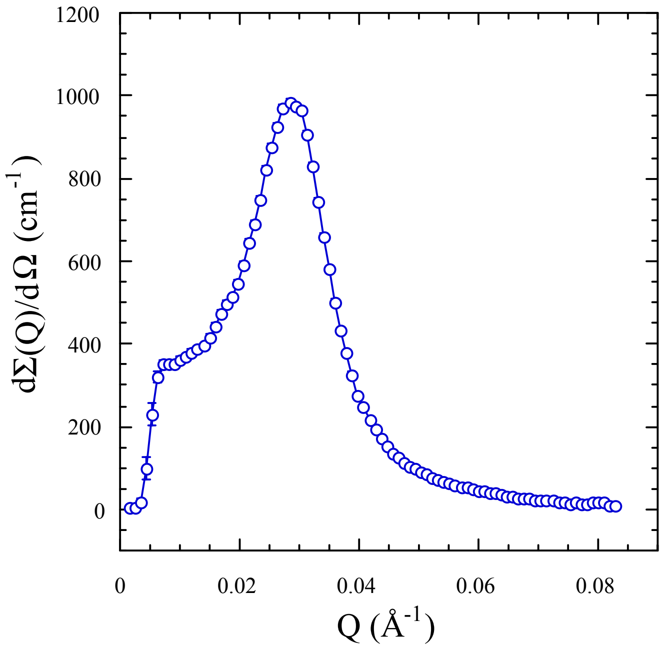
$$I(Q=0)_{STD} = \phi\, A\, d_{STD}\, T_{STD+cell} \left(\frac{d\Sigma(Q=0)}{d\Omega}\right)_{STD} \Delta\Omega\, \varepsilon\, t.$$

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

Circular 1D Average

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



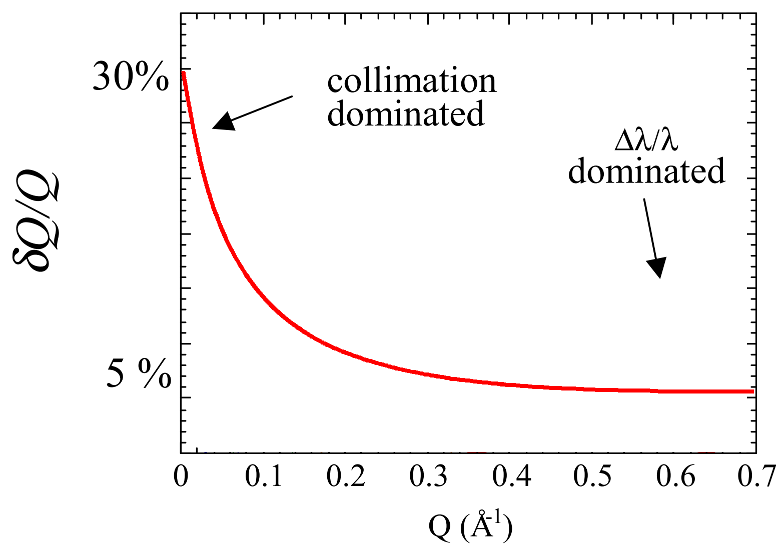


Q-Resolution Function

$$Q = \frac{4\pi}{\lambda}\sin\left(\frac{\theta}{2}\right) \approx \frac{2\pi}{\lambda}\theta \qquad \left(\frac{\delta Q}{Q}\right)^2 = \underbrace{\left(\frac{\delta\theta}{\theta}\right)^2}_{\text{collimation}} + \underbrace{\left(\frac{\delta\lambda}{\lambda}\right)^2}_{\text{wavelength spread}}$$

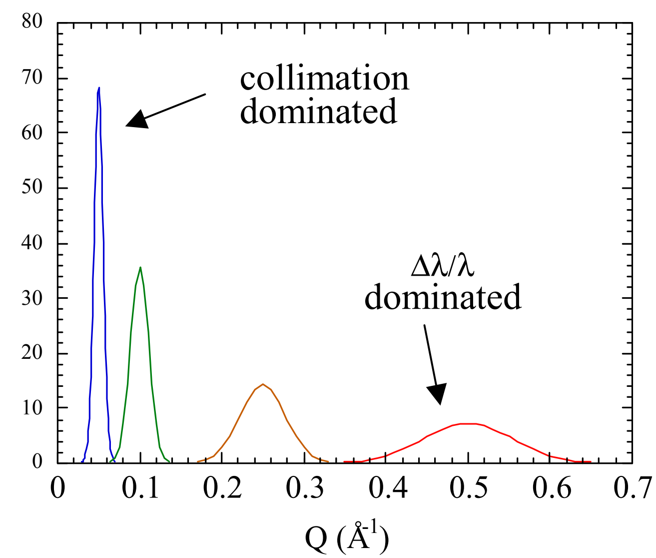
- Collimation : L_1, L_2, A_1, A_2, δD (detector resolution)

δQ/Q



Q-Resolution Function
in Gaussian Approximation

$R(Q,Q_o) = A \exp(-(Q-Q_o)^2/\delta Q^2)$



Smearing Effect

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

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

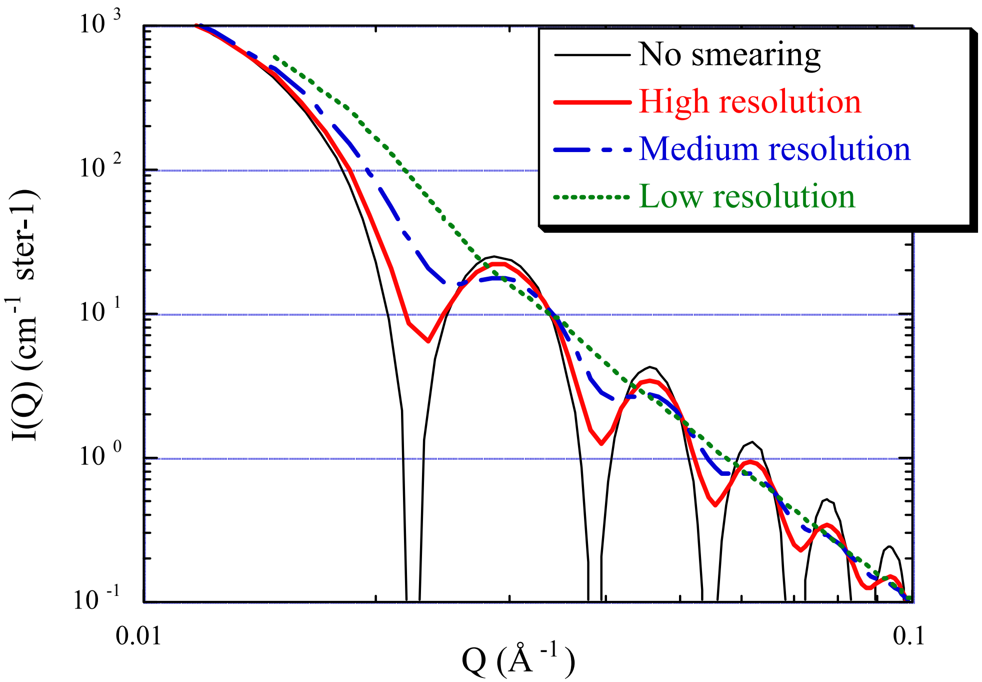
→ Sharp features are smeared the most.

<u>High resolution</u>

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

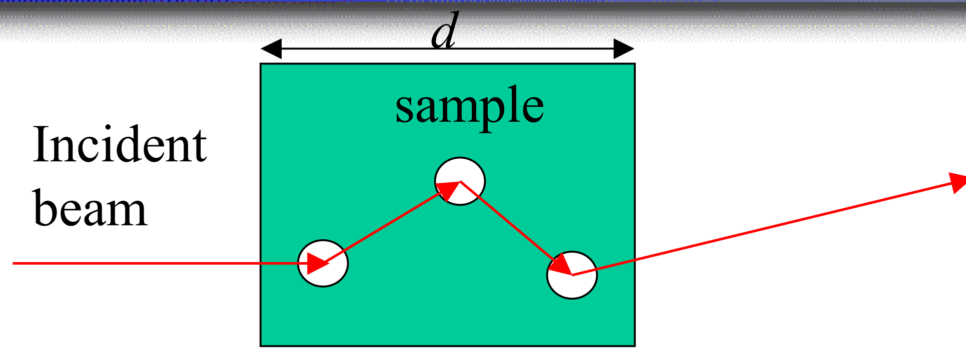
<u>Low Resolution</u>

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

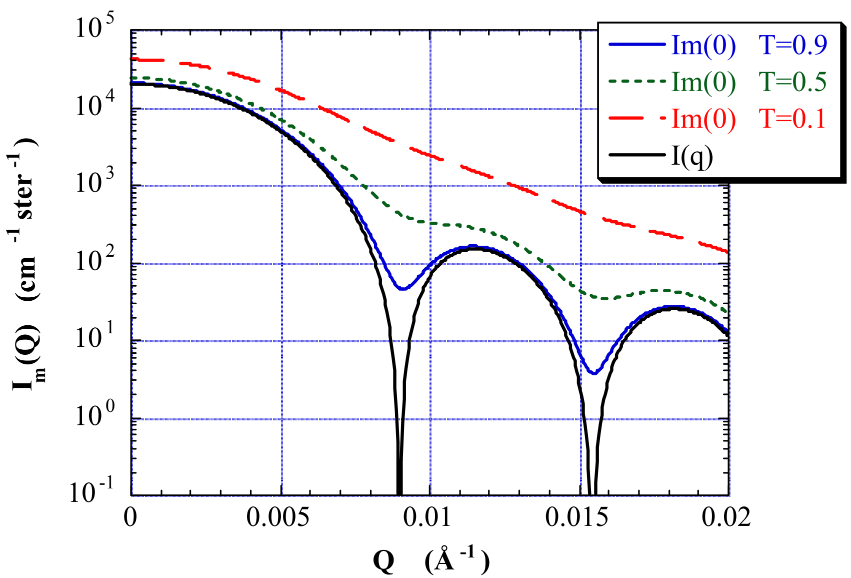


Form factor
of a monodisperse sphere
(R=200Å)

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

National Institute of Standards and Technology

- 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

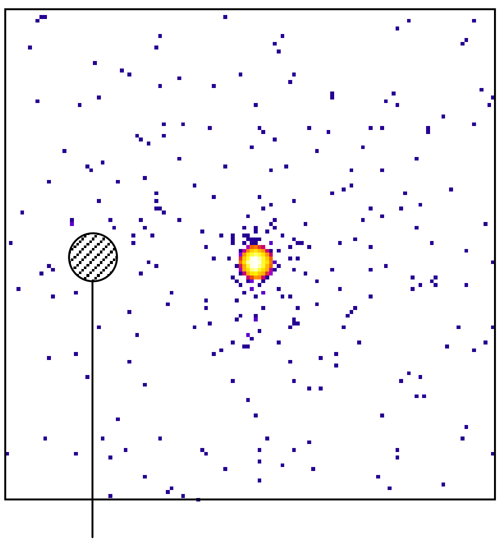
- Align the center of sample with neutron beam
  - laser beam and neutron camera

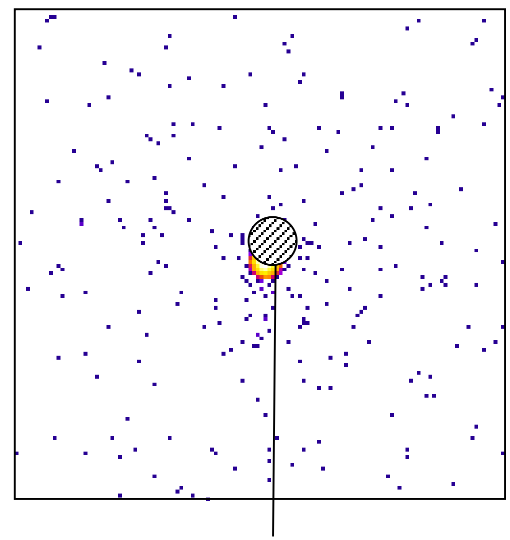
- Measure a beam center
  - Q = 0 position
  - Use a proper beam attenuator

- 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)

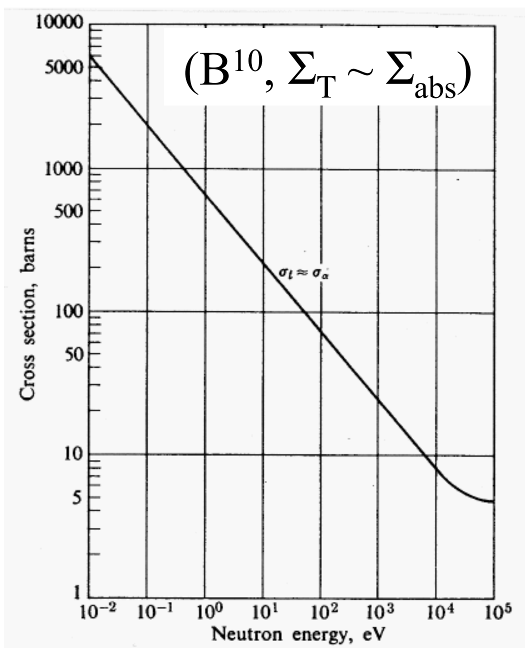
Wavelength Dependence of Cross-Section

- Neutron cross-section depends on neutron wavelength λ.

- Absorption Cross-Section

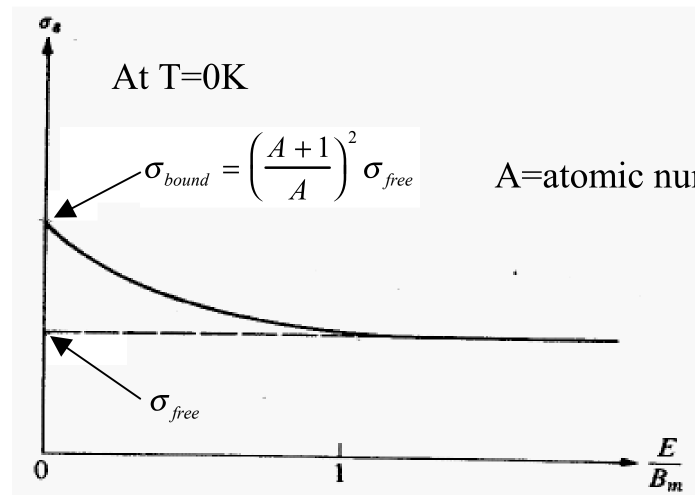
$$\boxed{\Sigma_{abs} \propto 1/v_n \propto \lambda}$$

where $v_n =$ neutron velocity



- Scattering Cross-Section

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



A=atomic number

Neutron Energy/Chemical Binding Energy

For light element $\sigma_{bound} > \sigma_{free}$

(Hydrogen, $\sigma_{bound} = 4\ \sigma_{free}$)

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

Appendix

Q-Resolution Function

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

$$I_{smeared}(Q_o) = \int R(Q,Q_o)I(Q)dQ$$

- Q-resolution Function $R(Q,Q_o)$ is determined by :

  1) Beam collimation 2) Detector resolution
  3) Wavelength 4) Wavelength spread

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

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