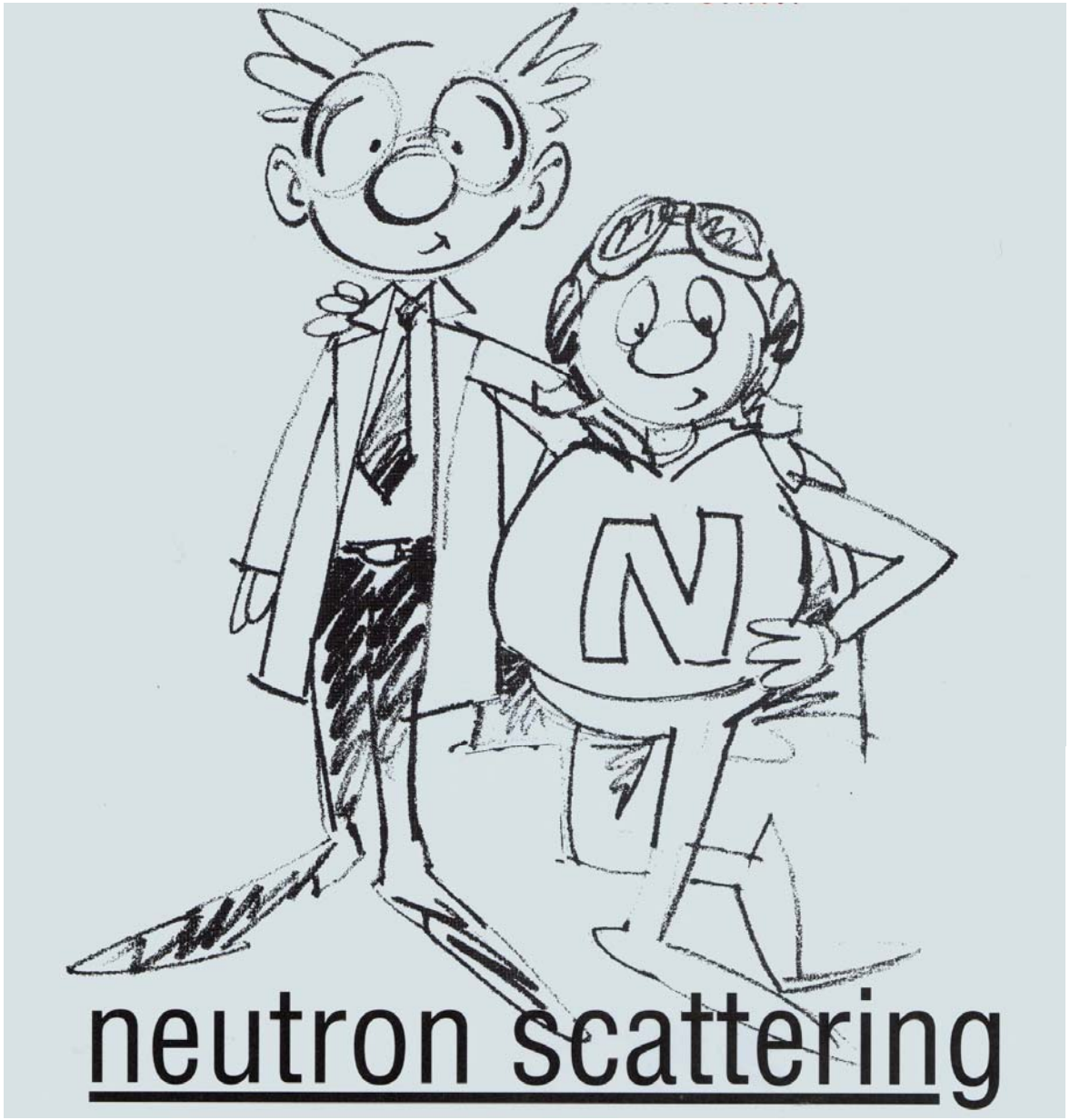


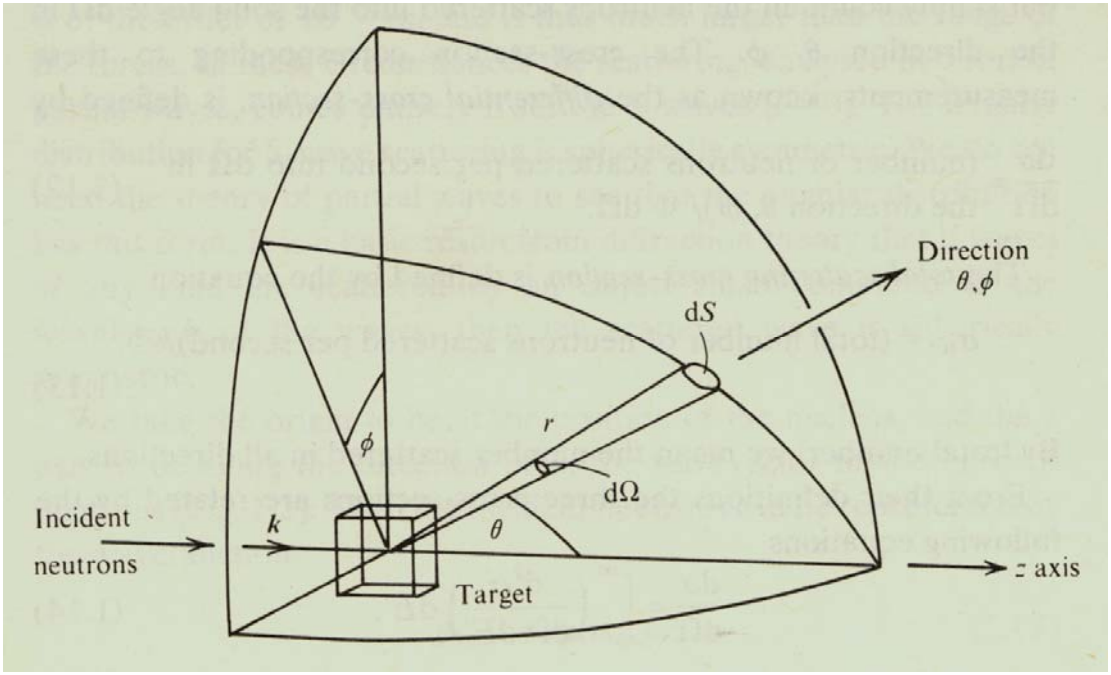
Introduction to Low-Q Neutron Scattering

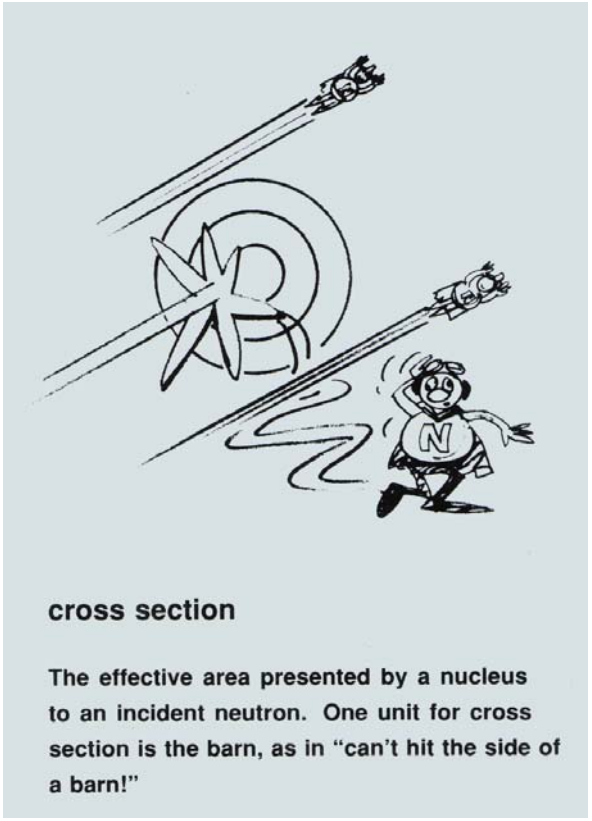
by

Roger Pynn

Indiana University
&
Spallation Neutron Source

We Measure Neutrons Scattered from a Sample





Φ = number of incident neutrons per cm^2 per second

σ = total number of neutrons scattered per second / Φ

$$\frac{d\sigma}{d\Omega} = \frac{\text{number of neutrons scattered per second into } d\Omega}{\Phi\, d\Omega}$$

$$\frac{d^2\sigma}{d\Omega dE} = \frac{\text{number of neutrons scattered per second into } d\Omega \text{ \& } dE}{\Phi\, d\Omega\, dE}$$

σ measured in barns:
1 barn = 10^{-24} cm^2

Attenuation = $\exp(-N\sigma t)$
N = # of atoms/unit volume
t = thickness

Scattering from Many Atoms

- Neutrons are scattered by nuclei
  - The range of nuclear forces is femtometers – much less than the neutron wavelength so the scattering is point like (ripples on a pond)
- Energy of (thermal) neutron is too small to change nuclear energy
  - If the nucleus is fixed, the scattering is elastic
- We can add up the (elastic) scattering from an assembly of nuclei:

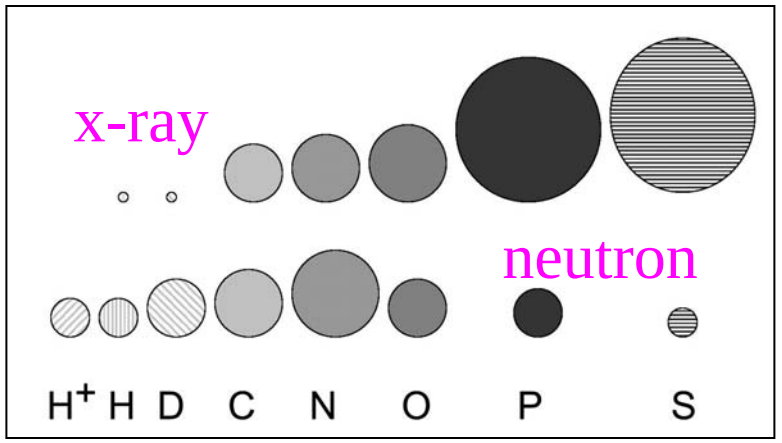
$$\frac{d\sigma}{d\Omega} = \sum_{i,j} b_i b_j e^{i(\vec{k}_0 - \vec{k}').(\vec{R}_i - \vec{R}_j)} = \sum_{i,j} b_i b_j e^{-i\vec{Q}.(\vec{R}_i - \vec{R}_j)}$$

where the wavevector transfer Q is defined by $\vec{Q} = \vec{k}' - \vec{k}_0$

  - b_i is called the coherent scattering length of nucleus i
  - $\boldsymbol{k}$ is the incident neutron wavevector ($2\pi/\lambda$); $\boldsymbol{k'}$ is the scattered wavevector
  - The calculation assumes the scattering is weak (called Born Approximation)

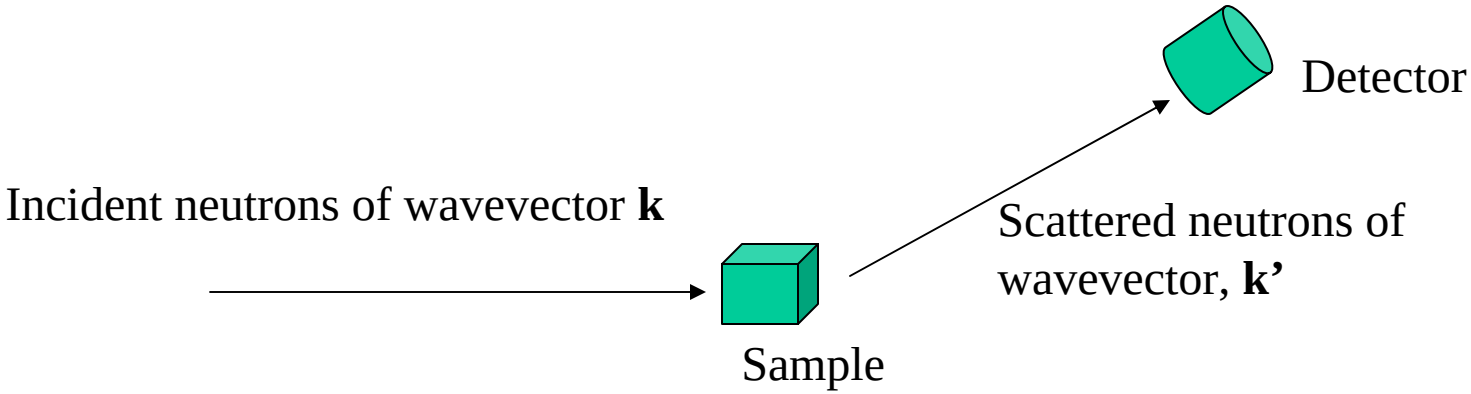
The Success of Neutron Scattering is Rooted in the Neutron's Interactions with Matter

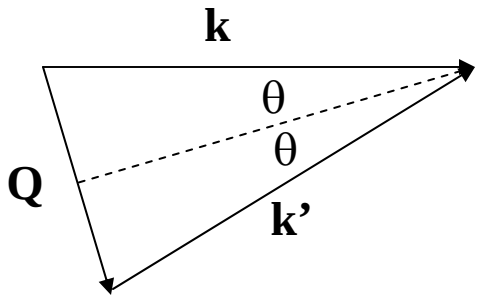
- Interact with nuclei not electrons
- Isotopic sensitivity (especially D and H)
- Penetrates sample containment
- Sensitive to bulk and buried structure



- Simple interpretation – provides statistical averages, not single instances
- Wavelength similar to inter-atomic spacings
- Energy similar to thermal energies in matter
- Nuclear and magnetic interactions of similar strength

Scattering Triangle





Neutron diffraction measures the differential scattering cross section $d\sigma/d\Omega$ as a function of the scattering wavevector (**Q**)

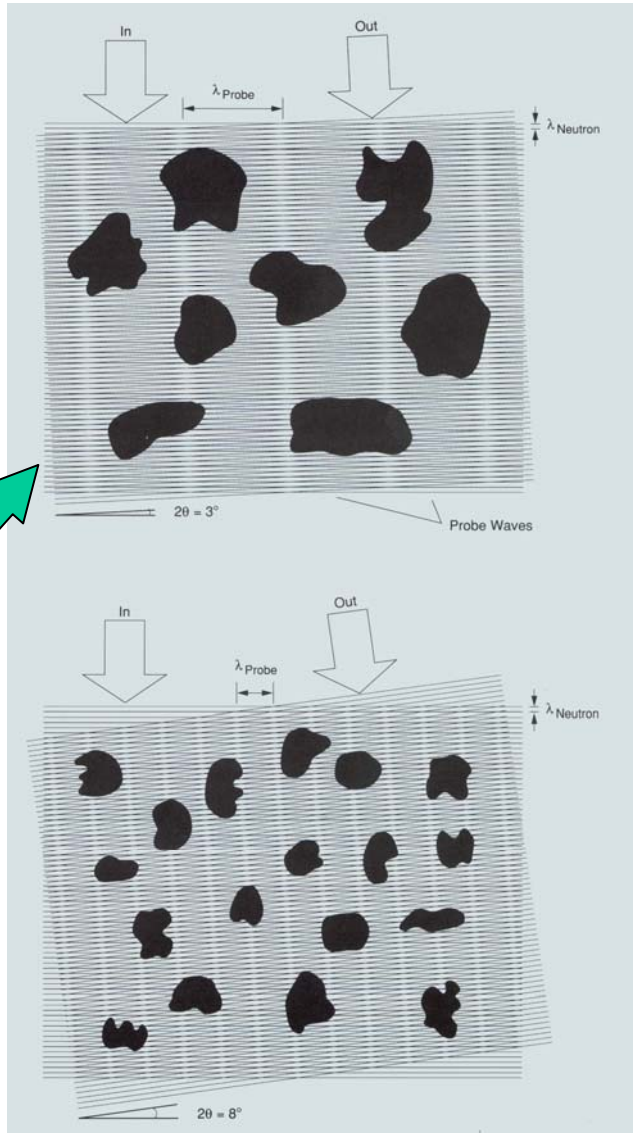
For elastic scattering, k = k' so $Q = 2\,k \sin\theta = (2\pi/\lambda)\sin\theta$
The distance probed in the sample is: $d = 2\pi / Q$
(Combining the two equations gives Bragg's Law: $\lambda = 2\,d \sin\theta$)

Low-Q Scattering (SANS & Reflectometry) is Used to Measure Large Objects (~10 nm to ~100 nm)

Small Q => large d (because $d=2\pi/Q$)

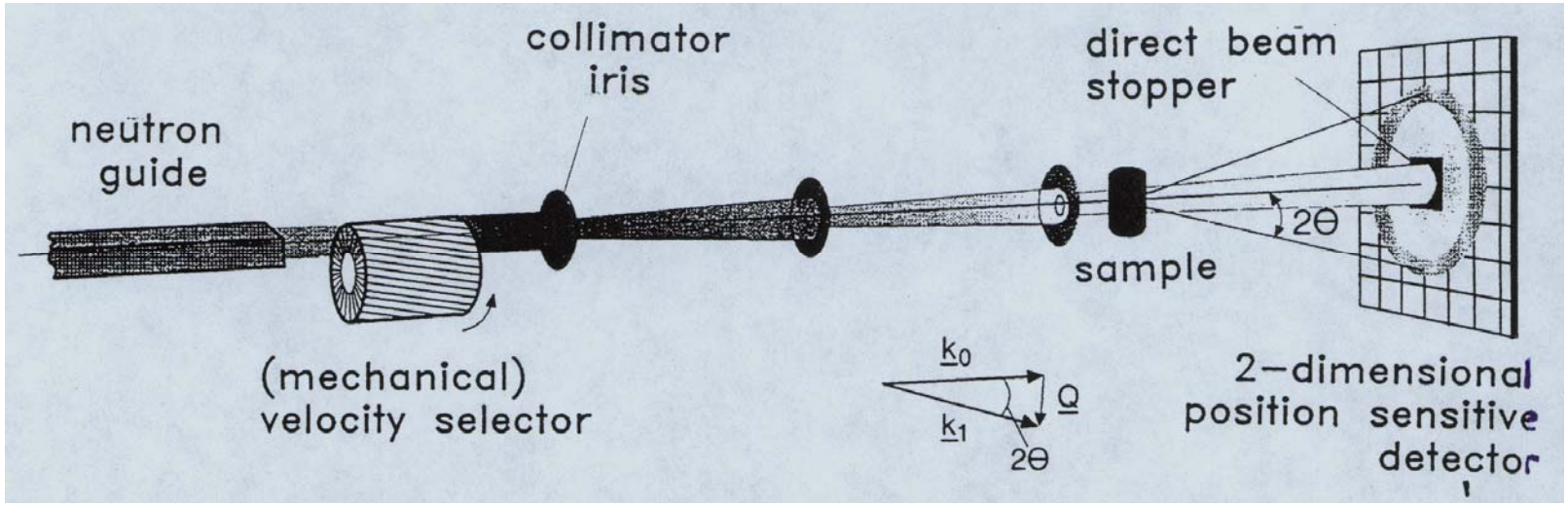
Large d => small θ (because $\lambda = 2\, d \sin\theta$)

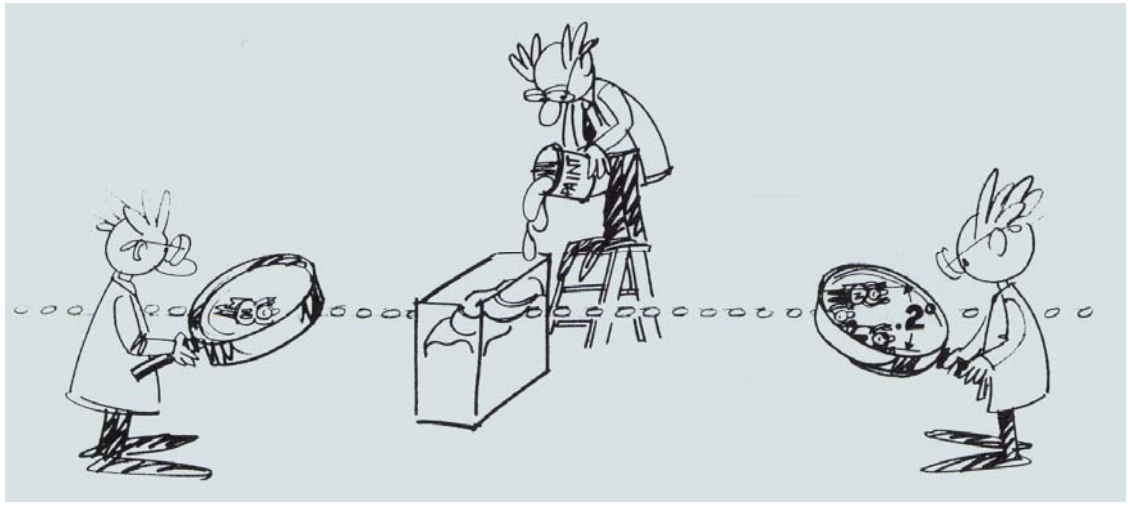
Scattering at small angles probes large length scales



Typical scattering angles for SANS are ~ 0.3º to 5º

Two Views of the Components of a Typical Reactor-based SANS Diffractometer





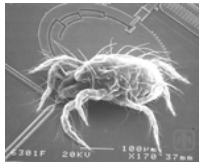
Note that SANS, like other diffraction methods, probes material structure in the direction of (vector) $\vec{Q}$

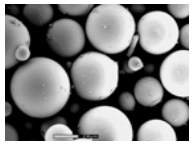
The NIST 30m SANS Instrument Under Construction

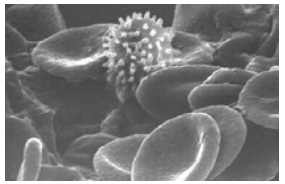
Neutron Scattering Measures the Structure of Materials from angstroms to microns!

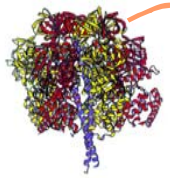
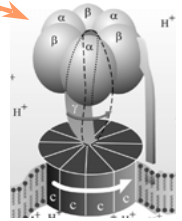

Human hair

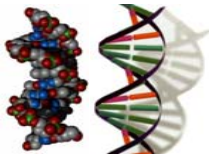

Ant


Dust mite


Fly ash


Red blood cells



ATP synthase


DNA


Atoms of silicon

10^{-2} m — 1 cm

10^{-3} m — 10^6 nanometers

Microworld

10^{-4} m — 0.1 mm, 100 μm

10^{-5} m — 0.01 mm, 10 μm

10^{-6} m — 10^3 nanometers

Visible spectrum

Neutron scattering

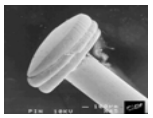
Nanoworld

10^{-7} m — 0.1 μm, 100 nm

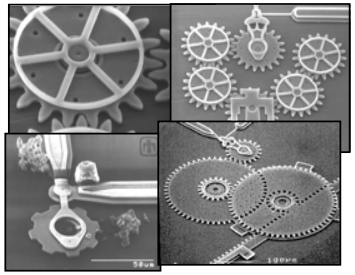
10^{-8} m — 0.01 μm, 10 nm

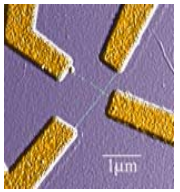
10^{-9} m — 1 nanometer (nm)

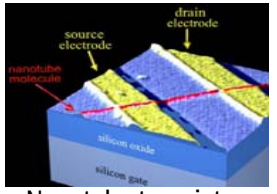
10^{-10} m — 0.1 nm, 1 angstrom


Head of a pin

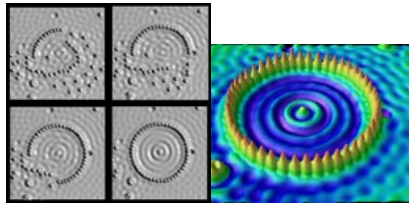
Microelectromechanical Devices

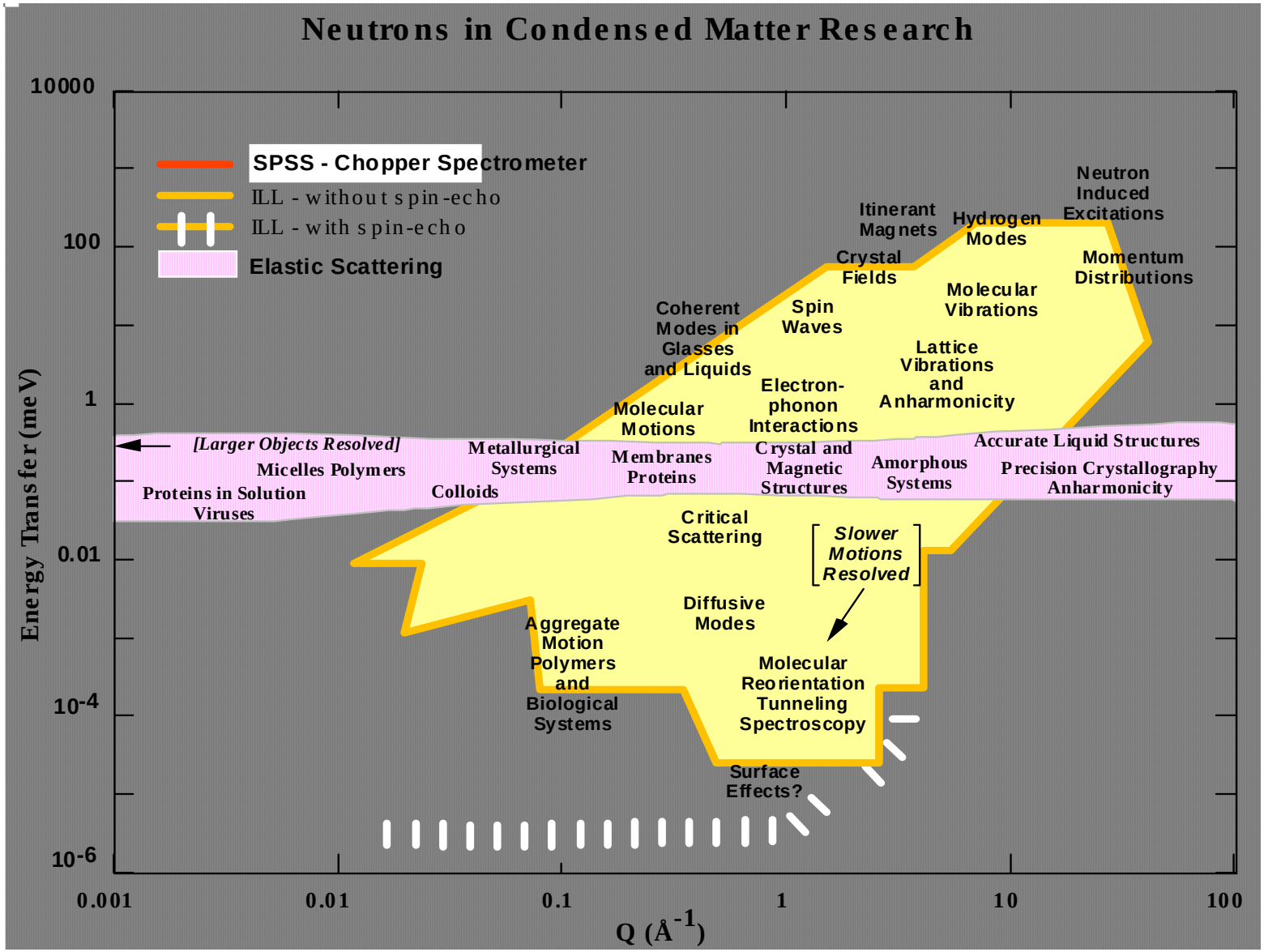



Nanotube electrode



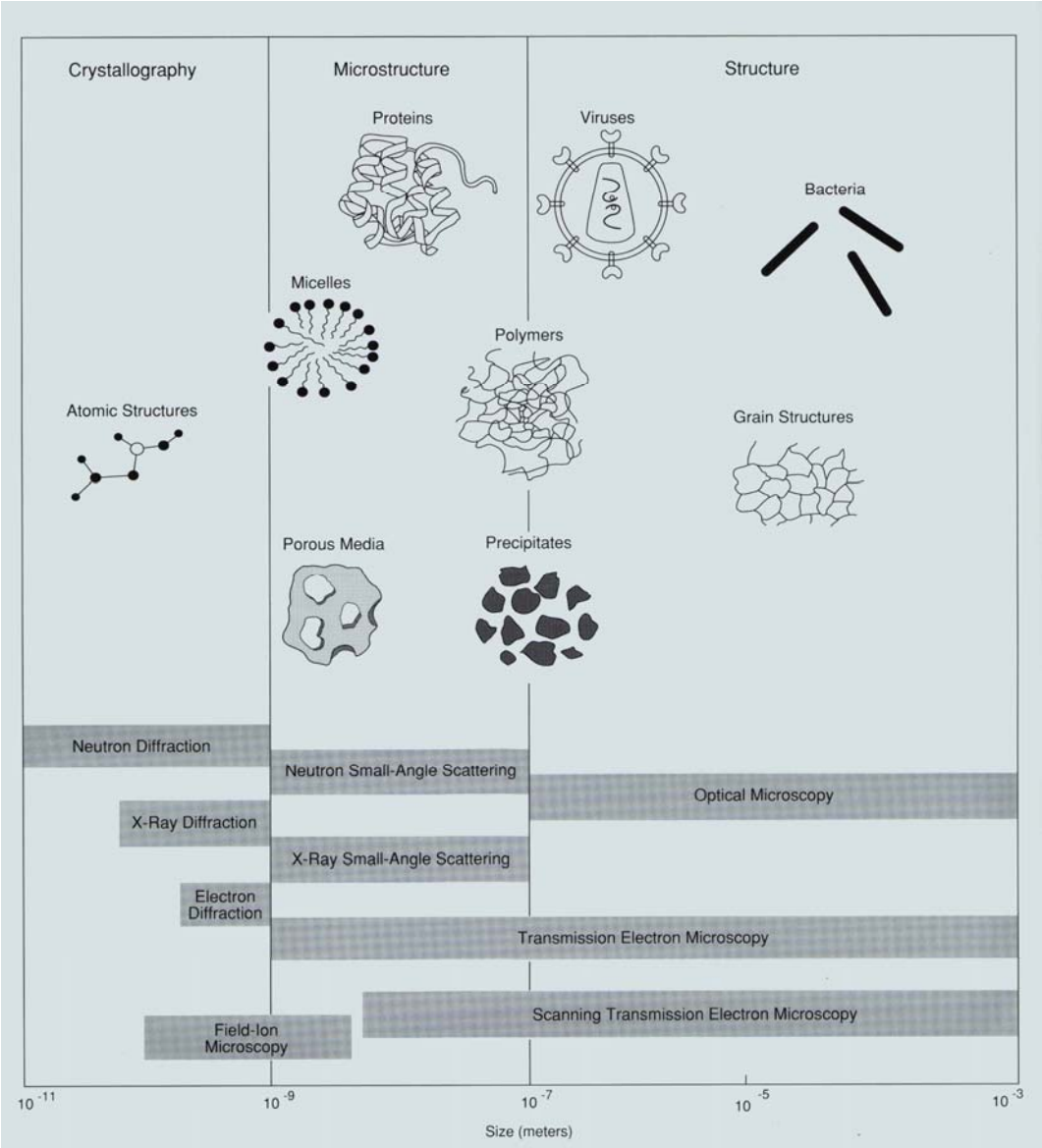
Nanotube transistor


Quantum corral of 48 iron atoms



Neutron scattering experiments measure the number of neutrons scattered at different values of the wavevector and energy transfered to the neutron, denoted Q and E. The phenomena probed depend on the values of Q and E accessed.

Where Does SANS Fit As a Structural Probe?



Typical SANS Applications

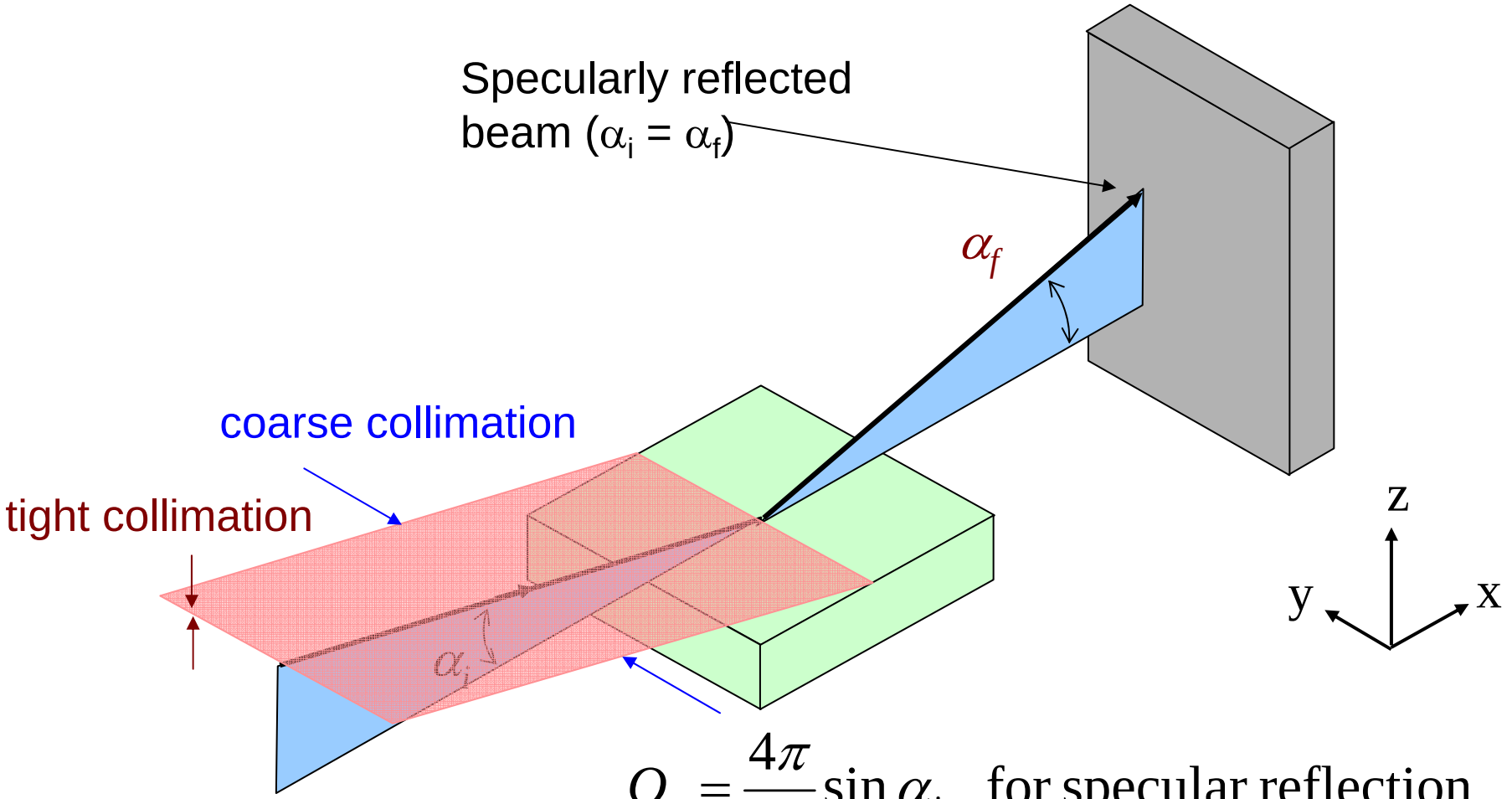
- Biology
  - Organization of biomolecular complexes in solution
  - Conformation changes affecting function of proteins, enzymes, protein/DNA complexes, membranes etc
  - Mechanisms and pathways for protein folding and DNA supercoiling
- Polymers
  - Conformation of polymer molecules in solution and in the bulk
  - Structure of microphase separated block copolymers
  - Factors affecting miscibility of polymer blends
- Chemistry
  - Structure and interactions in colloid suspensions, microemeulsions, surfactant phases etc
  - Mechanisms of molecular self-assembly in solutions

Reflectometry also Measures “Large” Structures

- Neutrons are perfectly reflected by most materials below a critical angle. For Ni (a “good” reflector), γ_c (in °) ~ 0.1 λ (in Å)
- Beyond the critical angle the reflectivity drops quickly, ~ q^{-4}
- If the material is layered, we get interference, just like we get with an oil slick or a soap bubble because neutrons reflected form the various interfaces interfere with each other

- Reflectometry can measure layer thicknesses up to ~ 1000 Å with ~ Å and can detect surface/interface roughness of less than 5 Å

Scattering Geometry for Reflectometry



$$Q_z = \frac{4\pi}{\lambda}\sin\alpha_i \quad \text{for specular reflection}$$

Typical reflection angle between 1° to 5°

Typical Reflectometry Applications

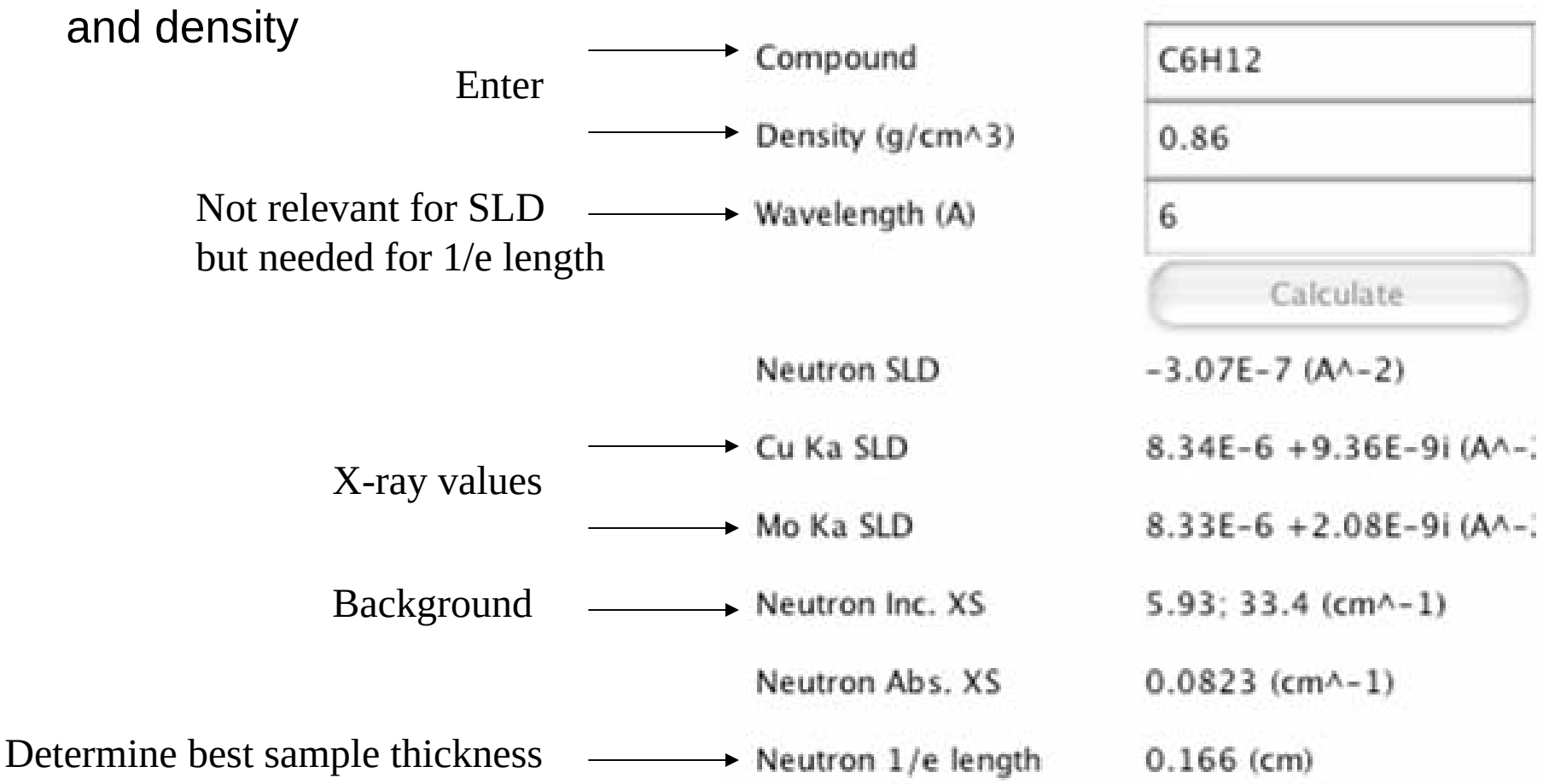
- Biology
  - Interaction of biomolecules with lipid layers
- Polymers
  - Interpenetration of neighboring polymer layers
  - Microstructures of block copolymers
  - Absorption of polymers to solid surfaces
- Chemistry
  - Structure of lipid layers at liquid-air interfaces
- Physics
  - Vector magnetization of various artificial multilayer structures

Scattering Length Density

- Remember $\frac{d\sigma}{d\Omega} = b_{coh}^2 \left\langle \left| \int d\vec{r}.e^{-i\vec{Q}.\vec{r}}\, n_{nuc}(\vec{r}) \right|^2 \right\rangle$
- What happens if Q is very small?
  - The phase factor will not change significantly between neighboring atoms
  - We can average the nuclear scattering potential over length scales ~$2\pi/10Q$
  - This average is called the scattering length density and denoted $\rho(\vec{r})$
- How do we calculate the SLD?
  - Easiest method: go to www.ncnr.nist.gov/resources/sldcalc.html
  - By hand: let us calculate the scattering length density for quartz – SiO_2
  - Density is 2.66 gm.cm^{-3}; Molecular weight is 60.08 gm. mole^{-1}
  - Number of molecules per Å^3 = N = $10^{-24}(2.66/60.08)*N_{avagadro}$ = 0.0267 molecules per Å^3
  - SLD=Σb/volume = $N(b_{Si} + 2b_O)$ = $0.0267(4.15 + 11.6)\ 10^{-5}$ Å^{-2} = 4.21 x10^{-6} Å^{-2}
- A uniform SLD causes scattering only at Q=0; spatial variations in the SLD cause scattering at finite values of Q
- The SLD is used both for SANS and reflectometry

SLD Calculation

- www.ncnr.nist.gov/resources/sldcalc.html
- Need to know chemical formula and density

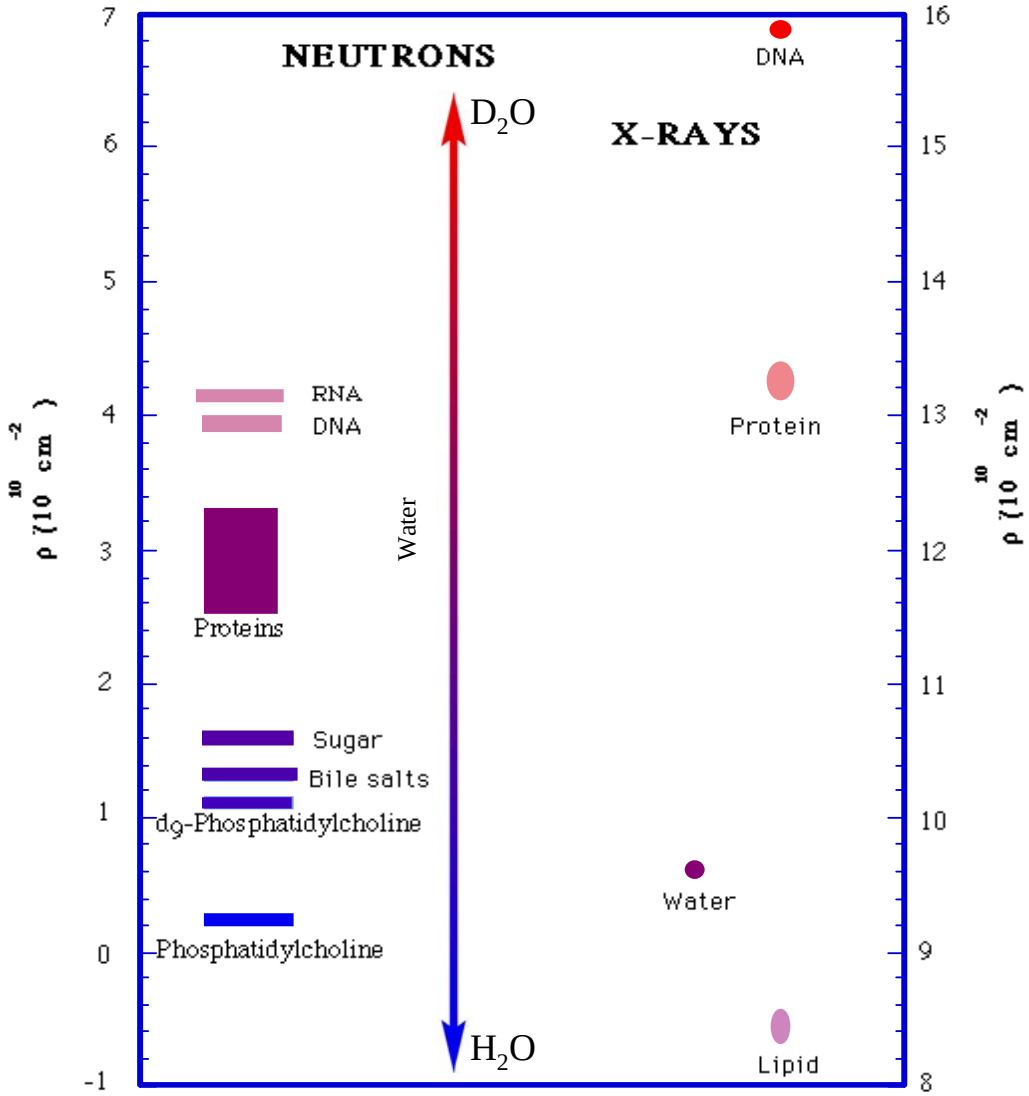


Note units of the cross section – this is cross section per unit volume of sample

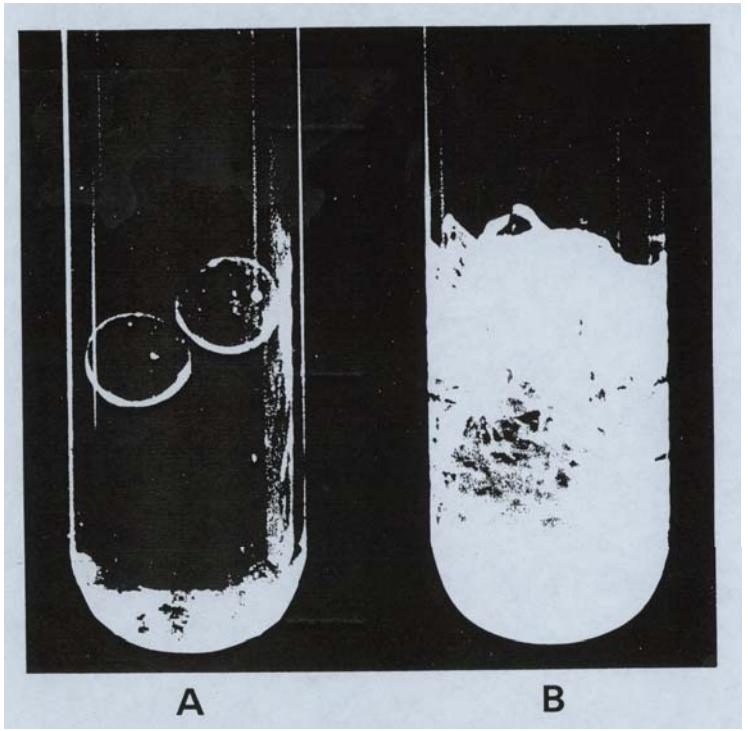
Incoherent Background and Absorption

- In addition to coherent (Q-dependent) scattering that is measured in SANS and reflectometry, neutrons may be scattered *incoherently*
- Incoherent scattering is not directionally (Q) dependent
  - In SANS (or reflectometry) measurements it is a uniform background
- Incoherent scattering arises from two sources:
  - Spin incoherent scattering (the neutron-nucleus state can be singlet or triplet and these have different scattering lengths)
  - Isotopic incoherent scattering
- Look up incoherent scattering lengths (included in NIST SLD calculator – see next VG)
- Neutrons may also be absorbed by some nuclei (Gd, Cd, B..)

Contrast & Contrast Matching

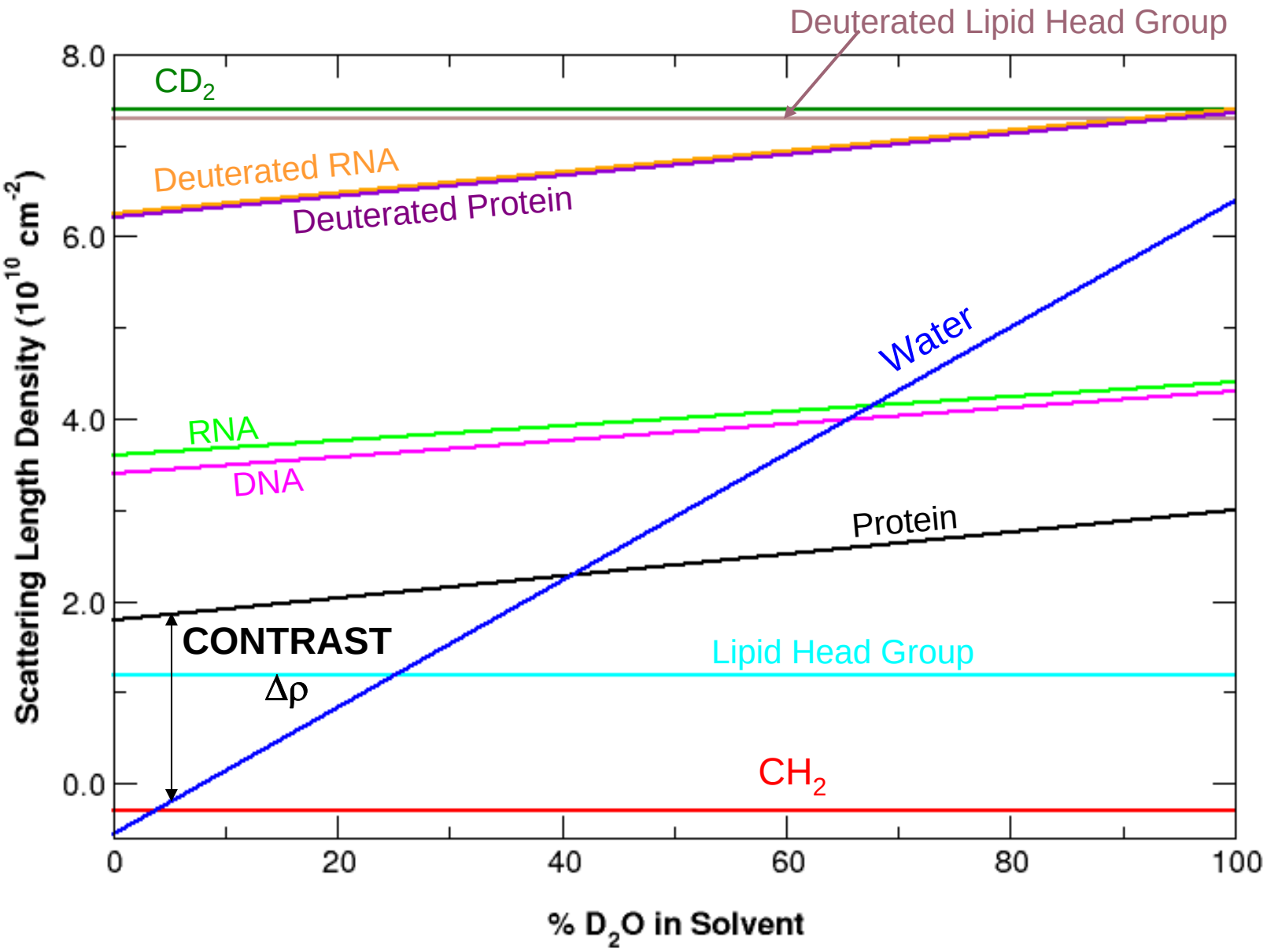


* Chart courtesy of Rex Hjelm



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

Contrast Variation

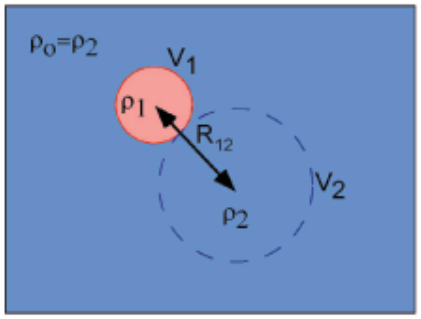


Isotopic Contrast for Neutrons

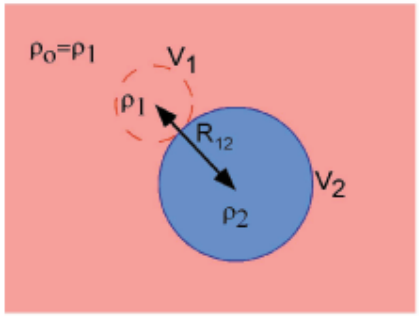
Hydrogen Isotope	Scattering Length b (fm)
^{1}H	-3.7409 (11)
^{2}D	6.674 (6)
^{3}T	4.792 (27)

Nickel Isotope	Scattering Lengths b (fm)
^{58}Ni	15.0 (5)
^{60}Ni	2.8 (1)
^{61}Ni	7.60 (6)
^{62}Ni	-8.7 (2)
^{64}Ni	-0.38 (7)

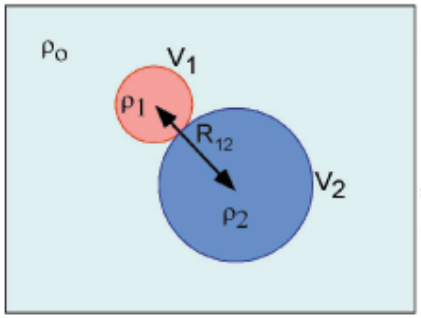
Using Contrast Variation to Study Compound Particles



$$I_1(Q) = (\rho_1 - \rho_2)^2 F_1^2$$



$$I_2(Q) = (\rho_2 - \rho_1)^2 F_2^2$$



$$I_3(Q) - \frac{(\rho_1 - \rho_o)^2}{(\rho_1 - \rho_2)^2} I_1(Q) - \frac{(\rho_2 - \rho_o)^2}{(\rho_1 - \rho_2)^2} I_2(Q)$$

$$= \underbrace{2(\rho_1 - \rho_o)(\rho_2 - \rho_o) F_1 F_2 \frac{\sin(QR_{12})}{QR_{12}}}$$

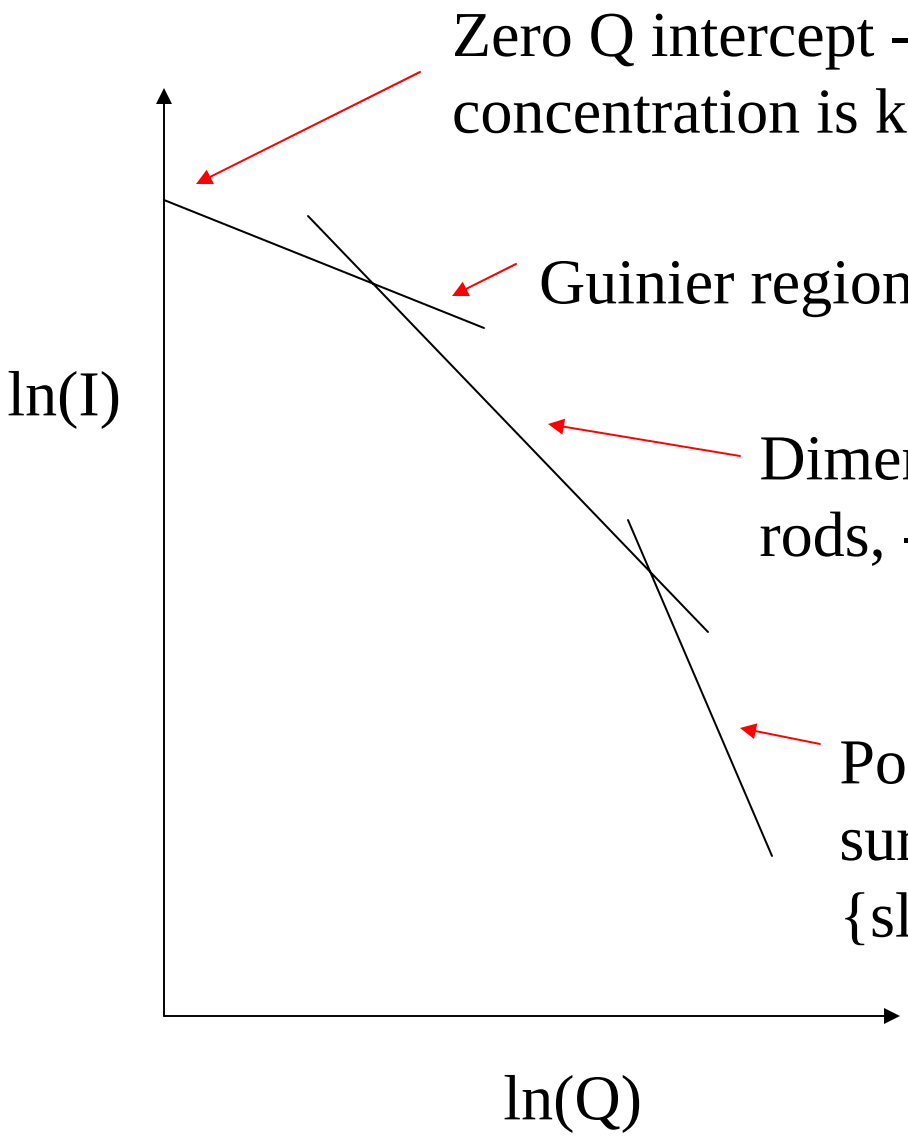
$=0$ at $Q = \pi/R_{12}$

Examples include nucleosomes (protein/DNA) and ribosomes (poteins/RNA)

The same trick can be used in reflectometry to label particular layers or interfaces

Viewgraph from Charles Glinka (NIST)

What can we Learn from SANS?



Zero Q intercept - gives particle volume if concentration is known

Guinier region (slope = $-r_g^2/3$ gives particle "size")

Dimensionality of particle (slope = -1 for rods, -2 for sheets, $-D_f$ for a mass fractal)

Porod region - gives surface area and surface fractal dimension {slope = $-(6-D_s)$}

Sample Requirements for SANS & Reflectometry

- SANS
  - Monodisperse particles less than ~100 nm in diameter
  - Concentration: 1-5 mg/ml at least
  - Volume: 350-700 μl per sample
  - Data collection time: 0.5-6 hrs per sample
  - Deuterated solvent is often highly desirable

- Reflectometry
  - Flat sample with roughness < 0.5 – 1 nm
  - Large enough sample (several square cm – 25 cm^2 is good)
  - Layers less than ~ 50 nm thick

- In both cases, calculate what you expect to see so that you know you will have enough signal (use the NIST website tools)

References

- Viewgraphs describing the NIST 30-m SANS instrument
  - www.ncnr.nist.gov/programs/sans/tutorials/30mSANS_desc.pdf
- SANS data can be simulated for various particle shapes using the programs available at:
  - www.ncnr.nist.gov/resources/simulator.html
- To choose instrument parameters for a SANS experiment at NIST go to:
  - www.ncnr.nist.gov/resources/sansplan.html
- A very good description of SANS experiments can be found at:
  http://www.strubi.ox.ac.uk/people/gilbert/sans.html

END