Characterization of Latex Microspheres Using Ultra-Small-Angle Neutron Scattering

Summer School on Neutron Scattering and Reflectometry From Submicron Structures

NIST Center for Neutron Research
June 10-12, 2004

New Capabilities obtainable using **USANS:**

q range: $3x10^{-5}$ Å^{-1} < q < 0.01 Å^{-1}

Particle Diameter: **0.1 μm < D < 10 μm**

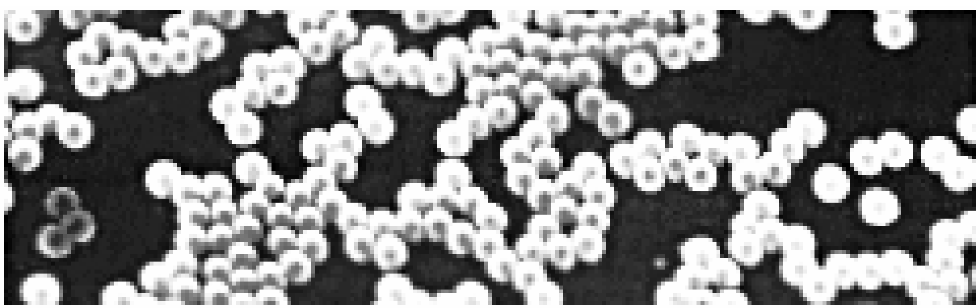
Pores

in rocks, cement, paper, gels, thermal barrier coatings, etc

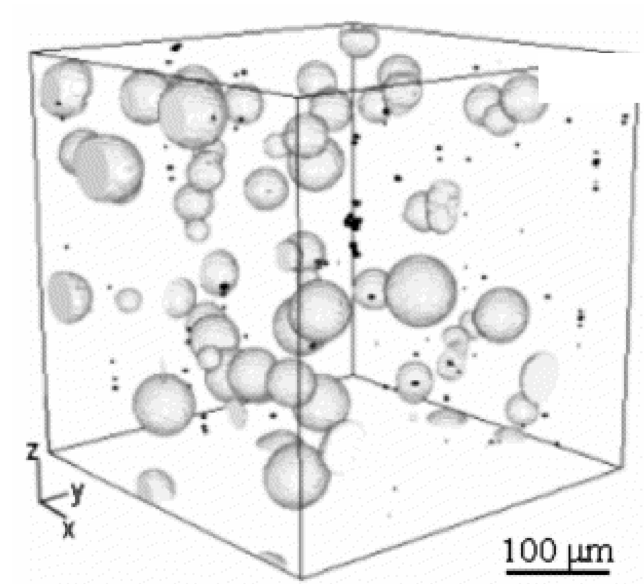
Dispersions

In alloys, ceramics, oil (soot), etc.

Emulsions (oil/water)



Polystyrene Latex Microspheres



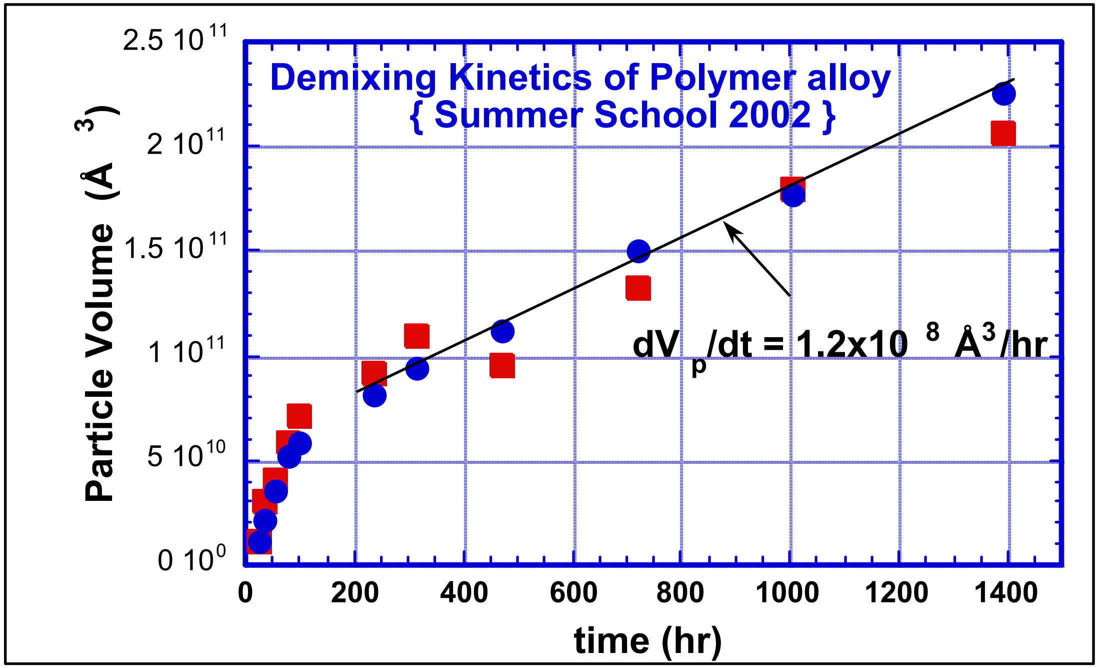
Dispersion in Alloy

Nondestructive Evaluation

SAS allows **nondestructive** *insitu* characterization of samples

Examples:

- Sintering of pores within ceramics or metals
- Second phase nucleation and growth in polymer or metal alloys
- Coarsening of particles during annealing "Ostwald Ripening"



Characterization of Two-Phase Particulate Systems

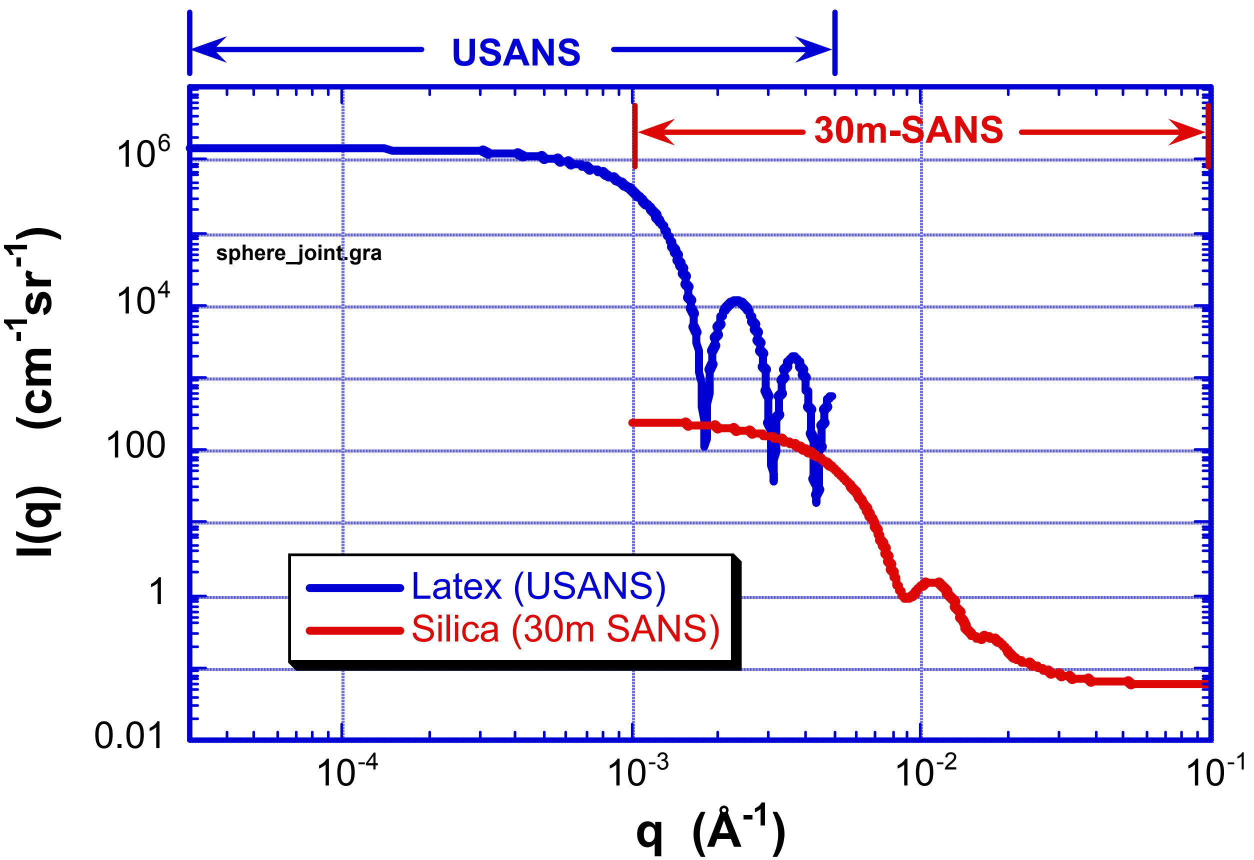
Things we can learn from small angle scattering:

- Radius of gyration from Guinier fit.
- Volume fraction from integration of scattering.
- Mean particle volume from forward cross-section.
- Total particle surface area from Porod’s law.
- Size distribution { if all particles are of the same shape }
- Particle shape { if all particles are of the same size }

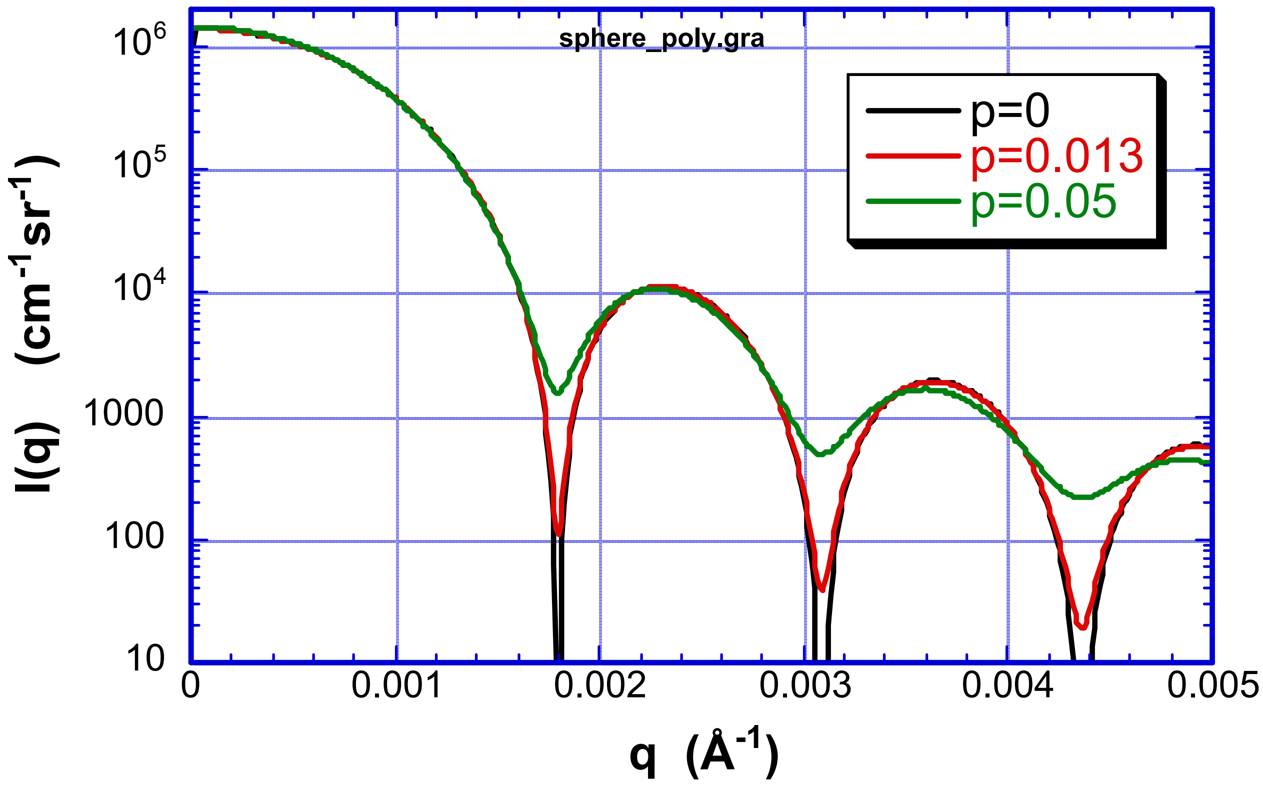
From this experiment, you will learn how we can measure all the above characterization parameters

Experiment Comparison

Value	Silica (30m-SANS)	Latex (USANS)
Diameter	100 nm	500 nm
Volume Fraction	0.05%	1.0%
Size Dispersity	10 %	1.3%

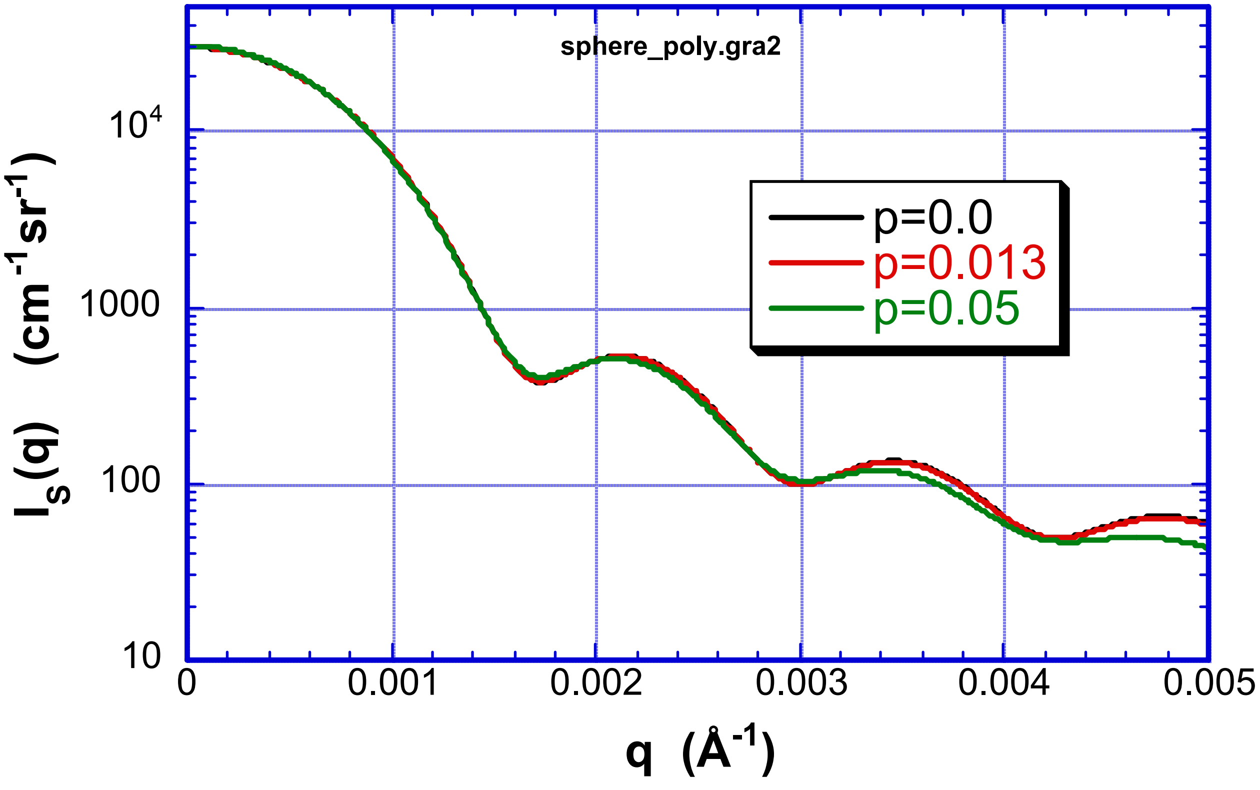


Scattering from 1.0 vol % 500 nm diameter latex spheres in D_2O



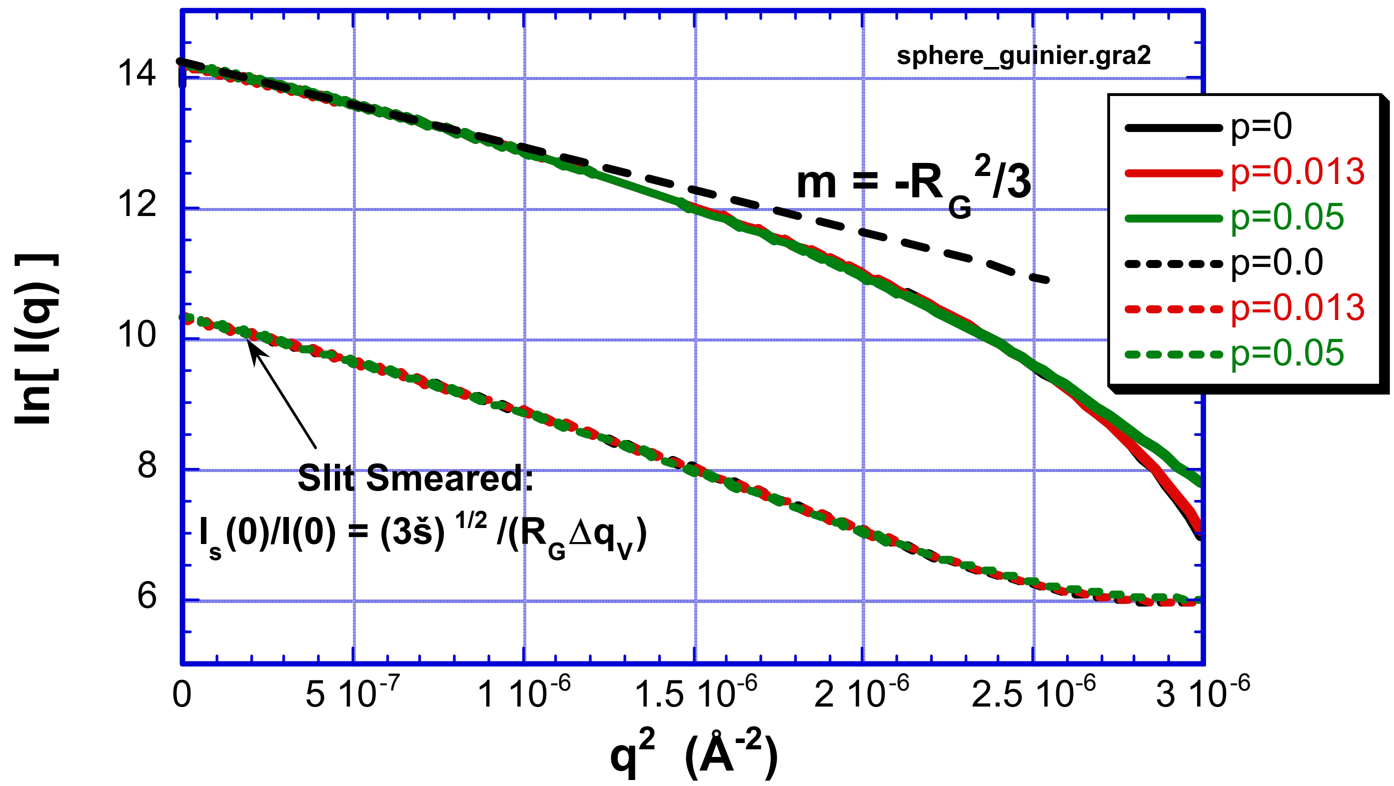
Slit-Smeared

Scattering from 1.0 vol % 500 nm diameter latex spheres in D_2O



Guinier fit to data

$R_G{}^2 = 3D^2/20$



Calculating Volume Fraction from Invariant

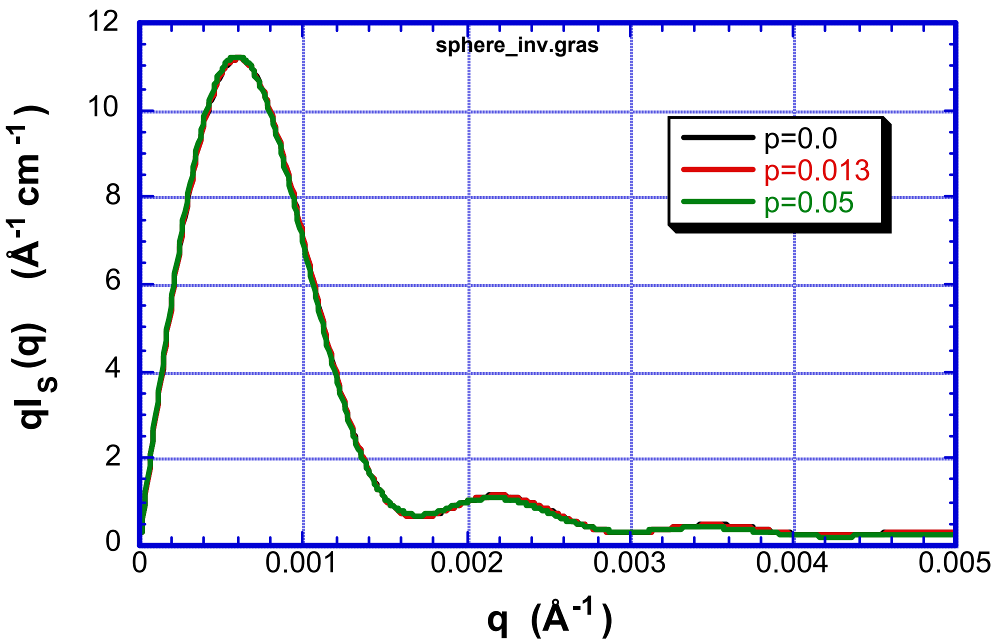
For all two phase systems having *uniform* scattering length densities in each phase, the volume fraction ϕ can be determined from the integration of all scattering

$$\phi(1-\phi) = \frac{Q_I}{2\pi^2 \Delta\rho^2}$$

The **invariant** is determined by

$$Q_I \equiv \int_0^\infty q^2 \frac{d\Sigma}{d\Omega}(q)dq$$

$$= \Delta q_v \int_0^\infty q \frac{d\Sigma_s}{d\Omega}(q)dq$$



Calculating Mean Particle Volume from Forward Cross-Section

For all two phase systems having *uniform* scattering length densities in each phase, the forward cross-section $d\Sigma/d\Omega(0)$ is

$$\frac{d\Sigma}{d\Omega}(0) = \phi < V > \Delta\rho^2$$

where ϕ is the volume fraction, $<V>$ is the mean particle volume. For a distribution of spherical particle sizes:

$$< V >= \tfrac{4}{3}\pi < R^3 >$$

We can use this relation to calculate either ϕ or $<R^3>$, and compare to values obtained from Guinier fit (R) and invariant (ϕ).

Calculating Particle Surface Area from Porod's Law

For all two phase systems having *uniform* scattering length densities in each phase, the asymptotic scattering at large q follows the relation

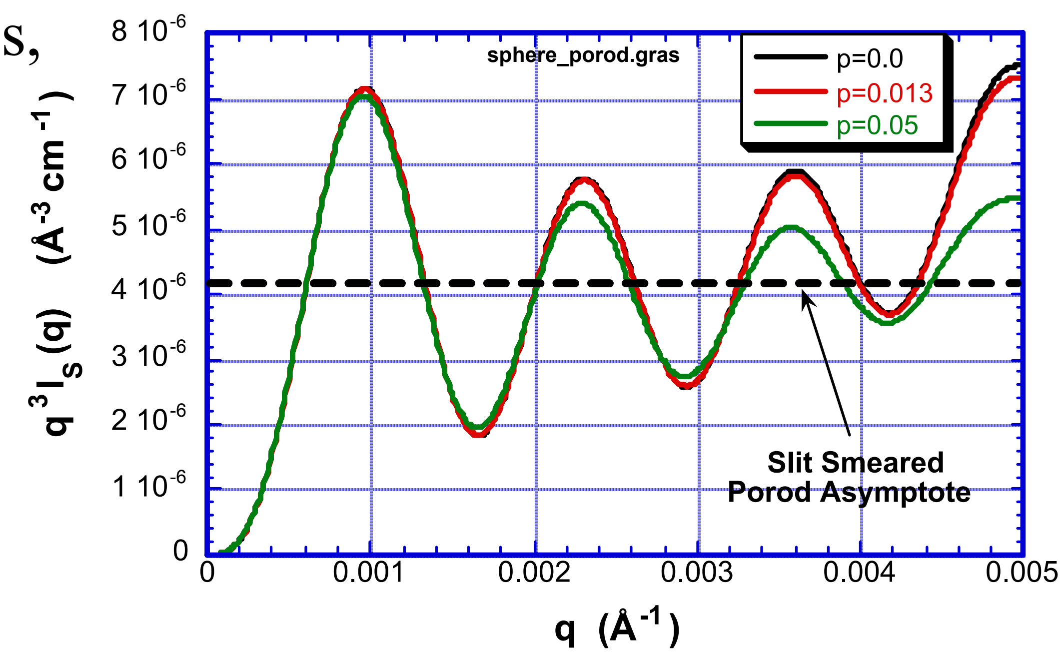
$$2\pi\Delta\rho^2 S_V = q^4 \frac{d\Sigma}{d\Omega}(q) = \Delta q_v q^3 \frac{d\Sigma_s}{d\Omega}(q)$$

Where S_V is the total particle surface area per unit sample volume.

For monodisperse spheres,

$$S_V = \frac{6\phi}{D}$$

Where D is the diameter.



Summary of Tasks

Data Acquisition:

>> Measure ~ 1 vol % latex in D_2O Sample.

>> Measure the empty beam background.

Data Reduction:

>> Run **IGOR Macro** to obtain slit-smeared data $I_S(q)$.

Data Analysis:

>> Fit $I_S(q)$ to Guinier law to obtain mean particle diameter.

>> Determine volume fraction from invariant Q_I

>> Determine volume fraction from $I_S(0)$

>> Determine surface area from large-q Porod asymptote

>> Determine mean **diameter**, **polydispersity** and **volume fraction** from fit of entire curve