Week 11

Characterization of Materials: Scattering

Exercise 1
Answer these questions by true or false:

1. SAXS signal derives from the electron-density contrast of the sample
   True/false

2. The Guinier regime operates at scattering-vector values for which $q > 2\pi/d$, whereby d is the characteristic size of the scattering elements
   True/false

3. In SAXS the field distribution measured is the Fourier transform of the electric field distribution in the exit plane of a sample
   True/false

4. A diffraction peak appears in powder diffraction measurements but never in a small-angle X-ray scattering experiment
   True/false

Exercise 2:
Select the correct answer(s) (more than one answer can be correct)

1. SAXS …
   a. Is used in determining atomic positions in crystals
   b. Involves elastic scattering processes
   c. Is used in determining general sizes and shapes of objects in the few nanometer-to-micron range
   d. Can only be applied for crystalline samples
   e. Can only be applied for amorphous samples

2. What is a structure factor in small-angle scattering
   a. the interparticle interference
   b. the intra-particle interference
   c. provides information on the spacing between scattering objects and their interaction

3. The Porod regime…
   a. Provides information on the shape of the scattering particles
   b. Provides information on the roughness of the interface of the scattering particles in their surrounding medium
   c. Provides information on the overall size of the scattering particles

4. Two samples are investigated at the same beamline using identical incident radiation. Sample A consists of an ensemble of particles with a difference in electron density relative to the medium in which they sit of = 0.2 e/Åand a number density of particles of = 1000/mm. Sample B on the other hand contains identically shaped particles, but with = 0.1 e/Å and = 4000/mm. Which sample produces a stronger SAXS pattern?
   a. Sample A
   b. Sample B

c. Both exhibit the same intensity

Exercise 3: Experimental setup

1. You want to measure bone using small-angle X-ray scattering with an energy of 12.4 keV, the interesting features you expect to be the spacing along the collagen fiber with a d-period of 67 nm, what is the minimum distance you need to place your detector at for measuring the collagen peak when you have a 1mm beamstop blocking the direct beam mounted 1cm in front of the detector?
2. If you want to also measure the diameter of the collagen fiber which you expect to be in the range of 150 – 200 nm, do you need to move the detector closer or further away?
3. How large do you need the detector area to be, to also measure the (002) peak of the hydroxy-apatite (hexagonal crystal structure) and a lattice parameter c = 6.88 Å?

Exercise 4: Scattering of nanoparticles and Guinier law

In Figure 1 you see the small-angle neutron scattering (SANS) curve of a colloidal dispersion consisting of silica spheres coated with octadecane in toluene.

The Guinier approximation is given by the equation: $I(q) \approx I(0)e^{-(1/3)q^2R_G^2}$

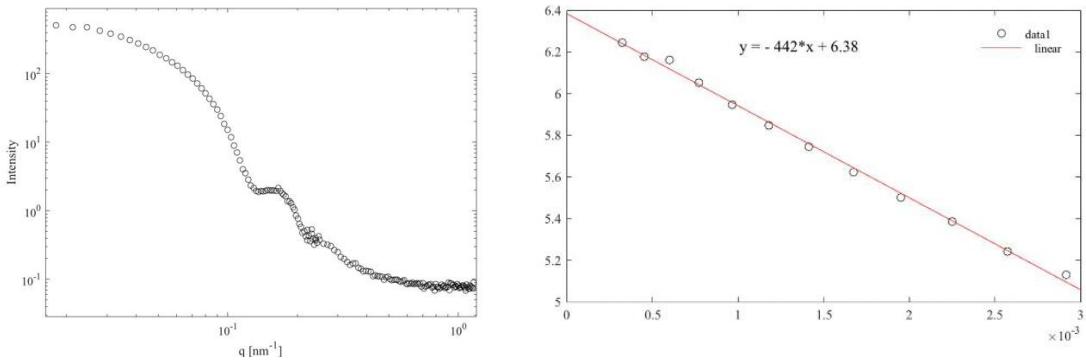
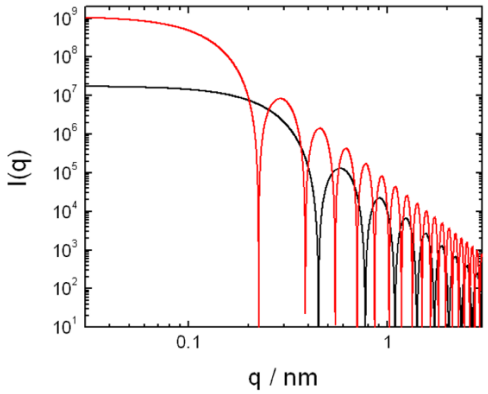


Figure 1. a) SANS curve of colloidal dispersion b) corresponding Guinier plot

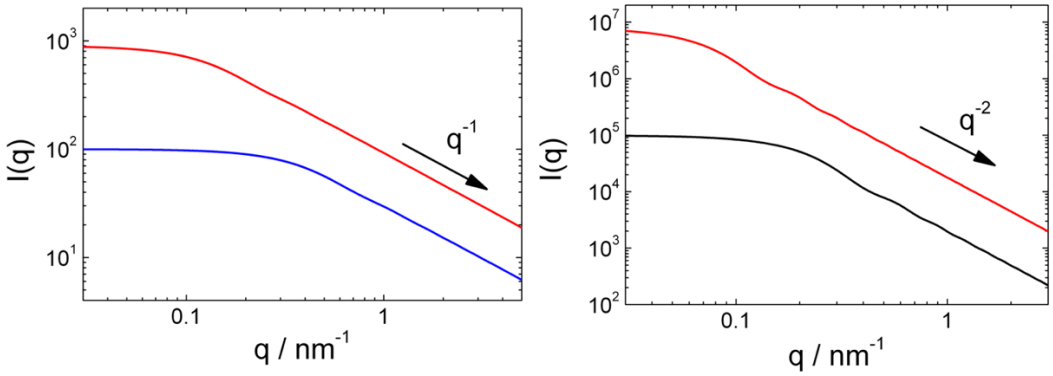
a. Label the x and y axis of Figure 1b, assuming that it is a standard Guinier plot.
b. Determine the radius of gyration using the Guinier approximation
c. Determine the radius of the colloidal particle
d. The colloidal particles have been stored for a long time and they started to agglomerate, which part of the scattering curve would you expect to change and why?
e. Consider Exercise 4 of week 10 where you calculated the expected end to end distance of a polyethyleneoxide assuming random walk. How can you measure this value with scattering?

Exercise 5: Scattering of nanoparticles and form factor

a. The following figure shows the simulated scattering of spheres. Which of the curves revers to larger spheres? Justify your answer



b. Which curves belong to a long cylinder and which belong to a flat disk. Justify your answer



c. Are the red curves in b. characteristic for the larger or smaller nanoparticles compared to black/blue?

Exercise 6: Synchrotron scattering/ diffraction experiments

List three advantages of performing scattering/diffraction experiments at synchrotrons rather than using a lab source