KWS-1 & KWS-2

Small Angle Neutron Scattering

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Manual of the JCNS Laboratory Course Neutron Scattering

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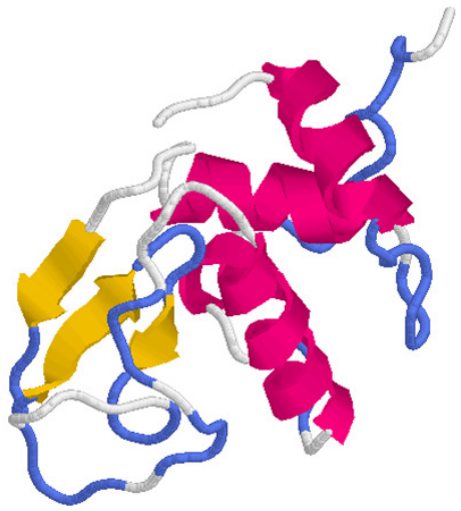

Fig. 1: *Representation of the protein lysozyme, which has a very compact form.*

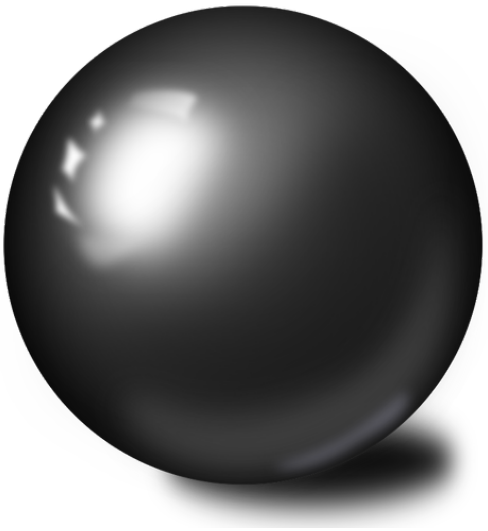

Fig. 2: *A spherical colloidal particle is the second sample of choice.*

1 Introduction

The objective of this lab course is to clarify the essential concepts of small-angle neutron scattering. Structures are only visible by a scattering experiment if there is an appropriate contrast. For neutrons one often uses the exchange of ^{1}H by ^{2}H = D, i.e. deuterium. The chosen contrast of this lab course is achieved by using heavy water (D_2O) as solvent. The materials (solutes) are natural ones having normal protons.

The globular, compact lysozyme (Fig. 1) appears in chicken eggs and has anti-bacterial function. The molecule is charged, which leads to repulsive interactions. So there is a short range order, and the distance between the molecules can be determined.

The other sample is a dispersion of colloidal, spherical particles. The sample will be diluted such that there is no interaction between the particles. So we can determine the size of the particles by the Guinier approximation, and secondly by the first “minimum”. More details are given below.

2 Preparing solutions in Water

A lysozyme solution of 0.02g per ml of water must be prepared. We will weigh 0.02g of Lysozyme and put it into a new Packard glas. With an Eppendorf pipette we will add exactly 1 ml D_2O. These pipettes are extremely accurate with respect to the volume. From the solution about 0.5 to 0.6ml are transferred to Hellma quartz cuvettes, which are 1mm thick. For the later evaluation we need a highly accurate concentration. So all weights need to be written down as exactly as possible.

For the colloidal suspension, we will prepare a 1% solution. So we will weigh 0.01 g or polystyrene particles. Then we will again add 1 ml of D_2O.

If some samples have been already prepared (possibly from earlier groups), we may also use those without own preparation.

3 The Measurement at KWS-1 and/or KWS-2

These two solutions (suspensions) are now being measured in the small-angle neutron scattering instrument KWS-1 (or KWS-2). The wavelength of neutrons is set to 7Å. The collimation is fixed to 8m. The samples are placed as close as possible to the detector, to measure the largest Q values possible. Both samples will be measured at detector distances 2m and 8m. The offset between the sample position and the detector of about 30cm leads to effective detector distances of about 1.7m and 7.7m.

The sample holder will be filled with the two samples. In addition, the empty beam and a plexiglass plate are measured for absolute calibration. For a good statistical measurement the following times are set: 8m detector distance for 20min, and 2m detector distance 10min. The total measuring time for the 4 positions will be about 2 hours. The measurement is typically started before lunch, and can be evaluated in the afternoon. It is quite likely that an internal employee will start separate measurements during the afternoon until the next morning in order to use the valuable measuring time overnight.

4 Evaluation of the Scattering Data: Absolute Calibration

The measured data is raw data at first and describes the intensity on the detector. The data has to be corrected for the effectiveness of the different detector channels. Then the empty beam measurement is subtracted to account for the zero effect of the instrument. Then the intensities are expressed as absolute units using Eq. 5.5 and are radially averaged, because for the isotropic scattering samples, the intensity does not depend on the polar angle. To perform all these steps we will be using a software available in our institute, called QtiKWS. However, since the understanding of the Eq. 5.5, as such, is more important than the exact technical understanding of the evaluation, the results are produced relatively quickly by the software, namely, $\mathrm{d}\Sigma/d\Omega$ as a function of the scattering vector Q for our samples. This data will be provided for the students to do the final evaluation. In the following, this evaluation is described.

5 Evaluation of Lysozyme Scattering Curves

The position of the maximum $Q_{\max}$ provides information on the typical distance of the proteins in solution. This can be calculated to $\ell = 2\pi/Q_{\max}$. Knowing the weight of the protein in water (0.02g/cm^3) there is an alternative way to calculate the average distance. The molar mass of the protein is 1.43×10^4g/mol. The number density of the protein is therefore $n/V = 0.02\text{g/cm}^3/(1.43 \times 10^4\text{g/mol}) = 1.40 \times 10^{-6}\text{mol/cm}^3 = 8.42 \times 10^{-7}\text{Å}^{-3}$. For a simple cubic packing the typical distance is given by $\ell = \sqrt[3]{V/n}$. For a hexagonal close packed lattice the typical distance is $\ell = \sqrt[6]{16/27}\sqrt[3]{V/n}$. This distance is the minimum distance of the planes

important for the scattering experiment, and the next neighbor distance of the hexagonal c.p. lattice is $\sqrt{3/2}\ \ell = \sqrt[6]{2}\sqrt[3]{V/n}$. Both calculated distances of the cubic and hexagonal structure are to be compared with the measured one.

6 Evaluation of the Scattering from Colloidal Particles

In a first step we have to prepare the scattering data for background subtraction. We plot the original data of the two detector distances in a log-log plot, i.e. $\log_{10}(d\Sigma/d\Omega) \rightarrow \log_{10} Q$. After this, we will see a plateau at high Q which indicates the constant incoherent scattering. Taking the average of the last (say 10) points will give us the estimate of the background. A new column with the background subtracted will be generated for the 8m and 2m measurements. Finally, the two data sets should be combined to yield a single data set.

Now, we will aim at the overall appearance of the colloids, i.e. we will determine the particle dimension. For this purpose the Guinier approximation can be applied. The general appearance of the Gunier scattering law was already given in eq. 5.35 and reads:

$$\frac{d\Sigma}{d\Omega}(\mathbf{Q}\rightarrow 0) = \frac{d\Sigma}{d\Omega}(0) \cdot \exp\left(-\frac{1}{3}Q^2R_g^2\right) \tag{1}$$

For this purpose we plot the logarithm of the background corrected intensity against the square of the scattering vector, i.e. $\ln(d\Sigma/d\Omega) \rightarrow Q^2$. The highest Q will lead to large values that we are not interested in. So the plot has to be truncated to the rather small Q, say $Q^2 = 0..4 \times 10^{-4}\text{Å}^{-2}$. Here, we do a linear regression and take the slope S as a result only. It has the units Å^2. From this we can calculate the radius of gyration using $R_g = \sqrt{-3S}$. Then, the relation to the full radius is used, i.e. $R = \sqrt{\frac{5}{3}}R_g$.

If the concentration was too high, or there are weak electrostatic repulsive interactions, we will try to apply the structure and formfactor fit. The presence of a structure factor can be seen by lower scattering intensities at smallest Q compared to slightly higher Q, i.e. there is a maximum in the scattering that would not be there without interactions. We will then have:

$$\frac{d\Sigma}{d\Omega}(\mathbf{Q}) = \frac{d\Sigma}{d\Omega}(0) \cdot S(Q) \cdot F(Q) \tag{2}$$

For the formfactor, we will take the ideal sphere expression:

$$F(Q) = \left[3\frac{\sin(QR) - QR\cos(QR)}{(QR)^3}\right]^2 \tag{3}$$

For the two particle structure factor we will then find the approximation:

$$S(Q) = 1 - 8\phi \cdot \left[3\frac{\sin(2QR) - 2QR\cos(2QR)}{(2QR)^3}\right] \tag{4}$$

The full expression will then be fitted to the whole scattering curve, and the radius will be one parameter that we obtain.

Then, we will try to read the Q-value of the first “minimum”. Due to smearing effects of the reolution and polydispersity, the “minimum” may only weakly be formed as a weak dip. The radius is now obtained from $R = 4.493/Q_{\min}$.

The last evaluation method will be the Porod scattering at high Q. It applies for any shape of particles with a smooth surface. Here the many heavily oscillating fringes are smeared out, such that the scattering has a simple power law:

$$\frac{d\Sigma}{d\Omega}(\mathbf{Q}) = P \cdot Q^{-4} = \frac{9}{2} \cdot \frac{d\Sigma}{d\Omega}(0) \cdot (QR)^{-4} \tag{5}$$

So, we need to determine the forward scattering from our previous evaluations, and read the coefficient P from a log-log plot of the intensity versus the scattering vector Q.

After we have obtained three different versions of the radius R, they should be compared and discussed. The imperfections of the different evaluations should have become clear by the use of the different approaches.

7 Preparatory Exercises

(I) Lysozyme in D_2O

The first sample of the Neutron Lab Course at the SANS instrument KWS-1 (KWS-2) will be Lysozyme in heavy water (D_2O). This protein is rather globular (diameter ca. 5 nm). The Coulomb interactions of this charged molecule lead to liquid-like short-range-ordering. This will be observed in the SANS scattering experiment by a correlation peak. Simple estimations will be made now:

1. Give the connection between the number density ϕ and the unit cell parameter assuming a simple cubic lattice!
2. The chemical concentration c is usually given in g/L or mg/ml. The molar mass of the molecule is 14307g/mol. What is the connection between the chemical concentration and the number density?
3. The correlation peak appears at a scattering vector $Q_{\max}$. How would it relate to the unit cell parameter of a simple cubic lattice? What is the dependence of $Q_{\max}$ as a function of the chemical concentration c?
4. Please rationalize the relations of the hexagonal close packed lattice with respect to the cubic packing! The spacing of the planes is shorter by a value of rougly 0.916 (larger Q value compared to cubic). The nearest neighbor has a larger distance of ca. 1.122 times the cubic packing.

(II) Colloidal Dispersion

The different approaches for the size determination is the main subject here.

1. The Appendix B derived the Guinier scattering law for any shape of particles while in the main manuscript the first application was the compact sphere. What is the general meaning of the radius of gyration R_g? What is the general understanding for other shapes of particles?
2. At large Q we observe a constant background from incoherent scattering. The hydrogen atom has a incoherent cross section of 80×10^{-24}cm^2, and the deuterium atom 2×10^{-24}cm^2. The concentration of hydrogen from the particles is roughly 100 times smaller than the concentration of deuterium from the heavy water. On the basis of these numbers estimate the ratio of background from the particles and the solvent!
3. We came across the Porod scattering at high Q for the spheres with smooth surfaces. The original expression for the scattering would describe heavy oscillations at high Q. Why does this part of the scattering curve smear out such that a simple power law is remaining? This reason holds for any type of power law at high Q.

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