Experimental report

15/08/2022

Proposal: 1-20-69 **Council:** 4/2021

Title: Developing new analysis methods for fixed window scans for soft colloidal suspensions

Research area: Soft condensed matter

This proposal is a resubmission of 1-20-64

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Samples: D2O
Myoglobin in D2O
bovine serum albumin in D2O

Instrument	Requested days	Allocated days	From	To
IN16B	4	3	10/09/2021	13/09/2021

Abstract:

The instrument design of IN16b allows to measure specific energy transfers with a high energy resolution by so-called elastic and inelastic fixed window scans (E/IFWS). This measurement method permits to reduce the acquisition time from the several hours needed for a single full spectrum down to less than a minute for an E/IFWS point. So far, this method has mainly been applied to polymer and glass systems as well as hydrated protein powders.

Here we propose to acquire accurate calibration data to extend this method to suspensions of soft colloids such as proteins.

In this way, the method can be applied to kinetically evolving systems such as proteins crystallizing from solution or assembly processes as e.g. in pathological protein aggregation diseases.

The challenge for these samples consists in the superposition of different contributions to the measured signal - notably from the colloid global and internal diffusion and from the solvent - which can only be achieved by accurate calibration data from well characterized systems. These systems would not by themselves make the case for a college 8 or 9 proposal, but the data will be crucial to proceed with the E/IFWS method.

Experimental Report: Beamtime IN16b exp 1-20-69
07/09/21-10/09/21

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1 General Comment

The beamtime was combined with Experiment 8-04-923. Calibration measurements were performed with the settings necessary for this beamtime and shared between the two beamtimes to optimize measurement time.

2 Performed Measurements

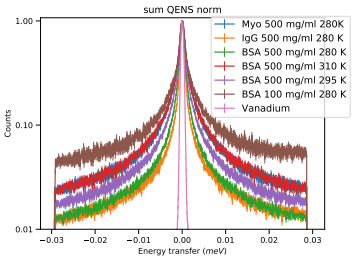
All samples were measured the same way: To be able to have a standard analysis, full QENS spectra (sinusoidal speed profile for the monochromator) were collected for in total 6.5 hours with saving the data every 5 minutes. Given the high saving rate of the spectra, it will be possible to bin data together in such a way that the errorbars are similar for different resolutions convoluted with theexperimental spectra. In addition, all samples were measured with fixed energy transfers (i.e. accurate velocity profile for the monochromator) with the measurement details indicated in Table 1. Cylindrical sample holders were used. The Calibration measurements (vanadium, empty can, D_2O) were performed at $T = 280$ K. Several protein solution samples were prepared and measured with the same protocol: polyclonal Immunoglobulin IgG $c_p = 500\frac{\text{mg}}{\text{ml}}$ ($T = 280$ K), Myoglobin $c_p = 500\frac{\text{mg}}{\text{ml}}$ ($T = 280$ K), Bovine Serum Albumin $c_p = 100\frac{\text{mg}}{\text{ml}}$ ($T = 280$ K), Bovine Serum Albumin $c_p = 500\frac{\text{mg}}{\text{ml}}$ ($T = 280\,\text{K}; 295\,\text{K}; 310\,\text{K}$). All given concentrations are nominal concentrations.

3 Data Reduction and first Analysis

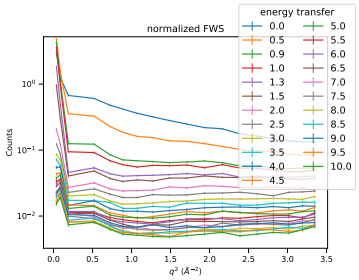
Data reduction was performed automatically using the Mantid nightly version 6.1.20210826.1625 via an ILL visa instance allowing to reduce also the data from the diffraction tubes. Figure 1a shows the full QENS spectra sumed over q for

Table 1: Settings for the measurements of the FWS with the energy transfer ΔE and the acquisition time t.

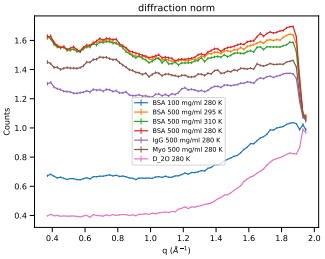
ΔE (μeV)	0	0.5	0.9	1	1.3	1.5	2	2.5	3	3.5	4	4.5
t (min)	0.5	0.925	1.265	1.35	1.605	1.775	2.2	2.625	3.05	3.475	3.9	4.325
ΔE (μeV)	5	5.5	6	6.5	7	7.5	8	8.5	9	9.5	10	
t (min)	4.75	5.175	5.6	6.025	6.45	6.875	7.3	7.725	8.15	8.575	9	



(a) Full QENS spectra collected during the beamtime. All spectra are sumed over q.



(b) Vanadium normalized FWS of polychlonal IgG as a function of q^2 for different energy offsets as indicated in the legend.



(c) Diffraction data from the different samples measured during the beamtime.

Figure 1: Reduced example data. All shown data contain error bars. The high data quality can be observed.

the different protein samples as well as the vanadium measurement. Since the volume fraction for the protein samples at $c_p = 500\ \frac{\text{mg}}{\text{ml}}$ between the different is comparable, the difference in the linewidth of the different protein signals can directely related to the different protein radii, which influence the diffusion coefficient. For BSA, the crowding effect as well as the temperature dependence can be observed for the different measurements.

Figure 1b displays the FWS as a function of q^2 of polyclonal immunoglobulin for different energy transfers normalized by the elastic vanadium scan to correct for the detector efficiency.

Figure 1c shows the diffraction data of the samples measured during the beamtime. Also here, the data is normalized by the vanadium scan. The high protein concentration and the therefore implied high hydrogen content does add a significant background to the diffraction data. For the BSA solution with $c_p = 100\ \frac{\text{mg}}{\text{ml}}$, the solution, the difraction signal is mainly dominated by the solvent, while for higher protein concentrations an additional frature is appearing around $q \approx 0.7\ \text{Å}^{-1}$.