ESRF	**Experiment title:** Collective dynamics of ordered polyelectrolyte solutions studied by means of X-ray photon correlation spectroscopy	**Experiment number**: SC-552
Beamline: Troika	**Date of experiment**: from: 6/5/99 to: 18/5/99	**Date of report**: 5/8/99
Shifts: 36	**Local contact(s)**: Gerhard Gruebel	*Received at ESRF:*

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Report:

The aim of the experiment was to study the protein Ferritin in aqueous solutions of different salt and protein concentrations. Furthermore, we planned to spend few shifts on test measurements (XPCS) of different DNA solutions (high molecular weight).

Previous SAXS studies had shown that Ferritin could be a promising biological molecule for investigations of dynamics by means of X-ray-photon-correlation spectroscopy. This technique requires high scattered intensity. Ferritin molecules consist of a strongly scattering iron core inside a protein shell.
Fig. 1 shows SAXS curves obtained from solutions of Ferritin in salted water. Using 200 mM aq. NaCl solution the forces between the charged proteins are screened. A fit with a spheric symmetric form factor gives the size of the iron core (ca. 7.5 nm in diameter) and the thickness of the protein shell (ca. 2.5 nm in diameter). Furthermore, our data show high scattered intensity, especially for small scattering vectors. On the other hand towards higher q's the intensity decreases rapidly according to the size of the molecule.
Restricting ourself to small scattering vectors we investigated the dynamics of several solutions of Ferritin in water and glycerol. Fig. 2 gives a typical correlation function of Ferritin in pure Glycerol at 20^0 C . An exponential fit to the data gives the decay time τ=9,1 seconds. This value is three orders of magnitude higher than decay times of the slow mode of aq. Ferritin solutions measured by means of dynamic light scattering. The viscosity of

Glycerol exceeds the viscosity of water by three orders of magnitude, too. But there remains a contradiction to solve in future, because the X-ray's scattering vector (ca. $2 \cdot 10^{-3}\,\dot{A}^{-1}$) was more than two times bigger than scattering vectors available by light scattering.
We postponed further investigations at different temperatures and higher q's, because of experimental difficulties concerning low flux of the monochromatic coherent beam.

From preceding SAXS experiments we knew about a peak arising with decreasing salt content of the solvent. This peak is due to weak ordering of the charged protein molecules and the peak's position changes with Ferritin concentration as shown in Fig. 3.
We tried to investigate dynamics at the peak position, but the scattered intensity was not high enough for XPCS measurements. Fig. 3 shows that the peaks are quite broad, even in solution with very low salt concentration. Thus, scattered intensity at peak position outranges scattering from high-salt solutions (Fig. 1) only by a factor of 10. With respect to the scattered intensity at small angles, there's a lack in intensity by factors of 5-10.
Instead of dynamic measurements we performed systematic studies of peak position and height for different protein concentrations and salt contents.
This work was fundamental preparation for future measurements which can be done when the new beamline Troika C is finished. This beamline will improve the available intensity by a factor 100. The resulting intensities will be sufficient for XPCS measurements at scattering vectors in the vicinity of the Ferritin's peak.

We have also performed preliminary measurements on DNA. To have enough intensity for XPCS measurements we first measured DNA of high molecular weight (10^6 d < M_W < 10^8 d). We restricted our measurements to small q values. Fig. 4 shows a typical correlation function ($M_W \approx 10^7$ d, q= $2{,}7 \cdot 10^{-3}\,\dot{A}^{-1}$). The fit shows two processes with decay times of 23 ms, respective 770 ms.
Though it was not possible to increase the scattered intensity by labelling with metallic ions, we did first steps in labelling DNA with Ferritin. Fig. 5 shows a correlation function of a mixture of Ferritin and DNA. There is a wide field for future work, for example systematic studies at different Q values, studies of monodisperse solutions and measurements at Q values of the peak position.

Figure 1

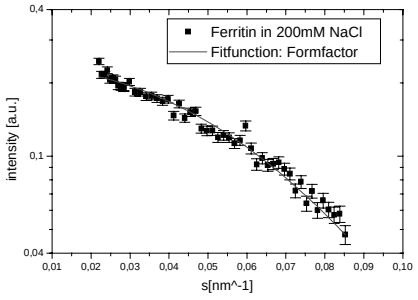

Figure 2

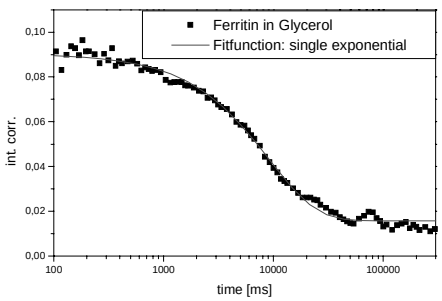

Figure 3

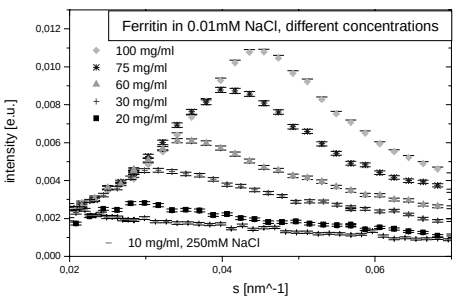

Figure 4

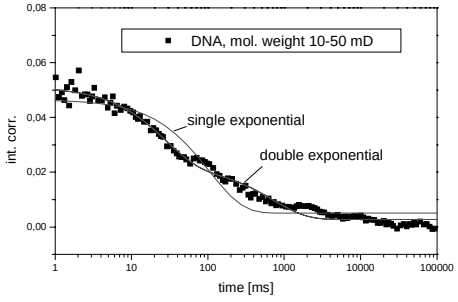

Figure 5