

**Experiment title:**

Study of size and shape fluctuations in micellar systems of block-copolymers by means of x-ray photon correlation spectroscopy

**Experiment number:**

SC-308

SC-308

**Beamline:**

ID10

**Date of experiment:**

from: 9.4.97

to:

16.4.97

**Date of report:**

20.2.98

**Shifts:**

11

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**Report:**

The aim of the experiment was to measure the dynamics of dilute and concentrated solutions of blockcopolymer micelles by x-ray-photon correlation spectroscopy.

In order to ensure sufficient scattering intensity we used blockcopolymer micelles labeled with gold particles in the core [1]. The polymer used is Polystyrene-block-Polyvinylpyridine, both blocks contain 300 monomer units, in toluene these polymers form micelles. Precursor micelles filled with  $\text{HAuCl}_4$  are referred to as sample 1. After chemical reduction the micellar core contains very monodisperse gold particles (sample 2). An additional controlled aggregation process leads to micelles containing several gold particles leading to even higher scattering intensities (sample 3).

Fig. 1 shows SAXS curves obtained from samples 1-3. The XPCS measurements had to be restricted to the micelles containing the final particles (sample 2 and 3), since strong radiation damage by the wide bandpass beam at ID10 was observed. After switching on the beam a strong increase in the scattered intensity on a time scale of minutes was observed (Fig. 2). A

similar effect could also be seen on the micelles containing gold particles, but here it was weaker. Correlation functions obtained on sample 3, containing the aggregated gold particles, are shown in Fig. 3. Here we were able to observe the expected  $q^2$ -dependence of the relaxation rates in the low  $q$  limit (Fig. 4). The apparent hydrodynamic radius determined from the correlation function amounts to 214 nm, a value which is considerably higher than the one observed by light scattering in a similar dilute solution. This decrease of the cooperative diffusion coefficient with increasing concentration could indicate a substantial influence of polymer interaction in the solution. For a final conclusion the data base has to be extended, preferably on a sample which is less sensitive to radiation damage.

Reference:

J. P. Spatz, S. Mößmer, M. Möller, Chem. Eur. J. 2, 1996, 1552.

