Anomalous Small-Angle X-ray Scattering Measurements on Polyacrylates Surrounded by TI⁺ Counterions

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Quantitative anomalous small-angle X-ray scattering turned out to be an excellent tool for the structural analysis of the conformation of macro-ions under the influence of surrounding counterions like Sr [1], Br [2] or Pb [3]. Recently ASAXS measurements on polyacrylates surrounded by Tl counter-ions have been performed at energies in the vicinity of the Tl-L_{III} absorption edge at 12658 eV. The polymer concentration in solution is kept constant with 1 wt% corresponding to a Tl⁺ concentration for the fully neutralised poly acrylic acid of 110 mMol. The thallium salts were prepared by hydrolysis of thallium I ethanolate with ice. To avoid disproportionation of the thallium hydroxide, neutralisation of the acrylic acids was performed directly afterwards. The samples were freeze dryed to remove the alcohol, and re-dissolved in water. Due to the disproportionation sensitivity of thallium I hydroxide the reference was neutralised with acetic acid previous to the freeze drying. In order to perform quantitative Raman corrections aqueous solutions with precisely the same Tl⁺ concentration have been prepared and measured together with the polymer samples. The samples were degased and filled in Hilgenberg mark tubes of 2 mm diameter no longer than two days in advance of the measurement.

The separation of the pure-resonant scattering contribution of the Tl⁺-ions especially in the q-range up to 10 nm⁻¹ in combination with precise corrections due to resonant Raman scattering, facilitates the direct analysis of the fluctuation scattering of the counter-ions indicating weak coupling of the counter-ions to the macro-ions. The mixed-resonant scattering exhibits experimentally the expected mean-field behaviour predicted by an earlier theoretical MD-study [4]. The experiments were performed at the B1 beam line (former JUSIFA) at HASYLAB, DESY, Hamburg, using a Pilatus 1M detector.



Figure 1: Total scattering curves of polyacrylates surrounded by Tl^+ counter-ions measured at 3 energies in the vicinity of the L_{III} absorption edge of thallium at 12658 eV.

References

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