



	Experiment title: Structure of the Pristine Oil/Water Interface: Surfactant-Free Emulsions	Experiment number: SC1841
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Report:

The aim of the experiment was to measure, using anomalous small angle x-ray scattering, the distribution function of the charge balancing ions (chosen to be Rb^+) at the interface of the recently discovered (Angew. Chem. Int. Ed 2004, 43, 3568- 3571 James K. Beattie and Alex M. Djerdjev) oil in water emulsions produced without surfactants. Measurements were performed in the vicinity of the Rb K-edge (15.2 keV) and data were analysed using core-shell model involving emulsion droplets and the shell of Rb^+ ions.

The experiment proved to be at the margin of sensitivity and stability for the system and the instrument but an upper level for the concentration of the ion sheet and its dimension have been set and an energy dependent crosssection was found. The polydispersity of the emulsion was far greater than expected and as a result the fringes in the form factor could not be observed. The actual size of the emulsion was determined by USAXS and a fit to polydisperse sphere model. Figure 1 shows typical USAXS plots for selected emulsions studied and the corresponding fits. Figure 2 depicts the combined SAXS and USAXS results for emulsion 12.1.

Much was learned about the emulsion physical chemistry in a series of concurrent experiments. The detectivity of the counterions can be improved by increasing the surface charge density and will be proposed.

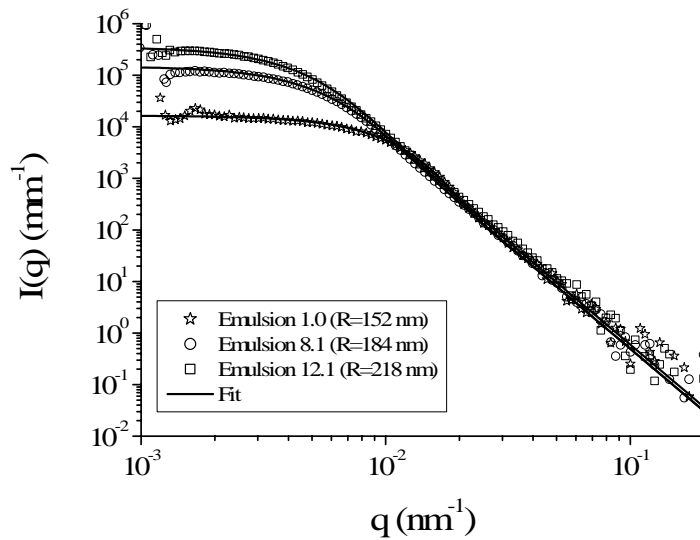


Figure 1: USAXS intensity for selected emulsions indicating the radius (R) and broad size distribution.

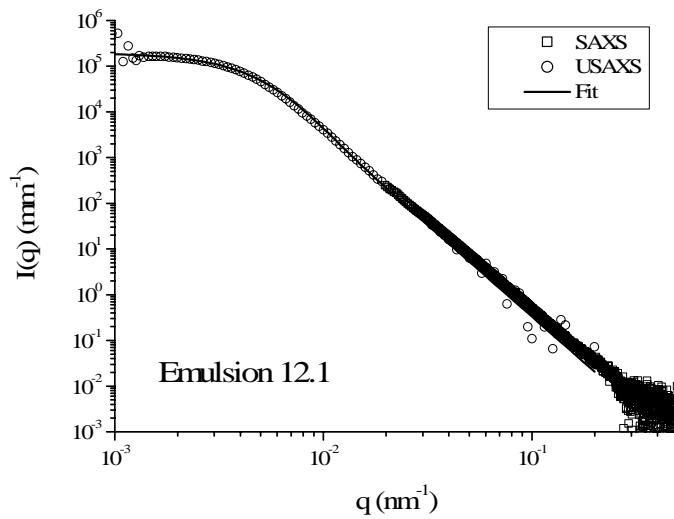


Figure 2: SAXS and USAXS intensities for emulsion 12.1. The slight mismatch in the absolute intensity level is because the USAXS measurements were done many hours (> 9 h) after the SAXS measurements.

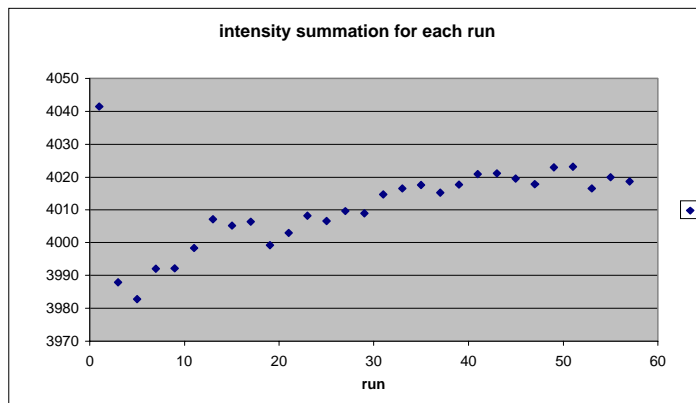


Figure 3: Stability in the absolute scattering intensity obtained by summing all q channels in subsequent runs over 30 minutes. This stability approaches 1/1000 and typical ASAXS scan spans less than 10 minutes.

The experiment required precision in the absolute integrated scattering intensity and stability of the

emulsion and measuring system to fractions of 1 nm^{-1} on the absolute scale. This was achieved and is a tribute to the quality of the ID-2 instrument and its team. Figure 3 shows typical stability in intensity reached within 1 h of the emulsion preparation. The stability of the emulsion was further checked by the constancy of the pH in the 100 ml emulsion container over 24 hours. The final scattering functions after all corrections for different incident energies are shown in Fig. 4. The corresponding difference intensity and a comparison with core-shell model is depicted in Fig. 5.

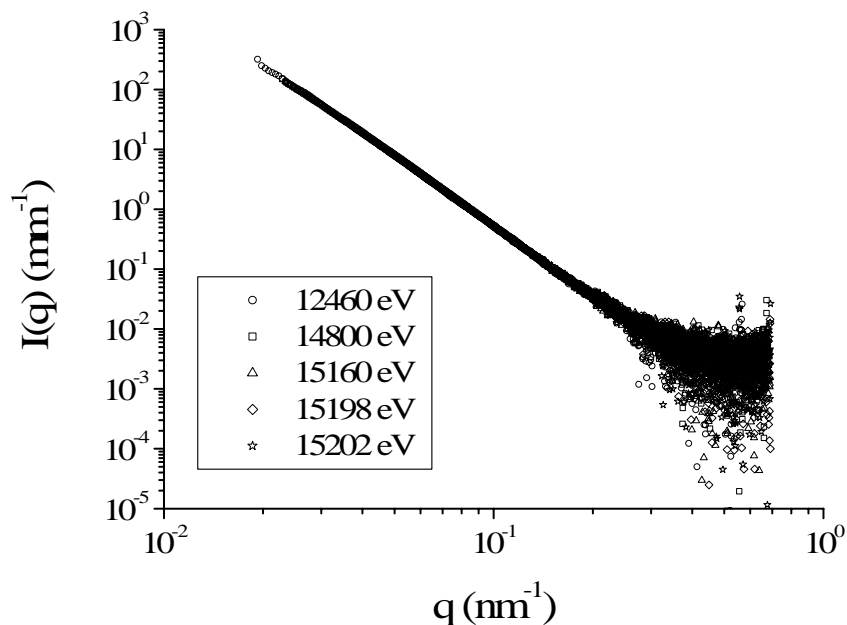


Figure 4: Energy dependence of the scattering intensity for emulsion 12.1. Similar results were obtained for other emulsions indicating very little contribution from the resonant scattering by the Rb ions.

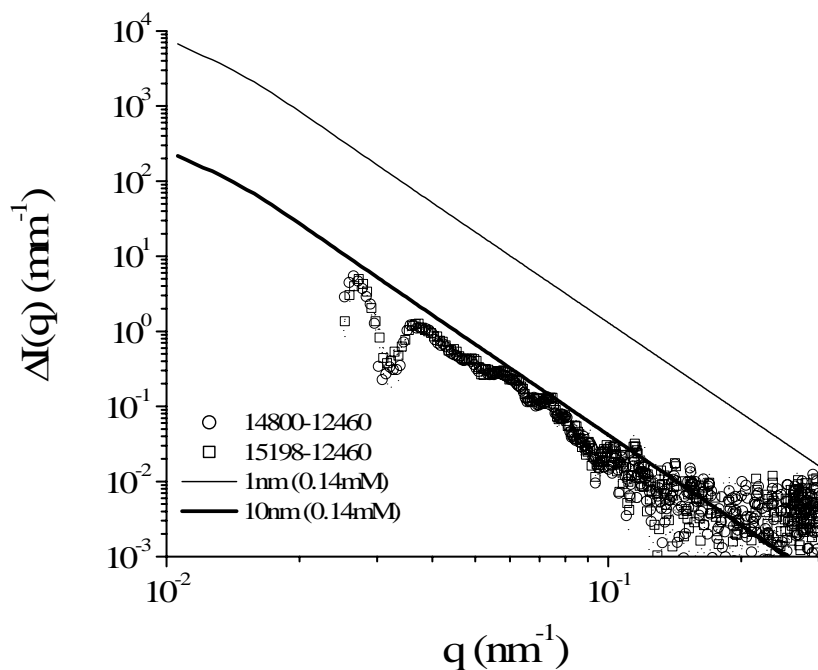


Figure 5: Comparison of difference intensity (ΔI) with a core-shell model involving 1 nm and 10 nm (lower curve) shell of Rb ions at the oil-water interface. The observed ΔI is smaller than that for 10 nm shell corresponding to a more extended distribution of Rb ions. The dips in ΔI matches with the minimum in the form factor for the corresponding monodisperse particles.