	Experiment title: The solid-liquid extraction	Experiment number: SC2636
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Report:

The solid-liquid extraction method with which we are working on, consists in selectively precipitate metal ions from a bulk mixture using cationic surfactant below their Krafft point. This technique has been shown to be very efficient for separation of actinides and lanthanides using long chain ammoniums or pyridiniums [4-5]. *A particular point of this process is the recognition of cationic metal ions by cationic surfactants.* The original proposed mechanism was the formation of a complex ion involving anionic counter-ions of the metal salt, in order to ensure electric neutrality.

We aim at understand these attractive interactions between positively charged polar head groups of the surfactants and metal cations before and after precipitation. Main concerns are about the effect of metal polarisability on its adsorption at the micelle surface, the role of counter-ions and further the structure of precipitated solid (amorphous, crystalline or mesoporous).

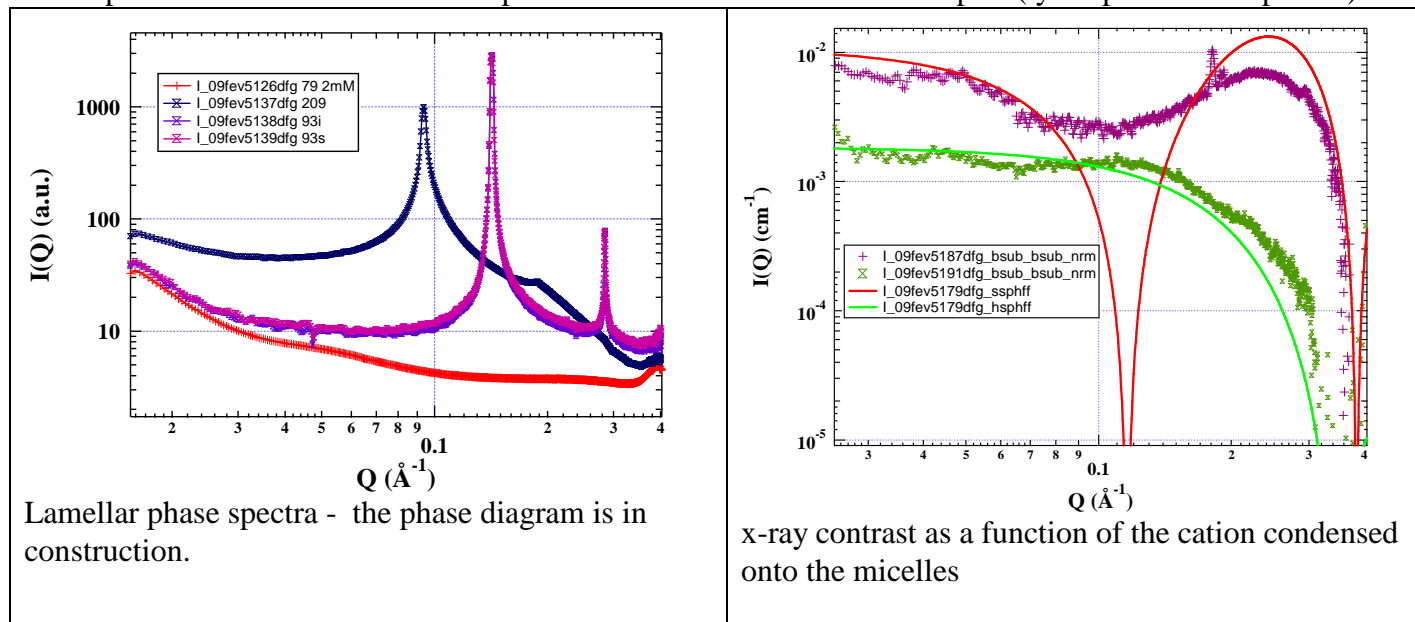
Preliminary experiments have been conducted using cetylpyridinium chloride as “complexing” surfactant. This compound has been shown to selectively precipitate uranyl but lanthanides when used above the critical micelle concentration (1 mM). At lower concentrations, metal separation is not complete. From dynamic light scattering experiment, the chloride form makes spherical aggregates while the nitrate form spherical aggregates at weak ionic strength and makes elongated aggregates at higher ionic strength. The ion has a strong effect on the size and the number of aggregation . Using X-ray scattering we wanted to probe the micelle surface in order to describe the adsorbed corona of metal ions adsorbed at that interface, making use

of the natural electronic contrast between light elements of the surfactants and heavy elements (metals). Unfortunately, although we observe an effect on the extraction, the ion distribution is weak around the micelles and then their contrast in the solvent full of ions remains weak.

The scattering from micelles is around 0.1 cm^{-1} with a Porod region at the limit of the detection.

To cover a maximum a Hoffmeister series of ions and ionic strength, hundred of samples were prepared in different capillaries. However the subtraction with blank sample was not obvious since all capillaries scatter differently. Nevertheless some differences are observable at least to extract size of the aggregates. Concerning the absolute scattering value, it is more difficult.

Some picture below shows two examples for diluted and concentrated samples (lyotropic lamellar phases).



By analysing in details the scattering curves, we expect an effect of the metal hardness (Hoffmeister's series) on the ion adsorption onto the size micelle.

References

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