	Experiment title: cobalt bis-dicarbollide, a new class of surfactant with an interest for the separative chemistry	**Experiment number**: SC2976
Beamline: ID2	**Date of experiment**: from: 02/02 to: 04/02/12	**Date of report**: 15/08/12
Shifts: 6	**Local contact(s)**: M. SZTUCKI	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

P. Bauduin, D. Brusselle, L. Girard, O. Diat from ICSM, 30 Bagnols sur Cèze

Report:

This was a study of self-aggregation on a new class of surfactant molecule with a large interest in separative chemistry and for medicine. A first study using neutron scattering allowed us to characterize the system in water. Some of the structural information was necessary to fully analyse the transition between a vesicle and a micellar organisation that can be only provided by x-rays.

Unfortunetly the concentration at which we observed these transitions as a function of salt and modifying the permittivity of the solvant (by adding alcohol) was to low (below 10-3M) to be able to subtract correctly the capillary scattering. Yet, we tried to use always the same capillary support but we had unfortunetly some deposit onto the glass walls due to beam damage.

The variation of the signal at low q was non coherent with various slope and very noisy and inconsistent with data observed by neutron scattering.

Nevertheless, the beamtime was used for another test using sugar based amphiphiles, an experiment which was very succesfull and published.

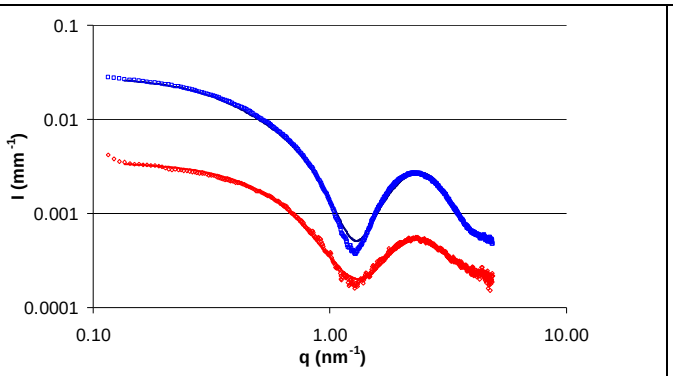
To summarize, the effect of adding salts to micelles made of a sugar based surfactant, n-octyl-beta-glucoside, has been investigated using small angle X-ray scattering (SAXS) and analysed taking into account dynamic light scattering (DLS) data. It was shown that adding salts leads to change the outer shell hydration of the micelles i.e. where the glucose moieties are situated.

The surfactant C8G1 is known to form small micelles in water. From the literature it was found that a biaxial ellipse shape or an end-capped cylinder shape are suitable to describe C8G1 micelles in water. As the electron density in the polar part of the surfactant, made of glucose moiety, differs from the electron densities in the surrounding aqueous phase and in the micellar core, made of n-C8 alkyl chains, a shell model was

considered. The shell of the micelle is composed of glucose moieties and the core is composed of C8G1 alkyl chains. For C8G1 prolate micelles are formed, i.e. with b~c and a>b describing a rugby ball shape. The electron density profile along the main micelle axis *a* is given in Fig. 1, the shell thickness is denoted *t* in the following. The electron density profile shows a higher electron density in the shell compared to the micellar core and to the aqueous phase, this is due to the strong hydration of the glucose moiety.

The fitting of the SAXS spectra was performed by separating the parameters in two classes: *(i)* known parameters: Φ_{C8G1}, ρ_{core} *and* $\rho_{Aqu.}$ are determined experimentally from chemical composition, *t* is the length of a glucose moiety 0.5 ± 0.02 nm, *b* and *c* were kept constant and equal to 1.19 ± 0.03 nm which corresponds to the length of the n-octyl chain of the surfactant in an extended conformation

(ii) fitting parameters*:* *a*, ρ_{shell} (only for samples containing salt), I_b, a prefactor (which is related to n_P), and R_{HS}. Parameter ranges, that correspond to the accuracy, were used for the fixed parameters in order to give some degrees of freedom to the fit. The fits were performed from 0.13 to 3 nm^{-1}. This *q*-range is sufficiently large to take into account the whole shape of the micelle.



SAXS spectra for C8G1 micelles (0.045 and 0.15 M) fitted by a model of biaxial ellipse with a shell

The results were analysed and published recently in Colloids and Surfaces A: Physicochemical and Engineering Aspects, in 2012