ESRF	Experiment title: LYOTROPIC LAMELLAR PHASE: STERIC STABILISATION BY GLYCOLIPIDS. PHASE DIAGRAM MAPPING IN CONCENTRATION GRADIENT	Experiment number: Sc 94
Beamline: **BL 4**	Date of experiment: from: 09/12/95 to: 11/12/95	Date of report: 20/02/96
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Report:

The determination of a ternary phase prism of a mixed system glycolipid/cationic surfactant/water in a large temperature range has been completed for the first time with a quantity of glycolipids as low as 400 mg in total. Up to now, the whole field of mixed system phase diagram had been reduced to industrial surfactants easily available in large quantities.

Experiment has been performed on the water, DDAB (didodecyldimethylammonium bromide) and LS (2-O-Louroylsaccharose) ternary system. Samples have been prepared in 2mm diameter borosilicate capillaries. An aliquot of a concentrated phase of a given composition was put in contact with a water solution in order to get a concentration gradient along the capillary axe. The beam size at sample has been set to 0,25mm x 0,25mm, allowing excellent mapping of the capillaries. Typical counting time to obtain a 512 x 512 frame was 60 seconds. Five capillaries with different initial composition of the concentrated phase have been scanned at six different temperatures.

Fig.1 shows au exemple of $t_{rall}s_{fll}issi_{oil}$ of the sample together with the $^{*J}C$ activity determined by autoradiography on the samc sample. From these two curves, composition along the capillary axe was deduced.

The first set of experiments performed have allowed identification of phase coexistence zones. These phase coexistence have been deduced from spectra as shown on Fig.2 and would probably not have been observed with classical methods implying preparation of one manipulable sample per composition point. Fig. 3 shows one of the phase triangles determined during this experiment. Feasability of rapid efficient ternary phase diagrams is demonstrated.

An unexpected observation has been made due to the fine focussing of the beam at the sample. Within a volume of 0,25mm x 0,25mm x2mm capillary thickness, the swclling of the lamellar phase proceeds by finite steps instead of the expected continuous swelling. The lamellar phase is present as multilayered vesicular

structure of very defined layering periodicity. Typical interbilayer thickness is 100Å, with negligible fluctuation. The dominant interaction is electrostatic and a typical frame showing the coexistence is shown in Fig. 4. We do not know the generality, or the eplanation for this observation, since it implies cooperative steps involving rapid swelling instead of the continuous process expected for a diffusion process of water molecules in multilayer structures.

This fist experiment has shown the feasibility of the proposed method which can now be applied to other molecules.

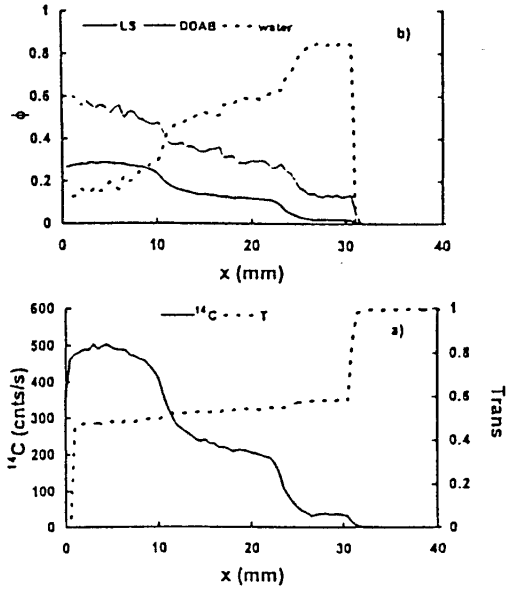

Fig. 1: In situ analysis of the sample content along the capillary axe for an initial molar ratio LS/DDAB=0.5 at the x origine. 1a): ^{14}C activity and the X-ray transmission. 1b): weight fractions deduced from 1a).

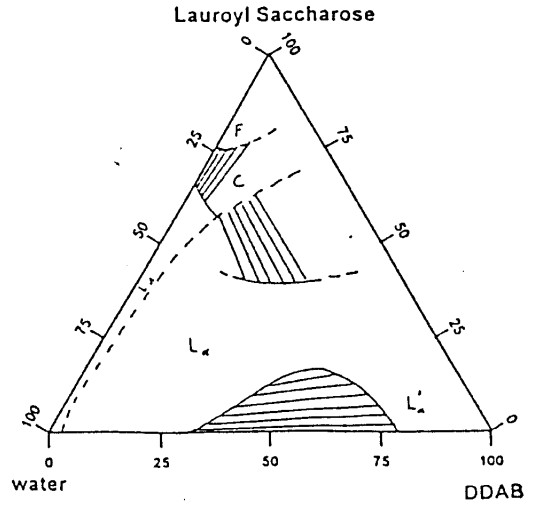

Fig. 3: First outline of the ternary phase diagram at T=25°C for the water/DDAB/LS system.

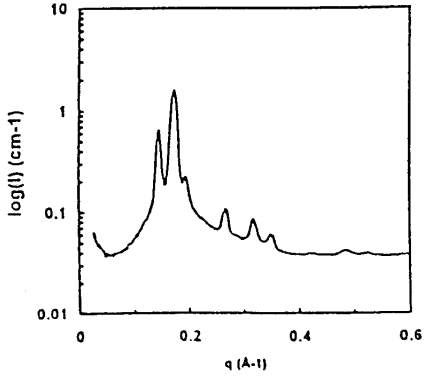

Fig. 2: Cubic and lamellar phase coexistence observed for a composition of 20% water, 24% DDAB and 56% LS.

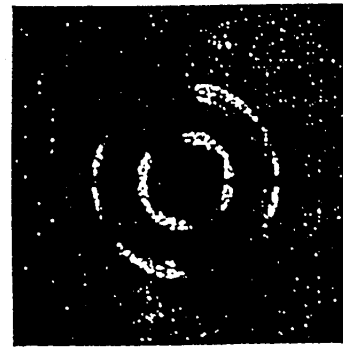

Fig. 4: 2D spectrum showing stepwise swelling for the lamellar structure along the water gradient.