ESRF	**Experiment title:** **Molecular criteria for aqueous boundary lubrication:** ***An XRR study of surfactant layer structures at mica-water interface***	**Experiment number**: SC-2575
Beamline: BM28	**Date of experiment**: from: 27 Nov 2008 to: 01 Dec 2008	**Date of report**: Feb 2009
Shifts: 48	**Local contact(s)**: Dr. Laurence Bouchenoire	*Received at ESRF:*

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Report:

This has been a very successful and highly productive experiment in several aspects, as summarised below.

1) **The measurements were carried out by using two liquid cells that house the samples. We designed and constructed the cells and the main body for which is shown in Fig. 2. They have been used for previous measurements (ID10B) and will be used for future related measurements, and could also be adapted to neutron reflectometry experiments.**

We carried out three different sets of experiments.

2) **We studied the salt ($NaNO_3$) effect on the adsorption on mica for three surfactants: one hydrogenated gemini ($(C12TAB)_2$-H12) and two semifluorinated gemini F-spacer surfactants ($(C12TAB)_2$-H4F4H4 and $(C12TAB)_2$-H3F6H3). As we increase the concentration of salt solution (20, 50, 100 and 200 mM) added to the surfactant solutions, by keeping the same surfactant concentration at 0.1 cmc, we found that the thickness and the density of the bilayer increase.**
3) **The second system we studied is the competitive adsorption between hydrogenated and semifluorinated single chain surfactants. We looked at three surfactants mixtures: C16TAB-F4H11(d)TAB, C16TAB-F5H10TAB and C18TAB-F8H6(d)TAB. We keep the total concentration of surfactants mixture at 1 cmc, while varying the composition. We started with a composition of 100%**

hydrogenated surfactant and as we gradually add the semifluorinated surfactant we believe that they displace the hydrogenated surfactants as they adsorb more strongly.

4) **Finally we looked at the interaction between three components: a polymer, Polyethyleneimine (PEI), a perfluorinated surfactant, Cesium Perfluorononanoate (CsPFN) and a salt, $NaNO_3$ Effect of varying surfactant and salt concentrations were studied in the following sequence: 1) 100 ppm PEI was adsorbed on mica. 2) 50 ppm PEI in water was injected in the liquid cell. 3) A mixture of 50 ppm PEI and CsPFN was added in different steps with increasing surfactant concentrations. 4) 50 mM $NaNO_3$ solution was added to the mixture varying the CsPFN concentration 5) CsPFN concentration keeped constant and varying $NaNO_3$ concentration. 6) The system was rinsed and the sequence repeated from step 4). We found a quite geometric response of the layer structure to the change in the solution conditions. Some reflectivity curves, for this system, are shown in Fig. 1. Distinct Keissig fringes are evident, and both the reflectivity and the fringe spacing vary as the concentration of the surfactant increases from 0.1 cmc to 1 cmc and as the 50 mM salt solution is added to the PEI-CsPFN mixture.**

5) **We found that our local contact, Dr Bouchenoire, extremely knowledgeable and helpful. The beam alignment was non-trival due our gently curved mica surface geometry. Dr Bouchenoire was very careful and patient with us.**

6) **Overall, we anticipate 3 manuscripts to be submitted as a result of every single set of experiment.**

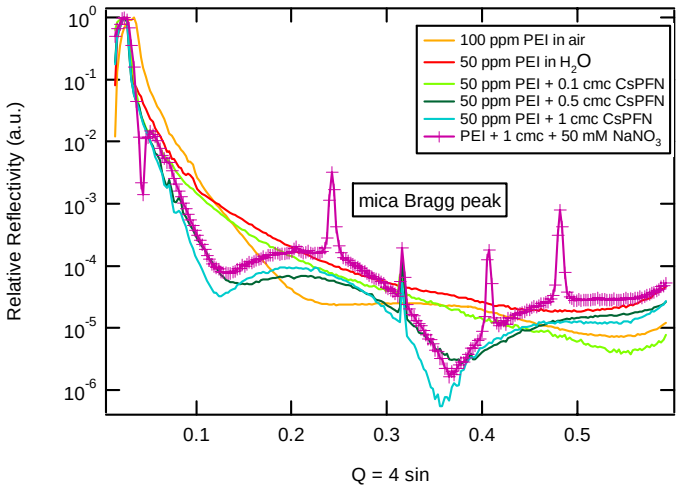

Fig. 1. Example XRR curves from the three component system: polymer PEI, surfactant CsPFN and $NaNO_3$ salt solution on mica

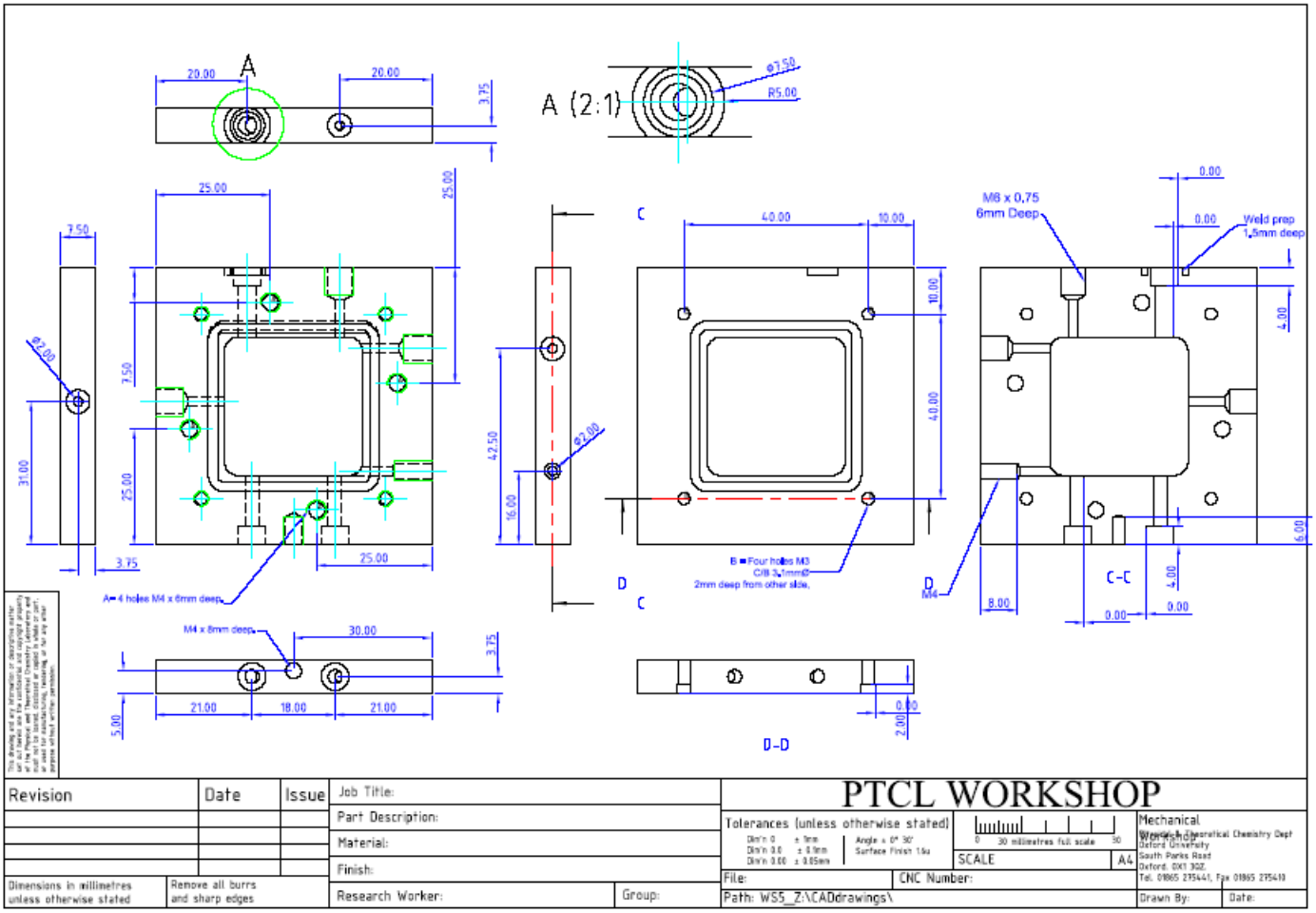

Fig. 2. Mechanical drawing for the main body of a liquid cell designed for ID10B beam line.