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Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

- fill in a separate form for each project or series of measurements.
- type your report, in English.
- include the reference number of the proposal to which the report refers.
- make sure that the text, tables and figures fit into the space available.
- if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** **Molecular criteria for aqueous boundary lubrication:** ***An XRR study of surfactant layer structures at mica-water interface***	**Experiment number**: 28-01-877
Beamline: BM28	**Date of experiment**: from: 09 Sept 2009 to: 13 Sept 2009	**Date of report**: Oct 2009
Shifts: 37	**Local contact(s)**: Dr. Oier Bikondoa	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Dr Wuge H. Briscoe*, University of Bristol and University of Oxford

Dr Robert Jacobs*, University of Oxford

Dr Robert K. Thomas, University of Oxford

Dr Peixun Li,* University of Oxford

Miss Francesca Speranza*, University of Bristol

Mr. Thomas Dane*, University of Bristol

Miss Jennifer Bulpett*, University of Bristol

Mr. Philip Cresswell*, University of Bristol

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 (at ID10B and BM28) and will be used for future related measurements, and could also be adapted to neutron reflectometry experiments.**

We carried out two main sets of experiments.

2) **We studied the interaction between three components: a polymer, Polyethyleneimine (PEI, linear, 50 ppm), a perfluorinated surfactant, Cesium Perfluorononanoate (CsPFN, 1 cmc) and a salt, $NaNO_3$ (50 mM). This system was previously examined and it led to the appearance of some interesting Bragg peaks. Hence, we firstly looked at the effects of varying the temperature of the system. PTC heaters were connected to the liquid cell, allowing to increase the system temperature from about 24°C (hutch temp.) to 39 °C. We found that the Bragg peaks disappear upon heating (see fig.1), suggesting that they are due to hydrated liquid crystal formation of the CsPFN surfactant below its Krafft temperature**

(~40°C). Secondly, since the PEI is a strong polyelectolyte at low pH, the effect of the pH on the system was studied. We found some significant differences between the reflectivity curves of the systems at pH 10 and 7 and the pH 4 system curve. Also by using solutions at different values of pH, the Bragg peaks disappear upon heating.

3) **Then we looked at the interaction between Sodium Perfluorononanoate (NaPFN), branched PEI and $NaNO_3$. Surfactant and salt concentrations were varied in order to study their effects on the system and we found a geometric response of the layer structure to the change in the solution conditions. The reflectivity varies as the concentration of the surfactant increases from 0.1 cmc to 1 cmc and as the salt solution is added to the PEI-NaPFN mixture. pH and temperature effects have also been examined, and an increase in the system temperature causes the disappearance of some small peaks.**
4) **We also looked at four C12 chain surfactants: sulphobetaine, phosphocoline, Na dodecyl sulphate and dodecyltrimethyl ammonium bromide. We looked at different surfactants mixtures, varying the total concentration of surfactants mixture and the composition. We found some interesting results, and we would like investigate this system further in possible future experiments.**
5) **We found that our local contact, Dr Oier Bikondoa, extremely knowledgeable and helpful. The beam alignment was non-trival due our gently curved mica surface geometry. Dr Bikondoa was very careful and patient with us.**
6) **Overall, we anticipate 2 manuscripts to be submitted as a result of every single set of experiment.**
7) **We find the analysis of the XRR data from mica non-trivial and are in the process of seeking help from several collaborators.**

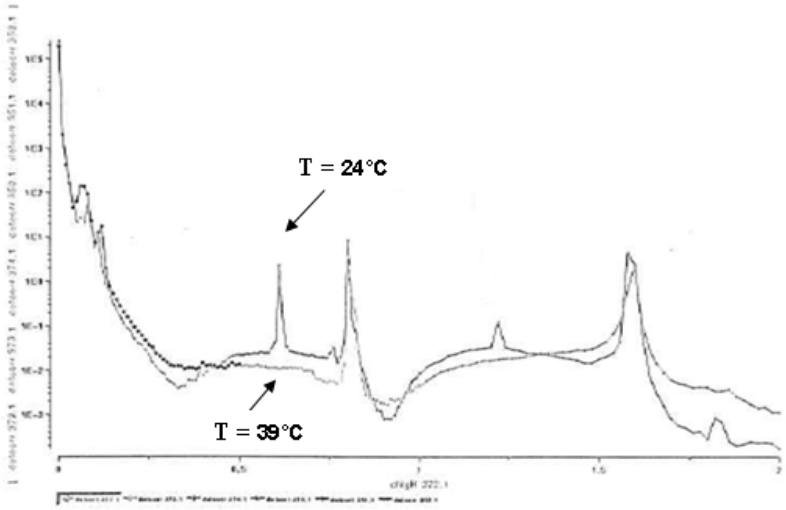

Fig. 1. Example XRR curves from the three component system: polymer PEI, surfactant CsPFN and $NaNO_3$ salt solution on mica at temperature values of 24°C and 39 °C.

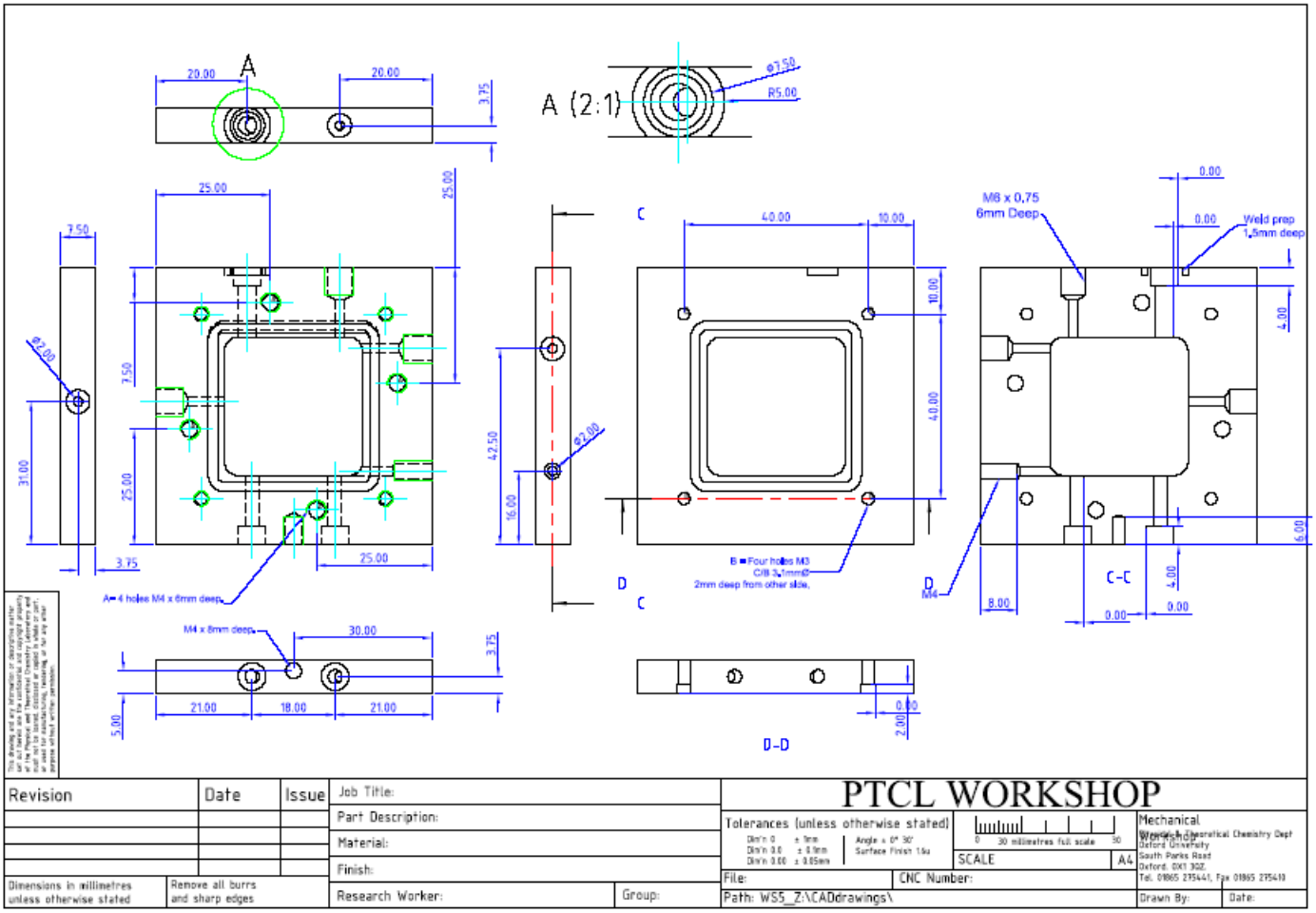

Fig. 2. Mechanical drawing for the main body of a liquid cell designed for BM28 beam line.