Experimental Report

07/11/2013

Proposal: **9-10-1240** **Council:** 4/2012

Title: Neutron Reflection at the Mica Water Interface

This proposal is a new proposal

Researh Area: Chemistry

Main proposer: **CLARKE Stuart M.**

Experimental Team: MILLER Kathryn
CLARKE Stuart M.
GRIFFIN Lucy

Local Contact: GUTFREUND Philipp

Samples: D2O
H2O
C16TAB (CH3(CH2)15N(Br)(CH3)3)
DiDAB ([CH3(CH2)11]2N(CH3)2(Br))
Si/Mica substrate

Instrument	Req. Days	All. Days	From	To
D17	3	3	06/12/2012	09/12/2012

Abstract:

Mica is an exceptionally important mineral in surface science, which until now has been inaccessible by neutron reflection. Here we propose a novel attachment method unlocking this mineral for the first time allowing the fine structure of adsorbed layers to be analysed.

Previously mica was not available to neutron reflection experiments for a number of reasons including defects in the crystal and a high absorption cross section. Experiments carried out by Rennie and Clarke 10 years ago attempted to float thin mica sheets on water but suffered from penetration issues. Here we propose to overcome this problem by decreasing the mica thickness to less than a micron.

Experimental Report:

Neutron Reflection at the Mica Water Interface

Mica is an exceptionally important mineral in surface science, which until now has been inaccessible by neutron reflection. We proposed a novel attachment method unlocking this mineral for the first time allowing the fine structure of adsorbed layers to be analysed.

We are pleased to report that the use of neutron reflection with mica was successfully achieved using a very thin mica film supported on a silicon substrate to avoid previous problems with crystal defects and surface waviness. We have fully characterised the bare mica- water interface using 4 water contrasts (Figure 1). The D_2O contrast (top data set in blue) shows a characteristic double critical edge relating to total reflection, first from the increase in SLD from silicon to mica and at higher Q from silicon to D_2O. The presence of the second plateau gives comfort that the surface of interest, the mica, is the adsorption surface being studied. The crystal must be thick enough to see a second plateau and also to dampen down oscillations that would otherwise swamp the measured data . The crystal however must not be so thick that it attenuates too much of the neutron beam as the reflectivity of adsorbed layers are hard to interpret as the reflectivity hits background faster. Therefore the optimum mica crystal thickness is between 0.5 and 25 μm to satisfy all conditions.

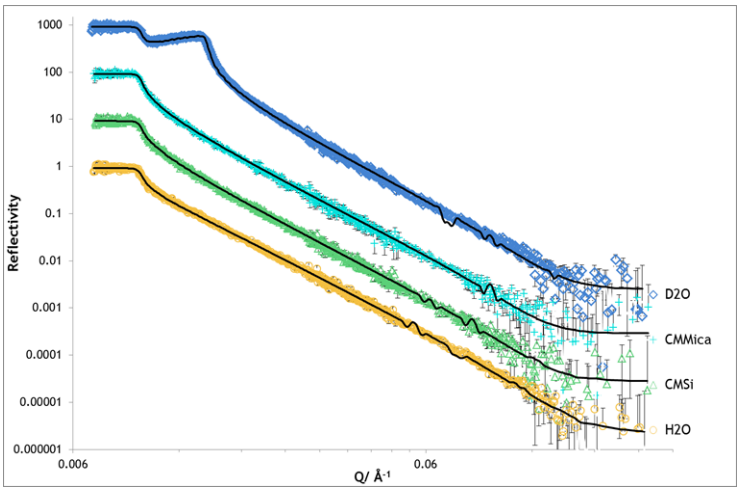

Figure 1: Reflectivity profiles of the bare mica interface in four water contrasts, D_2O, H_2O and water contrast matched to silicon and mica.

We have also showed the potential of this experimental set up to study soft condensed matter layers by characterising the adsorption and desorption of didodecyldimethylammonium bromide (DDAB), a cationic dichain surfactant. It is found that, at the twice the critical micelle concentration (CMC = 0.08mmol), a bilayer of DDAB with a thickness of 22Å, slightly less than twice the molecule length (16Å), is observed (Figure 2). The surfactant layer contains essentially no water, indicating that a uniform bilayer is formed and not adsorbed micelles. Interestingly the layer seems to be robustly bound to the surface even with extensive washing (removed <5% of the bilayer). Ion exchange with 1M KCl did remove 45% of the layer, however half of the original bilayer remains. The reflectivity of the bare interface could be recovered after the mica was UV-Ozone treated.

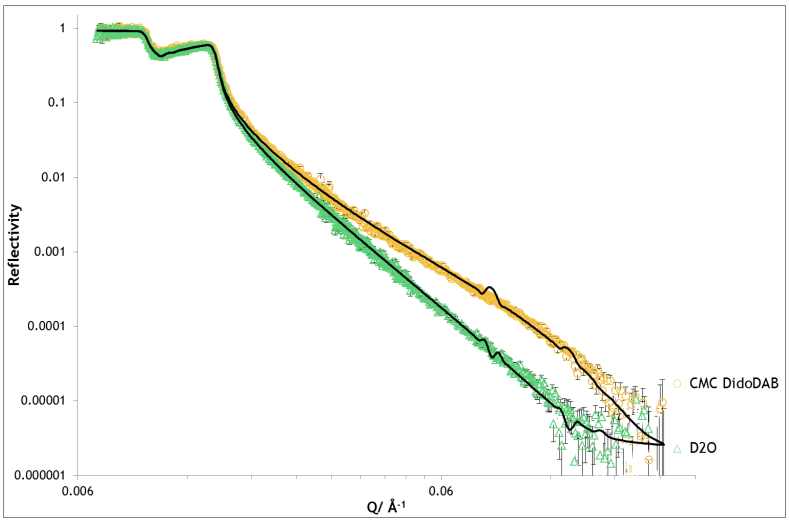

Figure 2: Reflectivity Profile of the bare mica interface (green) and in the presence of 0.16mM DDAB solution.