



	Experiment title: Locating counter ions at the muscovite mica - electrolyte interface	Experiment number: HC-2298
Beamline: ID03	Date of experiment: from: 3 March 2016 to: 9 March 2016	Date of report: 19 August 2016
Shifts: 18	Local contact(s): Maciej Jankowski	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Sander Brugman*, Mireille Smets*, Eleanor Townsend*, Elias Vlieg* Radboud University, Institute for Molecules and Materials, Heyendaalseweg 135, 6525AJ Nijmegen, The Netherlands		

Report:

This study is partly a continuation of the work conducted by Stelian Pinte, a joint PhD student between the Radboud University and the ESRF. In that project, the interfacial structure of muscovite mica in contact with different electrolyte solutions was determined. Pinte already finished his PhD thesis [1] and his first results have recently been submitted for publication.

These experiments gave the first indications of the presence of counter ions at the surface, which in many experiments and models is not taken into account. Here, by changing the anion to the heavily scattering iodide, we aim to establish if the counter ion is indeed present at the interface. Moreover, three different cations (Li^+ , K^+ and Cs^+) were used at three different concentrations (10, 50 and 200 mM) to obtain reliable results and to monitor the change in the layering behaviour and the structure of the hydration shells as a function of concentration.

This experiment went extremely well. A full dataset of 14 crystal truncation rods of muscovite mica was measured for fourteen different conditions. Data processing is still ongoing, but a feasible model for the CsI data has already been found (figure 1). For a 50 mM electrolyte solution, a Cs occupancy of 0.43 ± 0.05 monolayer was found, which is close to the expected value of 0.5 monolayer considering charge neutrality. Furthermore, a clear indication of the anion was found with an occupancy of 0.18 ± 0.10 monolayer. Further analyses are still ongoing.

In conclusion, this was a very successful experiment. Data analyses are in progress and will take time due to the large number of systems investigated, but preliminary results show that the counter ion indeed can be found at the interface. These results will be submitted for publication.

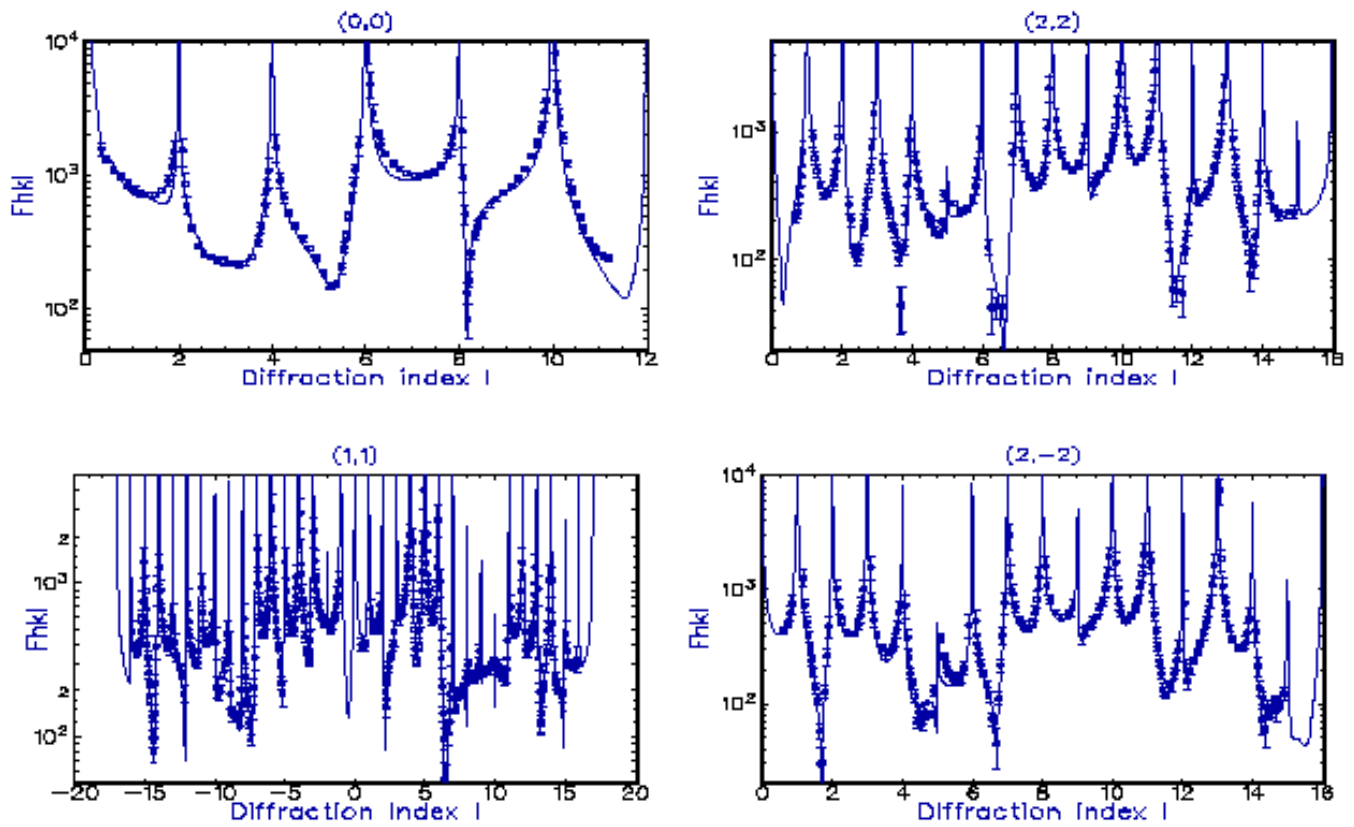


Figure 1: Four measured crystal truncation rods (dots) of muscovite mica in contact with a 50 mM CsI solution. A preliminary fit (line) is in good agreement with the experimental data. The vertical axis depicts the measured structure factor, the horizontal axis shows the l -value, indicated on top are the (h, k) diffraction indices of the measured crystal truncation rod.

[1] Stelian Pinteá, Interface Structure of Mica in Electrolyte Solutions - a surface X-ray diffraction study (Radboud University, Nijmegen, The Netherlands, 2015).