ESRF	**Experiment title:** Crystal Structure of Solid Monolayers of Dodecane Adsorbed at Sub-Monolayer Coverages and from the Liquid onto Graphite	**Experiment number**: SI-559
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Report:

This experiment was part of a continuing study of the structure and dynamics of simple molecules adsorbed to solid surfaces from liquids and solutions. From previous work we have identified the formation of crystalline adsorbed monolayers of dodecane onto graphite from its liquid and we wished to determine the orientation of the overlayer with respect to the underlying surface. This grazing incidence x-ray scattering experiment was a trial experiment to see if we could observe the scattering from a single monolayer of dodecane adsorbed on a single graphite surface.

It is clear from this experiment that we can indeed observe the scattering from the adsorbed overlayer which is characterised by peaks that occur at Q values as those observed with other methods (neutron diffraction) and by an intensity distribution indicative of a two-dimensional layer. These peaks also occur in a region of scattering space where there are no reflections from the bare surface or bulk liquid dodecane increasing our confidence in our assignment.

The experimental geometry turns out to be crucial in the observation of the monolayer pattern such that the incident angle of the synchrotron radiation should be just below the critical angle of the graphite surface while just above that of the liquid dodecane. These angles are, of course dependent upon the energy of the radiation. Initially we selected 0.62A to obtain good transmission through the liquid dodecane. However, a wavelength of 1.55A still allowed reasonable penetration but with the advantage of larger critical angles, faciliting alignment.

A significant issue with these measurements concerns the nature of the solid graphite substrate. During the experiment we attempted to characterise several graphites obtained from different sources both natural and synthetic. Despite, other less attractive features, the choice of graphite finally centred on the size of the surface available to be illuminated. Ultimately the best pattern was obtained from a highly oriented pyrolytic graphite and not a single crystal, although the pattern includes scattering from the adsorbed monolayer on 'mis oriented' crystal faces. It is clearly desirable to use a larger single crystal in any subsequent experiment although clearly these are difficult to obtain.

The precise alignment of the substrate was performed by diffraction from the (002) graphite reflections allowing the surface to be inclined to within 0.1 degrees. However, the inplane orientation of the graphite could not be performed because the in-plane graphite peak is well outside the accessible q-range of the multiwire, gas filled detector of ID01 which has a maximum scattering angle of approximately 10 degrees. This detector does have the advantage that a wide range of scattering space can be covered simultaneously, just at lower q. There is a scanning detector on ID01 that can go to wide enough angles to see the in-plnae graphite peak, however, this detector scans in the vertical direction so the sample would need to be vertical also, a configuration that does not allow liquid to remain on the solid surface. In a subsequent experiment both the scanning detector and the multiwire detector need to be used in combination to align the substrate then to investigate the adsorbed layer. However, this will demand more beam time to change repeatedly between detectors (e.g. make and break the vacuum/remove the nose cone etc.).

The temperature control was achieved using an aluminium block with a thermostated water bath. Only temperatures above 8C could be used without excessive condensation of water from the air (windows were avoided to reduce additional scattering). At these temperatures, although there was evidence of evapouration of the dodecane with time, most of the loss of liquid came from the inclination of the sample to be at the appropriate critical angle. Clearly this problem could be reduced in an experiment where the incident beam is inclined at the grazing angle. Incident beam intensity fluctuations and detector stability were also issues and prevented us using long counting times to get good statistics and the efficient subtraction of the bare graphite background.

It is envisgaed that during a subsequent experiment many of these issues will be resolved and the data that can obtained will be optmised.