ESRF		**Experiment number:** SI-960
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Shifts: 18	**Local contact(s):** Dr. S. Ramos	*Received at ESRF:*

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Report:

We carried out a surface reflection EXAFS experiment on $Ni(NO_3)_2$ aqueous solution to determine the ion concentration and structure as a function of depth. We were successful in measuring the ion specific concentration for four different concentrations (0.1molal to 4molal) in the first 10Å and down to a bulk depth of 3000Å.
A new experimental setup was developed for this experiment, in which the incoming beam was collimated to 0.2mm, then deflected towards the diffractometer using a 14cm Si crystal. The crystal was then aligned to get the most horizontal beam possible. After this stage, the diffractometer was aligned to this new beam so that the centre of rotation of the diffractometer was in the deflected beam. The sample stage was introduced and the angle of the beam was measured as half the difference between the deflected beam and reflected beam. New automatic alignment macros were written for this stage, so that after each scan the diffractometer could be positioned accurately again. This way evaporation (and loss of sample level) could be accurately checked. The sample was refilled with distilled water after a 2% loss in sample volume, to maintain concentration.
The reflection data were extremely encouraging, with data showing significant concentration variation as the measured depth approached the surface interface. For the deeper layers, there was sufficient statistical information to obtain EXAFS data. Figure 1 and 2 shows data from two angles of the 1 molal sample.

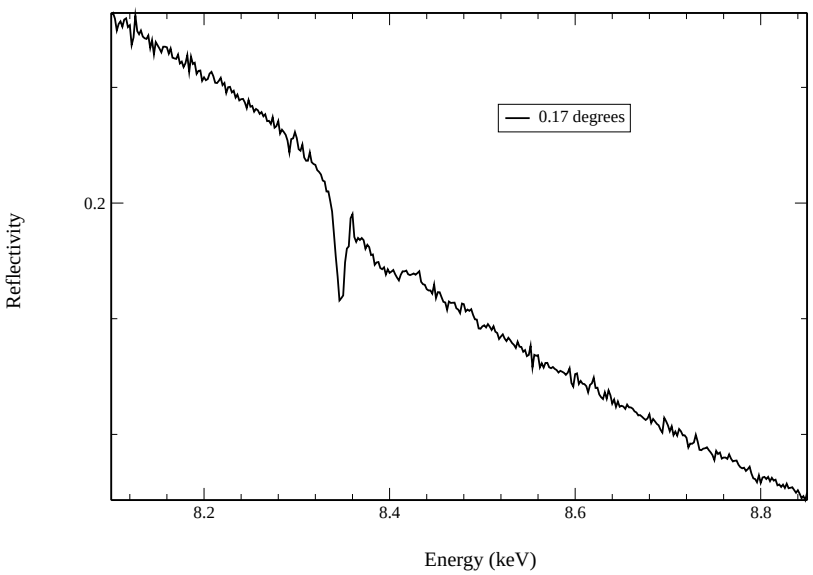

Figure 1: The 1 molal $Ni(NO_3)_2$ Reflectivity at 0.17 degrees, pentration depth of 200Å.

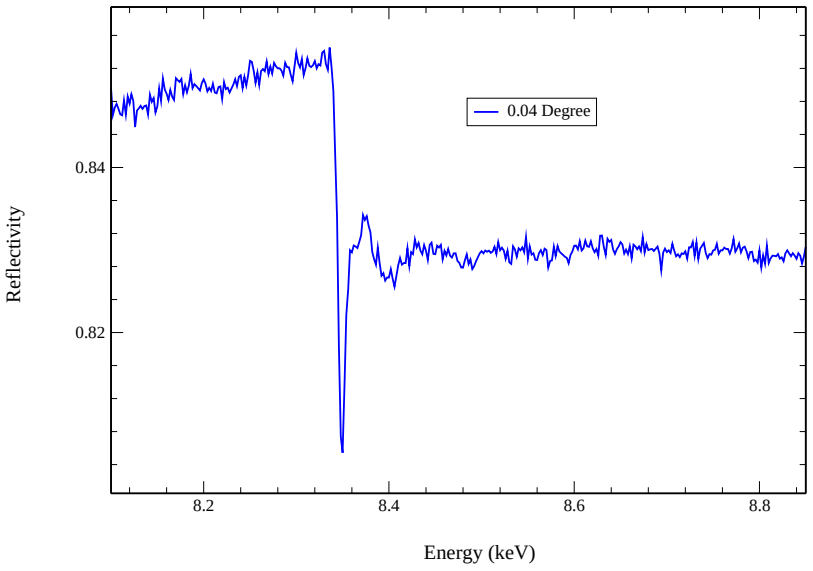

Figure 2: The 1 molal $Ni(NO_3)_2$ Reflectivity at 0.04 degrees, about 30Åpenetration depth. Clearly shows that there are ions very close to the surface and has sufficient statisitcal quality to determine a basic EXAFS spectrum.