ESRF	**Experiment title: Aqua-ion and solute structure in aq ueous electrolyte solutions: An Anomalous x-ray scattering study of l-1 electrolytes CsI and RbBr in**	**Experiment number:** SC417
Beamline: ID01	**Date of experiment:** from: 2 July 1998 to: 6-July 1998	**Date of report:** 14-August-1998
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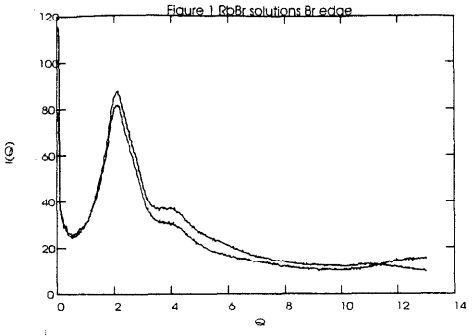
Report: The aim of this experiment was an attempt to measure the hydration and ion-ion structure of the Rb and Br ions in concentrated aqueous solution usings the anomalous X-ray diffraction technique. The high flux and high incident and diffracted beam energy resolution available on ID01 suggest these measurements should now be feasible.

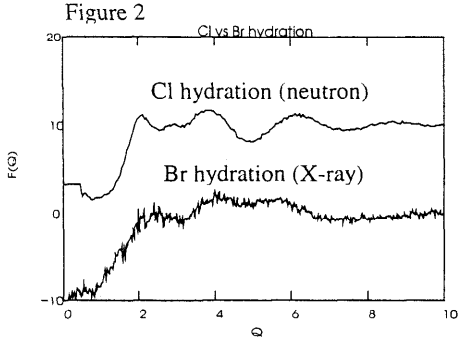
Diffraction experiments were made at 200 and 10 eV from the Rb (15.2 keV) and Br (13.474 keV) K-edges respectively. The initial experiments took place at the end of march 1998. However, due to a problem with the monochromator after the restart of the synchrotron we were unable to carry out the full experimental programme at that time. However, we were, at this time, able to establish that there was excessive beam heating of our sample due to the high flux. This lead our solutions (that were in sealed capillaries) to explode.

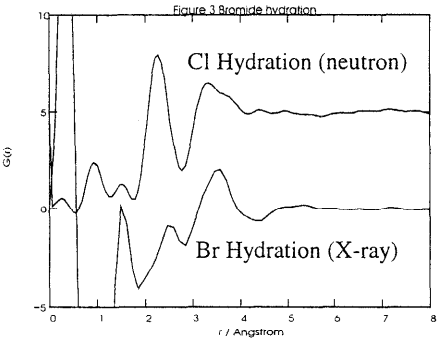
We were rescheduled beam time to complete the experiment at the beginning of July 1998. For these experiments we used silica capillary tubes through which the solutions were continually circulated using a peristaltic pump. Using this method we were able to completely avoid the problems with beam heating that we observed in the original measurements.

Figure 1 shows the raw intensities we obtained for 6 molal RbBr solutions at the two

Figure 2







wavelengths at the Br K-edge. A clear difference can be seen in the two diffraction patterns. Figure 2 shows the difference pattern obtained after a very crude normalisation of the two diffraction patterns. A clear signal in the differential scattering pattern is observed. Also shown in figure 2 is the first order difference function obtained from a neutron diffraction and isotopic substitution experiment on the Cl^- ion in aqueous sodium chloride. The functions show similar features (although the structure in the neutron difference is richer due to the contributions of the D atoms to the scattering). Figure 3 shows the transform of this function to real space along with the Cl hydration for neutrons. The prominent peak at -3.4 Å corresponds to the typical Br-0 distance expected. It can be noted how the Br-0 peak is at slightly further distance than the Cl-O distance observed in the neutron experiment and is more distinct due to the. absence of Br-H correlations in the X-ray difference functions.

There are still a few points that need to be addressed in this experiment, most notably the rather strange behaviour of the X-ray data at high Q values. Needless to say we find this first experiment very encouraging and we are currently analysing the data (including the Rb edge data) with more thorough correction procedures.