



Experiment title: X-ray diffraction investigations of the structure of concentrated aqueous solutions of aluminum, beryllium and magnesium bromides at low and room temperatures

Experiment number:
CH-931

Beamline: ID15b	Date of experiment: 22 nd -28 th November 2000 and 11 th -17 th April 2001	Date of report: 28-02-2002
Shifts: 18	Local contact(s): Dr. José Manuel Merino Alvarez and Dr. Thomas Buslaps	<i>Received at ESRF:</i>

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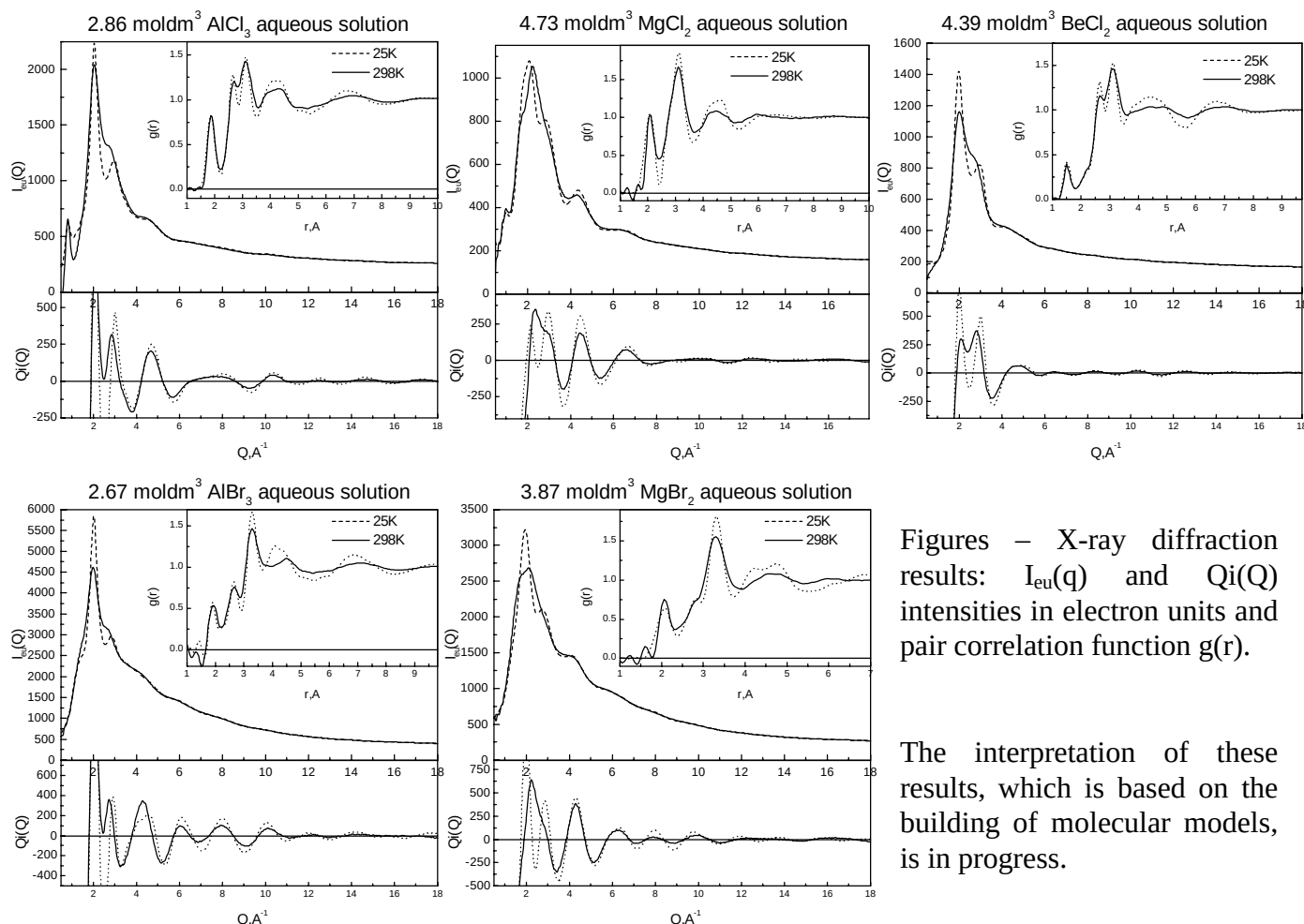
	and	
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Report:

We intended to obtain, at different temperatures, information about the structure of concentrated aqueous solutions of aluminium, beryllium and magnesium salts. The results from this experiments will be coordinated with the results obtained for these samples from inelastic neutron scattering [1] and the from spontaneous Raman scattering experiments [2].

High intensity monochromatized synchrotron radiation was used, in order to gather information with high accuracy in short acquisition times. Monochromatized synchrotron radiation of high energy (88.620keV, 0.140Å) was chosen, simplifying most of the corrections to be performed on the raw data. The samples were studied by transmission in layers of 2mm, contained in a plane parallel cell between kapton windows. The detection system was a MAR online image plate scanner (2300x2300 pixels: pixel size 0.15mm). The one-dimensional diffraction patterns were obtained by integration of the diffraction rings of the 2D patterns. The resolution and angular range covered could be modified by changing the distance from the sample to the detector. The CCR cooling system of the beamline allowed us to change and monitor the sample temperature between 25K and 300K.

The measured intensities, normalised to the intensity of the incident beam, were corrected from background, empty container and air scattering contributions, absorption and geometrical factors and then scaled to electron units using the Krogh-Moe method (for that the incoherent intensities were approximately calculated from data available in literature) [3,4]. A comparison between the experimental results obtained with the liquid and the glassy samples for the aqueous solutions of aluminium, magnesium and beryllium halides investigated are displayed bellow. These results are in fairly good agreement with the results obtained in the past, with a conventional X- ray tube and photographic detection, for aqueous solutions of these salts at room temperature [5].



Figures – X-ray diffraction results: $I_{eu}(q)$ and $Q_i(Q)$ intensities in electron units and pair correlation function $g(r)$.

The interpretation of these results, which is based on the building of molecular models, is in progress.

During these experiment we also had the opportunity to obtain room temperature X-ray diffraction patterns of aqueous solutions of other inorganic salts, namely concentrated aqueous solutions of strontium and barium chlorides and bromides, and of lithium and caesium sulphates. These results together with the ones obtained at room temperature for the aqueous solutions of magnesium salts have already been included in an article entitled “*Intermediate range order in aqueous solutions of salts constituted by one ion of valence two combined with a counter-ion of valence one*” sent for publication [6].

Results from this experiment were also presented in Vaals, Holland, during the 27th International Conference on Solution Chemistry (Aug 2001) and in Obernai, France during the ESF-EMLG Conference “Water at the New Millenium” (Sept 2001).

References

- [1] Alves Marques M, Gaspar A M and Kolesnikov A I, 2001 *ISIS Report on experiments RB11953 and RB11489*
- [2] da Silveira A, Alves Marques M and Macias Marques N 1965 *Mol Phys* **9** 271; Alves Marques, Sousa Oliveira M A and Resina Rodrigues J 1990 *J Chem Soc Farad Trans* **86** 471
- [3] Warren B E 1968 *X-Ray Diffraction* (Reading, MA: Addison-Wesley)
- [4] Wilson AJC (ed) 1992 *International Tables for Crystallography C*
- [5] Alves Marques M and de Barros Marques M I 1974 *Proc K Ned Akad Wetensch B* **77** 286; de Barros Marques M I 1982, *Thesis* (U. Lisboa)
- [6] Alves Marques M, Cabaço M I, de Barros Marques M I, Gaspar A M, submitted to publication

Note Unfortunately due to a problem with the front-end of the beamline the experiment could not be performed in November 2000 as initially scheduled and had to be rescheduled to April 2001 as indicated above.