

Proposal 25-02-643

Structural studies on polycarboxylate complexes of transition metal ions

Time allocated: 8 shifts

Our research is focused mainly in the study of the magnetic exchange between paramagnetic centres through polycarboxylic bridging ligands. Sometimes the synthesis of these complexes is quite difficult and only small or epitaxially twin crystals are obtained. In the latter case we have to cut these crystals to obtain a tiny (about 70 microns in all three directions) single crystals appropriate for X-ray diffraction. In this context, synchrotron radiation becomes the only way to obtain data, good enough, to solve and refine the crystal structure of these compounds.

There were four main tasks to be carried out in the time allocated at BM25. However, in our first scheduled time in October 2007, the experiments could not be performed due to some communications problem between the control software and the experimental set up. Thus, the experiment was rescheduled to May 2008.

The second attempt, in May 2008, was more successful and some tests could be carried out, but no structural determination of any complex was obtained.

The main problem during the measurements was the alignment of the crystal in the X-ray beam. This procedure is made with an optical telescope with a preset position for the X-ray beam, but although the data collection was good for the first image, after a couple of rotations, the crystal seemed to be out of the beam. The troubleshooting for this misalignment was very hard and took most of the time. Some other minor problems were added to this one, such as the data collection procedure or the crystal rotation system.

At the end, some images of doubtful quality were obtained, however, the images come out without headers and therefore, they cannot be read with usual software such as MOSFILM or HKL2000, and it seems that special software has to be developed to read the images until a header could be included with the image.

We think that the single crystal station at the BM25 is not fully operative yet, and that this kind of experiments is welcome by the staff in order to complete the setup of the beamline.