



	Experiment title: <b>Supramolecular Organic and Inorganic Architectures</b>	<b>Experiment number:</b> CH 1344
<b>Beamline:</b> ID11	<b>Date of experiment:</b> from: 06.09.02 to: 27.02.04	<b>Date of report:</b> 17.02.04
<b>Shifts:</b> 31	<b>Local contact(s):</b> Dr. Gavin Vaughan	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants</b> (* indicates experimentalists): <b>Dr. Mario Ruben</b> <b>ISIS Strasbourg/INT Karlsruhe</b> <b>Dr. Mihai Barboiu</b> <b>ISIS Strasbourg</b> <b>Michael Stadler</b> <b>ISIS Strasbourg</b> <b>Dr. Lindsay Uppadine</b> <b>ISIS Strasbourg</b> <b>Dr. Helmar Goerls</b> <b>University of Jena</b>		

## Report:

231 categories of samples were taken to Grenoble and of those, 156 crystals were mounted for study. X-ray diffraction measurements at normal temperature and also at variable temperatures (in the case of the magnetical switchable spin crossover Fe<sup>II</sup> compounds) were carried out. Some of the most important data sets are discussed below.

### 1. [4x4] Pb(II)

Because of the uniqueness of that molecule we continued the efforts of the previous experiments CH 832, CH 918 and CH 1026. During the experiment CH 1344 we succeeded finally to establish *and* to refine the structure of the [4x4]Pb<sub>16</sub> grid complex. Thus, this time we can locate all organic material correctly beside of the easily observable, grid-like organised 16 lead atoms. The results were published in a joint publication.<sup>1</sup>

Furthermore, the data set of second species of the [4x4] grid-like complexes (with a varied, more flexible ligand system) was recorded and the refinement is in work by Dr. Gavin Vaughan.

## 2. Redox-active [2x2] Co<sup>II</sup><sub>4</sub> complexes

The structural features of a series of tetranuclear Co<sup>II</sup><sub>4</sub> complexes displaying very interesting reduction behaviour was investigated and reported.<sup>2</sup> In addition a double modulation of the redox behaviour by potential and pH-value was found and also brought to publication.<sup>3</sup>

## 3. Switchable [2x2] Fe<sup>II</sup> compounds

We persisted also in obtaining good data sets of 1D and 2D molecular arrangements of the spin crossover [2x2] Fe<sup>II</sup> units. Although we were able to collect several data sets at different temperatures of a 1D compounds, we couldn't solve the structure this important coordination polymer. Nevertheless, we succeeded in determining the structure of an additional spin crossover grid-compounds and concluded this part of the investigations by a publication.<sup>4</sup> In forthcoming experiments, we would like to investigate the switching behaviour of multinuclear spin transition Fe<sup>II</sup><sub>4</sub> compounds in more detail (triggered by temperature and light).

## 4. Helices and helical codons

Linear ligands of type py-hyz-py-hyz-py (py = pyridine, hyz = hydrazone) gave helical complexes with AgOSO<sub>2</sub>CF<sub>3</sub>. Two such of structures were solved at ESRF, by Dr Gavin Vaughan: a trinuclear double-helical complex and a mononuclear helix assembled in an oligomeric structure (in solid state). Further investigations are being done on the complexes, in order to study their physico-chemical properties.

## 5. Publications

- [1] M. Barbiou, J.-M. Lehn, G. Vaughan, *J. Am. Chem. Soc.* **2003**, *125*, 10257-10265.
- [2] M. Ruben, J.-M. Lehn, G. Vaughan, *Chem. Comm.* **2003**, 1338-1339
- [3] L. H. Uppadine, J.-P. Gisselbrecht, J.-M. Lehn, *Chem. Comm.* **2004**, in press.
- [4] M. Ruben, E. Breuning, J.-M. Lehn, V. Ksenofontov, F. Renz, P. Gülich, G. Vaughan, *Chem. Eur. J.* **2003**, *9*, 4422-4429;
- [5] M. Ruben, E. Breuning, J.-M. Lehn, V. Ksenofontov, P. Gülich, G. Vaughan, *J. Mag. Mag. Mat.* **2003**, in press.
- [6] M. Ruben, U. Ziener, J.-M. Lehn, V. Ksenofontov, P. Gülich, G. Vaughan, *Chem. Eur. J.* **2004**, *submitted*.
- [7] M. Ruben, F. J. Rojo, J. Romero-Salguero, L. Uppadine, J.-M. Lehn, *Angew. Chem. Int. Ed.* **2004**, in press.
- [8] D. Walther, S. Liesicke, L. Böttcher, R. Fischer, H. Görls, G. Vaughan, *Inorg. Chem.* **2003**, 625-632.