



	Experiment title: Supramolecular Architectures with Incorporated Mixed Valence Metals	Experiment number: CH1026
Beamline: ID11	Date of experiment: from: 30.03.01 to: 02.04.01	Date of report: 30.08.01
Shifts: 09	Local contact(s): Dr. Gavin Vaughan	<i>Received at ESRF:</i>

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Report:

28 categories of samples were taken to Grenoble and of those, 13 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^{II} compounds) were carried out. Finally, nine data sets could be collected. The refinement of most of the data sets is either already finished or in course. 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 and CH 918. Most of the measuring time was dedicated once more to that problem. Finally, for the first time, we succeeded to obtain data set of this compound, which gives us the really expectation to a correct solution of that structure in the frame of the accepted parameters of X-ray structures. Thus, this time we can locate all organic material correctly beside of the easily observable, grid-like organised 16 lead atoms. Dr. Gavin Vaughan works at the moment on the refinement of that gigantic (for our branch of chemistry) molecule and we hope that he will finish soon. (over 600 non hydrogen will have to be refined).

In addition, we were able to determine the structure of one suspected intermediate during the formation of the [4x4] Pb(II). The intermediate consists of four organic ligands and has a

cross-like shape. Surprisingly, all coordination sites of those four ligands are completely occupied by lead atoms. A very important issue for the understanding of the course of self-assembly of large supramolecular entities.

2. [2x3] Zn^{II}

For the first time, a grid-like compound consisting of transition metals with a nuclearity larger than four was successfully investigated. Thus, an important gap in our research could be closed after long lasting efforts. The structural determination exhibits that the six Zn-atoms are aligned in two lateral lines with three members, while the central row is completely missing due of the steric strain of the whole entity. This result gives us important experience in avoiding this strain in the construction of high-nuclear grid-compounds.

3. Switchable [2x2] Fe^{II} compounds

We persisted also in obtaining a good data set of a, by Pb^{II} atoms, aligned 1D wire of spin crossover [2x2] Fe^{II} units. Although we were able to collect two data sets at two different temperatures of that compounds, we couldn't solve the structure of this important compound. In a forthcoming experiment we should persist in solving this nice exemple of a hierarchical coordination. Nevertheless, we succeeded in determining the structure of an additional spin crossover grid-compound.

4. Further compounds

In addition, data sets of one tetranuclear Mn^{II} compound, of one mononuclear Fe^{II} compound and of three M₂L₂- helices could be collected.

5. Publications

1. Recognition-Directed Supramolecular Assemblies of Metal Complexes of Terpyridine Derived Ligands with Self-Complementary Hydrogen Bonding Sites

Ulrich Ziener, Esther Breuning, Jean-Marie Lehn, Elina Wegelius, Kari Rissanen, Gerhard Baum, Dieter Fenske, Gavin Vaughan

Chemistry

Volume: 6, Issue: 22, Date: November 17, 2000, Pages: 4132-4139

Further publications are in preparation!

