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ESRF

Experiment Report Form

The double page inside this form is to be filled in by all users or groups of users who have had access to beam time for measurements at the ESRF.

Once completed, the report should be submitted electronically to the User Office using the **Electronic Report Submission Application:**

http://193.49.43.2:8080/smis/servlet/UserUtils?start

Reports supporting requests for additional beam time

Reports can now be submitted independently of new proposals – it is necessary simply to indicate the number of the report(s) supporting a new proposal on the proposal form.

The Review Committees reserve the right to reject new proposals from groups who have not reported on the use of beam time allocated previously.

Reports on experiments relating to long term projects

Proposers awarded beam time for a long term project are required to submit an interim report at the end of each year, irrespective of the number of shifts of beam time they have used.

Published papers

All users must give proper credit to ESRF staff members and proper mention to ESRF facilities which were essential for the results described in any ensuing publication. Further, they are obliged to send to the Joint ESRF/ ILL library the complete reference and the abstract of all papers appearing in print, and resulting from the use of the ESRF.

Should you wish to make more general comments on the experiment, please note them on the User Evaluation Form, and send both the Report and the Evaluation Form to the User Office.

Deadlines for submission of Experimental Reports

- 1st March for experiments carried out up until June of the previous year;
- 1st September for experiments carried out up until January of the same year.

Instructions for preparing your Report

?? fill in a separate form for each project or series of measurements.

?? type your report, in English.

?? include the reference number of the proposal to which the report refers.

?? make sure that the text, tables and figures fit into the space available.

?? if your work is published or is in press, you may prefer to paste in the abstract, and add full reference details. If the abstract is in a language other than English, please include an English translation.

ESRF	**Experiment title:** Interwoven Metal-Organic Framework with Nanoporous and Magnetic Properties	**Experiment number**: CH-1236
Beamline: ID11	**Date of experiment**: from: 12/07702 at 7:00 to: 16/07/02 at 7:00	**Date of report**: 29/08/02
Shifts: 12	**Local contact(s)**: Dr. Gavin Vaughan	*Received at ESRF:*

Names and affiliations of applicants (* indicates experimentalists):

Prof. Jaume Veciana

Dr. Daniel Ruiz-Molina

Dr. Klaus Wurst

Daniel Maspoch

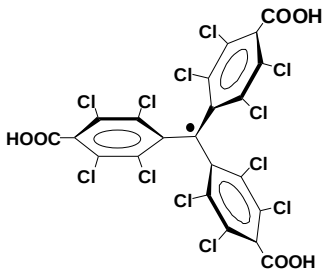
Report:

This is a preliminary report of the experiments carried out at the ESRF past July (CH-1236), which is being submitted together with a new application form. The full report will be submitted on due time next September according to the Instructions given in the web page (http://www.esrf.fr).

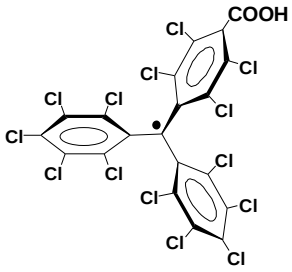
The main objective of the work was the structural resolution, using Synchrotron Radiation facilities, of magnetic two- and three-dimensional open-framework structures based on metal ions and organic radicals acting as ligands. The exceptional characteristics of nanoporous materials have prompted their application in different fields such as molecular sieves, sensors, ion-exchangers, and catalysis. The use of synchrotron radiation facilities was highly required due to recurrent obtaining of really small single crystal, even too small to be solved by conventional CCD techniques. Moreover, their high insolubility difficult any recrystallization experiment to improve the size as well as the quality of the crystals so far obtained.

The results obtained during the last experiment (12 shifts) were really impressive. During the four days used for the experiment, nine structures were resolved, three of them exhibiting interesting nanoporous structures with very large pores, in the 3-4 nm range, and intriguing intramolecular connections. Hereby is the list and a brief description of the aforementioned structures and the most remarkable results:

A) A complex of Co(II) with the radical **2** in a stoicheometric form of 3:2, with molecules of pyridine completing the octahedral geometry of the Co(II) ions. The unit cell of such complex has a large volume, nearly 27000Å^3. Moreover, its crystal packing shows the presence of nanopores.



(1)



(2)

B) Two different phases of a supramolecular complex composed of radical **1**, which acts as an anion, and a complex of Co(II) with 2,2'-bipyridine, which acts as a cation. The supramolecular interactions such as hydrogen bonds and ?-? interactions between these two components originate large nanopores with size of ca. 3 nm.

C) Two different crystalline phases of radical **1**. One of these two structures is very interesting because forms polar micropores due to the presence of eight carboxylic groups forming the walls of such channels.

D) A monomeric Co(II) complex with an octaedrical geometry formed by two molecules of radical **2** and four water ligands.

E) A monomeric Zn(II) complex with an octaedrical geometry formed by two molecules of radical **2** and four water ligands.

F) A complex of Co(II) with radical **2** and 4,4'-bipyridine.

G) A complex of TTF with Cu(I) and Cu(II) ions, which forms a three dimensional framework.

Now, we are working to refine all these structures. Moreover, we are analyzing the behavior of complexes with nanopores as zeotype materials and studying their magnetic properties. We expect that at the end of September three or four different papers containing the structures solved in ESRF will be completed and submitted.

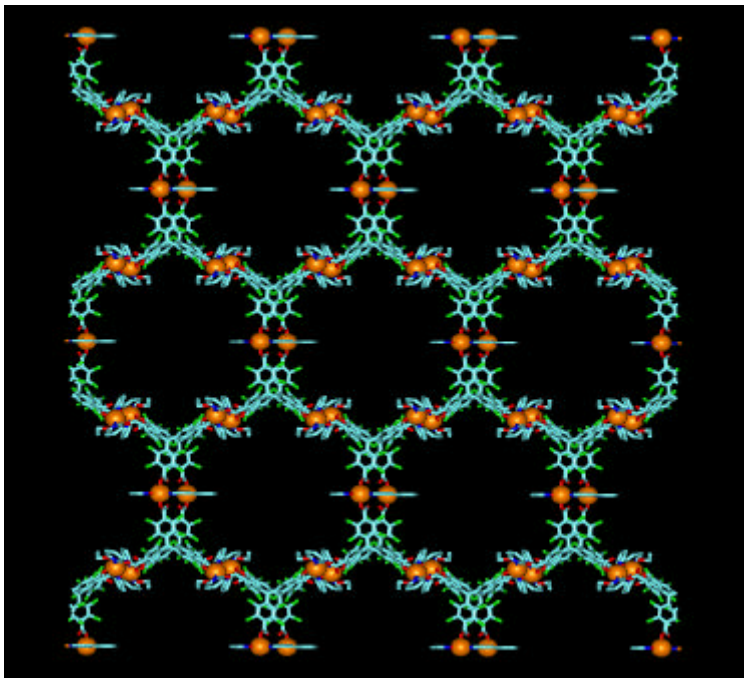


Figure 1. A previous complex synthesized with radical **2** showing open-framework structure with large nanopores.