EUROPEAN SYNCHROTRON RADIATION FACILITY

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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:** **Crystallographic structure of $Cu_7[W(CN)_8]_4 \cdot xD_2O$ molecular magnet** | **Experiment number**: HE-2962 |
|---|---|---|
| **Beamline**: | **Date of experiment**: from: 24/09/2008 to:29/09/2008 | **Date of report**: 28/08/2009 |
| **Shifts:** | **Local contact(s)**: Olga Safonova | *Received at ESRF:* |


Names and affiliations of applicants (* indicates experimentalists):

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**Report:**

The main aim of the project **HE -2962** was determination of nuclear structure and lattice parameters of the molecular magnet $Cu_7[W(CN)_8]_4 \cdot xD_2O$ at low temperatures.

On the basis of magnetic measurements we know that a sharp transition to the magnetically ordered state occurs at $T_c \sim 40$ K. It was important to check the crystal structure in the magnetic phase and to learn whether it changed upon the transition at $T_c$.

We used the BM01 synchrotron diffractometer for the experiment with the x-ray wavelength $\lambda = 0.4983$ Å. To avoid any extra peaks the powder sample was placed in the capillary without any additive.

We performed scans at temperatures 5, 35, 50, 100 and 200K, that is below and above the magnetic ordering temperature. Fig.1 shows the diffraction patterns at these temperatures. It is necessary to mention that we started measurements at room temperature (RT) and next continued them at decreasing temperatures. After returning to RT the sample turned out to be destroyed (the beam effect). So we decided to restart measurements with the new part of the sample and began with the run at 5K. Because the crystallographic structure at RT of the investigated sample was known from the XRD diffraction, we could recognize when the sample started to change the structure. The changes were observed at 200K – they are visible in the insert in Fig.1.

The FULLPROF refinements reveal that the tetragonal *I4/mmm* structure is preserved in the whole range temperature (5 - 300 K). Fig.2. gives the observed and the refined spectrum at the temperature of 5 K The lattice constants ***a*** and ***c*** obtained from the refinement are collected in Table 1.

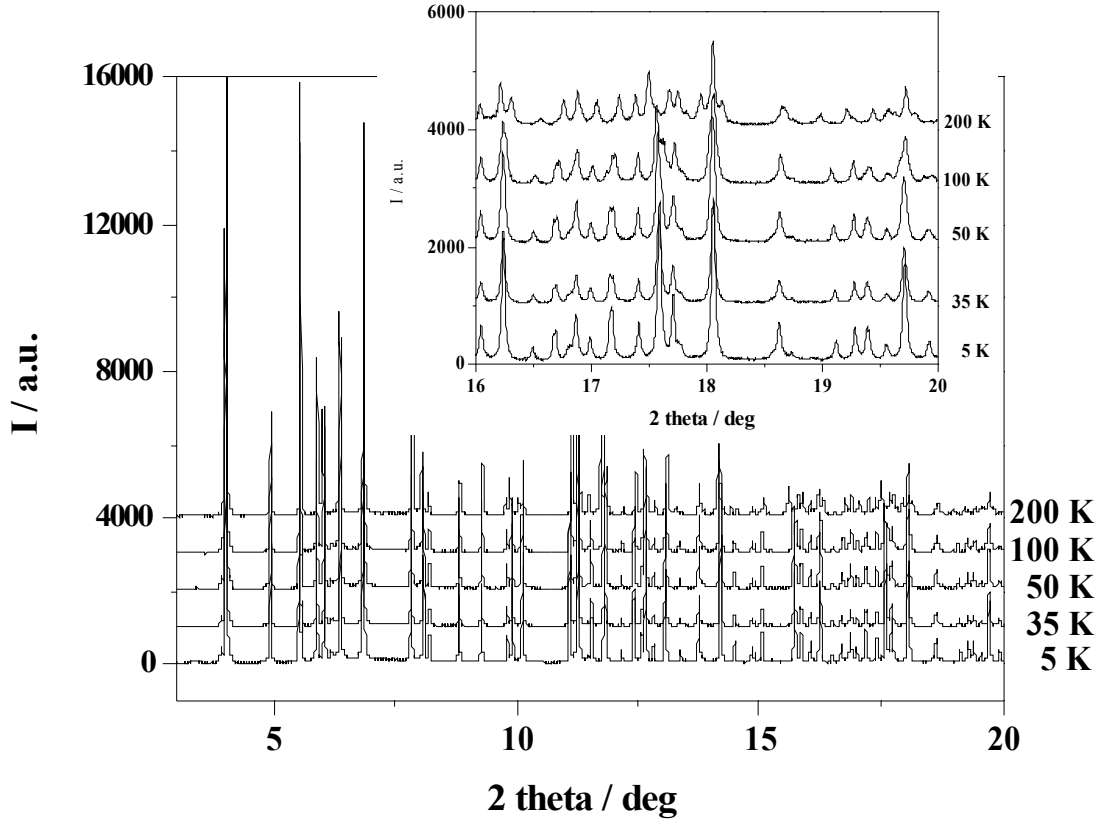


**Fig.1. Synchrotron-diffraction spectra of $Cu_7[W(CN)_8]_4 \cdot xD_2O$ taken at 5, 35, 50,100 and 200 K. The insert shows that at 200 K the sample changes its structure what is interpreted as a destruction of the material caused by the synchrotron beam.**

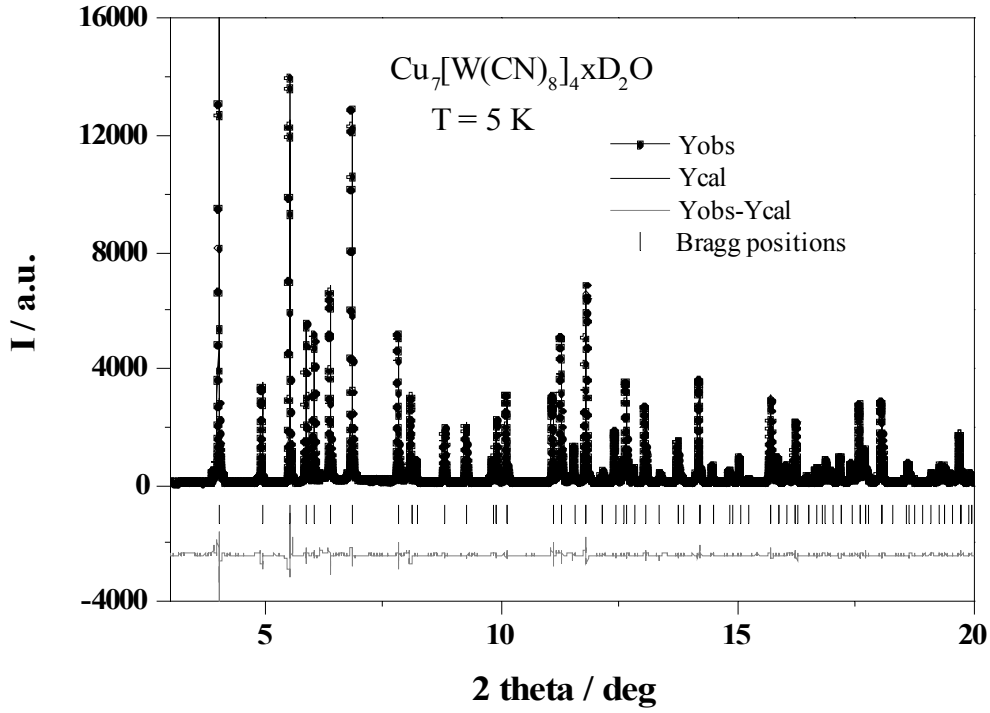


**Fig.2. The Fullprof refinement of the synchrotron pattern obtained at 5K for $Cu_7[W(CN)_8]_4 \cdot xD_2O$.**

**Table 1.**
**Lattice constants *a* and *c* obtained from the synchrotron experiment (5 – 200 K) and from the classical (300 K) x-ray diffraction.**

| *T* /K | *a* /Å | *c*/ Å | *V* /Å$^3$ |
|---|---|---|---|
| 5 | 7.2770 | 28.2103 | 1493.89 |
| 35 | 7.2771 | 28.2107 | 1493.93 |
| 50 | 7.2741 | 28.2374 | 1494.12 |
| 100 | 7.2745 | 28.2381 | 1494.30 |
| 300* | 7.2758 | 28.2609 | 1496.06 |

*** -** Result obtained at the X'Pert Pro Panalytical diffractometer for powder sample.

It follows from Table 1 that a small increase of lattice parameter ***c*** and a very slight decrease of lattice parameter ***a*** is observed when temperature is changed from below to above $T_c$. However, the volume of the unit cell increases monotonically as a function of temperature.

The experiment has shown that the magnetic transition at $T_c \sim 40$ K in the investigated molecular magnet $Cu_7[W(CN)_8]_4 \cdot xD_2O$ is not connected to the change of the crystal structure.