

## 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 via the User Portal:

<https://www.esrf.fr/misapps/SMISWebClient/protected/welcome.do>

### ***Reports supporting requests for additional beam time***

Reports can 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.



**Experiment title: Field induced internal Fe and Ln spin reorientation in “butterfly” {Fe<sub>3</sub>LnO<sub>2</sub>} Single Molecule Magnets**

**Experiment number:**  
HC-661

<b>Beamline:</b> ID08	<b>Date of experiment:</b> from: 11/09/2013 to: 16/09/2013	<b>Date of report:</b> 31/03/2014
<b>Shifts:</b> 18	<b>Local contact(s):</b> Erika Jimenez Romero	<i>Received at ESRF:</i>

**Names and affiliations of applicants (\* indicates experimentalists):**

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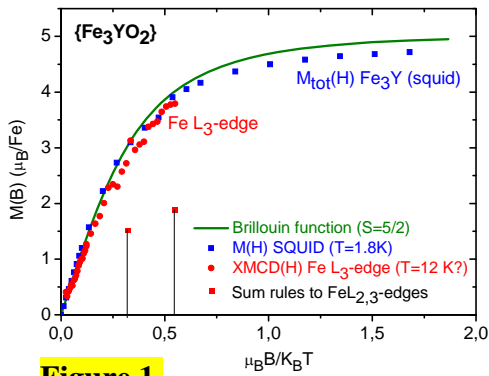
**Report:** Single Molecule Magnet (SMM) behaviour has been observed on a series of compounds of the type [Fe<sub>3</sub>Ln(μ<sub>3</sub>-O)<sub>2</sub>(CCl<sub>3</sub>COO)<sub>8</sub>(H<sub>2</sub>O)(THF)<sub>3</sub>], where Ln represents the lanthanide cations (Gd, Tb, Dy, and Ho) and Y(III) as pseudo-lanthanide<sup>1</sup>. The Ln atom provides the anisotropy, while the Fe<sub>3</sub> subcluster imposes the magnetic moment. In our previous work<sup>2</sup>, it was reported that upon application of an increasing magnetic field, the two magnetic subsystems (Ln and Fe<sub>3</sub> sublattice) undergo polarization in the field direction. In particular, it was shown that in the Gd and Dy compounds, the Ln is which dominates the total magnetization of the molecule. The present experiment was aimed at deeping in the study of the Fe<sub>3</sub> subcluster-Ln interaction within the {Fe<sub>3</sub>LnO<sub>2</sub>} cluster, by applying a competing external field and low temperatures, to provoke relative reorientation of the Fe and Ln magnetic moments.

In order to determine **directly** the spin and orbital moments of both sublattices (the Fe<sub>3</sub> subcluster and the Ln atom), as well as their dependence with the magnetic field, XAS and XMCD experiments were performed at the Fe L<sub>2,3</sub>-edges and Ln M<sub>4,5</sub>-edges on the {Fe<sub>3</sub>LnO<sub>2</sub>} compounds, with Ln= Y, Gd, Dy, Tb and Ho. Additionally, XMCD(H) measurements in the hysteresis mode were performed by varying the applied field (-9 T < H < 9 T), at the Fe L<sub>3</sub>-edge and Ln M<sub>4</sub> or M<sub>5</sub> edge, to determine the orientation of the Fe sublattice magnetization and the Ln one, respectively.

Powder samples were prepared by pressing them onto a double-face carbon tape, which was held in place on the sample platform by means of a copper mask with four teflon screws. Radiation damage tests were performed by plotting the XAS spectra as a function of the time, but no evolution of the XAS signal with time was observed.

The Fe sublattice hysteresis curves were obtained “point by point”. That is, by fixing an applied field and scanning in energy along the Fe L<sub>3</sub>-edge. Then, by following the high intensity XMCD peak obtained for several fields, ranging between -9 to 9 T, one obtains the XMCD(H) for the Fe sublattice contribution.

The first problem arises in the difference between sample and holder temperatures (T). Although, the thermometer placed closest to the sample indicated a T ≈ 3.6 K during the experiment, the analysis of the experimental data indicates a much higher one.

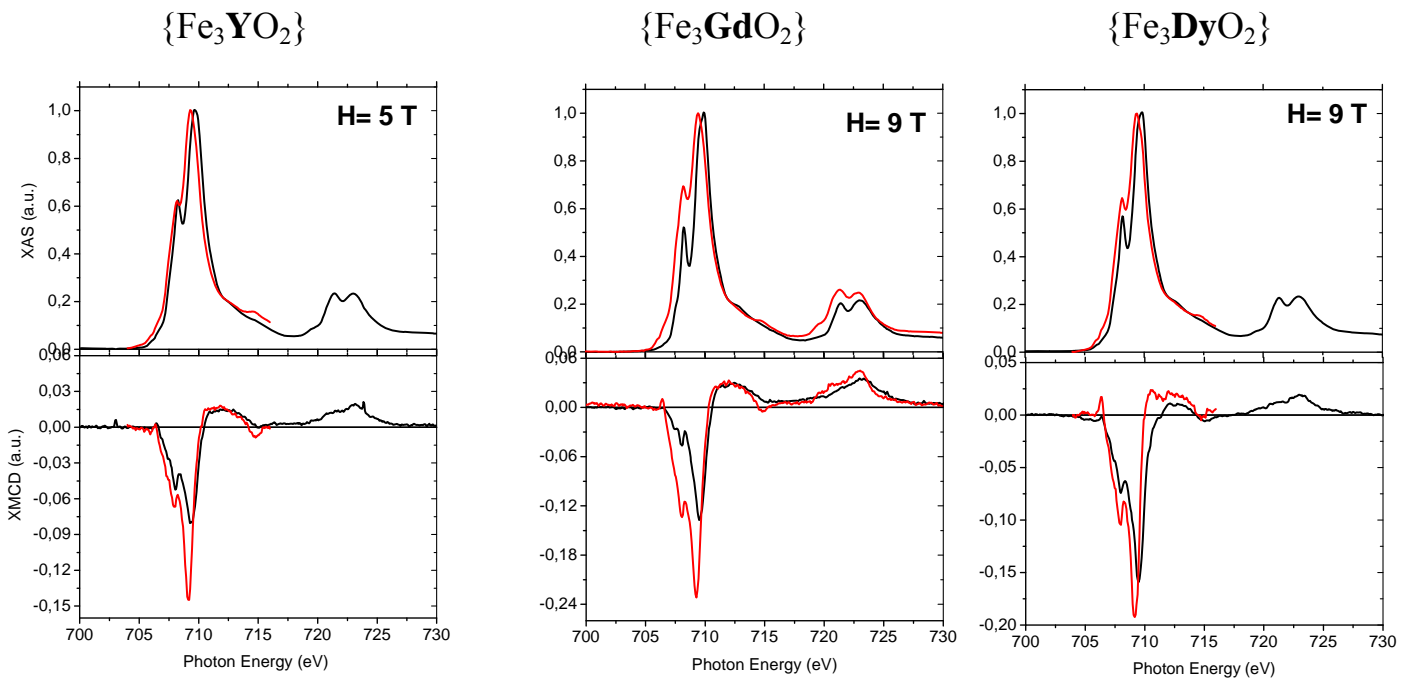


**Figure 1.**

is displayed in **Figure 1**, red circles. According to the ID08 beamline staff, a sample temperature of about 5 K would be reasonable, so the obtained  $T=12$  K indicates the existence of a lack of thermal contact between sample and holder.

The second aspect to report is the non-reproducibility of the XAS and XMCD spectra, obtained at the Fe  $L_{2,3}$ -edges. Comparing these spectra, measured at the highest available field (9 T), with those obtained from the XMCD(H) curve at the same field (**Figure 2**), we realize that both measurements are not consistent. Indeed, **Figure 2** shows that there is a little shift in energy on both, XAS and XMCD, spectra measured in two different moments, i.e. during the Fe  $L_{2,3}$  scan or during the hysteresis measurement. Moreover, some differences can be discerned in the spectra shape, which imply different areas for the Fe  $L_3$  and  $L_2$  peaks. If both experiments were performed in the same conditions, i.e., on the same sample and with the same pressure, temperature, applied field and procedure of measurement, the only difference is that they were performed at a different time. Our explanation is that the sample temperature is not controlled. The sample preparation method could have an influence on this effect, since when we took out the sample, we realized that the carbon tape was broken into pieces because of the low temperature, and detached from the holder. Moreover, less powder sample than in the initial state remained.

In view of the non-reproducibility of the experimental data, we conclude that the HC-661 results are not useful to our research.



**Figure 2.** Comparison of XAS and XMCD spectra at the Fe  $L_{2,3}$ -edges on different compounds. Black lines indicate the XAS and XMCD complete spectra measured at the Fe  $L_{2,3}$ -edges range of energies. While red lines refer to the spectra obtained from the hysteresis measurements, i.e. XMCD(H) at the Fe  $L_3$ -edge.

<sup>1</sup>J. Bartolomé et al. Phys. Rev. B 80, 014430 (2009)

<sup>2</sup>L. Badía-Romano et al. Phys. Rev. B 87, 014403 (2013)