Experimental report

07/12/2016

Proposal: 5-31-2460

Council: 4/2016

Title: Magneto-structural correlations onnew bimetallic-anionic paramagnetic ionic liquids with 3D magnetic ordering in its solid state.

Research area: Materials

This proposal is a new proposal

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Experimental team: Manuel DE PEDRO DEL VALLE
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Samples: compounds based on (Î¼-Oxo)bis[trihaloferrate(III)] anions and imidazolium cations

Instrument	Requested days	Allocated days	From	To
D2B	2	2	13/09/2016	15/09/2016
D1B	2	3	11/09/2016	13/09/2016

Abstract:

Magnetic ionic liquids (MILs) are molten salts formed entirely of ions, which have a melting point below 100 ºC. The great variety of possible combinations of anions and cations makes them a huge potential field of development of smart materials with a primary focus in Materials Science; supported by the growing interest of chemists, physicists, and materials scientists. In the search of developing new MILs with enhanced magnetic response at room temperature, we have synthetized a new family based on imidazolium cation and [Fe2OX6]- anion. Therefore, in this proposal we ask for 2 days at D2B high resolution diffraction instrument to obtain accurate magnetic structure characterization and phase transition of two new MILs with formula Dimin[Fe2OX6]- (X = Cl and Br). The magnetic structure characterization of these AF compounds with polar space group will be carried out using D1B high flux and medium resolution diffractometer, for that we ask for two days of beam-time.

Experimental report 7-03-155

Title of Experiment:	Magneto-structural correlations on new bimetallic-anionic paramagnetic ionic liquids with 3D magnetic ordering in its solid state.	**Local Contacts:** **Oscar Fabelo** **Fernandez diaz Maria Teresa**
Principal Proposer:	Manuel de Pedro del Valle	**Instrument: D1B y D2B**
Experimental Team:	Manuel de Pedro del Valle/Palmerina Gonzalez Izquierdo/Oscar Fabelo/ Fernandez Diaz Maria Teresa	**Date of Experiment:**

In the search of developing new Magnetic Ionic Liquids (MILs) with enhanced magnetic response at room temperature, we have synthetized a new family of MILs based on imidazolium cation and $[Fe_2OX_6]^-$ anion, $DiminFe_2OX_6$ (X = Cl and Br). They show a much larger effective magnetic moment due to the doubled concentration of iron centers per mole. In addition, at low temperatures, the magnetic susceptibility exhibit a 3D maximum at approximately 4.6 K and 8.2 K for $DiminFe_2OCl_6$ and $DiminFe_2OBr_6$ respectively.

We have two main objective of the proposed proposal: determine the magneto-structural correlations (D1B) and the solid-solid phase transitions (D2B) of $DiminFe_2OX_6$ (X = Cl and Br). Initially, about 2 g of $DiminFe_2OCl_6$ were milled and placed in a vanadium container at room temperature. After that, we introduced the sample in the cryostat and we try to cool down the sample up to 1.5 K (below the 3D ordering) to perform the magnetic study. Unfortunately, a problem with the cryostat did not allow it to cool below 5 K nor to stabilize the temperature. We contacted with the responsible of the instrument, (across the phone call because was Sunday), and she decided to change the cryostat on Monday. We can only perform a study of the structural part doing a ramp from 5 K to RT in continuous mode.

On Monday, after the new cryostat was installed (after lunch) we change the other sample, $DiminFe_2OBr_6$. Again, the problems were persisting. So we decided to measure this sample from 5 K to RT in continuous mode to observe the thermal variation in-detail. After these technical problems it was decided that we would be reimbursed with 1 day of beam time when it will be possible.

For the D2B instrument, we decided to measure these sample at selected temperatures (in the range $1.5<T<300$ K) to observe with good statistics above and below the magnetic and structural transitions. These experiments will allow us to determine the crystal structure and phase diagram. The presence of H atoms in the cation part (H = 16) predicted low statistic in the neutron spectroscopy pattern. Therefore, we measured above 8 hours per spectra (3 patterns for sample).