Experimental report

13/06/2016

Proposal: 5-51-502 **Council:** 10/2014

Title: Spin and charge densities in simple water-bridged TM-dimer complexes using a combination of polarized neutrons and X-ray diffraction

Research area: Chemistry

This proposal is a continuation of 5-51-498

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Samples: Co(3pic2)
Ni(py2)

Instrument	Requested days	Allocated days	From	To
D3	9	9	11/07/2015	20/07/2015
D9	7	7	04/05/2015	11/05/2015

Abstract:

In this continuation proposal we again plan to collect flipping ratios in order to obtain magnetization densities. The two compounds that we focus on this time are structurally simple water- and pivalate-bridged transition metal dimer complexes.

In parallel to these experiments we are already carrying out single-crystal X-ray diffraction with the same compounds at very low temperature at a dedicated synchrotron beam line at SPring8 and with the flipping ratios at hand we will co-refine multipole models of spin and charge densities (directly from measured flipping ratios and structure factors) for these compounds.

The properties of these spin and electron densities, derived using for instance Atoms in Molecules tools, will be correlated to the extensive magnetic properties already measured on this class of compounds, with the hope to see new connections between magnetism and spin/charge densities.

Experimental beamtime report for Proposal: 5-51-502

Title: Spin and charge densities in simple water-bridged TM-dimer complexes using a combination of polarized neutrons and X-ray diffraction

Proposal 5-51-502 involved the measurement of flipping ratios for the monoclinic crystal structure of the compound $Co_2(H_2O)(O_2CC(CH_3)_3)_4(HO_2CC(CH_3)_3)_2(3\text{-Mepy})_2$. The idea has been to use the experimentally derived spin density to study the magnetic exchange interaction between the two Co atoms, with particular focus on the pathway. From magnetic data, it seems to be a ferromagnetic interaction but this is very difficult to rationalize based on those measurements.

The initial attempts to cool down the crystal resulted in broken crystals and we had severe problems with obtaining the first data. At the time that we had found a suitable crystal, the neutron flux disappeared due to technical problems.

The crystal was kept cold until July where neutrons came back and the measurement was restarted. Local staff managed to find a suitable crystal and the data was eventually collected.

In the meantime, we have tried several synchrotron data collections to obtain matching X-ray data for joint charge and spin density refinement, but have not been successful. Therefore, the current study will only involve polarized neutron data and the spin density itself.

The data analysis is currently completed, but we are awaiting theoretical results to back up our analysis of the results. A publication will be submitted before the end of the year.