

Experimental report on previous beamtime usage

The present report summarizes the findings resulting from our previous experiment at SNBL, on the proposal submitted for the 4/2021 proposal round, and carried out in 2022.

1. Structural resolution of $Mg_5(en)_6(BH_4)_{10}$

A highly complex crystal structure of stoichiometric $Mg_5(en)_6(BH_4)_{10}$ was solved from single crystal synchrotron X-ray diffraction at SNBL, and later confirmed by neutron powder diffraction (NPD) on isotopically substituted $Mg(en)_{1.2}(^{11}BD_4)_2$. We highlighted the role of the amorphous $Mg(BH_4)_2$ in the reactivity of the $Mg(BH_4)_2$ -en system and characterized a previously overlooked phase, $Mg(en)_2(BH_4)_2$.

The results of this work have been published in *Dalton Transactions*, 52, 8, 2404-2411, **2023** "Structural insight into the magnesium borohydride–ethylenediamine solid-state Mg-ion electrolyte system"

2. Structure of a new Mn-based derivative of the MIL-53/47 MOFs

A new MOF, composed of infinity chains of Mn(III) bridged by terephthalate and methoxy ligands has been discovered by means of synchrotron powder and single crystal diffraction at SNBL. Owing to the similarities with those frameworks, we expect that this MOFs possesses rich redox chemistry, that might be similar to MIL-47, and that ligand modification might lead to pore size and modifications in breathing behaviour, as has been previously observed for the MIL-53 series. Work in this direction is currently underway and promising results, to be published soon, have already been obtained.

3. Thermally induced structural changes in NH_2 substituted LnBTC MOFs

We obtained a new kind of LnBTC (Ln = lanthanide) MOFs, also known as MOF-76, from aminotrimetic acid instead of the classical non-substituted trimetic acid. The influence of the presence of the amine group on the stability and fluorescent properties have been studied in house, and were complemented by variable-temperature synchrotron X-ray diffraction at SNBL to determine the structural changes occurring in these MOFs upon heating. Those changes have been monitored for the Tb and Eu-based MOFs with and without amine on the linkers, revealing an interesting flexible behavior. Furthermore, the experiments revealed a strong X-ray induced fluorescence in the green region of the spectrum for NH_2 -MOF-76(Tb). The results obtained from this work are currently part of a manuscript to be submitted soon.