Laboratory Experiment Proposal Submission

Experimental Details

Experiment location: A115
Experiment title: Control of Magnetization Using External Stimuli in Coordination Polymers
Experiment date: ongoing
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Chemicals Used

Chemical Name	Health	Flammability	Reactivity	Special Hazards
Deuterium Oxide	Blank	Blank	Blank	NONE
Potassium Hexacyanochromate(III)	1	Blank	Blank	
Potassium Hexacyanoferrate(III)	1	Blank	Blank	NONE
Iron(II) Chloride	2	Blank	1	NONE
Cobalt(II) Chloride	2	Blank	Blank	NONE
Nickel(II) Chloride	2	Blank	Blank	NONE
Copper(II) Chloride	2	Blank	Blank	NONE

Reactants and Resulting Samples

Chemical Name	Hazardous?	Known Hazards
Prussian Blue Analogues	N	

Required Safety Equipment

- [x] Hood Organics

Required Laboratory Equipment

- [x] Balance
- [x] Centrifuge
- [x] Hot Plate
- [x] Dls
- [x] Ftir
- [x] Glovebox
- [x] Uvvis Spectrometer
- [x] Xray Diffractometer

Experimental Write Up

1. Weigh all reagents. 2. Make solutions of reagents using D2O as the solvent, these solutions will be in stoppered flasks with two access ports, one for inert gas (nitrogen is OK) and one for general access. Specifically, the solutions are: - Solution (a). add 0.125 grams of mCl2 to 100 mL of D2O (where m =, Fe, Co, Ni, or Cu), resulting in a concentration of 10-1 M - Solution (b). add 0.328 grams of K3M(CN)6 to 50 mL of D2O (where M =Fe or Cr), resulting in a concentration of 2 x 10-2 M - Solution (c). add 0.746 grams of KCl to 50 mL of D2O, resulting in a concentration of 2 x 10-1 M 3. Stir solutions at room temperature via stir bar. 4. Pour solution (b) into solution (c) and continue mixing to yield solution (b+c), no chemical reaction takes place during this step. 5. Over a few hours, slowly and continuously add the contents of solution (a) to solution (b+c). During this addition, the system is not to be exposed to H2O containing atmosphere. All liquids will be transferred in tubes to and from stoppered flasks. This transfer may be achieved via peristaltic pumping. For some complexes (when m = and M =), it is necessary to apply external heat to facilitate the reaction, this is done via a hot plate turned to 50-75 degrees C. For the heating process, solution (a) is kept warm during the addition process. During this addition, the ions that are in solution form coordination networks. The formation of the product may be detected by eye as the solution changes from a colored liquid to a cloudy liquid of a different color, and the cloudiness is actually the product precipitating out. -Solution (b+c) contains "M(CN)6" ions and "K" ions (in addition to "Cl" and water-based ions) -Solution (a) contains "m" ions (in addition to "Cl" and water-based ions) The chemical reaction is simply: (b+c) + (a) = 4*m + 3*M(CN)6 + 6*D2O =4[M(CN)6]3[(D2O)6] Note, the written reaction is representative of what occurs, although non stoichiometric formulas are often the norm for these compounds. Also, there are many potassium ions that do not become incorporated into the lattice and simply remain in solution even after the product is formed; however, a high concentration of K is used to ensure the correct stoichiometry during the crystallization process due to the kinetics of the particle ripening. 6. Until the experiment is performed, samples will be stored in solution to avoid D to H exchange. One possibility is to keep the samples in sealed flasks filled with inert gas (nitrogen is OK) covered to protect from light exposure and stored in a refrigerator. 7. Prior to the experiment, samples are to be centrifuged and vacuum dried (to a degree) before transfer to an experimental sample canister, and this transferring of the product is to be performed in a glovebox. 8. In addition to neutron scattering experiments, XRD, FTIR, UV-VIS, and DLS will be performed on a portion of the product.

Experimenter Signature: _____ Date: _____

Lab Responsible Signature: _____ Date: _____