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Dehydration of Scandium for RERecycling and Synthesis of Nano Materials

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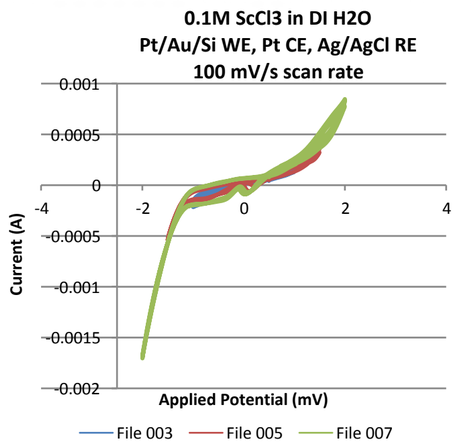
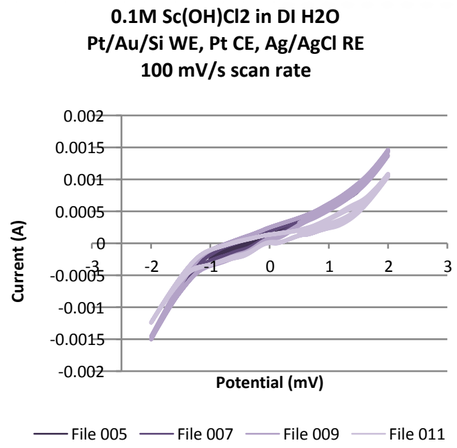
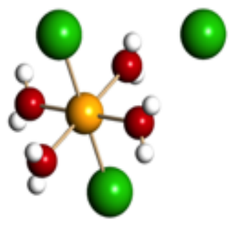
Introduction

Scandium is used in production of high powered lights, fuel cells and sonar equipment. Despite its many uses in industry, scandium is very expensive due to its low levels of pure ore found in nature. With Scandium's importance to industry and its rarity of pure metal it is considered to be nationally critical. Because of its cost very little research has been done on scandium and its properties. Scandium's electrical properties in nanoparticles and Nano-inks (N-inks) are of particular interest to be used in additive manufacturing.

Electrochemistry





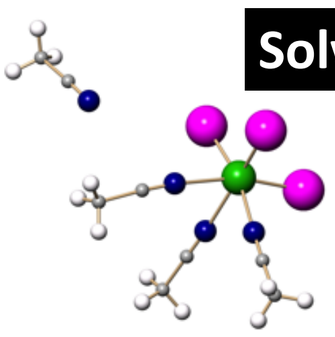


$[Sc(Cl)_2(H_2O)_4]Cl \bullet H_2O$

A 0.1M solution of $ScCl_3(H2O)_6$ in 8ml of deionized water and underwent electrochemical reduction in a cyclic voltammetry cell. No reduction peaks were observed so we sought to synthesize new precursors.

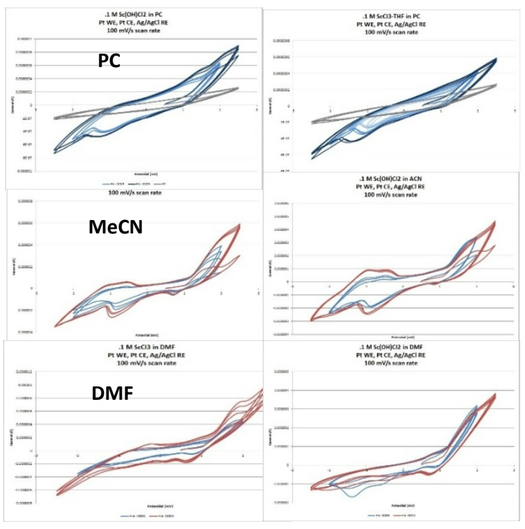
Research approach and results

Solvated species



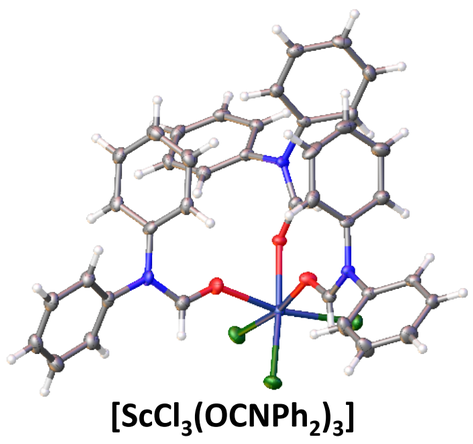
$[ScCl_3(DMF)_3]$

Dry solvated scandium species were synthesized by dissolving ScCl3(H2O)6 in a solution of SOCl2 and THF at room temperature. The dry precursor was then dissolved in various solvents



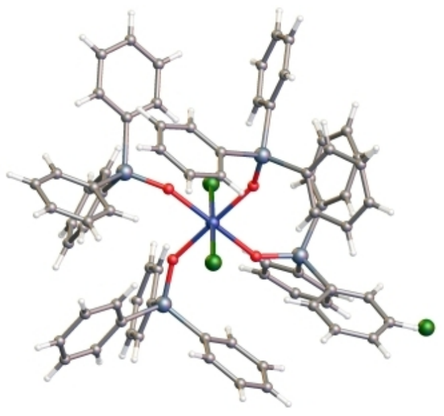
The lower than expected reduction peaks led us to believe that scandium was actually catalytically reducing the organic solvents present in each cell.

New route to dehydration



$[ScCl_3(OCNPh_2)_3]$

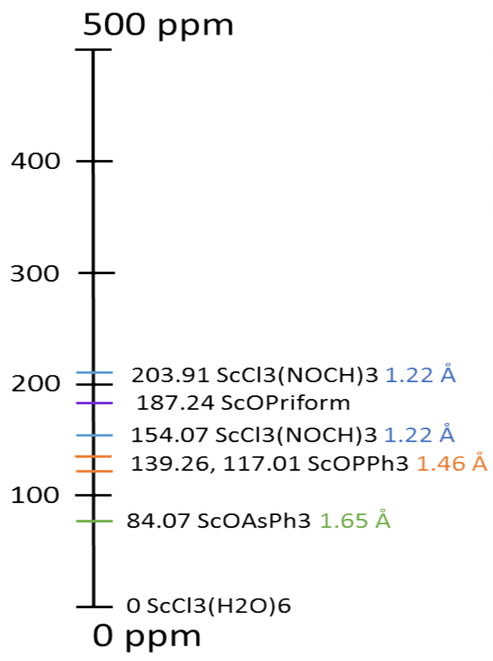
Ligands with oxygen double bonds to carbon, sulfur, nitrogen, phosphorus, and arsenic were shown to readily react with $ScCl_3(H2O)_6$.



$[ScCl_2(OAsPh_3)_4][Cl]$

This route requires use of only one precursor as opposed two precursors and longer reaction time with the solvated routes.

Patterns in 45Sc NMR shifts

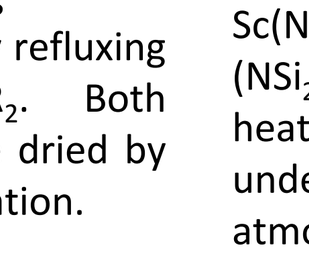


45Sc NMR is a valuable tool to characterize scandium complexes. 45Sc NMR shifts were investigated for patterns based on bond length of ligands with oxygen double bonded to carbon, arsenic, sulfur and phosphorous.

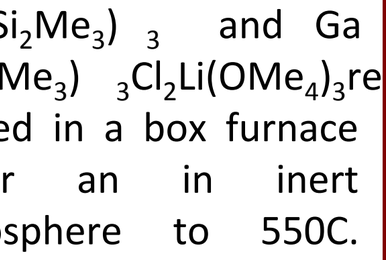
Scandium and Gallium Alloy Synthesis

$Sc(NR2)3 + Ga(NR2)3 \xrightarrow{550C}$

$Sc[N(Si_2Me_3)]_3$ was synthesized by refluxing $ScCl_3(THF)_3$ in KNR_2. $Ga[N(Si_2Me_3)]_2Cl_2Li(THF)_3$ was synthesized by refluxing $GaCl_3$ in $LiNR_2$. Both products were dried by vacuum distillation.



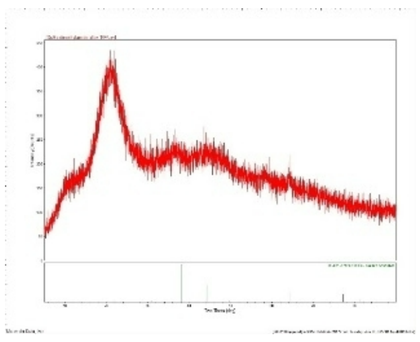
$Sc[N(Si_2Me_3)]_3$



$Ga[N(Si_2Me_3)]_2Cl_2Li(THF)_3$

$Sc(NSi_2Me_3)_3$ and $Ga(NSi_2Me_3)_3Cl_2Li(OMe_4)_3$ re heated in a box furnace under an in inert atmosphere to 550C. The resultant product was characterized using PXRD with a plastic dome (PeD).

The resultant PXRD spectrum shows a mixed Sc and Ga alloy in a 3:1 ratio.



Summary

Many goals have been accomplished in this project:

- Synthesis of dehydrated scandium solvates.
- Simple scandium dehydration with O= ligands.
- Correlate 45Sc NMR shifts to bond length of O double bonded to carbon, arsenic, sulfur and phosphorous.
- Synthesis of scandium and gallium alloys.

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