ASP Volunteer: Synthesis of Tin Phosphide Nano Particles

SAND2015-6392PE

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U.S. DEPARTMENT OF ENERGY

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Exceptional service in the national interest

1st Semester

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Safety before entering lab:

-BEA100- Beryllium Awareness Training
-CHM100-Chemical Safety Training
-CHM103-Site-Specific Chemical Safety Training
-NANO101- Nanotechnology Safety Awareness Training
-RAD214- Radiation Generating Device Safety Training
-ESH100- Environmental Safety and Health
-ENV112- Hazardous Waste & Environmental Management Training
-PRS150- Pressure Safety Orientation
-PRS115- Cryogen Safety Orientation

Safety In the lab:

- Always wear our PPE
- Familiarize ourselves with hazards in the lab
- Read the MSDS for all chemicals you are working with.

1st Semester Continued

Sandia National Laboratories

CSI Dognapping (T. J. Boyle & BAHS)

- hands on investigation for 4th graders
- magic show is cancelled due to someone stealing the magic chemistry dog
- make observations & collect clues on their journey to find the mysterious “dognapper”.

Research at the AML

Tin and Phosphorous Bulk Properties

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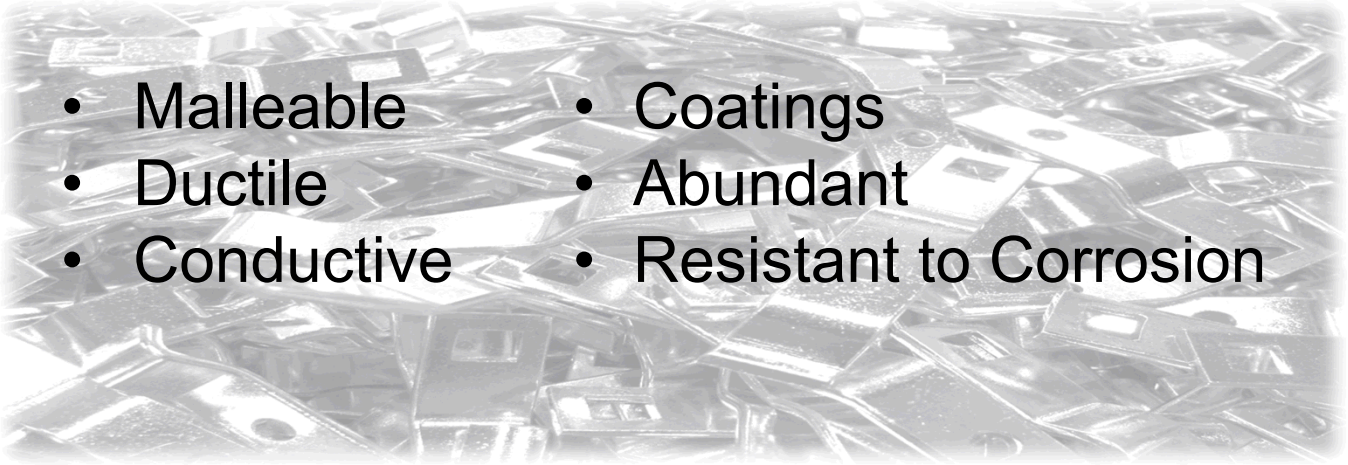
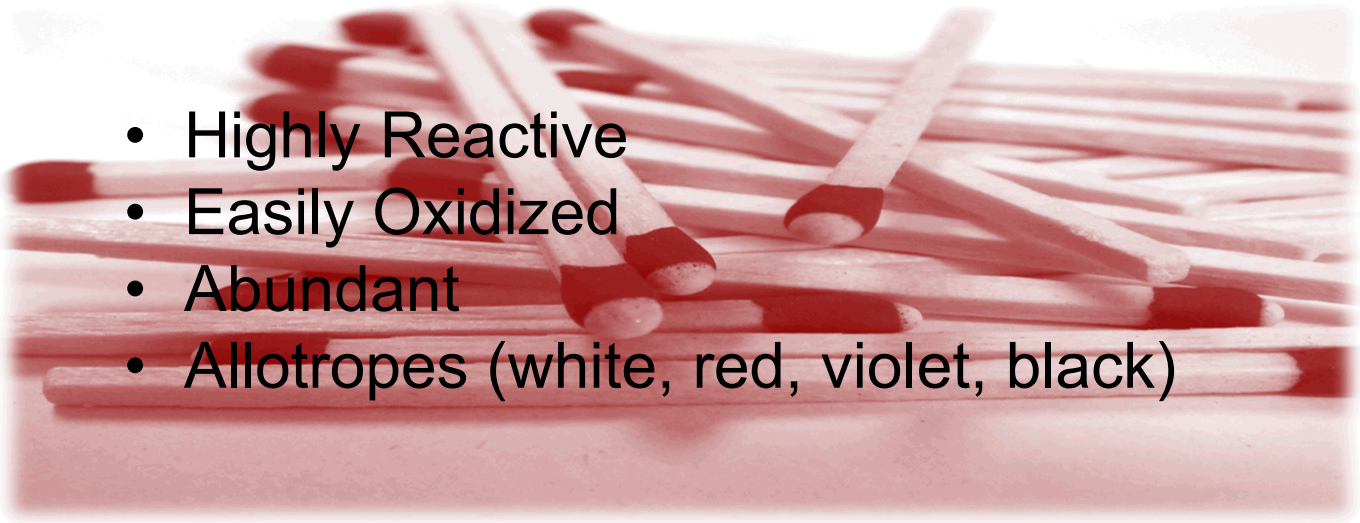
tin
50
Sn
118.71



- Malleable
- Ductile
- Conductive
- Coatings
- Abundant
- Resistant to Corrosion

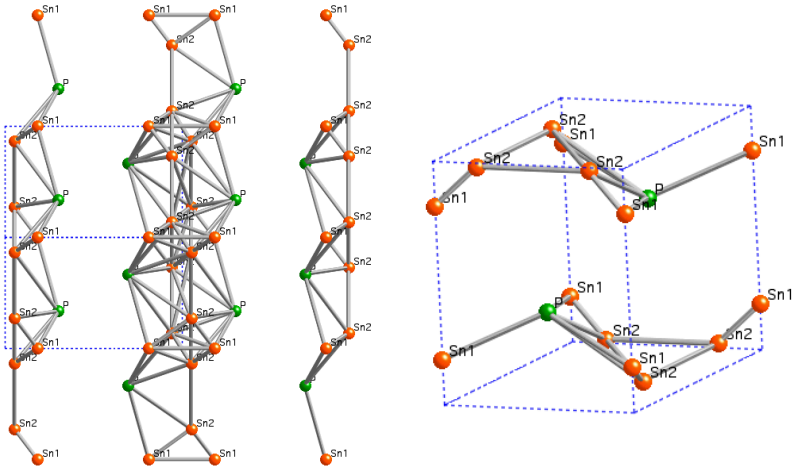
phosphorus
15
P
30.974



- Highly Reactive
- Easily Oxidized
- Abundant
- Allotropes (white, red, violet, black)

Tin Phosphides

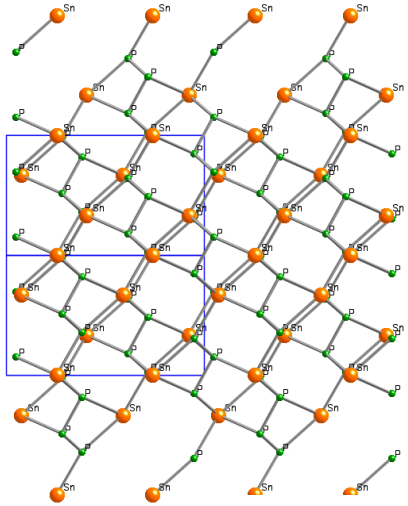
SnP
Trigonal unit cell
a =4.3922 Å
c = 6.040 Å

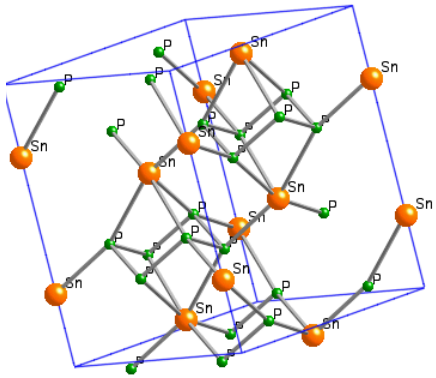


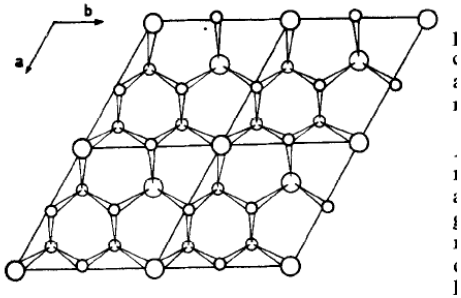
JAN GULLMAN, JOURNAL OF SOLID STATE CHEMISTRY 87,202-207 (1990)

SnP_3
Trigonal unit cell
a = 7.378 Å
c = 10.512 Å

Sn_3P_4
Sn_4P_3







JAN GULLMAN, JOURNAL OF SOLID STATE CHEMISTRY 5, 441-445 (1972)

Tin Phosphide Applications

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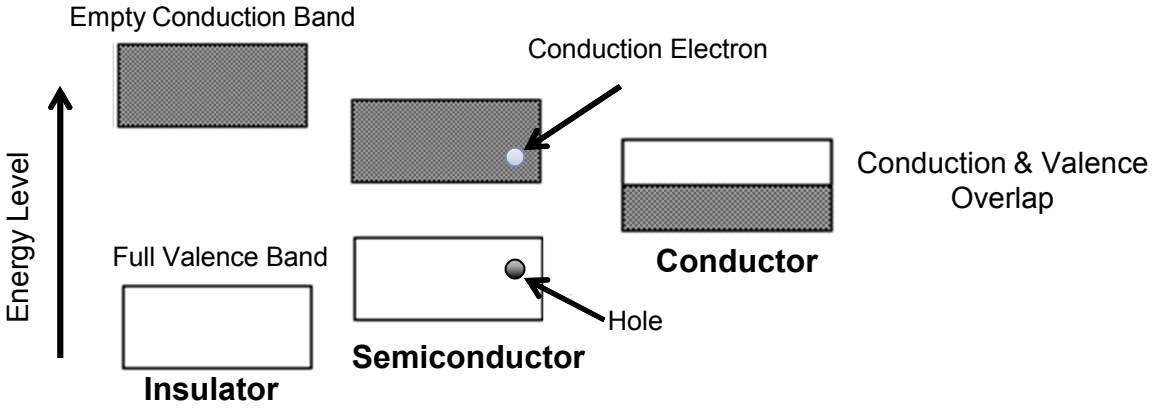
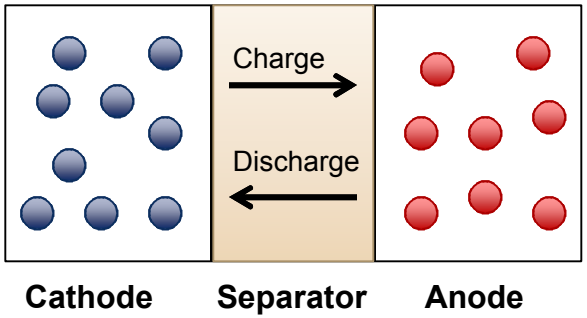
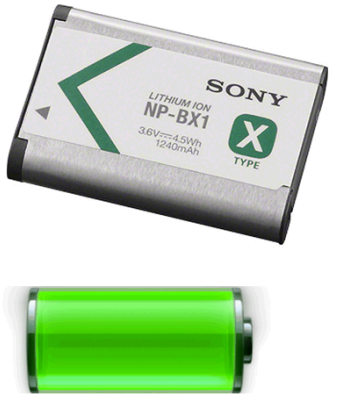
Semiconductor Properties



Anode Materials for Batteries

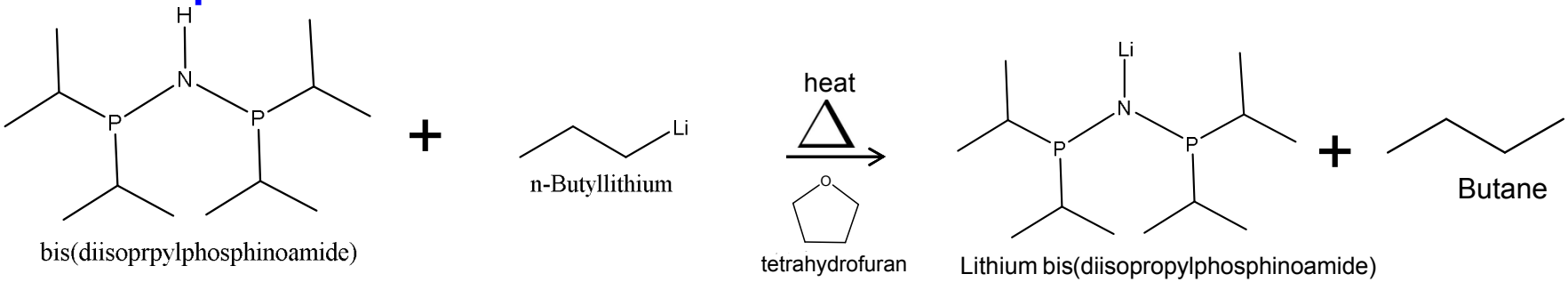




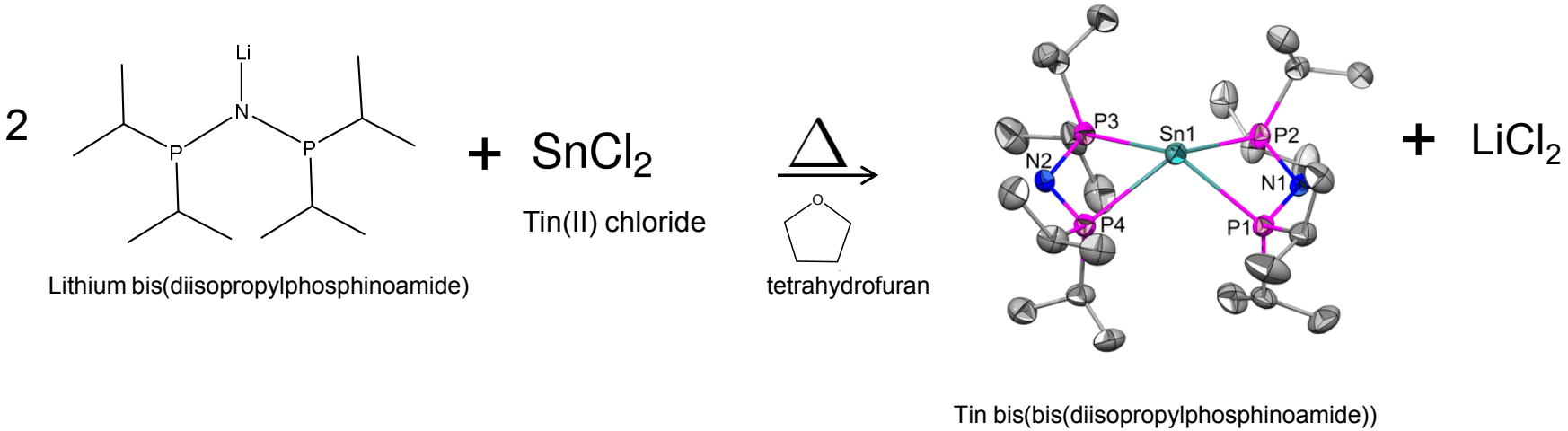
Youngjin Kim , Yongil Kim , Aram Choi , Sangwon Woo , Duckgyun Mok , Nam-Soon Choi ,Yoon Seok Jung , Ji Heon Ryu , Seung M. Oh , and Kyu Tae Lee, Adv. Mater. 2014, 26, 4139–4144

Synthesis Route:

First Step



Second Step



Dickie, D. A. et al Inorg. Chem., **2011**, *50* (22), pp 11288–11290

What kind of particles can we make from this precursor?

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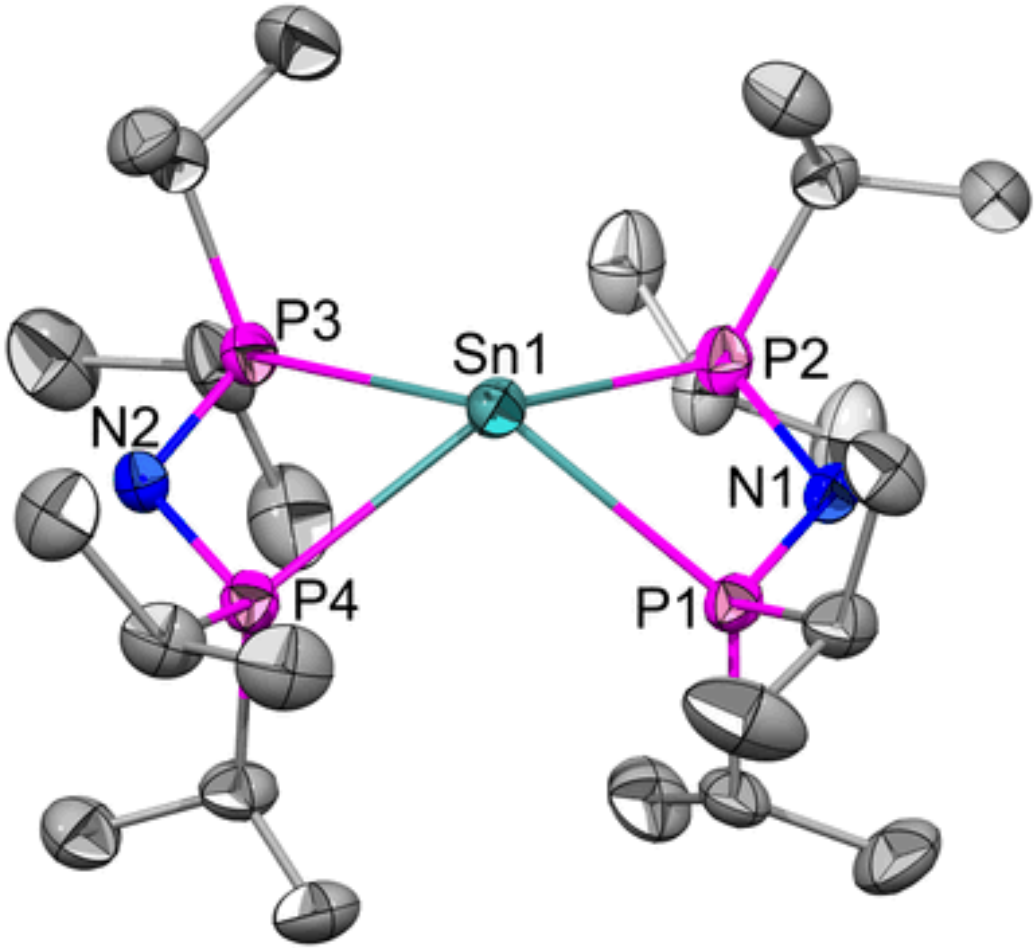
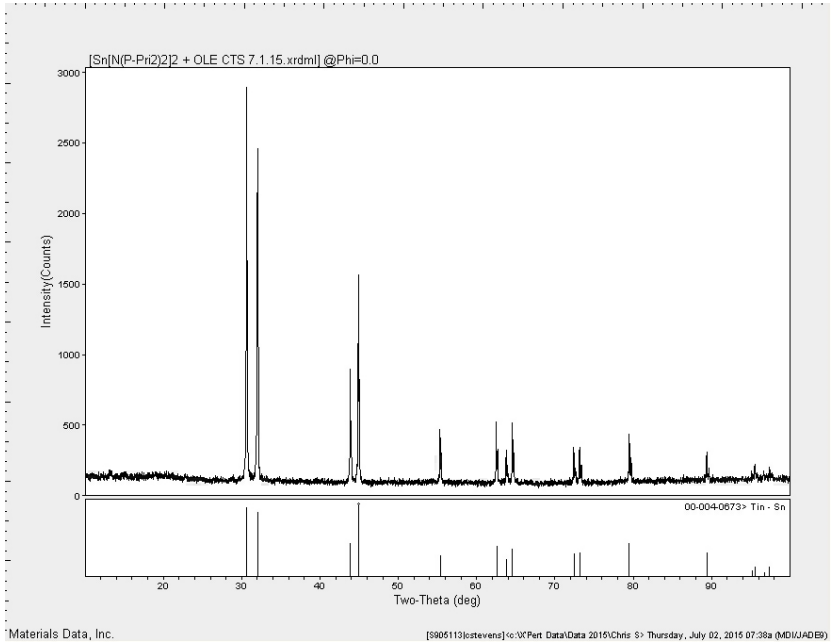


Tin bis[bis(diisopropylphosphino)]amide

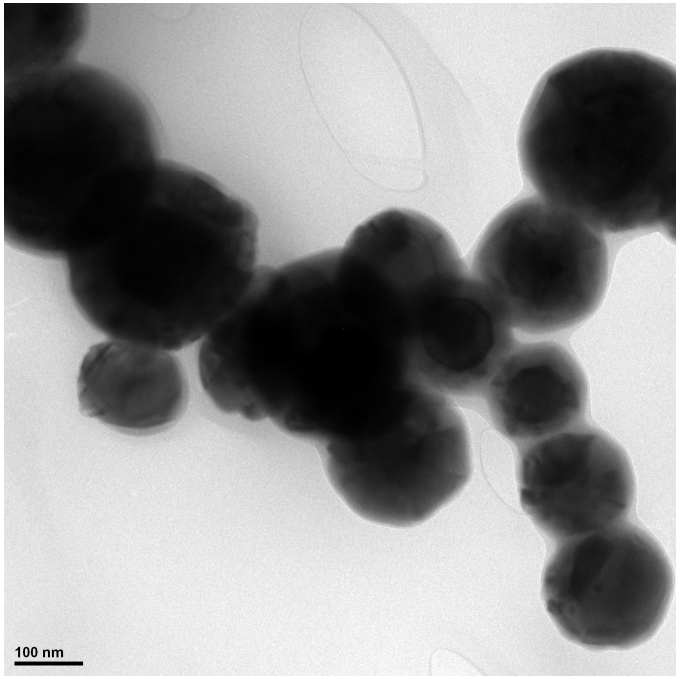
Dickie, D. A. et al Inorg. Chem., **2011**, *50* (22), pp 11288–11290

Precursor can be used to produce…

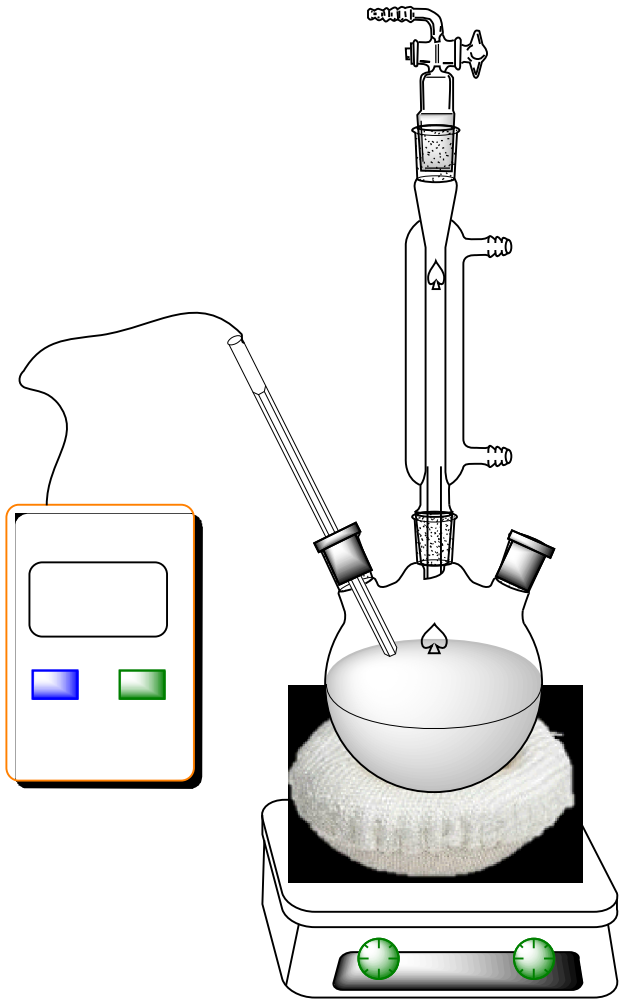
Tin Particles



Tin Phosphide Particles



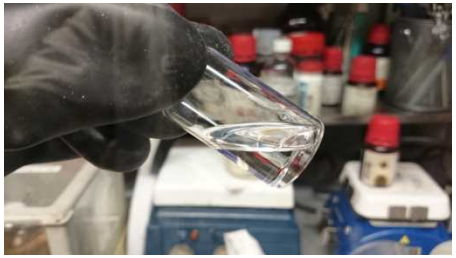
Solution Precipitation



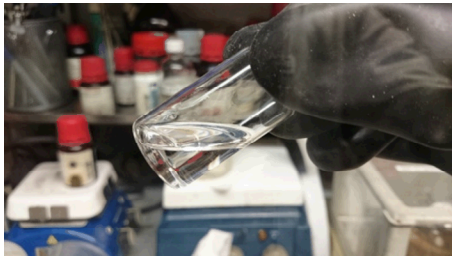
Explored

- Phosphorous Containing
- Phosphorus Free Solvents
- Temporal (Time & Temp)

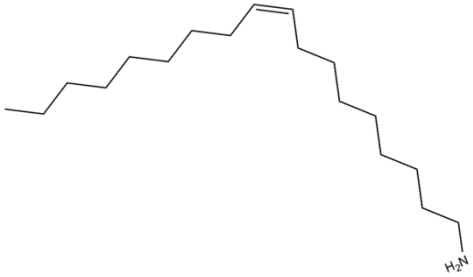
Phosphorus Free Solvents



Octadecene

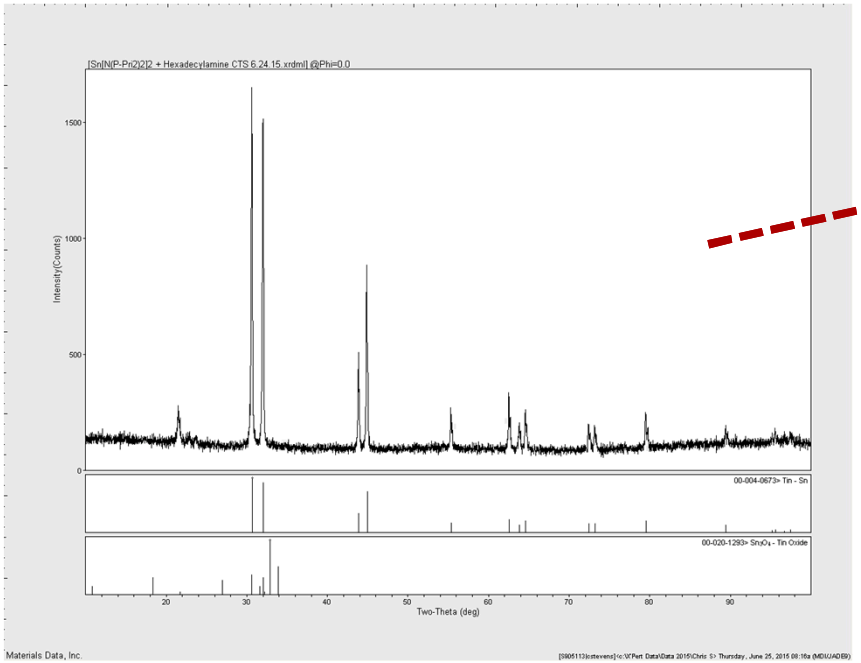


Oleylamine

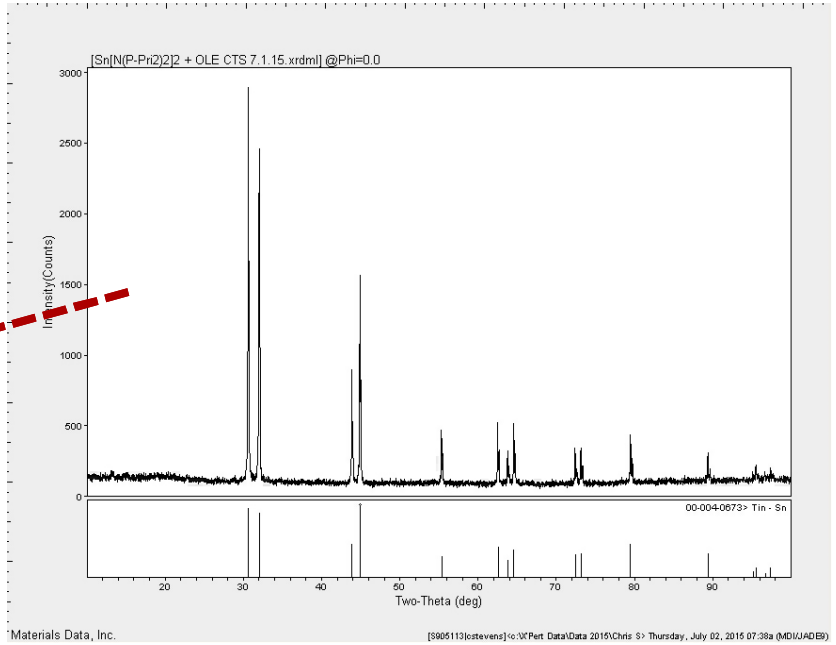


Hexadecylamine

Phosphorus Free Solvent Results



$Sn[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$ + Hexadecylamine
2 hours 315 ^{o}C



$Sn[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$ + Oleylamine
2 hours 5 minutes 330 ^{o}C

P Free: Octadecene Results

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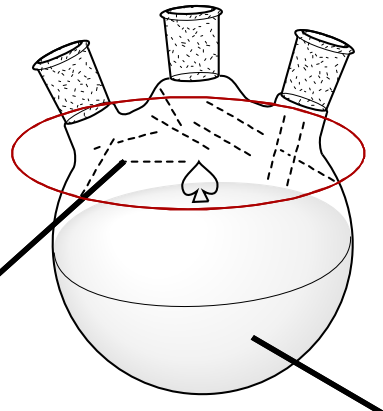
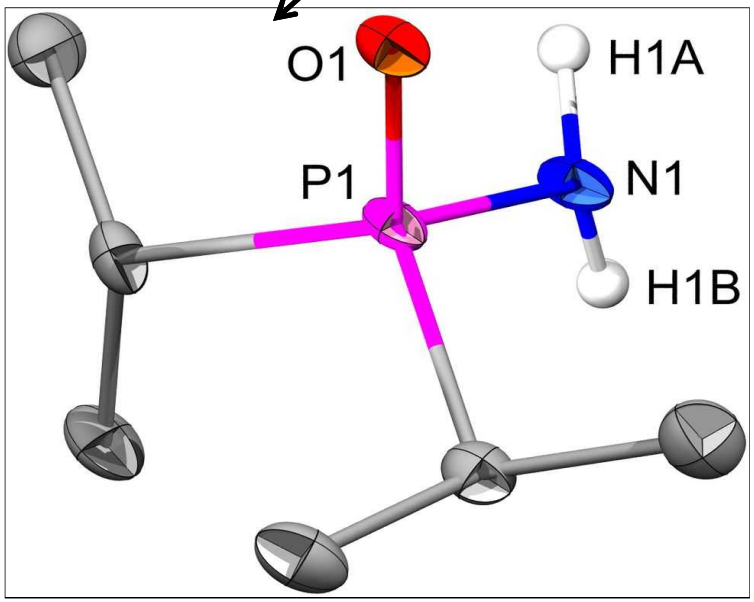
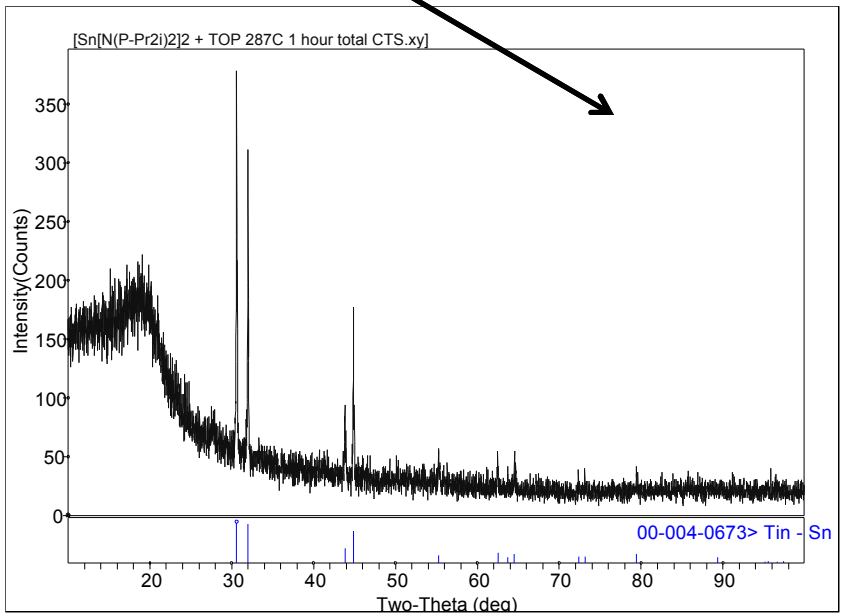
$Sn[N(P\text{-}Pr^{i}_{2})_2]_2$

Octadecene

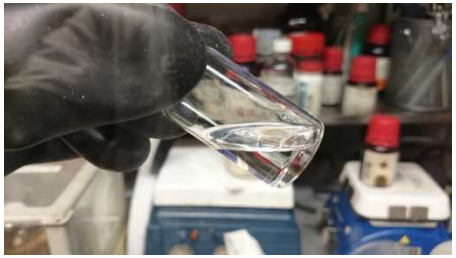
$\xrightarrow{\Delta}$



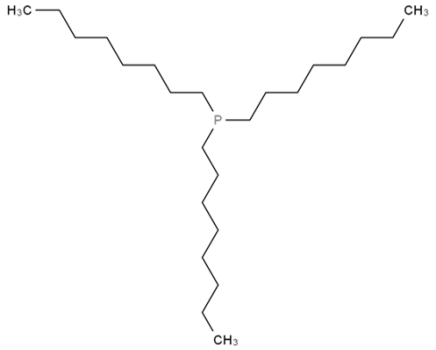




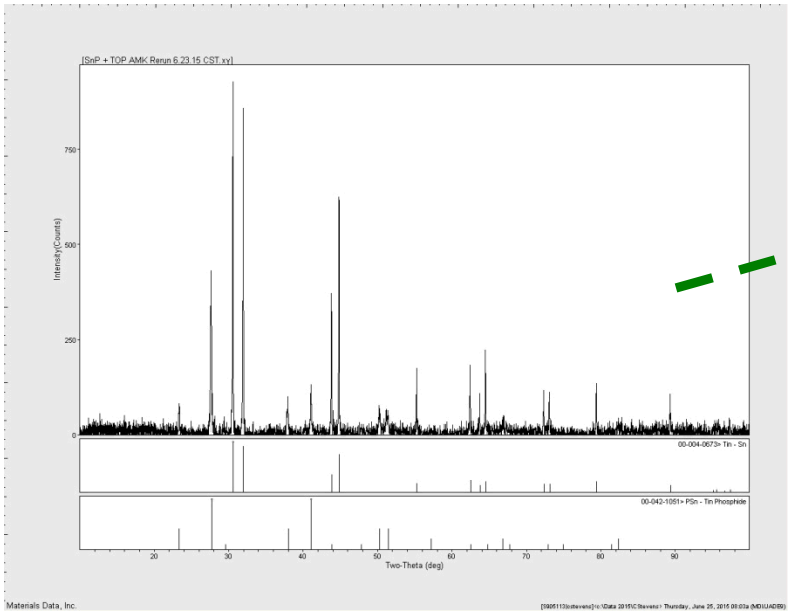
Phosphorous Rich Solvents



Trioctylphosphine

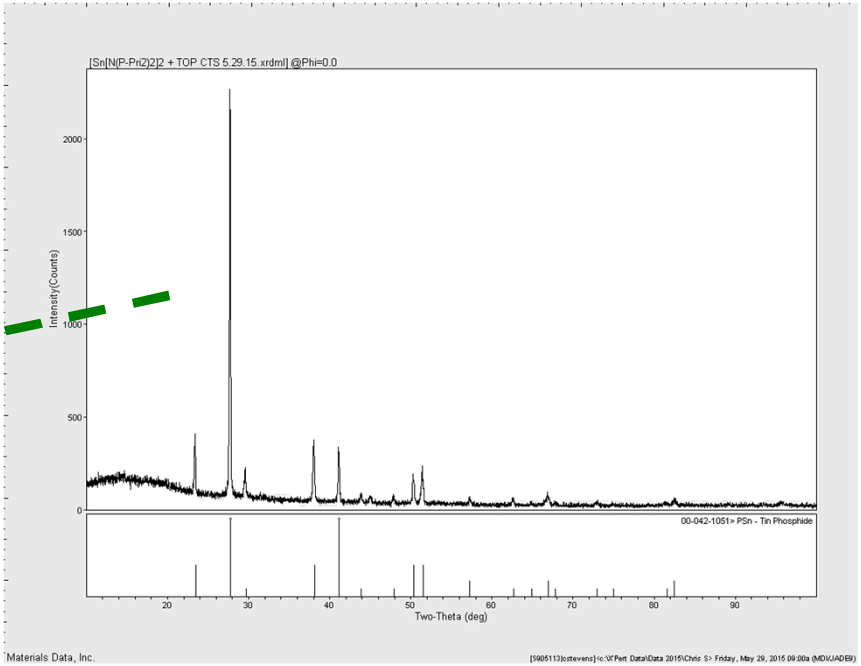


Results from Phosphorus Rich Solvents

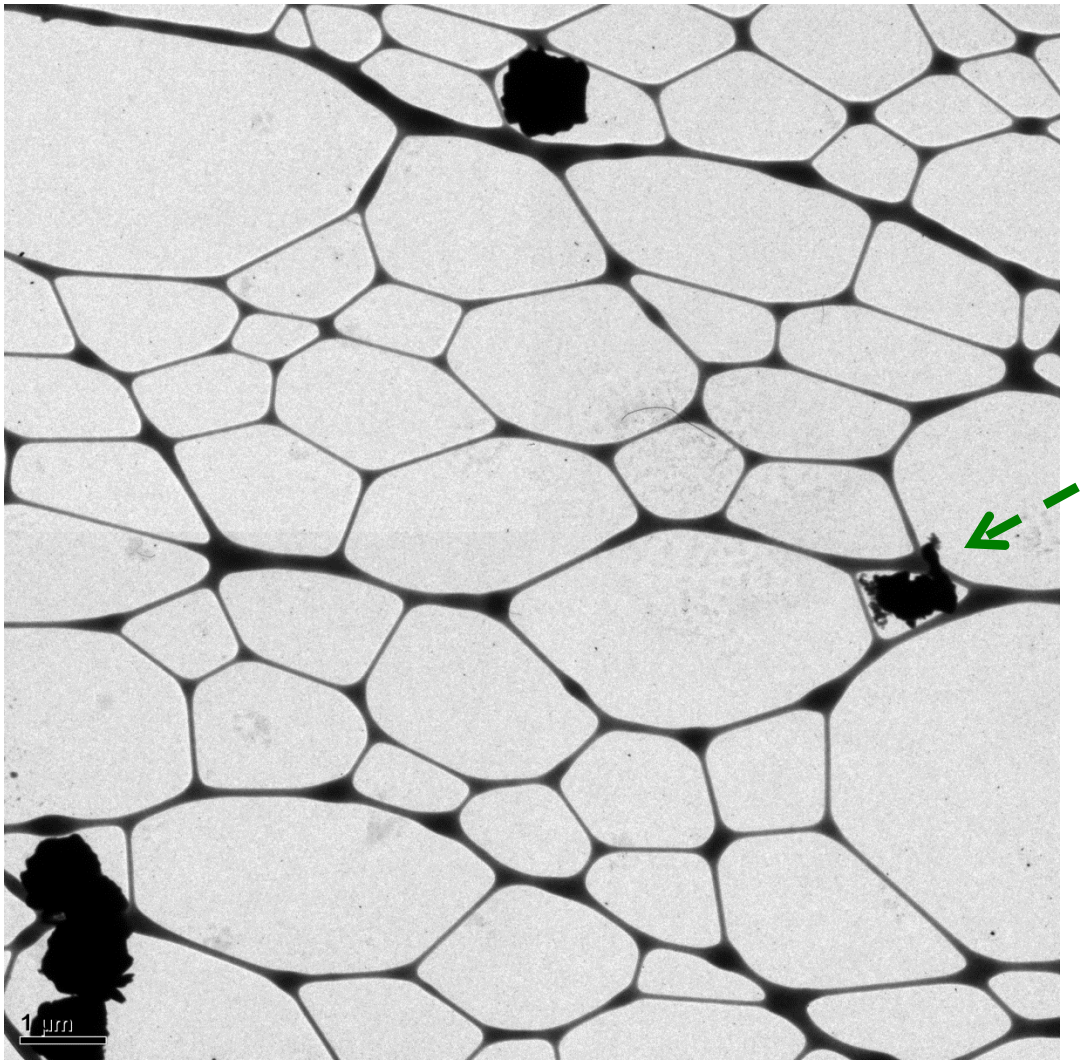


$Sn[N(P\text{-}Pr^{i}_{2})_2]_2$ + Trioctylphosphine
1 hour °(x) temp

$Sn[N(P\text{-}Pr^{i}_{2})_2]_2$ + Trioctylphosphine
6 hours 45 minutes 352°C

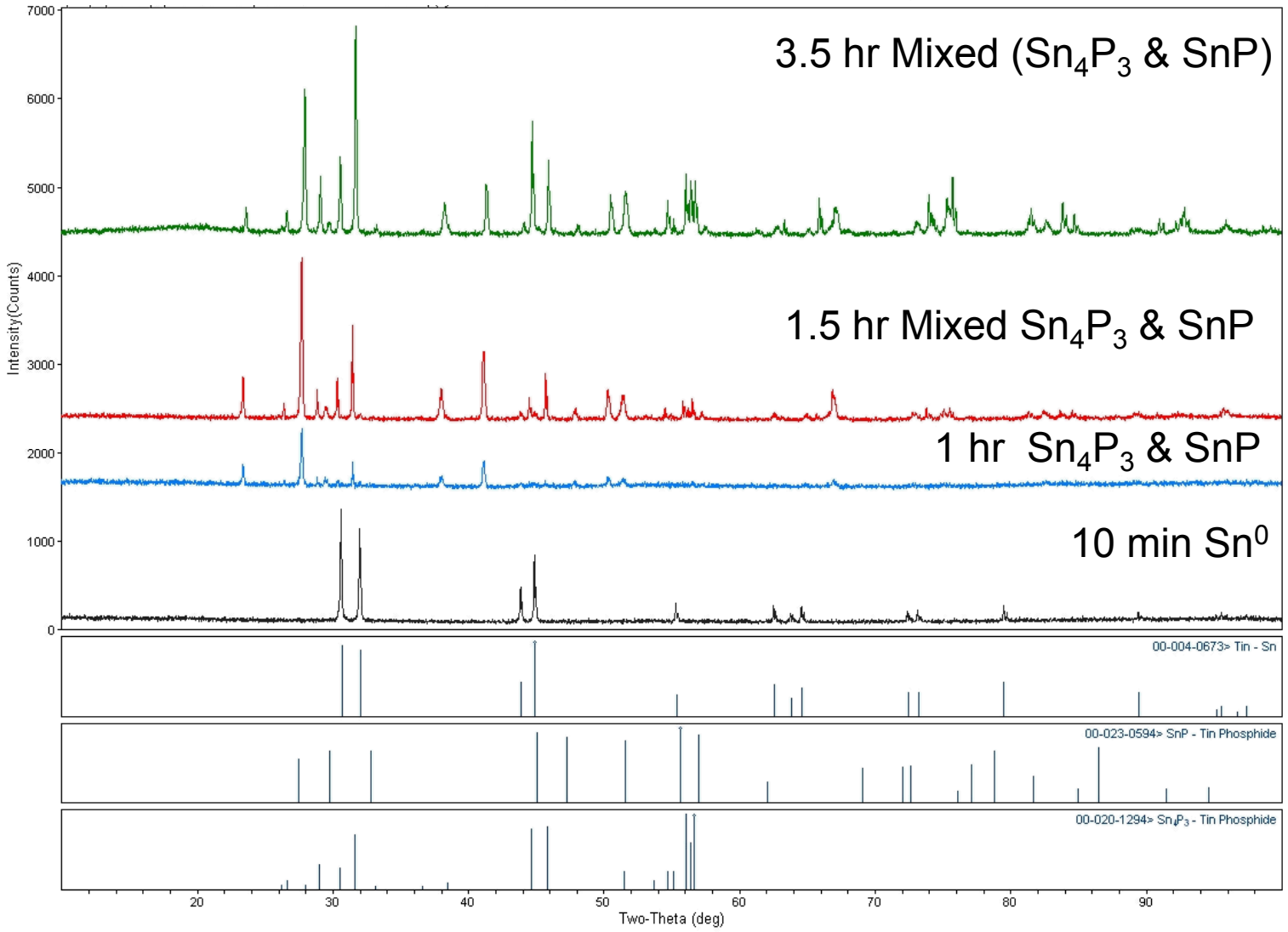


Transmission Electron Microscopy

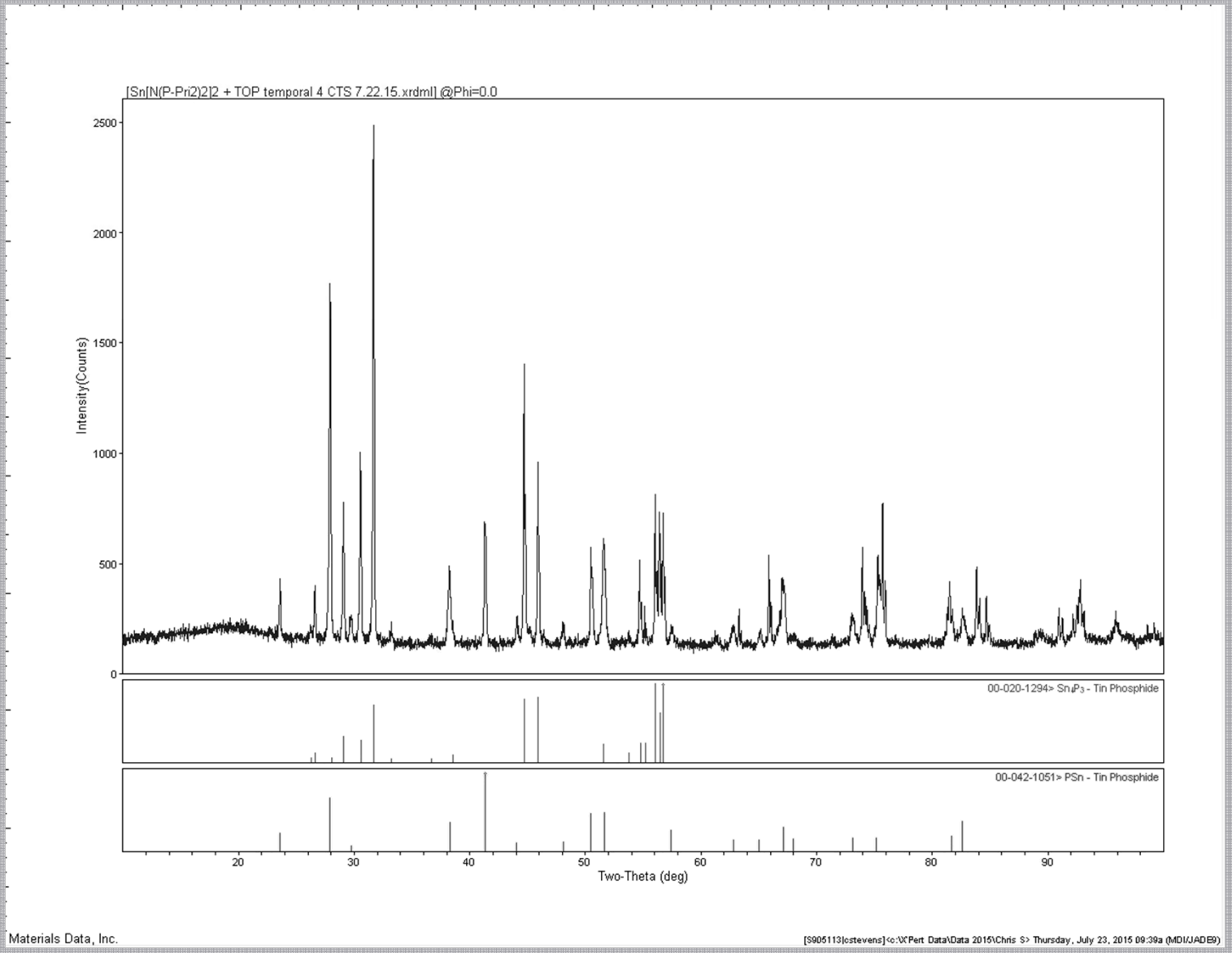


$Sn[N(P\text{-}Pr^{i}_{2})_2]_2$ +
Trioctylphosphine
6 hrs 45 minutes
352 °C

Temporal Study



Temporal Study 3.5 hrs



Recap

- Tin Phosphides are important for uses in Semiconductors
- Investigating the synthesis of a single source precursor to develop tin phosphide nano particles was unsuccessful
- Phosphorus rich solvents yield positively for Tin Phosphide Nano particles and can affect oxidation of tin

- http://easytimegallery.com/t/tin-periodic-table.html (tin Picture)
- https://www.google.com/search?q=phosphorus&biw=1600&bih=1063&source=lnms&tbm=isch&sa=X&ved=0CAYQ_AUoAWoVChMIhrnNhIrvxgIVwTWICh08uA7I#tbm=isch&q=matches&imgrc=oMjoNoAsi9dCBM%3A (phosphorous pic)

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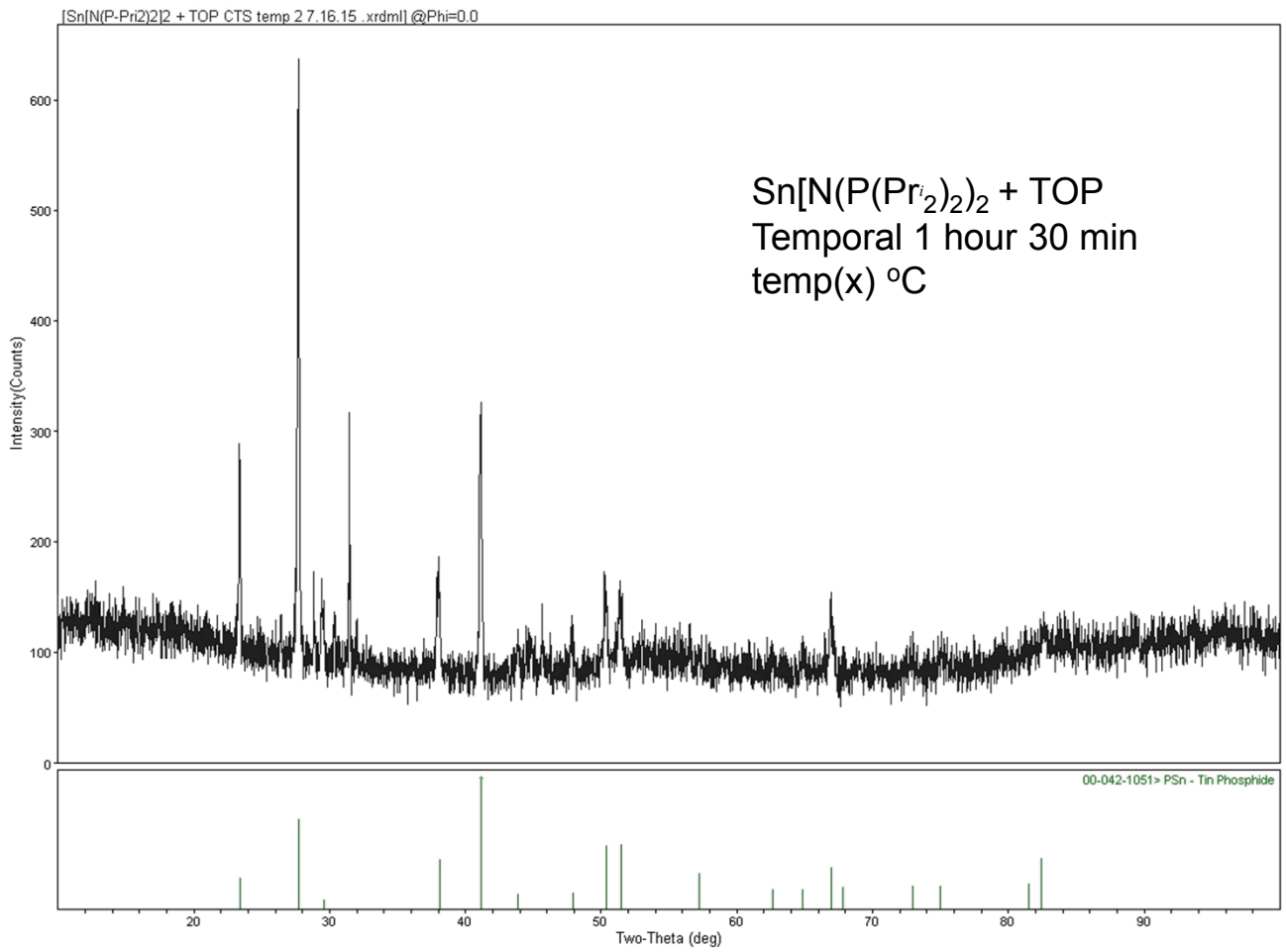
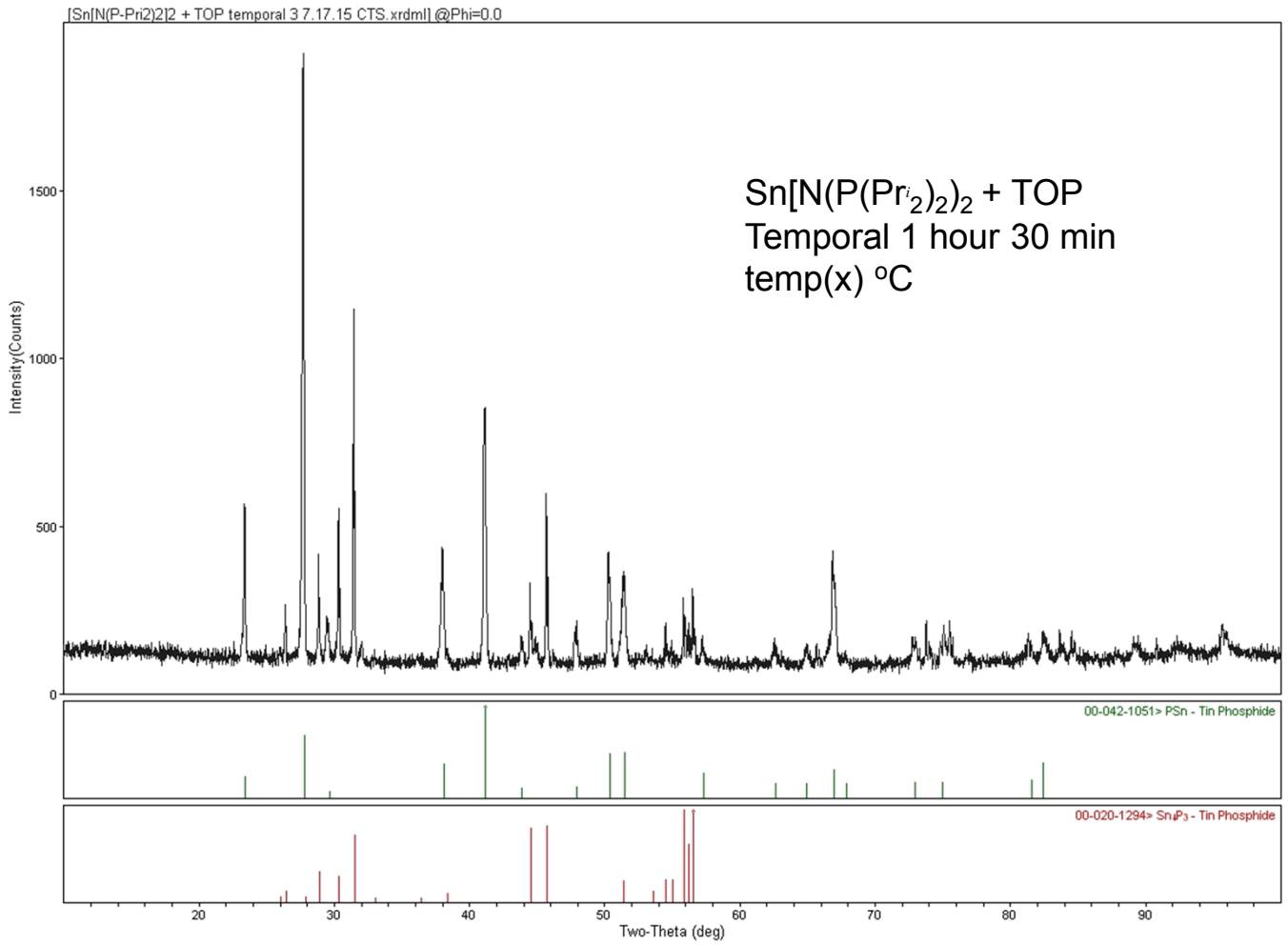
ADDITIONAL SLIDES

Temporal Study



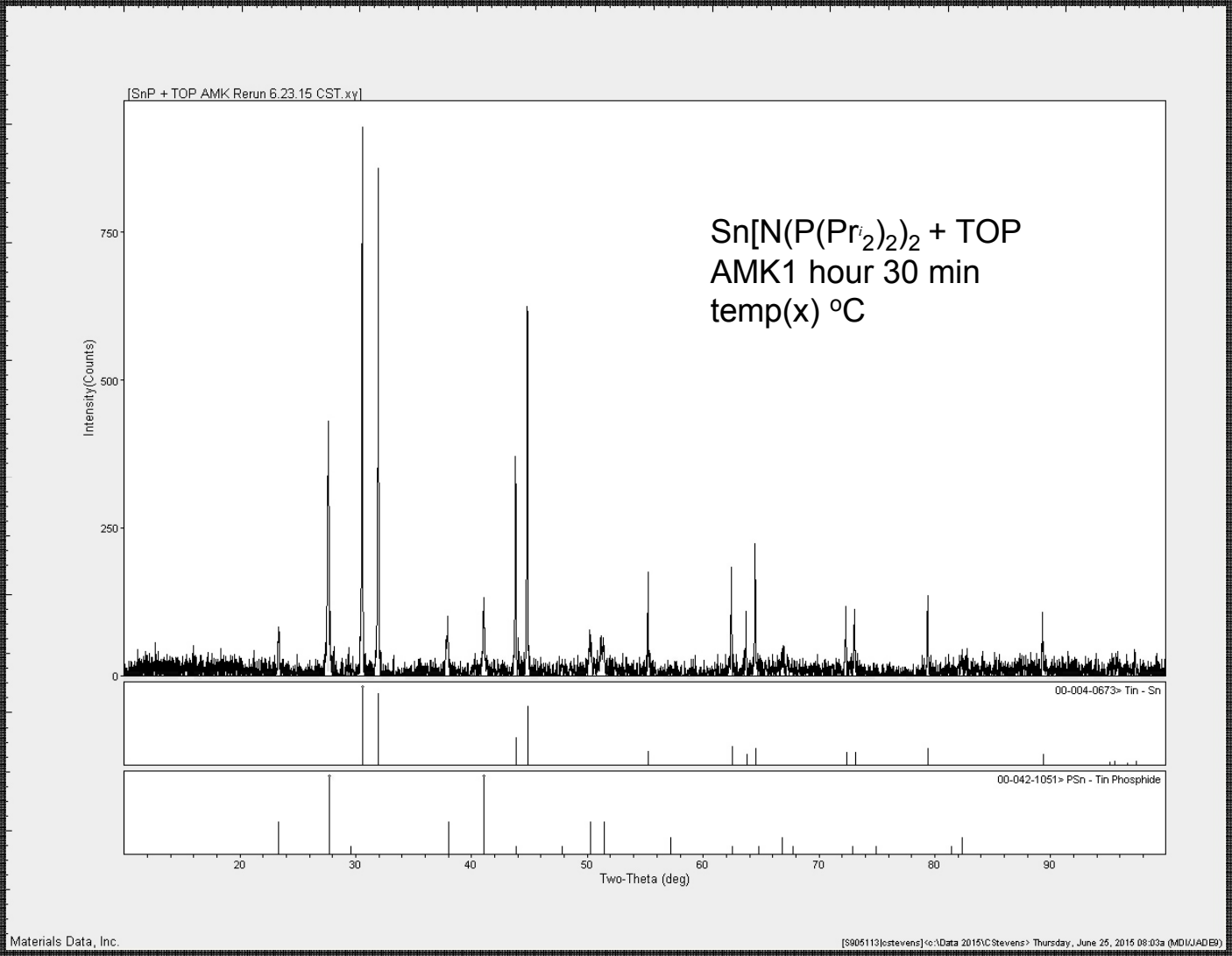
$Sn[N(P(Pr^{i}_{2})_{2})_{2}$ + TOP
Temporal 1 hour 30 min
temp(x) ^{o}C

Temporal Study

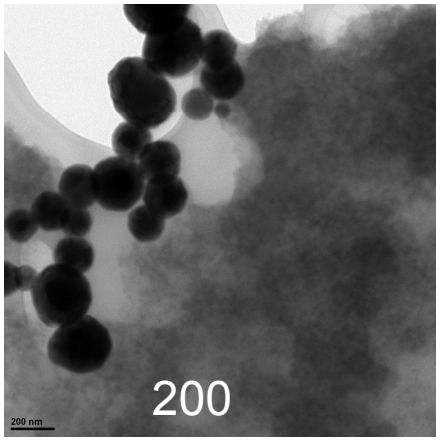


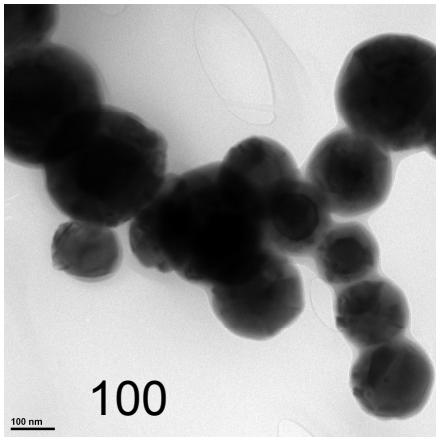
$Sn[N(P(Pr^{i}_{2})_{2})_{2}$ + TOP
Temporal 1 hour 30 min
temp(x) ^{o}C

Temporal Study



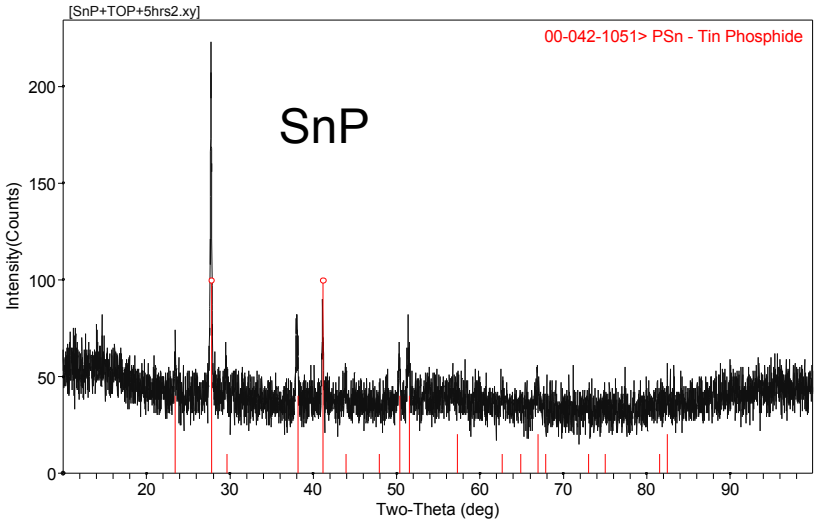
Previous Work



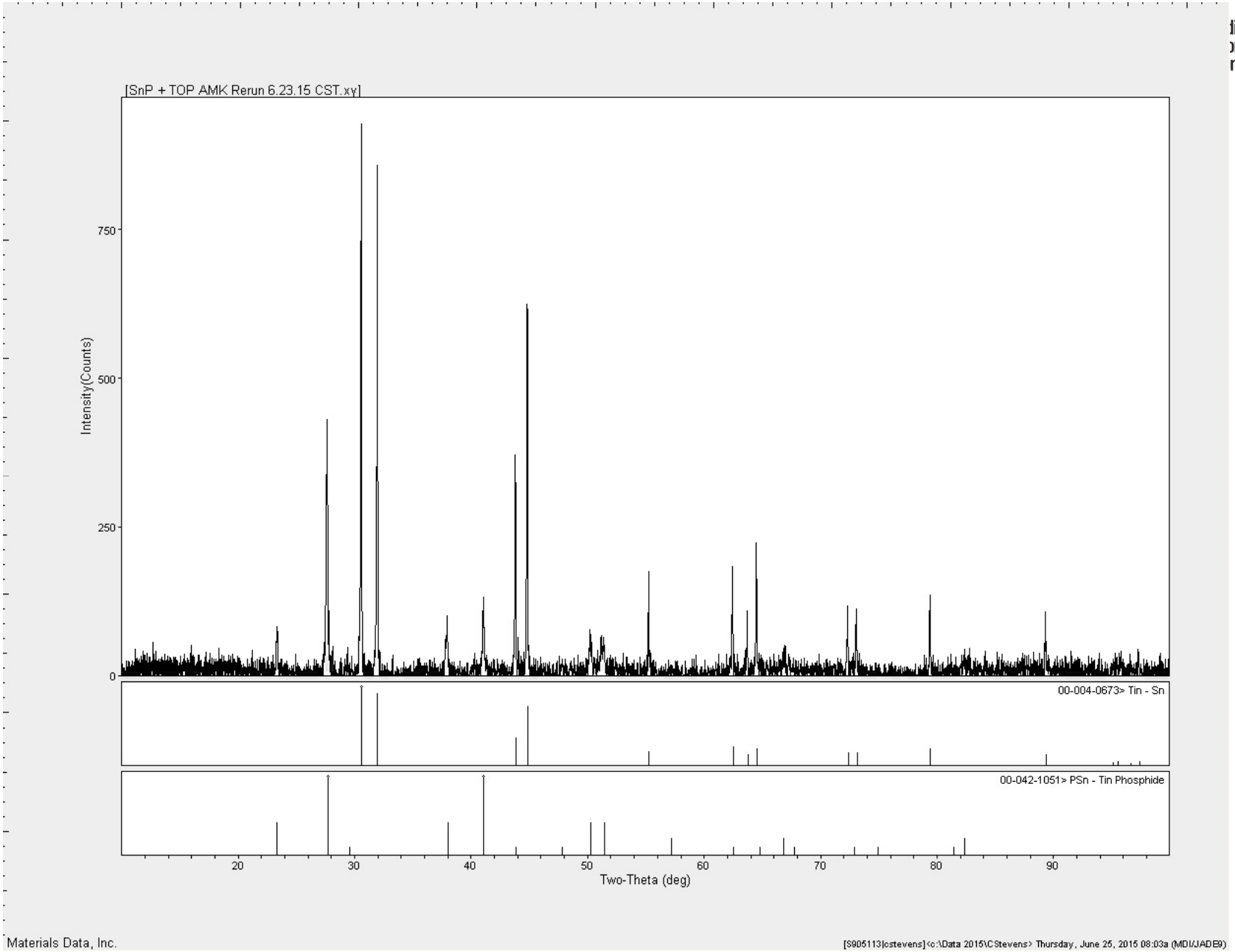


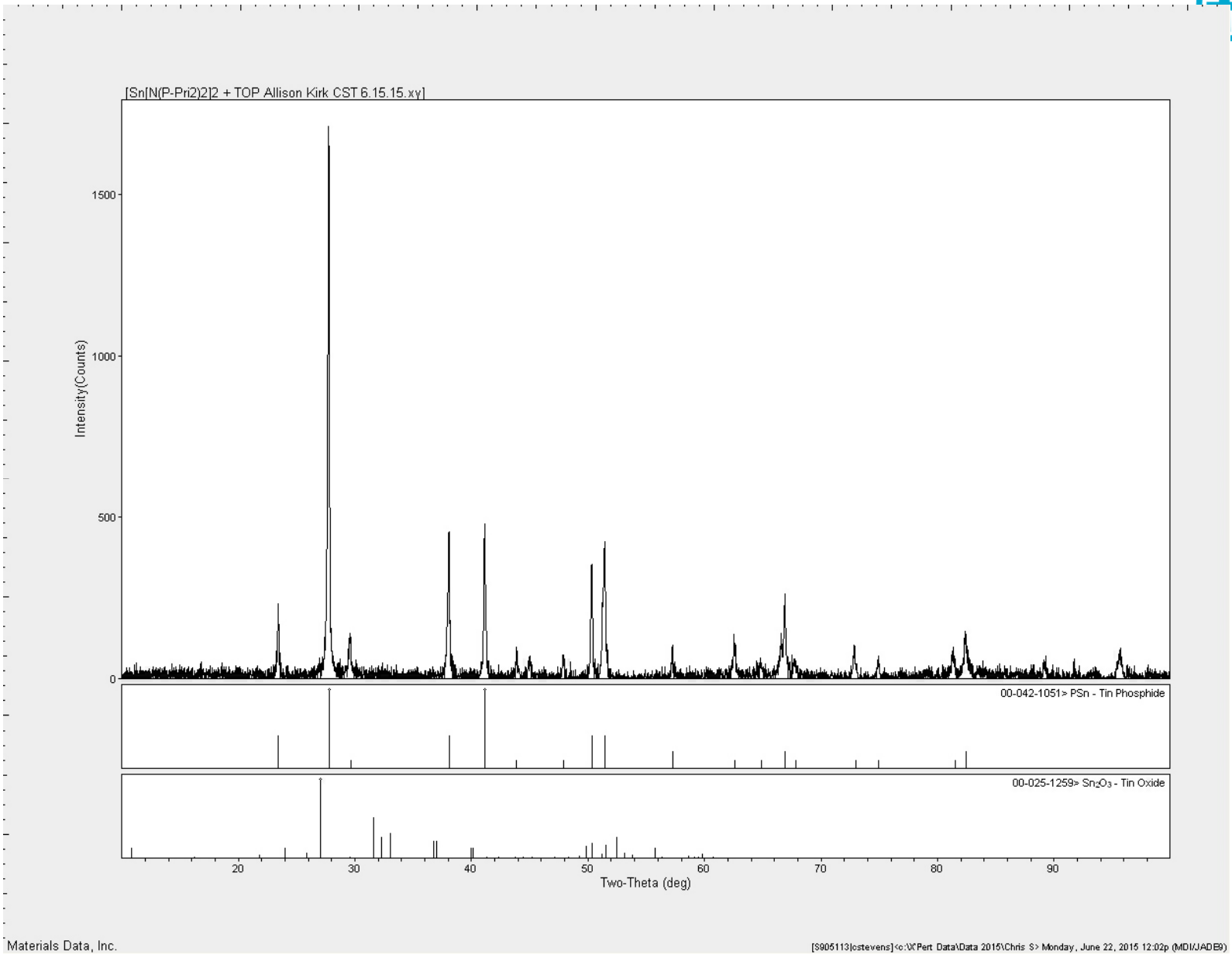
SnP TEM Images

A previous Intern had used a phosphorous containing solvent to synthesize SnP nanoparticles

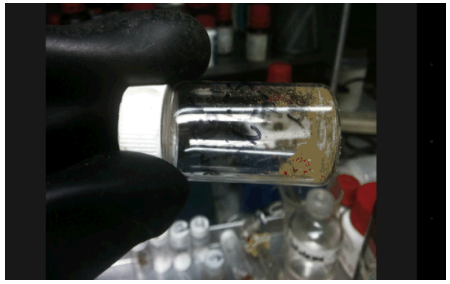


$Sn[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$ + TOP

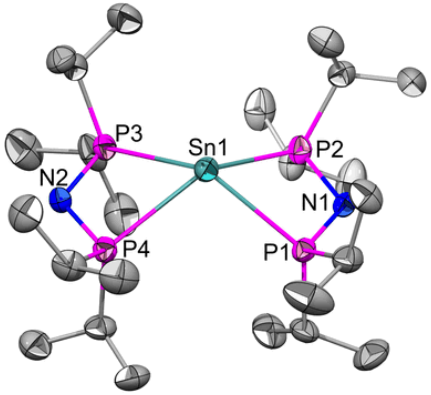

[SnP + TOP AMK Rerun 6.23.15 CST.xy]
Intensity(Counts)
750
500
250
0
00-004-0673> Tin - Sn
00-042-1051> PSn - Tin Phosphide
20
30
40
50
60
70
80
90
Two-Theta (deg)
Materials Data, Inc.
[S905113|cstevens]<c:\Data 2015\CStevens> Thursday, June 25, 2015 08:03a (MDI/JADE9)


[Sn[N(P-Pri2)2]2 + TOP Allison Kirk CST 6.15.15.xy]
Intensity(Counts)
1500
1000
500
0
00-042-1051> PSn - Tin Phosphide
00-025-1259> Sn2O3 - Tin Oxide
20
30
40
50
60
70
80
90
Two-Theta (deg)
Materials Data, Inc.
[S905113|cstevens]<c:\X'Pert Data\Data 2015\Chris S> Monday, June 22, 2015 12:02p (MDI/JADE9)

Precursors to (SnP) Reaction

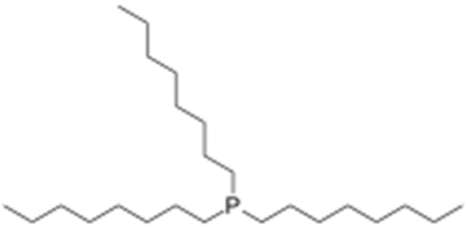


$Sn[N(P(Pr^{i}_{2})_{2})_{2}$



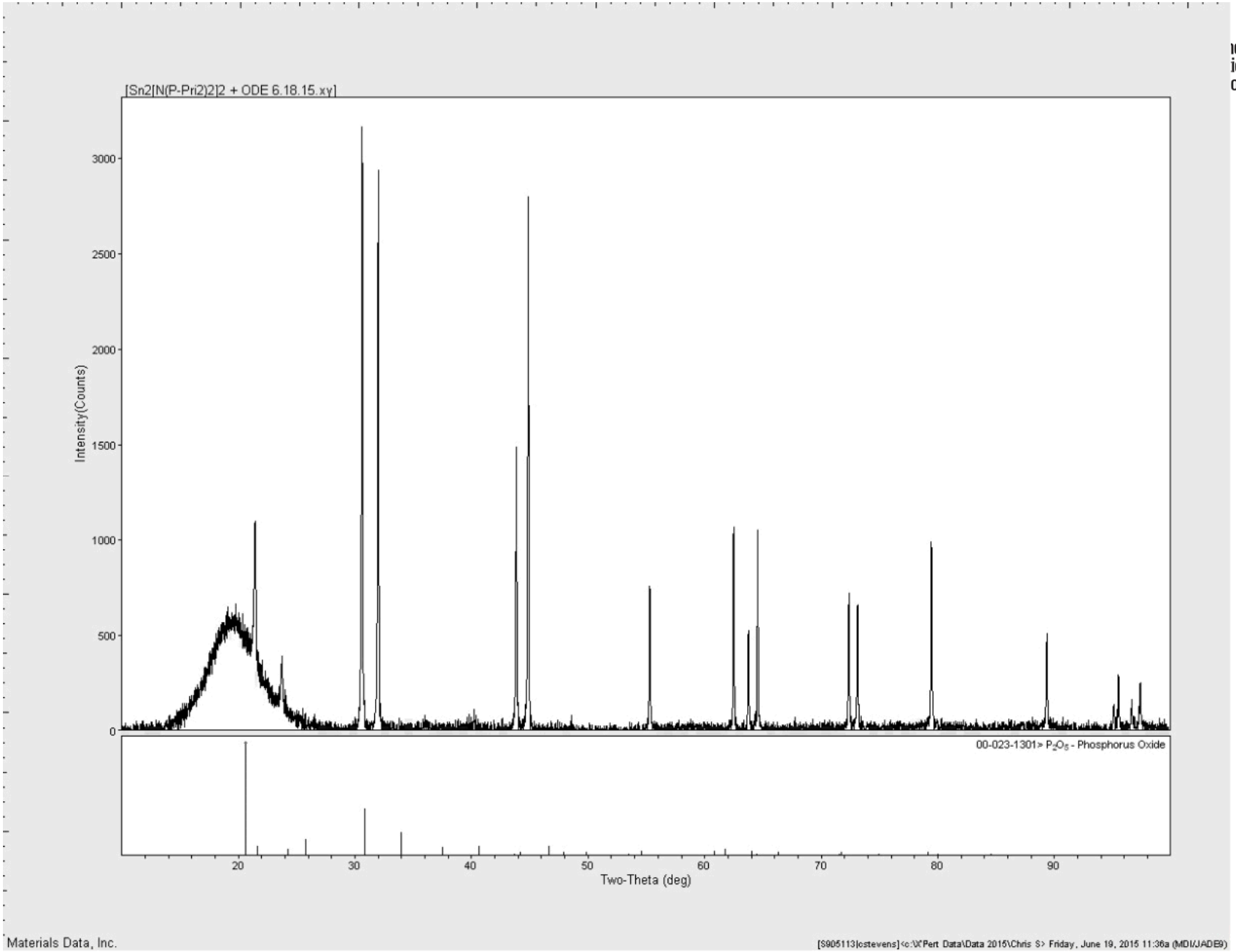
Octadecene

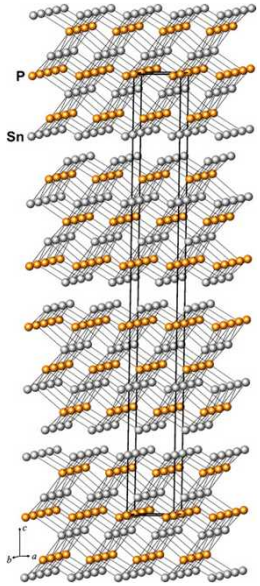
Trioctylphosphine



Dickie, D. A. et al Inorg. Chem., **2011**, *50* (22), pp 11288–11290

Precursor	Solvent	Method	Time & Temp	Identify	Tin Phosphide
$Sn[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$	Octadecene	Solution Precipitation	46 min 236.2 C°	XRD	
$Sn[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$	Octadecene	Solution Precipitation	51 min 305.0 C°	XRD	
$Sn[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$	Trioctylphosphine	Solution Precipitation	4 hours 24 min 303.2 C°	XRD	
$Sn[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$	Trioctylphoshine	Solution Precipitation	6 hours 45 min N/A	XRD TEM	
$Sn[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$	Octadecene	Solution Precipitation	2 hours 5 min 315 C	XRD	
$Sn_{2}[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$	Octadecene	Solution Precipitation	4 hours 5 min	XRD	
$Sn_{2}[N(P\text{-}Pr^{i}_{2})_{2}]_{2}$	Trioctylphophine	Solution Precipitation			


[Sn2[N(P-Pri2)2]2 + ODE 6.18.15.xy]
Intensity(Counts)
3000
2500
2000
1500
1000
500
0
00-023-1301> P2O5 - Phosphorus Oxide
20
30
40
50
60
70
80
90
Two-Theta (deg)
Materials Data, Inc.
[S905113|cstevens]<c:\X'Pert Data\Data 2015\Chris S> Friday, June 19, 2015 11:36a (MDI/JADE9)



Sn4P3

http://www.sciencedirect.com/science/article/pii/S0022459606004646