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The results of one year's effort to produce single source precursors that will generate high yields of semiconductor materials consisting of elements from groups 14 and 16 are summarized in this report. Laboratory studies demonstrate that compounds in which bonds between group 14 and 16 elements exist and in which remaining valences are occupied with phenyl groups are excellent sources of phase pure binary compounds such as tin sulfide, tin selenide and tin telluride. Mechanistic studies reveal that phenyl migration is the dominant reaction pathway allowing formation of the target compounds at temperatures as low as 300 °C.

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Principal Investigator: Philip Boudjouk F49620-92-J-0431

ANNUAL TECHNICAL REPORT for August 15, 1992 -August 14, 1993

I. List of Objectives

The objectives in this project arose from an accidental discovery during the work we began under another Air Force project (AFOSR Grant No 91-0197) in which we observed that heating of certain organotin sulfides and selenides produced high yields of tin sulfide and tin selenide in high purity, i.e., with carbon contamination of less than 1%. Under this grant we have the following objectives:

1-Determine the breadth and scope of the applicability of the relatively nontoxic **perphenylated** group 14-16 chalcogenides as single source precursors to phase pure binary and ternary materials such as SnS, SnSe and SnS_xSe_{1-x}, as well as the germanium, lead and tellurium analogues.

2-Investigate the possibility that group 13-15 analogues such as GaAs could be prepared by the low temperature pyrolysis of perphenylated precursors.

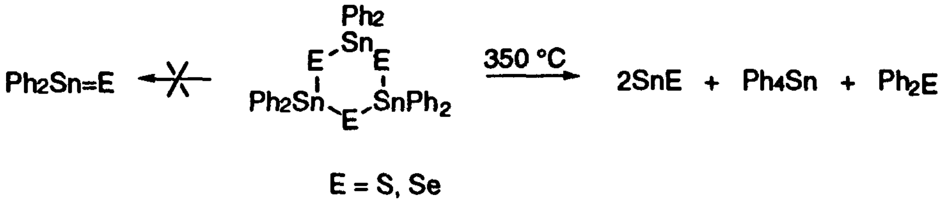
3-Attempt to determine the mechanism of the production of these materials.

II. Status of Research Effort

Good progress was made in the first year of this grant period. Below is a summary of the work accomplished using structural features as the means of organization of the report.

Cyclic Group 14-16 Chalcogenides

Cyclic compounds were the first investigated and were our choice by accident. Our original intentions were to generate reactive intermediates containing multiple bonds between tin and sulfur and tin and selenium. Instead, a novel rearrangement proved to be the preferred pathway and a new route to tin sulfide and tin selenide was found.



This methodology has several key advantages:

1) facile synthesis of the cyclic precursors;

2) low toxicity of those precursors;

3) ease of handling of the precursors because they are air and water stable solids;

4) only a simple tube furnace is required, valving systems and high temperature safegaurds are not needed;

5) the yields are very high, within a few percent of theoretical;
6) the pyrolysis product contains very little carbon (always less than 5%, often less than 1%);
7) there is no danger of explosion, a known hazard in current methodologies since the system operates at atmospheric pressure and much lower temperature than those approaches based on simple combination of the elements; and
8) the procedure is very easy to scale up.

The essential feature of this new reaction is the high mobility of the phenyl group. A reasonable mechanism was proposed in our first publication on the subject (Bahr, S. R.; Boudjouk, P.; McCarthy, G. J.; *Chem. Mater.* **1992**,*4*, 383).

One drawback we encountered was that we could not easily synthesize a wide variety of cyclic compounds that included elements such as lead from group 14; boron, aluminum, gallium and indium from group 13 and tellurium from group 16. We then explored linear systems.

Linear Group 14-16 Chalcogenides

The ease with which the phenyl groups migrated in the cyclic systems led us to believe that the cyclic structure may not be essential to the production of the binary systems. Thus we proceeded to prepare a series of linear group 14 - 16 compounds of the general formula, $(Ph_3M)_2X$, M = Ge, Sn, Pb; X = S, Se, Te, that might serve as useful single source precursors of metal chalcogenides. We had immediate success. The syntheses were much easier than the cyclic systems above and the pyrolyses gave the desired compounds in excellent yields.

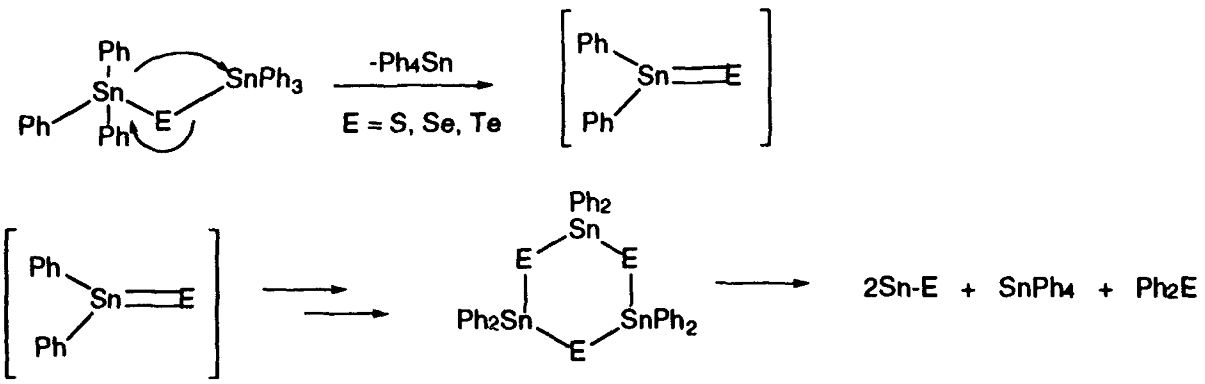
Synthesis of Linear Metallic Chalcogenides

We were able to prepare these compounds in large quantities using well established techniques. An added benefit to this approach is that now tellurium precursors could be easily made and investigated. We were unsuccessful in generating tellurium cyclic analogues to those compounds mentioned above. Indeed, using linear models, we were now able to access easily a much broader variety of compounds from groups 13, 14, 15, and 16. In this grant period we successfully prepared tin sulfides, selenides and tellurides in high yields and high purity under mild conditions from these linear precursors.

$$\mathrm{Ph_3M\text{-}Cl} + \mathrm{Na_2E} \longrightarrow \mathrm{Ph_3M\text{-}E\text{-}MPh_3} + \mathrm{NaCl}$$
$$M = \mathrm{Sn, Pb};\ E = \mathrm{S, Se, Te}$$

Mechanism of Pyrolysis of Linear Metallic Chalcogenides

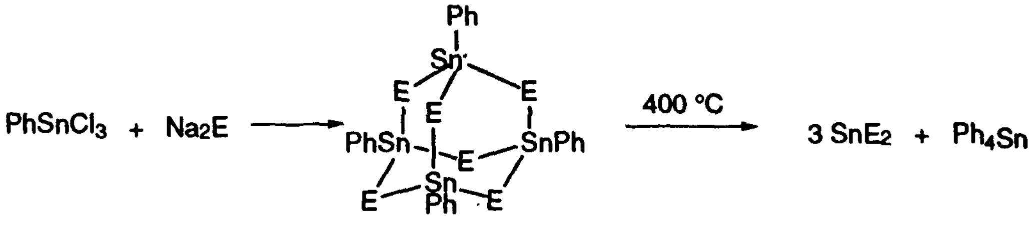
Pyrolyses of the linear compound proceeded efficiently and we have initiated efforts to determine the mechanism of the production of the binary compounds. By careful analysis of incompletely pryrolyzed substrate we detected the presence of ring systems as intermediates. Thus, we have proposed that a reasonable route to the product is through the unusual reactive intermediate, $Ph_2Sn{=}E$, the target of our earlier proposed research under grant 91-0197.



We have since completed a thorough analysis of this reaction that will be presented in the technical report for the period 8/15/93 - 8/14/94

Adamantyl Group 14-16 Chalcogenides

It is logical to extend this work to cage compounds for two reasons: 1) to determine if phenyl migration would still be an important mechanism in a rigid cage structure and, 2) to determine if the ratio of atoms in the cage structure would influence the nature of the products. Affecting the nature of the product could be manifested in two distinct ways: altering the ratio of atoms in the product binary and, altering the microstructure of the product binary. Initial studies were conducted in this grant period and we found that, indeed, the adamantyl cage structure produces a different ratio of atoms in the product than the linear and cyclic systems. We were able to produce tin disulfide and tin diselenide in good yields and high purity from the adamantyl system below.



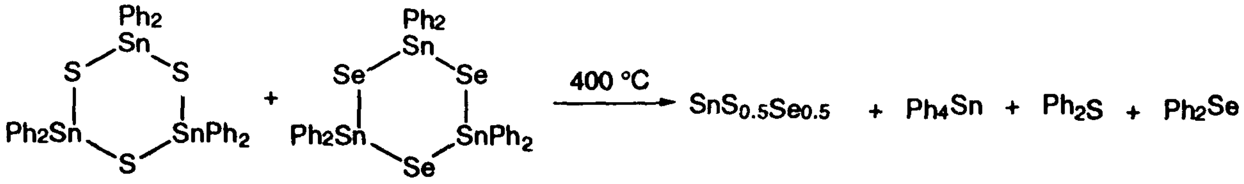
This example, when combined with the linear and cyclic compounds we investigated and summarized above, opens the way to an entirely new approach to the synthesis of novel materials comprised of main group atoms. We have now been able to demonstrate that we can easily control the stoichiometry of some group 14 - 16 compounds simply by apropriate design of the precursor.

Nonstoichiometric Ternaries of the Type SnS_xSe_{1-x} From Molecular Compounds

Doping of materials is a standard method of altering their physical properties, in particular, their electronic proporties. Normally, this is accomplished by mixing a stoichoimetric binary compound with the desired quantity of a third agent followed by repeated sequences of heating and grinding to ensure good mixing.

We have developed a method that circumvents this time consuming and tedious process for ternary compounds of groups 14 - 16. We found that simply by heating a mixture of different perphenylated precursors we can form ternary systems. *Moreover, we can control the ratio of elements.*

For example, we have demonstrated that a heating a 1:1 mixture of the two cyclics, $(Ph_2SnS)_3$ and $(Ph_2SnSe)_3$ produces high yields of $SnS_{0.5}Se_{0.5}$. By adjusting the ratios of the two cyclics



we have been able to control the amounts of sulfur and selenium in the final product. Details of that part of our study will be in the report covering the period 8/15/93 - 8/14/94.

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V. Interactions (research funded by F49620-929-J-0431)

A. Presentations at meetings, conferences and seminars

1. Papers

None

2. Posters

None

3. Seminars

None

4. Invited Lectures

None

5. Plenary Lectures

None

B. Consultative and Advisory Functions

None

C. New Discoveries, Inventions or Patent disclosures

None

D. Additional Information

None