to provide a diminished susceptibility to electrophilic attack may partly account for the observed stabilization.[30]

Acknowledgment. We thank the National Research Council of Canada and the University of Victoria for financial support.

Registry No. H_3GeSCF_3, 68457-15-8; $H_2Ge(SCF_3)_2$, 68457-16-9; $H_3GeSeCF_3$, 68457-17-0; $H_2Ge(SeCF_3)_2$, 68457-18-1; $H_2Ge(SeCF_3)Br$, 68457-19-2; $Ge_2H_5SCF_3$, 68457-20-5; $Ge_2H_5SeCF_3$, 68457-21-6; $Hg(SeCF_3)_2$, 60129-77-3; $Hg(SCF_3)_2$, 21259-75-6; GeH_3I, 13573-02-9; GeH_3Br, 13569-43-2; $AgSCF_3$, 811-68-7; GeH_2Br_2, 13769-36-3; GeH_2I_2, 14694-31-6; Ge_2H_5I, 19021-93-3.

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Reactions of Primary Alkylantimony Compounds. Formation of the Macromolecular Species $(RSb)_x$ and $(RSbI_{0.4})_x$

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Received April 28, 1978

Reactions of the primary alkylstibines CH_3SbH_2, $C_2H_5SbH_2$, and n-$C_4H_9SbH_2$ with organosilicon chlorides or HCl produce a pale green macromolecular species with a metallic sheen analyzing as $(RSb)_x$ while the same stibines with iodine-containing reagents, I_2, ICl, CI_4, and $(CH_3)_2AsI$, produce a purple-black species, $(RSbI_{0.4})_x$. With CH_3AsCl_2, the stibines yield only the all-arsenic macromolecule $(CH_3As)_x$ and $RSbCl_2$. No evidence for the formation of cyclooligomers of antimony is found.

Introduction

Low-dimensional organometallic macromolecules warrant study as potential sources of new materials with unusual electrical properties.[1] Earlier studies of a homoatomic ladder-structure catenate of arsenic, $(CH_3As)_x$,[2,3] exposed a rich variety of thermal and photoconductivity properties[4,5] that encouraged us to extend this study to antimony.

Few reports exist of compounds which analyze satisfactorily as multiples of the unit RSb; those that do are restricted to cyclooligomers with sterically demanding phenyl[6] or *tert*-butyl[7] R groups. Previous reports of high-molecular-weight organoantimony catenates invariably describe products containing excess antimony.[8,9] In two earlier papers we briefly reported procedures which provide the $(CH_3Sb)_x$ polymer, specifically the reactions of CH_3SbH_2 with dibenzylmercury[10] and with sulfur monochloride.[11] The present study concerns the reactions of primary alkylstibines with elemental halogen, halocarbons, and organometallic halides.

Since our primary interest was a study of the ability of the RSb unit to form macromolecular compositions, it was important that the primary alkylarsenic reagents be as free as possible from potentially chain-terminating dialkyl derivatives and chain-branching antimony trihalide. The results of our study of both existing and novel procedures to obtain high-purity organoantimony monomers is described in a recent review.[12]

Results

Reactions of Alkylstibines. Primary alkylstibines react rapidly with iodine, iodine monochloride, or carbon tetraiodide in benzene to form a microcrystalline purple-black solid and alkyldiiodostibine. A ^1H NMR spectrum of the reaction mixture within 1 min of combination shows a complete absence of alkylstibine. The ^1H NMR spectrum is consistent with $RSbI_2$ formation: R = Me, singlet at δ 1.84; R = Et, δ 1.95 (quartet) 1.65 (triplet) ($^3J_{HH'} = 7$ Hz). After opening of the reaction tube and removal of the purple-black solid by filtration, evaporation of solvent affords a bright yellow crystalline solid identified as $RSbI_2$. The purple-black solid showed consistent C, H, and I percentages near the empirical composition $RSbI_{0.4}$ (or $R_5Sb_5I_2$). We are seeking crystals large

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enough for X-ray structural analysis and electrical property characterization. No iodine could be displaced from $CH_3SbI_{0.4}$ by heating at 50 °C in vacuo (0.1 mmHg) for 2 days; in contrast, attempts to form this material by exposing crystals of halogen-free $(RSb)_x$ to iodine vapor ($\sim$1 mmHg at 40 °C) result in rapid and complete destruction of the original material.[13]

As monitored by ^1H NMR spectroscopy, alkylstibines combine with CCl_4 to form $CHCl_3$ and $RSbCl_2$, but in contrast to alkylarsines,[3] no Sb–Sb condensation products are found even when CH_3SbH_2 is in excess of the ratio shown in eq 1.

$$RSbH_2 + 2CCl_4 \rightarrow RSbCl_2 + 2CHCl_3 \qquad (1)$$

Ethylstibine and CH_3AsCl_2 undergo rapid hydrogen–chlorine exchange followed by slow HCl elimination to form the all-arsenic ladder homopolymer $(CH_3As)_x$. No other solid products were formed, and no antimony or ethyl group incorporation in $(CH_3As)_x$ could be detected by mass spectrometry.

$$C_2H_5SbH_2 + 2CH_3AsCl_2 \rightarrow C_2H_5SbCl_2 + 2CH_3AsHCl$$
$$CH_3AsHCl \rightarrow (CH_3As)_x + HCl \qquad (2)$$

There was no ^1H NMR evidence for the formation of C_2H_5SbHCl. With $(CH_3)_2AsI$, CH_3SbH_2 produces $(CH_3)_2AsH$, $(CH_3)_2AsAs(CH_3)_2$, and solid $CH_3SbI_{0.4}$; no CH_3SbI_2 or CH_3SbHI was found.

The reactions of $RSbH_2$ with three organosilicon chlorides, $(CH_3)_2SiCl_2$, $(CH_3)_3SiCl$, and $(C_6H_5)_3SiCl$, provided $(RSb)_x$ without apparent halogen incorporation. In each reaction, a crystalline solid with a pale green metallic luster and the formula $(RSb)_x$ was isolated. An X-ray diffraction powder pattern shows no bands in common with the major bands of elemental antimony. This polymeric compound is, in appearance, quite different from previously reported black materials with the claimed formula $(RSb)_x$ which are obtained from the thermal decomposition of $RSbH_2$.[8,9,14] We also isolated this black, amorphous material and found that it slowly turns gray in the presence of air and moisture. It is very likely a mixture of finely divided antimony metal and a variety of $(CH_3Sb)_x$ modifications. (X-ray powder patterns of these amorphous materials frequently show the major bands of elemental antimony.) In contrast, the new pale green solid is indefinitely air stable and consists of small, well-formed crystals.

These reactions of CH_3SbH_2 with organosilicon chlorides were monitored by ^1H NMR. Initially, a small amount of HCl is formed, but it eventually disappears. The concentrations of the silicon compounds remain nearly constant (within experimental error) at their initial levels. Only the concentration of CH_3SbH_2 decreases with time until it is consumed. On opening the reaction tube, we observed a pressure due to a noncondensable gas (hydrogen). These reactions can, therefore, better be described as catalytic. The initial formation of HCl is believed to occur via a halogen–hydrogen exchange (eq 3). Then the HCl produced can catalyze the

$$RSbH_2 + (CH_3)_2SiCl_2 \rightarrow RSbHCl + (CH_3)_2SiHCl \qquad (3)$$
$$RSbHCl \rightarrow (RSb)_x + HCl$$

decomposition of $RSbH_2$ (eq 4). With regard to reaction 3,

$$\begin{array}{c} RSbH_2 + HCl \rightarrow RSbHCl + H_2 \\ RSbHCl \rightarrow (RSb)_x + HCl \\ \hline RSbH_2 \rightarrow (RSb)_x + H_2 \end{array} \qquad (4)$$

it should be noted that CH_3AsH_2 and $(CH_3)_2SiCl_2$ do not undergo H/Cl exchange even at 100 °C.

We also found that CH_3SbH_2 reacts directly with HCl in a 2:1 molar ratio to give the same shiny pale green solid, $(CH_3Sb)_x$. The reaction, however, was much faster and the rapid formation of solid in the reaction tube precluded ^1H NMR monitoring.

Although we were unable to demonstrate with certainty the preparation of *n*-butylstibine due to its very rapid decomposition (the material obtained may also have contained some di-*n*-butyl ether), it did undergo reactions similar to the lighter alkylstibines and produced a product with $(CH_3)_2SiCl_2$ analyzing successfully as $(C_4H_9Sb)_x$.

Discussion

We have previously reported that methylarsine, CH_3AsH_2, with a large variety of halogen sources (both organic and inorganic), produces a purple-black crystalline "polymer", empirical formula $(CH_3As)_x$, possessing a novel ladder structure constructed entirely of arsenic atoms.[2,3] We have concluded from a variety of viewpoints that this macromolecular crystalline form is more thermodynamically stable than the much better known cyclooligomer pentamethylcyclopentaarsine, $(CH_3As)_5$.[9] Well-purified samples of the cyclooligomer convert slowly to the polymer over a period of months at 60 °C. At 200 °C, however, catena forms rapidly revert to cyclooligomer; this suggests that the free energy difference between catena and cyclo forms is small, and that, as could be reasonably assumed, the catena–cyclo transformation occurs with a positive change in entropy.

In contrast, all attempts to prepare analogue arsenic ladder structures with organic groups other than methyl fail to produce stable high-molecular-weight crystalline products; transient polymeric products inevitably give way to cyclooligomeric products. The only consistent explanation for the unique properties of the methyl derivative is based upon steric considerations, not along a ladder strand but rather between parallel ladder strands where the interladder approach distance would be forced by large substituents to an unstable length. The larger antimony-atom van der Waals radius, however, can accommodate larger substituents.

Few cyclopolystibines are known. Red amorphous tetra-*tert*-butylcyclotetrastibine is a minor product in the reduction of $(t\text{-}Bu)_2SbCl$,[7] and very recently an orange-red solid analyzing as $(C_6H_5Sb)_6 \cdot C_6H_6$ has been reported as a high-yield product of the reaction of $C_6H_5SbH_2$ and styrene in benzene.[6] (In remarkable contrast, no reaction occurs when diphenylstibine and styrene are similarly combined.[15]) While cyclopolyphosphines and arsines generally behave as feeble Lewis bases, this cyclostibine would appear to behave as a Lewis acid. Other than these two cyclooligomers, no other polystibine compounds with satisfactory analyses as RSb have previously been reported.

We have not found any evidence for the formation of cyclooligomers of antimony in the present study. We, therefore, conclude that, in continuation of the evident trend toward increasing degrees of association on descending the group 5 elements and homoatomic catenates, only in special cases where substituent bulk prevents macromolecular formation would cyclooligomers of antimony prevail.

The electrical properties of $(RSb)_x$ and $(RSbI_{0.4})_x$ will be described in future reports. Preliminary results (for compressed powders) suggest that $(RSb)_x$ is a better conductor than $(RAs)_x$ by several orders of magnitude, in keeping with the trend toward more metallic properties on descending a main-group family. We hope to expand our short list of R groups to include substituents possessing both +I and −I inductive effects, to ascertain the effect of inductive phenomena on charge carrier density and mobility.

Experimental Section

Published procedures were used for the synthesis of dichloro(methyl)arsine,[16] dimethyliodoarsine,[17] and carbon tetraiodide.[18] The preparations of dichloro(methyl)stibine, dichloro(ethyl)stibine, *n*-

butyldichlorostibine, methylstibine, ethylstibine, and *n*-butylstibine are described in an earlier publication.[12]

$SbCl_3$ (Alfa) was used as obtained. The organosilicon halides (Alfa) were fractionally distilled before use. Alkylstibines were stored at −196 °C and transferred into reaction tubes on an all-glass vacuum line of standard design as quickly as possible to reduce loss by decomposition. The benzene and carbon tetrachloride employed in all studies were spectrograde and were freshly distilled from molecular sieve. Before each experiment, benzene was degassed thoroughly by repeated freeze–pump–thaw cycles.

IR spectra were recorded on a Perkin-Elmer 221G spectrophotometer using a 10-cm path length gas cell fitted with NaCl windows. NMR spectra were recorded on a Perkin-Elmer R20B spectrometer in sealed 5-mm tubes. All NMR chemical shifts are referred to benzene ($Me_4Si = \delta\ 0.00$) which was the solvent in all NMR studies. Mass spectra were recorded on a Du Pont 21-492 spectrometer at 70 eV at 2×10^{-6} mmHg. Elemental analyses were performed by either Integral Analytical Laboratory, Raleigh, N.C., or Schwarzkopf Microanalytical Laboratory, Woodside, N.Y.

Reactions of Alkylstibines with Iodine Sources. In all cases, a 0.5 M solution of the halogen-containing reactant in benzene was introduced into a medium-wall 5-mm o.d. NMR tube and thoroughly degassed by repetitive freeze–pump–thaw cycles. The alkylstibine was condensed into the NMR tube so that the $RSbH_2$:reactant ratio was 2:1. The NMR tube was then sealed on the vacuum line. Solid products were isolated by opening the tubes on the vacuum line and removing volatile materials by distillation. With I_2, ICl, CI_4, and dimethyliodoarsine, the major antimony-containing reaction products are $RSbI_2$ (identified by ^1H NMR) and $RSbI_{0.4}$, the former being a yellow solid readily soluble in common organic solvents and the latter being an involatile purple-black solid insoluble in the same solvents.

For $RSbI_{0.4}$ the analytically determined R:Sb:I ratio of 1:1:0.4 was nearly constant despite variations in R group and molar ratio of reactants. (Anal. Calcd for $CH_3I_{0.4}Sb$: C, 6.40; H, 1.61; I, 27.07; Sb, 64.92. Found: C, 6.29; H, 1.57; I, 27.37; Sb, 65.09. These data are typical of all results.) The mass spectrum of $EtSbI_{0.4}$ curiously shows no fragments containing both iodine and more than one antimony atom. At 70 eV and 350 °C, the major peaks correspond to $(EtSb)_n^+$, $n = 2$–4.

Reaction of Alkylstibines with Organosilicon Chlorides. Equimolar ratios of alkylstibine and $(CH_3)_2SiCl_2$, $(CH_3)_3SiCl$, or $(C_6H_5)_3SiCl$ were combined as 10% solutions in benzene and monitored by ^1H NMR spectroscopy. In all cases a pale greenish purple, metallic-looking solid, $(RSb)_x$, was found. (Anal. Calcd for CH_3Sb: C, 8.77; H, 2.19. Found: C, 8.51; H, 2.16. Calcd for C_2H_5Sb: C, 15.93; H, 3.34. Found: C, 15.76; H, 3.29. Calcd for C_4H_9Sb: C, 26.85; H, 5.07. Found: C, 26.31; H, 4.94.)

Acknowledgment. We are grateful to the Ethyl Corp. for their generous gift of tetraalkyllead reagents used in the synthesis of the alkylantimony compounds and to the Office of Naval Research for partial support of this research.

Registry No. $(CH_3Sb)_x$, 62173-60-8; $(C_2H_5Sb)_x$, 68781-08-8; $(C_4H_9Sb)_x$, 68781-10-2; CH_3SbH_2, 23362-09-6; $C_2H_5SbH_2$, 68781-03-3; n-$C_4H_9SbH_2$, 68781-04-4; $(CH_3)_2SiCl_2$, 75-78-5; $(CH_3)_3SiCl$, 75-77-4; $(C_6H_5)_3SiCl$, 76-86-8; $MeSbI_2$, 30982-87-7; $EtSbI_2$, 68781-05-5; I_2, 7553-56-2; ICl, 7790-99-0; CI_4, 507-25-5; $(CH_3)_2AsI$, 676-75-5; CH_3AsCl_2, 593-89-5; $(CH_3As)_x$, 26403-94-1; CH_3SbCl_2, 42496-23-1; $C_2H_5SbCl_2$, 68781-06-6; $C_4H_9SbCl_2$, 5613-73-0.

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Electrochemistry of Iminodiacetate Complexes of Cobalt at Stationary Electrodes

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Received June 9, 1978

The redox behavior of *cis*- and *trans*-bis(iminodiacetato)cobaltate(III) has been studied in aqueous solution at stationary electrodes. The cis complex exhibits quasi-reversible behavior and a reduction potential estimated to be +0.36 V vs. the SHE. The trans isomer, however, exhibits more complex behavior, and upon reoxidation the cis isomer of the Co(III) system forms quantitatively. The results can be interpreted in terms of a model in which the cis isomer of the Co(II) system is the more stable and forms to the virtual exclusion of the corresponding trans species, whereas the trans species is preferred in the case of Co(III). The model also neatly rationalizes the synthetic methods which have been used to prepare the cis and trans isomers of the Co(III) complex. In addition, according to this model, an upper limit of 0.17 V vs. the SHE can be assigned to the reduction potential of the trans system. Finally, complications which occur in studying the complex at a mercury electrode are discussed, and conditions are described under which these effects are minimized.

Introduction

Two isomeric forms of a 2:1 complex of the iminodiacetate ligand with Co(III) have been characterized.[1] Both complexes are pseudooctahedral with the ligands coordinated on opposite triangular faces. The structures are distinguished by the relative disposition of the nitrogen donors. In one structure the nitrogens coordinate in a cis fashion and in the other trans. We became interested in the redox properties of *cis*- and *trans*-bis(iminodiacetato)cobaltate(III), denoted *cis*-$Co(IDA)_2^-$ and *trans*-$Co(IDA)_2^-$, respectively, when we found that these