

Table III. Crystallographic Data for $\text{Cp}_2\text{Nb}(\text{CO})\text{P}^i\text{PrPh}$ (1)

mol formula	$\text{C}_{20}\text{H}_{22}\text{NbOP}$
fw, g	402.28
cryst syst	monoclinic
space group	$P2_1/c$ (No. 14)
cell dimens	
<i>a</i> , Å	16.899 (3)
<i>b</i> , Å	15.409 (2)
<i>c</i> , Å	13.717 (3)
β, deg	90.94 (2)
<i>V</i> , Å ³	3571.2
<i>Z</i>	8
ρ_{calc} , g cm ⁻³	1.496
linear abs coeff, μ, cm ⁻¹	7.346
<i>F</i> (000)	1648
radiation, Å	$\lambda(\text{Mo K}\alpha) = 0.71073$
scan type	ω -2 θ
scan speed, deg min ⁻¹	1.5-8.3
scan width, deg	$\Delta\omega = 1.2 + 0.347 \tan \theta$
rflns measd	$\pm h, k, l$
θ range, deg	2-22
no. of rflns meads	4411
temp, K	296 (1)
decay, %	-3.6, cor
cutoff for obsd data	$I \geq 3\sigma(I)$
no. of unique obsd data (NO)	1920
no. of variables (NV)	225
transmissn coeff (DIFABS)	0.9107, 1.1340
<i>R</i> (<i>F</i>)	0.038
<i>R</i> _w (<i>F</i>)	0.039
weighting scheme	$w^{-1} = [\sigma^2(I) + (0.04I)^2]^{1/2}$
GO _F	2.780

antibonding orbital. This may contribute to an explanation of asymmetry observed for the P-Nb-CP angles in the structure of 1.

Finally, the low inversion barrier due to an easy antibonding-orbital-assisted planarization of the phosphorus atom may be responsible for the particularity observed in the crystal structure of 1 consisting of the presence of two enantiomers in the asymmetric unit of the unit cell.

Experimental Section

Syntheses were performed under an argon atmosphere by using standard Schlenk-tube techniques. ¹H and ³¹P NMR spectra were recorded on JEOL FX100 and Bruker WM400 spectrometers; chemical shifts are given relative to TMS and H₃PO₄. IR spectra were obtained on a Perkin-Elmer 580B spectrophotometer. $\text{Cp}_2\text{Nb}(\text{CO})\text{H}$ was prepared according to the literature procedure.²⁸

$[\text{Cp}_2\text{Nb}(\text{CO})\text{P}^i\text{PrPhH}]^+\text{Cl}^-$ (1'). To a toluene solution (20 mL) of $\text{Cp}_2\text{Nb}(\text{CO})\text{H}$ (0.3 g, 1.19 mmol) was added dropwise a toluene solution (2 mL) of P^iPrPhCl (0.27 g, 1.22 mmol). An orange precipitate was gradually formed which, after the system was stirred for 30 min, was collected, washed with heptane, and dried under vacuum (0.41 g, yield 78%). Anal. Calcd for $\text{C}_{20}\text{H}_{23}\text{OPClNb}$: C, 54.73; H, 5.21. Found: C, 54.3; H, 5.4. NMR (δ, ppm): ¹H (D₂O) 7.61-7.54 (m, Ph), 5.45 (d, *J* = 2.2 Hz, Cp), 5.53 (d, *J* = 2.2 Hz, Cp), 2.31 (m, CH), 1.22 (m, CH₃), 5.54 (dd, *J* = 7.7, 349 Hz, PH); ³¹P (D₂O) +36. IR (ν_{CO}, CH₂Cl₂): 1960 cm⁻¹.

$\text{Cp}_2\text{Nb}(\text{CO})\text{P}^i\text{PrPh}$ (1). A 1 M aqueous solution of NaOH (15 mL) was added to a suspension of 1' (0.4 g, 0.91 mmol) in 15 mL of toluene, and the mixture was stirred for 15 min. The organic layer was separated and evaporated, yielding a maroon product, which was washed with heptane and dried (0.26 g, yield 72%). Anal. Calcd for $\text{C}_{20}\text{H}_{22}\text{OPNb}$: C, 52.86; H, 4.85. Found: C, 51.7, H 4.3. NMR (δ, ppm): ¹H (C₆D₆) 7.61-6.89 (m, Ph), 4.42 (d, *J* = 1.3 Hz, Cp), 1.85 (m, CH), 1.20 (dd, *J* = 6.6, 13 Hz, CH₃); ³¹P (C₆D₆) -13.7. IR (ν_{CO}, toluene): 1922 cm⁻¹.

Crystallographic Studies. An orange irregularly shaped crystal grown from acetone solution (approximate dimensions 0.25 × 0.15 × 0.12 mm) was mounted on an Enraf-Nonius CAD4 diffractometer. The crystal data and data collection parameters are summarized in Table III. The unit cell was determined and refined from 25 randomly selected reflections obtained by use of the CAD4 automatic routines. Intensities were corrected for Lorentz and polarization effects. All calculations were carried out by use of the Enraf-Nonius SDP package²⁶ with neutral-atom scattering factors. The structure was solved and refined by conventional three-dimensional Patterson, difference Fourier, and full-matrix least-squares methods. The empirical absorption correction DIFABS³⁰ was applied. The Nb, P, O, and C (carbonyl) atoms were refined with anisotropic temperature factors and all other carbon atoms with isotropic factors. The positions of all the hydrogen atoms were calculated by the HYDRO program of SDP, and these atoms were placed riding on the carbon atoms bearing them and included in the final calculations with *B*_{iso} fixed at 5.0 Å².

Registry No. 1, 137966-97-3; 1', 137966-98-4; $\text{Cp}_2\text{Nb}(\text{CO})\text{H}$, 11105-68-3; P^iPrPhCl , 54006-34-7.

Supplementary Material Available: Tables of anisotropic thermal parameters, hydrogen atom coordinates, complete bond distances and angles, and least-squares planes (7 pages); a listing of observed and calculated structure factors (10 pages). Ordering information is given on any current masthead page.

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Convenient One-Pot Synthesis of *tert*-Butyldimethylantimony

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Summary: *tert*-Butyldimethylantimony, (*t*-Bu)Me₂Sb, was prepared by a one-pot reaction of SbCl₃ with 1 equiv of (*t*-Bu)MgCl at -50 °C followed by 2 equiv of MeMgBr at 0 °C in diethyl ether. (*t*-Bu)Me₂Sb was isolated in 64% yield (based on SbCl₃) after fractional vacuum distillation (63 °C at 30 Torr) and can be used as a precursor in the chemical vapor deposition of antimony-containing semiconductor materials.

The III/V antimony-containing semiconductor materials are useful in infrared detection, high-speed devices, and optical fiber communication systems. A variety of binary, ternary, and quaternary III/V semiconductor systems containing antimony have been investigated for use in

infrared detectors operating in the 3-5- and 8-12-μm spectral ranges.¹ These wavelength ranges are important since they are natural windows in the atmosphere for IR transmission.

The ternary alloy InAs_{1-x}Sb_x has the smallest band gap (*x* = 0.6, *E*_g ≈ 0.1 eV at 300 K) of any of the III/V semiconductor materials, and this has attracted interest for

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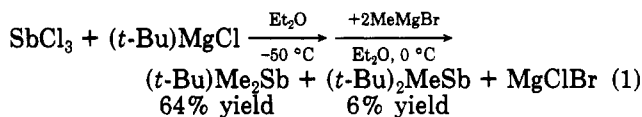
infrared detection in the 8–12- μm spectral range.² The cutoff wavelength at 77 K for $\text{InAs}_{1-x}\text{Sb}_x$ ($x = 0.6$) is $\sim 9 \mu\text{m}$. Further reduction of the band gap is required to extend the cutoff wavelength and can occur if the crystal lattice is dilated with the heavier group V element Bi.³

$\text{InAs}_{1-x}\text{Sb}_y\text{Bi}_z$ and $\text{InSb}_{1-x}\text{Bi}_x$ have been grown by organometallic vapor-phase epitaxy (OMVPE) using trimethylantimony, Me_3Sb , as the organoantimony source compound.³ Bismuth concentrations higher than 2 atom %, as well as growth temperatures above 400 °C, resulted in poor film morphology during the growth.^{3b} However, growth temperatures lower than 400 °C resulted in significant problems due to the incomplete pyrolysis of Me_3Sb . Me_3Sb is an inefficient antimony source compound at low film growth temperatures since it is only 15% pyrolyzed at 400 °C.⁴ In the case of the Bi-containing materials, the situation is further complicated since Bi tends to phase separate.

In order to minimize the tendency of the Bi to phase separate and to limit the diffusion in these alloys, an alternative organoantimony precursor for OMVPE is needed which has a lower pyrolysis temperature than Me_3Sb . Recently $(i\text{-Pr})_3\text{Sb}$ was used to grow InSb films by OMVPE as low as 300 °C, the lowest reported film growth temperature of InSb using a stable Sb source compound.⁵ However, the low vapor pressure of $(i\text{-Pr})_3\text{Sb}$ in comparison to that of Me_3Sb resulted in low film growth rates. Therefore, an organoantimony precursor with a higher vapor pressure and a low decomposition temperature is still required.

Results and Discussion

Although unsymmetric stibines with *tert*-butyl substituents such as $(t\text{-Bu})\text{R}_2\text{Sb}$ ($\text{R} = \text{OMe}, \text{OEt}$)⁶ and $(t\text{-Bu})_2\text{RSb}$ ($\text{R} = \text{C}\equiv\text{CH}, \text{H}^{\text{a}}$) are known, *tert*-butyldimethylantimony, $(t\text{-Bu})\text{Me}_2\text{Sb}$, has not been reported until now. $(t\text{-Bu})\text{Me}_2\text{Sb}$ was prepared by a one-pot reaction of SbCl_3 with 1 equiv of $(t\text{-Bu})\text{MgCl}$ at -50 °C followed by 2 equiv of MeMgBr at 0 °C in diethyl ether (eq 1). $(t\text{-Bu})_2\text{MeSb}$ was isolated in 6% yield as a major organoantimony byproduct from this reaction.



This one-pot synthetic methodology avoids the isolation of the alkylantimony dihalide intermediate (RSbX_2) prior to subsequent alkylation. Although $(t\text{-Bu})\text{SbBr}_2$ ⁶ and $(t\text{-Bu})\text{SbCl}_2$ ⁹ can be prepared by controlled substitution reactions of the corresponding antimony trihalide with (*t*-

Bu) MgCl at low temperatures, they have been isolated in only 31.5% and 37.6% yield, respectively. Furthermore, $(t\text{-Bu})\text{SbBr}_2$ is unstable even upon storage at 0 °C.⁶

Homolysis of metal–carbon bonds is an important pyrolytic mechanism in OMVPE.¹⁰ Breaking the weakest bond, typically the metal–carbon bond, is the rate-determining step in the pyrolysis of many organometallic source compounds. The $(t\text{-Bu})\text{-Sb}$ bond is weaker than the Me-Sb bond on the basis of comparative activation energies for the formation of a *tert*-butyl free radical (93 kcal/mol) versus that of a methyl free radical (105 kcal/mol).¹¹ $(t\text{-Bu})\text{Me}_2\text{Sb}$ decomposes in the gas phase at lower temperatures than Me_3Sb due to the reduced bond strength of the $(t\text{-Bu})\text{-Sb}$ bond.¹² Therefore, $(t\text{-Bu})\text{Me}_2\text{Sb}$ can be used as an alternative precursor to the low-temperature OMVPE growth of antimony-containing semiconductor materials.¹³ Furthermore, $(t\text{-Bu})\text{Me}_2\text{Sb}$ has a higher vapor pressure (3.6 Torr at 25 °C) than $(i\text{-Pr})_3\text{Sb}$ ¹⁴ (0.4 Torr at 25 °C). These properties as well as the herein reported convenient synthesis of $(t\text{-Bu})\text{Me}_2\text{Sb}$ make this compound an attractive alternative antimony source for the OMVPE of antimony-containing semiconductor materials.

Experimental Section

General Procedures. Organoantimony compounds should be handled with extreme caution since they are toxic.¹⁵ Organic solvents were distilled under Ar from sodium/benzophenone. Synthesis was carried out under purified Ar using inert-atmosphere techniques. Air- and moisture-sensitive materials were transferred inside a N_2 -filled Vacuum Atmospheres glovebox. SbCl_3 (99.99% metal basis) was purchased from Alfa. Methyl and *tert*-butyl Grignard reagents were purchased from Aldrich and used as received. Elemental analyses were performed by E + R Microanalytical Laboratory, Inc. NMR spectra were recorded on C_6D_6 solutions with a Bruker AMX-400 spectrometer. ¹³C NMR spectra were obtained at 100.6 MHz with ¹H-coupled ¹³C spectra obtained under gated decoupling conditions.

$(t\text{-Bu})\text{Me}_2\text{Sb}$. To a solution of 148.6 g (651.5 mmol) of SbCl_3 (99.99% pure by metal basis) in 800 mL of Et_2O at -50 °C was added dropwise (drop time 3 h) with vigorous stirring 342 mL (2.0 M in Et_2O , 684 mmol) of $(t\text{-Bu})\text{MgCl}$. After the addition was complete, the slurry was stirred at -50 °C for 1 h. Then the slurry was warmed to room temperature slowly (about 2 h) and stirred at room temperature for an additional 1.5 h. The slurry was then cooled to 0 °C, and 500 mL (2.7 M diethyl ether, 1350 mmol) of MeMgBr was added dropwise (drop time 3 h) to the slurry at 0 °C. The slurry was stirred at room temperature for 24 h and then refluxed for 3 h. The slurry was cooled to 0 °C, and 500 mL of deoxygenated distilled water was added dropwise (drop time 2 h) to the slurry with stirring. A condenser was used to condense the Et_2O from this exothermic step. The slurry was stirred 24 h and then allowed to settle. The upper organic layer was transferred to a Schlenk flask with anhydrous MgSO_4 and stirred 24 h at room temperature. The slurry was filtered, and the solvent was removed by fractional distillation. $(t\text{-Bu})\text{Me}_2\text{Sb}$, an extremely air-sensitive colorless liquid, was collected in a liquid-nitrogen trap down to 6 Torr using a 55 °C water bath. The remaining liquid residue in the distillation pot appeared to be primarily $(t\text{-Bu})_2\text{MeSb}$ (10.5 g, 6% yield based on SbCl_3) by ¹H NMR (C_6D_6): 1.17 ppm (s, 18 H, $(\text{CH}_3)_3\text{C}$), 0.58 ppm (s, 3 H, CH_3). $(t\text{-Bu})\text{Me}_2\text{Sb}$ was then purified by fractional vacuum distillation

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at 44 °C and 11 Torr (87.7 g, 64.4% yield based on SbCl_3). (*t*-Bu) Me_2Sb was purified by fractional vacuum distillation two more times (55 °C at 20 Torr and 63 °C at 30 Torr): vapor pressure equation $\log P$ (mmHg) = 8.7082 - 2429/[T (K)]; mp 12-13 °C. ^1H NMR (C_6D_6): 1.07 ppm (s, 9 H, $(\text{CH}_3)_3\text{C}$), 0.55 ppm (s, 6 H, CH_3). ^{13}C NMR (C_6D_6): 29.8 ppm (q of septets, $(\text{CH}_3)_3\text{C}$, $^1J_{\text{CH}} = 125$, $^3J_{\text{CH}} = 5.5$ Hz), 20.9 ppm (s, $(\text{CH}_3)_3\text{C}$), 4.7 ppm (q, CH_3 , $^1J_{\text{CH}} = 131$ Hz). Anal. Calcd for $\text{C}_8\text{H}_{16}\text{Sb}$: C, 34.49; H, 7.24; Sb, 58.27. Found: C, 34.63; H, 7.20; Sb, 57.50.

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Registry No. (*t*-Bu) Me_2Sb , 138260-00-1; SbCl_3 , 10025-91-9; (*t*-Bu) MgCl , 677-22-5; (*t*-Bu) $_2\text{MeSb}$, 138260-01-2.

Stabilization of Pentaborane(9) toward Air Oxidation and Facile Preparation of *nido*- $\text{R}_2\text{C}_2\text{B}_4\text{H}_6$ Clusters in Organic Solutions

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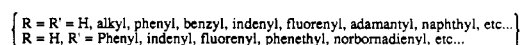
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Summary: Solutions of pentaborane(9) in tetrahydrofuran (THF) were found to be indefinitely stable under inert conditions and relatively stable to dry air. Solutions exposed to the air were found to decompose slowly to boric acid. Similar stabilities were also found for other pentaborane(9) organic solvent systems including diglyme, diethyl ether, and hexane. No vigorous reactions with air or moisture, including pyrophoric or shock-sensitive behavior, were observed for any of the solutions investigated. The reaction of pentaborane(9) with triethylamine and either 3-hexyne or phenylacetylene in THF at room temperature was found to produce up to 80% of *nido*-2,3-(CH_3CH_2) $_2\text{C}_2\text{B}_4\text{H}_6$ and 20% of *nido*-2,3-(C_6H_5)- $\text{HC}_2\text{B}_4\text{H}_6$, respectively. In addition, the reaction of diphenylacetylene with the pentaborane(9)-THF solution and triethylamine was also explored to determine if the preparation of the synthetically very difficult *nido*-2,3-(C_6H_5) $_2\text{C}_2\text{B}_4\text{H}_6$ carborane could be achieved. The reaction was found to give a 21% yield of the *nido*-2,3-(C_6H_5) $_2\text{C}_2\text{B}_4\text{H}_6$ carborane after 1 day at room temperature. The nature of the interaction between pentaborane(9) and THF has been investigated by ambient- and low-temperature ^{11}B NMR studies.

The synthetic and organometallic chemistry of many small borane, heteroborane, and related systems has been made accessible through the use of *nido*-pentaborane(9), B_5H_9 .¹ In fact, pentaborane(9) is an extremely versatile synthon in the preparation of a wide variety of boron clusters, including the larger boranes such as $[\text{B}_9\text{H}_{14}]^-$ anion, $\text{B}_{10}\text{H}_{14}$, and $\text{B}_{18}\text{H}_{22}$.^{1d,2} Pentaborane(9), however, requires extreme caution in handling even under high-vacuum or inert-atmosphere conditions due to its explosive reactivity with air.^{3,4} Thus, synthetic reactions requiring

Scheme I. Small Substituted Carborane Synthesis⁶



pentaborane(9) as a starting material are typically run on relatively small scales (typically less than 50 mmol per reaction vessel) and require laborious handling and safety precautions.^{1,3,5} An alternative or modified method in which pentaborane(9) is stabilized but its reactivity not significantly altered is therefore of great practical interest.

The small carboranes⁶ and phosphinoboranes⁷ derived from pentaborane(9) have been shown to exhibit a broad diversity in their structural and organometallic chemistry. The reactions of pentaborane(9) with unsaturated main-group compounds, such as alkynes⁵ and phosphalkenes,⁷ produce a variety of both cage-inserted and cage-substituted cluster compounds. The neat reaction of alkynes with pentaborane(9) in the presence of amine bases such as triethylamine produces *nido*-2,3- $\text{R}_2\text{C}_2\text{B}_4\text{H}_6$ carborane clusters in up to 30% yield (Scheme I).^{5,6} While this reaction has been shown to be a very general and useful

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