Synthesis and X-ray Structures of All-Alkyl-Substituted AlSb Ring Compounds

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*Summary: The dehalosilylation reaction between diorganoaluminum hydrides R_2AlH (R = Me, Et) and t-$Bu_2SbSiMe_3$ leads to the formation of the first all-alkyl-substituted Al−Sb heterocycles $[Me_2AlSb(t\text{-}Bu)_2]_3$ (**1**) and $[Et_2AlSb(t\text{-}Bu)_2]_2$ (**2**). In addition, the synthesis of $(Me_2Al)_3[Sb(t\text{-}Bu)_2]_2Sb(SiMe_3)_2$ (**3**) is described. **1** and **3** were characterized by single-crystal X-ray analysis.*

Introduction

Numerous amide, phosphide, and arsenide compounds of group 13 (Al, Ga, In) which form simple Lewis acid−base adducts or cyclic compounds have been synthesized within the past decade.[1] However, only a handful of group 13 antimonides have been prepared and structurally characterized (Ga− and In−Sb,[2] Al−Sb).[3] Wells et al. investigated in detail the synthesis of Ga and In antimonides by dehalosilylation reactions of diorganogallium or diorganoindium halides and $Sb(SiMe_3)_3$.[4] Our group was particularly interested in the synthesis and structural characterization of AlSb single-source precursors and recently reported investigations concerning reactions between $Sb(SiMe_3)_3$ and aluminum trialkyls, diorganoaluminum halides, and diorganoaluminum hydrides, leading to the simple Lewis acid−base adducts $R_3Al{\leftarrow}Sb(SiMe_3)_3$ and $R_2AlCl{\leftarrow}Sb(SiMe_3)_3$ and to cyclic compounds containing Al−Sb σ-bonds, $[R_2AlSb(SiMe_3)_2]_x$ (x = 2, 3).[5] The potential of the heterocycles to produce AlSb films in MOCVD experiments was demonstrated.[6] Such prepared films showed no contamination with carbon but a temperature-dependent incorporation of Si between 5 and 25%. To avoid any Si contamination, we were interested in the synthesis of potential precursors which do not contain silyl groups but, rather, are completely alkyl-substituted. The synthesis of the first all-alkyl-substituted Al−Sb ring compounds is reported here.

Table 1. Crystallographic Data and Measurements for $[Me_2AlSbt\text{-}Bu_2]_3$ (1) and $(Me_2Al)_3[Sb[t\text{-}Bu)_2]_2Sb(SiMe_3)_2$ (3)

	1	3
mol formula	$C_{30}H_{72}Al_3Sb_3$	$C_{28}H_{72}Al_3Sb_3Si_2$
fw	879.07	911.23
cryst syst	monoclinic	monoclinic
space group	$P2_1/c$ (No. 14)	$P2_1/c$ (No. 14)
a, Å	9.3705(2)	9.3949(2)
b, Å	21.8233(4)	22.2071(4)
c, Å	20.3768(3)	20.5648(5)
β, deg	91.744(1)	90.699(1)
V, Å^3	4561.03(13)	4290.19(16)
Z	4	4
radiation (wavelength, Å)	Mo Kα (0.710 73)	Mo Kα (0.710 73)
μ, mm^{-1}	2.011	2.008
temp. K	123(2)	123(2)
D_{calcd}, g cm^{-3}	1.402	1.411
crystal dimens, mm	0.20 × 0.15 × 0.10	0.20 × 0.15 × 0.05
$2\theta_{max}$, deg	56.6	50.0
no. of rflns recorded	42 896	50 401
no. of nonequiv rflns recorded	10 019	7547
R_{merg}	0.064	0.044
no. of params refined	331	331
R1,[a] wR2[b]	0.039, 0.107	0.040, 0.100
goodness of fit[c]	1.044	1.112
max, min transmissn	0.7777, 0.7129	0.7232, 0.6677
final max, min Δρ, e Å^{-3}	1.776, −1.743	1.311, −1.406

[a] $R1 = \sum(||F_o| - |F_c||)/\sum|F_o|$ (for $I > 2\sigma(I)$). [b] $wR2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{1/2}$. [c] Goodness of fit $= \{\sum[w(|F_o^2| - |F_c^2|)^2]/(N_{observns} - N_{params})\}^{1/2}$.

Experimental Section

General Considerations. All manipulations were performed in a glovebox under an N_2 atmosphere or by standard Schlenk techniques. Me_2AlH,[7] Et_2AlH,[8] and t-$Bu_2SbSiMe_3$[9]

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Table 2. Selected Bond Distances (Å) and Angles (deg) for [Me_2AlSb*t*-$Bu_2]_3$ (1) and ($Me_2Al)_3[Sb[$*t*-$Bu)_2]_2Sb(SiMe_3)_2$ (3)[a]

dist	**1**	**3**	angle	**1**	**3**
Sb1−Al1	2.7839(10)	2.7320(16)	Al1−Sb1−Al3	128.92(3)	123.17(5)
Sb1−Al3	2.7679(10)	2.7199(17)	Al1−Sb2−Al2	115.28(3)	128.37(5)
Sb2−Al1	2.7188(10)	2.7678(16)	Al2−Sb3−Al3	121.33(3)	115.38(5)
Sb2−Al2	2.7319(11)	2.7803(16)	X−Sb1−Y	105.07(13)	102.31(9)
Sb3−Al2	2.7306(11)	2.7194(16)	C21−Sb2−C25	106.02(14)	105.06(19)
Sb3−Al3	2.7428(10)	2.7245(17)	C31−Sb3−C35	104.87(13)	106.2(2)
Sb1−X	2.222(3)	2.457(3)	C1−Al1−Sb2	112.79(14)	108.23(19)
Sb1−Y	2.234(3)	2.442(3)	C2−Al1−Sb2	103.76(12)	110.54(19)
Sb2−C21	2.226(3)	2.246(5)	C1−Al1−Sb1	110.92(12)	107.7(2)
Sb2−C25	2.208(4)	2.263(5)	C2−Al1−Sb1	111.03(12)	108.81(18)
Sb3−C31	2.248(4)	2.244(5)	X−Sb1−Al1	104.37(9)	109.14(7)
Sb3−C35	2.254(3)	2.219(5)	Y−Sb1−Al1	100.81(9)	106.55(8)
Al1−C1	1.981(4)	1.968(6)	X−Sb1−Al3	107.00(9)	102.17(7)
Al1−C2	1.980(4)	1.964(6)	Y−Sb1−Al3	100.81(9)	111.52(7)
Al2−C3	1.982(5)	1.978(6)	Sb1−Al1−Sb2	102.80(3)	106.89(5)
Al2−C4	1.957(5)	1.980(6)	Sb2−Al2−Sb3	108.18(4)	103.10(5)
Al3−C5	1.974(4)	1.976(6)	Sb1−Al3−Sb3	107.80(3)	106.61(6)
Al3−C6	1.976(4)	1.965(6)	C1−Al1−C2	114.71(19)	114.4(3)
			C3−Al2−C4	121.4(2)	115.0(3)
			C5−Al3−C6	114.47(17)	120.4(3)

[a] X = C11 (**1**), Si1 (**3**); Y = C15 (**1**), Si2 (**3**).

were prepared by literature methods. ^1H and $^{13}C\{^1H\}$ spectra were recorded using a Bruker AMX 300 spectrometer and are referenced to internal C_6D_5H ($\delta(^1H)$ 7.154, $\delta(^{13}C)$ 128.0). Mass spectra were recorded on a VG Masslab 12-250 spectrometer in the electron ionization mode at 20 eV. Infrared spectra were recorded in Nujol between KBr plates with a Nicolet Magna 550 and are reported in reciprocal centimeters. Melting points were observed in sealed capillaries and were not corrected. Elemental analyses were performed at the Mikroanalytisches Labor der Universität Bonn.

Preparation of [Me_2AlSb(t-$Bu)_2]_3$ (1). Me_2AlH (0.29 g, 5 mmol) and t-$Bu_2SbSiMe_3$ (1.55 g, 5 mmol) were combined at room temperature and stirred for 10 min at 135 °C in a preheated oil bath. After gas evolution had stopped, the white solid which remained was dissolved in pentane/hexane (5:1; 30 mL) and stored at 0 °C. After 10 h **1** was obtained as colorless crystals (1.41 g, 1.6 mmol, 96%). Mp: 184 °C dec. Anal. Calcd (found) for $C_{30}H_{72}Al_3Sb_3$: C, 41.0 (40.93); H, 8.20 (8.17). ^1H NMR (300 MHz, C_6D_5H, 25 °C): δ 0.02 (s, 6H, Me), 1.54 (s, 18H, *t*-Bu). $^{13}C\{^1H\}$ NMR (80 MHz, C_6D_5H, 25 °C): δ −3.2 (Me), 32.3 (CMe$_3$), 35.3 (CMe_3). MS (EI, 20 eV; *m*/*z* (%)): 643 (1), $M^+ - Sb$-t-Bu_2; 571 (5), $^2/_3M^+ - Me$; 486 (4), $^2/_3M^+$; 446 (5), $Al_3Sb_3^+$; 349 (10), $(Me_2Al)_2Sb$-t-Bu_2^+; 292 (6), $^1/_3M^+$; 235 (15), Sb-t-Bu_2^+; 56 (100), Me_2Al^+. IR (Nujol, KBr): ν 1243, 1178, 1149, 1016, 937, 836, 806, 671, 561 cm^{-1}.

Preparation of [Et_2AlSb(t-$Bu)_2]_2$ (2). Et_2AlH (0.17 g, 2 mmol) was added to t-$Bu_2SbSiMe_3$ (0.62 g, 2 mmol), and the mixture was heated for 30 min at 155 °C in a preheated oil bath, while gas evolution was observable. After the mixture was cooled to room temperature, the resulting white solid was dissolved in 30 mL of pentane and stored at −30 °C. After 2 days, **2** was obtained as colorless crystals (0.58 g, 0.9 mmol, 90%). Mp: 192 °C dec. Anal. Calcd (found) for $C_{24}H_{56}Al_2Sb_2$: C, 44.90 (45.02); H, 8.73 (8.79). ^1H NMR (300 MHz, C_6D_5H, 25 °C): δ 0.60 (q,$^3J_{HH}$ = 8.1 Hz, 4H, CH_2), 1.44 (t, $^3J_{HH}$ = 8.1 Hz, 6H, CH_3), 1.50 (s, 18H, *t*-Bu). $^{13}C\{^1H\}$ NMR (80 MHz, C_6D_5H, 25 °C): δ 3.0 (CH_2CH_3), 10.7 (CH_2CH_3), 32.6 (CMe$_3$), 35.2 (CMe_3). MS (EI, 20 eV; *m*/*z* (%)): 640 (1), M^+; 405 (5), $M^+ - Sb$-t-Bu_2; 320 (25), $M^+/2$; 296 (6), $Al_2Sb_2^+$; 57 (100), t-Bu^+. IR (Nujol, KBr): ν 1243, 1224, 1149, 1016, 981, 948, 914, 836, 804, 723, 626, 592, 530 cm^{-1}.

Preparation of ($Me_2Al)_3(Sb$-t-$Bu_2)_2Sb(SiMe_3)_2$ (3). Me_2AlH (0.29 g, 5 mmol) and t-$Bu_2SbSiMe_3$ (1.55 g, 5 mmol) were combined at room temperature and slowly heated to 130 °C. After gas evolution had stopped, the residual white solid was dissolved in 25 mL of pentane and stored at 0 °C. Within 10 h colorless crystals (1.32 g) were formed. Mp: 186 °C dec. Anal. Calcd (found) for $C_{28}H_{72}Al_3Sb_3Si_2$: C, 36.91 (36.17); H, 7.96 (7.81). ^1H NMR (300 MHz, C_6D_5H, 25 °C): δ 0.01 (s, 12H, 4Me), 0.02 (**1**), 0.04 (s, 6H, 2Me), 0.52 (s, 18H, $2SiMe_3$), 1.37 (s, 36H, 4*t*-Bu), 1.54 (**1**). $^{13}C\{^1H\}$ NMR (80 MHz, C_6D_5H, 25 °C): δ −3.1 (Me), −3.2 (**1**), 5.7 ($SiMe_3$), 30.1 (CMe$_3$), 31.4 (CMe_3), 32.3 (**1**), 35.3 (**1**). MS (EI, 20 eV; *m*/*z* (%)): 853 (1), $M^+ - t$-Bu; 781 (1), $M^+ - t$-$Bu - SiMe_3$; 675 (3), $M^+ - Sb$-t-Bu_2; 602 (5), $M^+ - Sb$-t-$Bu_2 - SiMe_3$; 486 (10), Me_2AlSb-t-$Bu_2SbSiMe_3^+$; 446 (10), $Al_3Sb_3^+$; 292 (25), Me_2AlSb-t-Bu_2^+; 267 (20), $Sb(SiMe_3)_2^+$; 235 (35), Sb-t-Bu_2^+; 73 (20), $SiMe_3^+$; 56 (100), $AlMe_2^+$. IR (Nujol, KBr): ν 1149, 1016, 838, 721, 667, 559 cm^{-1}.

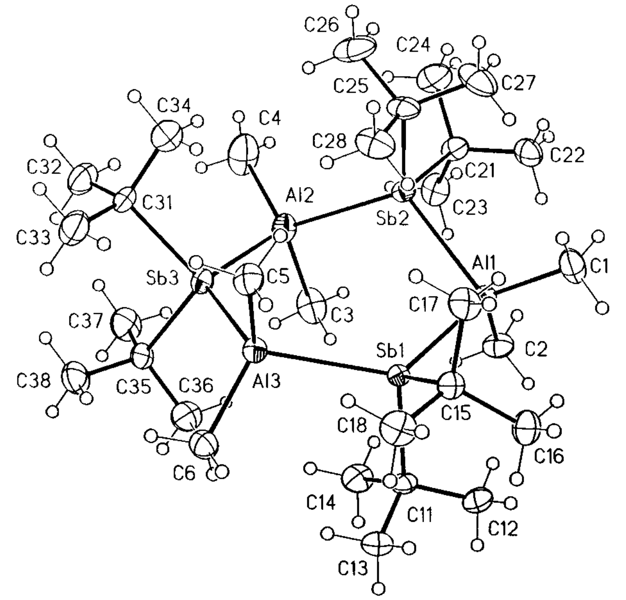


Figure 1. ORTEP diagram (50% probability ellipsoids) showing the solid-state structure and atom-numbering scheme for **1**.

X-ray Structure Solution and Refinement. Crystallographic data of **1** and **3** are summarized in Table 1 and selected bond lengths and angles in Table 2. Figures 1 and 2 show the ORTEP diagrams of the solid-state structures of **1** and **3**. Data were collected on a Nonius Kappa-CCD diffractometer. Structures were solved by direct methods (SHELXS-97)[10] and refined by full-matrix least squares on F^2. All non-hydrogen atoms were refined anisotropically, and hydrogen

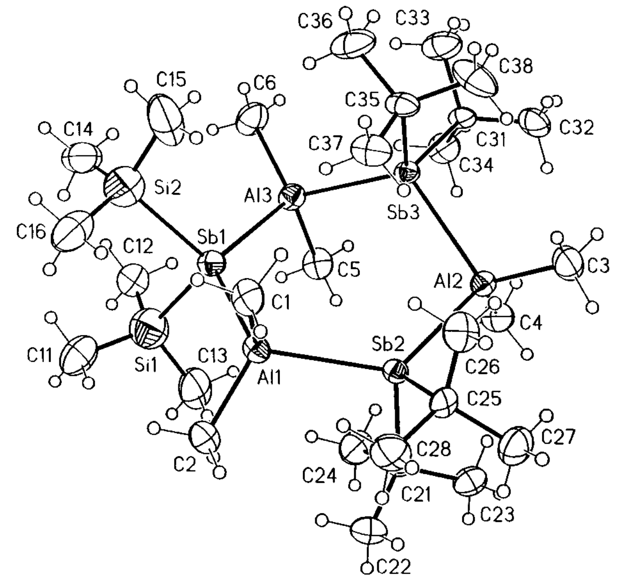


Figure 2. ORTEP diagram (50% probability ellipsoids) showing the solid-state structure and atom-numbering scheme for **3**.

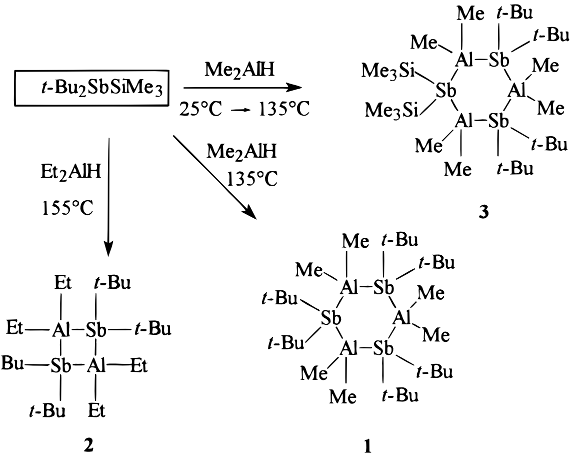
atoms were refined by a riding model (SHELXL-97).[11] An empirical absorption correction was applied.

Results and Discussion

Diorganoaluminum hydrides and *t*-$Bu_2SbSiMe_3$ reacted with evolution of Me_3SiH in almost quantitative yield, giving the four- and six-membered cyclic systems **1**−**3** (Scheme 1). To obtain pure **1** and **2**, it was necessary to perform the reaction in a preheated oil bath at 135 °C for **1** and at 155 °C for **2**. If the reaction was performed with a slower increase in temperature, partially silylated cyclic compounds were obtained, as detected by ^1H NMR investigations. This is due to the fact that *t*-$Bu_2SbSiMe_3$ starts to decompose at 80 °C, and this results in the formation of *t*-$BuSb(SiMe_3)_2$ and $Sb(SiMe_3)_3$.[9] These compounds react with the aluminum hydrides to give the partially silylated cyclics in addition to the all-alkylated compound. Separation of the all-alkyl and partially silylated compounds by sublimation or crystallization was not possible.

The ^1H spectra of **1** and **2** show the organic substituents at 0.02 (Me_2Al) and 1.54 ppm (*t*-Bu_2Sb) and at 0.61, 1.42 (Et_2Al) and 1.50 ppm (*t*-Bu_2Sb), respectively. The ^1H spectrum of **3** is more complicated, showing signals for MeAl groups at 0.01, 0.02, and 0.04 ppm. Silyl groups were detected at 0.52 ppm and *t*-BuSb groups at 1.37 and 1.54 ppm. The resonances at 0.02 and 1.54 ppm correspond to those of **1**, and the resonances at 0.01, 0.04, 0.52, and 1.37 ppm may indicate the partially silylated ring of **3**. The intensities of the Me_2Al resonances for **3** are, as expected, in the ratio 2:1. However, the presence of resonances corresponding to those of **1** does not necessarily mean that the solid product is a mixture of **1** and **3**. Unit cell determinations of 10 crystals of this product only show the presence of **3** in the solid state. Therefore, it is possible that **1** is formed only in solution by ligand exchange reactions which could proceed via dissociation/recombination processes.[12]

Scheme 1. Reaction Scheme and Line Drawings for 1−3



While the mass spectra of **1** and **3** do not show peaks due to the molecular ions, the mass spectrum of **2** does show a low intense molecular ion peak. The mass spectra of **1** and **3** show an $Al_3Sb_3^+$ fragment ion at *m*/*z* 446 with low intensity, indicating that the six-membered ring is relatively stable under these conditions. The mass spectrum of **1** shows an $M^+/3$ (*m*/*z* 292) ion and that of **2** an $M^+/2$ (*m*/*z* 320) ion. The mass spectrum of **3** also shows an $Sb(SiMe_3)_2^+$ ion (*m*/*z* 267).

Single crystals were obtained at 0 °C from a solution in pentane (**3**) or pentane/hexane (10:1 (**1**)). Obviously, the ring size depends on the bulkiness of the substituents on Al. Small Me groups lead to the formation of six-membered rings in **1** and **3**, while the somewhat greater size of the Et groups favors the formation of a four-membered ring in **2**. Analogous results were observed in reactions of the corresponding aluminum hydrides with $Sb(SiMe_3)_3$, which gave $[Me_2AlSb(SiMe_3)_2]_3$,[5b] and $[Et_2AlSb(SiMe_3)_2]_2$.[5a] **1** and **3** are isostructural with $[Me_2AlSb(SiMe_3)_2]_3$ and $[Me(Cl)AlSb(SiMe_3)_2]_3$.[5b] The nonplanar Al_3Sb_3 rings in **1** and **3** adopt distorted twist-boat conformations, and the Al and Sb atoms reside in distorted-tetrahedral environments. The ranges of the Al−Sb bond lengths observed are comparable (**1**, 2.719(1)−2.784(1) Å; **3**, 2.719(2)−2.780(2) Å) and are longer than those found in $[Me_2AlSb(SiMe_3)_2]_3$ (2.703(1)−2.738(1) Å) and $[Me(Cl)AlSb(SiMe_3)_2]_3$ (2.69−2.72 Å). The average values of 2.75 Å

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(12) Solid-state NMR investigations (^1H, ^{27}Al) were performed but did not solve the question whether **3** is a mixture of partially silylated and completely alkylated compounds or is pure, due to the line broadening of the resonances.

(13) For example: $[Me_2AlSb(SiMe_3)_2]_3$ (Al−Sb−Al, 118.5(1)−128.2(1)°; Sb−Al−Sb, 103.5(1)−106.5(1)°),[6b] $[Me_2AlAs(CH_2SiMe_3)Ph]_3$ (Al−As−Al, 118.2(2)−122.2(2)°; As−Al−As, 102.6(2)−104.8(2)°),[14] $[Me_2AlAsPh_2]_3 \cdot 2C_7H_8$ (Al−As−Al, 118.1(1)−122.7(1)°; As−Al−As, 99.1(1)−101.1(1)°),[14] $[Me_2AlN(CH_2)_2]_3$ (Al−N−Al, 119.9(5)°; N−Al−N, 102.0(5)°),[15] $[Me_2AlBi(SiMe_3)_2]_3$ (Al−Bi−Al, 121.7(1)−130.5(1)°; Bi−Al−Bi, 101.0(1)−104.1(1)°),[16] $[Me_2GaSb(SiMe_3)_2]_3$ (Ga−Sb−Ga, 118.3(2)−127.6(2)°; Sb−Ga−Sb, 103.7(2)−107.3(2)°),[17] $[Me_2InSb(t\text{-}Bu)_2]_3$ (In−Sb−In, 115.8(1)−127.8(1)°; Sb−In−Sb, 103.7(1)−109.4(1)°).[2c]

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(**1**) and 2.74 Å (**3**) compare well with the sum of the covalent radii of Al and Sb (2.73 Å). In agreement with the VSEPR model, the endocyclic Sb−Al−Sb bond angles are significantly smaller (**1**, 102.8(1)−108.2(1)°; **3**, 103.1(1)−106.9(1)°) and the Al−Sb−Al bond angles greater (**1**, 115.3(1)−128.9(1)°; **3,** 115.4(1)−128.4(1)°) than the tetrahedral value. Comparable values were found in other nonplanar six-membered heterocycles of group 13 and group 15 elements.[13]

Investigations concerning MOCVD experiments using **1** and **2** are in progress.

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Supporting Information Available: Tables of bond distances, bond angles, anisotropic temperature factor parameters, and fractional coordinates and figures giving additional views of the structures for **1** and **3**. This material is available free of charge via the Internet at http://pubs.acs.org.

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