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Notes

The Ate Complexes [M{C(SiMe$_3$)$_3$}(μ-SBu)$_2$Li(THF)$_2$] (M = Ge or Sn). The First Structural Characterization of Organometallic Ate Complexes of Group 14 Metals in Oxidation State II

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Summary: The ate complexes [M{C(SiMe$_3$)$_3$}(μ-SBu)$_2$Li(THF)$_2$], M = Ge or Sn, have been made from M(SBu)$_2$ and LiC(SiMe$_3$)$_3$ in tetrahydrofuran and their crystal structures determined. The LiS$_2$M rings are folded at the S⋯S axis, the ring in the tin compound more so than that in the germanium derivative. The latter has a conformation very like the ring in [(Me$_3$Si)$_3$CAl(SMe)(μ-SMe)$_2$Li(THF)$_2$]. NMR measurements show that the LiS$_2$M rings are preserved in toluene solution, but the Li−S bonds in the tin compound are transiently broken in THF.

Introduction

There has been much recent interest in stable germanium, tin, and lead carbene analogues of the types MR$_2$[1−12] and MRX,[13−17] where R is a bulky aryl or alkyl group and X is Cl or Br. Compounds of the type (MRCl)$_n$ [with (a) M = Ge or Sn, R = 2,6-{2,4,6-Me$_3$C$_6$H$_2$}$_2$C$_6$H$_3$, $n = 2$;[16] (b) M = Sn, R = C(SiMe$_2$Ph)$_3$, $n = 2$;[14] (c) M = Sn or Pb, R = C(SiMe$_3$)$_2$(SiMe$_2$OMe), $n = 2$,[14] (d) M = Ge, Sn, or Pb, R = C(SiMe$_3$)$_2$(SiMe$_2$C$_5$H$_4$N-2), $n = 1$[15] and (e) M = Pb, R = C(SiMe$_3$)$_3$, $n = 3$[14]], the bromide (PbRBr)$_2$, and the complex PbRBr·py[17] [R = 2,6-(2,6-Pr$^i_2$-C$_6$H$_3$)$_2$C$_6$H$_3$] have been structurally characterized. Attempts to make the compounds Sn[C(SiMe$_3$)$_3$]$_2$ and Sn[C(SiMe$_3$)$_3$]Cl have so far been unsuccessful. There are strong indications that the ate derivative Li[Sn{C(SiMe$_3$)$_3$}Cl$_2$] is the initial product from the reaction of the dihalides MX$_2$ with the organolithium reagent LiC(SiMe$_3$)$_3$,[18,19] and in the case of the reaction of GeCl$_2$·(1,4-dioxane) with LiC(SiMe$_3$)$_3$ in tetrahydrofuran (THF) the isolated product was shown spectroscopically to be the ate complex (Me$_3$Si)$_3$CGeCl·LiCl·3THF.[20] The crystal structures of these lithium compounds were, however, not determined. We describe below the ate complexes of the type Li[MRX$_2$], where M is Ge or Sn and X = SBu. As far as we are aware, these are the first σ-bonded organometalates of germanium(II) and tin(II) to be structurally characterized. The structures of the trithiolato complexes [Et$_4$N][Ge(SPh)$_3$][21] and [Ph$_4$As][Sn(SPh)$_3$][22] have been reported and those of a few other

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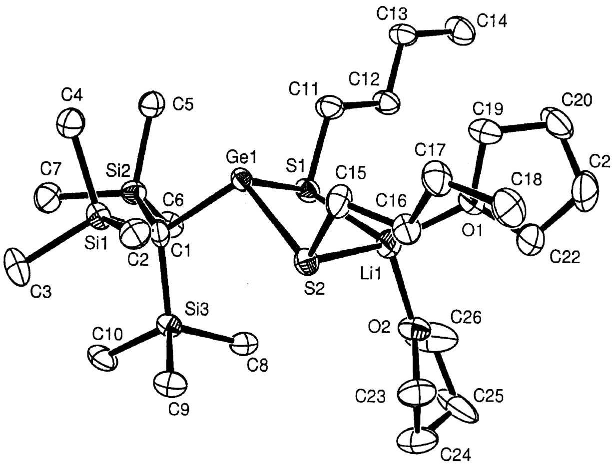
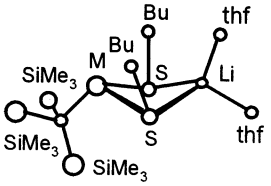


Figure 1. Molecular structure of **1** (50% probability ellipsoids).

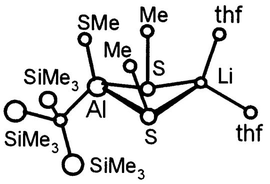
tert-butoxo,[23] aryloxo,[24] triorganosilanolato,[25,26] diorganoamido,[27–29] or diorganophosphanido[30] compounds of thegeneral type $MSn^{II}X_3$, where M = alkali metal. In the subsequent discussion the symbol Tsi denotes the "trisyl" ligand $(Me_3Si)_3C$.

Results and Discussion

In attempts to prepare compounds of the type M(Tsi)-SR (M = Ge or Sn) we treated the thiolates $M(SBu)_2$ (M = Ge or Sn) with LiTsi at room temperature in a mixture of diethyl ether and THF. The products were shown by NMR spectroscopy and X-ray diffraction studies to be the ate complexes $[M\{C(SiMe_3)_3\}(\mu\text{-}SBu)_2\text{-}Li(THF)_2]$ with M = Ge (**1**) or Sn (**2**). Details of the structures are presented in Figures 1 and 2 and Table 1.



1 M = Ge; **2** M = Sn



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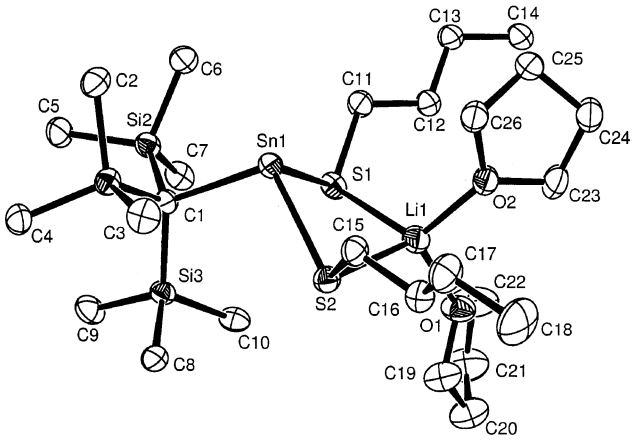

Figure 2. Molecular structure of **2** (50% probability ellipsoids).

Table 1. Selected Bond Lengths (Å) and Angles (deg) in Compounds 1 and 2

	1	2
M–C	2.096(5)	2.295(3)
M–S1	2.393(2)	2.568(1)
M–S2	2.386(2)	2.574(1)
Li–S1	2.447(9)	2.484(6),
Li–S2	2.456(9)	2.483(6)
Li–O	1.914(11)	1.925(7)
Li–O2	1.930(10)	1.918(6)
C–M–S1	101.8(2)	100.2(1)
C–M–S2	103.3(2)	102.6(1)
S1–M–S2	93.84(5)	87.79(3)
M–S1–Li	85.7(2)	81.55(14)
M–S2–Li	85.6(2)	81.43(15)
S1–Li–O2	114.8(5)	116.7(3)
S2–Li–O1	118.6(5)	115.6(3)
M–S1–C11	99.1(2)	101.9(1)
M–S2–C15	96.6(2)	99.0(1)
Li–S1–C11	108.2(3)	109.0(2)
Li–S2–C15	101.3(3)	106.0(2)
O1–Li–O2	102.7(5)	108.4(3)
S–Li–S	90.8(3)	91.7(2)

It can be seen that both compounds have butterfly structures, with almost symmetrical BuS bridges between M and Li. The Ge–S, Sn–S, and Li–S bond lengths are respectively ca. 0.13, 0.13, and 0.18 Å larger than the sums of the relevant covalent radii, suggesting that the Li–S bonds may be somewhat weaker than the Ge–S or Sn–S bonds. The Ge–S bonds in **1** are a little longer than the (nonbridge) Ge–S bonds in the ate species $[Et_4N][Ge(SPh)_3]$ [2.321(2), 2.367(2), and 2.375(2) Å][21] and fairly close to those (mean 2.39(1) Å) of the bridge bonds in the dimeric compound $^tBuSGe(\mu\text{-}SBu^t)_2\text{-}GeSBu^t$.[31] The bridge Sn–S bonds in **2** are likewise only a little longer than the (nonbridge) Sn–S bonds [2.532(1), 2.552(1), and 2.532(1) Å] in the salt $[Ph_4As][Sn(SPh)_3]$.[22] The departure of the four-membered ring from planarity is markedly larger for the tin compound **2** (sum of internal angles 342.5°, fold angle about the S····S vector 45.8(2)°) than for the germanium compound **1** (sum 355.9°, fold angle 22.3(2)°). The S–Ge–S angles are ca. 6° larger than the S–Sn–S angles and the Li–S–Ge angles are ca. 4° larger than the Li–S–Sn angles. The bonding at the Ge and Sn atoms can be considered as distorted tetrahedral with the lone pair occupying one of the sites. The geometry of the Tsi group is as expected for the group attached to Ge or Sn.[32] The Ge^{II}–Tsi bond in **1** (2.096(5) Å) is similar in length to those in the Ge^{II} compounds $Ge(Tsi)CH(SiMe_3)_2$ (2.067(4) Å)[33] or $Ge(Tsi)(\eta^5\text{-}C_5H_5)$ (2.135(17) Å)[34] and signifi-

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cantly longer than Ge^{IV}−Tsi bonds (1.94(1) −1.99(1) Å).[20,35−37] Likewise the Sn^{II}−Tsi bond in **2** (2.295(3) Å) is similar in length to the Sn−C bonds in the Sn^{II} compounds $[Sn\{C(SiMe_2Ph)_3\}Cl]_2$ (2.296(10) Å) and $[Sn\{C(SiMe_3)_2(SiMe_2OMe)\}Cl]_2$[14] (2.294(5) Å) and significantly longer than the Sn−C bonds in tris(triorganosilyl)methyl−Sn^{IV} derivatives (2.120(11) to 2.191(11) Å).[38−43]

The structures, especially that of **1**, show a striking resemblance to that of the recently reported tri(methylthiolato)aluminate **3**,[44] if the nonbridging MeS group in the latter is regarded as occupying the site taken up by the lone pair in **1** and **2**. Since the covalent radii of Ge (1.25 Å) and Al (1.22 Å) are not much different, comparison between **1** and **3** is especially interesting. In **3** the four-membered ring is a little closer to planarity (sum of angles 357.7°, fold angle 17°) than that in **1**. The M−S−Li (85.1, 85.7°) and S−Li−S (89.7°) angles are very close to those in **1**, but the S−M−S angle (98.4°) is some 5° larger than that in **1**. The Li−S bond lengths in **3** (mean 2.47(1) Å) are close to those in **1** (and **2**). There is one marked difference, however, namely that the C1−M−S angles in **1** (103.3(2), 101.8(2)°) are much smaller than those in **3** (115.1(2), 113.3(2)°), suggesting that the effective bulk of the lone pair in **1** is markedly larger than that of the terminal SMe group in **3**; that the S−M−S angle is significantly larger in **3** than in **1** may reflect the same influence.

The signal of the SCH_2 groups in the ^1H NMR spectra of **2** in THF-d_8 appears as a sharp triplet with $^3J_{HH}$ = 7.3 Hz, indicating that the two protons are equivalent. This implies (a) that the LiS_2Sn ring found in the solid state is broken in solution, probably by transient opening at Li−S bonds and coordination of extra THF at lithium, and (b) that effects of chirality at tin cannot be detected at the CH_2 group. The signals of the SCH_2 groups from samples of **1** and **2** in the less strongly coordinating toluene-d_8, however, appear as the AB part of an ABX_2-spin system. As the temperature is raised from −40 to +50 °C the chemical shift difference $\Delta\nu_{AB}$ for **2** decreases but the line width remains quite narrow, indicating that chemical exchange between the two sites within a CH_2 group is slow on the NMR time scale; the changes are reversed as the sample is cooled. The ^{119}Sn signal from **2** becomes broader at high temperatures ($\Delta\nu_{1/2}$ = 55 Hz at −40 °C and 140 Hz at +50 °C) but the chemical shift remains in the region expected for three-coordinate tin. These results are most simply explained by assuming that the predominant species in solution adopt the puckered ring structures found in the solid, in which the sulfur atoms are chiral. Reactions of **1** and **2** are under investigation.

Table 2. Crystallographic Data[a] for 1 and 2

	1	2
formula	$C_{26}H_{61}GeLiO_2S_2Si_3$	$C_{26}H_{61}LiO_2S_2Si_3Sn$
fw	633.67	679.77
temp/K	100(2)	100(2)
cryst syst	monoclinic	monoclinic
space group	$P2_1/c$	$P2_1/n$
a/Å	14.3450(16)	12.2299(7)
b/Å	18.1049(19)	16.9483(9)
c/Å	13.9628(16)	18.2285(10)
β/deg	90.452(3)	104.979(1)
Z	4	4
μ/mm^{-1}	1.079	0.932
measured/indep. reflns/R_{int}	28753/10430/0.098	26129/8759/0.030
reflns with $I > 2\sigma(I)$	4459	6349
R1; wR2 ($I > 2\sigma(I)$)	0.080, 0.137	0.052, 0.070
R1; wR2 (all data)	0.109, 0.179	0.123, 0.131
abs cor T_{min}; T_{max}	0.694/1.00	0.738/1.00

[a] SMART and Saint Release 5.0. Area detector control and integration software. Bruker AXS Analytical X-ray Instruments, Madison, WI, 1998.

Experimental Section

Air and moisture were excluded as much as possible from all reactions by use of Schlenk techniques with Ar as blanket gas. Solvents were dried by standard procedures and freshly distilled before use. NMR spectra were recorded at 360.1 (^1H), 90.5 (^{13}C), 99.4 (^{29}Si), 134.3 (^{119}Sn), and 194.5 MHz (^7Li). Chemical shifts are relative to $SiMe_4$ for H, C, and Si, $SnMe_4$, and LiCl in D_2O. The assignment of the ^{13}C signals was confirmed by INEPT and DEPT spectra. The compositions of **1** and **2** were confirmed by integration of ^1H NMR spectra, but analytical data for **1** suggested that one THF molecule had been lost during preparation of samples; **2** was extremely reactive and good analytical data could not be obtained.

$[Ge\{C(SiMe_3)_3\}(\mu\text{-}SBu)_2Li(THF)_2]$ (1). A solution of LiTsi, made from TsiH (3.68 g, 15.9 mmol) in THF (33 mL) and 5.7 mL of 2.8 M MeLi in Et_2O,[45,46] was added at room temperature to a stirred solution of $Ge(SBu)_2$ (3.98 g, 15.9 mmol) in THF (20 mL). The mixture was stirred for 1 h at room temperature and the solvents then removed to leave a dark red oil, which was extracted with pentane (50 mL). The filtered extract was reduced in volume to 30 mL and kept at −12 °C to give pale yellow crystals of **1** (4.0 g, 40%), mp 80−81 °C dec. Anal. Calcd for $C_{22}H_{53}LiOS_2Si_3Ge$ (**1**−THF**)**: C, 47.0; H, 9.5. Found: C, 47.1; H, 9.9. ^1H NMR (C_6D_6, 297 K): δ 0.71 (s, 27H, $SiMe_3$, $^2J_{SiH}$ = 6.0 Hz), 1.00 (t, 6H, CH_3, $^3J_{HH}$ = 7.3 Hz), 1.41−1.59 (m, 12H, CH_2 and THF), 1.64−1.85 (m, 4H, CH_2), 2.73−2.96 (4H, AB-part of ABX_2 spin system, $^2J_{AB}$ = 12 Hz; $^3J_{AX}$ = $^3J_{BX}$ = 7.4 Hz, $\Delta\nu_{AB}$ = 54.4 Hz, SCH_2), 3.62 (m, 8H, OCH_2, THF). $^{13}C\{^1H\}$ NMR (C_6D_6): δ 5.9 (Me_3Si, $^1J_{SiC}$ = 50.2 Hz), 14.2 (CH_3), 14.3 (CSi_3 $^1J_{SiC}$ = 35.4 Hz), 22.9 (CH_2), 25.3 (CH_2, THF), 33.0 (CH_2); 36.7 (SCH_2); 68.4 (OCH_2, THF). ^7Li NMR: δ 0.78. ^{29}Si NMR: δ −3.2.

$[Sn\{C(SiMe_3)_3\}(\mu\text{-}SBu)_2Li(THF)_2]$ (2). The procedure described above was used but starting from $Sn(SBu)_2$ (3.45 g, 11.6 mmol) in THF (22 mL) and a solution of LiTsi, made from TsiH (3.24 g, 14.0 mmol) in 17 mL of THF and 5 mL of 2.8 M MeLi in Et_2O. The pale yellow oil formed initially was

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extracted with pentane (20 mL), and the filtered extract was reduced in volume to 10 mL and kept at −12 °C to give pale yellow crystals of **2** (4.77 g, 60%), mp 45−47 °C (darkens and softens at 34 °C). ^{1}H NMR: δ ($C_6D_5CD_3$, 297 K) 0.44 (s, 27H, $SiMe_3$), 0.87 (t, 6H, CH_3, $^3J_{HH}$ = 7.4 Hz), 1.31−1.44 (m, 4H, CH_2), 1.49 (m, 8H, CH_2 and THF), 1.53−1.67 (m, 4H, CH_2), 2.55−2.72 (m, br, AB part of ABX_2-spin system, $^2J_{AB}$ = 12 Hz; $^3J_{AX}$ = $^3J_{BX}$ = 7.5; $\Delta\nu_{AB}$ = 21.5 Hz, 4H SCH_2), 3.56 (m, 8H, OCH_2, THF). δ (THF-d_8): 0.35 (s, 27H, $SiMe_3$), 1.02 (t, 6H, CH_3, $^3J_{HH}$ = 7.3 Hz), 1.51−1.64 (m, 4H, CH_2), 1.66−1.82 (m, 4H, CH_2), 2.71 (t, br, 4H, SCH_2, $^3J_{HH}$ 7.3 Hz). NMR: δ 6.3 (Me_3Si, $^1J_{CSi}$ = 50.0 Hz), 14.1 (CH_3), 17.7 (CSi_3), 22.6 (CH_2), 25.4 (THF), 31.1 (br, CH_2), 37.6 (br, CH_2S), 68.5 (OCH_2, THF). ^{29}Si NMR: δ −3.1 ($^1J_{SiC}$ = 50.0 Hz; $^2J_{SiSn}$ = 43.5 Hz). ^{119}Sn NMR ($C_6D_5CD_3$): δ 275.3. ^{7}Li NMR: δ 0.93.

X-ray Structure Determinations. Data were collected on a SMART CCD 1000 diffractometer and corrected for Lorentz and polarization effects and for absorption. For details see Table 2. The structures were determined by direct methods and by full-matrix least-squares refinement with anisotropic thermal parameters for non-hydrogen atoms.[47] The carbon atoms C(20) and C(25) of the tetrahydrofuran molecules in **1** and C(20) in **2** were disordered over two sites, with occupancies 0.7 and 0.3, 0.6 and 0.4, and 0.7 and 0.3, respectively. The hydrogen atoms were placed in calculated positions and refined in the riding model with fixed thermal parameters. All calculations were carried out by use of the SHELXTL PLUS program (PC Version 5.0).

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Supporting Information Available: Crystallographic data for **1** and **2**. This material is available free of charge via the Internet at http://pubs.acs.org. These data also have been deposited with the Cambridge Crystallographic Data Center, CCDC nos. 180388 and 180389, respectively, and may be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax +44 1223 336033; e-mail deposit@ccdc.cam.ac.uk or www.ccdc.cam.ac.uk).

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