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Synthesis and Characterization of R_2BiCl and $RBiCl_2$ [R = $CH(SiMe_3)_2$]

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Reaction of RLi [R = $CH(SiMe_3)_2$] with $BiCl_3$ in a 2:1 ratio gives R_2BiCl (**1**). $RBiPh_2$ is formed by the action of RMgCl on Ph_2BiCl. Reaction of $RBiPh_2$ with HCl in $CHCl_3$ gives $RBiCl_2$ (**2**), which crystallizes from diethyl ether as the solvate **2**·$0.5Et_2O$. The crystal structures of **1** and **2**·$0.5Et_2O$ have been determined by X-ray diffraction. Crystals of **1** consist of pyramidal R_2BiCl molecules, whereas in crystals of **2**·$0.5Et_2O$ there are ${}^1_\infty\{[RBi(Cl)Cl]_2\cdot Et_2O\}_x$ chains with chloro bridges. The diethyl ether molecules are weakly associated to neighboring bismuth atoms in the chain.

Introduction

Although some dialkylbismuth halides and alkylbismuth dihalides have a long history,[1] the knowledge of this class of compounds is rather limited. So far only one crystal structure of a dialkylbismuth halide[2] and no crystal structural data of alkylbismuth dichlorides have been reported. However, the structures of several alkylbismuth diiodides[3,4] as well as arylbismuth dihalides (including solvates) are known. Here we report the efficient syntheses of R_2BiCl (**1**) and $RBiCl_2$ (**2**)[5] [R = $CH(SiMe_3)_2$] and their crystal structures. An alternate synthesis reported for **2** used the reaction of RMgCl with $BiCl_3$ in 1:1 molar ratio. We found, however, that this method as described[5] yielded exclusively **1**. The formation of **1** and **2** as products of scrambling reactions of $BiCl_3$ and $[(Me_3Si)_2CH]_3Bi$ has been reported, but without details.[6] The crystal structure of the related trialkylbismuthine, R_3Bi, was described some years ago.[6] **1** and **2** are potential synthons in organobismuth chemistry. **2** has recently been used as the starting material for the synthesis of the first organobismuth rings, $[RBi]_n$ ($n = 3, 4$).[7]

Results and Discussion

The synthesis of $[(Me_3Si)_2CH]_2BiCl$ (**1**) was achieved in 93% yield by the reaction of RLi with $BiCl_3$ in 2:1 molar ratio in diethyl ether (eq 1).

$$2\,RLi + BiCl_3 \xrightarrow{Et_2O} \underset{\mathbf{1}}{R_2BiCl} + 2\,LiCl \qquad (1)$$

R = $(Me_3Si)_2CH$

1 is a yellow crystalline compound that is readily soluble in aliphatic and aromatic hydrocarbons and other organic solvents. The compound decomposes in air, but is stable in an inert atmosphere at low temperature. It melts at 71−73 °C and can be sublimed under reduced pressure without decomposition.

For the synthesis of $RBiCl_2$ (**2**) two steps are necessary (eq 2, 3). First, diphenylbismuth chloride was allowed to react with the Grignard reagent RMgCl to give $RBiPh_2$. Action of HCl on this intermediate yielded benzene and **2** with an overall yield of 77%.

$$Ph_2BiCl + RMgCl \xrightarrow{THF} RBiPh_2 + MgCl_2 \qquad (2)$$

$$RBiPh_2 + 2\,HCl \xrightarrow{CHCl_3} \underset{\mathbf{2}}{RBiCl_2} + 2\,C_6H_6 \qquad (3)$$

R = $(Me_3Si)_2CH$

In contrast to **1**, the dihalide **2** is insoluble in petroleum ether, but readily soluble in donor solvents such as tetrahydrofuran and diethyl ether. In benzene or chloroform **2** is slightly soluble. Crystallization from diethyl ether gave the adduct $(Me_3Si)_2CHBiCl_2\cdot 0.5Et_2O$. The latter is stable in the air for short periods of time, but solutions are hydrolyzed when exposed to the atmosphere. At low temperature in an inert atmosphere the adduct could be stored for a long time. Both halides **1** and **2**, as well as the intermediate, $(Me_3Si)_2CHBiPh_2$, have been characterized by ^1H and ^{13}C NMR spectroscopy. Characteristic mass spectra have been obtained by chemical ionization and electron impact techniques. The composition of the compounds has been proved by high-resolution mass spectrometry. Due to the low stability of **1** and **2** at room temperature, no satisfactory elemental analyses of these compounds could be obtained.

To establish the solid-state structure of **1** and **2**, X-ray diffraction studies (see Table 1 for details) have been carried out on single crystals obtained by cooling concentrated solutions of **1** in light petroleum and of **2** in diethyl ether. The structure of **1** (Figure 1) consists

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Table 1. Crystal Data, Data Collection, and Structure Refinement Parameters for 1 and 2

	1	**2**
formula	$C_{14}H_{38}BiClSi_4$	$C_9H_{24}BiCl_2O_{0.5}Si_2$
fw	563.23	476.34
color	lemon yellow	yellow
cryst size, mm	0.6 × 0.4 × 0.3	0.6 × 0.4 × 0.3
cryst syst	monoclinic	orthorhombic
space group	$P2_1/n$	$P2_1nb$
a, Å	1.2025(4)	8.9400(2)
b, Å	1.2393(3)	16.643(3)
c, Å	1.7382(4)	22.891(3)
a, deg	90	90
b, deg	108.88(2)	90
g, deg	90	90
V, Å^3	2451.0(12)	3405.9(11)
Z	4	8
d_{calcd}, g·cm^{-3}	1.526	1.858
μ(Mo Kα), mm^{-1}	7.491	10.784
scan method	$\omega/2\theta$	$\omega/2\theta$
F(000)	1112	1816
scan range (deg)	$2.97 \leq \theta \leq 27.51$	$2.6 \leq \theta \leq 27.5$
no. of measd data	6282	4664
no. of unique data	5039 ($R_{int} = 0.0356$)	4109 ($R_{int} = 0.0414$)
no. of params	196	266
no. of data with $I > 2\sigma(I)$	3812	4109
abs corr	DIFABS[17]	ψ-scans
wR2[a] (all data)	0.1035	0.0984
R1[a] (all data)	0.0680	0.0619
wR2[a] ($I > 2\sigma(I)$)	0.0941	0.0907
R1[a] ($I > 2\sigma(I)$)	0.0440	0.0437
GOF on F^2	1.022	1.036
residual density, e·Å^{-3}	+2.315; −1.153	+0.938; −0.847

[a] Definition of the R values: $R1 = (\sum||F_o| - |F_c||)/\sum|F_o|$; $wR2 = \{\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]\}^{1/2}$ with $w^{-1} = \sigma^2(F_o^2) + (aP)^2 + bP$.

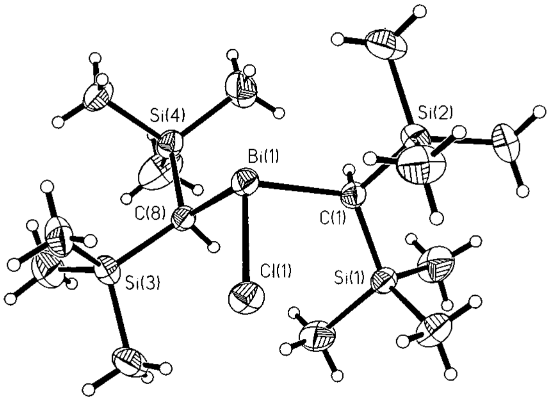

Figure 1. ORTEP-like representation at 50% probability of the molecular geometry and atom-numbering scheme for compound **1**. Important bond lengths (Å) and angles (deg) are as follows: Bi(1)−C(1) 2.273(7), Bi(1)−C(8) 2.277(7), Bi(1)−Cl(1) 2.530(2), C(1)−Bi(1)-Cl(1) 95.77(18); C(1)−Bi(1)−C(8) 100.7(2), C(8)−Bi(1)−Cl(1) 100.75(18), Bi(1)−C(1)−Si(1) 120.6(3), Bi(1)−C(1)−Si(2) 105.8(3), Bi(1)−C(8)−Si(3) 112.5(3), Bi(1)−C(8)−Si(4) 106.6(3), Si(4)−C(8)−Si(3) 117.0(4), Si(1)−C(1)−Si(2) 117.3(4).

of dialkylbismuth chloride molecules with a pyramidal environment around the bismuth center. The Bi−C bond lengths are 2.273(7) and 2.277(7) Å; the C−Bi−C angle is 100.7(2)°. These values are a little smaller than the corresponding values in R_3Bi (average C−Bi−C angle, 103°, av Bi−C distance 2.32 Å). The Bi−Cl distance in **1** is 2.530(2) Å. A slightly shorter Bi−Cl bond length (2.463 Å) has been reported for $[2,4,6\text{-}(CF_3)_3C_6H_2]_2BiCl$,[8] the only other monomeric diorganobismuth halide reported in the literature. Diorganobismuth halides with less bulky substitutents display considerable intermolecular interactions.[9,10] Diphenylbismuth chloride,[10] for example, is a coordination polymer with bridging chlorine atoms and a pseudo trigonal bipyramidal coordination at bismuth. As a consequence of the association, the Bi−Cl bond lengths in Ph_2BiCl are much longer (average 2.75 Å) than in **1**. Another interesting aspect of the structure of **1** is the dissimilarity of the bond angles: ∠C−Bi−Cl = 95.77(18)° and 100.75(18)°. This difference may be a consequence of the steric strain in the molecule, caused by the bulky $(Me_3Si)_2CH$ substituents. Also the Si−C−Bi bond angles of the alkyl substituents are dissimilar. The values for the two pairs of angles are 120.6(3)°, 105.8(3)° and 112.5(3)°, 106.6(3)°. Similar distortions have also been observed in the structure of $[(Me_3Si)_2CH]_3Bi$. For a detailed discussion see ref 6.

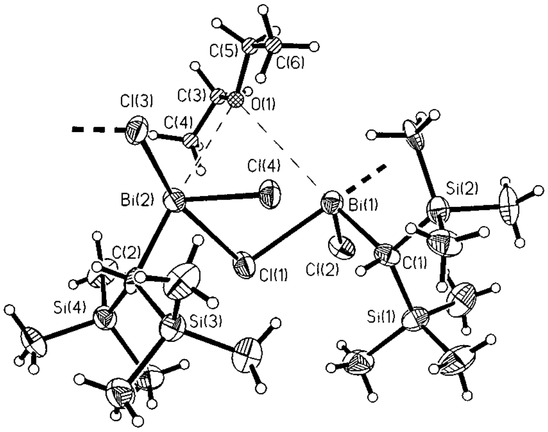

Figure 2. ORTEP-like representation at 50% probability of the molecular geometry and atom-numbering scheme for compound **2** (ball-and-stick representation of the ether molecule). Important bond lengths (Å) and angles (deg) are as follows: Bi(1)−C(1) 2.237(14), Bi(1)−Cl(1) 2.704(5), Bi(1)−Cl(2) 2.538(5), Bi(1)−Cl(3a) 2.824(4), Bi(1)···O(1) 3.557, Bi(2)−C(2) 2.244(14), Bi(2)−Cl(1) 2.852(4), Bi(2)−Cl(3) 2.729(4), Bi(2)−Cl(4) 2.522(4), Bi(2)···O(1) 3.431; C(1)−Bi(1)−Cl(1) 95.8(4), C(1)−Bi(1)−Cl(2) 99.7(2), C(1)−Bi(1)−Cl(3a) 95.4(4), Cl(2)−Bi(1)−Cl(1) 82.88(14), Cl(2)−Bi(1)−Cl(3a) 79.85(13), Cl(1)−Bi(1)−Cl(3a) 160.75(16), Bi(1)−Cl(1)−Bi(2) 98.88(16), C(2)−Bi(2)−Cl(1) 88.2(4), C(2)−Bi(2)−Cl(3) 94.8(4), C(2)−Bi(2)−Cl(4) 103.1(4), Cl(4)−Bi(2)−Cl(3) 81.50(13), Cl(1)−Bi(2)−Cl(3) 159.45(15), Bi(1)−C(1)−Si(1) 118.9(8), Bi(1)−C(1)−Si(2) 110.1(7), Bi(2)−C(2)−Si(3) 119.0(7), Bi(2)−C(2)−Si(4) 108.2(8).

The structure of $\mathbf{2}\cdot 0.5Et_2O$ is depicted in Figure 2. The asymmetric unit consists of $(Me_3Si)_2CHBiCl_2$ dimers and a diethyl ether molecule. In the crystal the $(Me_3Si)_2CHBiCl_2$ units are associated through single chlorine bridges between bismuth atoms to form a coordination polymer, ${}^{1}_{\infty}\{[(Me_3Si)_2CHBi(Cl)Cl]_2\cdot Et_2O\}_x$. With Cl−Bi−Cl bond angles involving bridging chlorine atoms (av 160°) not very far from linearity and Bi−Cl−Bi angles of 109.05(17)° and 98.88(16)° a zigzag chain results. The coordination around the bismuth atoms may be described as pseudo trigonal bipyramidal with the bridging chlorine ligands in axial positions and the alkyl groups and the terminal chlorine atoms in equato-

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rial positions. The diethyl ether molecules are coordinated as additional bridges between two neighboring bismuth centers in the $(Bi-Cl)_x$ chain. The Bi···O distances (3.431, 3.557 Å) are much closer to the sum of the van der Waals radii (3.9 Å) than to the sum of the covalent radii (2.16 Å) of bismuth and oxygen, indicating a loose attachment of the solvate. It should be noted that the smaller Bi−Cl−Bi angle in the polymeric chain corresponds to the bismuth atoms that are bridged by an ether molecule. The values for the Bi−C bond lengths increase from $RBiCl_2$ (2.24 Å), to R_2BiCl (2.28 Å), to R_3Bi (2.32 Å) because steric factors become more important. The average bond length between the bismuth atom and the nonbridging chlorine atom in **2**·$0.5Et_2O$ is 2.53 Å, practically identical with the Bi−Cl bond length in **1** (2.530(2) Å). In the polymeric chain shorter and longer Bi−Cl bonds (av 2.73, 2.84 Å) alternate. Therefore, even though the asymmetric unit is dimeric, it is possible to define $(Me_3Si)_2CHBiCl_2$ entities in the supramolecular chain.

Comparison of these compounds with other organobismuth dihalides, including solvates, reveals the structure of **2**·$0.5Et_2O$ to be novel. As a consequence of the various Lewis acidic and basic sites in the molecule, structural diversity is typical for this class of compounds. In fact, among the structures known so far, very few are analogues. However, a common feature is that no monomeric species are known in the solid state. There are either dimers or polymers with single or double halogen bridges. Single bridges as in **2**·$0.5Et_2O$ exist in $MesBiBr_2$[11] and $PhBiX_2$·THF (X = Cl, Br, I).[12,13] In the phenylbismuth dihalide solvates the tetrahydrofuran molecules act as terminal ligands at each bismuth center with Bi−O bond lengths of 2.671 Å, much shorter than the corresponding values in **2**·$0.5Et_2O$, where the ether molecules are bridging. It is also of interest to compare the structure of **2**·$0.5Et_2O$ with the antimony analogue of **2**. In the crystals of $(Me_3Si)_2CHSbCl_2$ even in the absence of donor solvents the alkylantimony dichloride molecules are only weakly associated to dimers.[14] Following the usual trend, the coordination number increases from antimony to bismuth, as does the degree of association.

Experimental Section

General Comments. The reactions and manipulations ware performed under an inert atmosphere of dry argon. Chemical shifts are reported in δ units (ppm) referenced to C_6D_5H (7.15 ppm, ^1H) and C_6D_6 (128.0 ppm, ^{13}C). The C,H correlation was performed with use of HSQC spectra. The superscripts a and b are related to the diastereotopic methyl groups in **1**.

Bis[bis(trimethylsilyl)methyl]bismuth Chloride (1). A solution of RCl (5.48 g, 28.12 mmol) in diethyl ether (30 mL) was added dropwise with stirring to 1.14 g (164 mmol) of Li wire (0.8 mm diameter) in refluxing diethyl ether (25 mL). The mixture was heated at reflux for 36 h, when the lithium partially dissolved and a fine white precipitate was formed. The reaction mixture was allowed to stand for several hours in order to allow the precipitate to settle. The milky solution was separated from the solid, transferred into a dropping funnel, and added dropwise with vigorous stirring and cooling to −40 °C to a solution of $BiCl_3$ (4.22 g, 13.38 mmol) in Et_2O (100 mL). Stirring was continued for 3 h at −40 °C and 12 h at ambient temperature. The solvent was removed at reduced pressure, and the residue was extracted with 2 × 250 mL of petroleum ether. The combined petroleum ether solutions were reduced to dryness in vacuo to give 7.00 g (93%) of **1** as lemon yellow crystals (mp: 71−73 °C) of sufficient purity for most purposes. Further purification was achieved by sublimation at 85 °C/5 × 10^{-3} mbar.

HRMS (EI, 70 eV): 562.1550 (calcd 562.1543 amu, $C_{14}H_{38}Bi^{35}ClSi_4$, M^+), 547.1311 (calcd 547.1309 amu, $C_{13}H_{35}Bi^{35}ClSi_4$, $M^+ - CH_3$). ^1H NMR (C_6D_6, 200 MHz, 23 °C): 0.09 (s, 9 H, CH_3^a, $^2J_{SiH} = 6.0$ Hz), 0.35 (s, 9 H, CH_3^b, $^2J_{SiH} = 6.1$ Hz), 1.30 (s, 1 H, CH, $^2J_{SiH} = 7.7$ Hz). ^{13}C NMR (C_6D_6, 50 MHz, 23 °C): 3.38 (CH_3^a), 5.44 (CH_3^b), 39.90 (CH). MS (EI, 70 eV): 562 (4) [M^+], 547 (16) [$M^+ - CH_4$], 403 (74) [$M^+ - R$], 129 (100), 73 (70). MS (CI^+, NH_3): 597 (22) [$M^+ + Cl$], 580 (100) [$M^+ + NH_4$], 562 (4) [M^+], 544 (55) [$M^+ - Cl + NH_3$], 527 (84) [$M^+ - Cl$], 420 (44) [$M^+ - R + NH_3$], 403 (75) [$RBiCl^+$], 146 (42) [$(Me_3Si)_2^+$]. MS (CI−, NH_3): 597 (35) [$M^- + Cl$], 562 (100) [M^-], 527 (12) [$M^- - Cl$], 403 (15) [$RBiCl^-$], 368 (20) [RBi^-].

Bis(trimethylsilyl)methylbismuth Dichloride (2). A solution of RCl (4.75 g, 24.38 mmol) in 70 mL of tetrahydrofuran (THF) was added dropwise with stirring during 1 h to magnesium filings (0.79 g, 32.50 mmol, activated with 0.5 mL of 1,2-dibromoethane) in 10 mL of THF. Refluxing the mixture for 2 h and stirring at ambient temperature for a further 15 h gave a Grignard solution. This was decanted from the unreacted magnesium and added dropwise with cooling in an ice bath over 1.5 h to a slurry of Ph_2BiCl (8.84 g, 22.18 mmol) in THF (120 mL). The mixture was stirred for another 2 h at 0 °C and 15 h at ambient temperature. A yellow solution containing a faintly yellow flaky solid formed. After evaporation of the solvent the residue was extracted twice with 280 mL of petroleum ether. Combining the solutions and evaporation of the solvent gave 9.78 g (84%) of the intermediate $RBiPh_2$ as a yellow oil, which solidified at ambient temperature. The crude product contained small amounts of Ph_2BiCl and RH and was used without purification for the synthesis of $RBiCl_2$.

^1H NMR (C_6D_6, 200 MHz, 23 °C): 0.07 (s, 18 H, CH_3, $^2J_{SiH} = 6.4$ Hz), 1.12 (s, 1 H, CH, $^2J_{SiH} = 7.7$ Hz), 7.03−7.25 (m, 6 H, m- + p-C_6H_5), 7.83 (dd, 4 H, o-C_6H_5, $^3J_{HH} = 7.9$ Hz, $^4J_{HH} = 1.4$ Hz). MS (EI, 70 eV): 522 (4) [M^+], 507 (15) [$M^+ - Me$], 492 (15) [$M^+ - 2\ Me$], 445 (15) [$RBiPh^+$], 430 (19) [$RBiPh^+ - Me$], 368 (100) [RBi^+], 363 (83) [Ph_2Bi^+], 286 (22) [$PhBi^+$], 209 (47) [Bi^+], 73 (26) [Me_3Si^+]. MS (CI+, NH_3): 507 (65) [$M^+ - Me$], 463 (95) [$M^+ - Ph + NH_4$], 445 (100) [$M^+ - Ph$], 380 (78) [$Ph_2Bi^+ + NH_4$], 303 (6) [$PhBi^+ + NH_4$], 209 (9) [Bi^+]. MS (CI−, NH_3): 445 (58) [$M^- - Ph$], 363 (20) [Ph_2Bi^-], 286 (5) [$PhBi^-$], 159 (100) [R^-].

A gentle flow of dry HCl was introduced for 2 h into a solution of $RBiPh_2$ (9.78 g, 18.71 mmol) in $CHCl_3$ (100 mL). The solvent was removed from the resulting clear yellow solution at reduced pressure, and the residue was extracted with 200 mL of diethyl ether. Reduction of the volume of this solution to 20 mL resulted in the crystallization of 8.18 g (92%) of **2**·$0.5Et_2O$ as yellow needles (mp: 132−134 °C).

HRMS (EI, 70 eV): 422.9972 (calcd 422.9972 amu, $C_6H_{16}Bi^{35}Cl_2Si_2$, $M^+ - CH_3$). ^1H NMR (C_6D_6, 200 MHz, 23 °C): −0.23 (s, 1 H, Si_2CH, $^2J_{SiH} = 7.8$ Hz), 0.19 (s, 18 H, $SiCH_3$, $^2J_{SiH} = 6.5$ Hz), 1.08 (t, 3 H, CH_2CH_3, $^3J_{HH} = 7.0$ Hz), 3.25 (q, 2 H, CH_2CH_3, $^3J_{HH} = 7.0$ Hz). ^{13}C NMR (C_6D_6, 200 MHz, 23 °C): 4.71 ($SiCH_3$, $^1J_{SiC} = 51.7$ Hz), 15.47 (CCH_3), 65.83 (CH_2), 69.69 (Si_2CH). MS (EI, 70 eV): 423 (100) [$M^+ - Me$], 408 (63) [$M^+ - 2\ Me$], 403 (10) [$M^+ - Cl$], 388 (6) [$M^+ - Cl - Me$], 279 (5)

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[$BiCl_2^+$], 244 (4) [$BiCl^+$], 209 (30) [Bi^+], 129 (81) [$C(SiMe_2)_2^+$], 85 (23) [$CSiMe_3^+$], 73 (55) [$SiMe_3^+$]. MS (CI+, NH_3): 456 (100) [$M + NH_4^+$], 439 (60) [$M^+ + H$], 420 (60) [$M - Cl + NH_3^+$], 400 (20). MS (CI−, NH_3): 475 (100) [$M^- + Cl$], 438 (14) [M^-], 279 (80) [$BiCl_2^-$].

X-ray Crystallography. The details of the crystal structure determination and refinement are given in the Table 1. Data were collected on a Siemens P4 four-circle diffractometer using graphite-monochromated Mo Kα radiation ($\lambda = 0.710\ 73$ Å). For this purpose the crystals were attached with Kel-F-oil to a glass fiber and cooled under a nitrogen stream to 173 K.

The structures were solved, after Lp correction, by direct methods. All non-hydrogen atoms were refined with anisotropic thermal parameters. The hydrogen atoms were refined with a riding model and a mutual isotropic thermal parameter. For structure solving and refinement the software package SHELX-97 was used.[15] The drawings were created with the XP Program of the SHELXTL program system.[16]

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Supporting Information Available: ^{1}H NMR spectra, tables of crystal data and structure refinement details, atom coordinates and *U* values, bond distances and angles, anisotropic thermal parameters, and dihedral angles (21 pages). See any current masthead page for ordering information and Internet access instructions.

OM9806444

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