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(Tris(trimethylsilyl)germyl)plumbanes: Crystal Structure of Diphenylbis(tris(trimethylsilyl)germyl)plumbane

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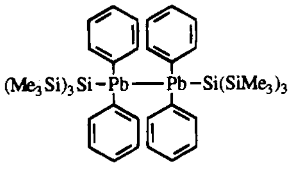
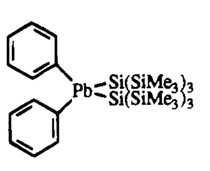
Reaction of $(C_6H_5)_3PbCl$ with 1 equiv of $(THF)_{2.5}LiGe(SiMe_3)_3$ in ether afforded $(C_6H_5)_3PbGe(SiMe_3)_3$ in 25% yield. An attempt to prepare $(C_6H_5)_2Pb(Ge(SiMe_3)_3)_2$ by the reaction of 2 equiv of $(THF)_{2.5}LiGe(SiMe_3)_3$ with Ph_2PbCl_2 gave a mixture of the expected product as a yellow solid and a red-orange solid identified as the diplumbane, $[(C_6H_5)_2Pb(Ge(SiMe_3)_3]_2$. The mono- and diplumbanes were separated by fractional crystallization from pentane and characterized by their elemental analyses, NMR spectra (^1H and ^{13}C), and infrared and UV–visible spectra. An X-ray structure determination on $[(C_6H_5)_2Pb(Ge(SiMe_3)_3]_2$ was not completed due to decomposition of the crystal but showed it to be isomorphous with $[(C_6H_5)_2Pb(Si(SiMe_3)_3]_2$, which is known to have a Pb–Pb bond. A low temperature X-ray structure determination on $(C_6H_5)_2Pb(Ge(SiMe_3)_3)_2$ revealed it to be orthorhombic, *Pnn*2, with $a = 13.999(5)$ Å, $b = 17.443(5)$ Å, $c = 10.404(2)$, $V = 2541$ Å^3, and $Z = 2$. The molecular structure exhibited marked distortion of the coordination geometry around Pb with $\angle$Ge–Pb–Ge = 135.0(1)° and $\angle$C–Pb–C = 99.8(7)°. NMR studies of the two products found that the monoplumbane was stable for 20 h in ether but that the diplumbane experienced marked decomposition during the same period.

Investigating the effects of bulky $-E(SiMe_3)_3$ (E = Si, Ge) substituents on the stability of group 14 derivatives, we reported that the products of the reaction of $(C_6H_5)_2PbCl_2$ with two equivalents of $(THF)_3LiSi(SiMe_3)_3$ included the diplumbane (**1**), which was structurally characterized, and the disubstituted, monoplumbane (**2**), which was not.[1] The diplumbane product was thought to have been formed from the intermediate $(Me_3Si)_3Si(C_6H_5)_2PbCl$ rather than from the decomposition of **2** which was found to be stable in solution under the reaction conditions. Since **2** could not be crystallized, a force field calculation was carried out to help assess the amount of steric crowding around the Pb atom.[2] The structure thus predicted exhibited a Si–Pb–Si angle of 134° indicative of significant crowding, even given the large radius of the metal atom. When $(C_6H_5)_2PbCl_2$ was treated with 2 equiv of $LiC(SiMe_3)_3$, the product was an unsymmetrical diplumbane, $(C_6H_5)_3PbPb(C_6H_5)_2C(SiMe_3)_3$.[2] Formation of this unexpected structure evidently involved a different mechanism that included phenyl group migration.



(1)



(2)

Seeking to compare the behavior induced by $-Ge(SiMe_3)_3$ substituents to that of their silyl and methyl counterparts, we carried out the reactions of $(C_6H_5)_3PbCl$ and $(C_6H_5)_2PbCl_2$ with 1 and 2 equiv, respectively, of $(THF)_{2.5}LiGe(SiMe_3)_3$.[3]

Experimental Section

Materials and Procedures. Diphenyllead dichloride and triphenyllead chloride were obtained from Johnson-Matthey and Organometallics Inc., respectively. The lithium germyl reagent, $(THF)_{2.5}LiGe(SiMe_3)_3$, was prepared according to the literature method[3] using low-halide methyl lithium (Aldrich). All syntheses were conducted using Schlenk techniques.[4] Other manipulations, analyses and characterizations were carried out as described earlier.[1] Force field calculations (MM2) were done on a CAChe system.

(Tris(trimethylsilyl)germyl)triphenylplumbane, $(Me_3Si)_3GePb(C_6H_5)_3$ (3). To 50 mL of a cold (−78 °C) ether suspension of $(C_6H_5)_3PbCl$ (1886 mg, 3.97 mmol) was slowly added with stirring 50 mL of an ether solution of $(THF)_{2.5}LiGe(SiMe_3)_3$ (1905 mg, 3.97 mmol). The mixture was stirred at −78 °C for 6 h and then allowed to warm to ambient temperature where stirring was continued another 14 h. Ether was removed *in vacuo*, the residue was stirred with 40 mL of pentane and the solid was separated by filtration. The colorless filtrate was concentrated to about 15 mL and held at 4 °C affording colorless, transparent rectangular crystals (735 mg, 25.3% isolated yield) after 4 d. Mp: 120 °C. Anal. Calcd for $C_{27}H_{42}Si_3GePb$: C, 44.38; H, 5.79. Found: C, 44.29; H, 5.75. NMR (C_6D_6): ^1H, δ0.294 (s, 27H, $Ge(SiMe_3)_3$), 7.08–7.27 and 7.7–7.9 (mult, 15H, $Pb(C_6H_5)_3$); $^{13}C\{^1H\}$, δ3.74 ($SiMe_3$), 129.5, 138.3, 152.9 (C_6H_5). IR (Nujol, cm^{-1}): 3050 sh, 2655 sh, 1795 w, 1730 w, 1560 w, 1435 m, 1310 w, 1255 s, 1200 vw, 1175 vw, 1100 sh, 1080 sh, 1030 m, 1020 sh, 975 s, 855s, 785 w, 725 m, 651 m, 645 sh, 570 w, 500 w, 475 m, 445 m, 375 m.

1,1,2,2-Tetraphenyl-1,2-bis((trimethylsilyl)germyl)diplumbane, $[(Me_3Si)_3GePb(C_6H_5)_2]_2$ (4). To 100 mL of a cold (−78 °C) ether suspension of $(C_6H_5)_2PbCl_2$ (1898 mg, 4.39 mmol) was added dropwise with stirring 150 mL of an ether solution of $(THF)_{2.5}LiGe(SiMe_3)_3$ (4209 mg, 8.78 mmol). The mixture was stirred at −78 °C for 6 h and then allowed to warm to ambient temperature where stirring was continued another 14 h. Ether was removed *in vacuo*, the residue was stirred with 80 mL of pentane and the solid was separated by filtration. The red filtrate was concentrated to about 25 mL and held at 4 °C for 2 h affording red-orange crystals (979 mg, 34.1% isolated yield). Mp: 120 °C dec. Anal. Calcd for $C_{42}H_{74}Si_6Ge_2Pb_2$: C, 38.59; H, 5.71. Found: C, 37.85; H, 5.60. NMR (C_6D_6): ^1H, δ0.155 (s, 54H, $Ge(SiMe_3)_3$), 7.13–7.35 and 8.06–8.16 (mult, 20H, $Pb(C_6H_5)_2$); $^{13}C\{^1H\}$, δ3.89 ($SiMe_3$), 129.5, 139.7, 156.0 (C_6H_5). UV–vis(pentane): 280 (sh), 375 (s) nm ($\epsilon = 3.05 \times 10^5$ M^{-1} cm^{-1}). IR (Nujol, cm^{-1}): 3035 sh, 2435 sh, 1565 vw, 1435 m, 1315 m, 1256 s, 1100 w, 1056 w, 1040 w, 887 vs, 775 s, 670 s, 665 sh, 510 w, 475 s, 390 sh.

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Table 1. Crystallographic Data for $(C_6H_5)_2Pb(Ge(SiMe_3)_3)_2$ (**5**)

$C_{30}H_{64}Ge_2Si_6Pb{\cdot}C_5H_{12}$	*Pnn*2 (orthorhombic)
fw = 1018.02	$T = -75$ °C
$a = 13.999$ (5) Å	$\lambda = 0.710\,73$ Å
$b = 17.443$ (5) Å	$\varrho_{\text{cald}} = 1.33$ g cm^{-3}
$c = 10.404$ (2) Å	$\mu = 46.5$ cm^{-1}
$V = 2541$ Å^3	$R = \Sigma\|\|F_o\| - \|F_c\|\|/\Sigma\|F_o\| = 0.035$
$Z = 2$	$R_w = [\Sigma_w(\|F_o\| - \|F_c\|)^2/\Sigma_w\|F_o\|^2]^{1/2} = 0.033$

Diphenylbis(tris(trimethylsilyl)germyl)plumbane, $((Me_3Si)_3Ge)_2Pb(C_6H_5)_2$ (5). The pentane solution from the crystallization of **4** was cooled to −78 °C, giving a yellow solid. This was filtered off, dissolved in a minimum volume of pentane and kept at 4 °C for 4 d, affording pale yellow needle crystals (1100 mg, 26.5% isolated yield). Mp: 116 °C dec. Anal. Calcd for $C_{30}H_{64}Si_6Ge_2Pb$: C, 38.10; H, 6.82. Found: C, 36.92; H, 6.77. The low percent carbon is probably due to inert carbide formation during analysis. NMR (C_6D_6): ^{1}H, δ0.310 (s, 54H, $Ge(SiMe_3)_3$), 7.10 to 7.30, 7.90 to 8.08 (mult, 10H, $Pb(C_6H_5)_2$); ^{13}C-{^{1}H}, δ4.30 ($SiMe_3$), 129.0, 139.5, 152.9 (C_6H_5). UV−vis (pentane): 268 (sh), 307 (s) nm ($\epsilon = 2.77 \times 10^5$ M^{-1} cm^{-1}). IR (Nujol, cm^{-1}): 3040 sh, 2710 vw, 1670 m, 1436 s, 1405 m, 1315 m, 1270 sh, 1250vs, 1085 m, 1045 m, 1030 m, 1090 w, 855 vs, 720 s, 651 s, 645 sh, 510 vw, 480 s, 380 m.

X-ray Structure of 5. A pale yellow square column having approximate dimensions 0.50 × 0.20 × 0.14 mm was mounted in a random orientation on a Nicolet R3m/V automatic diffractometer. Since the sample was temperature-sensitive, all handling was done in a watch glass that was in constant contact with a block of dry ice. The crystal was rapidly transferred to the goniometer and placed in a stream of dry nitrogen gas at −75 °C. The radiation used was Mo Kα monochromatized by a highly ordered graphite crystal. Final cell constants, as well as other information pertinent to data collection and refinement, are listed in Table 1. The Laue symmetry was determined to be mmm and from the systematic absences noted the space group was shown to be either *Pnn*2 or *Pnnm*. The overall quality of the crystal was poor, having broad and occasionally split peaks in ω. This was attributed to a combination of partial solvent loss during handling as well as the presence of several tiny satellite crystallites frozen to the surface of the sample, which could not be avoided due to the unusual procedures used. The intensities were measured using the Θ:2Θ scan technique, with the scan rate depending on the count obtained in rapid pre-scans of each reflection. Two standard reflections were monitored after every 2 h or every 100 data collected, and these showed no significant variation. During data reduction Lorentz and polarization corrections were applied, as well as a semiempirical absorption correction based on ψ scans of ten reflections having χ values between 70 and 90°.

Since the unitary structure factors displayed acentric statistics, space group, *Pnn*2 was assumed from the outset. The structure was solved by the SHELXTL Patterson interpretation program, which revealed the positions of the Pb and Ge atoms in the asymmetric unit, consisting of a half-molecule situated about a 2-fold axis. Remaining atoms were located in subsequent difference Fourier syntheses. The usual sequence of isotropic and anisotropic refinement was followed, after which all hydrogens were entered in ideal calculated positions and constrained to riding motion, with a single variable isotropic temperature factor for all of them. At this point, a partial molecule of pentane solvent was located, disordered about a 2-fold axis. This was treated by refining two separate ethyl groups defined by strong distance constraints. Each carbon atom was given a population of 40% (total occupancy 80%), which seemed to give the most reasonable range of temperature factors. All calculations in Table 1 are based on 100% occupancy however, under the assumption that some of the solvent was lost in handling.

The sense of direction in this polar, nonenantiomorphous space group was determined by refinement of a coefficient multiplying $\Delta f''$, which showed a very strong probability that the reported atomic coordinates are correct. After all shift/esd ratios were less than 0.1, convergence was reached at the agreement factors listed in Table 1. No unusually high correlations were noted between any of the variables in the last cycle of full-matrix least squares refinement, and the final difference density map showed a maximum peak of about 1.1 e/Å^3, located near Pb. All calculations were made using Nicolet's SHELXTL PLUS (1987) series of crystallographic programs.

Results and Discussion

Reaction of triphenyllead chloride with the lithium silylgermyl reagent according to eq 1, proceeds to give tris(trimethylsilyl)germyltriphenylplumbane (**3**) in 25% yield as transparent

$$(C_6H_5)_3PbCl + (THF)_{2.5}LiGe(SiMe_3)_3 \xrightarrow[-78\ °C\ \text{to RT}]{(C_2H_5)_2O} (Me_3Si)_3GePb(C_6H_5)_3 + LiCl \quad (1)$$

colorless crystals which are stable in air.[5] The melting point of 120 °C is lower than those of both $(Me_3Si)_3CPb(C_6H_5)_3$ (mp 195−197 °C) and $(Me_3Si)_3SiPb(C_6H_5)_3$ (mp 145 °C).[1] Although an X-ray structure determination was not carried out for **3**, we performed a molecular mechanics calculation on the molecule. Such calculations have been shown to reproduce major structural features of $(Me_3Si)_3SiGe(C_6H_5)_3$[6] and $(Me_3Si)_3SiSi(SiMe_3)_3$[7] with reasonable accuracy. In this case it was necessary to use estimated parameters for the Pb−Ge bond so the results should be regarded as approximate. The structure predicted for (**3**) exhibited a Ge−Pb bond distance of 2.7 Å, close to the sum of the covalent radii of Ge and Pb (2.68 Å[8]), and average Si−Ge−Si and Si−Ge−Pb angles near the tetrahedral value, suggesting minimal steric strain in the monosubstituted structure.

The sizable cone angle generated by the $(Me_3Si)_3Ge-$ group[9] is expected to cause considerable steric strain in *disubstituted* derivatives of the type $((Me_3Si)_3Ge)_2MX_n$ ($n \neq 0$). The structure of $((Me_3Si)_3Ge)_2SnCl_2$ shows conspicuous distortion from tetrahedral geometry around the tin[10] and $((Me_3Si)_3Ge)_2GeCl_2$ appears to be unstable at room temperature.[10] It was thus of interest to attempt the synthesis of a $((Me_3Si)_3Ge)_2PbX_2$ compound to ascertain whether the larger radius of Pb would reduce the steric strain sufficiently that the disubstituted Pb(IV) derivative, like that of Sn, would be stable. Since $PbCl_4$ is too strong an oxidizer to use with the lithium silyl reagent, $(C_6H_5)_2PbCl_2$ was chosen as the Pb(IV) starting material. The reaction was carried out according to eq 2. After 20 h of stirring,

$$(C_6H_5)_2PbCl_2 + 2(THF)_{2.5}LiGe(SiMe_3)_3 \xrightarrow[-78\ °C\ \text{to RT}]{(C_2H_5)_2O} (C_6H_5)_2Pb(Ge(SiMe_3)_3)_2 + 2LiCl \quad (2)$$

removal of ether, extraction of the residue into pentane, and cooling to 4 °C, transparent red-orange crystals were obtained that became a yellow-orange powder after drying under vacuum. This product was identified as the diplumbane, $[(C_6H_5)_2PbGe(SiMe_3)_3]_2$, (**4**) (34% based on total Pb) by elemental analyses, ^{1}H and ^{13}C NMR spectra. The low carbon percentage, also seen in related compounds, was thought to result from silicon and germanium carbide formation during analysis. An attempted X-ray structure determination on the orange crystals could not be completed due to crystal decomposition, but **4** was shown to be isomorphous[11] with $[(C_6H_5)_2PbSi(SiMe_3)_3]_2$.[1]

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(5) The crystals developed a gray coating after 2−3 months, even at 4 °C.

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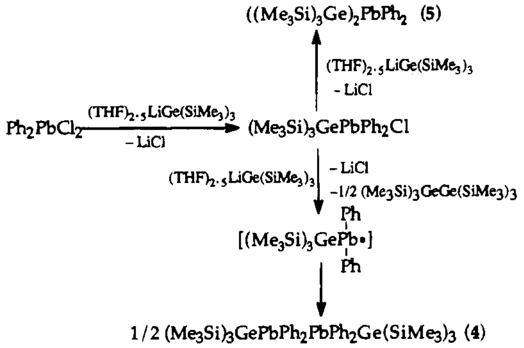
Scheme 1



After **4** was removed by crystallization, the remaining pentane solution was cooled to −78 °C causing precipitation of a yellow powder. This was crystallized from pentane yielding transparent, pale yellow crystals that were identified as the expected (eq 2) disubstituted, monoplumbane, $(C_6H_5)_2Pb(Ge(SiMe_3)_3)_2$ (**5**) (26.5% based on total Pb), by elemental analysis and ^1H and ^{13}C NMR spectra. The chemical shifts and the integrated areas of the proton spectra were consistent with the proposed formulation of **5**. Drying under vacuum yielded a pale yellow powder due to loss of solvation. An X-ray structure determination on the yellow crystals gave the structural parameters of **5** as a pentane solvate. Both **4** and **5** were stable to air except for the slow loss of pentane. In pentane solution, they exhibited UV−visible spectra consisting of strong peaks with λ_{max} at 375 (**4**) and 307 nm (**5**) accompanied by shoulders at 280 and 268 nm, respectively. In this series, diplumbanes have exhibited deeper colors than analogous monoplumbanes.[1]

The fact that both (**4**) and (**5**) were isolated from the reaction of $(C_6H_5)_2PbCl_2$ with 2 equiv of $(THF)_{2.5}LiGe(SiMe_3)_3$ closely parallels the results reported earlier using $(THF)_3LiSi(SiMe_3)_3$[1] giving the silyl counterparts **1** and **2**. In that case, a possible pathway to the diplumbane *via* decomposition of the strained, disubstituted monoplumbane was ruled out by NMR experiments. In order to similarly examine the stability of **4** and **5** under the conditions of our experiment, we made solutions of each compound individually in ether, held these for 20 h at ambient temperature, removed the ether, and obtained ^1H spectra of each residue in C_6D_6 solution. The ^1H spectrum of **5** changed minimally over 20 h and the resonance of **4** did not appear in the spectrum. (A solution of a mixture of **4** and **5** in C_6D_6, stored in the refrigerator several months exhibited a small amount of black deposit, presumably Pb.) After 20 h, the $SiMe_3$ resonance of **4** (δ 0.155) had been partially replaced by major ^1H signals at δ 0.138 and 0.167 (^{13}C, δ 3.847) that were not further identified, and smaller signals at δ 0.026, 0.172, 0.237, 0.293, and 0.389 ((probably $Me_3Si)_3GeGe(SiMe_3)_3$, lit.[12] 0.39 in C_6D_6). Thus, **4** is not formed from **5**, and the former decomposes to some extent in the reaction mixture accounting for the low isolated yield.

Table 2. Atomic Coordinates ($\times 10^4$) and Equivalent Isotropic Displacement Parameters ($Å^2 \times 10^3$)

	x	*y*	*z*	*U*(eq)[a]
Pb	5000	5000	5500	28(1)
Ge	3260(1)	4764(1)	4516(2)	36(1)
Si(1)	2466(4)	5973(3)	4461(6)	54(2)
Si(2)	3285(4)	4213(3)	2434(4)	51(2)
Si(3)	2379(4)	3949(3)	5956(5)	42(2)
C(1)	1360(11)	5866(9)	3355(19)	78(8)
C(2)	2139(13)	6261(10)	6173(18)	78(9)
C(3)	3223(13)	6744(8)	3699(20)	80(8)
C(4)	4230(15)	3464(11)	2323(19)	71(9)
C(5)	3553(13)	4648(10)	1201(12)	83(7)
C(6)	2093(13)	3871(11)	1905(17)	86(9)
C(7)	2663(13)	4152(9)	7705(13)	59(7)
C(8)	2554(10)	2892(6)	5622(20)	56(5)
C(9)	1090(10)	4144(8)	5735(23)	68(7)
C(10)	4780(9)	5978(5)	6903(13)	31(5)
C(11)	4970(15)	6276(6)	6512(13)	41(4)
C(12)	4867(14)	7355(7)	7314(14)	50(6)
C(13)	4566(11)	7246(8)	8549(17)	54(6)
C(14)	4310(15)	6506(10)	9001(18)	61(8)
C(15)	4436(10)	5882(7)	8148(13)	35(5)
C(16)	0	5000	9310(35)	120(13)
C(17)	152(30)	5728(12)	9938(25)	54(11)
C(17′)	908(19)	5299(19)	9744(43)	84
C(18)	338(29)	6387(19)	9148(36)	57(13)
C(18′)	853(33)	6116(19)	9527(44)	64(12)

[a] Equivalent isotropic *U* defined as one-third of the trace of the orthogonalized $\mathbf{U}_{ij}$ tensor.

A possible route to the observed products involving the reduction of an intermediate is shown in Scheme 1. In this scenario, the first equivalent of lithium silylgermyl reagent produces the expected substitution step forming a monochloro intermediate which can either undergo a second substitution to yield **5** or may be reduced by the second equivalent of lithium silylgermyl eventually forming the dilead derivative (**4**). Since the lithium reagent is a fairly strong reducing agent, the preparation of hexaphenyldilead *via* the reduction of triphenyllead chloride by an alkali metal serves as a precedent for the lower branch of Scheme 1.[13] A prominent ^1H signal at δ 0.390 and a corresponding ^{13}C signal at δ 4.8 show the presence of an appreciable quantity of $(Me_3Si)_3GeGe(SiMe_3)_3$ in the reaction mixture, as required by Scheme 1. The reducing action of the lithium silylgermyl reagent towards $PbCl_2$ (eq 3) lends support

$$PbCl_2 + 2(THF)_{2.5}LiGe(SiMe_3)_3 \xrightarrow{Et_2O} ((Me_3Si)_3Ge)_2 + Pb + 2LiCl \quad (3)$$

to the proposed reductive pathway to compound **4** in Scheme 1. The reaction in eq 3 represents a convenient synthesis of $(Me_3Si)_3GeGe(SiMe_3)_3$ in good yield.[12]

Description of the Structure of 5. Pale yellow crystals grown from pentane were found to contain one molecule of pentane of solvation that was lost under reduced pressure or upon standing at ambient so the crystals were maintained at low temperature. Crystal and data collection parameters are summarized in Table 1; atomic coordinates and geometric parameters for the heavy atoms are presented in Tables 2 and 3, respectively. Figure 1 shows the heavy atom molecular structure determined for (**5**). The Pb−Ge bond distance, 2.675(2) Å, is about that predicted from the sum of the covalent radii of Pb(IV), 1.46 Å, and Ge(IV), 1.22 Å.[8] With the exception of the large Ge−Pb−Ge′ angle, 135.0 (1)° and the small C(10)−Pb−C(10′) angle (99.8°), the heavy atom bond angles fall within a few degrees of the expected tetrahedral values. The Si−Ge−Si and Pb−Ge−Si angles average 109.0 and 109.8°, respectively, in contrast to the larger distortion of those angles seen in the more strained $((Me_3Si)_3Ge)_2SnCl_2$

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(11) $C_{42}H_{74}Ge_2Pb_2Si_6$, fw = 1307.13; space group $P2_1/n$ (molecule at inversion center); a = 13.259 Å, b = 17.023 Å, c = 14.06 Å, β = 97.43°, V = 3136.9 $Å^3$, Z = 2, d_{meas} = 1.39 g cm^{-3}, d_{calc} = 1.384 g cm^{-3} (no lattice solvent), μ = 64.624 cm^{-1}, T = 23 °C. These parameters may be compared to those of the analogous silyl compound, $[(Me_3Si)_3SiPb(C_6H_5)_2]_2$:[1] space group $P2_1/n$ (monoclinic), a = 13.208 Å, b = 16.586 Å, c = 13.896 Å, β = 99.29°, V = 3004$Å^3$, Z = 2, d_{calc} = 1.43 g cm^{-3}, showing that the two are isomorphous.

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Table 3. Selected Bond Lengths (Å), Bond Angles (deg), and Torsion Angles (deg) for **5**

	A. Bond Lengths		
Pb−Ge	2.675(2)	Pb−C(10)	2.266(13)
Ge−Si(1)	2.385(5)	Ge−Si(2)	2.370(5)
Ge−Si(3)	2.405(5)	Si(1)−C(1)	1.937(18)
Si(1)−C(2)	1.906(20)	Si(1)−C(3)	1.888(17)
Si(2)−C(4)	1.863(20)	Si(2)−C(5)	1.877(16)
Si(3)−C(7)	1.896(15)	Si(3)−C(8)	1.893(13)
C(10)−C(11)	1.392(17)	C(10)−C(15)	1.392(19)
C(11)−C(12)	1.386(18)	C(12)−C(13)	1.366(23)
	B. Bond Angles		
Ge−Pb−C(10)	103.8(3)	Ge−Pb−Ge′	135.0(1)
Ge−Pb−C(10′)	104.8(3)	C(10)−Pb−C(10′)	99.8(7)
Pb−Ge−Si(1)	107.3(1)	Pb−Ge−Si(2)	113.5(1)
Si(1)−Ge−Si(2)	110.1(2)	Pb−Ge−Si(3)	108.6(1)
Si(1)−Ge−Si(3)	107.4(2)	Si(2)−Ge−Si(3)	109.7(2)
Ge−Si(1)−C(1)	107.5(5)	Ge−Si(1)−C(2)	108.8(6)
C(1)−Si(1)−C(2)	112.9(8)	Ge−Si(1)−C(3)	112.2(6)
C(1)−Si(1)−C(3)	105.6(8)	C(2)−Si(1)−C(3)	109.8(8)
Ge−Si(2)−C(4)	110.6(7)	Ge−Si(2)−C(5)	110.1(5)
C(4)−Si(2)−C(5)	107.9(9)	Ge−Si(2)−C(6)	112.8(6)
Ge−Si(3)−C(7)	112.4(6)	Ge−Si(3)−C(8)	113.3(6)
C(7)−Si(3)−C(8)	109.3(8)	Ge−Si(3)−C(9)	108.3(6)
Pb−C(10)−C(11)	119.4(10)	Pb−C(10)−C(15)	123.8(9)
Pb−C(10)−C(11)	121.6(6)	Pb−C(10)−C(15)	119.7(6)
Pb−C(16)−C(17)	122.0(7)	Pb−C(16)−C(21)	117.7(7)
	C. Torsion Angles		
C(10)−Pb−Ge−Si(1)	32.2	Ge′−Pb−C(10′)−C(15′)	82.2
C(10)−Pb−Ge−Si(2)	154.1	Pb−C(10′)−C(11′)−C(12′)	−179.1
C(10)−Pb−Ge−Si(3)	−83.6		
C(10′)−Pb−Ge−Si(2)	−101.7	C(10′)−Pb−Ge−Si(1)	136.4
Ge′−Pb−Ge−Si(1)	−95.9	C(10′)−Pb−Ge−Si(3)	20.6
Ge′−Pb−Ge−Si(3)	148.3	Ge′−Pb−Ge−Si(2)	26.0
Si(1′)−Ge′−Pb−Ge	−95.9	C(1)−Si(1)−Ge−Pb	165.2
Si(1′)−Ge′−Pb−C(10)	136.4	C(1)−Si(1)−Ge−Si(2)	41.2
Si(1′)−Ge′−Pb−C(10′)	32.2	C(1)−Si(1)−Ge−Si(3)	−78.2
Si(2′)−Ge′−Pb−Ge	26.0	C(2)−Si(1)−Ge−Pb	−72.2
Si(2′)−Ge′−Pb−C(10)	−101.7	C(2)−Si(1)−Ge−Si(2)	163.8
Si(2′)−Ge′−Pb−C(10′)	154.1	C(2)−Si(1)−Ge−Si(3)	44.4
Si(3′)−Ge′−Pb−Ge	148.3	C(3)−Si(1)−Ge−Pb	49.5
Si(3′)−Ge′−Pb−C(10)	20.6	C(3)−Si(1)−Ge−Si(2)	−74.4
Si(3′)−Ge′−Pb−C(10′)	−83.6	C(3)−Si(1)−Ge−Si(3)	166.2

(Average Si−Ge−Si = 111.3°, Sn−Ge−Si = 107.6°).[1] Some steric repulsion between the trimethylsilyl groups in **5** is indicated by the staggering of the methyl groups (Figure 2). The dihedral angle between the two phenyl rings is 37°. These considerations suggest that the two large Ge$(SiMe_3)_3$ groups cause crowding even around the large Pb atom but that the steric strain is relieved by distortion of the weaker bonds to the lead rather than those in the germylsilyl substituents.

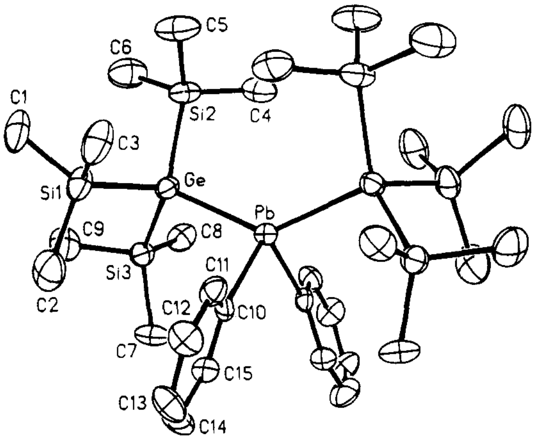

Figure 1. View of **5** showing the atom numbering scheme. Thermal ellipsoids are 40% equiprobability envelopes, with hydrogens omitted for clarity.

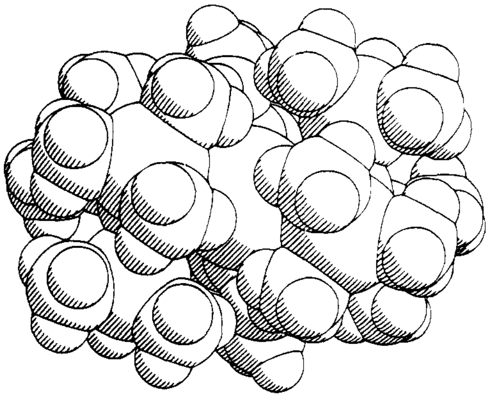

Figure 2. Space-filling view of **5** down the 2-fold axis showing the staggering of the methyl groups.

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Supplementary Material Available: For **5**, tables listing full data collection and processing parameters, full bond lengths and angles, torsion angles, anisotropic displacement parameters, H atom coordinates and isotropic displacement parameters (6 pages). Ordering information is given on any current masthead page.