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Anionic Cadmium Complexes Containing the Bulky Tris(trimethylsilyl)germyl and Tris(trimethylsilyl)silyl Ligands

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The first triiodo-bridged tris(trimethylsilyl)silyl- and tris(trimethylsilyl)germyldicadmium anionic complexes were prepared from CdI_2 and 2 equiv of $LiE(SiMe_3)_3$ (E = Si and Ge) in hexane. The crystal structure of the germyl derivative **1** was determined. It crystallizes as a lithium salt from pentane in the *Cmcm* space group (orthorhombic) with $a = 14.494(2)$, $b = 16.334(3)$, and $c = 27.361$ Å, $V = 6478$ Å^3, and $Z = 4$. The complex anion of **1**, $[(Me_3Si)_3GeCd(\mu\text{-}I_3)CdGe(SiMe_3)_3^-]$, exhibits two approximately tetrahedrally coordinated cadmiums with three iodides in trigonal bridging positions between the cadmiums. Efforts to prepare similar chloride-bridged structures were not fruitful.

Introduction

The development of a variety of metal−silicon reagents as silylating agents has generated increasing interest in sterically demanding substituents of the form $-E(SiMe_3)_3$ (E = C, Si, and Ge). In some cases, these bulky groups kinetically stabilize metal centers through the protective influence of their large "umbrella" of methyl groups.[1] In other cases, however, the steric strain they produce seems to cause structural rearrangements such as phenyl migration in the case of $-C(SiMe_3)_3$ with Ph_2PbCl_2[2] and trimethylsilyl group migration in the case of Ge(II).[3] Group 12 complexes with metal−silicon bonds were shown to be useful silylating agents.[4] Homoleptic tellurolates containing Te−Cd bonds can act as single-source precursors for thin films.[5]

Bis(tris(trimethylsilyl)silyl)cadmium and its zinc and mercury counterparts were among the early examples of metal−silyls with the bulky substituents.[6] To determine how substitution of Ge for the central Si affects the structure and stability of the complexes, we prepared several $-Ge(SiMe_3)_3$ analogues of known metal−$Si(SiMe_3)_3$ compounds.[1e,f] Our initial objective in the present work was to prepare $Cd(Ge(SiMe_3)_3)_2$ from $CdCl_2$ or CdI_2 and $(THF)_{2.5}LiGe(SiMe_3)_3$. In this paper we describe the results of those reactions along with that between CdI_2 and $(THF)_3LiSi(SiMe_3)_3$.

Experimental Section

Cadmium iodide and chloride were from Baker and Aldrich. Ethyl ether and tetrahydrofuran (THF) were distilled from $LiAlH_4$ under dry nitrogen. Pentane and hexane were distilled from sodium just before use. The reagents $(THF)_{2.5}LiGe(SiMe_3)_3$ and $(THF)_3LiSi(SiMe_3)_3$ were prepared according to literature methods[7,8] using low-halide (chloride) methyllithium from Aldrich. Elemental analyses were done by Atlantic Microlabs. All manipulations were conducted under argon dried with P_4O_{10}. NMR and IR spectra were recorded as described earlier.[1e,f]

Table 1. Data Collection and Processing Parameters

$Li^+\cdot C_{18}H_{54}Si_6Ge_2Cd_2I_3^-\cdot 4C_4H_8O$	*Cmcm* (orthorhombic)
fw = 1485.36	$T = -50$ °C
$a = 14.494(2)$ Å	$\lambda = 0.710\ 73$ Å
$b = 16.334(3)$ Å	ρ(calcd) = 1.52 g cm^{-3}
$c = 27.361(6)$ Å	$\mu = 31.04$ cm^{-1}
$V = 6478$ Å^3	$R = \Sigma\|\|F_o\| = \|F_c\|\|/\Sigma\|F_o\| = 0.049$
$Z = 4$	$R_w = [\Sigma_w{}^2(\|F_o\| - \|F_c\|)^2/\Sigma\|F_o\|^2]^{1/2} = 0.042$

Syntheses of Tetrakis(tetrahydrofuran)lithium Tris(-μ-iodo)bis[tris(trimethylsilyl)germylcadmate](1−), $[Li(THF)_4][(Me_3Si)_3GeCdI_3CdGe(SiMe_3)_3]$ (1). To a hexane suspension (50 mL, −78 °C) of CdI_2 (947.9 mg, 2.588 mmol) was slowly added 150 mL of a hexane solution of $(THF)_{2.5}LiGe(SiMe_3)_3$ (2481.8 mg, 5.176 mmol). The mixture was stirred at −78 °C for 4 h and allowed to warm slowly to room temperature where stirring continued for 15 h, and then the hexane was removed *in vacuo*. The residue was stirred for 1 h with pentane (50 mL) and filtered and the filtrate concentrated to about 40 mL and held at 0 °C for 30 min affording colorless, transparent, hygroscopic, needle crystals of **1** (1271 mg, isolated yield 60%). Mp: 150 °C dec. Anal. Calcd for $C_{26}H_{70}Cd_2Ge_2I_3LiO_2Si_6$ (bis(THF) solvate, *vide infra*): C, 23.28; H, 5.2. Found: C, 22.19; H, 4.94. ^1H NMR (C_6D_6): δ 0.558 (s, 54 H, $Ge(SiMe_3)_3$), 1.425 (mult) and 3.467 (mult 32 H, 4 THF). ^{13}C NMR (C_6D_6): δ 4.889 ($Ge(SiMe_3)_3$), 25.54, 68.42 (THF). IR (Nujol and neat, cm^{-1}): 3380 s br, 2960 s br, 2900 s br, 1920 w, 1745 m, 1600 s br, 1450 s, 1410 sh, 1285 w, 1250 vs, 1230 w, 1055 vs, 970 s, 850 vs br, 775 w, 695 m, 645 m, 635 sh, 425 w.

X-ray Structure Determination of 1. A colorless thick plate having approximate dimensions 0.40 × 0.45 × 0.12 mm was mounted in a random orientation on a Nicolet R3m/V automatic diffractometer. Since the crystals began to decompose in less than 1 min when exposed to the air at room temperature, all handling was done on a glass slide in constant contact with a block of dry ice, and the sample was then placed in a stream of dry nitrogen gas at −50 °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 absence noted the space group was shown to be either *Cmc*2_1, *C*2*cm*, or *Cmcm*. Intensities

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were measured using the ω scan technique, with the scan rate depending on the count obtained in rapid prescans of each reflection. Two standard reflections were monitored after every 2 h or every 100 data collected, and these showed no significant change. During data reduction, Lorentz and polarization corrections were applied, as well as a semiempirical absorption correction based on ψ scans of 10 reflections having χ angles between 70° and 90°. Since the unitary structure factors displayed centric statistics, space group *Cmcm* was assumed from the outset. The structure was solved by the SHELXTL Patterson interpretation program which revealed the positions of the I and Cd atoms in the asymmetric unit, consisting of one-quarter anion situated about an *m*2*m* site, one-quarter Li cation situated on an *m*2*m* site, and two independent half-THF molecules situated about mirror planes. Remaining atoms were located in subsequent difference Fourier syntheses. The usual sequence of isotropic and anisotropic refinement was followed, after which some of the methyl hydrogens were entered in ideal calculated positions and constrained to riding motion, with a single variable isotropic temperature factor for all of them. One of the two unique $SiMe_3$ groups in the anion was found to be disordered over two slightly different positions, and this was treated by introducing an ideal rigid body at this position having 50% occupancy, with the nearby mirror plane generating the other orientation. Both of the two independent THF molecules were found to be heavily disordered, and this was treated by refining atoms with partial occupancies at several different positions.

At this point, properly adjusted models were refined in both of the noncentrosymmetric space groups in order to see if any of the extensive disorder would disappear. Refinement in *Cmc*2$_1$ was very difficult due to extensive correlations between variables and resulted in no less disorder than in *Cmcm*. Refinement in *C*2*cm* proceeded smoothly, but again none of the disorder noted in the *Cmcm* refinement disappeared, and the bond distances seemed unrealistic compared to literature values. The *R* values were little changed despite the additional variables. Since no discernible improvement was achieved by refinement in either of the noncentrosymmetric space groups, the assumption was made that *Cmcm* is the best choice, and this was used for the final refinement. After all shift/ESD ratios were <0.1 (excepting the rigid group), 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 0.7 e/A^3. All calculations were made using Nicolet's SHELXTL PLUS (1987) series of crystallographic programs.

Reaction of $CdCl_2$ with $(THF)_{2.5}LiGe(SiMe_3)_3$ in Hexane. A hexane (−78 °C, 50 mL) suspension of $CdCl_2$ (553.8 mg, 3.02 mmol) and 150 mL of a hexane solution of $(THF)_{2.5}LiGe(SiMe_3)_3$ (2896.7 mg, 6.04 mmol) were combined as described in the synthesis of **1**. Workup in pentane produced copious quantities of metallic powder, presumably cadmium, along with decomposition products of the lithium germyl reagent including $(Me_3Si)_3Ge{-}Ge(SiMe_3)_3$, $HGe(SiMe_3)_3$, and $Ge(SiMe_3)_4$. No cadmium-containing product was isolated.

Reaction of $CdCl_2$ with $(THF)_{2.5}LiGe(SiMe_3)_3$ in Diethyl Ether. An ether solution (150 mL) of $(THF)_{2.5}LiGe(SiMe_3)_3$ (4550 mg, 9.48 mmol) was added to a cold ether suspension of $CdCl_2$ (870 mg, 4,74 mmol), as described for **1** in hexane. Workup in pentane gave only metallic powder and lithium germyl decomposition products. No cadmium-containing product was isolated.

Synthesis of Tetrakis(tetrahydrofuran)lithium Tris(μ-iodo)bis[tris(trimethylsilyl)silylcadmate](1−), $[Li(THF)_4][(Me_3Si)_3SiCdI_3CdSi(SiMe_3)_3]$ (2). A hexane (150 mL) solution of $(THF)_3LiSi(SiMe_3)_3$ (3435.7 mg, 7.29 mmol) was added to a hexane suspension (−78 °C, 50 mL) of CdI_2 (1330 mg, 3.647 mmol). After workup as described for **1**, colorless, transparent, hygroscopic crystals (2010 mg, isolated yield 79%) formed from pentane. Mp: 135 °C dec. Anal. Calcd for $C_{26}H_{70}Cd_2I_3LiO_2Si_8$ (bis(THF) solvate, *vide infra*): C, 24.94; H, 5.63. Found: C, 25.18; H, 5.62. ^{1}H NMR (C_6D_6): δ 0.538 (s, 54 H, $Si(SiMe_3)_3$), 1.451 (mult) and 3.481 (mult) (32 H, 4THF). ^{13}C NMR (C_6D_6): δ 4.266 ($Si(SiMe_3)_3$), 25.63 and 68.41 (THF). IR[9] (Nujol cm^{-1}) 3460 s br, 1910 w, 1620 m, 1410 s sh, 1370 sh, 1325 m, 1270 sh, 1260 vs, 1205 w, 1070 sh, 1065 vs, 940 s sh, 920 sh, 870 vs br, 785 m, 770 sh, 720 s, 660 s, 650 s, 595 m, 445 s.

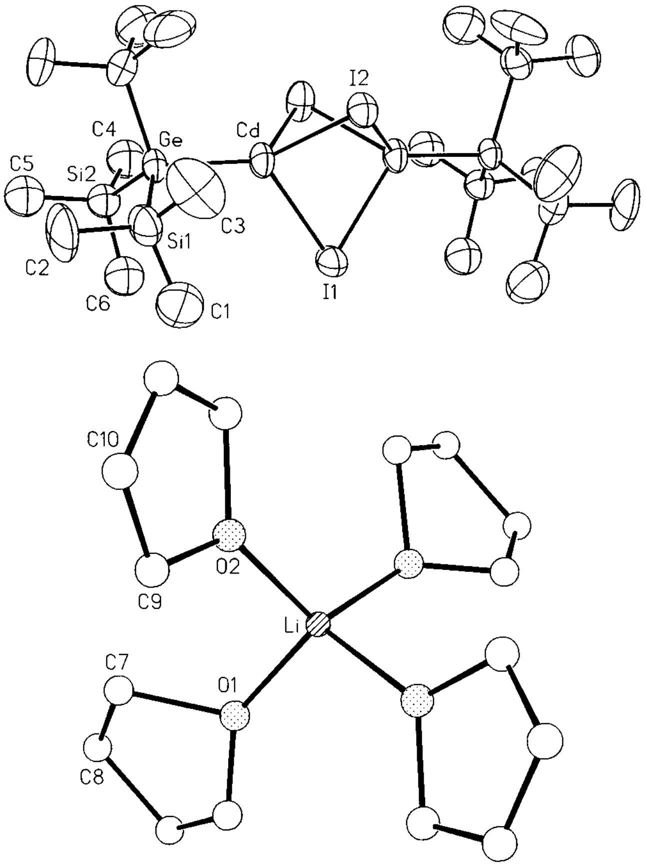
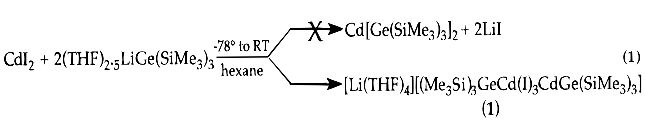


Figure 1. View of the cation and anion structures of **1**. Thermal ellipsoids are 40% equiprobability envelopes, with hydrogens omitted for clarity. Only one orientation of the disordered groups is shown.

Results and Discussion

Arnold, Tilley, Rheingold, and Geib[6] obtained bis(tris(trimethylsilyl)silyl)cadmium as a moderately stable solid from reaction of $CdCl_2$ with 2 equiv of $(THF)_3LiSi(SiMe_3)_3$ in diethyl ether. The more stable zinc and mercury silyls were similarly prepared and the former shown by X-ray crystallography to have the expected linear Si−Hg−Si backbone connecting the bulky "super silyl" groups. Seeking to prepare the analogous cadmium tris(trimethylsilyl)*germyl* derivative, we conducted the reaction of CdI_2 with 2 equiv of $(THF)_{2.5}LiGe(SiMe_3)_3$. The unexpected result, shown in eq 1, was the formation of the lithium salt of



$$CdI_2 + 2(THF)_{2.5}LiGe(SiMe_3)_3 \xrightarrow[\text{hexane}]{-78^\circ \text{ to RT}} \begin{cases} \not\to Cd[Ge(SiMe_3)_3]_2 + 2LiI \\ \to [Li(THF)_4][(Me_3Si)_3GeCd(I)_3CdGe(SiMe_3)_3] \ \textbf{(1)} \end{cases} \quad (1)$$

an unusual dicadmium anion.

Description of the Structure of 1. The anion in **1** (Figure 1) consists of two, approximately tetrahedral $RCdI_3$ (R = $(Me_3Si)_3Ge$) complexes sharing three trigonally disposed iodines (Table 2 gives atomic coordinates). The I−Cd−I angles average only about 91°, while the average of the Ge−Cd−I angles is near 125° (Table 3). This distortion of the expected tetrahedral angles around Cd is attributable, at least in part, to crowding of the iodines by the bulky R group. Figure 2 shows a CPK model of the anion where the steric demands of the group can be seen. The large space requirements of the iodines in the Cd(μ-I)$_3$Cd-bridging framework also favor small I−Cd−I angles. The Cd−Cd distance (3.345 Å) exceeds that in metallic cadmium (3.04 Å),[10] indicative of minimal interaction between the cadmiums. The Cd−Cd distance in **1** falls in the range of values recently

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[9] Both **1** and **2** reacted with the AgBr windows of the infrared cells.

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

	x	*y*	*z*	*U* (eq)[a]
I(1)	1466(1)	2791(1)	7500	92(1)
I(2)	0	609(1)	7500	73(1)
Cd	0	2099(1)	6889(1)	74(1)
Ge	0	2162(1)	5964(1)	64(1)
Si(1)	1330(3)	1469(3)	5702(2)	91(2)
C(1)	2397(10)	1964(9)	5964(6)	154(9)
C(2)	1413(10)	1405(9)	5036(5)	146(9)
C(3)	1318(10)	399(8)	5958(6)	165(10)
Si(2)	−170(7)	3550(3)	5703(2)	76(4)
C(4)	−1224	3972	5997	106(7)
C(5)	−287	3618	5027	106(7)
C(6)	854	4150	5903	106(5)
Li	0	3359(32)	2500	100(18)
O(1)	1079(11)	4052(9)	2572(17)	113(8)
O(2()	150(25)	2611(9)	1952(6)	104(7)
C(7)	1734(20)	4212(17)	2091(11)	89(10)
C(8)	2267(10)	4893(9)	2220(5)	116(6)
C(9)	834(19)	2041(18)	1867(11)	125(10)
C(10)	542(20)	1412(18)	1533(13)	129(12)
C(7′)	1214(22)	4649(18)	2057(12)	107(11)
C(9′)	468(27)	2561(25)	1511(16)	123(16)
C(10′)	−267(36)	1708(25)	1329(14)	121(19)
C(24)	5842(25)	272(34)	4811(64)	276(34)

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

Table 3. Selected Bond Lengths (Å), Bond Angles (deg), and Torsion Angles (deg) for **1**

	Bond Lengths		
Cd−I(1)	2.931(2)	Cd−I(2)	2.953(2)
Cd−Ge	2.533(2)	Cd−CdA	3.345(1)
Ge−Si(1)	2.348(5)	Si(1)−C(1)	1.887(16)
Si(1)−C(2)	1.830(16)	Si(1)−C(3)	1.884(15)
Li−O(1)	1.940(35)	Li−O(2)	1.947(36)
	Bond Angles		
Cd−I(1)−CdA	69.6(1)	Cd−I(2)−CdA	69.0(1)
I(1)−Cd−I(2)	89.7(1)	I(1)−Cd−Ge	123.7(1)
I(2)−Cd−Ge	126.8(1)	I(1)−Cd−I(1A)	92.9(1)
Cd−Ge−Si(1)	106.6(1)	Cd−Ge−Si(2)	109.7(2)
Si(1)−Ge−Si(2)	116.8(3)	Si(1)−Ge−Si(1A)	110.4(2)
O(1)−Li−O(2)	110.7(15)	O(1)−Li−O(1B)	108.6(26)
	Torsion Angles		
CdA−I(1)−Cd−I(2)	−46.7	CdA−I(1)−Cd−Ge	177.5
CdA−I(2)−Cd−I(1)	46.4	CdA−I(2)−Cd−Ge	180.0
I(1)−Cd−Ge−Si(1)	60.5	I(1)−Cd−Ge−Si(2)	−66.0
I(1)−Cd−Ge−Si(1)A	178.4	I(2)−Cd−Ge−Si(1)	−59.0
I(2)−Cd−Ge−Si(2)	174.6	I(2)−Cd−Ge−Si(1)A	59.0

found in three dicadmium complexes with tetrakis(μ-carboxylate-*O*,*O′*) dimetal cores.[11] Both the two Ge atoms and the two Cd atoms are colinear. The trimethylsilyl groups on the two Ge atoms are in an eclipsed conformation, probably due to steric interactions with the iodines. Taking into account the slight disorder found in the trimethylsilyl positions, the Si−Ge−Cd−I angles average about 60°. The THF positions in the cation of **1** were heavily disordered.

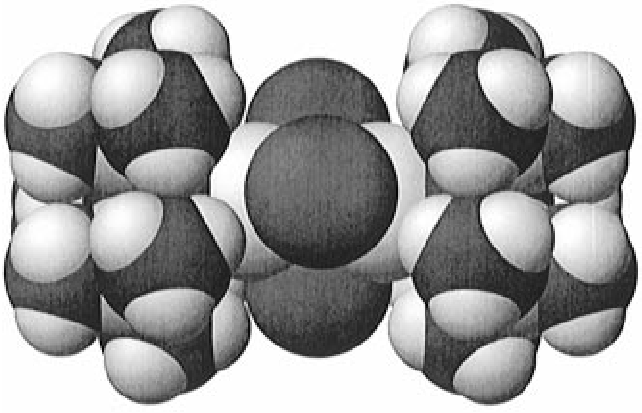

Figure 2. Space-filling (CPK) view of the anion of **1**.

Cadmium iodide complexes such as CdI_3^- and CdI_4^{2-} are known,[12] as are organodihalocadmates, $RCdX_2^-$,[13] but bridged dicadmium complexes appear to be uncommon, and we were unable to find examples of tribridged, dicadmates related to the anion of **1**. In principle, the anion can be regarded as a dimer of RCdI (R = $(Me_3Si)_3Ge$) that has formed a complex with an iodide. Sharing three bridging iodides relieves the coordinate unsaturation around the cadmiums in the putative dimer while achieving the tetrahedral coordination familiar in CdX_4^{2-} complex ions. RCdI (R = alkyl) compounds are known in the solid phase[14] but may decompose in solution unless stabilized by complex formation with either neutral ligands[15] or anions.[13] A possible pathway to **1** (eq 2) involves, first, formation of $(Me_3Si)_3GeCdI$, second, its dimerization, and, finally, its complexation with iodide from the reaction mixture. Although we did not see spectroscopic evidence for the RCdI dimer, the low complexing ability of the hexane solvent favors its formation. An alternate pathway in which RCdI first combines with I$^-$ to form an intermediate cadmate ion, $RCdI_2^-$, and then this reacts with another RCdI giving the anion of **1** does not seem as likely since known $RCdX_2^-$ salts, even with large cations, are substantially insoluble in organic solvents.[13]

$$CdI_2 + LiGe(SiMe_3)_3 \xrightarrow[\text{-LiI}]{} [(Me_3Si)_3GeCdI] \xrightarrow[\text{hexane}]{\text{dimerize}} [(Me_3Si)_3GeCdI]_2 \xrightarrow[+I^-]{} \text{anion of } (\mathbf{1}) \quad (2)$$

The novel structure of **1** prompted us to carry out the reaction of CdI_2 with 2 equiv of $(THF)_{2.5}LiSi(SiMe_3)_3$ to determine whether the disubstituted product, reported by Tilley et al.,[6] formed or if it would follow the course of the synthesis of **1**. The product (**2**) is assigned by its NMR spectra and elemental analyses[16] to be the $-Si(SiMe_3)_3$ counterpart of **1**. Although **2** was not structurally characterized, the major differences in spectra, melting point,[17] and composition between it and $Cd(Si(SiMe_3)_3)_2$, the other plausible product, support the assignment of its structure as also involving a triiodide-bridged dicadmium anion (eq 3). A small NMR peak at δ 0.330, as expected for $Cd(Si(SiMe_3)_3)_2$,[6] was observed in the reaction mixture, but isolation of that product was not attempted.

$$CdI_2 + 2(THF)_3LiSi(SiMe_3)_3 \xrightarrow[\text{hexane}]{\text{-78° to RT}} \underset{(\mathbf{2})}{[Li(THF)_4][(Me_3Si)_3SiCd(I)_3CdSi(SiMe_3)_3]} \quad (3)$$

The presence of THF was necessary for the purification of both products. Repeated crystallizations of either from pentane

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[16] Crystals of both **1** and **2** lost THF readily. Vacuum drying of analytical samples for 30 min gave results which correspond reasonably well to bis(THF) solvates. Crystals dried for only 1−2 min for NMR spectra gave integrations consistent with tetrakis(THF) solvation if the spectra were obtained immediately after preparation. After about 15 min, the C_6D_6 solutions became turbid.

[17] The mp of $Cd(Si(SiMe_3)_3)_2$ was reported to be 154−160 °C, whereas **2** melted with decomposition at 135 °C.

alone caused loss of THF and decomposition forming insoluble materials. Fortunately, conditions were found whereby one crystallization gave single crystals suitable for an X-ray structure determination.

Seeking a chloro-bridged, dicadmium anion analogous to the anion in **1**, we carried out reactions between $CdCl_2$ and 2 equiv of $(THF)_{2.5}LiGe(SiMe_3)_3$, both in hexane, as done in the other syntheses, and in diethyl ether. Unfortunately, both reactions gave only metallic powder, presumably cadmium, and decomposition products of the reagent, $HGe(SiMe_3)_3$, $(Me_3Si)_3Ge-Ge(SiMe_3)_3$, and $Ge(SiMe_3)_4$. There was no indication in NMR spectra of either $Cd(Ge(SiMe_3)_3)_2$ or $[Li(THF)_4][(Me_3Si)_3GeCdCl_3CdGe(SiMe_3)_3]$. The facile reduction of $CdCl_2$ by the germyl reagent was unexpected. Reduction was not reported when $CdCl_2$ and the silyl reagent were combined in ether and extracted into pentane.[6] Since chloro-bridged anions were not observed by the earlier workers either, it may be that the tribridged dicadmium anion core requires the large iodides for stability. The rather striking difference in products obtained from different cadmium halides has led us to examine the behavior of other metal iodides with the silyl and germyl reagents.

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Supporting Information Available: Tables listing full data collection and processing parameters, full bond lengths and angles, anisotropic displacement parameters, and H atom coordinates (9 pages). Ordering information is given on any current masthead page. Observed and calculated structure factors (11 pages) are available from the corresponding author.

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