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Synthesis, Structural Characterization, and Reactivity of $(Me_3CCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2}Cl$, $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]_2$, and $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$

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Abstract

Reaction of two equivalents of $(Me_3CCH_2)_2GaCl$ with $(Me_3Si)_3As$ affords $(Me_3CCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2}Cl$ (**1**), the fourth example of a compound containing the Ga-As-Ga-X (X = Cl, Br) core, but the first wherein the organogallium four-membered ring with arsenic, halogen mixed-bridging is not puckered. Compound **1**, as well as $(Me_3CCH_2)_2GaCl$, reacts with $LiAs(SiMe_3)_2\cdot 2THF$ (1:1 mole ratio) to produce the common product $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]_2$ (**2**) which on reaction with $(Me_3CCH_2)_2GaCl$ (1:2 mole ratio) gives **1** quantitatively. Mixing $(Me_3CCH_2)_2GaCl$ and $(Me_3Si)_3As$ in a 1:1 mole ratio yields the adduct $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (**3**). Thermolysis of **3** did not afford **2**, nor did its reaction with an additional equivalent of $(Me_3CCH_2)_2GaCl$ lead to **1** as the predominate product. Various physical and spectroscopic characterization data are presented for compounds **1-3**, as well as the results of their X-ray crystal structure determinations. Crystal data: **1**, monoclinic, space group $P2_1/c$ (C^5_{2h}), a = 12.412(1) Å, b = 17.306(1) Å, c = 20.238(2) Å, β = 119.34(1)°, V = 3790(1) Å^3, Z = 4; **2**, monoclinic, space group $P2/c$ (C^4_{2h}), a = 12.180(1) Å, b = 12.744(1) Å, c = 19.618(2) Å, β = 128.43(1)°, V = 2386(1) Å^3, Z = 2; **3**, monoclinic, space group $P2_1/c$ (C^5_{2h}), a = 12.486(1) Å, b = 12.544(1) Å, c = 19.985(2) Å, β = 101.73(1)°, V = 3065(1) Å^3, Z = 4.

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Reaction of two equivalents of $(Me_3CCH_2)_2GaCl$ with $(Me_3Si)_3As$ affords $(Me_3CCH_2)_2GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2Cl$ (**1**), the fourth example of a compound containing the Ga-As-Ga-X (X = Cl, Br) core, but the first wherein the organogallium four-membered ring with arsenic, halogen mixed-bridging is not puckered. Compound **1**, as well as $(Me_3CCH_2)_2GaCl$, reacts with $LiAs(SiMe_3)_2\cdot 2THF$ (1:1 mole ratio) to produce the common product $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]_2$ (**2**) which on reaction with $(Me_3CCH_2)_2GaCl$ (1:2 mole ratio) gives **1** quantitatively. Mixing $(Me_3CCH_2)_2GaCl$ and $(Me_3Si)_3As$ in a 1:1 mole

ratio yields the adduct $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (**3**). Thermolysis of **3** did not afford **2**, nor did its reaction with an additional equivalent of $(Me_3CCH_2)_2GaCl$ lead to **1** as the predominate product. Various physical and spectroscopic characterization data are presented for compounds **1-3**, as well as the results of their X-ray crystal structure determinations. Crystal data: **1**, monoclinic, space group $P2_1/c$ (C^5_{2h}), a = 12.412(1) Å, b = 17.306(1) Å, c = 20.238(2) Å, β = 119.34(1)°, V = 3790(1) Å^3, Z = 4; **2**, monoclinic, space group $P2/c$ (C^4_{2h}), a = 12.180(1) Å, b = 12.744(1) Å, c = 19.618(2) Å, β = 128.43(1)°, V = 2386(1) Å^3, Z = 2; **3**, monoclinic, space group $P2_1/c$ (C^5_{2h}), a = 12.486(1) Å, b = 12.544(1) Å, c = 19.985(2) Å, β = 101.73(1)°, V = 3065(1) Å^3, Z = 4.

Introduction

Recent work in our research group involving the formation of gallium-arsenic bonds has resulted in the preparation of the novel ring compounds $Ph_2\overline{GaAs(SiMe_3)_2Ga(Ph)_2X}$ [X = Cl (**4**)[1,2], Br (**5**)[2]] and $(Me_3SiCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2SiMe_3)_2Cl}$ (**6**)[3], as well as the dimers $[Ph_2GaAs(SiMe_3)_2]_2$ (**7**)[1,2] and $[(Me_3SiCH_2)_2GaAs(SiMe_3)_2]$ (**8**)[3]. In addition, we very recently reported the isolation of the indium-arsenic[4] and indium-phosphorus[5] analogs of **6** and **8**, $(Me_3SiCH_2)_2\overline{InAs(SiMe_3)_2In(CH_2SiMe_3)_2Cl}$ and $[(Me_3SiCH_2)_2InAs(SiMe_3)_2]_2$, and $(Me_3SiCH_2)_2\overline{InP(SiMe_3)_2In(CH_2SiMe_3)_2Cl}$ and $[(Me_3SiCH_2)_2InP(SiMe_3)_2]_2$, respectively. As part of a continuing effort to expand the scope of the gallium-arsenic series, the neopentyl group was chosen as the alkyl substituent on the gallium atoms with the expectation that its different steric bulk might manifest itself in a different ring

conformation or size in comparison to that of the previously studied (trimethylsilyl)methyl group. We also sought to investigate more fully the reactivity and interconvertibility of these types of compounds. We present herein our results obtained with the neopentyl ligand system.

Experimental Section

General Considerations. All manipulations and reactions were performed under vacuum, under argon in a Vacuum/Atmospheres HE-43 Dri-Lab, or under N_2 in standard Schlenk apparatus. Solvents were distilled from sodium/benzophenone ketyl under dry N_2 and degassed by several freeze-pump-thaw cycles. $(Me_3CCH_2)_2GaCl$,[6] $(Me_3Si)_3As$,[7] and $LiAs(SiMe_3)_2 \cdot 2THF$[7] were prepared by literature procedures. ^1H (300 MHz) and $^{13}C\{^1H\}$ (75.4 MHz) NMR spectra were recorded on a Varian XL-300 spectrometer at ambient temperatures using C_6D_6 or C_7D_8 solutions in 5 mm tubes which were flame-sealed under vacuum and were referenced to TMS using the residual protons or the carbons of deuterated benzene at δ 7.15 ppm or δ 128 ppm, respectively, or versus the upfield pentet of C_7D_8 at δ 2.09 ppm for ^1H NMR. Melting points (uncorrected) were obtained with a Thomas-Hoover Uni-melt apparatus and capillaries were flame-sealed under argon. Crystals used in the x-ray analyses were flame-sealed in 0.7 mm thin-walled glass capillaries. Elemental analyses were performed by E+R Microanalytical Laboratory, Inc., Corona, New York.

Preparation of $(Me_3CCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2}Cl$ (1). Reaction of $(Me_3CCH_2)_2GaCl$ with $(Me_3Si)_3As$ (2:1 Mole Ratio). $(Me_3CCH_2)_2GaCl$ (0.247 g, 1.00 mmol) in 20 mL of benzene and $(Me_3Si)_3As$ (0.147 g, 0.500 mmol) in 5 mL of benzene were combined in a 100-mL one-necked round-bottomed flask equipped with a Teflon® valve and a

magnetic stirbar. After one freeze-pump-thaw cycle of the solution, followed by stirring at room temperature for 5 days, removal of the volatiles *in vacuo* afforded a crude product which was dissolved in a small amount of pentane. On standing overnight at -15 °C, a very small amount of white material [mp 170 °C (dec); ^{1}H NMR (C_6D_6): δ 0.53 (s, 18 H, Si*Me*$_3$), 1.32 (s, 9 H, C*Me*$_3$), 1.42 (s, 2 H, CH_2)] precipitated from the pentane solution and, after filtration and concentration, cooling the mother liquor to -15 °C afforded colorless crystals of **1** suitable for X-ray analysis (0.14 g, 41% yield), mp 70-110 °C (dec). Anal. Calcd (Found) for $C_{26}H_{62}AsClGa_2Si_2$: C, 45.87 (45.74); H, 9.17 (9.27). ^{1}H NMR (C_6D_6): δ 0.45 (s, 18 H, Si*Me*$_3$), 1.27 (s, 36 H, C*Me*$_3$), 1.38 (s, 8 H, CH_2). ^{13}C {^{1}H} NMR (C_6D_6): δ 4.71 (Si*Me*$_3$), 32.92 (Me_3C), 34.50 (*Me*$_3$C), 40.57 (CH_2).

Preparation of [(Me$_3$CCH$_2$)$_2$GaAs(SiMe$_3$)$_2$]$_2$ (2). (a) Reaction of (Me$_3$CCH$_2$)$_2$GaCl with LiAs(SiMe$_3$)$_2$•2THF. Addition of a solution of $LiAs(SiMe_3)_2$•2THF (0.283 g, 0.800 mmol) in 10 mL of benzene to a solution of $(Me_3CCH_2)_2GaCl$ (0.200 g, 0.800 mmol) in 20 mL of benzene contained in a 100-mL one-necked round-bottomed flask equipped with a Teflon valve and a micro-stirbar gave a light brown reaction mixture which was stirred at room temperature for 24 h. Removal of the benzene *in vacuo* was followed by addition of 30 mL of pentane to the brownish-white residue; filtration and subsequent removal *in vacuo* of the pentane from the filtrate afforded crude product (**2**) (0.28 g, 82% yield). Cooling a pentane (5 mL) solution of the latter to -15 °C gave colorless crystals suitable for X-ray analysis, m.p. 160-170 °C. Anal. Calcd (Found) for $C_{32}H_{80}As_2Ga_2Si_4$: C, 44.35 (44.49); H, 9.30 (9.26). ^{1}H NMR (C_6D_6): δ 0.60 (s, 36 H, Si*Me*$_3$), 1.30 (s, 36 H, C*Me*$_3$), 1.47 (s, 8 H, CH_2).

(b) Reaction of 1 with $LiAs(SiMe_3)_2 \cdot 2THF$. Addition of a solution of $LiAs(SiMe_3)_2 \cdot 2THF$ (0.018 g, 0.050 mmol) in toluene-d_8 (1 mL) to a solution of **1** (0.034 g, 0.050 mmol) in toluene-d_8 (1 mL) contained in a 50-mL one-necked round-bottomed flask equipped with a Teflon valve resulted in the formation of white precipitate (presumably LiCl). After standing in the dry box for 12 h, followed by removal of the toluene-d_8 *in vacuo* and replacement with pentane (15 mL), the insoluble white solid was separated by filtration. Cooling the concentrated filtrate to -15 °C afforded **2** in nearly quantitative yield (mp and ^1H NMR spectrum same as those of an authentic sample of **2** *vide supra*).

Preparation of $(Me_3CCH_2)_2GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2Cl$ (1). Reaction of $(Me_3CCH_2)_2GaCl$ with 2 (2:1 Mole Ratio). Ten minutes after combining $(Me_3CCH_2)_2GaCl$ (7 mg, 3 x 10^{-2} mmol) and **2** (12 mg, 1.4 x 10^{-2} mmol) in C_6D_6 (1 mL) in an NMR tube, the ^1H NMR spectrum of the reaction mixture was identical to that of an authentic sample of **1**, thereby indicating there was complete conversion of the starting materials to **1**. The subsequently obtained $^{13}C\{^1H\}$ NMR spectrum was also identical to that of an authentic sample of **1**.

Preparation of $(Me_3CCH_2)_2Ga(Cl) \cdot As(SiMe_3)_3$ (3). Reaction of $(Me_3CCH_2)_2GaCl$ with $(Me_3Si)_3As$ (1:1 mole ratio). $(Me_3CCH_2)_2GaCl$ (0.247 g, 1.00 mmol) in 20 mL of benzene and $(Me_3Si)_3As$ (0.295 g, 1.00 mmol) in 5 mL of benzene were combined in a 100-mL one-necked round-bottomed flask equipped with a Teflon valve and a magnetic stirbar. After one freeze-pump-thaw cycle of the solution, followed by stirring at room temperature for 5 days, removal of the solvent *in vacuo* afforded a white solid which was dissolved in a small amount of pentane and, on storing at -15 °C, large colorless crystals of **3** suitable for X-ray analysis were

deposited (0.20 g, 40% yield), mp 78 °C (dec at 130 °C to a red material). Anal. (satisfactory analysis could not be obtained due to decomposition of sample). Calcd (Found) for $C_{19}H_{49}AsClGaSi_3$: C, 42.11 (37.97); H, 9.11 (8.08); Cl, 6.54 (3.32). ^1H NMR (C_6D_6): δ 0.34 (s, 27 H, Si*Me*$_3$), 1.23 (s with shoulder, 22 H, CH_2CMe_3). ^{13}C {^1H} NMR (C_6D_6): δ 3.49 (*Me*$_3$Si, 34.14 (*Me*$_3$C) [the methylene (CH_2) and tertiary (Me_3C) carbons was not observed).

Thermolysis of $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (3). A flame-sealed NMR tube containing **3** (10 mg) dissolved in C_7D_8 was heated at 110-120 °C over a period of 43 h with ^1H NMR spectra being recorded at various intervals. The resonance in the initial ambient temperature spectrum associated with **3** at δ 1.20 was replaced by one at δ 1.09 and a series of other peaks at δ 0.19 (possibly due to Me_3SiCl), 0.45, 0.54, and 1.03; however the one at δ 0.35 remained as a major peak. No resonances that could be attributed to **2** were evident.

Reaction of $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (3) with $(Me_3CCH_2)_2GaCl$ (1:1 mole ratio). $(Me_3CCH_2)_2GaCl$ (5 mg, 0.02 mmol) and **3** (95 mg, 0.02 mmol) were dissolved in C_7D_8 in an NMR tube. The ambient temperature ^1H NMR spectrum recorded immediately after combining the reactants and flame-sealing the tube, as well as that obtained 19 h later, was relatively simple with three major peaks at δ 0.35, 1.17, 1.23 which correspond to those associated with the adduct. By integration, the $(Me_3CCH_2)_2GaCl$ protons appeared to be degenerate with those of the adduct. After heating at 80 °C for 5 h and observing very little change in the spectrum, the mixture was heated at 100 °C for 29 h and new major resonances were observed at δ 1.09, 1.03, 0.54, 0.45, and 0.19 (possibly due to Me_3SiCl), with minor peaks being observed at δ1.27

and 1.38. The latter two peaks along with the one at 0.45 can be assigned to **1**, but the relative areas are not correct for that compound.

Structural Analyses of 1, 2, and 3. Crystallographic data and data collection parameters are presented in Table 1. Intensity data were corrected for the usual Lorentz and polarization effects; empirical absorption corrections, and for **2** a linear decay correction, were also applied. Each crystal structure was solved by direct methods (MULTAN11/82). For **1**, approximate non-hydrogen atom positions were obtained in part from an E-map and completed by a series of difference Fourier syntheses phased successively by an increasing number of atoms. In the case of **2**, with only two formula units in the unit cell of space group $P2/c$, the dimer is required to lie either on a center of symmetry or a C_2 axis. An E-map yielded Ga, As, and Si atom coordinates and, moreover, established that the As atoms lay on a crystallographic C_2 symmetry axis; C atoms were subsequently located in a difference Fourier synthesis. Approximate As, Ga, Cl, and Si atom positions for **3** were derived routinely from an E-map, and a weighted F_o Fourier synthesis phased by these atoms revealed C atom locations. Positional and thermal parameters (at first isotropic, then anisotropic) for each compound were adjusted by means of several rounds of full-matrix least-squares calculations. Hydrogen atoms were incorporated at their calculated positions (C-H = 1.05 Å) and an extinction correction was included as a variable during the final least-squares iterations. For structure-factor calculations, neutral atom scattering factors and their anomalous dispersion corrections were taken from reference 8.

Results and Discussion

Tris(trimethylsilyl)arsine, $(Me_3Si)_3As$, reacts with two equivalents of $(Me_3CCH_2)_2GaCl$ in benzene at room temperature to produce the mixed-bridge compound $(Me_3CCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2}Cl$ (**1**) in 41% crystalline yield (see Scheme I for a summary of this and the other reactions investigated in this study). This result is in accord with those obtained when the aryl/alkyl groups on the gallium starting material are phenyl or (trimethylsilyl)methyl, furnishing **4** and **6**, respectively. Compound **1** was characterized by ^1H and $^{13}C\{^1H\}$ NMR spectroscopy, partial elemental analysis, and single-crystal X-ray analysis (*vide infra*).

On the other hand, in stark contrast to the Ph_2GaCl and $(Me_3SiCH_2)_2GaCl$ cases, the reaction of $(Me_3Si)_3As$ with one equivalent of $(Me_3CCH_2)_2GaCl$ produces the 1:1 adduct $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (**3**) in 40% crystalline yield. The corresponding equimolar reaction with Ph_2GaCl leads to a mixture of mixed-bridge **4** and dimer **7**,[1] whereas the 1:1 reaction between the silylarsine and $(Me_3SiCH_2)_2GaCl$ affords mixed-bridge **6** and other uncharacterized products, but not dimer **8**.[3] These results led us to consider the possibility that **3** may be an intermediate in the synthesis of **1** or in the synthesis of the dimer $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]_2$ (**2**). To investigate this hypothesis, a toluene-d_8 solution of **3** sealed in an NMR tube was heated in an effort to eliminate Me_3SiCl and produce **2**; however, based on the NMR spectra obtained, only a small amount of what appeared to be Me_3SiCl and other products were present but, significantly, there was no evidence for **2** or **1**. A similar NMR tube reaction between **3** and one equivalent of $(Me_3CCH_2)_2GaCl$ at elevated temperatures gave what appeared to be the same products plus a trace of **1**, but no evidence for **2**. It thus appears that adduct **3** is not an

intermediate in the preparation of **2** and it is not likely that it is the predominate intermediate, in the preparation of **1**. By inference, analogous hypothetical adducts may not be intermediates in the synthesis of **4**, **6**, **7**, or **8**. Interestingly, it was eventually possible to characterize adduct **3** by ^{1}H NMR spectroscopy, and an X-ray crystallographic analysis (*vide infra*), but it was too unstable to allow a satisfactory elemental analysis to be obtained.

In a manner completely analogous to that previously reported by us to prepare **7**[2] and **8**,[3] dimer **2** was synthesized in 82% yield from the reaction of $(Me_3CCH_2)_2GaCl$ with $LiAs(SiMe_3)_2 \cdot 2THF$, and it was characterized by ^{1}H NMR spectroscopy, partial elemental analysis, and a single-crystal X-ray analysis (*vide infra*). This dimer can also be prepared by reaction of mixed-bridge **1** with one equivalent of the same lithium arsenide. Conversely, **2** reacts with two equivalents of $(Me_3CCH_2)_2GaCl$ to produce mixed-bridge **1**. The aforementioned conversion of **1** to **2** is the first reported example of the replacement of the chlorine atom in the Ga-As-Ga-Cl ring species by a salt-elimination reaction. The facile manner in which this reaction occurred indicates that other replacements may be possible, and efforts are being made to prepare compounds which contain two gallium centers bridged by two different Group V elements, another class of mixed-bridge compounds not yet reported.

For X-ray structural characterization of **1**, **2**, and **3**, crystals were grown from pentane solutions. Crystallographic data and data collection parameters are summarized in Table I. ORTEP diagrams showing the atom numbering schemes for **1**, **2**, and **3** are presented in Figures 1-3, respectively. Tables II-IV list the non-hydrogen atom fractional

coordinates and equivalent isotropic thermal parameters, while selected bond lengths for **1**, **2**, and **3** are provided in Tables V-VII, respectively.

Compound **1** crystallizes in the monoclinic system with four molecules occupying the general positions of the centrosymmetric space group $P2_1/c$. Atoms of the Ga-As-Ga-Cl ring deviate by only ±0.001 Å from their least-squares plane and thus they are coplanar. This geometry contrasts with the puckered forms encountered in the other previously reported gallium-arsenic-halogen mixed bridge compounds **4**,[1,2] **5**,[2] and **6**,[3] in all three of which the halogen atom is displaced significantly (Δ 0.256 Å, 0.293 Å, and 0.860 Å, respectively) from the Ga-As-Ga' plane. The mean Ga-As bond distance at 2.521 Å in the planar ring of **1** is longer than the mean of 2.466 Å in slightly puckered **4** but only slightly longer than the value of 2.504 Å in the very puckered ring of **6**. Less variation is apparent in the mean Ga-Cl distances in these same compounds (2.425 Å in **1**, 2.411 Å in **4**, 2.432 Å in **6**). The Ga-Cl-Ga angle at 94.41(4)° in **1** is considerably greater than the essentially equal values in **4** [91.3(1)°] and **6** [91.33(4)]°. The mean exocyclic C-Ga-C bond angle at 127.7° in **1** is also much enlarged over the corresponding angles in either **4** (121.3°) or **6** (123.6°).

Crystals of dimer **2** belong to the monoclinic system, space group $P2/c$, with the As atoms lying on a crystallographic C_2 axis of symmetry, and thus the Ga-As-Ga-As ring is strictly planar. The mean Ga-As bond length at 2.587 Å in **2** is only slightly longer than that of 2.567 Å in **8**. The Ga-As distances in dimeric **2** and **8** exceed those in the corresponding halogen-bridged species by comparable amounts (Δ = 0.066 Å for **2** vs. **1**; Δ = 0.063 Å for **8** vs. **6**), reflecting the more severe nature of the bond strain in group III-group V dimers vs. their chloro-bridged counterparts. Moreover, the Si-As-Si angle at 102.32(7)° in dimeric **2** is smaller than

that of 105.39(5)° in chloro-bridged **1**, indicating the increased steric compression present in the former. Like differences in corresponding bond lengths (Δ 0.051 Å, 0.052 Å) and angles (Δ 3.3°, 5.4°) have also been found recently in pairs of In-As and In-P analogues $[(Me_3SiCH_2)_2InM(SiMe_3)_2]_2$ and $(Me_3SiCH_2)_2InM(SiMe_3)_2In(CH_2SiMe_3)_2Cl$, M = As,[4] P[5].

Adduct **3** crystallizes in the centrosymmetric space group $P2_1/c$ with the four molecules in the unit cell lying in general positions. In the solid state, this molecule adopts the staggered conformation depicted in Figure 4. Surprisingly, a search of the literature revealed that although a number of gallium-arsenic adducts have been reported,[9] no complete crystal structure analysis has yet appeared. Consequently, the only Ga-As bond length comparison possible is with those found in cyclic species containing four-coordinate Ga and As atoms; the distance in **3** at 2.626(1) Å is longer than any of the Ga-As lengths in such compounds (see preceding discussion and cited references). The mean As-Si bond length at 2.367 Å lies close to the essentially equal values of 2.358 Å in **1** and 2.363 Å in **2**, whereas the mean Ga-C bond length at 1.997 Å is identical with that of 1.997 Å in **2** but longer than that of 1.966 Å in **1**. The Ga-Cl bond length at 2.242(1) Å is significantly shorter than the means of 2.411 Å - 2.432 Å in the Ga-As-Ga-Cl rings of **1**, **4**, and **6**. Intramolecular non-bonded interactions between substituents at As and Ga are minimized by a combination of bond angle deformation and rotation around the As-Ga bond. Thus, enlargement of the Cl-Ga-C and C-Ga-C angles (mean 116.4°), due at least in part to electronic effects, results in steric compression at the arsenic atom (mean Si-As-Si angle = 106.06° < mean Ga-As-Si angle = 112.70°). Rotation around the Ga-As bond aids in producing approximately

equidistant C...C separations of ca. 4.0 Å between the carbon atoms in each of the CH_2 groups at Ga and those in pairs of Me groups of the $SiMe_3$ substituents at As.

Conclusions

The facile preparation of compound **1** adds another member to the growing list of mixed-bridge compounds involving the heavier elements of Groups III and V. In addition, we believe it is significant that it has been clearly demonstrated that the Ga-As-Ga-Cl ring of **1** is easily converted to the Ga-As-Ga-As ring of **2**. This demonstrates the feasibility of using the same methodology to prepare compounds containing the Ga-As-Ga-E ring (E = N, P, or Sb). Moreover, the fact that adduct **3** was isolated from the 1:1 mole ratio reaction between $(Me_3CCH_2)_2GaCl$ and $(Me_3Si)_3As$, and that it could not be readily converted to **1** or **2**, gives some indication that as the bulk of the ligands on gallium increases, dehalosilyation occurs less readily and the adduct is not necessarily an intermediate in the formation of **1** and **2**.

Acknowledgment. The financial support for this work by the Office of Naval Research is gratefully acknowledged.

Supplementary Material Available: Tables of hydrogen atom coordinates and isotropic thermal parameters, anisotropic temperature factors, complete lists of interatomic distances and angles, including torsion angles, for **1**, **2** and **3**, as well as equations of least-squares planes through groups of atoms for **1** (20 pages); a listing of observed and calculated structure amplitudes for **1**, **2**, and **3** (89 pages). Ordering information is given on any current masthead page.

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Captions to Figures

Figure 1. ORTEP diagram (40% probability ellipsoids) showing the atom numbering scheme and solid-state conformation of $(Me_3CCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2SiMe_3)_2Cl}$ (**1**); hydrogen atoms have been omitted for clarity.

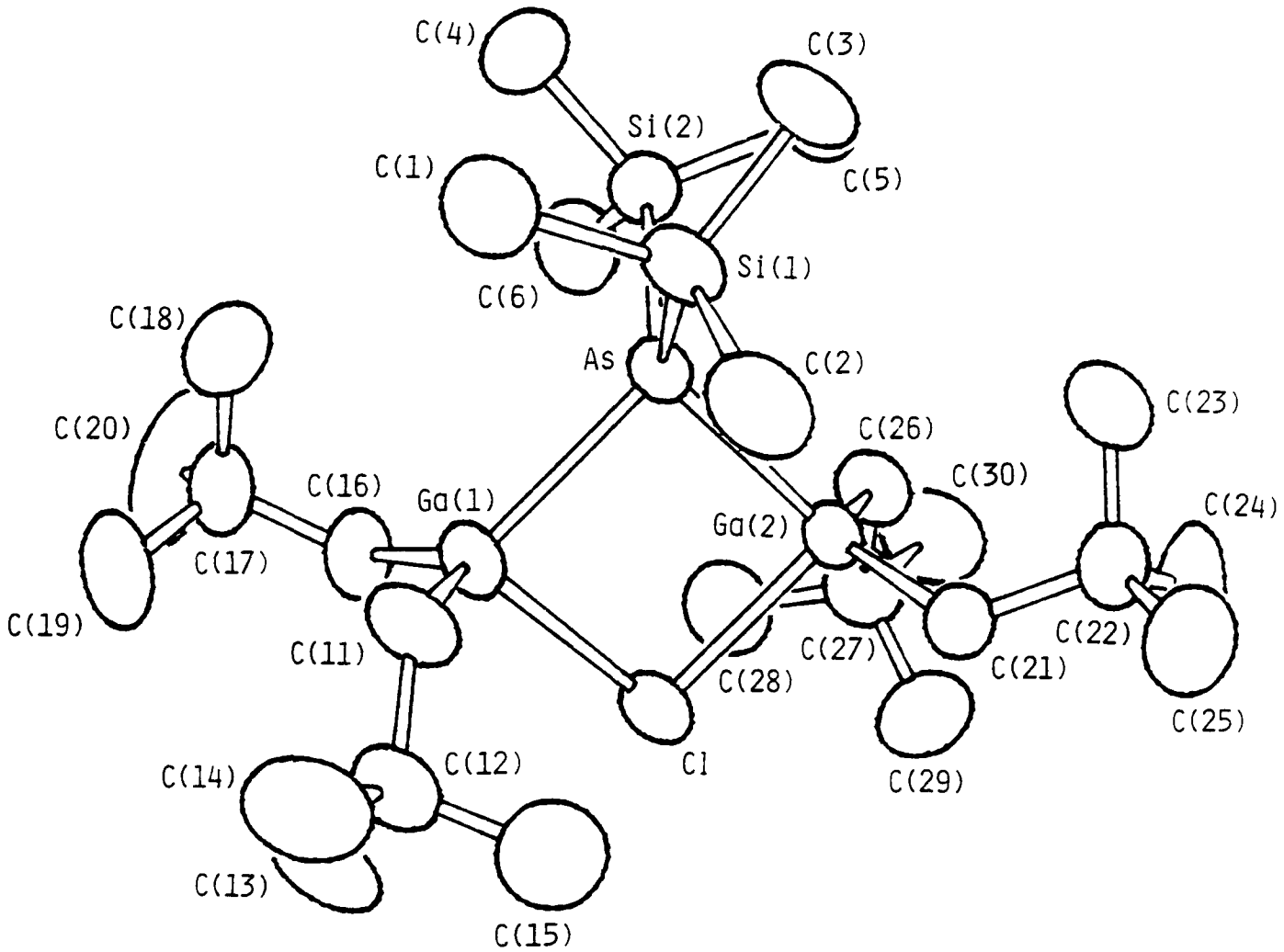
Figure 2. ORTEP diagram (40% probability ellipsoids) showing the atom numbering scheme and solid-state conformation of $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]$ (**2**); hydrogen atoms have been omitted for clarity. Primed atoms are related to the unprimed atoms by a crystallographic C_2 symmetry axis which passes through As(1) and As(2).

Figure 3. ORTEP diagram (40% probability ellipsoids) showing the atom numbering scheme and solid-state conformation of $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (**3**); hydrogen atoms have been omitted for clarity.

Figure 4. Newman projection down the Ga-As bond in $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$.

Caption to Scheme

Scheme I. Reaction Summary: Synthesis and Reactivity of $(Me_3CCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2Cl}$ (**1**), $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]_2$ (**2**), and $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (**3**).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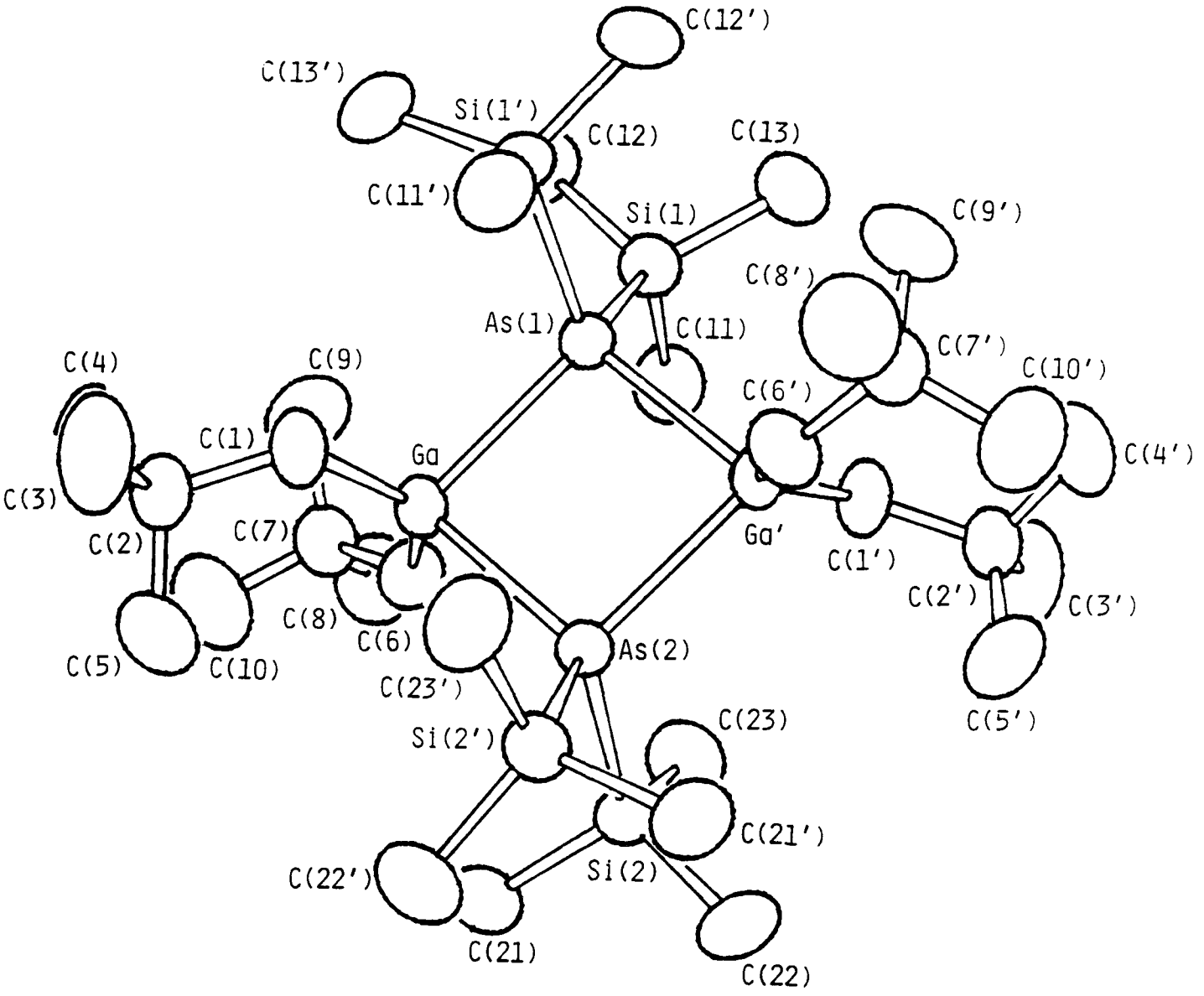
Si(2)
C(1)
C(5)
Si(1)
C(6)
C(18)
As
C(2)
C(20)
C(26)
C(23)
C(30)
C(16)
Ga(1)
Ga(2)
C(24)
C(17)
C(19)
C(11)
C(27)
C(22)
C(28)
C(21)
C(25)
C(14)
C(12)
Cl
C(29)
C(13)
C(15)

Figure 1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C(13')
Si(1')
C(12)
C(13)
C(11')
Si(1)
C(9')
C(8')
As(1)
C(11)
C(7')
C(10')
C(4)
C(9)
C(6')
C(1)
Ga
C(4')
C(3)
Ga'
C(7)
C(1')
C(2)
C(2')
C(3')
C(8)
C(6)
As(2)
C(5)
C(10)
C(23')
C(5')
Si(2')
C(23)
C(21')
C(22')
Si(2)
C(21)
C(22)

Figure 2

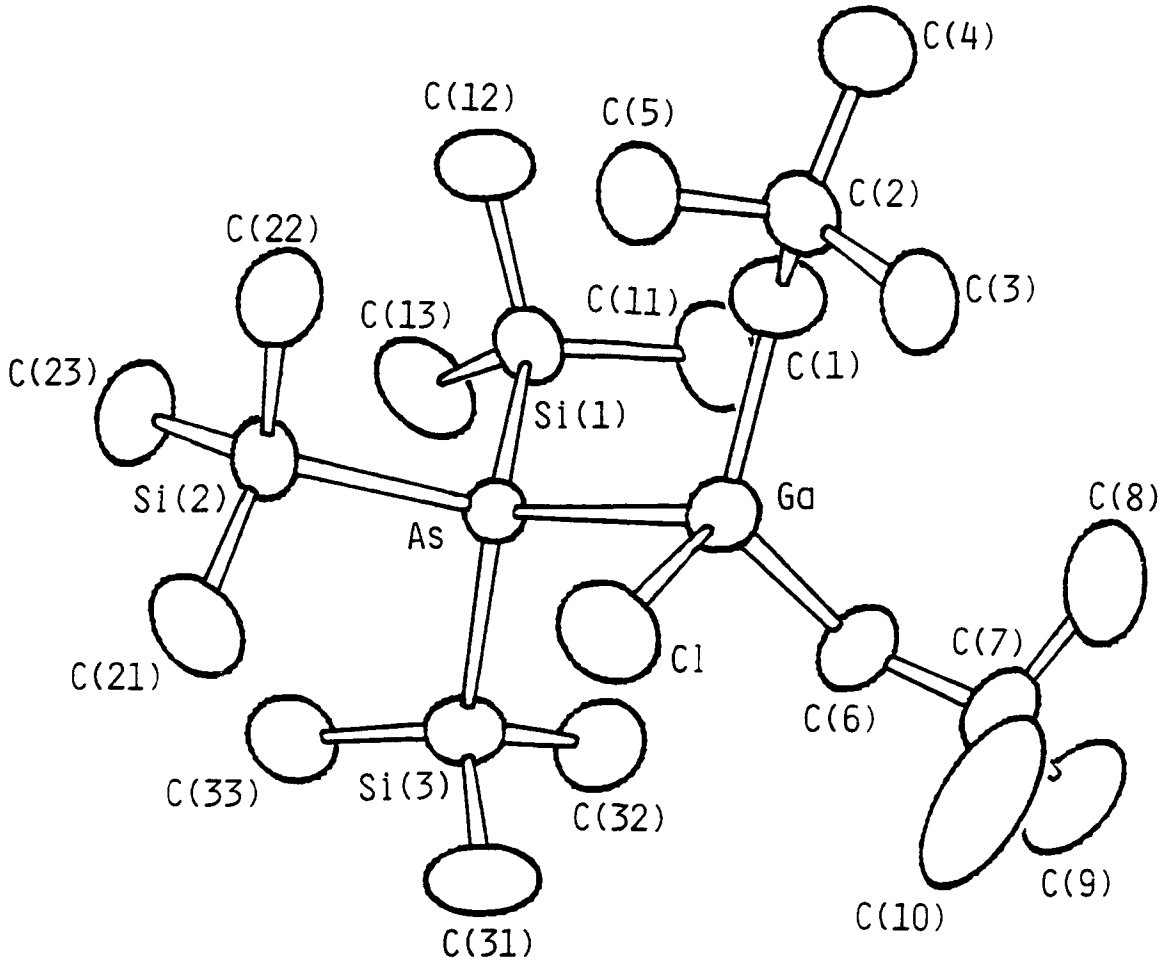

C(4)
C(12)
C(5)
C(2)
C(22)
C(3)
C(13)
C(11)
C(1)
C(23)
Si(1)
Si(2)
Ga
C(8)
As
C(21)
Cl
C(7)
C(6)
C(33)
Si(3)
C(32)
C(9)
C(10)
C(31)

Figure 3

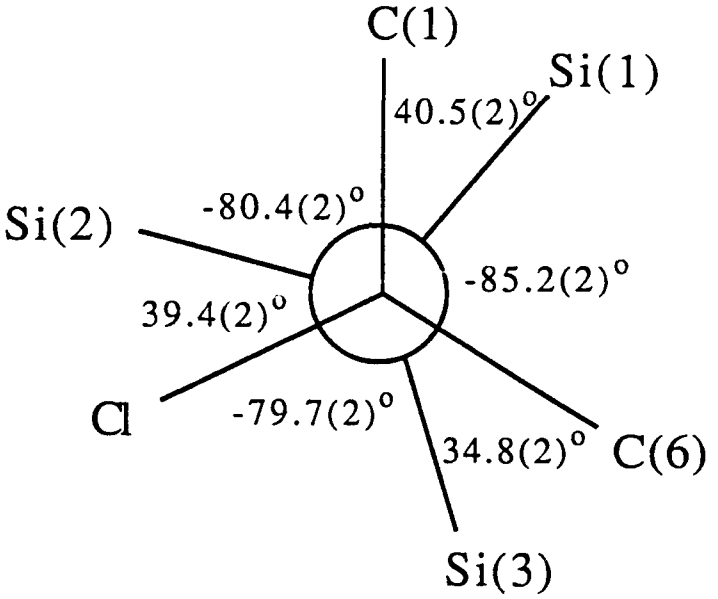
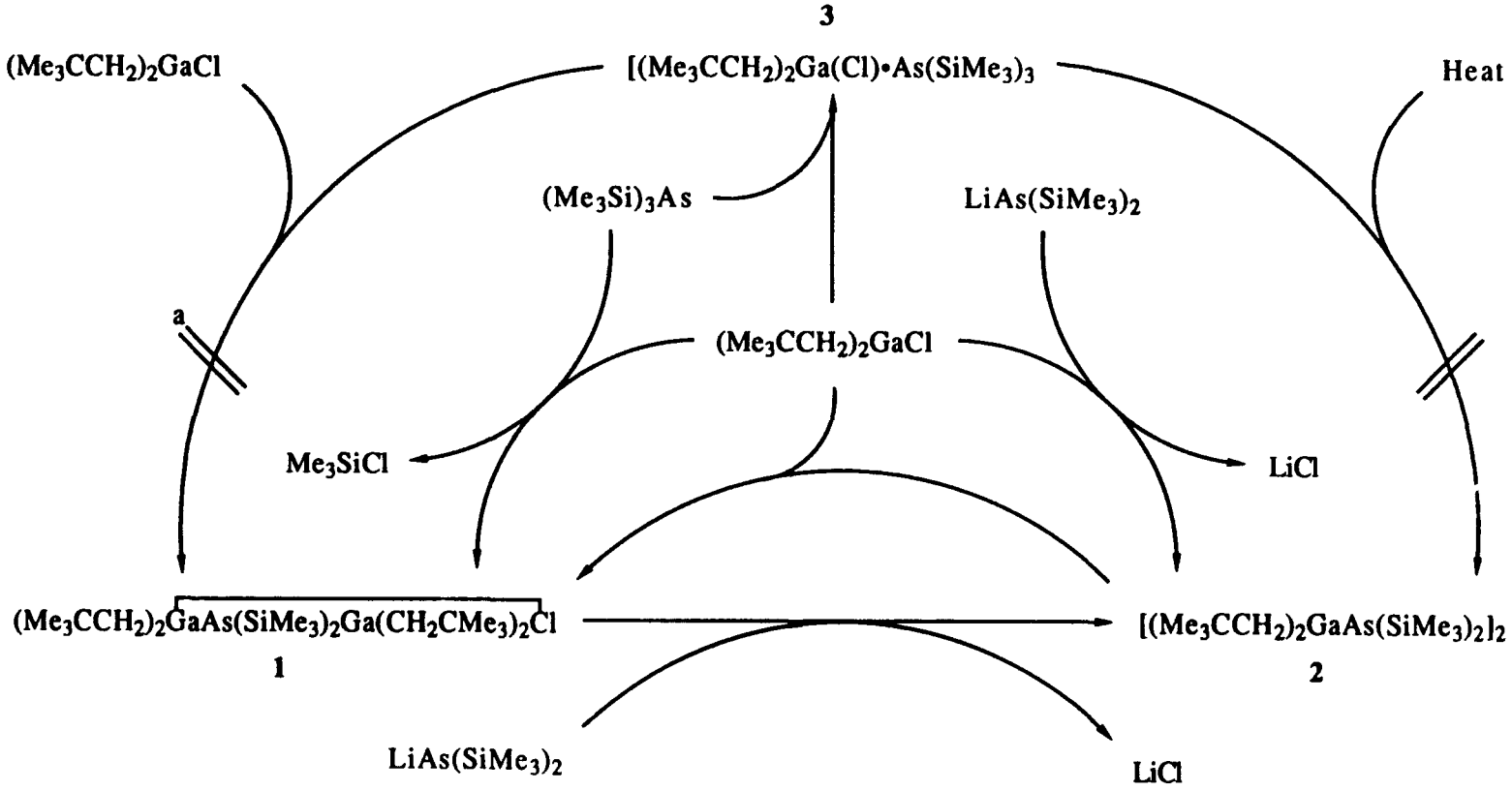


Figure 4



[a] Complex ^1H NMR spectrum of reaction mixture contains peaks which could be attributed to **1**

Scheme 1

Table I. Crystallographic Data and Measurements[a] for $(Me_3CCH_2)_2GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2Cl$ (1), $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]_2$ (2), and $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (3)

	1	2	3
mol formula	$C_{26}H_{62}AsClGa_2Si_2$	$C_{32}H_{80}As_2Ga_2Si_4$	$C_{19}H_{49}AsClGaSi_3$
formula wt	685.97	866.62	541.96
crystal system	monoclinic	monoclinic	monoclinic
space group	$P2_1/c(C^5_{2h})$	$P2/c(C^4_{2h})$	$P2_1/c(C^5_{2h})$
a, Å	12.412(1)	12.180(31)	12.486(1)
b, Å	17.306(1)	12.744(1)	12.544(2)
c, Å	20.238(2)	19.618(2)	19.9851)
β, deg	119.34(1)	128.43(1)	101.73.(1)
no. of orient. refls; θ, deg, range	25; 42-48	25; 42-48	25; 45-48
V, Å^3	3790(1)	2386(1)	3065(1)
Z	4	2	4
D_{calcd}, g cm^{-3}	1.202	1.206	1.174
temp, °C	25	25	25
crystal dims, mm	0.40x0.50x0.60	0.40x0.40x0.60	0.4x0.50x0.60
T_{max}:T_{min}	1.00:0.68	1.00:0.51	1.00:0.61
radiation (wavelength)	Cu-*K*α (1.5418 Å)	Cu-*K*α (1.5418 Å)	Cu-*K*α (1.5418 Å)
μ, cm-1	43.0	40.4	44.1
scan type	ω-2θ	ω-2θ	ω-2θ
scan width, deg	1.10 + 0.14tanθ	1.10 + 0.14tanθ	1.10 + 0.14tanθ

Table I. (continued)

θ_{max}, deg	75	75	75
intensity control reflns	$15\bar{1}$, $34\bar{1}$, 126, 208	$35\bar{5}$, $52\bar{2}$, 333, 262	224, $2\bar{2}4$, $2\bar{2}\bar{4}$, $22\bar{4}$
variation; repeat time, h	<2%; 2	-36%; 1	<2%; 2
no of rflns recorded	8216 ($+h,+k,\pm l$)	5138 ($+h,+k,\pm l$)	6594 ($+h,+k,\pm l$)
no of non-equiv reflns recorded	7775	4906	6299
R_{merge} (on I)	0.026	0.043	0.036
no of rflns retained, $I > 3.0\sigma(I)$	6153	2496	4351
no of parameters refined	290	183	227
extinction correction	$8.1(3)\times10^{-7}$	$1.55(5)\times10^{-6}$	$2.44(3)\times10^{-6}$
R (R_w)[b]	0.045 (0.065)	0.054 (0.072)	0.045 (0.060)
goodness-of-fit[c]	2.36	1.48	1.39
max shift; esd in final least-squares cycle	0.02	0.02	0.02
final $\Delta\rho$(e/Å^3) max; min	0.75; -0.69	1.05; -0.70	0.45; -0.62

[a] An Enraf-Nonius CAD-4 diffractometer equipped with a graphite monochromater was used for all measurements. Crystallographic calculations were performed on PDP11/44 and MicroVAX computers by use of the Enraf-Nonius Structure Determination Package (SDP).

[b] $R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$; $R_w = [\Sigma w\ (|F_o| - |F_c|)^2/\Sigma w\ |F_o|^2]^{1/2}$; $\Sigma w\Delta^2 [w = 1/\sigma^2(|F_o|), \Delta = (|F_o| - |F_c|)]$ was minimized.

[c] Goodness-of-fit = $[\Sigma w\Delta^2 / (N_{observations} - N_{parameters})]^{1/2}$.

Table II. Non-hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters for $(Me_3CCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2}Cl$ (1), with Estimated Standard Deviations in Parentheses.

atom	*x*	*y*	*z*	B_{eq}(Å^2)
As	0.33519(3)	0.26603(2)	0.29967(2)	3.68(1)
Ga(1)	0.55433(3)	0.24109(3)	0.40315(2)	4.24(1)
Ga(2)	0.29019(4)	0.12380(3)	0.29344(2)	4.04(1)
Cl	0.50217(8)	0.10515(7)	0.39309(6)	5.45(2)
Si(1)	0.33287(9)	0.33054(8)	0.19622(6)	5.05(3)
Si(2)	0.19465(9)	0.33408(7)	0.32466(6)	4.93(2)
C(1)	0.4319(5)	0.4180(4)	0.2337(3)	7.8(1)
C(2)	0.3991(4)	0.2663(4)	0.1504(2)	7.4(1)
C(3)	0.1722(4)	0.3586(4)	0.1244(3)	7.1(1)
C(4)	0.2149(5)	0.4408(3)	0.3198(3)	7.8(2)
C(5)	0.0349(4)	0.3043(3)	0.2511(3)	6.6(1)
C(6)	0.2196(4)	0.3105(4)	0.4211(3)	7.1(1)
C(11)	0.6711(3)	0.2577(3)	0.3635(2)	5.6(1)
C(12)	0.7764(3)	0.2014(3)	0.3805(2)	5.8(1)
C(13)	0.8403(5)	0.1732(5)	0.4620(4)	9.1(2)
C(14)	0.8676(5)	0.2401(5)	0.3616(4)	10.7(2)
C(15)	0.7250(5)	0.1310(5)	0.3297(4)	9.7(2)
C(16)	0.5838(4)	0.2598(3)	0.5071(2)	5.8(1)
C(17)	0.6402(4)	0.3368(3)	0.5457(2)	6.0(1)
C(18)	0.5726(6)	0.4042(4)	0.4921(4)	8.9(2)
C(19)	0.7727(6)	0.3450(5)	0.5641(4)	10.1(2)
C(20)	0.6279(7)	0.3472(5)	0.6165(3)	13.9(2)
C(21)	0.3024(4)	0.0693(3)	0.2117(2)	5.5(1)
C(22)	0.1839(5)	0.0490(3)	0.1373(3)	6.9(1)
C(23)	0.1077(6)	0.1226(4)	0.1034(4)	8.9(2)
C(24)	0.1072(7)	-0.0101(4)	0.1494(4)	11.6(3)
C(25)	0.2214(7)	0.0179(5)	0.0802(3)	10.7(2)
C(26)	0.1759(3)	0.1010(3)	0.3336(2)	5.3(1)
C(27)	0.2070(4)	0.0387(3)	0.3950(3)	6.2(1)
C(28)	0.3013(5)	0.0697(4)	0.4732(3)	8.0(2)
C(29)	0.2570(6)	-0.0341(3)	0.3774(3)	8.4(2)
C(30)	0.0889(5)	0.0181(4)	0.3977(4)	9.7(2)

Table III. Non-hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters for $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]_2$ (2), with Estimated Standard Deviations in Parentheses.

atom	*x*	*y*	*z*	B_{eq}(Å2)
As(1)	0.00000(-)[a]	0.11924(7)	0.25000(-)[a]	3.73(2)
As(2)	0.00000(-)[a]	0.39360(7)	0.25000(-)[a]	3.60(2)
Ga	-0.03287(5)	0.25610(6)	0.14143(3)	3.95(2)
Si(1)	-0.18629(14)	0.00320(14)	0.20167(9)	5.02(4)
Si(2)	-0.19277(14)	0.50923(14)	0.18669(9)	4.59(4)
C(1)	0.1440(5)	0.2395(6)	0.1566(3)	5.5(2)
C(2)	0.1675(5)	0.2798(5)	0.0939(3)	5.2(2)
C(3)	0.3247(6)	0.2788(9)	0.1384(5)	9.9(3)
C(4)	0.0968(7)	0.2115(8)	0.0154(4)	8.6(3)
C(5)	0.1145(7)	0.3917(7)	0.0678(4)	8.5(3)
C(6)	-0.2349(6)	0.2767(6)	0.0400(4)	5.7(2)
C(7)	-0.3127(6)	0.2270(6)	-0.0503(3)	5.5(2)
C(8)	-0.4731(8)	0.2336(7)	-0.0961(5)	8.8(3)
C(9)	-0.2701(7)	0.1127(7)	-0.0422(5)	8.1(3)
C(10)	-0.2872(8)	0.2871(9)	-0.1054(5)	9.1(3)
C(11)	-0.3564(6)	0.0765(7)	0.1373(4)	6.7(3)
C(12)	-0.1968(6)	-0.1020(6)	0.1327(4)	7.3(2)
C(13)	-0.1630(6)	-0.0595(6)	0.2959(4)	7.0(2)
C(21)	-0.2307(6)	0.5764(6)	0.0898(4)	6.0(2)
C(22)	-0.1545(6)	0.6126(6)	0.2666(4)	7.2(2)
C(23)	-0.3542(6)	0.4355(7)	0.1490(4)	7.1(2)

[a]Fixed by symmetry.

Table IV. Non-hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters for $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (3), with Estimated Standard Deviations in Parentheses.

atom	*x*	*y*	*z*	B_{eq}(Å^2)
As	0.18503(3)	0.26048(3)	0.40668(2)	3.60(1)
Ga	0.28703(4)	0.10447(4)	0.35816(2)	4.18(1)
Si(1)	0.24582(11)	0.27807(11)	0.52610(6)	5.13(3)
Si(2)	0.00773(9)	0.24102(10)	0.38670(7)	4.95(3)
Si(3)	0.21298(12)	0.42719(10)	0.35757(7)	5.31(3)
Cl	0.16634(12)	0.07974(12)	0.25979(6)	6.78(3)
C(1)	0.2965(4)	-0.0119(4)	0.4277(2)	5.3(1)
C(2)	0.2584(3)	-0.1261(3)	0.4099(2)	4.7(1)
C(3)	0.3082(4)	-0.1695(4)	0.3515(2)	5.8(1)
C(4)	0.2941(5)	-0.1976(4)	0.4719(3)	6.4(1)
C(5)	0.1338(4)	-0.1312(4)	0.3888(3)	6.2(1)
C(6)	0.4209(4)	0.1806(5)	0.3445(3)	6.4(1)
C(7)	0.5009(4)	0.1322(5)	0.3055(3)	6.9(1)
C(8)	0.5974(6)	0.2048(8)	0.3079(4)	12.5(2)
C(9)	0.5500(6)	0.0339(7)	0.3449(5)	12.4(2)
C(10)	0.4492(7)	0.1047(13)	0.2362(4)	19.8(5)
C(11)	0.3955(5)	0.2535(5)	0.5423(3)	7.9(2)
C(12)	0.1788(5)	0.1734(5)	0.5695(3)	7.9(2)
C(13)	0.2163(6)	0.4139(5)	0.5565(3)	8.6(2)
C(21)	-0.0634(5)	0.2701(5)	0.2959(3)	8.0(2)
C(22)	-0.0401(4)	0.1024(4)	0.4057(3)	7.0(1)
C(23)	-0.0671(4)	0.3329(4)	0.4422(3)	7.8(1)
C(31)	0.1989(6)	0.4074(5)	0.2641(3)	8.2(2)
C(32)	0.3533(5)	0.4741(5)	0.3958(3)	7.6(1)
C(33)	0.1115(5)	0.5289(4)	0.3736(3)	6.9(1)

Table V. Selected Bond Distances (Å) and Angles (deg) for $(Me_3CCH_2)_2\overline{GaAs(SiMe_3)_2Ga(CH_2CMe_3)_2Cl}$ (1), with Estimated Standard Deviations in Parentheses.

Bond Lengths			
As-Ga(1)	2.528(1)	Ga(1)-C(11)	1.992(5)
As-Ga(2)	2.514(1)	Ga(1)-C(16)	1.977(4)
As-Si(1)	2.360(1)	Ga(2)-C(21)	1.974(5)
As-Si(2)	2.3561)	Ga(2)-C(26)	1.990(5)
Ga(1)-Cl	2.422(1)		
Ga(2)-Cl	2.428(1)		
Bond Angles			
Ga(1)-As-Ga(2)	89.82(2)	Cl-Ga(1)-C(11)	108.9(2)
Ga(1)-As-Si(1)	110.87(3)	Cl-Ga(1)-C(16)	98.7(2)
Ga(1)-As-Si(2)	121.12(3)	C(11)-Ga(1)-C(16)	127.8(2)
Ga(2)-As-Si(1)	120.85(4)	Ga(2)-As-Si(2)	109.30(4)
As-Ga(2)-Cl	87.98(3)	As-Ga(2)-C(21)	113.8(2)
Si(1)-As-Si(2)	105.39(5)	As-Ga(2)-C(26)	111.0(2)
As-Ga(1)-Cl	87.79(2)	As-Ga(1)-C(11)	110.0(1)
Cl-Ga(2)-C(21)	97.8(1)	Cl-Ga(2)-C(26)	109.5(1)
As-Ga(1)-C(16)	114.6(1)	C(21)-Ga(2)-C(26)	127.6(2)
		Ga(1)-Cl-Ga(2)	94.41(4)

Table VI. Selected Bond Distances (Å) and Angles (deg) for $[(Me_3CCH_2)_2GaAs(SiMe_3)_2]_2$ (2), with Estimated Standard Deviations in Parentheses.

Bond Lengths			
As(1)-Ga	2.584(1)	Ga-C(1)	1.994(7)
As(1)-Si(1)	2.358(2)	Ga-C(6)	1.999(5)
As(2)-Ga	2.590(1)		
As(2)-Si(2)	2.367(2)		
Bond Angles			
Ga-As(1)-Ga'	95.11(4)	As(1)-Ga-C(1)	101.9(2)
Ga-As(1)-Si(1)	118.32(4)	As(1)-Ga-C(6)	111.9(3)
Ga'-As(1)-Si(1)	111.84(5)	As(2)-Ga-C(1)	110.4(2)
Si(1)-As(1)-Si(1')	102.32(7)	As(2)-Ga-C(6)	101.3(2)
Ga-As(2)-Ga'	94.84(4)	C(1)-Ga-C(6)	135.2(3)
As(1)-Ga-As(2)	85.02(3)		

Table VII. Selected Bond Distances (Å) and Angles (deg) for $(Me_3CCH_2)_2Ga(Cl)\cdot As(SiMe_3)_3$ (3), with Estimated Standard Deviations in Parentheses.

	Bond Lengths		
As-Ga	2.626(1)	Ga-Cl(1)	2.242(1)
As-Si(1)	2.363(1)	Ga-C(1)	2.002(5)
As-Si(2)	2.371(1)	Ga-C(6)	1.991(6)
As-Si(3)	2.366(1)		
	Bond Angles		
Ga-As-Si(1)	111.24(4)	As-Ga-Cl	97.87(4)
Ga-As-Si(2)	114.40(4)	As-Ga-C(1)	104.6(1)
Ga-As-Si(3)	112.46(4)	As-Ga-C(6)	100.2(2)
Si(1)-As(1)-Si(2)	106.64(5)	Cl-Ga-C(1)	116.2(1)
Si(1)-As-Si(3)	106.99(3)	Cl-Ga-C(6)	112.3(2)
Si(2)-As-Si(3)	104.56(5)	C(1)-Ga-C(6)	120.8(2)

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