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[19. ABSTRACT]

$[\text{Cl}_2\text{GaP}(\text{SiMe}_3)_2]_2$ (1) has been prepared from the 1:1 reaction of GaCl_3 with $\text{P}(\text{SiMe}_3)_3$. Thermal decomposition of 1 produces a brown powder which contains GaP, as evidenced by an X-ray powder pattern and partial elemental analysis. Compound 1 crystallizes in the monoclinic space group $\text{P}2_1/\text{n}$ (#14) with $a = 9.754(2)$, $b = 15.585(5)$, $c = 9.839(2)$ Å, and $\beta = 96.18(1)^\circ$, is composed of a planar $\overline{\text{Ga-P-Ga-P}}$ ring, with Ga-P bond distances of 2.378(2) and 2.380(2) Å, and contains exocyclic chlorine and SiMe_3 ligands. The ring core is a slightly distorted square, with Ga-P-Ga' and P-Ga-P' bond angles of 86.41(7) and 93.59(7)°, respectively. Additionally, ^1H NMR confirms that 1 exhibits monomer-dimer equilibrium in solution.

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SYNTHESIS, CHARACTERIZATION, AND THERMAL DECOMPOSITION OF
[Cl₂GAP(SiMe₃)₂]₂, A POTENTIAL PRECURSOR TO GALLIUM PHOSPHIDE

by

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**Synthesis, Characterization, and Thermal Decomposition of
[Cl₂GaP(SiMe₃)₂]₂, a Potential Precursor to Gallium Phosphide.**

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Summary: [Cl₂GaP(SiMe₃)₂]₂ (1) has been prepared from the 1:1 reaction of GaCl₃ with P(SiMe₃)₃. Thermal decomposition of 1 produces a brown powder which contains GaP, as evidenced by an X-ray powder pattern and partial elemental analysis. Compound 1 crystallizes in the monoclinic space group P2₁/n (#14) with *a* = 9.754(2), *b* = 15.585(5), *c* = 9.839(2) Å, and β = 96.18(1)°, is composed of a planar $\overline{\text{Ga-P-Ga-P}}$ ring, with Ga-P bond distances of 2.378(2) and 2.380(2) Å, and contains exocyclic chlorine and SiMe₃ ligands. The ring core is a slightly distorted square, with Ga-P-Ga' and P-Ga-P' bond angles of 86.41(7) and 93.59(7)°, respectively. Additionally, ¹H NMR confirms that 1 exhibits monomer-dimer equilibrium in solution.

Previous research in our laboratory has proven the utility of silylarsines to prepare compounds containing the gallium-arsenic bond. Using reagents of the general formula R_nAs(SiMe₃)_{3-n} and R'_nGaX_{3-n} (n = 0,1,2), a number of monomeric, dimeric and trimeric arsinogallanes have been prepared.¹ Subsequent work has explored the more fundamental reactions in the series, namely those between As(SiMe₃)₃ and GaX₃ (X = Cl, Br). We have shown that reactions involving these species in a 1:1 mole ratio proceed at relatively low temperatures to eliminate Me₃SiX with concomitant formation of GaAs.²

Recent research has applied analogous reaction schemes to gallium-phosphorus systems, yielding both 1:1 adducts and a compound exhibiting mixed-bridging of two

gallium centers by a phosphorus atom and a halogen.³ Herein we report the isolation and characterization of a remarkably different species resulting from the 1:1 reaction of GaCl₃ and P(SiMe₃)₃, [Cl₂GaP(SiMe₃)₂]₂ (**1**), which, through thermolysis yields a GaP containing powder, (84.8% purity based on C, H, Cl, Ga, P).

Experimental Section

General Considerations. All manipulations were performed using general Schlenk, dry box and/or high vacuum techniques. Solvents (including those used for NMR spectra) were appropriately dried and distilled under argon prior to use. Literature methods were used to prepare P(SiMe₃)₃.⁴ GaCl₃ was purchased from Alfa Products and purified by sublimation. ¹H, ¹³C{¹H} and ³¹P{¹H} NMR spectra were obtained on a Varian XL-300 at 300.0, 75.4 and 121.4 MHz, respectively. ¹H and ¹³C spectra were referenced to TMS *via* the residual protons or carbons of C₆D₆ or C₇D₈. ³¹P spectra were referenced to external 80% H₃PO₄ at δ 0.00 ppm. Mass spectra were obtained on a Hewlett-Packard 5988A GC/MS/DS system. Melting points (uncorrected) were taken in sealed capillaries (Thomas-Hoover Uni-melt). Elemental analysis was performed by E + R Microanalytical Laboratory, Inc., Corona, NY. The X-ray powder pattern of GaP was recorded at the Crystal Structure Center, Duke University, while the X-ray Crystal Structure of **1** was performed the New England Molecular Structure Center, Keene State College.

Preparation of [Cl₂GaP(SiMe₃)₂]₂ (1**).** GaCl₃ (0.2646 g, 1.503 mmol) in 150 mL of dry pentane was placed in a 250 mL round-bottom flask, equipped with a Teflon screw cap and sidearm valve. P(SiMe₃)₃ (0.381 g, 1.521 mmol) dissolved in 15 mL toluene was slowly added to the flask dropwise. The flask was capped and placed in a sonicating water bath for 15 h, after which the solvent and volatiles were removed *in vacuo*, affording a grayish-yellow solid. The volatiles were then hydrolyzed with deionized water and titrated with NaOH (0.102M, 14.40 mL corresponding to elimination of 1.47 mmol (97.8%) of Me₃SiCl). X-ray quality crystals of **1** were grown from toluene

(0.3714 g, 77.7% yield); mp. decomposes above 220 °C. Anal. Calcd (found) for $C_6H_{18}Cl_2GaPSi_2$: C, 22.66 (22.60); H, 5.71 (5.59); Cl, 22.30 (22.06). 1H NMR: δ 0.29 [d, 18H, $SiMe_3$ (monomer) ($J_{P-H} = 6.11$ Hz)], δ 0.45 [t, 36 H, $SiMe_3$ (dimer) ($J_{P-H} = 3.33$ Hz)]. $^{13}C\{^1H\}$ NMR: δ 2.7-3.0 [overlapping multiplet, $PSiMe_3$]. $^{31}P\{^1H\}$ NMR: δ -158.4 (s).

Thermal Decomposition of $[Cl_2GaP(SiMe_3)_2]_2$ (1). $[Cl_2GaP(SiMe_3)_2]_2$ (0.3437 g, 0.540 mmol) was placed in a water cooled sublimator with attached Teflon valve. Under vacuum, the sublimator, in a sand bath and opened to a trap cooled to -196 °C, was slowly warmed over a 3 h period to 300 °C, where it remained overnight. Finally, the powder was annealed for 2 h with a cool flame. Over the course of the decomposition, the color of the initially white material changed to yellow then orange, and eventually to brown. The brown material was collected in quantitative yield. Collected volatiles were hydrolyzed with deionized water and titrated with NaOH, (0.102 M, 18.20 mL), corresponding to the elimination of 1.86 mmol (85.8%) of Me_3SiCl . The brown powder was tightly packed into a thin-walled glass capillary for X-ray powder pattern analysis. Anal. Calcd (Found) for GaP: Ga, 69.24 (58.59); P, 30.76 (25.19); C, 0.00 (7.24); H, 0.00 (1.22); Cl, 0.00 (6.88); (Ga:P mol ratio = 1.03). X-ray powder diffraction pattern identical with that of a bona fide sample of GaP.

Structural Analysis. A single crystal of 1 was mounted in a 0.7 mm thin-walled glass capillary under an inert atmosphere of argon and flame sealed. Relevant crystallographic data are given in Table I. All measurements and calculations were carried out as in previous work.³ Cell constants and an orientation matrix for data collection, obtained from a least-squares refinement using the setting angles of 20 carefully centered reflections in the range $30.5^\circ < 2\theta < 35.8^\circ$, corresponded to a monoclinic cell. The space group $P2_1/n$ was defined uniquely by the Laue symmetry and systematic absences, $h0l$ when $h + l \neq 2n$, $0k0$ when $k \neq 2n$. The intensities of three reference reflections, remeasured after every 150 reflections, remained constant

throughout data collection indicating crystal and electronic stability (no decay correction was necessary).

Final fractional atomic coordinates are given in Table II. Selected bond distances and angles are given in Table III. A thermal ellipsoid diagram showing the atom labeling scheme for **1** is presented in Figure 1.

Results and Discussion

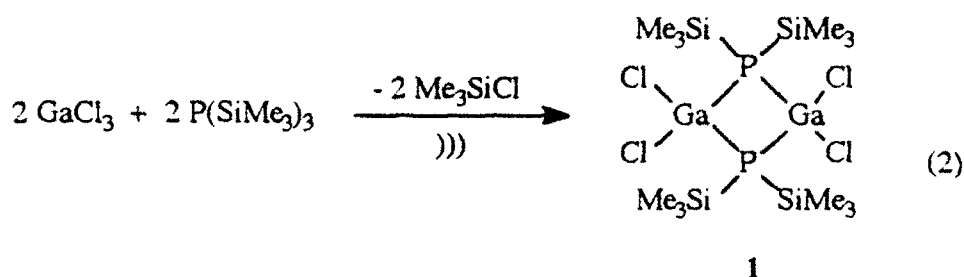
In an ongoing effort to synthesize possible single-source precursors to Group 13-15 semiconductors, dehalosilylations between metal halides and silylnictides, and salt elimination between metal halides and lithium silylnictides have been investigated.¹ Previously, the formation of GaAs from the 1:1 reaction of GaCl₃ with As(SiMe₃)₃ was reported.²



This facile reaction proceeds at room temperature, to initially yield a white precipitate which rapidly turns to yellow and finally, upon removal of solvent, to an orange powder. Upon heating the orange powder of equation 1, GaAs of about 95% purity is obtained. It is unfortunate that no suitable analysis for the orange powder could be obtained, as it decomposes rapidly upon standing.

Barron and coworkers have reported the isolation of the product of the analogous reaction in the indium-phosphorus system.⁵ The product of the reaction of InCl₃ with P(SiMe₃)₃ is a bright orange powder, which has been proposed as an oligomer, [Cl₂InP(SiMe₃)₂]_x (**2**), and undergoes thermal decomposition to eliminate Me₃SiCl, with concomitant formation of InP. However, no X-ray crystal structure of Compound **2** has been reported.

It is interesting that the analogous reaction involving GaCl_3 and $\text{P}(\text{SiMe}_3)_3$ affords a highly stable and completely characterizable intermediate (1).



The core of **1** consists of a planar Ga-P-Ga-P ring that is a slightly distorted square [Ga-P-Ga' and P-Ga-P' bond angles of $86.41(7)$ and $93.59(7)^\circ$, respectively]. The Ga-P and Ga'-P bond lengths of $2.378(2)$ and $2.380(2)$ Å, respectively, are the shortest reported to date for compounds having the general formula $[\text{R}_2\text{GaPR}'_2]_2$.⁶⁻⁸ Cowley and coworkers have reported a number of these dimeric gallium-phosphorus compounds.^{6,7} Of these, $[\textit{t}\text{-Bu}_2\text{Ga}(\mu\text{-(C}_5\text{H}_9)\text{PH})]_2$, exhibits the shortest Ga-P bond length of 2.451 Å.⁷ Compound **1** is the first reported example of a gallium-phosphorus dimer containing all exocyclic halogen ligands on the metal center. Due to the increased Lewis acidity of the gallium centers from the attached chlorine atoms, the shortened Ga-P distances are reasonable.

Cowley, *et al.*, have also reported a compound similar to **1**, $(\textit{t}\text{-BuGa}(\text{Cl})\text{P}(\text{H})\text{Ar}')_2$ (**3**),⁹ from the 1:1 reaction of $\textit{t}\text{-BuGaCl}$ with $\text{Ar}'\text{P}(\text{H})\text{Li}$, ($\text{Ar}' = 2,4,6\text{-}\textit{t}\text{-Bu}_3\text{C}_6\text{H}_2$). Surprisingly, **3** is a chloride-bridged dimer, with exocyclic phosphorus ligands, whereas **1** dimerizes through bridging phosphorus atoms, and contains exocyclic halogen ligands. Based on purely steric considerations, the chloride-bridged dimer may be anticipated, and it is unclear as to why this does not occur, but may be due to electronic factors of $\text{P}(\text{H})\text{Ar}'$ versus $\text{P}(\text{SiMe}_3)_2$.

As for the solution properties of **1**, two separate resonances are observed for the methyl protons in the ^1H NMR spectrum of $[\text{Cl}_2\text{GaP}(\text{SiCH}_3)_3]_2$, a downfield triplet (δ 0.45, $J_{\text{P-H}} = 3.33$ Hz) and an upfield doublet (δ 0.29, $J_{\text{P-H}} = 6.11$ Hz). These resonances suggest that both dimeric and monomeric forms of **1** exist in solution. A variable temperature study from 20° to 95 °C, in 10° increments, begins to demonstrate coalescence, indicating the equilibrium state. In addition, the relative area of the triplet resonance (dimer) to the doublet resonance (monomer) increases with elevated temperature, indicating that the dimeric species is favored at higher temperatures. The ^{13}C NMR spectra of **1** exhibits an overlapping multiplet in the methyl region, which is indicative of both species. Interestingly, the ^{31}P spectrum exhibits only a singlet, which may actually consist of two overlapping resonances. The dimeric nature of **1** in the gas phase is established by the parent peak of the direct introduction probe 25 eV electron impact mass spectrum, $[\text{M}^+, m/z = 632 (^{69}\text{Ga}, ^{35}\text{Cl})]$.

Compound **1** has been thermally decomposed at 300 °C to yield a powder containing GaP (84.8% based on C, H, Cl, Ga, and P). The relatively low temperature decomposition was not conditionally optimized, nor was the product purified, therefore the large amount of impurities shown by elemental analysis is not unexpected. Based on the high yield of the pyrolyzed volatiles, (85.8%) and the results of the X-ray powder pattern studies, we conclude that the title compound undergoes facile elimination of its ligands to produce a GaP- containing powder in a good yield. Furthermore, the presence of the exocyclic halogen ligands of **1** should provide a viable route for further dehalosilylation and salt elimination reactions to substitute alternative ligands onto the gallium centers.

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Grant No. 8818307 for establishment of the New England Molecular Structure Center. The assistance of Ray Butcher (X-ray crystallography) at Howard University, and George Dubay (mass spectrometry) at Duke University is greatly appreciated.

Supplementary Material Available: Tables of hydrogen atom coordinates and isotropic thermal parameters, anisotropic temperature factors complete lists of interatomic distances and angles (5 pages). Ordering information is given on any current masthead page.

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Captions to Figures

Figure 1. A thermal ellipsoid diagram (30 % probability level) showing the solid state conformation and atom numbering scheme of $[\text{Cl}_2\text{GaP}(\text{SiMe}_3)_2]_2$ (I). Hydrogen atoms have been omitted for clarity.

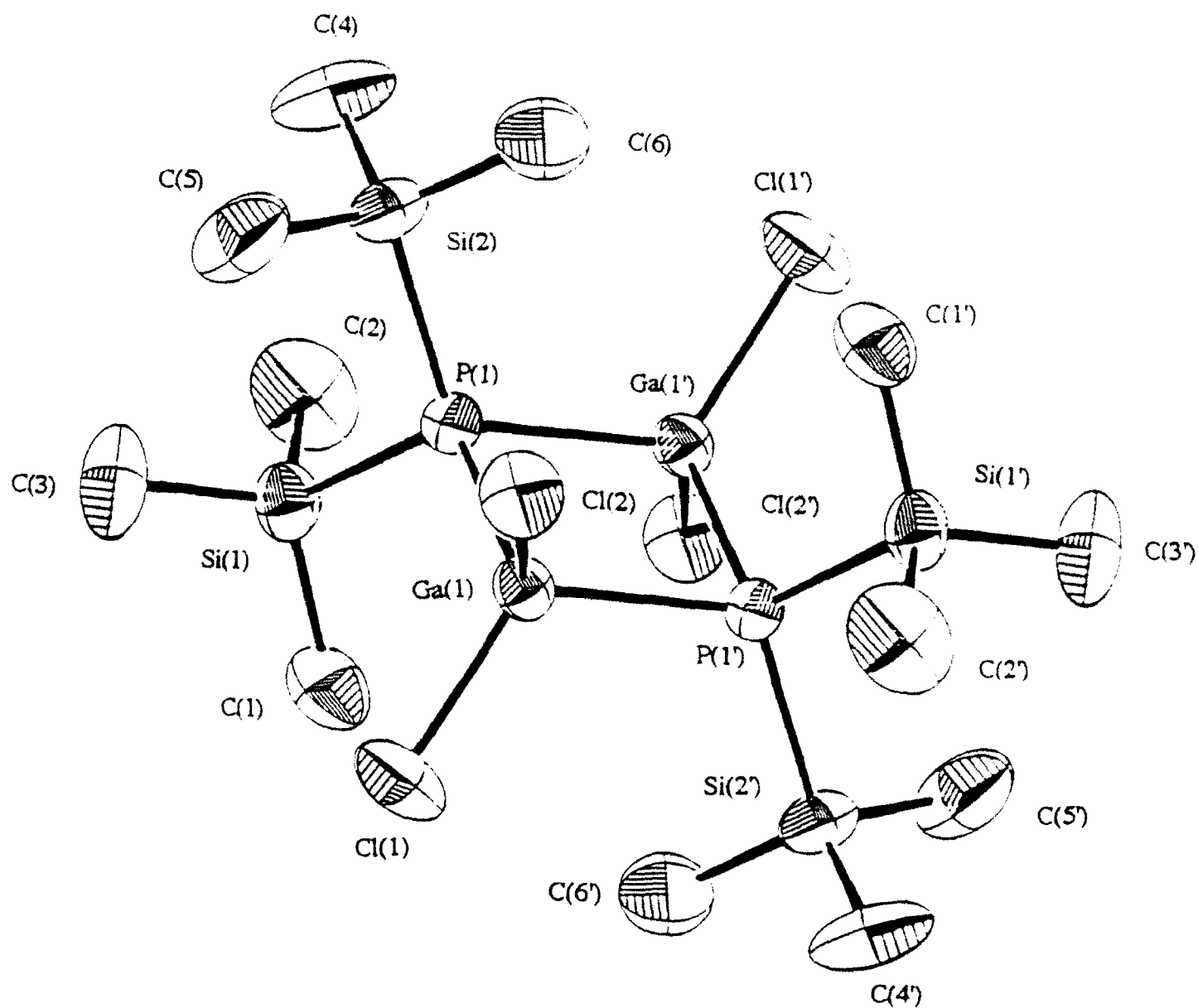


Figure 1

Table I. Crystallographic Data and Data Collection Parameters for
 $[\text{Cl}_2\text{GaP}(\text{SiMe}_3)_2]_2$ (1)

Molecular formula	$\text{C}_{12}\text{H}_{36}\text{Cl}_4\text{Ga}_2\text{P}_2\text{Si}_4$
Formula weight	635.96
Crystal system	monoclinic
Space group	$P2_1/n(C_{2h}^5)$ - No. 14
$a(\text{\AA})$	9.754(2)
$b(\text{\AA})$	15.585(5)
$c(\text{\AA})$	9.839(2)
$\beta(^{\circ})$	96.18(1)
$V(\text{\AA}^3)$	1487(1)
Z	2
$D_{\text{calcd.}} (\text{g cm}^{-3})$	1.420
$\mu(\text{Mo-}K\alpha \text{ radiation, } \lambda = 0.71069 \text{ \AA})$	24.3
F_{000}	648
Temp. ($^{\circ}\text{C}$)	23
Crystal dimensions (mm)	0.50 x 0.60 x 0.70
$T_{\text{max.}}:T_{\text{min.}}$	1.00:0.94
Scan type	ω^2
$2\theta_{\text{max.}}$, deg	55.0
Scanwidth, deg	$1.10 + 0.30 \tan \theta$
Total no. of refls. ($+h, +k, \pm l$) recorded	3727
No. of non-equiv. refls. recorded	3532
R_{int}	0.044
No. of refls. retained [$I > 3.0\sigma(I)$]	1669
No. of parameters refined	182
$R(R_w)^b$	0.043 (0.042)
Goodness-of-fit ^c	1.26
Max. shift/esd in final least-squares cycle	3.45
Final $\Delta\rho(\text{e/\AA}^3)$ max.;min.	0.42; -0.47

^a with profile analysis.

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

^cGoodness-of-fit = $[\sum w\Delta^2 / (N_{\text{observations}} - N_{\text{parameters}})]^{1/2}$.

Table II. Non-hydrogen Atom Fractional Coordinates and Equivalent Isotropic Thermal Parameters for $[\text{Cl}_2\text{GaP}(\text{SiMe}_3)_2]_2$ (1), with Estimated Standard Deviations in Parentheses

atom	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> _{eq} (Å ²)
Ga(1)	-0.13396(7)	0.04238(5)	-0.08827(7)	2.96(3)
Cl(1)	-0.2275(2)	0.0140(1)	-0.2939(2)	5.5(1)
Cl(2)	-0.2534(2)	0.1455(1)	-0.0095(2)	4.9(1)
P(1)	0.1056(2)	0.0750(1)	-0.0670(2)	2.94(7)
Si(1)	0.2164(2)	0.0496(1)	-0.2567(2)	3.83(9)
Si(2)	0.1533(2)	0.2083(1)	0.0224(2)	4.4(1)
C(1)	0.140(1)	-0.0480(6)	-0.3393(8)	5.1(4)
C(2)	0.403(1)	0.034(1)	-0.200(1)	7.4(6)
C(3)	0.188(1)	0.1430(7)	-0.371(1)	6.7(6)
C(4)	0.340(1)	0.2242(7)	0.032(1)	7.6(6)
C(5)	0.059(1)	0.2874(7)	-0.091(1)	7.2(6)
C(6)	0.092(1)	0.2134(7)	0.193(1)	7.1(6)

Table III. Interatomic Distances (Å) and Angles (deg.) for $[\text{Cl}_2\text{GaP}(\text{SiMe}_3)_2]_2$ (1),^a with Estimated Standard Deviations in Parentheses

(a) Bond Lengths			
Ga(1)-Cl(1)	2.174(2)	Si(1)-C(1)	1.84(1)
Ga(1)-Cl(2)	2.176(2)	Si(1)-C(2)	1.86(1)
Ga(1)-P(1)	2.378(2)	Si(1)-C(3)	1.84(1)
Ga(1)-P(1')	2.380(2)	Si(2)-C(4)	1.83(1)
P(1)-Si(1)	2.290(3)	Si(2)-C(5)	1.84(1)
P(1)-Si(2)	2.284(2)	Si(2)-C(6)	1.84(1)
(b) Bond Angles			
Cl(1)-Ga(1)-Cl(2)	107.14(7)	P(1)-Si(1)-C(1)	107.3(3)
Cl(1)-Ga(1)-P(1)	115.81(8)	P(1)-Si(1)-C(2)	108.1(3)
Cl(1)-Ga(1)-P(1')	116.69(7)	P(1)-Si(1)-C(3)	107.9(3)
Cl(2)-Ga(1)-P(1)	111.68(7)	C(1)-Si(1)-C(2)	111.1(6)
Cl(2)-Ga(1)-P(1')	111.64(7)	C(1)-Si(1)-C(3)	110.8(4)
P(1)-Ga(1)-P(1')	93.59(7)	C(2)-Si(1)-C(3)	111.4(5)
Ga(1)-P(1)-Ga(1')	86.41(7)	P(1)-Si(2)-C(4)	107.7(4)
Ga(1)-P(1)-Si(1)	115.90(9)	P(1)-Si(2)-C(5)	107.9(3)
Ga(1)-P(1)-Si(2)	112.69(9)	P(1)-Si(2)-C(6)	108.5(4)
Ga(1')-P(1)-Si(1)	110.97(8)	C(4)-Si(2)-C(5)	111.7(5)
Ga(1')-P(1)-Si(2)	116.55(9)	C(4)-Si(2)-C(6)	111.5(4)
Si(1)-P(1)-Si(2)	112.1(1)	C(5)-Si(2)-C(6)	109.5(5)

^aPrimed atoms are related to the unprimed atoms by a crystallographic center of symmetry.

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