

[Bis(trimethylsilyl)methyl]chloro(phenyl)phosphine**Fang Xi, Xia Chen,* Jian-Ping Guo and Dian-Sheng Liu**

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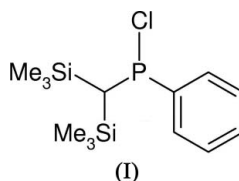
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Key indicatorsSingle-crystal X-ray study
 $T = 183\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$
 R factor = 0.051
 wR factor = 0.118
Data-to-parameter ratio = 18.6For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

The title compound, $\text{C}_{13}\text{H}_{24}\text{ClPSi}_2$, was synthesized *via* the reaction of bis(trimethylsilyl)methyl lithium and dichlorophenylphosphine. The coordination geometry around the P atom is distorted tetrahedral.

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The presence of heteroatoms such as Si or P in the α position of carbanions can increase their stability by hyperconjugation. In recent years, new organic phosphinyl compounds have been synthesized, including $\text{P}\equiv$ and $-\text{P}=\text{C}$ units, *etc.* (Edwards, 1995). The unique patterns of bonding in these compounds have enriched basic theories in the phosphine subject area and have also provided inspiration for the preparation of new structures. In the past 20 years, many alkali-metal compounds have been synthesized incorporating such species; for example, phosphinyl compounds substituted by benzyl can exhibit unique character when forming complexes with metal cations because of stereochemical effects (Clegg *et al.*, 1998). Much research has also been carried out on the special chemical reactivity and applications of heteroatom compounds in organic synthesis, developing new reactions in which the heteroatom-organic molecule plays the role of substrate or catalyst (White *et al.*, 1999; Chan *et al.*, 1995). Recently, we have synthesized the title phosphinyl compound, (I), and we report here its crystal structure.



In (I), the coordination geometry around P1 is distorted tetrahedral, with the P atom bonded to two C atoms and one Cl atom, and having a lone pair of electrons (Fig. 1 and Table 1). The $\text{C}2-\text{P}1-\text{C}1-\text{Si}1$, $\text{Cl}1-\text{P}1-\text{C}1-\text{Si}1$, $\text{C}2-\text{P}1-\text{C}1-\text{Si}2$ and $\text{Cl}1-\text{P}1-\text{C}1-\text{Si}2$ torsion angles are $82.15(15)$, $-177.22(10)$, $-150.30(15)$ and $-49.67(15)^\circ$, respectively.

Experimental

All reactions were carried out under Ar using standard Schlenk techniques. Diethyl ether was distilled from drying agents and degassed with Ar prior to use. In a 1000 ml round-bottomed flask, bromoform (20.0 ml, 0.23 mol) and trimethylchlorosilane (58.1 ml, 0.46 mol) were added to tetrahydrofuran (300 ml). To this solution, *n*-butyllithium in *n*-hexane (157.0 ml, 0.46 mol) was added dropwise

at 195 K, and the mixture was allowed to warm to room temperature, stirred for 4 h and filtered. Diethyl ether was distilled from the filtrate, and the product bromobis(trimethylsilyl)methane, a colourless liquid, was then collected by distillation in a vacuum at 321–333 K. In a subsequent reaction, *n*-butyllithium in *n*-hexane (19.6 ml, 57.37 mmol) was added quickly to a solution of bromobis(trimethylsilyl)methane (13.66 g, 57.37 mmol) in Et₂O (20 ml) at 195 K, and the solvent was removed immediately. The raw product was sublimed to give pure bis(trimethylsilyl)methylolithium. Finally, dichlorophenylphosphine (0.46 ml, 3.39 mmol) was added to a solution of bis(trimethylsilyl)methylolithium (0.563 g, 3.39 mmol) in Et₂O (20 ml) at 273 K, and the temperature was allowed to warm to room temperature. The mixture was stirred for 4 h, yielding a white precipitate, which was removed by filtration. The filtrate was concentrated to *ca* 10 ml and then cooled at 243 K for 3 d to give colourless crystals of the title compound.

Crystal data

C ₁₃ H ₂₄ ClPSi ₂	$V = 859.3 (3) \text{ \AA}^3$
$M_r = 302.93$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.171 \text{ Mg m}^{-3}$
$a = 8.5958 (19) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.069 (2) \text{ \AA}$	$\mu = 0.44 \text{ mm}^{-1}$
$c = 11.794 (3) \text{ \AA}$	$T = 183 (2) \text{ K}$
$\alpha = 75.783 (3)^\circ$	Block, colourless
$\beta = 74.661 (3)^\circ$	$0.40 \times 0.30 \times 0.20 \text{ mm}$
$\gamma = 87.322 (3)^\circ$	

Data collection

Bruker SMART CCD diffractometer	3549 measured reflections
ω scans	2972 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	2485 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.844$, $T_{\max} = 0.917$	$R_{\text{int}} = 0.017$
	$\theta_{\max} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0587P)^2 + 0.1605P]$
$R[F^2 > 2\sigma(F^2)] = 0.051$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.118$	$(\Delta/\sigma)_{\max} < 0.001$
$S = 1.08$	$\Delta\rho_{\max} = 0.41 \text{ e \AA}^{-3}$
2972 reflections	$\Delta\rho_{\min} = -0.22 \text{ e \AA}^{-3}$
160 parameters	
H-atom parameters constrained	

Table 1

Selected geometric parameters (\AA , $^\circ$).

Cl1–P1	2.1067 (11)	Si1–C1	1.903 (3)
P1–C1	1.823 (3)	Si2–C12	1.861 (3)
P1–C2	1.837 (3)	Si2–C13	1.861 (3)
Si1–C9	1.853 (4)	Si2–C11	1.870 (3)
Si1–C10	1.863 (3)	Si2–C1	1.909 (3)
Si1–C8	1.866 (3)		
C1–P1–C2	103.64 (13)	C2–P1–Cl1	96.79 (10)
C1–P1–Cl1	103.71 (9)		

H atoms were positioned geometrically and allowed to ride during subsequent refinement, with C–H = 0.93, 0.96 and 0.98 \AA for those on phenyl, tertiary and methyl C atoms, respectively. $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for the methyl groups and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ otherwise. The methyl groups were also allowed to rotate about their local threefold axes.

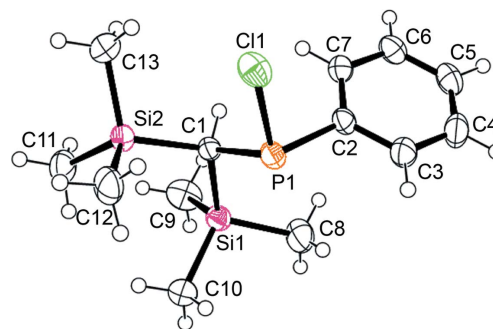


Figure 1

Molecular unit in (I), showing displacement ellipsoids at the 30% probability level. H atoms are shown as spheres of arbitrary radii.

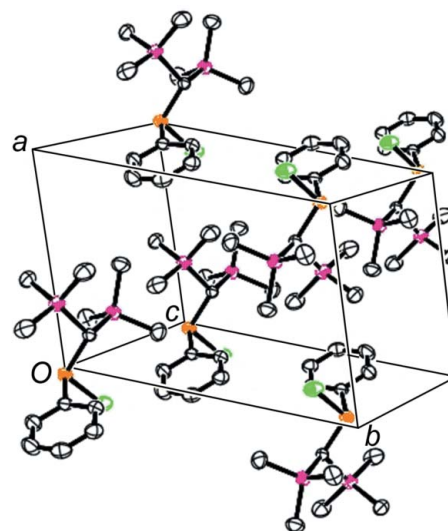


Figure 2

View of the unit-cell contents of (I). H atoms have been omitted.

Data collection: SMART (Bruker, 1998); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 1999); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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