Compounds containing Platinum–Carbon Bonds. Part III.[1] Crystal and Molecular Structure of *trans*-Chlorobis(dimethylphenylphosphine)(trimethylsilylmethyl)platinum(II)

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The crystal structure of the title compound has been determined from *X*-ray diffractometric data. Crystals are monoclinic, $a = 8{\cdot}919(4)$, $b = 21{\cdot}723(8)$, $c = 13{\cdot}865(6)$ Å, $\beta = 113{\cdot}88(3)°$, space group *Cc*. The structure was solved by the heavy-atom method and refined by full-matrix least squares to *R* 0·039 for 2573 independent reflections. The crystals contain discrete square planar $[PtCl(CH_2SiMe_3)(PMe_2Ph)_2]$ molecules. Pt–P, is 2·293(3), Pt–Cl 2·415(5), and Pt–C 2·079(14) Å.

As part of our investigations into the relative *trans*-influencing abilities of organic ligands [1] we have determined the crystal structure of the title compound by *X*-ray analysis. We expected that this work would also shed some light on the electronic effects of $Me_3SiCH_2^-$ and related ligands in transition-metal complexes. A preliminary report has appeared.[2]

Spectroscopic data indicate that Me^-, $Me_3SiCH_2^-$, and $MePh_2Si^-$ are ligands of high *trans*-influence in platinum(II) complexes and that the relative ability to weaken a *trans*-bond increases along the series: $Me^- \simeq Me_3SiCH_2^- \ll MePh_2Si^-$.[2] The exceptional *trans*-influence of $MePh_2Si^-$ has been confirmed by a structure analysis of *trans*-$[PtCl(SiMePh_2)(PMe_2Ph)_2]$.[3]

There is evidence that Me_3SiCH_2 and Me substituents in organic compounds release electrons by an inductive mechanism to π-bonded or electron-deficient carbon atoms; however Me_3SiCH_2 is appreciably more electron-releasing than Me, and hyperconjugation involving the $Me_3Si{-}CH_2$ bond has been invoked to explain this difference.[4,5] Transition-metal complexes of the type ML_n ($L = Me_3SiCH_2^-$) are known to be appreciably more stable than corresponding complexes with $L = Me^-$.[6-8] However, this effect is thought to be kinetic in origin and the importance of hyperconjugative electron release in $Me_3SiCH_2^-$ complexes of transition metals has yet to be demonstrated.

EXPERIMENTAL

Crystal Data.—$C_{20}H_{33}ClP_2PtSi$, $M = 594{\cdot}1$, Monoclinic, $a = 8{\cdot}919(4)$, $b = 21{\cdot}723(8)$, $c = 13{\cdot}865(6)$ Å, $\beta = 113{\cdot}88(3)°$, $U = 2456$ Å^3, D_m (by flotation) $= 1{\cdot}63$, $Z = 4$, $D_c = 1{\cdot}606$, $F(000) = 1168$. Space group *Cc*. Mo-K_α radiation, $\lambda = 0{\cdot}71069$ Å; $\mu(\text{Mo-}K_\alpha) = 62{\cdot}9$ cm^{-1}.

Approximate unit-cell constants and the Bragg reflections extinguished because of space-group symmetry were determined from Weissenberg and precession photographs. The absences hkl, $h + k = 2n + 1$ and $h0l$, $l = 2n + 1$, are consistent with the space groups *C*2/*c* and *Cc*. With $Z = 4$ the former space group would require the platinum atom to have C_2 or C_i site symmetry; either is incompatible, assuming no disorder, with the expected chemical structure. Accordingly, the space group *Cc* was initially chosen and this choice has been confirmed by a successful structure analysis.

Final values of the cell parameters and the intensities of all 3135 independent reflections with $\theta(\text{Mo-}K_\alpha) \leqslant 28°$ were measured on a Hilger and Watts Y 290 four-circle diffractometer by standard methods which we have previously described in detail.[9] The crystal employed was plate-shaped, with dimensions $0{\cdot}53 \times 0{\cdot}52 \times 0{\cdot}22$ mm, and was set so that its *a* axis was approximately coincident with the diffractometer φ axis. To monitor crystal and electronic stability the intensities of two strong reflections were measured periodically throughout the experiment, but only random fluctuations of up to $\pm 3\%$ of their mean values were observed.

The integrated intensities and their standard deviations were derived as described previously,[9] and were corrected for Lorentz, polarisation, and absorption effects. The transmission factors on $|F_o|$, the observed structure amplitude, calculated by an analytical method,[10] ranged from 0·34 to 0·57.

A total of 2573 reflections for which $I \geqslant 3\sigma(I)$ were used in the analysis.

Structure Analysis.—The position of the platinum atom was derived from a Patterson synthesis, and those of the other non-hydrogen atoms from subsequent difference syntheses.

The structure was refined by the method of full-matrix least squares. The function minimised was $\Sigma w\Delta^2$, where $\Delta = \big||F_o| - |F_o|\big|$. The weights *w* were taken as the reciprocals of the variances of $|F_o|$, as previously described.[9] Atomic scattering factors from ref. 11 were used, except for those of hydrogen [12] and platinum.[13] Allowance was made for the anomalous scattering of the platinum,

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† No reprints available.

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chlorine, phosphorus, and silicon atoms, using values of $\Delta f'$ and $\Delta f''$ from ref. 14.

Refinement of the positional and isotropic thermal parameters of all non-hydrogen atoms converged at R 0·073 and R' 0·100 [$R' = \{\Sigma w\Delta^2/\Sigma wF_o^2\}^{\frac{1}{2}}$]. Anisotropic thermal parameters for the atoms Pt, Cl, P(1), P(2), and C(1) were then introduced into the refinement and R and R' decreased to 0·047 and 0·060. At this stage the chemically equivalent Pt–P bond lengths were 2·227(8) and 2·362(7) Å. The co-ordinates of all atoms were reversed and with no increase in the number of parameters the inverted model was now refined; this led to R 0·040 and R' 0·053, and the two Pt–P distances became equal. Accordingly the second model was assumed to represent correctly the absolute configuration of the structure in the crystal used for the analysis. This model was further refined; the twelve non-methyl hydrogen atoms were included in the structure-factor calculations in positions consistent with the known stereochemistry of sp^2 and sp^3 hybridised carbon atoms; adjustment of 129 positional and thermal parameters of the non-hydrogen atoms yielded final values of R 0·039 and R' 0·052. In the final cycle of refinement all parameters shifted by $<0·6\sigma$, except for one which shifted by $1·5\sigma$. The final difference synthesis was featureless, except in the region of the platinum atom where the extreme function values were +0·96 and −2·43 eÅ^{-3}. The appropriateness of the weighting scheme was confirmed by an analysis of the mean $w\Delta^2$ as a bivariate function of $|F_o|$ and $\sin\theta/\lambda$. Final atomic parameters and a selection of functions derived from them are presented in Tables 1—4. Observed and calculated structure factors are listed in Supplementary Publication No. SUP 20868 (5 pp., 1 microfiche).* A view of the molecular structure is shown in the Figure.

Computer programs used were C. K. Johnson's ORTEP, the ATLAS versions of J. M. Stewart's *X*-Ray '63 and

TABLE 1

Fractional atomic co-ordinates and vibrational parameters

	x/a	y/b	z/c	B/Å^2
Pt	0	−0·13318(2)	−1/4	*
Cl	−0·0030(8)	−0·0387(2)	−0·1589(4)	*
P(1)	−0·0353(6)	−0·1820(2)	−0·1139(3)	*
P(2)	0·0315(5)	−0·0737(2)	−0·3771(3)	*
Si	0·2160(6)	−0·2486(2)	−0·2881(4)	*
C(1)	0·0103(21)	−0·2150(6)	−0·3248(11)	*
C(2)	−0·2254(29)	−0·1538(10)	−0·1008(18)	6·1(4)
C(3)	0·1331(26)	−0·1652(10)	0·0136(17)	5·4(4)
C(4)	−0·0667(21)	−0·2666(7)	−0·1121(13)	4·0(3)
C(5)	0·0428(26)	−0·3032(10)	−0·0342(17)	5·5(4)
C(6)	0·0038(38)	−0·3657(12)	−0·0327(23)	7·4(6)
C(7)	−0·1321(35)	−0·3883(12)	−0·1062(23)	7·7(6)
C(8)	−0·2522(40)	−0·3496(13)	−0·1894(24)	7·2(6)
C(9)	−0·2095(26)	−0·2901(10)	−0·1854(17)	5·2(4)
C(10)	0·3234(26)	−0·2141(9)	−0·3665(16)	5·3(4)
C(11)	0·1913(38)	−0·3353(14)	−0·3162(23)	7·2(6)
C(12)	0·3526(23)	−0·2370(8)	−0·1461(15)	4·7(3)
C(13)	−0·0960(21)	−0·0041(8)	−0·4101(14)	4·5(3)
C(14)	−0·0168(23)	−0·1063(8)	−0·5061(14)	4·5(3)
C(15)	0·2378(20)	−0·0443(7)	−0·3351(12)	3·8(3)
C(16)	0·3460(23)	−0·0509(8)	−0·2296(14)	4·5(3)
C(17)	0·5119(27)	−0·0286(9)	−0·1981(16)	5·2(4)
C(18)	0·5464(30)	0·0034(12)	−0·2660(20)	6·5(5)
C(19)	0·4454(33)	0·0114(12)	−0·3665(22)	6·9(5)
C(20)	0·2865(27)	−0·0114(10)	−0·4026(18)	5·6(4)

* These atoms were assigned anisotropic temperature factors of the form: $\exp[-10^{-4}(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + 2hk\beta_{12} + 2hl\beta_{13} + 2kl\beta_{23})]$.

The final values of the β_{ij} parameters are:

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
Pt	134(1)	15(1)	39(1)	0(1)	24(1)	1(1)
Cl	321(12)	18(1)	68(3)	11(3)	62(5)	−9(1)
P(1)	185(8)	20(1)	43(2)	8(2)	43(3)	3(1)
P(2)	140(6)	15(1)	42(2)	−6(2)	19(3)	2(1)
Si	178(8)	18(1)	68(3)	7(2)	56(4)	1(1)
C(1)	204(29)	14(3)	40(8)	−5(7)	45(12)	1(4)

TABLE 2

Selected interatomic distances (Å) and angles (°)

(*a*) Bond lengths

Pt–Cl	2·415(5)	Pt–C(1)	2·079(14)
Pt–P(1)	2·294(5)	Pt–P(2)	2·292(5)
P(1)–C(2)	1·880(28)	P(2)–C(13)	1·835(19)
P(1)–C(3)	1·834(18)	P(2)–C(14)	1·807(20)
P(1)–C(4)	1·860(17)	P(2)–C(15)	1·807(17)
Si–C(1)	1·844(19)	Si–C(11)	1·918(30)
Si–C(10)	1·872(27)	Si–C(12)	1·866(18)
C(4)–C(5)	1·38(2)	C(15)–C(16)	1·40(2)
C(5)–C(6)	1·40(4)	C(16)–C(17)	1·45(3)
C(6)–C(7)	1·32(4)	C(17)–C(18)	1·30(4)
C(7)–C(8)	1·48(4)	C(18)–C(19)	1·33(3)
C(8)–C(9)	1·34(4)	C(19)–C(20)	1·39(4)
C(9)–C(4)	1·37(2)	C(20)–C(15)	1·38(3)

(*b*) Bond angles

P(1)–Pt–C(1)	93·6(5)	P(2)–Pt–C(1)	93·1(5)
P(1)–Pt–Cl	85·9(2)	P(2)–Pt–Cl	87·3(2)
P(1)–Pt–P(2)	173·2(2)	Cl–Pt–C(1)	178·1(4)
Pt–P(1)–C(2)	111·0(8)	Pt–P(2)–C(13)	113·6(8)
Pt–P(1)–C(3)	111·7(8)	Pt–P(2)–C(14)	119·1(7)
Pt–P(1)–C(4)	122·6(7)	Pt–P(2)–C(15)	112·2(5)
C(2)–P(1)–C(3)	105·1(11)	C(13)–P(2)–C(14)	101·5(8)
C(2)–P(1)–C(4)	100·0(10)	C(13)–P(2)–C(15)	103·7(8)
C(3)–P(1)–C(4)	104·6(8)	C(14)–P(2)–C(15)	105·1(9)
P(1)–C(4)–C(5)	121(1)	P(2)–C(15)–C(16)	119(2)
P(1)–C(4)–C(9)	117(1)	P(2)–C(15)–C(20)	122(1)
C(9)–C(4)–C(5)	121(2)	C(20)–C(15)–C(16)	191(2)
C(4)–C(5)–C(6)	118(2)	C(15)–C(16)–C(17)	118(2)
C(5)–C(6)–C(7)	120(2)	C(16)–C(17)–C(18)	118(2)
C(6)–C(7)–C(8)	123(3)	C(17)–C(18)–C(19)	125(2)
C(7)–C(8)–C(9)	114(2)	C(18)–C(19)–C(20)	119(3)
C(8)–C(9)–C(4)	124(2)	C(19)–C(20)–C(15)	120(2)
C(1)–Si–C(10)	111·6(8)	C(10)–Si–C(11)	108·6(14)
C(1)–Si–C(11)	108·2(11)	C(10)–Si–C(12)	106·9(9)
C(1)–Si–C(12)	112·9(9)	C(11)–Si–C(12)	108·6(10)
Pt–C(1)–Si	116·1(7)		

(*c*) Intramolecular contacts

Cl · · · P(1)	3·21	C(1) · · · P(1)	3·19
Cl · · · P(2)	3·25	C(1) · · · P(2)	3·18
Cl · · · C(2)	3·48	C(1) · · · C(4)	3·47
Cl · · · C(13)	3·32	C(1) · · · C(14)	3·39
P(1) · · · C(12)	3·85	P(2) · · · C(10)	3·97
Pt · · · Si	3·33		

(*d*) Intermolecular contacts <4 Å

C(17) · · · C(2^{I})	3·49	Cl · · · C(14^{II})	3·83
Cl · · · C(20^{II})	3·61	Cl · · · C(13^{II})	3·97
C(18) · · · Cl$^{\mathrm{I}}$	3·79	C(12) · · · C(2^{I})	3·99

Superscripts refer to the following transformations of fractional co-ordinates:

I $1 + x, y, z$ II $x, -y, \frac{1}{2} + z$

TABLE 3

Root-mean-square amplitudes of motion along principal axes of vibrational ellipsoid (Å)

	Axis (1)	Axis (2)	Axis (3)
Pt	0·178	0·188	0·220
Cl	0·174	0·252	0·337
P(1)	0·178	0·210	0·256
P(2)	0·178	0·184	0·239
Si	0·200	0·221	0·252
C(1)	0·164	0·183	0·264

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* See Notice to Authors No. 7 in *J.C.S. Dalton*, 1972, Index issue (items less than 10 pp. are supplied as full size copies).

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TABLE 4

Equations of mean planes through various sets of atoms, and, in square brackets, deviations (Å) of relevant atoms from the planes

Plane (*A*): Pt, Cl, P(1) P(2), C(1) $7{\cdot}686x - 0{\cdot}911y + 1{\cdot}573z = -0{\cdot}263$

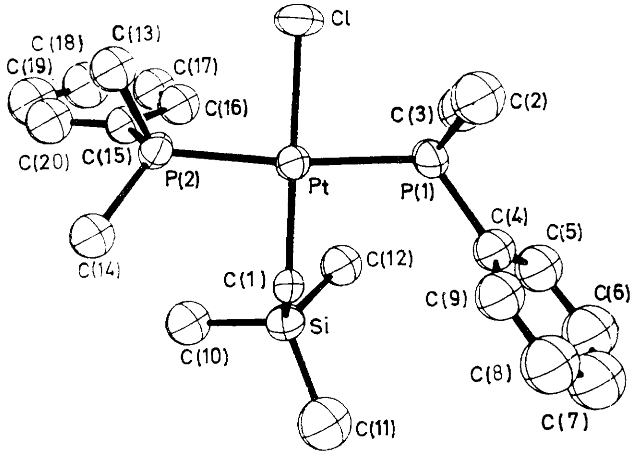
[Pt 0·009, Cl −0·025, P(1) 0·022, P(2) 0·021, C(1) −0·026, Si −1·696]

Plane (*B*): C(4)—(9) $-6{\cdot}955x + 4{\cdot}354y + 11{\cdot}896z = -2{\cdot}022$

[C(4) 0·009, C(5) 0·003, C(6) −0·014, C(7) 0·013, C(8) −0·001, C(9) −0·010, P(1) −0·119]

Plane (*C*): C(15)—(20) $-4{\cdot}032x + 18{\cdot}553y + 5{\cdot}799z = 3{\cdot}706$

[C(15) 0·019, C(16) −0·034, C(17) 0·037, C(18) −0·023, C(19) 0·005, C(20) −0·003, P(2) −0·025]



A perspective view of the molecular structure. The thermal motion ellipsoids enclose 50% of probability. The numbering scheme is shown

X-Ray '70 systems, the Hilger and Watts software package for the Y 290 diffractometer, and the HILGA data processing program of M. A. Bush, K. W. Muir, and D. White.

DISCUSSION

The crystal structure contains discrete $[PtCl(Me_3SiCH_2)(PMe_2Ph)_2]$ molecules separated by van der Waals contacts (Table 2). The platinum co-ordination is distorted slightly but significantly from ideal square-planar geometry. The C–Pt–P angles average 93·3° whereas the mean Cl–Pt–P angle is 86·6°. Alternate ligand-donor atoms deviate in opposite directions from the mean co-ordination plane of the platinum atom by 0·02—0·03 Å (Table 4). We consider these minor distortions to be a consequence of overcrowding of the bulky phosphine and $Me_3SiCH_2^-$ ligands.

The geometries of the phosphine ligands are as expected. The mean P–C(sp^3), P–C(sp^2), and C(sp^2)–C(sp^2) bond lengths are 1·84(2), 1·83(3), and 1·38(2) Å. Interbond angles at the phosphorus atoms show the expected deviations from tetrahedral geometry. The mean interbond angle at a phenyl-ring carbon atom is 120°, and the deviations of these carbon atoms from the mean plane through each ring (Table 4) are without significance.

The four Si–C bonds are equal in length (χ^2 4·2 on three degrees of freedom). The weighted mean Si–C distance of 1·868(13) Å agrees well with the corresponding value of 1·860(13) Å in $Nb_2(CSiMe_3)_2(CH_2SiMe_3)_4$.[9] The silicon co-ordination is almost exactly tetrahedral; the mean C–Si–C interbond angle is 109·5°, and the root-mean-square deviation from this mean is 2·1°. The Pt–C–Si angle of 116·1(7)° is appreciably greater than normal interbond angles at sp^3 carbon atoms; comparable angles in $Nb_2(CSiMe_3)_2(CH_2SiMe_3)_4$ and $Mo_2(CH_2SiMe_3)_6$ are 119·8(6) and 121·1°.[8,9] We share the view[8,9] that this effect is steric, rather than electronic, in origin. Opening of the bond angle at C(1) leads to an intramolecular Pt ··· Si contact of 3·33 Å which would appear to preclude any significant bonding interaction between these atoms.

The mutually *trans*-Pt–P bonds are equal in length, mean 2·293(3) Å. Comparable Pt–P(*trans* to P) distances are: 2·268(8) in *trans*-$[PtHCl(PPh_2Et)_2]$,[15] 2·28 in *trans*-$[PtCl(SiMePh_2)(PMe_2Ph)_2]$,[3] and 2·315(4) Å in *trans*-$[PtBr_2(PEt_3)_2]$.[16] These results suggest that Pt–P (*trans* to P) bonds lengthen slightly when aryl substituents on phosphorus are replaced by alkyl substituents. The greater inductive electron release from alkyl groups would, on current theories,[3] be expected to increase the *trans*-influencing ability of a phosphine ligand. However, the range of Pt–P(*trans* to P) distances is fairly small, and reliable conclusions concerning the effects of substituents on, for example, Pt–Cl(*trans* to P) distances cannot be drawn from the bond-length data available at present.[17] Plainly more results are needed to substantiate the phosphine *trans*-influence series suggested here.

The Pt–C distance [2·079(14) Å] agrees well with the sum of the appropriate covalent radii[18] (2·08 Å) and with the Pt^{II}–C(sp^3) distance of 2·106(17) Å in $K[Pt(acac)_2Cl]$.[19] The Pt–C distance in $[PtCl(CH_2SiMe_3)(PMe_2Ph)_2]$ and the covalent radii of sp^3 and sp^2 hybridised carbon atoms yield a predicted value of 2·05 Å for a Pt^{II}–C(sp^2) single bond, in agreement with the value of 2·043(13) Å in $[Pt(Ph)(PEt_3)_2(GePh_2OH)]$[20] where the σ-bonded phenyl group is *trans* to phosphine. In *cis*- and *trans*-$[PtCl_2\{C(NPhCH_2)_2\}(PEt_3)]$ the Pt–C distances have also been found to be insensitive to replacement of chlorine by phosphine in the *trans*-co-ordination site.[21] On the other hand, the sensitivity of Pt–C(π-olefin) distances to the nature of the *trans*-ligand is well established.[22]

The Pt–Cl(*trans* to C) distance [2·415(5) Å] agrees

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well with the comparable value (2·430 Å) in *cis*-[$PtCl_2CH(CH_2{\cdot}NH_2{\cdot}CHMePh)\{CH_2\}_2CH{:}CH_2$].[23] Pt–Cl (*trans* to P) distances typically lie in the range 2·36—2·39 Å[17] whereas the Pt–Cl(*trans* to Si) distance in [$PtCl(SiMePh_2)(PMe_2Ph)_2$] is 2·45 Å.[3] The Pt–Cl distances thus lead to a relative *trans*-influence series: $SiMePh_2^-$ > alkyl > tertiary phosphine. An identical series has been obtained from i.r. and n.m.r. data.[2] There is no suggestion from either structural or spectroscopic results that $CH_2SiMe_3^-$ is appreciably different in its electronic effects from other alkyl ligands in platinum(II) complexes.

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