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The structure of *cis*-dichloro-bis-(triphenylphosphine)platinum(II); a product in the reaction of *cis*-[$PtCl_2(CO)(PPh_3)$] with tin(II) chloride

G. K. ANDERSON, H. C. CLARK, J. A. DAVIES,
G. FERGUSON,* and M. PARVEZ
Chemistry Department
The University of Guelph,
Guelph, Ontario N1G 2W1, Canada

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Abstract

The reaction of *cis*-[$PtCl_2(CO)(PPh_3)$] with two equivalents of $SnCl_2 \cdot 2H_2O$ in acetone solution allows isolation of *cis*-[$PtCl_2(PPh_3)_2$] as the acetone solvate, the crystal structure of which has been determined. Crystals of *cis*-[$PtCl_2(PPh_3)_2$] · C_3H_6O are monoclinic, space group $P2_1/c$ (No. 14) with four formula units in a cell of dimensions $a = 10.288(3)$, $b = 24.372(5)$, $c = 15.367(3)$ Å, $\beta = 98.07(2)°$. The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations to a final R of 0.033 for 3761 reflections with $I > 3\sigma(I)$. The crystal structure contains discrete well-resolved *cis*-$PtCl_2(PPh_3)_2$ molecules with acetone of solvation filling cavities in the crystal structure. The Pt coordination is slightly distorted square-planar with Pt–Cl 2.333(2) and 2.356(2), Pt–P 2.251(2) and 2.265(2) Å, P–Pt–P 97.8(1), and Cl–Pt–Cl 87.1(1)°.

Introduction

During the course of our studies on the catalytic activity of platinum(II) halide complexes in the presence of tin(II) chloride (Clark *et al.*, 1980; Anderson *et al.*, 1982a; Clark and Davies, 1981), we observed that acetone solutions of *cis*-[$PtCl_2(CO)(PPh_3)$], with two equivalents of $SnCl_2 \cdot 2H_2O$, affect the catalytic hydroformylation of terminal monoenes under mild

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conditions. In order to gain some insight into the chemistry of such platinum(II):tin(II) catalyst systems, the reaction between *cis*-[$PtCl_2(CO)(PPh_3)$] and two equivalents of $SnCl_2 \cdot 2H_2O$ was performed in acetone solution and the resulting orange-red solution was allowed to evaporate until well-formed crystals appeared.

Attempts to identify portions of the solid product by solution $^{31}P\{^1H\}$ NMR spectroscopy were initially frustrated by the insolubility of the compound in a range of organic solvents and, accordingly, a single-crystal X-ray structure determination was performed. The results of the structural determination, described below, were elucidated simultaneously with the collection of $^{31}P\{^1H\}$ NMR data for a dilute solution of the complex in dichloromethane. Both sets of data establish that the complex is *cis*-[$PtCl_2(PPh_3)_2$] (**1**), evidently formed by a rearrangement process occurring during the reaction of *cis*-[$PtCl_2(CO)(PPh_3)$] with tin(II) chloride. Consideration of the stoichiometry of the reaction demonstrates that *cis*-[$PtCl_2(PPh_3)_2$] is not the sole product of this rearrangement reaction and, indeed, subsequent studies of the *cis*-[$PtCl_2(CO)(PPh_3)$]:$SnCl_2 \cdot 2H_2O$ system by multinuclear magnetic resonance techniques (Anderson *et al.*, 1982b) have shown that the formation of *cis*-[$PtCl_2(PPh_3)_2$] is preceded by a set of rearrangement reactions which yields a number of ionic species.

Although *cis*-[$PtCl_2(PPh_3)_2$] (**1**) is a well-known compound, frequently employed as a synthetic precursor in coordination chemistry, no structural determination has been reported. Undoubtedly, the insolubility of this complex has been a major factor in preventing the preparation of suitable crystals for study by X-ray diffraction. In the present case, the slow rearrangement reaction that occurs in acetone solution, in which (**1**) is insoluble, affords crystals which proved to be (**1**) · acetone.

Experimental

cis-[$PtCl_2(CO)(PPh_3)$] was prepared as described previously (Anderson *et al.*, 1981). $^{31}P\{^1H\}$ NMR spectra were obtained for a $CH_2Cl_2/10\%CD_2Cl_2$ solution at 24.3 MHz on a Brüker WP60 spectrometer operating in the Fourier transform mode. More positive shifts represent deshielding.

Reaction of cis-[$PtCl_2(CO)(PPh_3)$] *with* $SnCl_2 \cdot 2H_2O$.

cis-[$PtCl_2(CO)(PPh_3)$] (0.1014 g) and $SnCl_2 \cdot 2H_2O$ (0.0825 g, 2 mol. equiv.) were mixed in acetone (5 ml). The orange-red solution was filtered and allowed to stand. Slow evaporation, alternated with cooling to 278 K, produced the crystalline product (**1**) · acetone in ~15% yield. The crystals were filtered, washed twice with diluted mother liquors, and allowed to air-dry. $^{31}P\{^1H\}$ NMR data: $\delta = 15.5$, $^1J(^{195}Pt, ^{31}P) = 3679$ Hz.

Crystal data. $C_{39}H_{36}Cl_2OP_2Pt$, $[PtCl_2(PPh_3)_2] \cdot C_3H_6O$, $M_r = 848.7$, monoclinic, $a = 10.288(3)$, $b = 24.372(5)$, $c = 15.367(3)$ Å, $\beta = 98.07(2)°$, $V = 3814.9$ Å^3, $Z = 4$, $D_c = 1.48$ g cm^{-3}, $F(000) = 1792$, Mo K_α radiation, $\lambda = 0.71069$ Å, μ(Mo K_α) = 41.1 cm^{-1}; space group $P2_1/c$ from systematic absences ($h0l$ when $l = 2n + 1$, $0k0$ when $k = 2n + 1$).

Accurate cell parameters were obtained by a least-squares refinement of the setting angles of 25 reflections (with θ in the range 10–15°) measured on an Enraf-Nonius CAD4 diffractometer. Intensity data were collected by the $\omega/2\theta$ method with a small (0.48 × 0.14 × 0.10 mm) crystal to a maximum θ of 22, and 5100 unique data were obtained. After corrections for Lorentz and polarization effects and for absorption, the data with $I > 3\sigma(I)$ (3761) were labeled observed and used in structure solution and refinement. The maximum and minimum values of transmission coefficients in the absorption correction were 0.694 and 0.531, respectively.

Structure Solution and Refinement

The coordinates of the Pt atom were obtained from an analysis of a three-dimensional Patterson function and the remaining nonhydrogen atoms were located in a heavy-atom-phased Fourier summation which revealed the presence of an acetone of solvation. Refinement was by full-matrix least-squares calculations (Sheldrick, 1976) initially with isotropic and then with anisotropic vibration parameters. A difference map computed at an intermediate stage in the refinement revealed maxima (0.6–1.0 eÅ^{-3}) in positions expected for all the phenyl hydrogen atoms; these were then allowed for in geometrically idealized positions (C–H, 0.95 Å) and included in the final rounds of calculations. Only an overall isotropic thermal parameter was refined for these hydrogens. In the final four refinement cycles a weighting scheme of the form $\sqrt{\omega} = 1/[\sigma^2 F + pF^2]^{1/2}$ was employed where the final p parameter was 0.0031 and the phenyl C atoms were constrained to form regular planar hexagons (C–C 1.395 Å). The acetone molecule had large thermal parameters for C and O and, as we could not discern any acetone protons from our difference maps, we did not include these in the final calculations. The scattering factors of Stewart *et al.* (1965) were used for the H atoms and those of Cromer and Mann (1968) for the nonhydrogen atoms; allowance was made for anomalous dispersion (Cromer and Liberman, 1970). Refinement converged with $R = 0.033$ and $R_w = [\Sigma w\Delta^2/\Sigma wF_o^2]^{1/2} = 0.037$ for the 3761 reflections with $I > 3\sigma(I)$. A final difference map was devoid of any significant features. Atomic coordinates and standard deviations are given in Table 1, and temperature factors in Table 2. Figure 1 shows an ORTEP (Johnson, 1971) view of the molecule with our numbering scheme, and Fig. 2 shows a stereoview of the crystal structure; details of molecular geometry are given in Table 3.

Table 1. *cis*-$[PtCl_2(PPh_3)_2]$ · acetone. Final fractional coordinates (Pt × 10^5, remainder × 10^4) with estimated standard deviations in parentheses[a]

Atom	*x*	*y*	*z*
Pt	9525(3)	65004(1)	5450(2)
Cl(1)	−432(2)	6135(1)	−647(1)
Cl(2)	−822(2)	7060(1)	800(1)
P(1)	2579(2)	5947(1)	237(1)
P(2)	2063(2)	6920(1)	1739(1)
O	2647(25)	3816(10)	4286(14)
C(1)	966(29)	3532(9)	3381(20)
C(2)	2412(38)	3470(10)	3784(19)
C(3)	2980(30)	3035(12)	3257(28)
C(11)	3110(4)	5500(2)	1158(4)
C(12)	4414(4)	5380(2)	1482(4)
C(13)	4705(4)	5006(2)	2169(4)
C(14)	3691(4)	4752(2)	2533(4)
C(15)	2387(4)	4872(2)	2209(4)
C(16)	2096(4)	5246(2)	1522(4)
C(21)	3939(5)	6311(2)	−116(4)
C(22)	3843(5)	6877(2)	−239(4)
C(23)	4870(5)	7165(2)	−533(4)
C(24)	5992(5)	6887(2)	−703(4)
C(25)	6087(5)	6320(2)	−580(4)
C(26)	5060(5)	6032(2)	−286(4)
C(31)	2179(6)	5476(2)	−678(3)
C(32)	2042(6)	5691(2)	−1527(3)
C(33)	1764(6)	5344(2)	−2251(3)
C(34)	1622(6)	4782(2)	−2125(3)
C(35)	1759(6)	4566(2)	−1276(3)
C(36)	2037(6)	4914(2)	−552(3)
C(41)	1033(6)	6938(2)	2610(4)
C(42)	494(6)	6445(2)	2851(4)
C(43)	−332(6)	6441(2)	3498(4)
C(44)	−620(6)	6930(2)	3903(4)
C(45)	−81(6)	7423(2)	3662(4)
C(46)	745(6)	7427(2)	3015(4)
C(51)	2458(5)	7630(2)	1546(3)
C(52)	3330(5)	7909(2)	2170(3)
C(53)	3706(5)	8444(2)	2006(3)
C(54)	3211(5)	8701(2)	1217(3)
C(55)	2340(5)	8422(2)	593(3)
C(56)	1963(5)	7886(2)	757(3)
C(61)	3627(5)	6662(2)	2306(4)
C(62)	3676(5)	6365(2)	3086(4)
C(63)	4883(5)	6202(2)	3540(4)
C(64)	6042(5)	6335(2)	3214(4)
C(65)	5993(5)	6632(2)	2434(4)
C(66)	4786(5)	6796(2)	1980(4)
H(12)	5104	5553	1234
H(13)	5593	4925	2389
H(14)	3889	4498	3001
H(15)	1697	4699	2457
H(16)	1208	5328	1301
H(22)	3079	7067	−123
H(23)	4805	7551	−617

Table 1. Continued

Atom	x	y	z
H(24)	6691	7083	−903
H(25)	6851	6130	−696
H(26)	5125	5646	−202
H(32)	2139	6075	−1612
H(33)	1671	5491	−2829
H(34)	1433	4545	−2618
H(35)	1662	4183	−1191
H(36)	2130	4767	26
H(42)	690	6113	2576
H(43)	−699	6105	3662
H(44)	−1182	6927	4343
H(45)	−277	7756	3938
H(46)	1112	7763	2851
H(52)	3667	7734	2707
H(53)	4300	8634	2431
H(54)	3468	9066	1106
H(55)	2005	8597	56
H(56)	1370	7696	332
H(62)	2886	7275	3308
H(63)	4917	6000	4071
H(64)	6865	6224	3523
H(65)	6783	6723	2212
H(66)	4752	6998	1449

[a]Hydrogen atoms are in their calculated positions.

Discussion

The analysis establishes unambiguously that the product isolated from the reaction of *cis*-$[PtCl_2(CO)PPh_3]$ with $SnCl_2 \cdot 2H_2O$ is *cis*-$[PtCl_2(PPh_3)_2]$ (Fig. 1) and shows that the crystal structure (Fig. 2) also contains acetone of solvation. There are no untoward intermolecular contacts, which are all of the van der Waals type; the acetone of solvation (loosely) fills what would have otherwise been voids in the crystal lattice.

The Pt-coordination is essentially planar (Table 3) and slightly distorted from square by the bulk of the phosphine ligands. In Table 4 the dimensions of other *cis*-$PtCl_2(L)_2$ systems (L = phosphine ligand) are compared. The Pt–P dimensions reported here for (**1**) [2.251 and 2.265(2)Å] lie within the range of values given in Table 4. The P–Pt–P angle in (**1**) [97.8(1)°] is significantly enlarged from 90° to relieve intraphosphine overcrowding, a feature common to many of the complexes listed in Table 4. The effects of intramolecular overcrowding are also evident in the P–Pt–Cl and Pt–P–C angles and in the orientation adopted by the phenyl rings. In the $Ph_3P(1)$ moiety, phenyl carbon C(31) is close to being fully eclipsed with Cl(1) [torsion angle Cl(1)–Pt-

Table 2. Anisotropic temperature fators[a] ($\times 10^3$) with esd's in parentheses

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
Pt	25(1)	37(1)	43(1)	−2(1)	4(1)	2(1)
Cl(1)	32(1)	64(1)	60(1)	−16(1)	−2(1)	−1(1)
Cl(2)	36(1)	67(1)	60(1)	−9(1)	5(1)	15(1)
P(1)	27(1)	37(1)	49(1)	−2(1)	6(1)	1(1)
P(2)	35(1)	44(1)	47(1)	−7(1)	1(1)	2(1)
O	318(26)	270(24)	239(21)	10(19)	−9(17)	−80(23)
C(1)	173(21)	180(22)	270(31)	28(17)	−33(21)	−53(16)
C(2)	304(37)	133(18)	181(23)	−19(15)	86(24)	−139(22)
C(3)	231(31)	203(25)	484(64)	42(30)	196(39)	2(21)
C(11)	38(4)	40(4)	53(5)	1(4)	4(4)	4(4)
C(12)	43(5)	57(6)	67(6)	1(4)	−2(4)	3(4)
C(13)	57(6)	67(6)	89(7)	16(6)	−12(5)	9(5)
C(14)	96(9)	60(6)	91(8)	29(6)	−15(7)	18(6)
C(15)	72(7)	67(7)	93(8)	28(6)	18(6)	−1(5)
C(16)	47(5)	53(5)	75(6)	0(5)	6(5)	−2(4)
C(21)	36(5)	53(5)	47(5)	−1(4)	4(4)	−3(4)
C(22)	49(5)	51(5)	66(6)	−7(4)	13(4)	−4(4)
C(23)	69(7)	69(7)	73(7)	7(5)	12(5)	−25(6)
C(24)	44(6)	122(10)	90(8)	31(7)	13(5)	−32(6)
C(25)	40(6)	118(9)	95(8)	11(7)	14(5)	9(6)
C(26)	44(5)	65(6)	75(6)	5(5)	23(5)	9(4)
C(31)	31(4)	46(5)	55(5)	−7(4)	6(4)	2(4)
C(32)	61(6)	46(5)	65(6)	−6(4)	15(4)	−6(4)
C(33)	70(7)	104(9)	52(6)	−12(6)	12(5)	−14(5)
C(34)	64(6)	60(6)	98(8)	−41(6)	19(6)	−15(5)
C(35)	62(6)	51(5)	90(7)	−14(5)	13(5)	−13(5)
C(36)	42(5)	50(5)	68(6)	−12(4)	6(4)	−6(4)
C(41)	38(5)	59(5)	38(4)	−10(4)	−4(4)	1(4)
C(42)	52(6)	49(6)	71(6)	−4(4)	9(5)	−4(4)
C(43)	61(6)	79(7)	60(6)	−13(5)	22(5)	−24(5)
C(44)	53(6)	100(8)	56(6)	−25(6)	12(5)	−12(6)
C(45)	64(6)	84(7)	60(6)	−20(5)	10(5)	10(5)
C(46)	57(5)	55(6)	60(6)	−20(5)	6(5)	1(4)
C(51)	44(5)	43(5)	57(5)	−7(4)	11(4)	3(4)
C(52)	54(5)	52(5)	76(6)	−13(5)	−2(5)	−7(5)
C(53)	65(7)	65(7)	114(10)	−13(6)	8(7)	−15(5)
C(54)	78(7)	51(6)	136(11)	13(7)	30(8)	−12(6)
C(55)	96(9)	48(6)	85(8)	1(5)	5(7)	−1(5)
C(56)	57(5)	51(5)	61(6)	6(4)	4(4)	3(4)
C(61)	41(5)	46(5)	45(5)	−8(4)	0(4)	4(4)
C(62)	53(6)	67(6)	71(7)	7(5)	3(5)	2(5)
C(63)	63(7)	88(8)	95(8)	8(6)	−18(6)	16(6)
C(64)	50(7)	112(9)	107(10)	14(7)	18(6)	30(7)
C(65)	28(5)	93(7)	104(9)	−8(6)	0(5)	−6(5)
C(66)	46(5)	55(5)	69(6)	−3(5)	−3(5)	6(4)

[a]Anisotropic thermal parameters U_{ij} ($Å^2 \times 10^3$) in the expression $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$.

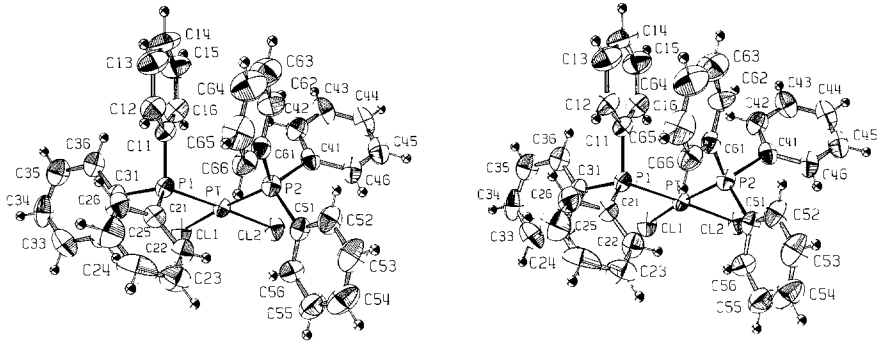

Fig. 1. A stereoview of *cis*-[$PtCl_2(PPh_3)_2$] showing the crystallographic numbering scheme.

-P(1)-C(31) −2.3(3)°] and the angle P(1)-Pt-Cl(1) [89.8(1)°] is significantly larger than the corresponding angle with the other P and Cl atoms [P(2)-Pt-Cl(2) 85.3(1)°] where *no* phenyl carbon comes close to being eclipsed with Cl(2) [e.g., Cl(2)-Pt-P(2)-C(41) 46.0(3)°]. As a consequence C(61) is almost eclipsed with P(1) [torsion angle P(1)-Pt-P(2)-C(61) −14.0(3)°]. The Cl(1)-Pt-P(1)-C(31) eclipsed conformation also results in the Pt-P(1)-C(31) angle [116.5(2)°] being larger than the other Pt-P(1)-C angles [110.3 and 113.5(2)°]; the P(1)-Pt-P(2)-C(61) orientation causes the Pt-P(2)-C(61) angle to be much enlarged [to 122.7(2)°] over the other Pt-P(2)-C angles [109.1 and 113.5(2)°]. All C-P-C angles are much less than tetrahedral [101.7-105.3(2)°] except for C(11)-P(1)-C(21) which is enlarged to 111.7(2)° to accommodate the approach of phenyl ring C(61)-C(66). The remaining

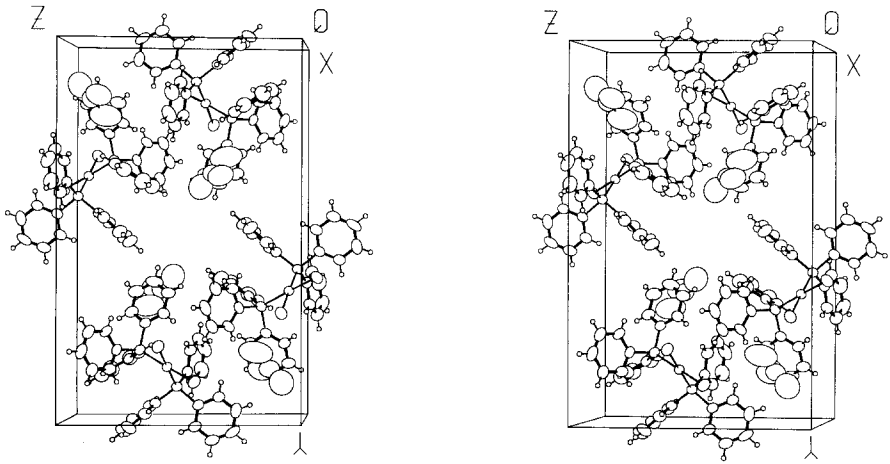

Fig. 2. A stereoview of the contents of a unit cell of *cis*-[$PtCl_2(PPh_3)_2$] · acetone.

Table. 3. *cis*-$[PtCl_2(PPh_3)_2]$ · acetone. Interatomic distances (Å) and angles (deg) with esd's in parentheses

(a) Distances

Pt–Cl(1)	2.333(2)	P(2)–C(41)	1.821(5)
Pt–Cl(2)	2.356(2)	P(2)–C(51)	1.810(6)
Pt–P(1)	2.251(2)	P(2)–C(61)	1.830(6)
Pt–P(2)	2.265(2)	O–C(2)	1.15(3)
P(1)–C(11)	1.808(6)	C(1)–C(2)	1.54(4)
P(1)–C(21)	1.802(5)	C(2)–C(3)	1.50(4)
P(1)–C(31)	1.816(5)		

Aromatic C–C constrained to be 1.395 Å.

(b) Angles

Cl(1)–Pt–Cl(2)	87.1(1)	Pt–P(2)–C(61)	122.7(2)
Cl(1)–Pt–P(1)	89.8(1)	C(11)–P(1)–C(21)	111.7(2)
Cl(1)–Pt–P(2)	172.4(1)	C(11)–P(1)–C(31)	103.7(2)
Cl(2)–Pt–P(1)	176.9(1)	C(21)–P(1)–C(31)	100.3(3)
Cl(2)–Pt–P(2)	85.3(1)	C(41)–P(2)–C(51)	105.3(2)
P(1)–Pt–P(2)	97.8(1)	C(41)–P(2)–C(61)	102.8(3)
Pt–P(1)–C(11)	110.3(2)	C(51)–P(2)–C(61)	101.7(2)
Pt–P(1)–C(21)	113.5(2)	O–C(2)–C(1)	107(4)
Pt–P(1)–C(31)	116.5(2)	O–C(2)–C(3)	145(4)
Pt–P(2)–C(41)	109.1(2)	C(1)–C(2)–C(3)	106(3)
Pt–P(2)–C(51)	113.5(2)		

Phenyl C–C–C angles constrained to be 120°.

(c) Torsion angles

P(2)Pt–P(1)C(11)	59.5(3)	P(1)Pt–P(2)C(51)	108.8(3)
P(2)Pt–P(1)C(21)	−66.8(3)	P(1)Pt–P(2)C(61)	−14.0(3)
P(2)Pt–P(1)C(31)	177.3(3)	Cl(1)Pt–P(1)C(31)	−2.3(3)
P(1)Pt–P(2)C(41)	−134.1(3)	Cl(2)Pt–P(2)C(41)	46.0(3)

(d) Deviations (Å) of atoms from the least-squares plane for Pt, P(1), P(2), Cl(1), and Cl(2)

Atom	deviation (Å)
Pt	0.003
P(1)	−0.006
P(2)	0.005
Cl(1)	0.005
Cl(2)	−0.006

dimensions [e.g., P–C 1.802–1.830(6) Å] in the phosphine moieties are unexceptional.

The orientation of the individual phenyl rings appear to be dictated by attempts to minimize intramolecular steric effects and there are no unduly short *ortho*-hydrogen · · · Pt interactions [shortest distance is Pt · · · H(22) 2.90 Å].

Del Pra and Zanotti (1979) comment on the difficulty of predicting

Table 4. Bond lengths and angles involving Pt, Cl, and P in *cis*-dichloro-bis(phosphine) platinum complexes

L in *cis*-$PtCl_2L_2$	Pt–Cl	Cl–Pt–Cl	Pt–P	P–Pt–P	Reference
P Ph_3	2.333(2) 2.356(2)	87.1(1)	2.251(2) 2.265(2)	97.8(1)	This work
P Me_3	2.368(3) 2.377(3)	87.9(1)	2.233(3) 2.243(3)	96.2(1)	Del Pra and Zanotti (1979)
$(Bu^t)_2P(CH_2)_2P(Bu^t)_2$	2.365(4) 2.374(3)	86.3(1)	2.262(3) 2.265(3)	89.4(1)	Harada *et al.* (1976)
$(Bu^t)_2P(CH_2)_3P(Bu^t)_2$	2.359(3) 2.362(3)	83.2(1)	2.281(3) 2.282(3)	99.1(1)	Harada *et al.* (1979)
$Ph_2PC{:}CCF_3$	2.357(5) 2.367(5)	88.6(1)	2.202(5) 2.248(4)	91.7(1)	Carty *et al.* (1976)
$P(C_6F_5)(CH_3)_2$	2.355(1) 2.332(1)	88	2.231(1) 2.240(1)	97	Manojlovic-Muir *et al.* (1978)

trans-influences in overcrowded phosphine complexes on the basis of Pt–P bond lengths. In (**1**), the Pt–Cl distances are significantly different [Pt–Cl(1) 2.333(2), Pt–Cl(2) 2.356(2) Å] even though both Cl atoms are *trans* to PPh_3 moieties. It is interesting to note that in (**1**) the longer Pt–P distance is *trans* to the shorter Pt–Cl distance and *vice-versa*. Thus, for example, $Ph_3P(2)$ [which suffers the greater overcrowding as measured by the Pt–P(2)–C(61) angle, 122.7(2)°] has the longer Pt–P distance [2.265(2) Å] and is *trans* to Cl(1), which has the shorter Pt–Cl distance [2.333(2) Å]. Notwithstanding the differences in the Pt–P and Pt–Cl bond lengths, it is worthwhile noting that the sums of the *trans* Pt–P and Pt–Cl distances are very similar [4.598(3) and 4.607(3) Å, mean 4.603 Å].

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