Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(*cis*-1,4-diphenyl-1,4-diphosphacyclohexane-κ^2P,P')platinum(II) bis(tetrafluoridoborate)

Tara S. Morey,[a] Susie M. Miller[b] and Monte L. Helm[a]*

[a]Chemistry Department, 1000 Rim Drive, Durango, CO 81301, USA, and [b]Chemistry Department, Colorado State University, Fort Collins, CO 80523, USA
Correspondence e-mail: helm_m@fortlewis.edu

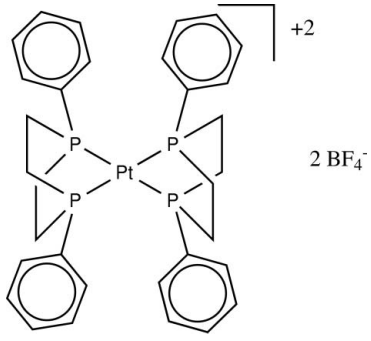
Received 31 May 2007; accepted 20 June 2007

Key indicators: single-crystal X-ray study; T = 373 K; mean σ(C–C) = 0.002 Å; R factor = 0.016; wR factor = 0.033; data-to-parameter ratio = 30.6.

The title complex, $[Pt(C_{16}H_{18}P_2)_2](BF_4)_2$, possesses a crystallographically imposed center of symmetry occupied by a Pt^{II} ion, which is coordinated by four P atoms from two 1,4-diphenyl-1,4-diphosphacyclohexane ligands in a distorted square-planar geometry.

Related literature

The corresponding complexes with both Pt^{II} and Pd^{II} have been previously reported as chloride salts (Mason *et al.*, 2006). For related literature, see: Brooks *et al.* (1989).



Experimental

Crystal data

$[Pt(C_{16}H_{18}P_2)_2](BF_4)_2$
M_r = 913.20
Tetragonal, $P4_12_12$
a = 10.83270 (15) Å
c = 29.1244 (6) Å
V = 3417.67 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 4.36 mm^{-1}
T = 373 (2) K
0.47 × 0.25 × 0.10 mm

Data collection

Bruker APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.281, T_{max} = 0.647
41078 measured reflections
6509 independent reflections
6151 reflections with $I > 2\sigma(I)$
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.016
$wR(F^2)$ = 0.033
S = 0.98
6509 reflections
213 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.69 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}
Absolute structure: Flack (1983)
Flack parameter: 0.007 (3) with 2711 Friedel pairs

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

The authors thank the American Chemical Society Petroleum Research Fund (grant No. 42268-GB3) for funding that supported this research.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RK2020).

References

Brooks, P. J., Gallagher, M. J., Sarroff, A. & Bowyer, M. (1989). *Phosphorus Sulfur Silicon Relat. Elem.* **44**, 235–247.
Bruker (2000). *SHELXTL* (Version 6.14) and *SADABS* (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (2006). *APEX2*. Version 2.0-2. Bruker AXS Inc., Madison, Wisconsin, USA.
Flack, H. D. (1983). *Acta Cryst.* A**39**, 876–881.
Mason, L. J., Perrault, E. M., Miller, S. M. & Helm, M. L. (2006). *Inorg. Chem. Commun.* **9**, 946–948.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

 doi:10.1107/S1600536807030115

supplementary materials

Acta Cryst. (2007). E**63**, m1983 [**doi:10.1107/S1600536807030115**]

Bis(*cis*-1,4-diphenyl-1,4-diphosphacyclohexane-κ^2P,P')platinum(II) bis(tetrafluoridoborate)

T. S. Morey, S. M. Miller and M. L. Helm

Comment

The crystal structure of (I) consists of $[Pt(C_{16}H_{18}P_2)_2]^{2+}$ cations and BF_4^- anions (Fig. 1). The cation shows a distorted square planar geometry around the metal center with two non-coordinating BF_4^- anions. The P—Pt—P bite angle observed in the structure is 72.908 (13)°. Additionally, the phenyl substituents on the phosphorus atoms are arranged in a parallel fashion, with a distance between the center of the phenyl rings of 3.727 Å and a dihedral angle of 2.15 (4)° between the rings. The parallel arrangement of phenyl rings is not, however, repeated in the unit cell between individual molecules. The parallel arrangement of the phenyl rings within the molecule was also observed in the previously reported analogous Pt^{II} an Pd(II) chloride complexes (Mason *et al.*, 2006).

Experimental

The synthesis of (I) was completed through reaction of the previously reported chloride salt with an excess of $AgBF_4$, followed by filtration to remove the solid AgCl (Mason *et al.*, 2006, Brooks *et al.*, 1989). Crystals where grown by slow vapor diffusion of diethylether into a saturated chloroform solution of (I).

Refinement

All H atoms were placed in calculated positions and constrained to ride on their parent atoms, with C—H distances in the range of 0.93–0.97 Å, with $U_{iso}(H)$ = 1.2 times $U_{eq}(C)$.

Figures

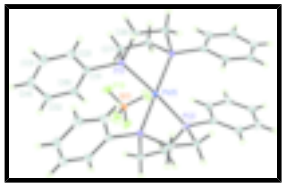


Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids. H–atoms are drawn as spheres with arbitrary radius.

Bis(*cis*-1,4-diphenyl-1,4-diphosphacyclohexane-κ^2P,P')platinum(II) bis(tetrafluoridoborate)

Crystal data

$[Pt(C_{16}H_{18}P_2)_2](BF_4)_2$	Z = 4
M_r = 913.20	F_{000} = 1792
Tetragonal, $P4_12_12$	D_x = 1.775 Mg m^{-3}
Hall symbol: P 4abw 2nw	Melting point: not measured K

a = 10.83270 (15) Å	Mo $K\alpha$ radiation λ = 0.71073 Å
b = 10.83270 (15) Å	Cell parameters from 8065 reflections
c = 29.1244 (6) Å	θ = 2.3–33.2º
α = 90º	μ = 4.36 mm^{-1}
β = 90º	T = 373 (2) K
γ = 90º	Prismatic, colorless
V = 3417.67 (10) $Å^3$	0.47 × 0.25 × 0.10 mm

Data collection

Bruker APEXII area-detector diffractometer	6509 independent reflections
Radiation source: fine-focus sealed tube	6151 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.028
T = 373(2) K	θ_{max} = 33.2º
φ and ω scans	θ_{min} = 2.0º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −16→16
T_{min} = 0.281, T_{max} = 0.647	k = −16→16
41078 measured reflections	l = −44→44

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.016	$w = 1/[\sigma^2(F_o^2) + (0.0084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.033	$(\Delta/\sigma)_{max}$ = 0.003
S = 0.98	$\Delta\rho_{max}$ = 0.69 e $Å^{-3}$
6509 reflections	$\Delta\rho_{min}$ = −0.87 e $Å^{-3}$
213 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983)
Secondary atom site location: difference Fourier map	Flack parameter: 0.007 (3)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R–factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R–factors based on F^2 are statistically about twice as large as those based on F, and R–factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Pt1	0.832348 (5)	0.832348 (5)	0.0000	0.01048 (2)
P1	0.84079 (4)	1.00450 (4)	0.045922 (11)	0.01195 (6)
P2	0.84132 (4)	0.64859 (3)	−0.040032 (12)	0.01251 (7)
C1	0.97255 (15)	1.10328 (14)	0.05191 (5)	0.0148 (3)
C2	0.96273 (16)	1.23257 (15)	0.04781 (6)	0.0184 (3)
H2A	0.8872	1.2691	0.0411	0.022*
C3	1.06751 (18)	1.30451 (16)	0.05401 (6)	0.0234 (4)
H3A	1.0617	1.3900	0.0518	0.028*
C4	1.18032 (18)	1.25058 (17)	0.06340 (6)	0.0247 (4)
H4A	1.2497	1.2999	0.0676	0.030*
C5	1.19046 (15)	1.12267 (17)	0.06662 (6)	0.0221 (3)
H5A	1.2667	1.0865	0.0725	0.026*
C6	1.08678 (15)	1.04949 (15)	0.06097 (5)	0.0180 (3)
H6A	1.0934	0.9641	0.0632	0.022*
C7	0.80596 (14)	0.94231 (14)	0.10316 (5)	0.0150 (3)
H7A	0.8759	0.8950	0.1143	0.018*
H7B	0.7912	1.0097	0.1244	0.018*
C8	0.70129 (14)	1.09169 (15)	0.03316 (5)	0.0163 (3)
H8A	0.6819	1.1469	0.0584	0.020*
H8B	0.7130	1.1407	0.0056	0.020*
C9	0.85797 (14)	0.68966 (14)	−0.10070 (5)	0.0158 (3)
H9A	0.8950	0.6219	−0.1176	0.019*
H9B	0.7778	0.7074	−0.1140	0.019*
C10	0.99765 (16)	0.59454 (13)	−0.02600 (5)	0.0171 (3)
H10A	1.0004	0.5669	0.0057	0.021*
H10B	1.0192	0.5254	−0.0456	0.021*
C11	0.73590 (14)	0.52277 (14)	−0.03228 (5)	0.0152 (3)
C12	0.70222 (14)	0.49227 (15)	0.01255 (5)	0.0158 (3)
H12A	0.7375	0.5341	0.0371	0.019*
C13	0.61647 (15)	0.40002 (15)	0.02062 (5)	0.0180 (3)
H13A	0.5941	0.3799	0.0505	0.022*
C14	0.56379 (15)	0.33727 (16)	−0.01647 (6)	0.0192 (3)
H14A	0.5052	0.2762	−0.0112	0.023*
C15	0.59824 (17)	0.36542 (15)	−0.06104 (6)	0.0223 (4)
H15A	0.5640	0.3220	−0.0855	0.027*
C16	0.68378 (16)	0.45819 (14)	−0.06942 (5)	0.0200 (3)
H16A	0.7064	0.4775	−0.0994	0.024*
B1	1.00964 (16)	0.65886 (17)	0.10947 (5)	0.0167 (3)
F1	1.01041 (11)	0.72699 (9)	0.14989 (4)	0.0289 (2)
F2	1.07033 (10)	0.54795 (10)	0.11657 (4)	0.0334 (3)
F3	0.88695 (10)	0.63397 (11)	0.09696 (4)	0.0319 (3)
F4	1.06599 (11)	0.72618 (12)	0.07492 (4)	0.0350 (3)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pt1	0.01143 (2)	0.01143 (2)	0.00858 (3)	0.00085 (3)	0.00064 (2)	−0.00064 (2)
P1	0.01367 (15)	0.01235 (14)	0.00981 (13)	0.00177 (18)	−0.00029 (13)	−0.00078 (13)
P2	0.01565 (16)	0.01150 (16)	0.01038 (13)	0.00070 (17)	0.00073 (13)	−0.00081 (12)
C1	0.0182 (7)	0.0147 (7)	0.0114 (6)	−0.0019 (6)	0.0002 (5)	−0.0015 (5)
C2	0.0240 (8)	0.0171 (7)	0.0143 (7)	0.0012 (7)	0.0012 (6)	0.0002 (6)
C3	0.0348 (9)	0.0178 (8)	0.0177 (7)	−0.0063 (8)	0.0012 (7)	−0.0003 (6)
C4	0.0259 (9)	0.0275 (8)	0.0208 (7)	−0.0122 (8)	0.0007 (7)	−0.0016 (6)
C5	0.0172 (8)	0.0300 (8)	0.0190 (7)	−0.0040 (7)	−0.0025 (6)	−0.0013 (6)
C6	0.0197 (7)	0.0180 (7)	0.0165 (7)	0.0010 (7)	−0.0025 (6)	−0.0010 (6)
C7	0.0180 (7)	0.0175 (7)	0.0094 (6)	0.0006 (6)	−0.0012 (5)	−0.0007 (5)
C8	0.0183 (7)	0.0147 (6)	0.0158 (7)	0.0039 (6)	−0.0004 (5)	−0.0013 (5)
C9	0.0192 (7)	0.0165 (7)	0.0116 (6)	−0.0016 (6)	−0.0002 (5)	−0.0010 (5)
C10	0.0197 (7)	0.0142 (6)	0.0174 (6)	0.0037 (7)	−0.0004 (6)	0.0008 (5)
C11	0.0186 (7)	0.0125 (7)	0.0145 (6)	0.0016 (6)	0.0003 (5)	−0.0006 (5)
C12	0.0210 (7)	0.0154 (6)	0.0111 (5)	0.0010 (7)	−0.0018 (5)	−0.0008 (5)
C13	0.0216 (8)	0.0157 (7)	0.0168 (6)	0.0002 (6)	0.0004 (6)	0.0037 (6)
C14	0.0212 (7)	0.0143 (6)	0.0222 (7)	−0.0010 (7)	−0.0016 (6)	0.0014 (6)
C15	0.0280 (8)	0.0193 (7)	0.0196 (7)	−0.0043 (7)	−0.0037 (6)	−0.0041 (6)
C16	0.0257 (8)	0.0188 (7)	0.0154 (6)	−0.0020 (7)	−0.0001 (6)	−0.0014 (5)
B1	0.0159 (7)	0.0186 (8)	0.0157 (6)	−0.0001 (7)	0.0008 (6)	−0.0001 (6)
F1	0.0368 (6)	0.0281 (5)	0.0220 (5)	0.0017 (6)	0.0052 (5)	−0.0078 (4)
F2	0.0316 (6)	0.0203 (5)	0.0484 (7)	0.0082 (5)	−0.0111 (5)	−0.0084 (5)
F3	0.0193 (5)	0.0343 (6)	0.0422 (7)	−0.0048 (5)	−0.0082 (5)	0.0085 (5)
F4	0.0357 (6)	0.0486 (7)	0.0208 (5)	−0.0118 (6)	0.0064 (5)	0.0078 (5)

Geometric parameters (Å, °)

Pt1—P1	2.2967 (4)	C8—C10^{i}	1.555 (2)
Pt1—P1^{i}	2.2967 (4)	C8—H8A	0.9700
Pt1—P2	2.3089 (3)	C8—H8B	0.9700
Pt1—P2^{i}	2.3089 (3)	C9—C7^{i}	1.558 (2)
P1—C1	1.7924 (16)	C9—H9A	0.9700
P1—C8	1.8204 (16)	C9—H9B	0.9700
P1—C7	1.8372 (15)	C10—C8^{i}	1.555 (2)
P2—C11	1.7924 (16)	C10—H10A	0.9700
P2—C9	1.8309 (15)	C10—H10B	0.9700
P2—C10	1.8379 (17)	C11—C12	1.395 (2)
C1—C6	1.393 (2)	C11—C16	1.407 (2)
C1—C2	1.410 (2)	C12—C13	1.384 (2)
C2—C3	1.389 (2)	C12—H12A	0.9300
C2—H2A	0.9300	C13—C14	1.398 (2)
C3—C4	1.382 (3)	C13—H13A	0.9300
C3—H3A	0.9300	C14—C15	1.384 (2)
C4—C5	1.393 (3)	C14—H14A	0.9300

C4—H4A	0.9300	C15—C16	1.389 (2)
C5—C6	1.385 (2)	C15—H15A	0.9300
C5—H5A	0.9300	C16—H16A	0.9300
C6—H6A	0.9300	B1—F4	1.3845 (19)
C7—C9^{i}	1.558 (2)	B1—F2	1.385 (2)
C7—H7A	0.9700	B1—F1	1.3894 (18)
C7—H7B	0.9700	B1—F3	1.404 (2)
P1—Pt1—P1^{i}	105.93 (2)	C10^{i}—C8—P1	107.75 (11)
P1—Pt1—P2	172.928 (14)	C10^{i}—C8—H8A	110.2
P1^{i}—Pt1—P2	72.908 (13)	P1—C8—H8A	110.2
P1—Pt1—P2^{i}	72.908 (13)	C10^{i}—C8—H8B	110.2
P1^{i}—Pt1—P2^{i}	172.928 (13)	P1—C8—H8B	110.2
P2—Pt1—P2^{i}	109.12 (2)	H8A—C8—H8B	108.5
C1—P1—C8	111.79 (7)	C7^{i}—C9—P2	107.38 (10)
C1—P1—C7	107.11 (7)	C7^{i}—C9—H9A	110.2
C8—P1—C7	101.83 (7)	P2—C9—H9A	110.2
C1—P1—Pt1	124.97 (5)	C7^{i}—C9—H9B	110.2
C8—P1—Pt1	105.63 (5)	P2—C9—H9B	110.2
C7—P1—Pt1	102.85 (5)	H9A—C9—H9B	108.5
C11—P2—C9	111.66 (7)	C8^{i}—C10—P2	109.69 (10)
C11—P2—C10	108.47 (7)	C8^{i}—C10—H10A	109.7
C9—P2—C10	101.61 (7)	P2—C10—H10A	109.7
C11—P2—Pt1	124.41 (5)	C8^{i}—C10—H10B	109.7
C9—P2—Pt1	106.38 (5)	P2—C10—H10B	109.7
C10—P2—Pt1	101.61 (5)	H10A—C10—H10B	108.2
C6—C1—C2	119.91 (16)	C12—C11—C16	119.80 (14)
C6—C1—P1	118.43 (12)	C12—C11—P2	117.68 (12)
C2—C1—P1	121.66 (13)	C16—C11—P2	122.48 (12)
C3—C2—C1	119.01 (16)	C13—C12—C11	120.34 (14)
C3—C2—H2A	120.5	C13—C12—H12A	119.8
C1—C2—H2A	120.5	C11—C12—H12A	119.8
C4—C3—C2	120.76 (16)	C12—C13—C14	119.59 (14)
C4—C3—H3A	119.6	C12—C13—H13A	120.2
C2—C3—H3A	119.6	C14—C13—H13A	120.2
C3—C4—C5	120.23 (17)	C15—C14—C13	120.48 (15)
C3—C4—H4A	119.9	C15—C14—H14A	119.8
C5—C4—H4A	119.9	C13—C14—H14A	119.8
C6—C5—C4	119.84 (17)	C14—C15—C16	120.28 (15)
C6—C5—H5A	120.1	C14—C15—H15A	119.9
C4—C5—H5A	120.1	C16—C15—H15A	119.9
C5—C6—C1	120.24 (15)	C15—C16—C11	119.50 (15)
C5—C6—H6A	119.9	C15—C16—H16A	120.2
C1—C6—H6A	119.9	C11—C16—H16A	120.2
C9^{i}—C7—P1	109.84 (10)	F4—B1—F2	110.86 (14)
C9^{i}—C7—H7A	109.7	F4—B1—F1	109.47 (14)
P1—C7—H7A	109.7	F2—B1—F1	109.35 (12)

C9^{i}—C7—H7B	109.7	F4—B1—F3	109.26 (13)
P1—C7—H7B	109.7	F2—B1—F3	108.75 (14)
H7A—C7—H7B	108.2	F1—B1—F3	109.12 (13)

Symmetry codes: (i) *y*, *x*, −*z*.

Fig. 1