Table 2. *Selected geometric parameters* (Å, °)

P1—Ni	2.190 (1)	C2′—C2	1.502 (5)
P2—Ni	2.182 (1)	C3—C2	1.390 (6)
N—Ni	1.932 (3)	C4—C3	1.379 (6)
C1—Ni	1.910 (4)	C4′—C4	1.512 (6)
C13—P1	1.807 (5)	C5—C4	1.376 (6)
C14—P1	1.789 (5)	C6—C5	1.394 (6)
C15—P1	1.811 (4)	C6′—C6	1.511 (5)
C16—P2	1.819 (4)	C8—C7	1.404 (5)
C17—P2	1.809 (4)	C12—C7	1.394 (5)
C18—P2	1.819 (5)	C9—C8	1.378 (6)
C7—N	1.354 (5)	C10—C9	1.372 (6)
C2—C1	1.411 (5)	C11—C10	1.366 (7)
C6—C1	1.405 (5)	C12—C11	1.372 (7)
P1—Ni—P2	177.78 (8)	C6—C1—Ni	121.5 (3)
P1—Ni—N	94.36 (10)	C2′—C2—C3	119.2 (4)
P2—Ni—N	87.29 (10)	C2′—C2—C1	119.6 (4)
P2—Ni—C1	88.50 (10)	C3—C2—C1	121.0 (3)
N—Ni—C1	175.72 (13)	C4—C3—C2	122.1 (4)
C1—Ni—P1	89.87 (10)	C4′—C4—C5	120.9 (4)
C13—P1—C14	103.0 (3)	C4′—C4—C3	121.8 (4)
C13—P1—C15	99.8 (2)	C5—C4—C3	117.2 (4)
C13—P1—Ni	116.32 (12)	C6—C5—C4	122.5 (4)
C14—P1—C15	102.5 (2)	C6′—C6—C1	121.2 (3)
C14—P1—Ni	115.0 (2)	C6′—C6—C5	118.1 (3)
C15—P1—Ni	117.8 (2)	C1—C6—C5	120.6 (3)
C16—P2—C17	103.5 (2)	C8—C7—C12	115.2 (4)
C16—P2—C18	101.8 (2)	C8—C7—N	123.0 (3)
C16—P2—Ni	113.29 (15)	C12—C7—N	121.8 (4)
C17—P2—C18	102.6 (2)	C9—C8—C7	121.8 (4)
C17—P2—Ni	112.2 (2)	C10—C9—C8	120.9 (4)
C18—P2—Ni	121.42 (12)	C11—C10—C9	118.6 (5)
C7—N—Ni	129.9 (2)	C12—C11—C10	120.8 (4)
C2—C1—C6	116.5 (3)	C7—C12—C11	122.6 (4)
C2—C1—Ni	121.6 (3)		

The ω scan width was symmetrical over 1.2° about the $K\alpha_{1,2}$ maximum and the background was offset 1.0 and −1.0 in ω from the $K\alpha_{1,2}$ maximum. The scan speed was a variable 3–6° min^{-1} (depending upon intensity). The linear absorption coefficient was calculated using values from *International Tables for X-ray Crystallography* (1974, Vol. IV).

The structure was solved by the heavy-atom method from which the position of the Ni atom was found. The remainder of the non-H atoms were obtained from a difference Fourier map. The amide H and aromatic H atoms were obtained from a subsequent difference Fourier map and refined without constraints; their distances range from 0.86 (4) to 0.99 (4) Å, and their angles between 117 (2) and 121 (2)°, except for atom H10 which makes angles of 114 (2) and 127 (2)°. Their isotropic displacement parameters range from 0.043 (9) to 0.073 (12) Å^2. The methyl H atoms were calculated in idealized positions (0.96 Å) and given fixed displacement parameters.

Programs used: *SHELXTL-Plus* (Sheldrick, 1990) for cell refinement, data collection, data reduction, structure solution (direct methods) and molecular graphics; *SHELX*76 (Sheldrick, 1976) for structure refinement (full-matrix least squares); *FUER* (Larson, 1982) for geometric and parameter tables.

The authors would like to acknowledge the University of Florida, Division of Sponsored Research, for support of the crystallography work, and the donors of the Petroleum Research Fund, administered by the American Chemical Society.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and bond distances and angles involving H atoms have been deposited with the IUCr (Reference: BK1063). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.

Boncella, J. M. & Villanueva, L. A. (1994). *J. Organomet. Chem.* **465**, 297–304.

Bryndza, H. E. & Tam, W. (1988). *Chem. Rev.* **88**, 1163–1188.

Carmona, E., Paneque, M. & Poveda, M. L. (1989). *Polyhedron*, **8**, 285–291.

Fryzuk, M. D., MacNeil, P. A., Rettig, S. J., Secco, A. S. & Trotter, J. (1982). *Organometallics*, **1**, 918–930.

Fryzuk, M. D. & Montgomery, D. C. (1989). *Coord. Chem. Rev.* **95**, 1–40.

Lappert, M. F., Power, P. P., Sanger, A. R. & Srivastava, R. C. (1980). In *Metal and Metalloid Amides*. Chatham: Halsted Press.

Larson, S. B. (1982). *FUER. Program for Generating Positional and Thermal Parameters and Geometrical Quantities*. Univ. of Texas, Austin, TX, USA.

Penney, J. M., VanderLende, D. D., Boncella, J. M. & Abboud, K. A. (1995). *Acta Cryst.* Submitted.

Rahim, M., White, C., Rheingold, A. L. & Ahmed, K. J. (1993). *Organometallics*, **12**, 2401–2403.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Sheldrick, G. M. (1990). *SHELXTL-Plus*. Version 4.21/V. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

VanderLende, D. D., Abboud, K. A. & Boncella, J. M. (1994). *Inorg. Chem.* In preparation.

Villanueva, L. A., Abboud, K. A. & Boncella, J. M. (1994). *Organometallics*. Submitted.

Acta Cryst. (1995). C**51**, 593–595

cis-Dichloro[bis(diphenylphosphino)-ethane]palladium(II)

SATNAM SINGH AND NARENDRA KUMAR JHA*

Department of Chemistry, Indian Institute of Technology, New Delhi 110016, India

PUNIT NARULA AND TEJ PAL SINGH

Department of Biophysics, All India Institute of Medical Sciences, New Delhi 110029, India

(*Received* 1 *March* 1994; *accepted* 28 *June* 1994)

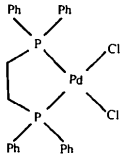
Abstract

In *cis*-[$PdCl_2(Ph_2PCH_2CH_2PPh_2)$], the Pd atom is at the centre of an approximately square-planar arrangement of two P and two Cl atoms. The P—Pd—P angle is 88.3 (1)° and the two Pd—P distances are 2.284 (3) and 2.264 (3) Å.

© 1995 International Union of Crystallography
Printed in Great Britain – all rights reserved

Comment

Only a few complexes are known in which $SbCl_3$ acts as a donor, *e.g.* $[Ni(CO)_3].SbCl_3$ and $[Fe(CO)_3].(SbCl_3)_2$ (Wilkinson, 1951). It was intended to explore the donor property of $SbCl_3$ further by reacting it with the d^8 square-planar complex $[PdCl_2(dppe)]$ (dppe = $Ph_2PCH_2CH_2PPh_2$). The 1:1 molar reaction of $[PdCl_2(dppe)]$ with $SbCl_3$ yielded a diamagnetic complex of the composition $[PdCl_2(dppe)].SbCl_3$. In order to obtain a single crystal for X-ray diffraction work, this complex was recrystallized from a mixture of CH_2Cl_2 and hexane. However, the composition of the crystals thus obtained proved to be $[PdCl_2(dppe)]$, (I), and not $[PdCl_2(dppe)].SbCl_3$. Nevertheless, we decided to determine the crystal structure of $[PdCl_2(dppe)]$ to compare it with that of $[PdCl_2(dppe)].CH_2Cl_2$ (Steffen & Palenik, 1976).



(I)

The crystals of the title complex are monoclinic and belong to the same space group ($P2_1/c$) as those of $[PdCl_2(dppe)].CH_2Cl_2$ (Steffen & Palenik, 1976). However, packing of the molecules in the unit cells of the solvated and unsolvated complexes is different. The unit-cell volume and density of the non-solvated complex are less than those of the solvated complex. Similar results have been reported in the analogous cases of $[ReCl_2(C_{26}H_{22}P_2)_2].C_6H_{14}$ (Lewis, Luck & Silverton, 1993) and $[ReCl_2(C_{26}H_{22}P_2)_2]$ (Bakir, Fanwick & Walton, 1986). Another interesting observation is that the Pd—P bond lengths and P—Pd—P angle are larger in the present case than in that of the solvated complex, *viz.* Pd—P1 and Pd—P2 are 2.284 (3) and 2.264 (3) Å, respectively, compared with 2.233 (2) and 2.226 (2) Å, respectively, in the solvated complex, and the P—Pd—P angle is 88.3 (1)° compared with 85.82 (7)° in the solvated complex. In addition, some P—C bond lengths are longer in the unsolvated complex. These differences may arise from the packing effects of the solvent molecule.

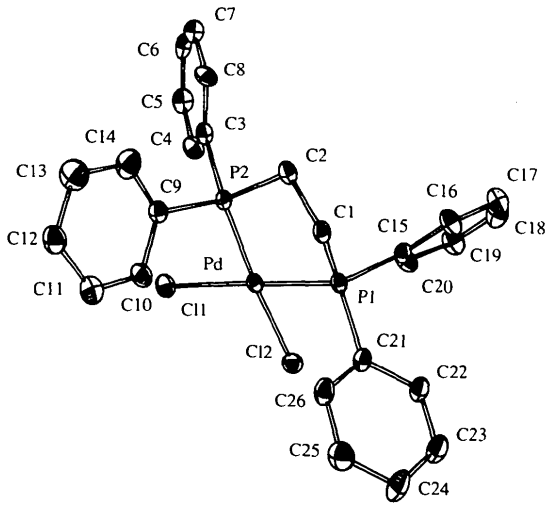


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the title compound, with displacement ellipsoids shown at the 50% probability level.

Experimental

Crystal data

$[PdCl_2(C_{26}H_{24}P_2)]$
M_r = 575.729
Monoclinic
$P2_1/c$
a = 11.925 (1) Å
b = 13.293 (3) Å
c = 16.314 (2) Å
β = 99.21 (1)°
V = 2552.7 (7) Å^3
Z = 4
D_x = 1.498 (5) Mg m^{-3}

Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 10–40°
μ = 9.69 mm^{-1}
T = 295 K
Plate
1.00 × 0.75 × 0.25 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical
T_{min} = 0.707, T_{max} = 0.995
3425 measured reflections
3243 independent reflections

2603 observed reflections [$F > 4\sigma(F)$]
R_{int} = 0.050
θ_{max} = 59.8°
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 14$
$l = -18 \rightarrow 18$
3 standard reflections frequency: 60 min intensity decay: 5%

Refinement

Refinement on F
R = 0.084
wR = 0.084
S = 3.22
2593 reflections
280 parameters
H-atom parameters not refined
$w = 0.5669/\sigma^2(F)$

$(\Delta/\sigma)_{max}$ = 0.072
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.10 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) for Pd and Cromer & Mann (1968) for all other non-H atoms

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Pd	0.12844 (7)	0.21945 (5)	0.70806 (5)	0.059 (4)
P1	−0.0296 (2)	0.2116 (2)	0.6086 (2)	0.035 (1)
P2	0.2338 (2)	0.1799 (2)	0.6089 (2)	0.040 (1)

Cl1	0.2981 (3)	0.2041 (2)	0.8102 (2)	0.056 (1)
Cl2	0.0060 (3)	0.2667 (2)	0.8044 (2)	0.059 (1)
C1	0.0171 (9)	0.1395 (8)	0.5228 (7)	0.042 (5)
C2	0.1346 (10)	0.1806 (8)	0.5079 (7)	0.047 (6)
C3	0.3567 (10)	0.2630 (8)	0.5915 (7)	0.046 (6)
C4	0.4024 (12)	0.3295 (9)	0.6589 (7)	0.052 (7)
C5	0.4930 (12)	0.3954 (10)	0.6429 (10)	0.054 (8)
C6	0.5324 (11)	0.3972 (10)	0.5680 (10)	0.074 (8)
C7	0.4855 (11)	0.3326 (10)	0.5056 (8)	0.061 (7)
C8	0.3966 (12)	0.2681 (9)	0.5187 (7)	0.055 (5)
C9	0.2873 (9)	0.0495 (8)	0.6173 (7)	0.045 (6)
C10	0.2465 (12)	−0.0124 (8)	0.6757 (8)	0.068 (7)
C11	0.2767 (13)	−0.1130 (10)	0.6793 (9)	0.074 (8)
C12	0.3508 (12)	−0.1514 (9)	0.6295 (9)	0.066 (8)
C13	0.3875 (15)	−0.0909 (11)	0.5708 (13)	0.102 (11)
C14	0.3533 (14)	0.0119 (10)	0.5658 (12)	0.102 (11)
C15	−0.0863 (9)	0.3299 (8)	0.5661 (7)	0.043 (5)
C16	−0.1414 (13)	0.3391 (8)	0.4836 (8)	0.057 (7)
C17	−0.1842 (14)	0.4323 (11)	0.4532 (9)	0.083 (9)
C18	−0.1792 (14)	0.5127 (10)	0.5040 (14)	0.093 (11)
C19	−0.1273 (14)	0.5065 (9)	0.5857 (10)	0.070 (8)
C20	−0.0781 (13)	0.4137 (8)	0.6154 (8)	0.069 (7)
C21	−0.1506 (9)	0.1410 (8)	0.6391 (8)	0.046 (6)
C22	−0.2615 (11)	0.1867 (9)	0.6427 (9)	0.061 (7)
C23	−0.3503 (12)	0.1332 (12)	0.6665 (11)	0.085 (9)
C24	−0.3341 (12)	0.0319 (12)	0.6893 (12)	0.094 (10)
C25	−0.2286 (15)	−0.0114 (10)	0.6848 (10)	0.088 (9)
C26	−0.1375 (11)	0.0431 (9)	0.6589 (9)	0.065 (7)

Table 2. *Selected geometric parameters* (Å, °)

Pd—Cl1	2.415 (3)	Pd—Cl2	2.394 (3)
Pd—P1	2.284 (3)	Pd—P2	2.264 (3)
P1—C1	1.85 (1)	P2—C2	1.87 (1)
C1—C2	1.56 (2)	P1—C15	1.81 (1)
P1—C21	1.85 (1)	P2—C3	1.89 (1)
P2—C9	1.84 (1)		
Cl2—Pd—Cl1	95.8 (1)	C15—P1—Pd	116.6 (3)
P1—Pd—Cl2	87.6 (1)	C15—P1—C1	107.5 (5)
P1—Pd—Cl1	172.4 (1)	C18—C19—C20	119 (1)
C2—P2—Pd	106.5 (4)	P1—C21—C22	123 (1)
C9—P2—Pd	113.0 (4)	C22—C21—C26	118 (1)
P2—Pd—Cl2	175.5 (1)	C22—C23—C24	119 (1)
P2—Pd—Cl1	88.5 (1)	C24—C25—C26	123 (1)
P2—Pd—P1	88.3 (1)	C9—P2—C3	107.0 (5)
C3—P2—Pd	120.2 (4)	C21—P1—Pd	114.9 (4)
C2—P2—C3	105.1 (5)	C21—P1—C1	106.4 (5)
C9—P2—C2	103.5 (5)	C15—P1—C21	106.5 (5)
C1—P1—Pd	104.3 (3)		

Data were corrected for Lorentz and polarization factors. The structure was solved by direct methods with *SHELXS*86 (Sheldrick, 1985). The structure was refined using *SHELX*76 (Sheldrick, 1976). H atoms were fixed geometrically. The perspective view of the title molecule was drawn using *ORTEP*II (Johnson, 1976) and geometrical analysis was performed using *PARST* (Nardelli, 1983).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1120). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Bakir, M., Fanwick, P. E. & Walton, R. A. (1987). *Polyhedron*, **6**, 907–913.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Lewis, D. J., Luck, R. L. & Silverton, J. V. (1993). *Acta Cryst.* C**49**, 1424–1426.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Sheldrick, G. M. (1976). *SHELX*76. *Program for Crystal Structure Determination*. Univ. of Cambridge, England.

Sheldrick, G. M. (1985). *SHELXS*86. *Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.

Steffen, W. L. & Palenik, G. J. (1976). *Inorg. Chem.* **15**, 2432–2438.

Wilkinson, G. (1951). *J. Am. Chem. Soc.* **73**, 5502.

©1995 International Union of Crystallography
Printed in Great Britain – all rights reserved

Acta Cryst. (1995). C**51**, 595–598

Azidotetrakis(trimethylphosphine)nickel(II) Tetrafluoroborate

CARYN C. CARSON AND ROBERT D. PIKE*

Department of Chemistry, College of William and Mary, Williamsburg, Virginia 23187, USA

GENE B. CARPENTER

Department of Chemistry, Brown University, Providence, Rhode Island 02912, USA

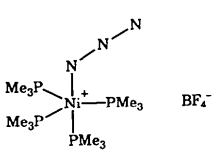
(*Received* 20 *June* 1994; *accepted* 15 *September* 1994)

Abstract

The title complex, $[Ni(N_3)(C_3H_9P)_4]BF_4$, is a nearly perfect trigonal bipyramid with the azide group at an apical position. The metal–azide bond angle, Ni1—N1—N2, of 138.6 (5)° is the largest observed for a terminal azide ligand.

Comment

Nickel(0) and nickel(I) reagents have significant utility in aryl-coupling reactions (Zembayashi, Tamao, Yoshida & Kumada, 1977; Semmelhack *et al.*, 1981; Rollin, Troupel, Tuck & Perichon, 1986; Amatore & Jutand, 1988; Zhou & Yamamoto, 1991). A potential source of such low-valency metal complexes are metal azides. This results from the ability of the azide to undergo photo-induced reductive elimination. As part of a study of nickel–azide–phosphine complexes, the azidotetrakis(trimethylphosphine)nickel(II) cation was synthesized as its BF_4^- salt, (1), and examined by X-ray crystallography. To our knowledge, this species is the only known five-coordinate nickel–azide complex.



(1)