Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

[(*R*)-4,4′-Bis(diphenylphosphino)-2,2′,6,6′-tetramethoxy-3,3′-bipyridine-κ^2P,P']dichloropalladium(II)

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Received 13 May 2002
Accepted 28 May 2002
Online 8 June 2002

Key indicators

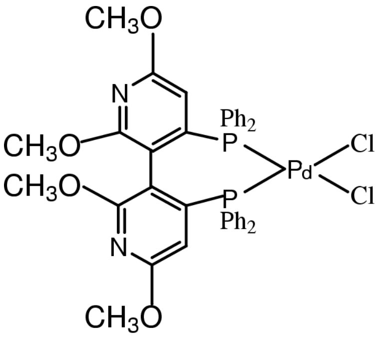
Single-crystal X-ray study
T = 294 K
Mean σ(C–C) = 0.009 Å
R factor = 0.068
wR factor = 0.155
Data-to-parameter ratio = 19.3

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, [$PdCl_2(C_{38}H_{34}N_2O_4P_2)$], is effective in enantioselective bis-alkoxycarbonylation of styrene. The Pd atoms are located on twofold rotation axes, and there are two independent half-molecules in the asymmetric unit. In the bipyridine systems, the pyridine rings make dihedral angles of 65.2 (6) and 67.9 (7)° with respect to each other.

Comment

In recent years, cationic palladium(II) complexes have attracted a great deal of attention in organometallic chemistry, especially because of their catalytic reactions (Drent & Budzelaar, 1996; Sen, 1993) and their application in the self-assembly of various metallamacrocycles (Stang & Olenyuk, 1996). Very recently, we used cationic palladium complexes with novel dipyridylphosphine ligands in the bis-methoxycarbonylation of styrene. In our studies, the enantiomer excess of dimethyl (*R*)-phenylbutanedioates has been improved to 88%. As part of our efforts in investigating these catalytic reactions and the molecular structure of the cation formed by palladium and 4,4′-bis(diphenylphosphine)-2,2′,6,6′-tetramethoxy-3,3′-bipyridine (P-Phos), we present the molecular structure of the title compound, (I).



(I)

The Pd1 and Pd2 atoms lie on twofold rotation axes. Fig. 1 shows the structure of one of two independent molecules. For the Pd1 complex, the least-squares planes of the two pyridine rings of the bipyridine system in the P-Phos ligand exhibit an interplanar angle of 65.2 (6)°, and the C5—C5^{i} distance is 1.511 (8) Å (see Table 1 for symmetry code). For the Pd2 complex, the corresponding dihedral angle is 67.9 (7)° with a C21—C21^{ii} distance of 1.510 (7) Å.

Experimental

All reactions and manipulations were carried out under N_2, using Schlenk techniques or in a glove-box. A solution of [{(*R*)-P-Phos}-

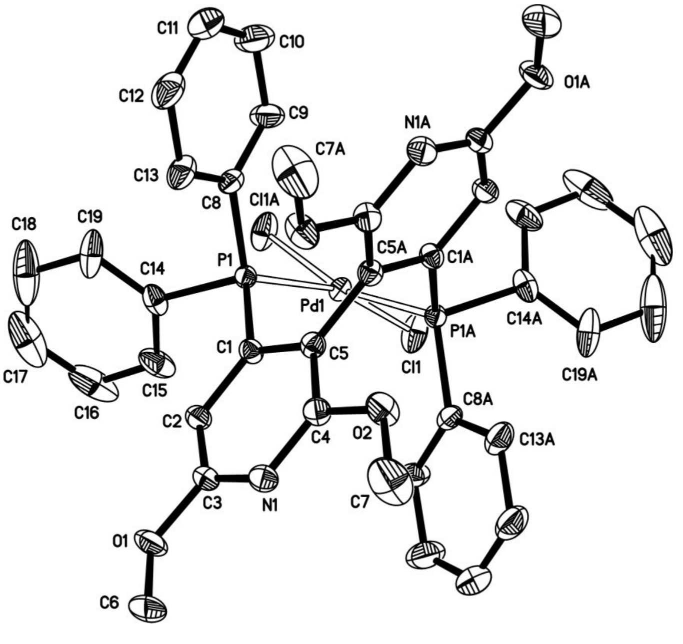


Figure 1
The structure of one of the independent molecules of (I), showing 30% probability displacement ellipsoids.

$Pd(H_2O)_2](SO_3CF_3)_2$ (87 mg, 0.085 mmol) in methanol (15 ml) was transferred under a nitrogen atmosphere to a 25 ml stainless steel autoclave. The autoclave was heated to 323 K under a CO pressure of 50 bar for 20 h. After the gas was released, the resulting red solution was evaporated *in vacuo*, and the red residue was dried overnight. Red crystals of (I), suitable for X-ray diffraction analysis, were obtained by recrystallization from a solution in $CHCl_3$. ^{31}P NMR ($CDCl_3$, p.p.m.): δ 30.6.

Crystal data

$[PdCl_2(C_{38}H_{34}N_2O_4P_2)]$
$M_r = 821.91$
Monoclinic, $C2$
$a = 19.551$ (3) Å
$b = 12.1135$ (19) Å
$c = 18.410$ (3) Å
$\beta = 119.529$ (3)°
$V = 3793.7$ (10) Å^3
$Z = 4$
$D_x = 1.439$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 7972 reflections
$\theta = 1$–27.5°
$\mu = 0.76$ mm^{-1}
$T = 294$ (2) K
Plate, red
0.28 × 0.24 × 0.22 mm

Data collection

Bruker CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.816$, $T_{max} = 0.851$
12 943 measured reflections

6304 independent reflections
5366 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 27.5°$
$h = -25 \rightarrow 24$
$k = -15 \rightarrow 9$
$l = -23 \rightarrow 23$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.068$
$wR(F^2) = 0.155$
$S = 1.13$
8354 reflections
433 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.05P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.64$ e Å^{-3}
$\Delta\rho_{min} = -0.35$ e Å^{-3}
Absolute structure: Flack (1983); 1740 Friedel pairs
Flack parameter = 0.01 (10)

Table 1
Selected geometric parameters (Å, °).

Pd1—P1	2.2504 (11)	Pd2—P2^{ii}	2.2681 (11)
Pd1—Cl1	2.3433 (14)	Pd2—Cl2	2.3167 (12)
C5—C5^{i}	1.511 (8)	C21—C21^{ii}	1.510 (7)
P1^{i}—Pd1—P1	94.57 (6)	P2^{ii}—Pd2—P2	93.26 (6)
P1—Pd1—Cl1^{i}	89.15 (5)	P2—Pd2—Cl2	89.97 (4)
Cl1^{i}—Pd1—Cl1	91.52 (8)	Cl2—Pd2—Cl2^{ii}	87.69 (6)

Symmetry codes: (i) $-x, y, -z$; (ii) $1 - x, y, 1 - z$.

The large elongated ellipsoids of the atom displacement parameters of C16 and C31 suggest orientational disorder of the phenyl groups.

Data collection: *SMART* (Bruker, 1997); cell refinement: *SMART*; data reduction: *SHELXTL-NT* (Bruker, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL-NT*; software used to prepare material for publication: *SHELXTL-NT*.

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