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trans-Dichloro(2-*n*-propylsulfinylpyridine-*N*)(triethylphosphine-*P*)palladium(II)

Ana M. G. Dias Rodrigues,[a] M. Teresa do P. Gambardella,[a] Regina H. P. Francisco,[a]* Gerimário F. de Sousa[b] and Carlos A. L. Filgueiras[c]

[a]*DQFM, IQSC, Universidade de São Paulo, Caixa Postal 780, 13560 250 São Carlos, SP, Brazil,* [b]*DQ, ICC, Universidade de Brasilia, 70910 900 Brasilia, DF, Brazil, and* [c]*DQ, ICEx, Universidade Federal de Minas Gerais, 30270 910 Belo Horizonte, MG, Brazil. E-mail: porto@ifqsc.sc.usp.br*
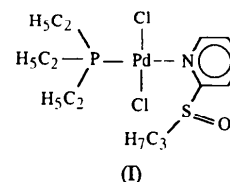
Abstract

The structure determination of the title compound, [PdCl$_2$(C$_6$H$_{15}$P)(C$_8$H$_{11}$NOS)], revealed the occurrence of discrete molecules, in which the Pd atom has square-planar coordination geometry, with distances Pd—P 2.232 (1), Pd—N 2.128 (3) and Pd—Cl 2.293 (2) and 2.294 (2) Å. The sulfoxide derivative acts as a monodentate ligand.

Comment

Investigations of the coordination chemistry of some transition and post-transition metals, namely Ni, Pd, Pt, Sn and Sb, with ambidentate S-, N- and O-donor ligands have revealed a variety of structural features,

with sulfoxide behaving as a mono- or bidentate ligand, or bridging two metal cations (Zhu, Shao, Yao, Wang & Wang, 1990; de Sousa & Filgueiras, 1990*a,b*; de Sousa, Filgueiras, Darensbourg & Reibenspies, 1992; Adedapo *et al.*, 1993; Francisco, Gambardella, Rodrigues, de Sousa & Filgueiras, 1995).

In the title compound, (I), the N-bonded 2-*n*-propyl-sulfinylpyridine group is monodentate. The coordination around the metal atom is completed by a P atom and two chloride anions, the four atoms arranged with *trans* square-planar geometry. Distances between each of these five atoms and their least-squares plane are in the range −0.0289 (3) to 0.032 (4) Å. The pyridine ring is almost perpendicular [dihedral angle 81.9 (1)°] to the plane containing the metal and its ligands. The displacement parameters are larger for the C atoms of the alkyl groups than for those of the ring. A view of one molecule is shown in Fig. 1.

(I)

The proton-decoupled ^{31}P NMR spectrum showed a chemical shift at 39.86 p.p.m. (relative to H$_3$PO$_4$) which agrees with a *trans*-Cl—Pd—Cl arrangement (Kunz & Pregosin, 1979). The IR spectra (CsI pellets) showed SO absorptions at 1055 and 1050 cm^{-1}, which were attributed to the stretching frequencies of the free ligand and the complex, respectively, suggesting little or no change of frequency upon complexation.
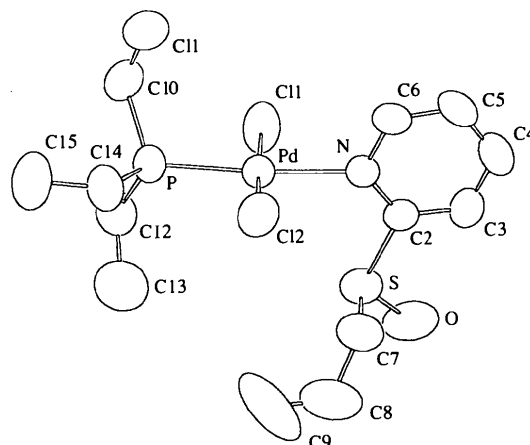

Fig. 1. *ORTEP* (Johnson, 1965) view of (I) showing the labelling of the non-H atoms and ellipsoids plotted at the 50% probability level.

Experimental

$[PdCl_2(C_6H_{15}P)(C_8H_{11}NOS)]$

Crystal data

$[PdCl_2(C_6H_{15}P)(C_8H_{11}NOS)]$
$M_r = 464.69$
Triclinic
$P\bar{1}$
$a = 8.0420\,(8)$ Å
$b = 8.4045\,(8)$ Å
$c = 15.174\,(1)$ Å
$\alpha = 105.021\,(8)°$
$\beta = 95.128\,(9)°$
$\gamma = 95.326\,(8)°$
$V = 979.4\,(2)$ Å^3
$Z = 2$
$D_x = 1.576$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 8$–$15°$
$\mu = 1.406$ mm^{-1}
$T = 298$ K
Prism
$0.40 \times 0.30 \times 0.20$ mm
Orange

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: refined from ΔF (DIFABS; Walker & Stuart, 1983)
$T_{min} = 0.836$, $T_{max} = 1.000$
4886 measured reflections
4711 independent reflections

3307 observed reflections $[F > 3\sigma(F)]$
$R_{int} = 0.0220$
$\theta_{max} = 27.97°$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 10$
$l = 0 \rightarrow 20$
2 standard reflections frequency: 120 min intensity decay: 3.7%

Refinement

Refinement on F
$R = 0.0427$
$wR = 0.0476$
$S = 1.33$
3307 reflections
202 parameters
H atoms riding with C—H = 1.08 Å
$w = 1/[\sigma^2(F) + 0.000738F^2]$

$(\Delta/\sigma)_{max} = 0.2$
$\Delta\rho_{max} = 0.685$ e Å^{-3}
$\Delta\rho_{min} = -0.862$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV, Table 2.3.1)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Pd	0.37603 (4)	0.32836(4)	0.21476 (3)	3.66 (1)
Cl1	0.2903 (2)	0.4029 (2)	0.0844 (1)	6.05 (4)
Cl2	0.4618 (2)	0.2467 (2)	0.3428 (1)	5.71 (4)
N	0.2816 (4)	0.0786 (5)	0.1438 (3)	3.7 (1)
C2	0.1192 (5)	0.0160 (5)	0.1283 (3)	3.8 (1)
C3	0.0578 (6)	−0.1369 (6)	0.0700 (3)	4.6 (1)
C4	0.1728 (8)	−0.2358 (7)	0.0281 (4)	5.5 (2)
C5	0.3417 (7)	−0.1743 (6)	0.0442 (4)	4.9 (2)
C6	0.3916 (6)	−0.0180 (6)	0.1026 (3)	4.2 (1)
S	−0.0252 (2)	0.1453 (2)	0.1860 (1)	5.71 (4)
O	−0.1970 (4)	0.0624 (7)	0.1461 (3)	7.6 (2)
C7	0.0144 (7)	0.088 (1)	0.2942 (4)	6.8 (2)
C8	−0.086 (1)	0.162 (1)	0.3692 (8)	10.9 (4)
C9	−0.042 (2)	0.318 (1)	0.407 (1)	14.5 (6)
P	0.4824 (2)	0.5929 (2)	0.2773 (1)	4.47 (4)
C10	0.6274 (8)	0.6639 (8)	0.2072 (4)	6.1 (2)
C11	0.7742 (9)	0.5660 (9)	0.1918 (6)	7.9 (3)
C12	0.3201 (8)	0.7322 (7)	0.2820 (5)	6.5 (2)
C13	0.205 (1)	0.715 (1)	0.3541 (7)	9.7 (3)
C14	0.5932 (8)	0.6419 (7)	0.3915 (4)	6.1 (2)
C15	0.667 (1)	0.8209 (9)	0.4326 (5)	8.9 (3)

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

Pd—Cl1	2.294 (2)	S—O	1.488 (4)
Pd—Cl2	2.293 (2)	S—C7	1.837 (8)
Pd—N	2.128 (3)	P—C10	1.808 (7)
Pd—P	2.232 (1)	P—C12	1.827 (7)
S—C2	1.794 (5)	P—C14	1.803 (7)
Cl1—Pd—Cl2	178.45 (5)	C2—S—O	106.4 (2)
Cl1—Pd—N	88.3 (1)	C2—S—C7	94.4 (3)
Cl1—Pd—P	87.60 (5)	O—S—C7	104.8 (4)
Cl2—Pd—N	90.3 (1)	Pd—P—C10	111.5 (2)
Cl2—Pd—P	93.80 (6)	Pd—P—C12	111.7 (2)
N—Pd—P	174.8 (1)	Pd—P—C14	116.5 (3)
Pd—N—C2	125.2 (3)	C10—P—C12	103.5 (3)
Pd—N—C6	117.0 (3)	C10—P—C14	106.1 (3)
C2—N—C6	117.4 (4)	C12—P—C14	106.7 (3)

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (Frenz, 1978). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELX76* (Sheldrick, 1976). Molecular graphics: *ORTEP* (Johnson, 1965).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: FG1058). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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