[N,N-Bis(diphenylphosphanyl)-(R)-1,2,3,4-tetrahydro-1-naphthylamine-k²P,P']dichloroplatinum(II)

Citation for published version (APA):

Tooke, D. M., Zijp, E. J., Vlugt, van der, J. I., Vogt, D., & Spek, A. L. (2007). [N,N-Bis(diphenylphosphanyl)-(R)-1,2,3,4-tetrahydro-1-naphthylamine-k²P,P']dichloroplatinum(II). *Acta Crystallographica, Section E: Structure Reports*, 63(1), m86-m88. <https://doi.org/10.1107/S1600536806052044>

DOI:

[10.1107/S1600536806052044](https://doi.org/10.1107/S1600536806052044)

Document status and date:

Published: 01/01/2007

Document Version:

Publisher's PDF, also known as Version of Record (includes final page, issue and volume numbers)

Please check the document version of this publication:

- A submitted manuscript is the version of the article upon submission and before peer-review. There can be important differences between the submitted version and the official published version of record. People interested in the research are advised to contact the author for the final version of the publication, or visit the DOI to the publisher's website.
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[*N,N*-Bis(diphenylphosphanyl)-(*R*)-1,2,3,4-tetrahydro-1-naphthylamine- κ^2P,P']-dichloroplatinum(II)

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Key indicators

Single-crystal X-ray study

T = 150 K

Mean $\sigma(\text{C}-\text{C})$ = 0.009 Å

R factor = 0.034

wR factor = 0.054

Data-to-parameter ratio = 19.7

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

The title compound, $[\text{PtCl}_2(\text{C}_{34}\text{H}_{31}\text{NP}_2)]$, crystallizes with four crystallographically independent molecules. All four molecules have the same *R* chirality, with $\text{P}-\text{Pt}-\text{P}$ bite angles ranging from 72.14 (5) to 72.38 (5)°.

Comment

The structure determination of the title compound, (I), was carried out in order to determine and study its coordination geometry and $\text{P}-\text{Pt}-\text{P}$ bite angles. The asymmetric unit was found to contain four crystallographically independent molecules with the expected *R* chirality but with slightly differing conformations of the ring systems (Fig. 1). The metal is in a slightly distorted square-planar environment (Table 1). The chelating ligand forms a four-membered ring with Pt. The geometry of (I) is in close agreement with that of the *sec*-butylamine analogue (Calabrò *et al.*, 2004).

The packing diagram (Fig. 2) illustrates the pseudo-translation symmetry in the *c*-axis direction. About 83% of the atoms in the unit cell are, within 0.4 Å, related by this pseudo-translation. $\text{C}-\text{H} \cdots \text{Cl}$ short interactions are listed in Table 2.

Experimental

N,N-Bis(diphenylphosphanyl)-(*R*)-1,2,3,4-tetrahydro-1-naphthylamine (72 mg, 140 µmol), prepared following the procedure described by Kuhlmann *et al.* (2006) was reacted with $\text{Pt}(\text{cod})\text{Cl}_2$ (52 mg, 139 µmol) in dichloromethane (5 ml). After 1 h of stirring at room temperature, the solvent was removed *in vacuo*. The white solid was washed twice with additional dichloromethane to remove traces of remaining cyclooctadiene. Colorless crystals were obtained by diffusion of Et_2O into a CHCl_3 solution. ^{31}P NMR (CDCl_3): δ 19.9 (*s*, $^1J_{\text{Pt}-\text{P}}$ = 3281 Hz).

Received 30 November 2006

Accepted 1 December 2006

Figure 1

The structures of the four crystallographically independent molecules of the title compound with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 30% probability level.

Figure 2

Packing diagram, viewed down the monoclinic *b* axis. The four independent molecules are colour-coded as black, red, green and blue. H atoms have been omitted.

Crystal data

[PtCl₂(C₃₄H₃₁NP₂)]
 $M_r = 781.53$
 Monoclinic, $P2_1$
 $a = 19.107(1) \text{ \AA}$
 $b = 15.9176(7) \text{ \AA}$
 $c = 20.8122(11) \text{ \AA}$
 $\beta = 101.266(5)^\circ$
 $V = 6207.8(5) \text{ \AA}^3$

$Z = 8$
 $D_x = 1.672 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 4.82 \text{ mm}^{-1}$
 $T = 150 \text{ K}$
 Block, colourless
 $0.33 \times 0.13 \times 0.06 \text{ mm}$

Data collection

Nonius KappaCCD diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 2002)
 $T_{\min} = 0.44$, $T_{\max} = 0.75$

151899 measured reflections
 28340 independent reflections
 21447 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.065$
 $\theta_{\max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.034$
 $wR(F^2) = 0.054$
 $S = 1.01$
 28340 reflections
 1441 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0146P)^2 + 3.8989P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 1.42 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.87 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 13646 Friedel pairs
 Flack parameter: $-0.021(3)$

Table 1

Selected geometric parameters (Å, °).

Pt1—Cl1	2.3580 (16)	Pt3—Cl5	2.3673 (16)
Pt1—Cl2	2.3613 (15)	Pt3—Cl6	2.3577 (13)
Pt1—P1	2.2061 (15)	Pt3—P6	2.2128 (14)
Pt1—P2	2.2087 (14)	Pt3—P5	2.2040 (13)
Pt2—Cl3	2.3644 (14)	Pt4—P8	2.2158 (14)
Pt2—Cl4	2.3521 (13)	Pt4—Cl8	2.3575 (13)
Pt2—P3	2.2171 (13)	Pt4—Cl7	2.3617 (16)
Pt2—P4	2.1964 (14)	Pt4—P7	2.2142 (13)
Cl1—Pt1—Cl2	92.22 (5)	P5—Pt3—P6	72.38 (5)
P1—Pt1—P2	72.33 (5)	Cl5—Pt3—Cl6	91.69 (5)
P3—Pt2—P4	72.37 (5)	P7—Pt4—P8	72.14 (5)
Cl3—Pt2—Cl4	91.34 (5)	Cl7—Pt4—Cl8	91.21 (5)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C15—H15...Cl4 ⁱ	0.95	2.72	3.635 (6)	162
C24—H24...Cl2	0.95	2.78	3.626 (6)	149
C38—H38B...Cl6 ⁱⁱ	0.99	2.78	3.624 (5)	144
C54—H54...Cl6 ⁱⁱⁱ	0.95	2.83	3.573 (6)	136
C59—H59...Cl6 ^{iv}	0.95	2.78	3.518 (6)	136
C92—H92...Cl1	0.95	2.77	3.658 (6)	155
Cl116—H116...Cl2	0.95	2.80	3.689 (7)	156
Cl130—H130...Cl3 ⁱⁱⁱ	0.95	2.82	3.706 (6)	155

Symmetry codes: (i) $x, y, z - 1$; (ii) $x - 1, y, z$; (iii) $-x + 1, y + \frac{1}{2}, -z + 1$; (iv) $-x + 1, y - \frac{1}{2}, -z + 1$.

All H atoms were placed at geometrically idealized positions (C—H = 0.95 Å for Csp^2 , C—H = 0.99 Å for methylene and C—H =

1.00 Å for tertiary) and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$. The absolute configuration is as expected from that of the starting material; the data set contains a 99.8% coverage of Friedel pairs

Data collection: *COLLECT* (Hooft, 1998); cell refinement: *DIRAX* (Duisenberg, 1992); data reduction: *EVALCCD* (Duisenberg *et al.*, 2003); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

This work was supported in part (DMT and ALS) by the Council for the Chemical Sciences of the Netherlands Organization for Scientific Research (CW-NWO), Avantium Technologies (EZ) and the National Research School Combination for Catalysis (NRSCC) (JlvdV). OMG AG is acknowledged for the generous loan of platinum complexes.

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