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## Key indicators

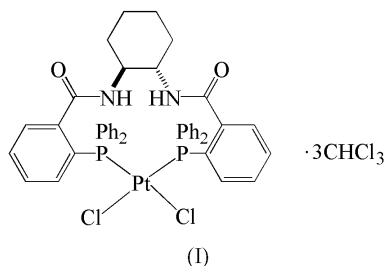
Single-crystal X-ray study  
 $T = 153$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å  
 $R$  factor = 0.036  
 $wR$  factor = 0.092  
Data-to-parameter ratio = 15.5For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.*cis*-{*trans*-1,2-Bis[2-(diphenylphosphino)-  
benzamido]cyclohexane- $\kappa^2P,P'$ }dichloro-  
platinum(II) chloroform trisolvate

The two P atoms in the square-planar title complex,  $[\text{PtCl}_2(\text{C}_{44}\text{H}_{40}\text{N}_2\text{O}_2\text{P}_2)] \cdot 3\text{CHCl}_3$ , adopt a *cis* configuration, forming a 13-membered metallacycle. The H atoms of the amide functions are oriented towards the Cl atoms, whereas the carbonyl groups interact with the solvent chloroform molecules by means of hydrogen bonds.

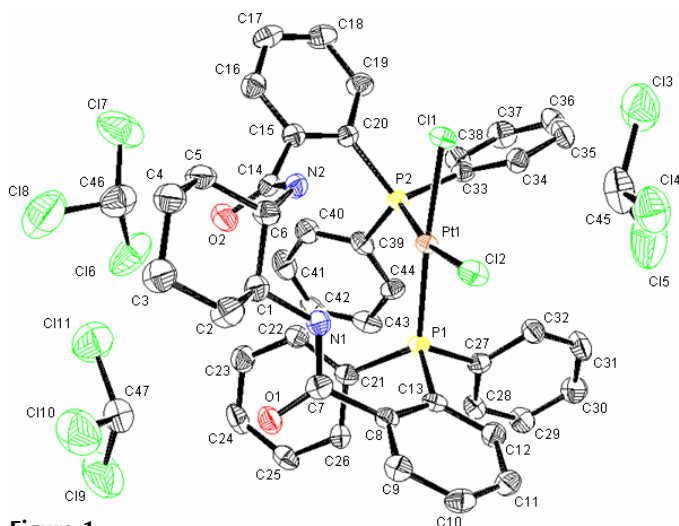
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## Comment

Square-planar complexes of palladium(II) and, to a minor extent, platinum(II) have long been recognized to play a central role in catalytic reactions (Parshall & Ittel, 1992). Recently, we reported the synthesis of a new diphosphine ligand, 1,2-bis[2-(diphenylphosphino)benzamido]benzene (dppbH), a close relative of *trans*-1,2-bis[2-(diphenylphosphino)benzamido]cyclohexane, a ligand developed by Trost in the early 1990's (Trost & Van Vranken, 1992). We have shown that the dppbH ligand reacts with  $[\text{MCl}_2(\text{cod})]$  ( $M = \text{Pd}, \text{Pt}$ ; cod = cyclooctadiene) to form square-planar complexes (Burger *et al.*, 2003). In the case of palladium, where the dppbH ligand coordinates through the two P atoms,  $[\text{PdCl}_2(\text{dppbH})]$  was obtained, while in the case of platinum, after deprotonation of the two amino functions (dppb), the ligand coordinates through the two P atoms and through the two N atoms to form  $[\text{Pt}(\text{dppb})]$ . Therefore, we were interested in the coordination behaviour of the Trost ligand compared with that of dppbH. We present here the single-crystal structure analysis of the platinum complex, (I), obtained with *trans*-1,2-bis[2-(diphenylphosphino)benzamido]cyclohexane.



Reaction of  $[\text{PtCl}_2(\text{cod})]$  with racemic *trans*-1,2-bis[2-(diphenylphosphino)benzamido]cyclohexane (*L*) in chloroform affords the corresponding racemic complex  $[\text{PtCl}_2(\text{L})]$ . The Pt atom is in a square-planar geometry, surrounded by two Cl atoms and two P atoms (Fig. 1). The chelating diphosphine ligand adopts a *cis* configuration. The formation of a 13-membered chelate ring imposes distortion around the Pt atom. The P—Pt—P angle  $[101.15(4)^\circ]$  is larger than the expected value of  $90^\circ$ . The atoms Pt1, P1, P2, Cl1 and Cl2 are essentially coplanar, with an average deviation of 0.0435 Å.



**Figure 1**  
The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

In the crystal structure, intramolecular hydrogen bonds are observed between the NH groups and the Cl atoms (see Table 2). Intermolecular interactions also take place. It is noteworthy that the three chloroform molecules interact with the complex molecule. Two of the chloroform solvent molecules form hydrogen bonds with the carbonyl groups, whereas the third interacts with a Cl atom (Fig. 2).

## Experimental

In a test tube, *trans*-1,2-bis[2-(diphenylphosphino)benzamido]cyclohexane (2 mg) was added to a chloroform solution (2 ml) of [PtCl<sub>2</sub>(cod)] (1 mg). The mixture was stirred overnight, then left at room temperature for several days, the test tube being slightly open, until colourless needles of the product were observed.

### Crystal data

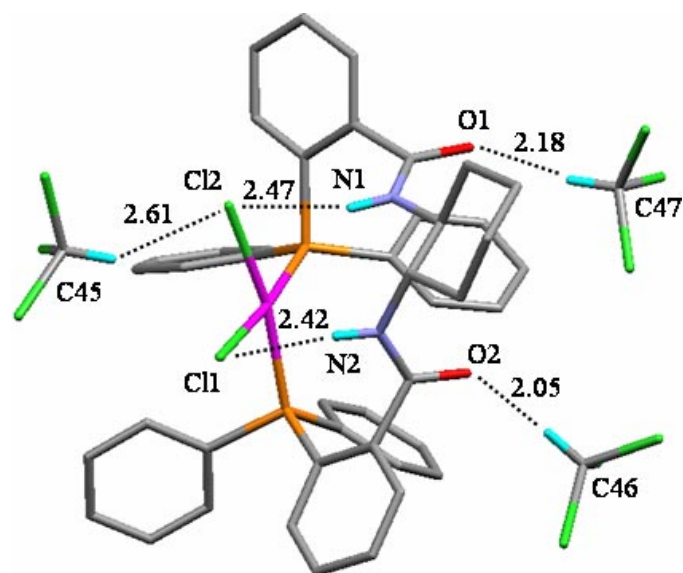
[Pt(C <sub>44</sub> H <sub>40</sub> Cl <sub>2</sub> N <sub>2</sub> O <sub>2</sub> P <sub>2</sub> )]·3CHCl <sub>3</sub>	$D_x = 1.670 \text{ Mg m}^{-3}$
$M_r = 1314.81$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 58409 reflections
$a = 19.3200 (9) \text{ \AA}$	$\theta = 1.6\text{--}24.9^\circ$
$b = 11.1284 (4) \text{ \AA}$	$\mu = 3.35 \text{ mm}^{-1}$
$c = 25.6676 (12) \text{ \AA}$	$T = 153 (2) \text{ K}$
$\beta = 108.638 (4)^\circ$	Block, colourless
$V = 5229.2 (4) \text{ \AA}^3$	$0.50 \times 0.27 \times 0.11 \text{ mm}$
$Z = 4$	

### Data collection

Stoe IPDS-2 diffractometer	7600 reflections with $I > 2\sigma(I)$
$\theta$ - $2\theta$ scans	$R_{\text{int}} = 0.134$
Absorption correction: multi-scan (Blessing, 1995)	$\theta_{\text{max}} = 24.9^\circ$
$T_{\text{min}} = 0.324$ , $T_{\text{max}} = 0.692$	$h = -22 \rightarrow 22$
45 476 measured reflections	$k = -13 \rightarrow 13$
9068 independent reflections	$l = -30 \rightarrow 30$

### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0454P)^2 + 9.7597P]$
$R[F^2 > 2\sigma(F^2)] = 0.036$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.092$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.06$	$\Delta\rho_{\text{max}} = 1.90 \text{ e \AA}^{-3}$
9068 reflections	$\Delta\rho_{\text{min}} = -1.19 \text{ e \AA}^{-3}$
586 parameters	
H-atom parameters constrained	



**Figure 2**  
The intra- and intermolecular interactions (dashed lines) observed in (I) (MERCURY; Bruno *et al.*, 2002). H atoms have been omitted.

**Table 1**

Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ ).

P1—Pt1	2.2631 (12)	Cl1—Pt1	2.3540 (12)
P2—Pt1	2.2535 (11)	Cl2—Pt1	2.3509 (11)
P2—Pt1—P1	101.15 (4)	P2—Pt1—Cl1	84.11 (4)
P1—Pt1—Cl2	89.35 (4)	Cl2—Pt1—Cl1	85.17 (4)

**Table 2**

Hydrogen-bonding geometry ( $\text{\AA}$ ,  $^\circ$ ).

$D\text{—}H\cdots A$	$D\text{—}H$	$H\cdots A$	$D\cdots A$	$D\text{—}H\cdots A$
C1—H1 $\cdots$ O1	0.98	2.66	2.814 (8)	89
C1—H1 $\cdots$ O2	0.98	2.65	3.187 (8)	115
C2—H2A $\cdots$ O1	0.97	2.60	3.163 (9)	117
C22—H22 $\cdots$ O2	0.93	2.47	3.178 (8)	133
C5—H5B $\cdots$ Cl1 <sup>i</sup>	0.97	2.80	3.669 (7)	150
C9—H9 $\cdots$ Cl1X <sup>ii</sup>	0.93	2.82	3.71 (1)	162
C34—H34 $\cdots$ Cl1	0.93	2.72	3.330 (6)	124
C45—H45 $\cdots$ Cl2	0.98	2.61	3.445 (9)	144
C46—H46 $\cdots$ O2	0.98	2.05	3.015 (10)	170
C47—H47 $\cdots$ O1	0.98	2.18	3.151 (9)	171
N2—H2N $\cdots$ Cl1	0.86	2.42	3.254 (5)	164
N1—H1N $\cdots$ Cl2	0.86	2.46	3.296 (5)	163

Symmetry codes: (i)  $-x, -y, 1 - z$ ; (ii)  $x, 1 + y, z$ . Cl1X is the centroid of the C39—C44 phenyl ring.

H atoms were positioned geometrically ( $N\text{—}H = 0.86$  and  $C\text{—}H = 0.93\text{--}0.98 \text{ \AA}$ ) and refined as riding, with  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C}, \text{N})$ . The two electron density maxima greater than  $1 \text{ e \AA}^{-3}$  were observed around the chloroform molecules, at distances of 1.20 and 1.25  $\text{\AA}$ , respectively. The deepest hole in the final difference map is located less than 1  $\text{\AA}$  from the Pt atom.

Data collection: X-Area (Stoe & Cie, 2000); cell refinement: X-Area; data reduction: X-RED32 (Stoe & Cie, 2000); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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## References

- Blessing, R. H. (1995). *Acta Cryst.* **A51**, 33–38.
- Bruno, I. J., Cole, J. C., Edgington, P. R., Kessler, M., Macrae, C. F., McCabe, P., Pearson, J. & Taylor, R. (2002). *Acta Cryst.* **B58**, 389–397.
- Burger, S., Therrien, B. & Süss-Fink, G. (2003). *Eur. J. Inorg. Chem.* pp. 3099–3103.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Parshall, G. W. & Ittel, S. D. (1992). *Homogeneous Catalysis*, 2nd ed., ch. 6, p. 137. New York: Wiley-Interscience.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Stoe & Cie (2000). *IPDS Software*. Stoe & Cie GmbH, Darmstadt, Germany.
- Trost, B. M. & Van Vranken, D. L. (1992). *Angew. Chem. Int. Ed. Engl.* **31**, 228–230.