# cis-Dichloridobis(dimethoxyphenyl-phosphino)platinum(II)

## Alexandra M. Z. Slawin, Paul G. Waddell and J. Derek Woollins*

Department of Chemistry, University of St Andrews, St Andrews KY16 9ST, Scotland
Correspondence e-mail: jdw3@st-and.ac.uk

The title compound, $[PtCl_2(C_8H_{11}O_2P)_2]$, resides on a crystallographic twofold rotation axis and adopts a *cis*-square-planar geometry. Molecules are linked by C—H···Cl interactions forming one-dimensional chains along the [010] direction.

## Related literature

For related literature on $PtCl_2L_2$ complexes, see: Bao *et al.* (1987); Fun *et al.* (2006); Slawin *et al.* (2007).
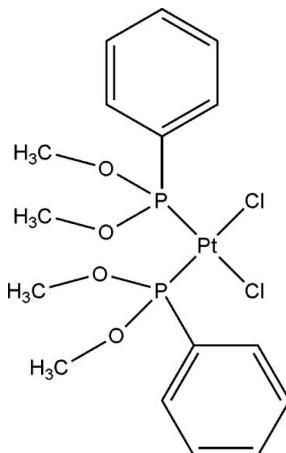


## Experimental

### Crystal data

| | |
|---|---|
| $[PtCl_2(C_8H_{11}O_2P)_2]$ | $V = 2047.5$ (2) Å$^3$ |
| $M_r = 606.27$ | $Z = 4$ |
| Monoclinic, $C2/c$ | Mo $K\alpha$ radiation |
| $a = 10.9734$ (7) Å | $\mu = 7.29$ mm$^{-1}$ |
| $b = 9.2290$ (6) Å | $T = 125$ (2) K |
| $c = 20.7002$ (14) Å | $0.20 \times 0.16 \times 0.07$ mm |
| $\beta = 102.398$ (2)° | |

### Data collection

| | |
|---|---|
| Rigaku SCXmini diffractometer | 5869 measured reflections |
| Absorption correction: multi-scan | 1869 independent reflections |
| (*ABSCOR*; Higashi, 1995) | 1772 reflections with $I > 2\sigma(I)$ |
| $T_{min} = 0.261$, $T_{max} = 0.602$ | $R_{int} = 0.035$ |

### Refinement

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.021$ | 117 parameters |
| $wR(F^2) = 0.043$ | H-atom parameters constrained |
| $S = 1.07$ | $\Delta\rho_{max} = 0.62$ e Å$^{-3}$ |
| 1869 reflections | $\Delta\rho_{min} = -0.69$ e Å$^{-3}$ |

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Pt1—P1 | 2.2190 (9) | Pt1—Cl1 | 2.3629 (9) |
| P1—Pt1—P1$^i$ | 101.16 (5) | P1$^i$—Pt1—Cl1 | 84.90 (3) |
| P1—Pt1—Cl1 | 173.78 (3) | Cl1—Pt1—Cl1$^i$ | 89.08 (4) |

Symmetry code: (i) $-x + 1, y, -z + \frac{1}{2}$.

**Table 2**
Hydrogen-bond geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| C7—H7B···Cl1$^{ii}$ | 0.98 | 2.82 | 3.778 (4) | 166 |

Symmetry code: (ii) $-x + 1, y - 1, -z + \frac{1}{2}$.

Data collection: *SCXmini* (Rigaku/MSC, 2006); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *CrystalStructure* (Rigaku/MSC, 2006); software used to prepare material for publication: *CrystalStructure*.

## References

Bao, Q.-B., Geib, S. J., Rheingold, A. L. & Brill, T. B. (1987). *Inorg. Chem.* **26**, 3453–3458.
Fun, H.-K., Chantrapromma, S., Liu, Y.-C., Chen, Z.-F. & Liang, H. (2006). *Acta Cryst.* E**62**, m1252–m1254.
Higashi, T. (1995). *ABSCOR.* Rigaku Corporation, Tokyo, Japan.
Rigaku (1998). *PROCESS-AUTO.* Rigaku Corporation, Tokyo, Japan.
Rigaku/MSC (2006). *CrystalStructure* (Version 3.8) and *SCXmini* (Version 1.0). Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXL97.* University of Göttingen, Germany.
Slawin, A. M. Z., Waddell, P. G. & Woollins, J. D. (2007). *Acta Cryst.* E**63**, m2018.

**supplementary materials**

### *cis*-Dichloridobis(dimethoxyphenylphosphino)platinum(II)

### A. M. Z. Slawin, P. G. Waddell and J. D. Woollins

#### Comment

The title complex (I) is comparable to similar platinum dichloride complexes containing trimethoxy phosphite; $[PtCl_2(P(OMe)_3)_2]$ (Bao *et al.*, 1987) and triphenyl phosphine $[PtCl_2(PPh_3)_2]$ (Fun *et al.*, 2006). The central platinum atom in (I) can be seen to bear a closer resemblance to the triphenyl phosphine complex. These measurement are also similar when compared to the structure of the complex $[PtCl_2(P(OMe)Ph_2)_2]$ (Slawin *et al.*, 2007) with the exception of the P—Pt—P bond angle which is unusually large.

#### Experimental

1 g (2.67 mmol) of $PtCl_2(COD)$ was dissolved in the minimum volume of dichloromethane in a round-bottomed flask. To this, 0.840 ml (5.34 mmol) of dimethylphenylphosphonite was added. The solution was then stirred for 0.5 h at room temperature. The product was precipitated *via* slow diffusion of hexane and was then filtered off and dried under vacuum, $[PtCl_2(P(OMe)_2Ph)_2]$ (1.86 mmol,*ca* 69%). $^{31}P$-$\{^{1}H\}$ NMR: δ 91.2 p.p.m. *J*{Pt—P} 4819 Hz.

#### Refinement

All H atoms were included in calculated positions (C—H distances are 0.96 Å for methyl H atoms, 0.97 Å for methylene H atoms and 0.98 Å for methine H atoms) and were refined as riding atoms with $U_{iso}(H) = 1.2 \, U_{eq}$(parent atom, methylene and methine H atoms) or $U_{iso}(H) = 1.5 \, U_{eq}$(parent atom, methyl H atoms).

#### Figures
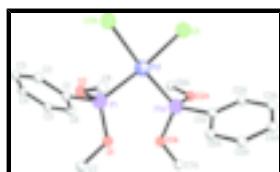


Fig. 1. The structure of (**1**) with displacement ellipsoids drawn at the 50% probability level, H atoms omitted for clarity.

### *cis*-Dichloridobis(dimethoxyphenylphosphino)platinum(II)

*Crystal data*

| | |
|---|---|
| $[PtCl_2(C_8H_{11}O_2P)_2]$ | $F_{000} = 1168$ |
| $M_r = 606.27$ | $D_x = 1.967$ Mg m$^{-3}$ |
| Monoclinic, $C2/c$ | Mo *K*α radiation<br>λ = 0.71073 Å |
| Hall symbol: -C 2yc | Cell parameters from 6759 reflections |

# supplementary materials

| | |
|---|---|
| $a = 10.9734\ (7)$ Å | $\theta = 3.0–27.5°$ |
| $b = 9.2290\ (6)$ Å | $\mu = 7.29$ mm$^{-1}$ |
| $c = 20.7002\ (14)$ Å | $T = 125\ (2)$ K |
| $\beta = 102.398\ (2)°$ | Prism, colourless |
| $V = 2047.5\ (2)$ Å$^3$ | $0.20 \times 0.16 \times 0.07$ mm |
| $Z = 4$ | |

## Data collection

| | |
|---|---|
| Rigaku SCXmini diffractometer | 1772 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.035$ |
| Monochromator: graphite | $\theta_{max} = 25.3°$ |
| $T = 125(2)$ K | $\theta_{min} = 3.2°$ |
| ω scans | $h = -13 \rightarrow 13$ |
| Absorption correction: multi-scan (ABSCOR; Higashi, 1995) | $k = -11 \rightarrow 10$ |
| $T_{min} = 0.261,\ T_{max} = 0.602$ | $l = -24 \rightarrow 24$ |
| 5869 measured reflections | Standard reflections: ? |
| 1869 independent reflections | |

## Refinement

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.021$ | H-atom parameters constrained |
| $wR(F^2) = 0.043$ | $w = 1/[\sigma^2(F_o^2) + (0.0167P)^2 + 0.5089P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.07$ | $(\Delta/\sigma)_{max} = 0.007$ |
| 1869 reflections | $\Delta\rho_{max} = 0.62$ e Å$^{-3}$ |
| 117 parameters | $\Delta\rho_{min} = -0.69$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

## Special details

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit S are based on $F^2$, conventional $R$-factors $R$ are based on F, with F set to zero for negative $F^2$. The threshold expression of $F^2 > 2$sigma$(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

|  | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| Pt1 | 0.5000 | 0.40682 (2) | 0.2500 | 0.01234 (7) |
| Cl1 | 0.59209 (8) | 0.58930 (10) | 0.32480 (4) | 0.0204 (2) |
| P1 | 0.40029 (8) | 0.25413 (10) | 0.17390 (4) | 0.0141 (2) |
| C1 | 0.4614 (3) | 0.2496 (4) | 0.09966 (17) | 0.0156 (8) |
| C2 | 0.4028 (3) | 0.3176 (4) | 0.04180 (18) | 0.0217 (9) |
| H2A | 0.3264 | 0.3677 | 0.0397 | 0.026* |
| C3 | 0.4560 (3) | 0.3125 (5) | −0.01343 (19) | 0.0270 (10) |
| H3A | 0.4166 | 0.3598 | −0.0532 | 0.032* |
| C4 | 0.5671 (4) | 0.2379 (4) | −0.01014 (19) | 0.0300 (10) |
| H4A | 0.6027 | 0.2337 | −0.0481 | 0.036* |
| C5 | 0.6258 (4) | 0.1707 (5) | 0.0466 (2) | 0.0314 (10) |
| H5A | 0.7017 | 0.1200 | 0.0480 | 0.038* |
| C6 | 0.5744 (3) | 0.1764 (4) | 0.10287 (19) | 0.0244 (9) |
| H6A | 0.6158 | 0.1312 | 0.1428 | 0.029* |
| O1 | 0.3959 (2) | 0.0909 (2) | 0.19814 (12) | 0.0195 (6) |
| C7 | 0.3218 (4) | −0.0145 (4) | 0.15439 (19) | 0.0261 (9) |
| H7A | 0.2341 | −0.0059 | 0.1573 | 0.039* |
| H7B | 0.3518 | −0.1124 | 0.1676 | 0.039* |
| H7C | 0.3294 | 0.0037 | 0.1088 | 0.039* |
| O2 | 0.2590 (2) | 0.2966 (3) | 0.14365 (11) | 0.0170 (5) |
| C8 | 0.1756 (3) | 0.3272 (5) | 0.18759 (19) | 0.0266 (10) |
| H8A | 0.1804 | 0.2485 | 0.2198 | 0.040* |
| H8B | 0.0899 | 0.3353 | 0.1617 | 0.040* |
| H8C | 0.2001 | 0.4185 | 0.2109 | 0.040* |

*Atomic displacement parameters (Å$^2$)*

|  | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Pt1 | 0.01451 (11) | 0.01079 (12) | 0.01187 (12) | 0.000 | 0.00317 (8) | 0.000 |
| Cl1 | 0.0245 (5) | 0.0150 (5) | 0.0195 (5) | −0.0003 (4) | −0.0004 (4) | −0.0036 (4) |
| P1 | 0.0172 (5) | 0.0127 (5) | 0.0125 (5) | −0.0014 (4) | 0.0033 (4) | 0.0006 (4) |
| C1 | 0.0178 (17) | 0.012 (2) | 0.017 (2) | −0.0047 (14) | 0.0040 (16) | −0.0026 (15) |
| C2 | 0.022 (2) | 0.023 (2) | 0.019 (2) | 0.0008 (16) | 0.0039 (18) | −0.0017 (17) |
| C3 | 0.031 (2) | 0.035 (3) | 0.012 (2) | −0.0028 (19) | −0.0005 (18) | 0.0011 (18) |
| C4 | 0.037 (2) | 0.036 (3) | 0.020 (2) | −0.003 (2) | 0.012 (2) | −0.0060 (19) |
| C5 | 0.023 (2) | 0.040 (3) | 0.034 (3) | 0.0069 (19) | 0.013 (2) | 0.001 (2) |
| C6 | 0.024 (2) | 0.027 (2) | 0.022 (2) | 0.0081 (17) | 0.0031 (18) | 0.0058 (18) |
| O1 | 0.0272 (13) | 0.0134 (14) | 0.0167 (14) | −0.0058 (11) | 0.0021 (11) | −0.0003 (11) |
| C7 | 0.040 (2) | 0.015 (2) | 0.022 (2) | −0.0068 (18) | 0.004 (2) | −0.0039 (17) |
| O2 | 0.0157 (12) | 0.0190 (15) | 0.0167 (14) | −0.0006 (10) | 0.0048 (11) | −0.0002 (11) |
| C8 | 0.022 (2) | 0.031 (3) | 0.030 (2) | −0.0002 (17) | 0.0135 (19) | 0.0001 (19) |

*Geometric parameters (Å, °)*

| Pt1—P1 | 2.2190 (9) | C4—C5 | 1.362 (5) |
|---|---|---|---|

| | | | |
|---|---|---|---|
| Pt1—P1[i] | 2.2190 (9) | C4—H4A | 0.9500 |
| Pt1—Cl1 | 2.3629 (9) | C5—C6 | 1.400 (5) |
| Pt1—Cl1[i] | 2.3629 (9) | C5—H5A | 0.9500 |
| P1—O1 | 1.592 (2) | C6—H6A | 0.9500 |
| P1—O2 | 1.591 (2) | O1—C7 | 1.453 (4) |
| P1—C1 | 1.804 (3) | C7—H7A | 0.9800 |
| C1—C2 | 1.382 (5) | C7—H7B | 0.9800 |
| C1—C6 | 1.402 (5) | C7—H7C | 0.9800 |
| C2—C3 | 1.392 (5) | O2—C8 | 1.450 (4) |
| C2—H2A | 0.9500 | C8—H8A | 0.9800 |
| C3—C4 | 1.388 (5) | C8—H8B | 0.9800 |
| C3—H3A | 0.9500 | C8—H8C | 0.9800 |
| | | | |
| P1—Pt1—P1[i] | 101.16 (5) | C5—C4—H4A | 119.5 |
| P1—Pt1—Cl1 | 173.78 (3) | C3—C4—H4A | 119.5 |
| P1[i]—Pt1—Cl1 | 84.90 (3) | C4—C5—C6 | 120.0 (4) |
| P1—Pt1—Cl1[i] | 84.90 (3) | C4—C5—H5A | 120.0 |
| P1[i]—Pt1—Cl1[i] | 173.78 (3) | C6—C5—H5A | 120.0 |
| Cl1—Pt1—Cl1[i] | 89.08 (4) | C5—C6—C1 | 119.3 (4) |
| O1—P1—O2 | 105.34 (13) | C5—C6—H6A | 120.3 |
| O1—P1—C1 | 106.75 (15) | C1—C6—H6A | 120.3 |
| O2—P1—C1 | 100.32 (14) | C7—O1—P1 | 119.2 (2) |
| O1—P1—Pt1 | 114.89 (10) | O1—C7—H7A | 109.5 |
| O2—P1—Pt1 | 114.40 (10) | O1—C7—H7B | 109.5 |
| C1—P1—Pt1 | 113.76 (11) | H7A—C7—H7B | 109.5 |
| C2—C1—C6 | 120.1 (3) | O1—C7—H7C | 109.5 |
| C2—C1—P1 | 122.6 (3) | H7A—C7—H7C | 109.5 |
| C6—C1—P1 | 117.3 (3) | H7B—C7—H7C | 109.5 |
| C1—C2—C3 | 119.8 (3) | C8—O2—P1 | 119.6 (2) |
| C1—C2—H2A | 120.1 | O2—C8—H8A | 109.5 |
| C3—C2—H2A | 120.1 | O2—C8—H8B | 109.5 |
| C4—C3—C2 | 119.7 (4) | H8A—C8—H8B | 109.5 |
| C4—C3—H3A | 120.2 | O2—C8—H8C | 109.5 |
| C2—C3—H3A | 120.2 | H8A—C8—H8C | 109.5 |
| C5—C4—C3 | 121.1 (4) | H8B—C8—H8C | 109.5 |
| | | | |
| P1[i]—Pt1—P1—O1 | 14.27 (10) | P1—C1—C2—C3 | 178.7 (3) |
| Cl1[i]—Pt1—P1—O1 | −167.15 (11) | C1—C2—C3—C4 | 0.6 (6) |
| P1[i]—Pt1—P1—O2 | 136.32 (11) | C2—C3—C4—C5 | −0.7 (6) |
| Cl1[i]—Pt1—P1—O2 | −45.10 (10) | C3—C4—C5—C6 | −0.1 (7) |
| P1[i]—Pt1—P1—C1 | −109.19 (13) | C4—C5—C6—C1 | 1.1 (6) |
| Cl1[i]—Pt1—P1—C1 | 69.39 (13) | C2—C1—C6—C5 | −1.3 (6) |
| O1—P1—C1—C2 | 129.0 (3) | P1—C1—C6—C5 | −179.6 (3) |
| O2—P1—C1—C2 | 19.4 (3) | O2—P1—O1—C7 | 46.7 (3) |
| Pt1—P1—C1—C2 | −103.2 (3) | C1—P1—O1—C7 | −59.3 (3) |
| O1—P1—C1—C6 | −52.7 (3) | Pt1—P1—O1—C7 | 173.6 (2) |
| O2—P1—C1—C6 | −162.3 (3) | O1—P1—O2—C8 | 75.5 (3) |
| Pt1—P1—C1—C6 | 75.1 (3) | C1—P1—O2—C8 | −173.8 (3) |

| C6—C1—C2—C3 | 0.4 (5) | Pt1—P1—O2—C8 | −51.6 (3) |

Symmetry codes: (i) −$x$+1, $y$, −$z$+1/2.


*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| C7—H7B···Cl1[ii] | 0.98 | 2.82 | 3.778 (4) | 166 |

Symmetry codes: (ii) −$x$+1, $y$−1, −$z$+1/2.

**Fig. 1**