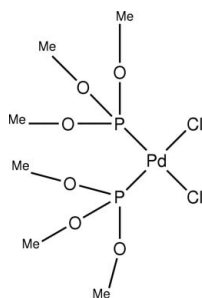


cis-Dichloridobis(trimethoxyphosphine)palladium(II) at 125 K

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Key indicators: single-crystal X-ray study; $T = 125$ K; mean $\sigma(\text{O}-\text{C}) = 0.010$ Å; R factor = 0.054; wR factor = 0.090; data-to-parameter ratio = 16.4.The title compound, $[\text{PdCl}_2(\text{C}_3\text{H}_9\text{O}_3\text{P})_2]$, which is isotypic with its platinum analogue, adopts a slightly distorted *cis* square-planar geometry for the Pd centre.**Related literature**For the platinum analogue, see: Bao *et al.* (1987). For related platinum complexes, see: Slawin *et al.* (2007*a,b*). For *cis*-bis-(triisopropoxyphosphino)platinum dichloride, see: Slawin *et al.* (2009).**Experimental***Crystal data* $[\text{PdCl}_2(\text{C}_3\text{H}_9\text{O}_3\text{P})_2]$ $M_r = 425.46$ Monoclinic, Cc $a = 6.8059$ (19) Å $b = 16.897$ (5) Å $c = 13.374$ (4) Å $\beta = 100.086$ (7)° $V = 1514.2$ (7) Å³ $Z = 4$ Mo $K\alpha$ radiation $\mu = 1.80$ mm⁻¹ $T = 125$ K $0.22 \times 0.16 \times 0.13$ mm*Data collection*

Rigaku SCXmini diffractometer

Absorption correction: multi-scan

(ABSCOR; Higashi, 1995)

 $T_{\min} = 0.662$, $T_{\max} = 0.791$

6333 measured reflections

2639 independent reflections

2338 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.072$ *Refinement* $R[F^2 > 2\sigma(F^2)] = 0.054$ $wR(F^2) = 0.090$ $S = 1.07$

2639 reflections

161 parameters

H-atom parameters constrained

 $\Delta\rho_{\text{max}} = 1.26$ e Å⁻³ $\Delta\rho_{\text{min}} = -0.71$ e Å⁻³

Absolute structure: Flack (1983),

1308 Friedel pairs

Flack parameter: -0.01 (5)**Table 1**

Selected geometric parameters (Å, °).

| | | | |
|-------------|------------|------------|------------|
| Pd1—Cl1 | 2.356 (2) | Pd1—P1 | 2.241 (2) |
| Pd1—Cl2 | 2.358 (2) | Pd1—P2 | 2.233 (2) |
| Cl1—Pd1—Cl2 | 89.71 (8) | Cl2—Pd1—P1 | 90.32 (8) |
| Cl1—Pd1—P1 | 179.04 (9) | Cl2—Pd1—P2 | 177.30 (8) |
| Cl1—Pd1—P2 | 87.59 (8) | P1—Pd1—P2 | 92.38 (8) |

Data collection: *SCXmini* (Rigaku, 2006); cell refinement: *PROCESS-AUTO* (Rigaku, 1998); data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2006); software used to prepare material for publication: *CrystalStructure*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: FI2085).

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cis-Dichloridobis(trimethoxyphosphine)palladium(II) at 125 K

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Comment

[PdCl₂(P(OMe)₃)₂] is isomorphous with its platinum analogue [PtCl₂(P(OMe)₃)₂] (**2**) (Bao *et al.*, 1987) and adopts a *cis* square planar geometry. Whereas the Pt complex was reported to have quite dissimilar bond lengths for Pt—P1/Pt—P2 and Pt—Cl1/PtCl2 bond lengths these pairs of bonds are equivalent in the title compound.

The Pd—Cl bond lengths in (**1**) (Pd(1)—Cl(1) 2.356?(2), Pd(1)—Cl(2) 2.358?(2) Å) are shorter than the Pt—Cl bonds in (**2**) whilst the Pd—P bonds (Pd(1)—P(1) 2.241?(2), Pd(1)—P(2) 2.233?(2) Å) are longer than the Pt—P bonds in (**2**). The P—M—P angle (P(1)—Pd(1)—P(2) 92.38?(8) °) is reduced and the Cl—M—Cl angle is enlarged (Cl(1)—Pd(1)—Cl(2) 89.71?(8) °) on going from Pt to Pd with the palladium compound reported here having angles which are closer to ideal square planar .

Experimental

0.5 g (1.75 mmol) of PdCl₂(COD) was dissolved in dichloromethane (5 mL) in a round-bottomed flask. To this 0.41 mL (3.5 mmol) of trimethylphosphite was added. The solution was 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, [PdCl₂(P(OMe)₃)₂] (1.36 mmol, *ca* 77%). ³¹P-¹H}NMR: δ 97.9 p.p.m..

Refinement

All H atoms were included in calculated positions and refined as riding atoms with U_{iso}(H) = 1.5 U_{eq}. The highest peak in the difference map is 1.097 Å from atom Pd1.

Figures

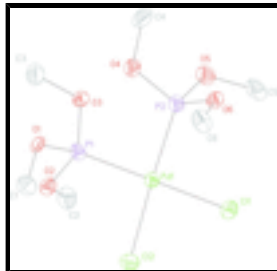


Fig. 1. The structure of the title compound with displacement ellipsoids drawn at the 50% probability level, hydrogen atoms omitted for clarity.

cis-Dichloridobis(trimethoxyphosphine)palladium(II)

Crystal data

| | |
|--|---|
| [PdCl ₂ (C ₃ H ₉ O ₃ P) ₂] | $F_{000} = 848.00$ |
| $M_r = 425.46$ | $D_x = 1.866 \text{ Mg m}^{-3}$ |
| Monoclinic, <i>Cc</i> | Mo <i>K</i> α radiation, $\lambda = 0.71075 \text{ \AA}$ |
| Hall symbol: C -2yc | Cell parameters from 7139 reflections |
| $a = 6.8059 (19) \text{ \AA}$ | $\theta = 3.1\text{--}27.7^\circ$ |
| $b = 16.897 (5) \text{ \AA}$ | $\mu = 1.80 \text{ mm}^{-1}$ |
| $c = 13.374 (4) \text{ \AA}$ | $T = 125 \text{ K}$ |
| $\beta = 100.086 (7)^\circ$ | Chunk, yellow |
| $V = 1514.2 (7) \text{ \AA}^3$ | $0.22 \times 0.16 \times 0.13 \text{ mm}$ |
| $Z = 4$ | |

Data collection

| | |
|---|--|
| Rigaku SCXmini diffractometer | 2639 independent reflections |
| Radiation source: fine-focus sealed tube | 2338 reflections with $F^2 > 2.0\sigma(F^2)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.072$ |
| Detector resolution: 6.85 pixels mm^{-1} | $\theta_{\text{max}} = 25.4^\circ$ |
| $T = 125 \text{ K}$ | $\theta_{\text{min}} = 2.1^\circ$ |
| ω scans | $h = -8 \rightarrow 8$ |
| Absorption correction: multi-scan (ABSCOR; Higashi, 1995) | $k = -20 \rightarrow 20$ |
| $T_{\text{min}} = 0.662$, $T_{\text{max}} = 0.791$ | $l = -16 \rightarrow 16$ |
| 6333 measured reflections | |

Refinement

| | |
|--|--|
| Refinement on F^2 | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.054$ | $w = 1/[\sigma^2(F_o^2) + (0.0247P)^2]$ |
| $wR(F^2) = 0.090$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.07$ | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 2639 reflections | $\Delta\rho_{\text{max}} = 1.26 \text{ e \AA}^{-3}$ |
| 161 parameters | $\Delta\rho_{\text{min}} = -0.71 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |
| Hydrogen site location: inferred from neighbouring sites | Absolute structure: Flack,(1983), 1308 Friedel pairs |
| | Flack parameter: $-0.01 (5)$ |

Special details

Geometry. ENTER SPECIAL DETAILS OF THE MOLECULAR GEOMETRY

Refinement. Refinement was performed using all reflections. The weighted R -factor (wR) and goodness of fit (S) are based on F^2 . R -factor (gt) are based on F . The threshold expression of $F^2 > 2.0 \sigma(F^2)$ is used only for calculating R -factor (gt).

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-------|--------------|---------------|--------------|----------------------------------|
| Pd(1) | 0.50973 (10) | 0.10743 (4) | 0.45788 (7) | 0.01777 (16) |
| Cl(1) | 0.4602 (3) | 0.23766 (14) | 0.39555 (17) | 0.0298 (6) |
| Cl(2) | 0.2407 (3) | 0.06430 (14) | 0.33413 (17) | 0.0277 (6) |
| P(1) | 0.5605 (3) | -0.01677 (14) | 0.51540 (16) | 0.0183 (5) |
| P(2) | 0.7614 (3) | 0.15409 (14) | 0.57243 (17) | 0.0201 (5) |
| O(1) | 0.7314 (8) | -0.0612 (3) | 0.4734 (4) | 0.0215 (14) |
| O(2) | 0.3841 (8) | -0.0766 (3) | 0.4858 (4) | 0.0210 (14) |
| O(3) | 0.6237 (9) | -0.0227 (3) | 0.6348 (4) | 0.0252 (15) |
| O(4) | 0.9294 (8) | 0.0917 (3) | 0.6089 (4) | 0.0259 (15) |
| O(5) | 0.6928 (8) | 0.1816 (3) | 0.6740 (4) | 0.0267 (15) |
| O(6) | 0.8710 (8) | 0.2305 (3) | 0.5430 (4) | 0.0202 (14) |
| C(1) | 0.7285 (16) | -0.0650 (6) | 0.3643 (6) | 0.034 (2) |
| C(2) | 0.1966 (14) | -0.0676 (5) | 0.5230 (7) | 0.030 (2) |
| C(3) | 0.6711 (15) | -0.0993 (5) | 0.6843 (7) | 0.033 (2) |
| C(4) | 1.1106 (14) | 0.1149 (5) | 0.6777 (7) | 0.036 (2) |
| C(5) | 0.5731 (15) | 0.2526 (5) | 0.6788 (6) | 0.032 (2) |
| C(6) | 0.9618 (13) | 0.2290 (5) | 0.4527 (8) | 0.032 (2) |
| H(1) | 0.7662 | -0.1183 | 0.3459 | 0.041* |
| H(2) | 0.8233 | -0.0265 | 0.3455 | 0.041* |
| H(3) | 0.5939 | -0.0527 | 0.3281 | 0.041* |
| H(4) | 0.2211 | -0.0723 | 0.5972 | 0.037* |
| H(5) | 0.1031 | -0.1090 | 0.4935 | 0.037* |
| H(6) | 0.1394 | -0.0155 | 0.5034 | 0.037* |
| H(7) | 0.6000 | -0.1045 | 0.7417 | 0.040* |
| H(8) | 0.8152 | -0.1028 | 0.7089 | 0.040* |
| H(9) | 0.6298 | -0.1419 | 0.6354 | 0.040* |
| H(10) | 1.2047 | 0.1387 | 0.6388 | 0.043* |
| H(11) | 1.1716 | 0.0681 | 0.7138 | 0.043* |
| H(12) | 1.0774 | 0.1534 | 0.7269 | 0.043* |
| H(13) | 0.6614 | 0.2980 | 0.6975 | 0.038* |
| H(14) | 0.4874 | 0.2454 | 0.7298 | 0.038* |
| H(15) | 0.4901 | 0.2621 | 0.6123 | 0.038* |
| H(16) | 1.0930 | 0.2546 | 0.4672 | 0.038* |
| H(17) | 0.8764 | 0.2575 | 0.3977 | 0.038* |
| H(18) | 0.9775 | 0.1740 | 0.4321 | 0.038* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-------|-------------|-------------|-------------|--------------|--------------|-------------|
| Pd(1) | 0.0217 (3) | 0.0152 (3) | 0.0159 (3) | 0.0006 (3) | 0.0018 (2) | -0.0001 (3) |
| Cl(1) | 0.0375 (15) | 0.0183 (13) | 0.0301 (14) | 0.0010 (12) | -0.0040 (11) | 0.0048 (10) |
| Cl(2) | 0.0321 (14) | 0.0253 (14) | 0.0223 (13) | -0.0027 (12) | -0.0050 (11) | 0.0006 (10) |

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|------|-------------|-------------|-------------|--------------|-------------|--------------|
| P(1) | 0.0228 (14) | 0.0171 (13) | 0.0157 (13) | -0.0017 (11) | 0.0056 (10) | -0.0012 (9) |
| P(2) | 0.0234 (15) | 0.0173 (14) | 0.0193 (13) | -0.0010 (11) | 0.0031 (11) | -0.0002 (10) |
| O(1) | 0.031 (3) | 0.019 (3) | 0.014 (3) | 0.013 (3) | 0.003 (2) | 0.002 (2) |
| O(2) | 0.024 (3) | 0.019 (3) | 0.020 (3) | 0.001 (2) | 0.004 (2) | 0.004 (2) |
| O(3) | 0.038 (4) | 0.019 (3) | 0.016 (3) | 0.005 (3) | -0.002 (2) | 0.002 (2) |
| O(4) | 0.021 (3) | 0.017 (3) | 0.037 (3) | 0.000 (2) | -0.003 (3) | -0.001 (2) |
| O(5) | 0.036 (4) | 0.026 (3) | 0.019 (3) | 0.000 (3) | 0.006 (2) | -0.002 (2) |
| O(6) | 0.025 (3) | 0.019 (3) | 0.017 (3) | 0.000 (2) | 0.005 (2) | 0.004 (2) |
| C(1) | 0.050 (7) | 0.038 (6) | 0.014 (5) | 0.015 (5) | 0.006 (5) | -0.010 (4) |
| C(2) | 0.030 (6) | 0.026 (5) | 0.036 (6) | -0.002 (5) | 0.008 (4) | -0.002 (5) |
| C(3) | 0.046 (6) | 0.024 (5) | 0.030 (5) | 0.006 (5) | 0.006 (4) | -0.002 (4) |
| C(4) | 0.026 (5) | 0.029 (6) | 0.047 (6) | 0.006 (4) | -0.007 (4) | 0.007 (5) |
| C(5) | 0.045 (6) | 0.036 (6) | 0.016 (5) | -0.003 (5) | 0.011 (4) | -0.009 (4) |
| C(6) | 0.037 (8) | 0.028 (5) | 0.033 (5) | -0.005 (4) | 0.013 (5) | -0.008 (5) |

Geometric parameters (Å, °)

| | | | |
|----------------------------|------------|------------------------------|-------|
| Pd(1)—Cl(1) | 2.356 (2) | C(1)—H(2) | 0.980 |
| Pd(1)—Cl(2) | 2.358 (2) | C(1)—H(3) | 0.980 |
| Pd(1)—P(1) | 2.241 (2) | C(2)—H(4) | 0.980 |
| Pd(1)—P(2) | 2.233 (2) | C(2)—H(5) | 0.980 |
| P(1)—O(1) | 1.568 (6) | C(2)—H(6) | 0.980 |
| P(1)—O(2) | 1.567 (6) | C(3)—H(7) | 0.980 |
| P(1)—O(3) | 1.583 (5) | C(3)—H(8) | 0.980 |
| P(2)—O(4) | 1.568 (6) | C(3)—H(9) | 0.980 |
| P(2)—O(5) | 1.582 (6) | C(4)—H(10) | 0.980 |
| P(2)—O(6) | 1.575 (6) | C(4)—H(11) | 0.980 |
| O(1)—C(1) | 1.457 (10) | C(4)—H(12) | 0.980 |
| O(2)—C(2) | 1.457 (12) | C(5)—H(13) | 0.980 |
| O(3)—C(3) | 1.464 (10) | C(5)—H(14) | 0.980 |
| O(4)—C(4) | 1.458 (10) | C(5)—H(15) | 0.980 |
| O(5)—C(5) | 1.458 (11) | C(6)—H(16) | 0.980 |
| O(6)—C(6) | 1.451 (12) | C(6)—H(17) | 0.980 |
| C(1)—H(1) | 0.980 | C(6)—H(18) | 0.980 |
| Cl(1)···O(5) ⁱ | 3.473 (5) | H(4)···O(6) ⁱⁱⁱ | 3.594 |
| Cl(1)···C(5) ⁱ | 3.563 (8) | H(4)···C(4) ^{viii} | 3.466 |
| O(1)···C(2) ⁱⁱ | 3.121 (11) | H(4)···C(5) ⁱⁱⁱ | 3.367 |
| O(2)···O(6) ⁱⁱⁱ | 3.352 (7) | H(4)···H(8) ^{viii} | 3.402 |
| O(2)···C(6) ⁱⁱⁱ | 3.367 (10) | H(4)···H(11) ^{viii} | 2.891 |
| O(3)···C(1) ^{iv} | 3.369 (10) | H(4)···H(13) ⁱⁱⁱ | 2.636 |
| O(4)···C(2) ⁱⁱ | 3.550 (11) | H(4)···H(15) ⁱⁱⁱ | 3.232 |
| O(5)···Cl(1) ^v | 3.473 (5) | H(5)···Cl(1) ⁱⁱⁱ | 2.989 |
| O(5)···C(1) ^{iv} | 3.194 (11) | H(5)···O(1) ^{viii} | 2.624 |
| O(6)···O(2) ^{vi} | 3.352 (7) | H(5)···O(6) ⁱⁱⁱ | 3.271 |
| C(1)···O(3) ^{vii} | 3.369 (10) | H(5)···C(1) ^{viii} | 2.915 |
| C(1)···O(5) ^{vii} | 3.194 (11) | H(5)···C(5) ⁱⁱⁱ | 3.440 |

| | | | |
|-------------------------------|------------|------------------------------|-------|
| C(1)···C(2) ⁱⁱ | 3.506 (13) | H(5)···H(1) ^{viii} | 2.756 |
| C(2)···O(1) ^{viii} | 3.121 (11) | H(5)···H(2) ^{viii} | 2.858 |
| C(2)···O(4) ^{viii} | 3.550 (11) | H(5)···H(13) ⁱⁱⁱ | 3.113 |
| C(2)···C(1) ^{viii} | 3.506 (13) | H(5)···H(15) ⁱⁱⁱ | 2.879 |
| C(5)···Cl(1) ^v | 3.563 (8) | H(5)···H(17) ⁱⁱⁱ | 3.321 |
| C(6)···O(2) ^{vi} | 3.367 (10) | H(6)···O(1) ^{viii} | 2.842 |
| Pd(1)···H(7) ^{vii} | 3.060 | H(6)···O(4) ^{viii} | 2.832 |
| Pd(1)···H(10) ^{viii} | 3.493 | H(6)···C(1) ^{viii} | 3.188 |
| Cl(1)···H(1) ^{ix} | 2.793 | H(6)···C(4) ^{viii} | 3.238 |
| Cl(1)···H(5) ^{vi} | 2.989 | H(6)···H(1) ^{viii} | 3.469 |
| Cl(1)···H(7) ^{vii} | 3.300 | H(6)···H(2) ^{viii} | 2.745 |
| Cl(1)···H(12) ⁱ | 3.121 | H(6)···H(10) ^{viii} | 3.158 |
| Cl(1)···H(13) ⁱ | 3.103 | H(6)···H(11) ^{viii} | 3.121 |
| Cl(1)···H(14) ⁱ | 3.585 | H(6)···H(18) ^{viii} | 3.466 |
| Cl(1)···H(16) ^{viii} | 2.843 | H(7)···Pd(1) ^{iv} | 3.060 |
| Cl(1)···H(18) ^{viii} | 3.571 | H(7)···Cl(1) ^{iv} | 3.300 |
| Cl(2)···H(2) ^{viii} | 3.255 | H(7)···Cl(2) ^{iv} | 3.005 |
| Cl(2)···H(4) ^{vii} | 3.151 | H(7)···C(1) ^{iv} | 3.339 |
| Cl(2)···H(7) ^{vii} | 3.005 | H(7)···H(2) ^{iv} | 2.897 |
| Cl(2)···H(8) ^x | 3.149 | H(7)···H(3) ^{iv} | 2.900 |
| Cl(2)···H(11) ^x | 2.747 | H(7)···H(13) ⁱⁱⁱ | 3.370 |
| Cl(2)···H(13) ⁱ | 2.949 | H(7)···H(18) ^{iv} | 3.489 |
| Cl(2)···H(18) ^{viii} | 3.031 | H(8)···Cl(2) ^{xiv} | 3.149 |
| O(1)···H(4) ⁱⁱ | 3.457 | H(8)···C(5) ^{xi} | 3.076 |
| O(1)···H(5) ⁱⁱ | 2.624 | H(8)···H(2) ^{iv} | 2.842 |
| O(1)···H(6) ⁱⁱ | 2.842 | H(8)···H(3) ^{iv} | 3.543 |
| O(1)···H(16) ⁱⁱⁱ | 3.250 | H(8)···H(4) ⁱⁱ | 3.402 |
| O(2)···H(16) ⁱⁱⁱ | 3.216 | H(8)···H(13) ^{xi} | 2.917 |
| O(2)···H(17) ⁱⁱⁱ | 3.038 | H(8)···H(14) ^{xi} | 2.814 |
| O(3)···H(2) ^{iv} | 3.022 | H(8)···H(15) ^{xi} | 2.973 |
| O(3)···H(3) ^{iv} | 2.920 | H(8)···H(18) ^{iv} | 3.229 |
| O(4)···H(1) ^{iv} | 3.567 | H(9)···O(6) ⁱⁱⁱ | 2.917 |
| O(4)···H(2) ^{iv} | 3.543 | H(9)···C(5) ^{xi} | 3.465 |
| O(4)···H(4) ⁱⁱ | 3.429 | H(9)···C(6) ⁱⁱⁱ | 3.325 |
| O(4)···H(6) ⁱⁱ | 2.832 | H(9)···H(13) ⁱⁱⁱ | 3.578 |
| O(5)···H(1) ^{iv} | 2.506 | H(9)···H(14) ^{xi} | 3.173 |
| O(5)···H(2) ^{iv} | 3.494 | H(9)···H(15) ^{xi} | 3.001 |
| O(5)···H(3) ^{iv} | 3.151 | H(9)···H(16) ⁱⁱⁱ | 2.826 |
| O(5)···H(10) ^{viii} | 3.352 | H(10)···Pd(1) ⁱⁱ | 3.493 |
| O(6)···H(4) ^{vi} | 3.594 | H(10)···O(5) ⁱⁱ | 3.352 |
| O(6)···H(5) ^{vi} | 3.271 | H(10)···C(5) ⁱⁱ | 3.132 |

supplementary materials

| | | | |
|------------------------------|-------|-------------------------------|-------|
| O(6)···H(9) ^{vi} | 2.917 | H(10)···H(6) ⁱⁱ | 3.158 |
| C(1)···H(5) ⁱⁱ | 2.915 | H(10)···H(14) ⁱⁱ | 2.759 |
| C(1)···H(6) ⁱⁱ | 3.188 | H(10)···H(15) ⁱⁱ | 2.914 |
| C(1)···H(7) ^{vii} | 3.339 | H(11)···Cl(2) ^{xiv} | 2.747 |
| C(1)···H(12) ^{vii} | 3.575 | H(11)···C(2) ⁱⁱ | 3.457 |
| C(1)···H(16) ⁱⁱⁱ | 3.534 | H(11)···H(2) ^{iv} | 3.268 |
| C(2)···H(1) ^{viii} | 3.535 | H(11)···H(3) ^{xiv} | 3.021 |
| C(2)···H(2) ^{viii} | 3.235 | H(11)···H(4) ⁱⁱ | 2.891 |
| C(2)···H(11) ^{viii} | 3.457 | H(11)···H(6) ⁱⁱ | 3.121 |
| C(2)···H(13) ⁱⁱⁱ | 3.293 | H(12)···Cl(1) ^v | 3.121 |
| C(2)···H(15) ⁱⁱⁱ | 3.502 | H(12)···C(1) ^{iv} | 3.575 |
| C(3)···H(2) ^{iv} | 3.075 | H(12)···H(1) ^{iv} | 2.925 |
| C(3)···H(3) ^{iv} | 3.305 | H(12)···H(2) ^{iv} | 3.327 |
| C(3)···H(14) ^{xi} | 3.381 | H(12)···H(14) ⁱⁱ | 3.188 |
| C(3)···H(15) ^{xi} | 3.443 | H(12)···H(17) ^v | 3.161 |
| C(4)···H(1) ^{iv} | 3.522 | H(13)···Cl(1) ^v | 3.103 |
| C(4)···H(2) ^{iv} | 3.554 | H(13)···Cl(2) ^v | 2.949 |
| C(4)···H(4) ⁱⁱ | 3.466 | H(13)···C(2) ^{vi} | 3.293 |
| C(4)···H(6) ⁱⁱ | 3.238 | H(13)···H(4) ^{vi} | 2.636 |
| C(4)···H(14) ⁱⁱ | 3.361 | H(13)···H(5) ^{vi} | 3.113 |
| C(5)···H(1) ^{iv} | 3.293 | H(13)···H(7) ^{vi} | 3.370 |
| C(5)···H(4) ^{vi} | 3.367 | H(13)···H(8) ^{ix} | 2.917 |
| C(5)···H(5) ^{vi} | 3.440 | H(13)···H(9) ^{vi} | 3.578 |
| C(5)···H(8) ^{ix} | 3.076 | H(14)···Cl(1) ^v | 3.585 |
| C(5)···H(9) ^{ix} | 3.465 | H(14)···C(3) ^{ix} | 3.381 |
| C(5)···H(10) ^{viii} | 3.132 | H(14)···C(4) ^{viii} | 3.361 |
| C(5)···H(17) ^{xii} | 3.430 | H(14)···C(6) ^{xii} | 3.047 |
| C(6)···H(9) ^{vi} | 3.325 | H(14)···H(1) ^{iv} | 3.097 |
| C(6)···H(14) ^{xiii} | 3.047 | H(14)···H(3) ^{iv} | 3.538 |
| H(1)···Cl(1) ^{xi} | 2.793 | H(14)···H(8) ^{ix} | 2.814 |
| H(1)···O(4) ^{vii} | 3.567 | H(14)···H(9) ^{ix} | 3.173 |
| H(1)···O(5) ^{vii} | 2.506 | H(14)···H(10) ^{viii} | 2.759 |
| H(1)···C(2) ⁱⁱ | 3.535 | H(14)···H(12) ^{viii} | 3.188 |
| H(1)···C(4) ^{vii} | 3.522 | H(14)···H(16) ^{xii} | 3.131 |
| H(1)···C(5) ^{vii} | 3.293 | H(14)···H(17) ^{xii} | 2.492 |
| H(1)···H(5) ⁱⁱ | 2.756 | H(14)···H(18) ^{xii} | 3.040 |
| H(1)···H(6) ⁱⁱ | 3.469 | H(15)···C(2) ^{vi} | 3.502 |
| H(1)···H(12) ^{vii} | 2.925 | H(15)···C(3) ^{ix} | 3.443 |
| H(1)···H(14) ^{vii} | 3.097 | H(15)···H(4) ^{vi} | 3.232 |
| H(1)···H(16) ⁱⁱⁱ | 3.050 | H(15)···H(5) ^{vi} | 2.879 |
| H(1)···H(17) ⁱⁱⁱ | 3.545 | H(15)···H(8) ^{ix} | 2.973 |

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| H(2)···Cl(2) ⁱⁱ | 3.255 | H(15)···H(9) ^{ix} | 3.001 |
| H(2)···O(3) ^{vii} | 3.022 | H(15)···H(10) ^{viii} | 2.914 |
| H(2)···O(4) ^{vii} | 3.543 | H(15)···H(16) ^{viii} | 3.041 |
| H(2)···O(5) ^{vii} | 3.494 | H(16)···Cl(1) ⁱⁱ | 2.843 |
| H(2)···C(2) ⁱⁱ | 3.235 | H(16)···O(1) ^{vi} | 3.250 |
| H(2)···C(3) ^{vii} | 3.075 | H(16)···O(2) ^{vi} | 3.216 |
| H(2)···C(4) ^{vii} | 3.554 | H(16)···C(1) ^{vi} | 3.534 |
| H(2)···H(5) ⁱⁱ | 2.858 | H(16)···H(1) ^{vi} | 3.050 |
| H(2)···H(6) ⁱⁱ | 2.745 | H(16)···H(9) ^{vi} | 2.826 |
| H(2)···H(7) ^{vii} | 2.897 | H(16)···H(14) ^{xiii} | 3.131 |
| H(2)···H(8) ^{vii} | 2.842 | H(16)···H(15) ⁱⁱ | 3.041 |
| H(2)···H(11) ^{vii} | 3.268 | H(17)···O(2) ^{vi} | 3.038 |
| H(2)···H(12) ^{vii} | 3.327 | H(17)···C(5) ^{xiii} | 3.430 |
| H(3)···O(3) ^{vii} | 2.920 | H(17)···H(1) ^{vi} | 3.545 |
| H(3)···O(5) ^{vii} | 3.151 | H(17)···H(5) ^{vi} | 3.321 |
| H(3)···C(3) ^{vii} | 3.305 | H(17)···H(12) ⁱ | 3.161 |
| H(3)···H(7) ^{vii} | 2.900 | H(17)···H(14) ^{xiii} | 2.492 |
| H(3)···H(8) ^{vii} | 3.543 | H(18)···Cl(1) ⁱⁱ | 3.571 |
| H(3)···H(11) ^x | 3.021 | H(18)···Cl(2) ⁱⁱ | 3.031 |
| H(3)···H(14) ^{vii} | 3.538 | H(18)···H(6) ⁱⁱ | 3.466 |
| H(4)···Cl(2) ^{iv} | 3.151 | H(18)···H(7) ^{vii} | 3.489 |
| H(4)···O(1) ^{viii} | 3.457 | H(18)···H(8) ^{vii} | 3.229 |
| H(4)···O(4) ^{viii} | 3.429 | H(18)···H(14) ^{xiii} | 3.040 |
| Cl(1)—Pd(1)—Cl(2) | 89.71 (8) | O(2)—C(2)—H(4) | 109.5 |
| Cl(1)—Pd(1)—P(1) | 179.04 (9) | O(2)—C(2)—H(5) | 109.5 |
| Cl(1)—Pd(1)—P(2) | 87.59 (8) | O(2)—C(2)—H(6) | 109.5 |
| Cl(2)—Pd(1)—P(1) | 90.32 (8) | H(4)—C(2)—H(5) | 109.5 |
| Cl(2)—Pd(1)—P(2) | 177.30 (8) | H(4)—C(2)—H(6) | 109.5 |
| P(1)—Pd(1)—P(2) | 92.38 (8) | H(5)—C(2)—H(6) | 109.5 |
| Pd(1)—P(1)—O(1) | 113.9 (2) | O(3)—C(3)—H(7) | 109.5 |
| Pd(1)—P(1)—O(2) | 116.9 (2) | O(3)—C(3)—H(8) | 109.5 |
| Pd(1)—P(1)—O(3) | 113.8 (2) | O(3)—C(3)—H(9) | 109.5 |
| O(1)—P(1)—O(2) | 100.6 (3) | H(7)—C(3)—H(8) | 109.5 |
| O(1)—P(1)—O(3) | 104.1 (3) | H(7)—C(3)—H(9) | 109.5 |
| O(2)—P(1)—O(3) | 106.0 (3) | H(8)—C(3)—H(9) | 109.5 |
| Pd(1)—P(2)—O(4) | 113.9 (2) | O(4)—C(4)—H(10) | 109.5 |
| Pd(1)—P(2)—O(5) | 112.8 (2) | O(4)—C(4)—H(11) | 109.5 |
| Pd(1)—P(2)—O(6) | 117.3 (2) | O(4)—C(4)—H(12) | 109.5 |
| O(4)—P(2)—O(5) | 103.9 (3) | H(10)—C(4)—H(11) | 109.5 |
| O(4)—P(2)—O(6) | 106.1 (3) | H(10)—C(4)—H(12) | 109.5 |
| O(5)—P(2)—O(6) | 101.4 (3) | H(11)—C(4)—H(12) | 109.5 |
| P(1)—O(1)—C(1) | 119.9 (5) | O(5)—C(5)—H(13) | 109.5 |
| P(1)—O(2)—C(2) | 121.6 (5) | O(5)—C(5)—H(14) | 109.5 |
| P(1)—O(3)—C(3) | 120.7 (5) | O(5)—C(5)—H(15) | 109.5 |

supplementary materials

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| P(2)—O(4)—C(4) | 120.4 (5) | H(13)—C(5)—H(14) | 109.5 |
| P(2)—O(5)—C(5) | 122.1 (5) | H(13)—C(5)—H(15) | 109.5 |
| P(2)—O(6)—C(6) | 118.9 (5) | H(14)—C(5)—H(15) | 109.5 |
| O(1)—C(1)—H(1) | 109.5 | O(6)—C(6)—H(16) | 109.5 |
| O(1)—C(1)—H(2) | 109.5 | O(6)—C(6)—H(17) | 109.5 |
| O(1)—C(1)—H(3) | 109.5 | O(6)—C(6)—H(18) | 109.5 |
| H(1)—C(1)—H(2) | 109.5 | H(16)—C(6)—H(17) | 109.5 |
| H(1)—C(1)—H(3) | 109.5 | H(16)—C(6)—H(18) | 109.5 |
| H(2)—C(1)—H(3) | 109.5 | H(17)—C(6)—H(18) | 109.5 |
| Cl(1)—Pd(1)—P(2)—O(4) | -151.9 (2) | O(1)—P(1)—O(2)—C(2) | 170.3 (5) |
| Cl(1)—Pd(1)—P(2)—O(5) | 90.0 (2) | O(2)—P(1)—O(1)—C(1) | 73.0 (6) |
| Cl(1)—Pd(1)—P(2)—O(6) | -27.2 (2) | O(1)—P(1)—O(3)—C(3) | -52.8 (7) |
| Cl(2)—Pd(1)—P(1)—O(1) | 98.3 (2) | O(3)—P(1)—O(1)—C(1) | -177.4 (6) |
| Cl(2)—Pd(1)—P(1)—O(2) | -18.5 (2) | O(2)—P(1)—O(3)—C(3) | 52.8 (7) |
| Cl(2)—Pd(1)—P(1)—O(3) | -142.6 (2) | O(3)—P(1)—O(2)—C(2) | 62.1 (6) |
| P(1)—Pd(1)—P(2)—O(4) | 27.1 (2) | Pd(1)—P(2)—O(4)—C(4) | 175.4 (5) |
| P(1)—Pd(1)—P(2)—O(5) | -91.0 (2) | Pd(1)—P(2)—O(5)—C(5) | -71.2 (6) |
| P(1)—Pd(1)—P(2)—O(6) | 151.9 (2) | Pd(1)—P(2)—O(6)—C(6) | -58.1 (5) |
| P(2)—Pd(1)—P(1)—O(1) | -81.8 (2) | O(4)—P(2)—O(5)—C(5) | 165.0 (6) |
| P(2)—Pd(1)—P(1)—O(2) | 161.4 (2) | O(5)—P(2)—O(4)—C(4) | -61.6 (7) |
| P(2)—Pd(1)—P(1)—O(3) | 37.3 (2) | O(4)—P(2)—O(6)—C(6) | 70.5 (5) |
| Pd(1)—P(1)—O(1)—C(1) | -52.9 (6) | O(6)—P(2)—O(4)—C(4) | 44.8 (7) |
| Pd(1)—P(1)—O(2)—C(2) | -65.9 (6) | O(5)—P(2)—O(6)—C(6) | 178.7 (5) |
| Pd(1)—P(1)—O(3)—C(3) | -177.3 (5) | O(6)—P(2)—O(5)—C(5) | 55.1 (6) |

Symmetry codes: (i) $x-1/2, -y+1/2, z-1/2$; (ii) $x+1, y, z$; (iii) $x-1/2, y-1/2, z$; (iv) $x, -y, z+1/2$; (v) $x+1/2, -y+1/2, z+1/2$; (vi) $x+1/2, y+1/2, z$; (vii) $x, -y, z-1/2$; (viii) $x-1, y, z$; (ix) $x-1/2, y+1/2, z$; (x) $x-1, -y, z-1/2$; (xi) $x+1/2, y-1/2, z$; (xii) $x-1/2, -y+1/2, z+1/2$; (xiii) $x+1/2, -y+1/2, z-1/2$; (xiv) $x+1, -y, z+1/2$.

Fig. 1

