

metal-organic compounds



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***trans*-Dichloridobis[tris(4-methoxyphenyl)phosphane]palladium(II) toluene solvate**

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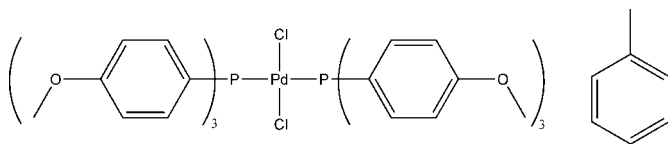
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in solvent or counterion; R factor = 0.029; wR factor = 0.074; data-to-parameter ratio = 20.4.

In the title compound, *trans*-[PdCl₂[P(4-MeOC₆H₄)₃]₂]-C₇H₈, the Pd(II) atom lies on a center of symmetry, resulting in a distorted *trans*-square planar geometry. The Pd—P and Pd—Cl bond lengths are 2.3409 (4) and 2.2981 (4) Å, respectively. An intramolecular C—H···Cl hydrogen bond occurs. In the crystal, weak C—H···O interactions are observed between the aromatic rings of adjacent molecules. The toluene solvate molecule is equally disordered over two sets of sites.

Related literature

For a review on related compounds, see: Spessard & Miessler (1996). For related compounds, see: Meijboom & Omondi (2010). For the synthesis of the starting materials, see: Drew & Doyle (1990).



Experimental

Crystal data

[PdCl₂(C₂₁H₂₁O₃P)₂]-C₇H₈
 $M_r = 974.13$
Triclinic, $P\bar{1}$
 $a = 7.8545$ (4) Å

$b = 12.1231$ (7) Å
 $c = 12.4024$ (8) Å
 $\alpha = 85.666$ (2)°
 $\beta = 78.762$ (2)°

$\gamma = 75.919$ (2)°
 $V = 1123.03$ (11) Å³
 $Z = 1$
Mo $K\alpha$ radiation

$\mu = 0.65$ mm⁻¹
 $T = 100$ K
 $0.27 \times 0.20 \times 0.08$ mm

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer
Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.844$, $T_{\max} = 0.950$

19639 measured reflections
5573 independent reflections
5169 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.074$
 $S = 1.06$
5573 reflections
273 parameters

4 restraints
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 1.28$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.67$ e Å⁻³

Table 1

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> |
|----------------------------|-------------|---------------|-----------------------|-------------------------|
| C01—H01B···O2 ⁱ | 0.98 | 2.36 | 3.327 (7) | 170 |
| C3—H3A···O2 ⁱⁱ | 0.98 | 2.57 | 3.255 (3) | 127 |
| C36—H36···Cl | 0.95 | 2.79 | 3.5402 (19) | 136 |

Symmetry codes: (i) $x + 1, y - 1, z$; (ii) $-x, -y + 1, -z + 2$.

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT-Plus and XPREP (Bruker, 2004); program(s) used to solve structure: SIR97 (Altomare *et al.*, 1999); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 2005); software used to prepare material for publication: WinGX (Farrugia, 1999).

Financial assistance from the South African National Research Foundation (SA NRF), the Research Fund of the University of Johannesburg and SASOL is gratefully acknowledged. The University of the Free State (Professor A. Roodt) is thanked for the use of its diffractometer.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2723).

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supplementary materials

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***trans*-Dichloridobis[tris(4-methoxyphenyl)phosphane]palladium(II) toluene solvate**

A. Muller and R. Meijboom

Comment

Transition metal complexes containing phosphine, arsine and stibine ligands are widely being investigated in various fields of organometallic chemistry (Spessard & Miessler, 1996). As part of a systematic investigation involving complexes with the general formula *trans*-[MX₂(L)₂] (*M* = Pt or Pd; *X* = halogen, Me, Ph; *L* = Group 15 donor ligand), crystals of the title compound, were obtained.

[PdCl₂(L)₂] (*L* = tertiary phosphine, arsine or stibine) complexes can conveniently be prepared by the substitution of 1,5-cyclooctadiene (COD) from [PdCl₂(COD)]. The title compound, *trans*-[PdCl₂{P(4-MeOC₆H₄)₃}₂], crystallizes in the triclinic spacegroup *P* $\bar{1}$, with the Pd atom on a center of symmetry and each pair of equivalent ligands in a mutually *trans* orientation. The geometry is, therefore, slightly distorted square planar and the Pd atom is not elevated out of the coordinating atom plane. All angles in the coordination polyhedron are close to the ideal value of 90°, with P—Pd—Cl = 88.422 (15) and P—Pd—Cl^{*i*} = 91.578 (15)°. As required by the crystallographic symmetry, the P—Pd—P^{*i*} and Cl—Pd—Cl^{*i*} angles are 180°. Some weak intermolecular interactions were observed and are reported in Table 1.

The title compound compares well with other closely related Pd^{II} complexes from the literature containing two chloro and two tertiary phosphine ligands in a *trans* geometry. The title compound, having a Pd—Cl bond length of 2.2981 (4) Å and a Pd—P bond length of 2.3409 (4) Å, fits well into the typical range for complexes of this kind. Notably the title compound crystallized as a solvated complex; these type of Pd^{II} complexes have a tendency to crystallize as solvates (Meijboom & Omondi, 2010). The solvate molecule, toluene, is found 50:50 disordered molecule.

Experimental

Dichloro(1,5-cyclooctadiene)palladium(II), [PdCl₂(COD)], was prepared according to the literature procedure of Drew & Doyle (1990). A solution of tris(4-methoxyphenyl)phosphine (0.2 mmol) in dichloromethane (2.0 cm³) was added to a solution of [PdCl₂(COD)] (0.1 mmol) in dichloromethane (3.0 cm³). Slow evaporation of the solvent gave the parent palladium compound. Recrystallization from toluene/hexane afforded crystals of the title compound.

Refinement

The aromatic and methyl H atoms were placed in geometrically idealized positions (C—H = 0.95–0.98) and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl H atoms respectively. Methyl torsion angles were refined from electron density

Figures

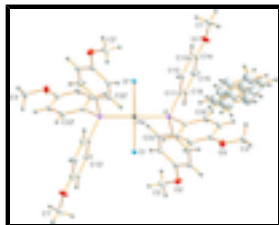


Fig. 1. The structure (I), showing 50% probability displacement ellipsoids. For the C atoms, the first digit indicates ring number and the second digit indicates the position of the atom in the ring. Accented lettering indicate atoms generated by symmetry ($1 - x, 1 - y, 1 - z$).

trans-Dichloridobis[tris(4-methoxyphenyl)phosphane]palladium(II) toluene monosolvate

Crystal data

[PdCl₂(C₂₁H₂₁O₃P)₂]·C₇H₈

$M_r = 974.13$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.8545$ (4) Å

$b = 12.1231$ (7) Å

$c = 12.4024$ (8) Å

$\alpha = 85.666$ (2)°

$\beta = 78.762$ (2)°

$\gamma = 75.919$ (2)°

$V = 1123.03$ (11) Å³

$Z = 1$

$F(000) = 502$

$D_x = 1.44$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 5142 reflections

$\theta = 2.4$ – 28.3 °

$\mu = 0.65$ mm⁻¹

$T = 100$ K

Plate, yellow

$0.27 \times 0.2 \times 0.08$ mm

Data collection

Bruker X8 APEXII 4K Kappa CCD diffractometer

Radiation source: fine-focus sealed tube graphite

Detector resolution: 8.4 pixels mm⁻¹

φ and ω scans

Absorption correction: multi-scan (SADABS; Bruker, 2004)

$T_{\min} = 0.844$, $T_{\max} = 0.950$

19639 measured reflections

5573 independent reflections

5169 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.037$

$\theta_{\max} = 28.3$ °, $\theta_{\min} = 2.4$ °

$h = -10 \rightarrow 10$

$k = -16 \rightarrow 16$

$l = -16 \rightarrow 16$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.074$

$S = 1.06$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0261P)^2 + 1.197P]$

5573 reflections
273 parameters
4 restraints

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 1.27 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.67 \text{ e } \text{\AA}^{-3}$$

Special details

Experimental. The intensity data was collected on a Bruker X8 Apex II 4 K Kappa CCD diffractometer using an exposure time of 6 s/frame. A total of 1637 frames were collected with a frame width of 0.5° covering up to $\theta = 28.31^\circ$ with 99.8% completeness accomplished.

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 $F^2 > \sigma(F^2)$ 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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|-----|-------------|--------------|--------------|----------------------------------|-----------|
| Pd | 0.5 | 0.5 | 0.5 | 0.01073 (6) | |
| P | 0.32288 (6) | 0.43533 (4) | 0.65434 (4) | 0.01130 (9) | |
| Cl | 0.44125 (6) | 0.67186 (4) | 0.58287 (4) | 0.01752 (10) | |
| C11 | 0.2359 (2) | 0.31163 (15) | 0.64258 (14) | 0.0129 (3) | |
| C12 | 0.3519 (2) | 0.20484 (15) | 0.62301 (15) | 0.0150 (3) | |
| H12 | 0.4766 | 0.1972 | 0.6168 | 0.018* | |
| C13 | 0.2882 (2) | 0.10942 (15) | 0.61244 (15) | 0.0162 (3) | |
| H13 | 0.3689 | 0.0375 | 0.599 | 0.019* | |
| C14 | 0.1045 (3) | 0.11999 (16) | 0.62176 (16) | 0.0179 (4) | |
| C15 | -0.0126 (3) | 0.22615 (16) | 0.64053 (17) | 0.0198 (4) | |
| H15 | -0.1373 | 0.2339 | 0.6462 | 0.024* | |
| C16 | 0.0528 (2) | 0.32029 (15) | 0.65097 (15) | 0.0159 (3) | |
| H16 | -0.0282 | 0.3922 | 0.6641 | 0.019* | |
| C21 | 0.1254 (2) | 0.54510 (14) | 0.70174 (14) | 0.0124 (3) | |
| C22 | 0.0282 (2) | 0.60449 (16) | 0.62413 (15) | 0.0161 (3) | |
| H22 | 0.0638 | 0.5836 | 0.5493 | 0.019* | |
| C23 | -0.1195 (2) | 0.69346 (15) | 0.65361 (15) | 0.0163 (3) | |
| H23 | -0.187 | 0.7315 | 0.6002 | 0.02* | |
| C24 | -0.1672 (2) | 0.72600 (15) | 0.76289 (15) | 0.0159 (3) | |
| C25 | -0.0732 (3) | 0.66642 (16) | 0.84167 (15) | 0.0180 (4) | |
| H25 | -0.1083 | 0.6878 | 0.9163 | 0.022* | |
| C26 | 0.0716 (2) | 0.57583 (15) | 0.81157 (15) | 0.0151 (3) | |
| H26 | 0.1343 | 0.5347 | 0.8658 | 0.018* | |
| C31 | 0.4441 (2) | 0.39989 (15) | 0.76733 (14) | 0.0131 (3) | |
| C32 | 0.3972 (3) | 0.32619 (17) | 0.85375 (15) | 0.0186 (4) | |

supplementary materials

| | | | | | |
|------|---------------|---------------|--------------|--------------|-----|
| H32 | 0.3015 | 0.2914 | 0.8524 | 0.022* | |
| C33 | 0.4873 (3) | 0.30242 (17) | 0.94193 (16) | 0.0194 (4) | |
| H33 | 0.4539 | 0.2516 | 0.9999 | 0.023* | |
| C34 | 0.6271 (2) | 0.35369 (16) | 0.94457 (15) | 0.0169 (4) | |
| C35 | 0.6740 (3) | 0.42844 (16) | 0.85931 (15) | 0.0180 (4) | |
| H35 | 0.7683 | 0.4642 | 0.8614 | 0.022* | |
| C36 | 0.5843 (2) | 0.45100 (16) | 0.77175 (15) | 0.0161 (3) | |
| H36 | 0.6181 | 0.5018 | 0.7139 | 0.019* | |
| C1 | 0.1436 (3) | -0.07965 (17) | 0.6040 (2) | 0.0291 (5) | |
| H1A | 0.2311 | -0.082 | 0.5355 | 0.044* | |
| H1B | 0.0727 | -0.1355 | 0.602 | 0.044* | |
| H1C | 0.206 | -0.0977 | 0.6665 | 0.044* | |
| O1 | 0.0278 (2) | 0.03200 (12) | 0.61544 (14) | 0.0259 (3) | |
| C2 | -0.3886 (3) | 0.88867 (18) | 0.72033 (18) | 0.0257 (4) | |
| H2A | -0.3003 | 0.9208 | 0.6689 | 0.039* | |
| H2B | -0.4815 | 0.9505 | 0.7574 | 0.039* | |
| H2C | -0.4428 | 0.8442 | 0.6799 | 0.039* | |
| O2 | -0.30260 (18) | 0.81656 (12) | 0.80035 (12) | 0.0217 (3) | |
| C3 | 0.6746 (3) | 0.2635 (2) | 1.11707 (17) | 0.0274 (5) | |
| H3A | 0.5497 | 0.2941 | 1.1507 | 0.041* | |
| H3B | 0.7509 | 0.2604 | 1.1716 | 0.041* | |
| H3C | 0.6883 | 0.1867 | 1.0916 | 0.041* | |
| O3 | 0.7257 (2) | 0.33564 (13) | 1.02543 (11) | 0.0231 (3) | |
| C01 | 0.7355 (9) | -0.0476 (6) | 1.0145 (5) | 0.0481 (14)* | 0.5 |
| H01A | 0.7192 | -0.1018 | 1.0762 | 0.072* | 0.5 |
| H01B | 0.734 | -0.0824 | 0.946 | 0.072* | 0.5 |
| H01C | 0.6386 | 0.0211 | 1.0261 | 0.072* | 0.5 |
| C02 | 0.9034 (10) | -0.0180 (6) | 1.0076 (6) | 0.0624 (18)* | 0.5 |
| C03 | 0.9981 (10) | -0.0566 (5) | 1.0829 (5) | 0.0409 (14)* | 0.5 |
| H03 | 0.9624 | -0.1095 | 1.1379 | 0.049* | 0.5 |
| C04 | 1.1548 (11) | -0.0191 (6) | 1.0822 (6) | 0.0619 (18)* | 0.5 |
| H04 | 1.2331 | -0.0426 | 1.1334 | 0.074* | 0.5 |
| C05 | 1.1757 (14) | 0.0568 (8) | 0.9956 (8) | 0.074 (2)* | 0.5 |
| H05 | 1.2674 | 0.0949 | 0.9974 | 0.088* | 0.5 |
| C06 | 1.0987 (9) | 0.0869 (6) | 0.9139 (6) | 0.0521 (15)* | 0.5 |
| H06 | 1.1435 | 0.1327 | 0.8548 | 0.063* | 0.5 |
| C07 | 0.9368 (10) | 0.0471 (5) | 0.9159 (5) | 0.0410 (14)* | 0.5 |
| H07 | 0.865 | 0.0644 | 0.8607 | 0.049* | 0.5 |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|---------------|---------------|---------------|
| Pd | 0.01295 (9) | 0.00955 (9) | 0.00981 (9) | -0.00301 (6) | -0.00176 (6) | -0.00060 (6) |
| P | 0.0125 (2) | 0.0109 (2) | 0.0107 (2) | -0.00299 (16) | -0.00240 (15) | 0.00014 (15) |
| Cl | 0.0238 (2) | 0.0120 (2) | 0.0163 (2) | -0.00615 (16) | 0.00094 (16) | -0.00323 (15) |
| C11 | 0.0160 (8) | 0.0127 (8) | 0.0108 (7) | -0.0056 (6) | -0.0026 (6) | 0.0017 (6) |
| C12 | 0.0149 (8) | 0.0147 (8) | 0.0157 (8) | -0.0044 (7) | -0.0034 (6) | 0.0012 (6) |
| C13 | 0.0194 (9) | 0.0117 (8) | 0.0172 (8) | -0.0027 (7) | -0.0046 (7) | 0.0009 (6) |

| | | | | | | |
|-----|-------------|-------------|-------------|--------------|--------------|-------------|
| C14 | 0.0232 (9) | 0.0140 (8) | 0.0196 (9) | -0.0072 (7) | -0.0086 (7) | 0.0018 (7) |
| C15 | 0.0156 (9) | 0.0180 (9) | 0.0278 (10) | -0.0054 (7) | -0.0070 (7) | -0.0003 (7) |
| C16 | 0.0155 (8) | 0.0136 (8) | 0.0186 (9) | -0.0020 (7) | -0.0046 (7) | -0.0010 (7) |
| C21 | 0.0119 (8) | 0.0110 (8) | 0.0148 (8) | -0.0034 (6) | -0.0029 (6) | 0.0002 (6) |
| C22 | 0.0189 (9) | 0.0165 (9) | 0.0132 (8) | -0.0034 (7) | -0.0042 (7) | -0.0008 (6) |
| C23 | 0.0173 (9) | 0.0148 (8) | 0.0172 (8) | -0.0023 (7) | -0.0073 (7) | 0.0017 (7) |
| C24 | 0.0126 (8) | 0.0145 (8) | 0.0208 (9) | -0.0033 (6) | -0.0028 (7) | -0.0022 (7) |
| C25 | 0.0173 (9) | 0.0212 (9) | 0.0147 (8) | -0.0021 (7) | -0.0025 (7) | -0.0039 (7) |
| C26 | 0.0153 (8) | 0.0161 (8) | 0.0141 (8) | -0.0023 (7) | -0.0045 (6) | -0.0003 (6) |
| C31 | 0.0134 (8) | 0.0138 (8) | 0.0115 (8) | -0.0016 (6) | -0.0033 (6) | -0.0005 (6) |
| C32 | 0.0196 (9) | 0.0223 (9) | 0.0173 (9) | -0.0108 (7) | -0.0056 (7) | 0.0043 (7) |
| C33 | 0.0233 (10) | 0.0206 (9) | 0.0174 (9) | -0.0104 (8) | -0.0067 (7) | 0.0049 (7) |
| C34 | 0.0180 (9) | 0.0192 (9) | 0.0147 (8) | -0.0041 (7) | -0.0055 (7) | -0.0014 (7) |
| C35 | 0.0172 (9) | 0.0210 (9) | 0.0185 (9) | -0.0088 (7) | -0.0054 (7) | 0.0012 (7) |
| C36 | 0.0166 (8) | 0.0170 (9) | 0.0150 (8) | -0.0052 (7) | -0.0032 (7) | 0.0024 (7) |
| C1 | 0.0310 (11) | 0.0126 (9) | 0.0482 (14) | -0.0063 (8) | -0.0169 (10) | 0.0006 (9) |
| O1 | 0.0244 (7) | 0.0132 (7) | 0.0448 (9) | -0.0067 (6) | -0.0144 (7) | -0.0006 (6) |
| C2 | 0.0239 (10) | 0.0197 (10) | 0.0302 (11) | 0.0043 (8) | -0.0088 (8) | -0.0007 (8) |
| O2 | 0.0191 (7) | 0.0194 (7) | 0.0228 (7) | 0.0045 (5) | -0.0052 (5) | -0.0033 (5) |
| C3 | 0.0359 (12) | 0.0337 (12) | 0.0201 (10) | -0.0178 (10) | -0.0149 (9) | 0.0094 (8) |
| O3 | 0.0266 (7) | 0.0310 (8) | 0.0178 (7) | -0.0141 (6) | -0.0122 (6) | 0.0064 (6) |

Geometric parameters (Å, °)

| | | | |
|--------------------|-------------|----------|------------|
| Pd—Cl | 2.2981 (4) | C33—H33 | 0.95 |
| Pd—Cl ⁱ | 2.2981 (4) | C34—O3 | 1.356 (2) |
| Pd—P ⁱ | 2.3409 (4) | C34—C35 | 1.393 (3) |
| Pd—P | 2.3409 (4) | C35—C36 | 1.382 (3) |
| P—C21 | 1.8112 (17) | C35—H35 | 0.95 |
| P—C31 | 1.8124 (18) | C36—H36 | 0.95 |
| P—C11 | 1.8185 (18) | C1—O1 | 1.435 (2) |
| C11—C12 | 1.397 (2) | C1—H1A | 0.98 |
| C11—C16 | 1.400 (2) | C1—H1B | 0.98 |
| C12—C13 | 1.391 (2) | C1—H1C | 0.98 |
| C12—H12 | 0.95 | C2—O2 | 1.431 (2) |
| C13—C14 | 1.399 (3) | C2—H2A | 0.98 |
| C13—H13 | 0.95 | C2—H2B | 0.98 |
| C14—O1 | 1.360 (2) | C2—H2C | 0.98 |
| C14—C15 | 1.394 (3) | C3—O3 | 1.435 (2) |
| C15—C16 | 1.384 (3) | C3—H3A | 0.98 |
| C15—H15 | 0.95 | C3—H3B | 0.98 |
| C16—H16 | 0.95 | C3—H3C | 0.98 |
| C21—C22 | 1.394 (2) | C01—C02 | 1.434 (10) |
| C21—C26 | 1.396 (2) | C01—H01A | 0.98 |
| C22—C23 | 1.389 (3) | C01—H01B | 0.98 |
| C22—H22 | 0.95 | C01—H01C | 0.98 |
| C23—C24 | 1.394 (3) | C02—C03 | 1.296 (10) |
| C23—H23 | 0.95 | C02—C07 | 1.353 (10) |
| C24—O2 | 1.367 (2) | C03—C04 | 1.410 (10) |

supplementary materials

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|------------------------------------|-------------|---------------|-------------|
| C24—C25 | 1.391 (3) | C03—H03 | 0.95 |
| C25—C26 | 1.389 (2) | C04—C05 | 1.372 (12) |
| C25—H25 | 0.95 | C04—H04 | 0.95 |
| C26—H26 | 0.95 | C05—C06 | 1.265 (11) |
| C31—C32 | 1.394 (2) | C05—H05 | 0.95 |
| C31—C36 | 1.400 (3) | C06—C07 | 1.462 (9) |
| C32—C33 | 1.391 (3) | C06—H06 | 0.95 |
| C32—H32 | 0.95 | C07—H07 | 0.95 |
| C33—C34 | 1.393 (3) | | |
| Cl—Pd—Cl ⁱ | 180 | C34—C33—H33 | 120.3 |
| Cl—Pd—P ⁱ | 91.578 (15) | O3—C34—C35 | 115.85 (17) |
| Cl ⁱ —Pd—P ⁱ | 88.422 (15) | O3—C34—C33 | 124.53 (17) |
| Cl—Pd—P | 88.422 (15) | C35—C34—C33 | 119.62 (17) |
| Cl ⁱ —Pd—P | 91.578 (15) | C36—C35—C34 | 120.49 (17) |
| P ⁱ —Pd—P | 180.00 (2) | C36—C35—H35 | 119.8 |
| C21—P—C31 | 106.76 (8) | C34—C35—H35 | 119.8 |
| C21—P—C11 | 103.93 (8) | C35—C36—C31 | 120.74 (17) |
| C31—P—C11 | 105.02 (8) | C35—C36—H36 | 119.6 |
| C21—P—Pd | 110.78 (6) | C31—C36—H36 | 119.6 |
| C31—P—Pd | 110.54 (6) | O1—C1—H1A | 109.5 |
| C11—P—Pd | 118.96 (6) | O1—C1—H1B | 109.5 |
| C12—C11—C16 | 118.07 (16) | H1A—C1—H1B | 109.5 |
| C12—C11—P | 120.39 (14) | O1—C1—H1C | 109.5 |
| C16—C11—P | 121.53 (13) | H1A—C1—H1C | 109.5 |
| C13—C12—C11 | 121.28 (17) | H1B—C1—H1C | 109.5 |
| C13—C12—H12 | 119.4 | C14—O1—C1 | 117.21 (16) |
| C11—C12—H12 | 119.4 | O2—C2—H2A | 109.5 |
| C12—C13—C14 | 119.65 (17) | O2—C2—H2B | 109.5 |
| C12—C13—H13 | 120.2 | H2A—C2—H2B | 109.5 |
| C14—C13—H13 | 120.2 | O2—C2—H2C | 109.5 |
| O1—C14—C15 | 115.85 (17) | H2A—C2—H2C | 109.5 |
| O1—C14—C13 | 124.46 (17) | H2B—C2—H2C | 109.5 |
| C15—C14—C13 | 119.68 (17) | C24—O2—C2 | 117.50 (15) |
| C16—C15—C14 | 120.00 (18) | O3—C3—H3A | 109.5 |
| C16—C15—H15 | 120 | O3—C3—H3B | 109.5 |
| C14—C15—H15 | 120 | H3A—C3—H3B | 109.5 |
| C15—C16—C11 | 121.33 (17) | O3—C3—H3C | 109.5 |
| C15—C16—H16 | 119.3 | H3A—C3—H3C | 109.5 |
| C11—C16—H16 | 119.3 | H3B—C3—H3C | 109.5 |
| C22—C21—C26 | 118.89 (16) | C34—O3—C3 | 117.05 (15) |
| C22—C21—P | 118.30 (13) | C02—C01—H01A | 109.5 |
| C26—C21—P | 122.75 (14) | C02—C01—H01B | 109.5 |
| C23—C22—C21 | 121.52 (17) | H01A—C01—H01B | 109.5 |
| C23—C22—H22 | 119.2 | C02—C01—H01C | 109.5 |
| C21—C22—H22 | 119.2 | H01A—C01—H01C | 109.5 |
| C22—C23—C24 | 118.83 (17) | H01B—C01—H01C | 109.5 |
| C22—C23—H23 | 120.6 | C03—C02—C07 | 130.7 (7) |
| C24—C23—H23 | 120.6 | C03—C02—C01 | 119.1 (7) |

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| O2—C24—C25 | 115.53 (16) | C07—C02—C01 | 110.2 (7) |
| O2—C24—C23 | 124.16 (17) | C02—C03—C04 | 119.2 (7) |
| C25—C24—C23 | 120.30 (17) | C02—C03—H03 | 120.4 |
| C26—C25—C24 | 120.24 (17) | C04—C03—H03 | 120.4 |
| C26—C25—H25 | 119.9 | C05—C04—C03 | 108.9 (8) |
| C24—C25—H25 | 119.9 | C05—C04—H04 | 125.5 |
| C25—C26—C21 | 120.14 (17) | C03—C04—H04 | 125.5 |
| C25—C26—H26 | 119.9 | C06—C05—C04 | 133.6 (10) |
| C21—C26—H26 | 119.9 | C06—C05—H05 | 113.2 |
| C32—C31—C36 | 118.22 (16) | C04—C05—H05 | 113.2 |
| C32—C31—P | 121.90 (14) | C05—C06—C07 | 116.0 (8) |
| C36—C31—P | 119.82 (13) | C05—C06—H06 | 122 |
| C33—C32—C31 | 121.47 (17) | C07—C06—H06 | 122 |
| C33—C32—H32 | 119.3 | C02—C07—C06 | 110.4 (7) |
| C31—C32—H32 | 119.3 | C02—C07—H07 | 124.8 |
| C32—C33—C34 | 119.46 (17) | C06—C07—H07 | 124.8 |
| C32—C33—H33 | 120.3 | | |
| Cl—Pd—P—C21 | 40.71 (6) | C23—C24—C25—C26 | 1.6 (3) |
| Cl ⁱ —Pd—P—C21 | -139.29 (6) | C24—C25—C26—C21 | 0.9 (3) |
| Cl—Pd—P—C31 | -77.43 (6) | C22—C21—C26—C25 | -1.9 (3) |
| Cl ⁱ —Pd—P—C31 | 102.57 (6) | P—C21—C26—C25 | 175.13 (14) |
| Cl—Pd—P—C11 | 160.99 (7) | C21—P—C31—C32 | 81.11 (16) |
| Cl ⁱ —Pd—P—C11 | -19.01 (7) | C11—P—C31—C32 | -28.83 (17) |
| C21—P—C11—C12 | -170.72 (14) | Pd—P—C31—C32 | -158.32 (14) |
| C31—P—C11—C12 | -58.74 (16) | C21—P—C31—C36 | -96.04 (15) |
| Pd—P—C11—C12 | 65.57 (16) | C11—P—C31—C36 | 154.02 (14) |
| C21—P—C11—C16 | 10.31 (17) | Pd—P—C31—C36 | 24.53 (16) |
| C31—P—C11—C16 | 122.28 (15) | C36—C31—C32—C33 | -0.7 (3) |
| Pd—P—C11—C16 | -113.40 (14) | P—C31—C32—C33 | -177.86 (15) |
| C16—C11—C12—C13 | -0.2 (3) | C31—C32—C33—C34 | 0.4 (3) |
| P—C11—C12—C13 | -179.23 (14) | C32—C33—C34—O3 | -179.07 (18) |
| C11—C12—C13—C14 | -0.1 (3) | C32—C33—C34—C35 | 0.4 (3) |
| C12—C13—C14—O1 | -178.36 (18) | O3—C34—C35—C36 | 178.74 (17) |
| C12—C13—C14—C15 | 0.6 (3) | C33—C34—C35—C36 | -0.7 (3) |
| O1—C14—C15—C16 | 178.38 (18) | C34—C35—C36—C31 | 0.4 (3) |
| C13—C14—C15—C16 | -0.6 (3) | C32—C31—C36—C35 | 0.3 (3) |
| C14—C15—C16—C11 | 0.3 (3) | P—C31—C36—C35 | 177.54 (14) |
| C12—C11—C16—C15 | 0.1 (3) | C15—C14—O1—C1 | -175.70 (19) |
| P—C11—C16—C15 | 179.15 (15) | C13—C14—O1—C1 | 3.3 (3) |
| C31—P—C21—C22 | 166.77 (14) | C25—C24—O2—C2 | 172.50 (17) |
| C11—P—C21—C22 | -82.53 (15) | C23—C24—O2—C2 | -6.5 (3) |
| Pd—P—C21—C22 | 46.36 (15) | C35—C34—O3—C3 | 176.95 (18) |
| C31—P—C21—C26 | -10.31 (17) | C33—C34—O3—C3 | -3.6 (3) |
| C11—P—C21—C26 | 100.39 (16) | C07—C02—C03—C04 | 7.9 (11) |
| Pd—P—C21—C26 | -130.73 (14) | C01—C02—C03—C04 | -174.1 (6) |
| C26—C21—C22—C23 | 0.4 (3) | C02—C03—C04—C05 | 0.4 (9) |
| P—C21—C22—C23 | -176.82 (14) | C03—C04—C05—C06 | -9.8 (14) |
| C21—C22—C23—C24 | 2.2 (3) | C04—C05—C06—C07 | 10.3 (14) |

supplementary materials

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| C22—C23—C24—O2 | 175.75 (17) | C03—C02—C07—C06 | -7.4 (10) |
| C22—C23—C24—C25 | -3.2 (3) | C01—C02—C07—C06 | 174.4 (5) |
| O2—C24—C25—C26 | -177.36 (17) | C05—C06—C07—C02 | -1.1 (9) |

Symmetry codes: (i) $-x+1, -y+1, -z+1$.

Hydrogen-bond geometry (\AA , $^\circ$)

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|------------------------------------|-------|-------------|-------------|---------------|
| C01—H01B \cdots O2 ⁱⁱ | 0.98 | 2.36 | 3.327 (7) | 170 |
| C3—H3A \cdots O2 ⁱⁱⁱ | 0.98 | 2.57 | 3.255 (3) | 127 |
| C36—H36 \cdots Cl | 0.95 | 2.79 | 3.5402 (19) | 136 |

Symmetry codes: (ii) $x+1, y-1, z$; (iii) $-x, -y+1, -z+2$.

