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Di- μ -chlorido-bis{[2-(di-*tert*-butylphosphanyl)biphenyl-3-yl- κ^2C^3,P]-palladium(II)} dichloromethane disolvate

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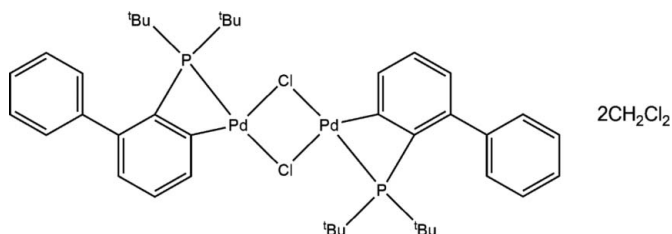
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(C-C) = 0.005$ Å; R factor = 0.039; wR factor = 0.094; data-to-parameter ratio = 22.9.

The asymmetric unit of the title compound, $[Pd_2Cl_2(C_{20}H_{26}P)_2] \cdot 2CH_2Cl_2$, contains one half-molecule of the palladium complex and a dichloromethane solvent molecule. In the complex, two Pd^{II} atoms are bridged by two Cl atoms, with the other two coordination sites occupied by a C atom of the biphenyl system and a P atom, resulting in a distorted square-planar coordination geometry of the Pd^{II} atom and a cyclometallated four-membered ring. The Pd₂Cl₂ unit is located about an inversion center. The planes of the rings of the biphenyl system make a dihedral angle of 66.36 (11)°.

Related literature

For background to palladacycles, see: Beletskaya & Cheprakov (2004); Orlye & Jutland (2005); Herrmann *et al.* (2003). For their applications as catalysts for methoxy-carbonylation, see: Omondi *et al.* (2011); Williams *et al.* (2008). For related structures with Pd^{II} in *ortho*-position, see: Sole *et al.* (2004); Mohr *et al.* (2006); Bennett *et al.* (2010); Christmann *et al.* (2006); Garrou *et al.* (1981).



Experimental

Crystal data

$[Pd_2Cl_2(C_{20}H_{26}P)_2] \cdot 2CH_2Cl_2$
 $M_r = 1048.31$
 Triclinic, $P\bar{1}$
 $a = 9.4741$ (15) Å
 $b = 10.1805$ (15) Å
 $c = 12.0677$ (18) Å
 $\alpha = 103.250$ (4)°
 $\beta = 95.443$ (3)°
 $\gamma = 97.080$ (3)°
 $V = 1115.2$ (3) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 1.27$ mm⁻¹
 $T = 100$ K
 $0.08 \times 0.07 \times 0.02$ mm

Data collection

Bruker X8 APEXII 4K KappaCCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2008)
 $T_{min} = 0.905$, $T_{max} = 0.975$
 15104 measured reflections
 5515 independent reflections
 4344 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.049$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.094$
 $S = 1.01$
 5515 reflections
 241 parameters
 1 restraint
 H-atom parameters constrained
 $\Delta\rho_{max} = 1.16$ e Å⁻³
 $\Delta\rho_{min} = -1.09$ e Å⁻³

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT-Plus (Bruker, 2008); data reduction: SAINT-Plus and XPREP (Bruker, 2008); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 (Farrugia, 2012); software used to prepare material for publication: WinGX (Farrugia, 2012).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2217).

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supporting information

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Di- μ -chlorido-bis{[2-(di-*tert*-butylphosphanyl)biphenyl-3-yl- κ^2C^3,P]palladium(II)} dichloromethane disolvate

Cedric W. Holzapel and Bernard Omondi

S1. Comment

Palladacycles (Beletskaya & Cheprakov, 2004; Orlye & Jutland, 2005; Herrmann *et al.*, 2003) appear as long-sought thermally stable and structurally defined catalysts for Heck, cross-coupling and for methoxycarbonylation of alkenes (Omondi *et al.*, 2011, Williams *et al.*, 2008). However, with the exception of 2-(2'-di-*tert*-butylphosphine)biphenyl palladium(II) acetate, shown in the Fluka and Aldrich catalogues, this complex and other four membered ring phosphapalladacycles (Garrou, 1981) have shown little success in this regard.

The title compound crystallizes as a dichloromethane solvate, with half a molecule of the complex in the asymmetric unit, in which the two Pd centres are bridged by two chlorides and are part of a constrained four-membered ring along with two carbon atoms of a phenyl ring and a phosphorus atom (Fig. 1). The four-membered rings are *trans* across the planar Pd₂Cl₂ unit, the planar Pd₂Cl₂ unit being on the center of inversion at 1/2, 0, 0. The coordination around the palladium atom is distorted square planar, with the angles around the metal deviating significantly from orthogonality yet very planar. The smallest of the angles around the metal centre is 68.84 (10)° and is similar to that previously reported dinuclear cyclopalladate, 68.89 (3)° (Christmann *et al.*, 2006) and of related *ortho*-metallated Pd-structures from literature (Sole *et al.*, 2004; Mohr *et al.*, 2006; Bennett *et al.*, 2010). The other angles around the Pd centers are 85.85 (11), 95.0 (2) and 106.44 (3)° all probably due to steric hindrance caused by *tert*-butyl groups. The Pd—C distance (1.978 (3) Å) is shorter than the Pd—P distance (2.2327 (9) Å) which is in good agreement with the Pd—P separation in similar four-membered compounds with a *sp*²-hybridized carbon. The carbon-phosphorus distance that is part of the constrained ring is significantly shorter [1.824 (3) Å] compared to the other two [1.860 (3) and 1.879 (4) Å] as observed in most cyclopalladates. The planes of the phenyl rings of the biphenyl moiety have a dihedral angle of 66.36 (11)°.

S2. Experimental

(2-Biphenyl)-di-*tert*-butylphosphine (597 mg, 2 mmol) was added to the solution of lithium chloride (170 mg, 4 mmol) and palladium chloride (355 mg, 2 mmol) in 2:1 methanol-dichloromethane (15 ml) under argon. The mixture was heated under reflux under argon for 1 h. The solvent was evaporated *in vacuo*. To the residue was added dichloromethane (15 ml) and water (10 ml) and the mixture stirred for 30 minutes. The organic phase was separated, dried (anhydrous sodium sulfate) and the solvent evaporated to leave a residue (823 mg) which was chromatographed over silica gel (Davisil, 20 g) using 5% methanol in dichloromethane as mobile phase. The eluent (120 ml) was evaporated to leave 1 as a colourless crystalline mass (676 mg, 82%). Good quality crystals (m.p. 162–165 °C, decomp) were obtained by diffusion of the vapours of ether into a solution of the title compound in dichloromethane at room temperature.

¹H NMR (300 MHz, CDCl₃): δ = 1.37 (d, *J*_{PH} = 1.3 Hz, 36H, C(CH₃)₃), 7.05 (d, *J* = 3.2 Hz, 2H), 7.25 (dd, *J* = 7.5 and 3.2 Hz, 2H), 7.30–7.50 (m, 8H), 7.58 (t, *J* = 7.5 Hz, 2H), 7.85 (t, *J* = 7.5 Hz, 2H);

$^{13}\text{C}\{^1\text{H}\}$ NMR (75 Mz, CDCl_3): $\delta = 30.37$ (d, $J_{\text{PC}} = 5$ Hz, $\text{C}(\text{CH}_3)_3$), 35.97 (d, $J_{\text{PC}} = 12$ Hz, $\text{C}(\text{CH}_3)_3$), 127.44 (d, $J_{\text{PC}} = 8.5$ Hz, C-aromatic), 127.78 (s, C-aromatic), 127.44 (d, $J_{\text{PC}} = 8.5$ Hz, C-aromatic), 127.78 (s, C-aromatic), 128.18 (s, C-aromatic), 129.19 (s, C-aromatic), 129.28 (s, C-aromatic), 133.55 (d, $J_{\text{PC}} = 26.0$ Hz, C-aromatic), 138.55 (d, $J_{\text{PC}} = 32.0$, C-aromatic), 141.90 (d, $J_{\text{PC}} = 3.9$ Hz, C-aromatic), 142.81 (s, C-aromatic);

$^{31}\text{P}\{^1\text{H}\}$ (97 MHz, CDCl_3): $\delta = -17.80$.

The title compound was also obtained in essentially quantitative yield by reacting the above phosphine with an equimolar amount of (*cis*, *cis*-1,5-cyclooctadiene) palladium (II)-chloride in dichloromethane under reflux (1 hr) or at room temperature (18 hrs).

S3. Refinement

Carbon-bound H-atoms were placed in calculated positions [$\text{C}-\text{H} = 0.98$ Å for Me H atoms, 0.99 Å for Methylene H atoms and 0.95 Å for aromatic H atoms; $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ (1.5 for Me groups)] and were included in the refinement in the riding model approximation.

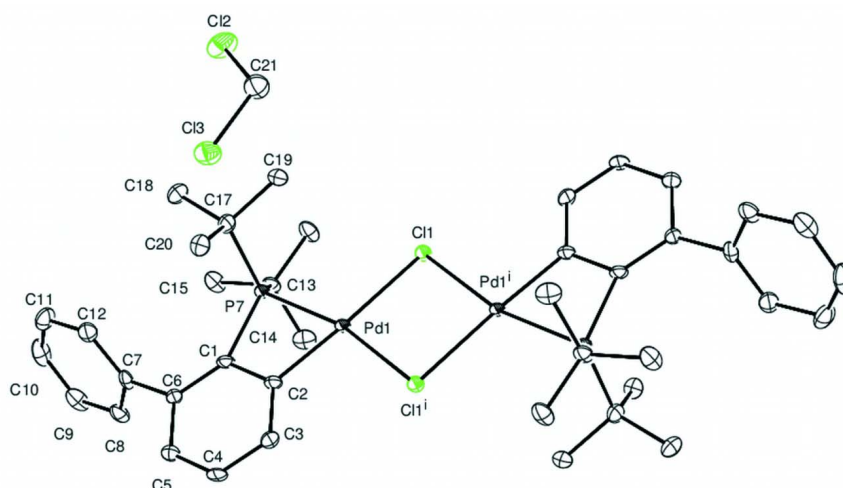


Figure 1

The molecular structure and atom-numbering scheme for (I), with displacement ellipsoids drawn at the 50% probability level. Hydrogen atoms have been omitted for clarity. The asymmetric unit only has only half a molecule of the palladium complex, the other half generated by the symmetry operator $-x, -y, -z$.

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Crystal data

$[\text{Pd}_2\text{Cl}_2(\text{C}_{20}\text{H}_{26}\text{P})_2] \cdot 2\text{CH}_2\text{Cl}_2$

$M_r = 1048.31$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 9.4741$ (15) Å

$b = 10.1805$ (15) Å

$c = 12.0677$ (18) Å

$\alpha = 103.250$ (4) $^\circ$

$\beta = 95.443$ (3) $^\circ$

$\gamma = 97.080$ (3) $^\circ$

$V = 1115.2$ (3) Å 3

$Z = 1$

$F(000) = 532$

$D_x = 1.561$ Mg m $^{-3}$

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

Cell parameters from 15104 reflections

$\theta = 1.8\text{--}28.3$ $^\circ$

$\mu = 1.27$ mm $^{-1}$

$T = 100$ K

Block, yellow

$0.08 \times 0.07 \times 0.02$ mm

Data collection

Bruker X8 APEXII 4K KappaCCD
diffractometer
Graphite monochromator
 φ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2008)
 $T_{\min} = 0.905$, $T_{\max} = 0.975$
15104 measured reflections

5515 independent reflections
4344 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.049$
 $\theta_{\max} = 28.3^\circ$, $\theta_{\min} = 1.8^\circ$
 $h = -12 \rightarrow 12$
 $k = -13 \rightarrow 13$
 $l = -15 \rightarrow 16$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.039$
 $wR(F^2) = 0.094$
 $S = 1.01$
5515 reflections
241 parameters
1 restraint
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
 $w = 1/[\sigma^2(F_o^2) + (0.0476P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 1.16 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -1.09 \text{ e } \text{\AA}^{-3}$

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 $F^2 > \sigma(F^2)$ 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. >>> The Following ALERTS were generated <<<< Format: alert-number_ALERT_alert-type_alert-level text 232_ALERT_2_C Hirshfeld Test Diff (M-X) Pd1 - C11.. 5.30 su Apllying DELU restraints does not remove this alert. 250_ALERT_2_C Large U3/U1 Ratio for Average U(i,j) Tensor ... 2.23 Visual inspections of ellipsoids show no abnormalities. $R = 3.8\%$. 911_ALERT_3_C Missing # FCF Refl Between THmin & STh/L= 0.600 3 912_ALERT_4_C Missing # of FCF Reflections Above STh/L= 0.600 62 Noted, no measures taken

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

| | x | y | z | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|----|------------|------------|------------|----------------------------------|
| C1 | 0.4506 (3) | 0.2051 (4) | 0.3645 (3) | 0.0143 (7) |
| C2 | 0.5538 (3) | 0.1684 (4) | 0.2933 (3) | 0.0144 (7) |
| C3 | 0.6977 (3) | 0.1820 (4) | 0.3385 (3) | 0.0178 (7) |
| H3 | 0.7687 | 0.1578 | 0.2902 | 0.021* |
| C4 | 0.7336 (3) | 0.2319 (4) | 0.4564 (3) | 0.0189 (7) |
| H4 | 0.831 | 0.2437 | 0.4889 | 0.023* |
| C5 | 0.6289 (3) | 0.2652 (4) | 0.5278 (3) | 0.0178 (7) |
| H5 | 0.6566 | 0.2975 | 0.6082 | 0.021* |
| C6 | 0.4844 (3) | 0.2521 (3) | 0.4840 (3) | 0.0133 (7) |
| C7 | 0.3771 (3) | 0.2810 (4) | 0.5662 (3) | 0.0161 (7) |
| C8 | 0.3568 (4) | 0.1982 (4) | 0.6430 (3) | 0.0198 (8) |
| H8 | 0.4105 | 0.1251 | 0.6417 | 0.024* |

| | | | | |
|------|--------------|--------------|---------------|--------------|
| C9 | 0.2587 (4) | 0.2219 (4) | 0.7209 (3) | 0.0245 (8) |
| H9 | 0.2457 | 0.1656 | 0.773 | 0.029* |
| C10 | 0.1794 (4) | 0.3281 (4) | 0.7226 (3) | 0.0288 (9) |
| H10 | 0.1098 | 0.3426 | 0.7741 | 0.035* |
| C11 | 0.2018 (4) | 0.4135 (4) | 0.6489 (3) | 0.0259 (9) |
| H11 | 0.1495 | 0.4878 | 0.6517 | 0.031* |
| C12 | 0.3009 (4) | 0.3899 (4) | 0.5708 (3) | 0.0201 (8) |
| H12 | 0.3164 | 0.4484 | 0.5207 | 0.024* |
| C13 | 0.1586 (3) | 0.0291 (4) | 0.2698 (3) | 0.0161 (7) |
| C14 | 0.2346 (4) | -0.0917 (4) | 0.2839 (3) | 0.0221 (8) |
| H14A | 0.3069 | -0.0628 | 0.3518 | 0.033* |
| H14B | 0.2812 | -0.1234 | 0.2156 | 0.033* |
| H14C | 0.1642 | -0.1661 | 0.2933 | 0.033* |
| C15 | 0.0822 (4) | 0.0747 (4) | 0.3750 (3) | 0.0215 (8) |
| H15A | 0.0348 | 0.1531 | 0.3674 | 0.032* |
| H15B | 0.1524 | 0.1008 | 0.4443 | 0.032* |
| H15C | 0.0104 | -0.0006 | 0.3809 | 0.032* |
| C16 | 0.0503 (4) | -0.0164 (4) | 0.1600 (3) | 0.0210 (8) |
| H16A | -0.0155 | -0.097 | 0.1637 | 0.032* |
| H16B | 0.1018 | -0.0389 | 0.0931 | 0.032* |
| H16C | -0.0043 | 0.0577 | 0.1532 | 0.032* |
| C17 | 0.2358 (4) | 0.3161 (4) | 0.2158 (3) | 0.0178 (7) |
| C18 | 0.1118 (4) | 0.3638 (4) | 0.2795 (3) | 0.0239 (8) |
| H18A | 0.1377 | 0.3755 | 0.3622 | 0.036* |
| H18B | 0.0258 | 0.2954 | 0.2527 | 0.036* |
| H18C | 0.0927 | 0.451 | 0.2645 | 0.036* |
| C19 | 0.1888 (4) | 0.2829 (4) | 0.0851 (3) | 0.0217 (8) |
| H19A | 0.1082 | 0.208 | 0.0641 | 0.033* |
| H19B | 0.2692 | 0.2557 | 0.0442 | 0.033* |
| H19C | 0.1593 | 0.3638 | 0.0641 | 0.033* |
| C20 | 0.3659 (4) | 0.4295 (4) | 0.2461 (3) | 0.0207 (8) |
| H20A | 0.3418 | 0.5083 | 0.2185 | 0.031* |
| H20B | 0.4469 | 0.3958 | 0.2095 | 0.031* |
| H20C | 0.3922 | 0.4568 | 0.3295 | 0.031* |
| C21 | 0.3306 (4) | 0.6222 (4) | -0.0511 (3) | 0.0265 (9) |
| H21A | 0.3451 | 0.6825 | -0.1041 | 0.032* |
| H21B | 0.3437 | 0.5292 | -0.0916 | 0.032* |
| Cl1 | 0.32540 (8) | -0.02556 (9) | -0.05326 (7) | 0.01603 (17) |
| Cl2 | 0.15439 (10) | 0.61906 (11) | -0.01468 (10) | 0.0332 (2) |
| Cl3 | 0.46013 (9) | 0.68133 (10) | 0.07196 (8) | 0.0267 (2) |
| P7 | 0.30128 (8) | 0.16081 (9) | 0.24901 (7) | 0.01217 (18) |
| Pd1 | 0.46067 (2) | 0.08342 (3) | 0.13497 (2) | 0.01217 (8) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|----|-------------|-------------|-------------|-------------|--------------|-------------|
| C1 | 0.0082 (14) | 0.0204 (18) | 0.0149 (16) | 0.0042 (13) | -0.0005 (12) | 0.0051 (14) |
| C2 | 0.0097 (15) | 0.0189 (17) | 0.0145 (16) | 0.0039 (13) | -0.0009 (12) | 0.0040 (14) |

| | | | | | | |
|-----|--------------|--------------|--------------|--------------|--------------|--------------|
| C3 | 0.0091 (15) | 0.0250 (19) | 0.0186 (18) | 0.0056 (14) | 0.0036 (13) | 0.0016 (15) |
| C4 | 0.0058 (14) | 0.029 (2) | 0.0196 (18) | 0.0048 (14) | -0.0031 (13) | 0.0029 (16) |
| C5 | 0.0126 (16) | 0.0218 (18) | 0.0170 (17) | 0.0037 (14) | -0.0014 (13) | 0.0018 (15) |
| C6 | 0.0103 (15) | 0.0157 (17) | 0.0134 (16) | 0.0029 (13) | 0.0014 (12) | 0.0022 (13) |
| C7 | 0.0079 (14) | 0.0224 (18) | 0.0123 (16) | -0.0026 (13) | 0.0005 (12) | -0.0042 (14) |
| C8 | 0.0131 (16) | 0.027 (2) | 0.0156 (17) | -0.0011 (14) | -0.0021 (13) | 0.0022 (15) |
| C9 | 0.0169 (17) | 0.037 (2) | 0.0172 (18) | -0.0062 (16) | 0.0018 (14) | 0.0069 (17) |
| C10 | 0.0174 (18) | 0.040 (2) | 0.021 (2) | -0.0046 (17) | 0.0088 (15) | -0.0054 (18) |
| C11 | 0.0153 (17) | 0.027 (2) | 0.032 (2) | 0.0062 (15) | 0.0051 (15) | -0.0019 (18) |
| C12 | 0.0136 (16) | 0.025 (2) | 0.0191 (18) | 0.0023 (14) | 0.0015 (14) | 0.0012 (15) |
| C13 | 0.0090 (15) | 0.0232 (19) | 0.0148 (16) | 0.0007 (13) | -0.0006 (12) | 0.0036 (14) |
| C14 | 0.0157 (17) | 0.0221 (19) | 0.029 (2) | -0.0014 (15) | 0.0012 (15) | 0.0091 (16) |
| C15 | 0.0147 (16) | 0.032 (2) | 0.0166 (18) | -0.0025 (15) | 0.0038 (13) | 0.0055 (16) |
| C16 | 0.0121 (16) | 0.031 (2) | 0.0161 (17) | -0.0043 (15) | 0.0001 (13) | 0.0023 (16) |
| C17 | 0.0142 (16) | 0.0217 (18) | 0.0171 (17) | 0.0050 (14) | 0.0038 (13) | 0.0017 (15) |
| C18 | 0.0174 (17) | 0.029 (2) | 0.026 (2) | 0.0107 (16) | 0.0057 (15) | 0.0034 (17) |
| C19 | 0.0218 (18) | 0.028 (2) | 0.0159 (18) | 0.0101 (16) | -0.0006 (14) | 0.0053 (16) |
| C20 | 0.0211 (18) | 0.0208 (19) | 0.0203 (18) | 0.0043 (15) | 0.0045 (14) | 0.0039 (15) |
| C21 | 0.0205 (18) | 0.031 (2) | 0.027 (2) | 0.0032 (16) | 0.0027 (15) | 0.0062 (18) |
| C11 | 0.0066 (3) | 0.0255 (5) | 0.0139 (4) | 0.0042 (3) | 0.0010 (3) | -0.0001 (3) |
| C12 | 0.0144 (4) | 0.0346 (6) | 0.0501 (7) | 0.0044 (4) | 0.0032 (4) | 0.0093 (5) |
| C13 | 0.0176 (4) | 0.0300 (5) | 0.0311 (5) | 0.0015 (4) | -0.0002 (4) | 0.0073 (4) |
| P7 | 0.0049 (3) | 0.0191 (4) | 0.0116 (4) | 0.0028 (3) | 0.0008 (3) | 0.0014 (3) |
| Pd1 | 0.00434 (11) | 0.01965 (14) | 0.01151 (13) | 0.00282 (9) | 0.00054 (8) | 0.00138 (10) |

Geometric parameters (Å, °)

| | | | |
|---------|-----------|----------|-----------|
| C1—C2 | 1.391 (5) | C14—H14C | 0.98 |
| C1—C6 | 1.403 (5) | C15—H15A | 0.98 |
| C1—P7 | 1.824 (3) | C15—H15B | 0.98 |
| C2—C3 | 1.398 (4) | C15—H15C | 0.98 |
| C2—Pd1 | 1.978 (3) | C16—H16A | 0.98 |
| C3—C4 | 1.389 (5) | C16—H16B | 0.98 |
| C3—H3 | 0.95 | C16—H16C | 0.98 |
| C4—C5 | 1.397 (5) | C17—C18 | 1.532 (5) |
| C4—H4 | 0.95 | C17—C20 | 1.535 (5) |
| C5—C6 | 1.399 (4) | C17—C19 | 1.544 (5) |
| C5—H5 | 0.95 | C17—P7 | 1.879 (4) |
| C6—C7 | 1.495 (4) | C18—H18A | 0.98 |
| C7—C12 | 1.389 (5) | C18—H18B | 0.98 |
| C7—C8 | 1.400 (5) | C18—H18C | 0.98 |
| C8—C9 | 1.387 (5) | C19—H19A | 0.98 |
| C8—H8 | 0.95 | C19—H19B | 0.98 |
| C9—C10 | 1.388 (6) | C19—H19C | 0.98 |
| C9—H9 | 0.95 | C20—H20A | 0.98 |
| C10—C11 | 1.391 (6) | C20—H20B | 0.98 |
| C10—H10 | 0.95 | C20—H20C | 0.98 |
| C11—C12 | 1.396 (5) | C21—C12 | 1.764 (4) |

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|-------------|-----------|---------------------------|------------|
| C11—H11 | 0.95 | C21—C13 | 1.771 (4) |
| C12—H12 | 0.95 | C21—H21A | 0.99 |
| C13—C15 | 1.529 (5) | C21—H21B | 0.99 |
| C13—C14 | 1.534 (5) | C11—Pd1 ⁱ | 2.4154 (8) |
| C13—C16 | 1.539 (4) | C11—Pd1 | 2.4444 (9) |
| C13—P7 | 1.860 (3) | P7—Pd1 | 2.2328 (9) |
| C14—H14A | 0.98 | Pd1—C11 ⁱ | 2.4154 (8) |
| C14—H14B | 0.98 | | |
| C2—C1—C6 | 122.1 (3) | H15A—C15—H15C | 109.5 |
| C2—C1—P7 | 95.0 (2) | H15B—C15—H15C | 109.5 |
| C6—C1—P7 | 142.8 (3) | C13—C16—H16A | 109.5 |
| C1—C2—C3 | 120.7 (3) | C13—C16—H16B | 109.5 |
| C1—C2—Pd1 | 109.9 (2) | H16A—C16—H16B | 109.5 |
| C3—C2—Pd1 | 129.1 (3) | C13—C16—H16C | 109.5 |
| C4—C3—C2 | 117.9 (3) | H16A—C16—H16C | 109.5 |
| C4—C3—H3 | 121 | H16B—C16—H16C | 109.5 |
| C2—C3—H3 | 121 | C18—C17—C20 | 110.1 (3) |
| C3—C4—C5 | 121.1 (3) | C18—C17—C19 | 109.4 (3) |
| C3—C4—H4 | 119.4 | C20—C17—C19 | 108.6 (3) |
| C5—C4—H4 | 119.4 | C18—C17—P7 | 115.1 (3) |
| C4—C5—C6 | 121.7 (3) | C20—C17—P7 | 106.2 (2) |
| C4—C5—H5 | 119.1 | C19—C17—P7 | 107.3 (2) |
| C6—C5—H5 | 119.1 | C17—C18—H18A | 109.5 |
| C5—C6—C1 | 116.4 (3) | C17—C18—H18B | 109.5 |
| C5—C6—C7 | 118.7 (3) | H18A—C18—H18B | 109.5 |
| C1—C6—C7 | 124.8 (3) | C17—C18—H18C | 109.5 |
| C12—C7—C8 | 119.3 (3) | H18A—C18—H18C | 109.5 |
| C12—C7—C6 | 121.9 (3) | H18B—C18—H18C | 109.5 |
| C8—C7—C6 | 118.7 (3) | C17—C19—H19A | 109.5 |
| C9—C8—C7 | 120.5 (4) | C17—C19—H19B | 109.5 |
| C9—C8—H8 | 119.8 | H19A—C19—H19B | 109.5 |
| C7—C8—H8 | 119.8 | C17—C19—H19C | 109.5 |
| C8—C9—C10 | 119.9 (4) | H19A—C19—H19C | 109.5 |
| C8—C9—H9 | 120.1 | H19B—C19—H19C | 109.5 |
| C10—C9—H9 | 120.1 | C17—C20—H20A | 109.5 |
| C9—C10—C11 | 120.1 (4) | C17—C20—H20B | 109.5 |
| C9—C10—H10 | 120 | H20A—C20—H20B | 109.5 |
| C11—C10—H10 | 120 | C17—C20—H20C | 109.5 |
| C10—C11—C12 | 120.0 (4) | H20A—C20—H20C | 109.5 |
| C10—C11—H11 | 120 | H20B—C20—H20C | 109.5 |
| C12—C11—H11 | 120 | C12—C21—C13 | 111.6 (2) |
| C7—C12—C11 | 120.2 (4) | C12—C21—H21A | 109.3 |
| C7—C12—H12 | 119.9 | C13—C21—H21A | 109.3 |
| C11—C12—H12 | 119.9 | C12—C21—H21B | 109.3 |
| C15—C13—C14 | 109.0 (3) | C13—C21—H21B | 109.3 |
| C15—C13—C16 | 110.7 (3) | H21A—C21—H21B | 108 |
| C14—C13—C16 | 108.7 (3) | Pd1 ⁱ —C11—Pd1 | 92.33 (3) |

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| C15—C13—P7 | 114.4 (2) | C1—P7—C13 | 112.74 (15) |
| C14—C13—P7 | 105.4 (2) | C1—P7—C17 | 112.13 (16) |
| C16—C13—P7 | 108.4 (2) | C13—P7—C17 | 114.90 (15) |
| C13—C14—H14A | 109.5 | C1—P7—Pd1 | 85.84 (11) |
| C13—C14—H14B | 109.5 | C13—P7—Pd1 | 115.73 (12) |
| H14A—C14—H14B | 109.5 | C17—P7—Pd1 | 112.17 (11) |
| C13—C14—H14C | 109.5 | C2—Pd1—P7 | 68.84 (10) |
| H14A—C14—H14C | 109.5 | C2—Pd1—Cl1 ⁱ | 97.07 (10) |
| H14B—C14—H14C | 109.5 | P7—Pd1—Cl1 ⁱ | 165.87 (3) |
| C13—C15—H15A | 109.5 | C2—Pd1—Cl1 | 174.81 (10) |
| C13—C15—H15B | 109.5 | P7—Pd1—Cl1 | 106.44 (3) |
| H15A—C15—H15B | 109.5 | Cl1 ⁱ —Pd1—Cl1 | 87.67 (3) |
| C13—C15—H15C | 109.5 | | |
| | | | |
| C6—C1—C2—C3 | -2.4 (5) | C14—C13—P7—C1 | -55.2 (3) |
| P7—C1—C2—C3 | 179.9 (3) | C16—C13—P7—C1 | -171.4 (2) |
| C6—C1—C2—Pd1 | 172.0 (3) | C15—C13—P7—C17 | -65.6 (3) |
| P7—C1—C2—Pd1 | -5.8 (2) | C14—C13—P7—C17 | 174.7 (2) |
| C1—C2—C3—C4 | 0.5 (5) | C16—C13—P7—C17 | 58.4 (3) |
| Pd1—C2—C3—C4 | -172.6 (3) | C15—C13—P7—Pd1 | 161.1 (2) |
| C2—C3—C4—C5 | 1.2 (6) | C14—C13—P7—Pd1 | 41.4 (3) |
| C3—C4—C5—C6 | -1.2 (6) | C16—C13—P7—Pd1 | -74.9 (2) |
| C4—C5—C6—C1 | -0.6 (5) | C18—C17—P7—C1 | -93.1 (3) |
| C4—C5—C6—C7 | 176.1 (3) | C20—C17—P7—C1 | 28.9 (3) |
| C2—C1—C6—C5 | 2.4 (5) | C19—C17—P7—C1 | 144.9 (2) |
| P7—C1—C6—C5 | 178.7 (3) | C18—C17—P7—C13 | 37.4 (3) |
| C2—C1—C6—C7 | -174.1 (3) | C20—C17—P7—C13 | 159.4 (2) |
| P7—C1—C6—C7 | 2.2 (7) | C19—C17—P7—C13 | -84.6 (3) |
| C5—C6—C7—C12 | 114.5 (4) | C18—C17—P7—Pd1 | 172.3 (2) |
| C1—C6—C7—C12 | -69.1 (5) | C20—C17—P7—Pd1 | -65.7 (2) |
| C5—C6—C7—C8 | -63.4 (4) | C19—C17—P7—Pd1 | 50.3 (3) |
| C1—C6—C7—C8 | 113.0 (4) | C1—C2—Pd1—P7 | 5.1 (2) |
| C12—C7—C8—C9 | 1.8 (5) | C3—C2—Pd1—P7 | 178.8 (4) |
| C6—C7—C8—C9 | 179.8 (3) | C1—C2—Pd1—Cl1 ⁱ | -176.0 (2) |
| C7—C8—C9—C10 | 0.3 (5) | C3—C2—Pd1—Cl1 ⁱ | -2.3 (3) |
| C8—C9—C10—C11 | -2.2 (6) | C1—P7—Pd1—C2 | -3.63 (15) |
| C9—C10—C11—C12 | 1.9 (6) | C13—P7—Pd1—C2 | -116.91 (16) |
| C8—C7—C12—C11 | -2.0 (5) | C17—P7—Pd1—C2 | 108.59 (16) |
| C6—C7—C12—C11 | -180.0 (3) | C1—P7—Pd1—Cl1 ⁱ | -8.08 (19) |
| C10—C11—C12—C7 | 0.2 (5) | C13—P7—Pd1—Cl1 ⁱ | -121.36 (17) |
| C2—C1—P7—C13 | 121.0 (2) | C17—P7—Pd1—Cl1 ⁱ | 104.15 (17) |
| C6—C1—P7—C13 | -55.8 (5) | C1—P7—Pd1—Cl1 | 174.08 (11) |
| C2—C1—P7—C17 | -107.4 (2) | C13—P7—Pd1—Cl1 | 60.80 (12) |
| C6—C1—P7—C17 | 75.7 (5) | C17—P7—Pd1—Cl1 | -73.70 (12) |
| C2—C1—P7—Pd1 | 4.8 (2) | Pd1 ⁱ —Cl1—Pd1—P7 | 179.47 (3) |

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|---------------|------------|--|---|
| C6—C1—P7—Pd1 | -172.0 (4) | Pd1 ⁱ —C11—Pd1—C11 ⁱ | 0 |
| C15—C13—P7—C1 | 64.6 (3) | | |

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