

Dibromidobis[2-(dicyclohexylphosphanyl)-biphenyl- κ P]palladium(II)

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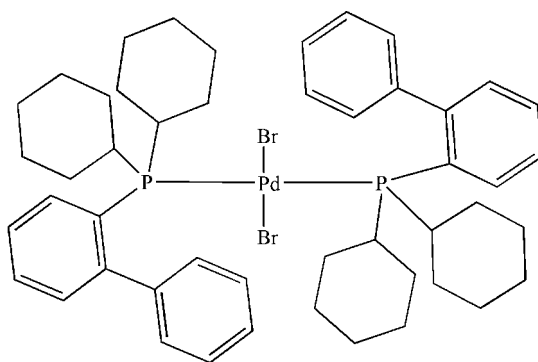
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Key indicators: single-crystal X-ray study; $T = 291$ K; mean $\sigma(\text{C}-\text{C}) = 0.014$ Å; R factor = 0.066; wR factor = 0.180; data-to-parameter ratio = 15.8.

The title compound, $[\text{PdBr}_2(\text{C}_{24}\text{H}_{31}\text{P})_2]$, has a distorted *trans* square-planar coordination of the Pd atom, which occupies an inversion centre. The most important bond distances include Pd–P of 2.380 (2) Å and Pd–Br of 2.515 (2) Å. Weak intermolecular π – π interactions between the benzene rings of adjacent molecules [centroid–centroid distance = 3.949 (6) Å] are present *via* crystallographic inversion centres, resulting in a one-dimensional supramolecular architecture.

Related literature

For related literature, see: Barder *et al.* (2005); Christmann *et al.* (2006); Stark & Whitmire (1997); Tomori *et al.* (2000); Tsuji (1995); Xu *et al.* (2007).



Experimental

Crystal data

$[\text{PdBr}_2(\text{C}_{24}\text{H}_{31}\text{P})_2]$
 $M_r = 967.14$
 Triclinic, $P\bar{1}$
 $a = 9.817$ (8) Å
 $b = 9.827$ (8) Å
 $c = 11.957$ (10) Å
 $\alpha = 91.582$ (11)°
 $\beta = 108.822$ (10)°

$\gamma = 103.713$ (10)°
 $V = 1053.9$ (15) Å³
 $Z = 1$
 Mo $K\alpha$ radiation
 $\mu = 2.44$ mm⁻¹
 $T = 291$ (2) K
 $0.14 \times 0.10 \times 0.09$ mm

Data collection

Bruker SMART APEX CCD diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.723$, $T_{\max} = 0.803$

7316 measured reflections
 3811 independent reflections
 2810 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.038$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.066$
 $wR(F^2) = 0.180$
 $S = 1.10$
 3811 reflections

241 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.71$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.42$ e Å⁻³

Data collection: SMART (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: SHELXTL (Sheldrick, 2008); software used to prepare material for publication: PLATON (Spek, 2003) and SHELXTL.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SI2114).

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supplementary materials

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Comment

Phosphine complexes of palladium have widely been used as catalysts for various reactions (Tsuji, 1995). These complexes are easily prepared from palladium(II) salts and an excess of phosphine ligands. Among them, monophosphinobiaryl complexes of palladium are one of the most important ones (Barder *et al.*, 2005; Christmann *et al.*, 2006; Xu *et al.*, 2007).

The title complex has crystallographic inversion symmetry C_i (Fig.1). The Pd atom is in a square-planar environment, while the *trans* 2-(Dicyclohexylphosphanyl)biphenyl ligands are in an eclipsed conformation. The dihedral angles of the benzene rings are $60.8 (2)^\circ$. The Pd—P [2.380 (2) Å] and Pd—Br [2.515 (5) Å] bond lengths are longer than the related triphenylphosphine complex of palladium [2.336 (2)Å and 2.4169 (13) Å](Stark & Whitmire, 1997) possibly due to the steric bulk of the ligand. Weak intermolecular $\pi \cdots \pi$ interactions between the benzene rings C19 - C24 (Cg4) of inversion related adjacent molecules [centroid-centroid distance Cg4 \cdots Cg4ⁱⁱ is 3.949 (6) Å, the perpendicular distance Cg4 on ring Cg4ⁱⁱ is 3.582 Å, and the slippage is 1.663 Å, symmetry code ii = 1 - x, 1 - y, 1 - z] were calculated for the structure of the title complex with the programme PLATON (Spek, 2003), resulting in a one-dimensional supramolecular architecture.

Experimental

2-(Dicyclohexylphosphanyl)biphenyl was prepared as described in the literature (Tomori *et al.*, 2000). A solution of PdBr₂(PhCN)₂ (1 mmol) and 2-(Dicyclohexylphosphanyl)biphenyl (2 mmol) in dry benzene (5 ml) was stirred for 1 day, removal of solvent resulted in a yellow powder that was recrystallized from dichloromethane-petroleum ether solution at room temperature to give the desired product as yellow crystals suitable for single-crystal X-ray diffraction.

Refinement

H atoms were placed in calculated positions ($C_{sp^2}-H = 0.93$ Å, $C_{sp^3}-H = 0.97-0.98$ Å) and refined as riding on their carriers with isotropic displacement parameters $U_{iso}(H) = 1.2$ times $U_{eq}(C)$.

Figures

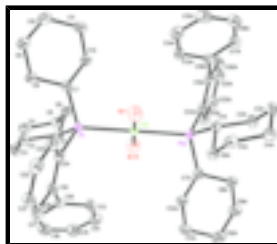


Fig. 1. The molecular structure of the title compound with displacement ellipsoids of the non-hydrogen atoms drawn at the 30% probability level. Inversion related atoms are labelled with an A. (Symmetry code: 2 - x, 1 - y, 1 - z).

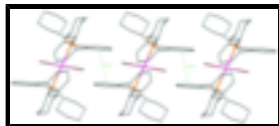


Fig. 2. Partial view of the crystal packing showing the formation of the chain motif of molecules formed by the intermolecular $\pi \cdots \pi$ interactions, extending along the a axis.

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Crystal data

| | |
|---|---|
| [PdBr ₂ (C ₂₄ H ₃₁ P) ₂] | $Z = 1$ |
| $M_r = 967.14$ | $F_{000} = 496$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.524 \text{ Mg m}^{-3}$ |
| $a = 9.817 (8) \text{ \AA}$ | Mo $K\alpha$ radiation |
| $b = 9.827 (8) \text{ \AA}$ | $\lambda = 0.71073 \text{ \AA}$ |
| $c = 11.957 (10) \text{ \AA}$ | Cell parameters from 1503 reflections |
| $\alpha = 91.582 (11)^\circ$ | $\theta = 2.4\text{--}21.7^\circ$ |
| $\beta = 108.822 (10)^\circ$ | $\mu = 2.45 \text{ mm}^{-1}$ |
| $\gamma = 103.713 (10)^\circ$ | $T = 291 (2) \text{ K}$ |
| $V = 1053.9 (15) \text{ \AA}^3$ | Block, yellow |
| | $0.14 \times 0.10 \times 0.09 \text{ mm}$ |

Data collection

| | |
|---|--|
| Bruker SMART APEX CCD diffractometer | 3811 independent reflections |
| Radiation source: fine-focus sealed tube | 2810 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{\text{int}} = 0.039$ |
| $T = 291(2) \text{ K}$ | $\theta_{\text{max}} = 25.5^\circ$ |
| φ and ω scans | $\theta_{\text{min}} = 2.4^\circ$ |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | $h = -11 \rightarrow 11$ |
| $T_{\text{min}} = 0.723$, $T_{\text{max}} = 0.803$ | $k = -11 \rightarrow 11$ |
| 7316 measured reflections | $l = -14 \rightarrow 14$ |

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.066$ | H-atom parameters constrained |
| $wR(F^2) = 0.180$ | $w = 1/[\sigma^2(F_o^2) + (0.0609P)^2 + 7.3363P]$ |
| $S = 1.10$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3811 reflections | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 241 parameters | $\Delta\rho_{\text{max}} = 0.71 \text{ e \AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | $\Delta\rho_{\text{min}} = -1.42 \text{ e \AA}^{-3}$ |
| | 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 $F^2 > \sigma(F^2)$ 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> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|--------------|--------------|----------------------------------|
| Pd1 | 1.0000 | 0.5000 | 0.5000 | 0.0252 (2) |
| Br1 | 0.96825 (13) | 0.71510 (13) | 0.59715 (10) | 0.0612 (4) |
| P1 | 0.8353 (2) | 0.5540 (2) | 0.32229 (17) | 0.0267 (5) |
| C1 | 0.7959 (9) | 0.4354 (9) | 0.1852 (7) | 0.0306 (18) |
| H1 | 0.7703 | 0.3392 | 0.2060 | 0.037* |
| C2 | 0.6642 (9) | 0.4454 (10) | 0.0766 (7) | 0.038 (2) |
| H2A | 0.5763 | 0.4378 | 0.0990 | 0.045* |
| H2B | 0.6870 | 0.5358 | 0.0468 | 0.045* |
| C3 | 0.6343 (10) | 0.3264 (10) | -0.0199 (8) | 0.044 (2) |
| H3A | 0.6021 | 0.2366 | 0.0078 | 0.053* |
| H3B | 0.5546 | 0.3355 | -0.0903 | 0.053* |
| C4 | 0.7702 (10) | 0.3287 (10) | -0.0518 (8) | 0.045 (2) |
| H4A | 0.7945 | 0.4131 | -0.0893 | 0.054* |
| H4B | 0.7488 | 0.2479 | -0.1090 | 0.054* |
| C5 | 0.9032 (10) | 0.3258 (9) | 0.0551 (8) | 0.038 (2) |
| H5A | 0.9903 | 0.3344 | 0.0315 | 0.046* |
| H5B | 0.8843 | 0.2369 | 0.0879 | 0.046* |
| C6 | 0.9318 (9) | 0.4476 (9) | 0.1490 (7) | 0.0362 (19) |
| H6A | 1.0167 | 0.4456 | 0.2180 | 0.043* |
| H6B | 0.9546 | 0.5366 | 0.1171 | 0.043* |
| C7 | 0.9148 (9) | 0.7385 (9) | 0.2981 (7) | 0.0330 (18) |
| H7 | 0.9069 | 0.7981 | 0.3616 | 0.040* |
| C8 | 0.8334 (10) | 0.7900 (9) | 0.1839 (8) | 0.044 (2) |
| H8A | 0.8407 | 0.7375 | 0.1171 | 0.052* |
| H8B | 0.7288 | 0.7724 | 0.1751 | 0.052* |
| C9 | 0.8963 (11) | 0.9454 (10) | 0.1819 (10) | 0.056 (3) |
| H9A | 0.8471 | 0.9723 | 0.1048 | 0.067* |

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|------|-------------|-------------|-------------|-------------|
| H9B | 0.8761 | 0.9987 | 0.2415 | 0.067* |
| C10 | 1.0617 (11) | 0.9819 (11) | 0.2059 (10) | 0.057 (3) |
| H10A | 1.0808 | 0.9410 | 0.1396 | 0.068* |
| H10B | 1.0993 | 1.0835 | 0.2124 | 0.068* |
| C11 | 1.1426 (10) | 0.9292 (10) | 0.3177 (10) | 0.054 (3) |
| H11A | 1.1345 | 0.9794 | 0.3853 | 0.064* |
| H11B | 1.2474 | 0.9482 | 0.3268 | 0.064* |
| C12 | 1.0801 (9) | 0.7721 (9) | 0.3166 (8) | 0.039 (2) |
| H12A | 1.1322 | 0.7419 | 0.3914 | 0.047* |
| H12B | 1.0957 | 0.7210 | 0.2531 | 0.047* |
| C13 | 0.6373 (9) | 0.2210 (10) | 0.3590 (8) | 0.043 (2) |
| H13 | 0.7063 | 0.2713 | 0.4296 | 0.052* |
| C14 | 0.6340 (11) | 0.0838 (10) | 0.3309 (9) | 0.048 (2) |
| H14 | 0.7007 | 0.0422 | 0.3832 | 0.058* |
| C15 | 0.5362 (12) | 0.0082 (11) | 0.2291 (10) | 0.055 (3) |
| H15 | 0.5373 | -0.0841 | 0.2107 | 0.066* |
| C16 | 0.4362 (12) | 0.0670 (11) | 0.1533 (9) | 0.056 (3) |
| H16 | 0.3689 | 0.0148 | 0.0829 | 0.067* |
| C17 | 0.4337 (10) | 0.2035 (10) | 0.1801 (8) | 0.047 (2) |
| H17 | 0.3618 | 0.2412 | 0.1292 | 0.056* |
| C18 | 0.5374 (9) | 0.2858 (9) | 0.2823 (7) | 0.0338 (19) |
| C19 | 0.5333 (9) | 0.4319 (9) | 0.3139 (7) | 0.0324 (18) |
| C20 | 0.4018 (9) | 0.4472 (10) | 0.3274 (8) | 0.043 (2) |
| H20 | 0.3219 | 0.3678 | 0.3114 | 0.051* |
| C21 | 0.3852 (10) | 0.5738 (11) | 0.3630 (8) | 0.047 (2) |
| H21 | 0.2967 | 0.5795 | 0.3733 | 0.056* |
| C22 | 0.4983 (11) | 0.6902 (11) | 0.3831 (8) | 0.046 (2) |
| H22 | 0.4871 | 0.7770 | 0.4056 | 0.056* |
| C23 | 0.6325 (10) | 0.6814 (10) | 0.3703 (8) | 0.040 (2) |
| H23 | 0.7104 | 0.7626 | 0.3862 | 0.048* |
| C24 | 0.6521 (9) | 0.5536 (9) | 0.3340 (7) | 0.0306 (18) |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|------------|------------|------------|
| Pd1 | 0.0260 (5) | 0.0262 (5) | 0.0248 (5) | 0.0099 (4) | 0.0083 (3) | 0.0043 (3) |
| Br1 | 0.0626 (7) | 0.0682 (8) | 0.0511 (7) | 0.0210 (6) | 0.0141 (5) | 0.0072 (5) |
| P1 | 0.0255 (10) | 0.0281 (11) | 0.0275 (10) | 0.0093 (8) | 0.0084 (8) | 0.0049 (8) |
| C1 | 0.032 (4) | 0.035 (5) | 0.031 (4) | 0.014 (4) | 0.014 (3) | 0.007 (3) |
| C2 | 0.032 (4) | 0.046 (5) | 0.036 (5) | 0.015 (4) | 0.007 (4) | 0.004 (4) |
| C3 | 0.043 (5) | 0.054 (6) | 0.029 (5) | 0.009 (4) | 0.007 (4) | 0.000 (4) |
| C4 | 0.051 (6) | 0.045 (6) | 0.036 (5) | -0.001 (4) | 0.021 (4) | -0.005 (4) |
| C5 | 0.047 (5) | 0.036 (5) | 0.043 (5) | 0.015 (4) | 0.028 (4) | 0.004 (4) |
| C6 | 0.038 (5) | 0.038 (5) | 0.034 (4) | 0.012 (4) | 0.013 (4) | 0.005 (4) |
| C7 | 0.032 (4) | 0.035 (5) | 0.036 (5) | 0.014 (4) | 0.012 (4) | 0.007 (4) |
| C8 | 0.040 (5) | 0.034 (5) | 0.056 (6) | 0.010 (4) | 0.013 (4) | 0.020 (4) |
| C9 | 0.047 (6) | 0.039 (6) | 0.077 (7) | 0.012 (5) | 0.013 (5) | 0.024 (5) |
| C10 | 0.056 (6) | 0.041 (6) | 0.072 (7) | 0.007 (5) | 0.023 (6) | 0.019 (5) |

| | | | | | | |
|-----|-----------|-----------|-----------|------------|-----------|------------|
| C11 | 0.033 (5) | 0.050 (6) | 0.073 (7) | 0.002 (4) | 0.019 (5) | 0.013 (5) |
| C12 | 0.031 (4) | 0.037 (5) | 0.051 (5) | 0.010 (4) | 0.015 (4) | 0.014 (4) |
| C13 | 0.031 (5) | 0.059 (6) | 0.039 (5) | 0.016 (4) | 0.008 (4) | 0.010 (4) |
| C14 | 0.052 (6) | 0.037 (6) | 0.059 (6) | 0.020 (5) | 0.017 (5) | 0.013 (5) |
| C15 | 0.059 (6) | 0.032 (6) | 0.068 (7) | 0.005 (5) | 0.021 (6) | -0.002 (5) |
| C16 | 0.055 (6) | 0.046 (6) | 0.052 (6) | -0.005 (5) | 0.011 (5) | -0.004 (5) |
| C17 | 0.037 (5) | 0.045 (6) | 0.044 (5) | 0.001 (4) | 0.002 (4) | 0.007 (4) |
| C18 | 0.030 (4) | 0.031 (5) | 0.039 (5) | 0.003 (3) | 0.015 (4) | 0.003 (4) |
| C19 | 0.029 (4) | 0.034 (5) | 0.035 (4) | 0.010 (3) | 0.011 (3) | 0.004 (4) |
| C20 | 0.027 (4) | 0.048 (6) | 0.052 (6) | 0.010 (4) | 0.012 (4) | 0.007 (4) |
| C21 | 0.030 (5) | 0.067 (7) | 0.053 (6) | 0.023 (5) | 0.018 (4) | 0.009 (5) |
| C22 | 0.055 (6) | 0.051 (6) | 0.051 (6) | 0.034 (5) | 0.026 (5) | 0.012 (5) |
| C23 | 0.038 (5) | 0.040 (5) | 0.047 (5) | 0.015 (4) | 0.019 (4) | 0.005 (4) |
| C24 | 0.033 (4) | 0.034 (5) | 0.029 (4) | 0.014 (4) | 0.012 (3) | 0.006 (3) |

Geometric parameters (Å, °)

| | | | |
|----------------------|------------|----------|------------|
| Pd1—P1 ⁱ | 2.380 (2) | C9—H9B | 0.9700 |
| Pd1—P1 | 2.380 (2) | C10—C11 | 1.495 (14) |
| Pd1—Br1 ⁱ | 2.515 (2) | C10—H10A | 0.9700 |
| Pd1—Br1 | 2.515 (2) | C10—H10B | 0.9700 |
| P1—C24 | 1.848 (8) | C11—C12 | 1.518 (13) |
| P1—C1 | 1.862 (8) | C11—H11A | 0.9700 |
| P1—C7 | 1.866 (8) | C11—H11B | 0.9700 |
| C1—C6 | 1.510 (11) | C12—H12A | 0.9700 |
| C1—C2 | 1.533 (11) | C12—H12B | 0.9700 |
| C1—H1 | 0.9800 | C13—C14 | 1.371 (13) |
| C2—C3 | 1.527 (12) | C13—C18 | 1.398 (12) |
| C2—H2A | 0.9700 | C13—H13 | 0.9300 |
| C2—H2B | 0.9700 | C14—C15 | 1.347 (14) |
| C3—C4 | 1.496 (12) | C14—H14 | 0.9300 |
| C3—H3A | 0.9700 | C15—C16 | 1.358 (14) |
| C3—H3B | 0.9700 | C15—H15 | 0.9300 |
| C4—C5 | 1.511 (12) | C16—C17 | 1.378 (14) |
| C4—H4A | 0.9700 | C16—H16 | 0.9300 |
| C4—H4B | 0.9700 | C17—C18 | 1.391 (12) |
| C5—C6 | 1.527 (11) | C17—H17 | 0.9300 |
| C5—H5A | 0.9700 | C18—C19 | 1.488 (12) |
| C5—H5B | 0.9700 | C19—C20 | 1.393 (11) |
| C6—H6A | 0.9700 | C19—C24 | 1.410 (11) |
| C6—H6B | 0.9700 | C20—C21 | 1.367 (13) |
| C7—C8 | 1.512 (11) | C20—H20 | 0.9300 |
| C7—C12 | 1.518 (11) | C21—C22 | 1.345 (14) |
| C7—H7 | 0.9800 | C21—H21 | 0.9300 |
| C8—C9 | 1.509 (12) | C22—C23 | 1.396 (12) |
| C8—H8A | 0.9700 | C22—H22 | 0.9300 |
| C8—H8B | 0.9700 | C23—C24 | 1.394 (12) |
| C9—C10 | 1.505 (14) | C23—H23 | 0.9300 |
| C9—H9A | 0.9700 | | |

supplementary materials

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|---------------------------------------|-------------|---------------|------------|
| P1 ⁱ —Pd1—P1 | 180.0 | C10—C9—C8 | 111.9 (8) |
| P1 ⁱ —Pd1—Br1 ⁱ | 85.15 (7) | C10—C9—H9A | 109.2 |
| P1—Pd1—Br1 ⁱ | 94.85 (7) | C8—C9—H9A | 109.2 |
| P1 ⁱ —Pd1—Br1 | 94.85 (7) | C10—C9—H9B | 109.2 |
| P1—Pd1—Br1 | 85.15 (7) | C8—C9—H9B | 109.2 |
| Br1 ⁱ —Pd1—Br1 | 180.000 (2) | H9A—C9—H9B | 107.9 |
| C24—P1—C1 | 106.1 (4) | C11—C10—C9 | 111.8 (8) |
| C24—P1—C7 | 104.9 (4) | C11—C10—H10A | 109.2 |
| C1—P1—C7 | 108.6 (4) | C9—C10—H10A | 109.2 |
| C24—P1—Pd1 | 112.3 (3) | C11—C10—H10B | 109.2 |
| C1—P1—Pd1 | 115.8 (3) | C9—C10—H10B | 109.2 |
| C7—P1—Pd1 | 108.5 (3) | H10A—C10—H10B | 107.9 |
| C6—C1—C2 | 109.2 (7) | C10—C11—C12 | 111.6 (8) |
| C6—C1—P1 | 112.5 (6) | C10—C11—H11A | 109.3 |
| C2—C1—P1 | 116.7 (5) | C12—C11—H11A | 109.3 |
| C6—C1—H1 | 105.9 | C10—C11—H11B | 109.3 |
| C2—C1—H1 | 105.9 | C12—C11—H11B | 109.3 |
| P1—C1—H1 | 105.9 | H11A—C11—H11B | 108.0 |
| C3—C2—C1 | 109.1 (7) | C11—C12—C7 | 110.5 (7) |
| C3—C2—H2A | 109.9 | C11—C12—H12A | 109.6 |
| C1—C2—H2A | 109.9 | C7—C12—H12A | 109.6 |
| C3—C2—H2B | 109.9 | C11—C12—H12B | 109.6 |
| C1—C2—H2B | 109.9 | C7—C12—H12B | 109.6 |
| H2A—C2—H2B | 108.3 | H12A—C12—H12B | 108.1 |
| C4—C3—C2 | 111.7 (7) | C14—C13—C18 | 120.4 (9) |
| C4—C3—H3A | 109.3 | C14—C13—H13 | 119.8 |
| C2—C3—H3A | 109.3 | C18—C13—H13 | 119.8 |
| C4—C3—H3B | 109.3 | C15—C14—C13 | 121.4 (9) |
| C2—C3—H3B | 109.3 | C15—C14—H14 | 119.3 |
| H3A—C3—H3B | 107.9 | C13—C14—H14 | 119.3 |
| C3—C4—C5 | 112.5 (7) | C14—C15—C16 | 119.8 (10) |
| C3—C4—H4A | 109.1 | C14—C15—H15 | 120.1 |
| C5—C4—H4A | 109.1 | C16—C15—H15 | 120.1 |
| C3—C4—H4B | 109.1 | C15—C16—C17 | 120.4 (10) |
| C5—C4—H4B | 109.1 | C15—C16—H16 | 119.8 |
| H4A—C4—H4B | 107.8 | C17—C16—H16 | 119.8 |
| C4—C5—C6 | 109.5 (7) | C16—C17—C18 | 120.9 (9) |
| C4—C5—H5A | 109.8 | C16—C17—H17 | 119.6 |
| C6—C5—H5A | 109.8 | C18—C17—H17 | 119.6 |
| C4—C5—H5B | 109.8 | C17—C18—C13 | 117.0 (8) |
| C6—C5—H5B | 109.8 | C17—C18—C19 | 121.4 (8) |
| H5A—C5—H5B | 108.2 | C13—C18—C19 | 121.4 (8) |
| C1—C6—C5 | 110.0 (7) | C20—C19—C24 | 118.2 (8) |
| C1—C6—H6A | 109.7 | C20—C19—C18 | 116.4 (7) |
| C5—C6—H6A | 109.7 | C24—C19—C18 | 125.4 (7) |
| C1—C6—H6B | 109.7 | C21—C20—C19 | 122.8 (9) |
| C5—C6—H6B | 109.7 | C21—C20—H20 | 118.6 |
| H6A—C6—H6B | 108.2 | C19—C20—H20 | 118.6 |

| | | | |
|------------------------------|------------|-----------------|------------|
| C8—C7—C12 | 109.9 (7) | C22—C21—C20 | 119.2 (8) |
| C8—C7—P1 | 117.0 (6) | C22—C21—H21 | 120.4 |
| C12—C7—P1 | 113.5 (5) | C20—C21—H21 | 120.4 |
| C8—C7—H7 | 105.1 | C21—C22—C23 | 120.5 (9) |
| C12—C7—H7 | 105.1 | C21—C22—H22 | 119.8 |
| P1—C7—H7 | 105.1 | C23—C22—H22 | 119.8 |
| C9—C8—C7 | 111.9 (8) | C24—C23—C22 | 121.3 (9) |
| C9—C8—H8A | 109.2 | C24—C23—H23 | 119.3 |
| C7—C8—H8A | 109.2 | C22—C23—H23 | 119.3 |
| C9—C8—H8B | 109.2 | C23—C24—C19 | 117.9 (7) |
| C7—C8—H8B | 109.2 | C23—C24—P1 | 117.6 (6) |
| H8A—C8—H8B | 107.9 | C19—C24—P1 | 124.5 (6) |
| P1 ⁱ —Pd1—P1—C24 | 103 (35) | C9—C10—C11—C12 | 54.5 (12) |
| Br1 ⁱ —Pd1—P1—C24 | 120.1 (3) | C10—C11—C12—C7 | -57.0 (11) |
| Br1—Pd1—P1—C24 | -59.9 (3) | C8—C7—C12—C11 | 57.1 (10) |
| P1 ⁱ —Pd1—P1—C1 | -20 (33) | P1—C7—C12—C11 | -169.7 (7) |
| Br1 ⁱ —Pd1—P1—C1 | -2.1 (3) | C18—C13—C14—C15 | 0.5 (15) |
| Br1—Pd1—P1—C1 | 177.9 (3) | C13—C14—C15—C16 | -1.5 (16) |
| P1 ⁱ —Pd1—P1—C7 | -142 (33) | C14—C15—C16—C17 | -0.2 (16) |
| Br1 ⁱ —Pd1—P1—C7 | -124.4 (3) | C15—C16—C17—C18 | 2.9 (15) |
| Br1—Pd1—P1—C7 | 55.6 (3) | C16—C17—C18—C13 | -3.7 (14) |
| C24—P1—C1—C6 | 168.1 (6) | C16—C17—C18—C19 | -178.3 (9) |
| C7—P1—C1—C6 | 55.8 (6) | C14—C13—C18—C17 | 2.1 (13) |
| Pd1—P1—C1—C6 | -66.5 (6) | C14—C13—C18—C19 | 176.6 (8) |
| C24—P1—C1—C2 | 40.7 (7) | C17—C18—C19—C20 | 57.9 (11) |
| C7—P1—C1—C2 | -71.6 (7) | C13—C18—C19—C20 | -116.4 (9) |
| Pd1—P1—C1—C2 | 166.1 (5) | C17—C18—C19—C24 | -123.8 (9) |
| C6—C1—C2—C3 | 59.6 (9) | C13—C18—C19—C24 | 61.9 (12) |
| P1—C1—C2—C3 | -171.5 (6) | C24—C19—C20—C21 | -2.3 (13) |
| C1—C2—C3—C4 | -55.9 (10) | C18—C19—C20—C21 | 176.1 (8) |
| C2—C3—C4—C5 | 54.7 (11) | C19—C20—C21—C22 | 1.9 (14) |
| C3—C4—C5—C6 | -55.3 (10) | C20—C21—C22—C23 | -1.4 (14) |
| C2—C1—C6—C5 | -61.9 (9) | C21—C22—C23—C24 | 1.5 (14) |
| P1—C1—C6—C5 | 166.9 (6) | C22—C23—C24—C19 | -1.8 (12) |
| C4—C5—C6—C1 | 58.9 (9) | C22—C23—C24—P1 | -179.1 (7) |
| C24—P1—C7—C8 | -64.7 (7) | C20—C19—C24—C23 | 2.2 (12) |
| C1—P1—C7—C8 | 48.4 (7) | C18—C19—C24—C23 | -176.0 (8) |
| Pd1—P1—C7—C8 | 175.1 (6) | C20—C19—C24—P1 | 179.2 (6) |
| C24—P1—C7—C12 | 165.6 (6) | C18—C19—C24—P1 | 1.0 (12) |
| C1—P1—C7—C12 | -81.3 (7) | C1—P1—C24—C23 | -139.6 (6) |
| Pd1—P1—C7—C12 | 45.3 (7) | C7—P1—C24—C23 | -24.7 (7) |
| C12—C7—C8—C9 | -56.0 (10) | Pd1—P1—C24—C23 | 93.0 (6) |
| P1—C7—C8—C9 | 172.6 (7) | C1—P1—C24—C19 | 43.4 (8) |
| C7—C8—C9—C10 | 54.0 (12) | C7—P1—C24—C19 | 158.3 (7) |
| C8—C9—C10—C11 | -52.8 (13) | Pd1—P1—C24—C19 | -84.1 (7) |

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

Fig. 1

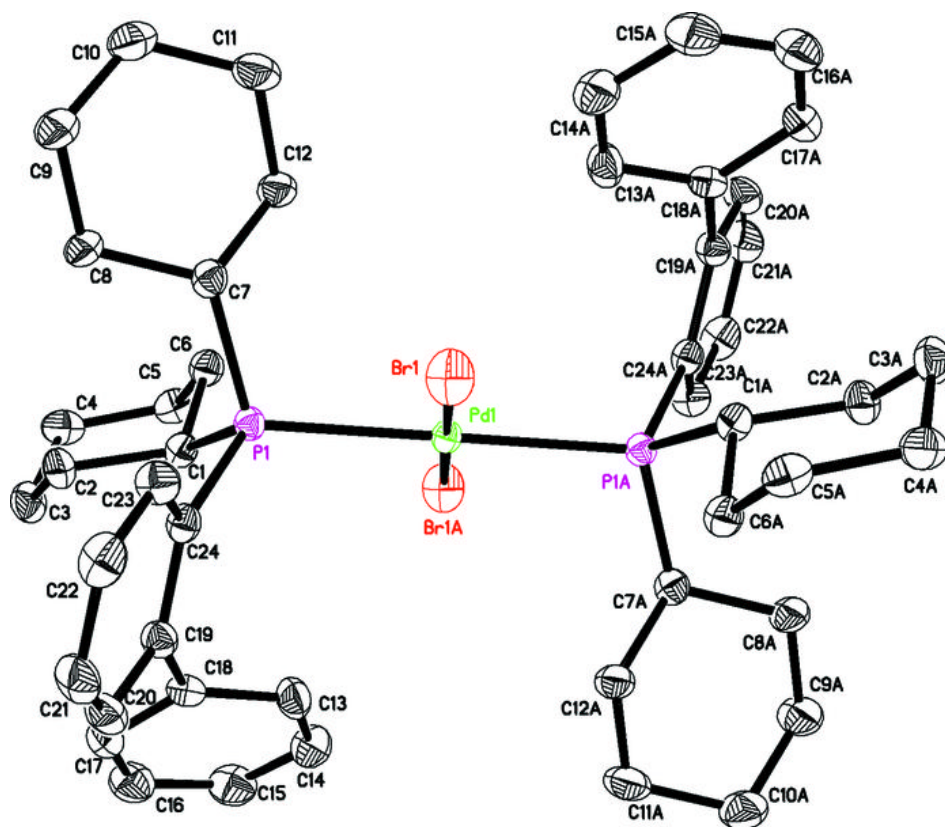


Fig. 2

