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# Dibromidobis[1-(2,4,6-trimethylphenyl)-1,4,5,6-tetrahydropyrimidine-$\kappa N^3$]-palladium(II)

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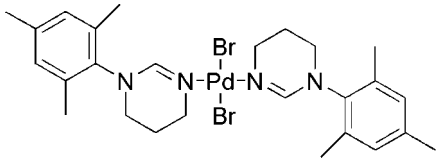
Key indicators: single-crystal X-ray study; $T$ = 293 K; mean $\sigma$(C–C) = 0.008 Å; $R$ factor = 0.045; $wR$ factor = 0.114; data-to-parameter ratio = 15.5.

In the title complex, $[PdBr_2(C_{13}H_{18}N_2)_2]$, the $Pd^{II}$ atom is situated on an inversion center. The tetrahydropyrimidine group of the *N*-(2,4,6-trimethylphenyl)-1,4,5,6-tetrahydropyrimidine ligand is twisted from the square ($PdN_2Br_2$) coordination plane with a C—N—Pd—Br torsion angle of 81.8 (4)°; this is different from the angle of 43.47 (14)°, reported in a closely related structure, dichloridobis(1-methyl-1,4,5,6-tetrahydropyrimidine)palladium(II).

## Related literature

For the related structure, dichlorobis(1-methyl-1,4,5,6-tetrahydropyrimidine)palladium(II), see: Chang & Lee (2007).




## Experimental

### Crystal data

$[PdBr_2(C_{13}H_{18}N_2)_2]$
$M_r$ = 670.81
Monoclinic, $P2_1/c$
$a$ = 7.1348 (14) Å
$b$ = 21.308 (4) Å
$c$ = 8.9704 (18) Å
$\beta$ = 94.60 (3)°
$V$ = 1359.4 (5) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 3.64 mm$^{-1}$
$T$ = 293 K
0.20 × 0.20 × 0.20 mm

### Data collection

Rigaku Saturn 724 CCD area-detector diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2006)
$T_{min}$ = 0.530, $T_{max}$ = 0.530
6809 measured reflections
2393 independent reflections
1931 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.034

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.114
$S$ = 1.08
2393 reflections
154 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.76 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.50 e Å$^{-3}$

Data collection: *CrystalClear* (Rigaku/MSC, 2006); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2226).

## References

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Rigaku/MSC (2006). *CrystalClear*. Rigaku/MSC Inc., Tokyo, Japan.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

# supporting information

*Acta Cryst.* (2011). E**67**, m62 [https://doi.org/10.1107/S1600536810050968]

## Dibromidobis[1-(2,4,6-trimethylphenyl)-1,4,5,6-tetrahydropyrimidine-$\kappa N^3$]palladium(II)

**Pu Mao, Xiujun Liu, Liangru Yang, Jinwei Yuan and Maoping Song**

### S1. Comment

Our group is interested in the preparation of new N-heterocyclic carbene (NHC) ligands based on substituted 1,4,5,6-tetrahydropyrimidine and their palladium complexes. In the course of preparing the palladium complex of a bidentate NHC ligand, we observed that the reaction of the corresponding tetrahydropyrimidine salt and $Pd(OAc)_2$, unexpectedly, under unoptimized reaction conditions, afforded the title compound.

In the title compound the Pd atom is situated on a center of inversion (Fig. 1). The organic ligands twist away from the square (PdN2Br2) coordination plane with a C1—N1—Pd1—Br1 torsion angle of 81.8 (4)°. The corresponding angle in the closely related structure, dichlorobis(1-methyl-1,4,5,6-tetrahydropyrimidine)palladium(II) [Chang & Lee, 2007], is 43.47 (14)°.

### S2. Experimental

$Pd(OAc)_2$ (101 mg, 0.45 mmol) was added to a solution of N,N-methylene-N′,N′-bis-2,4,6-trimethylphenyl-1,4,5,6-tetrahydropyrimidine (260 mg, 0.45 mmol) in DMSO (3 ml). The mixture was then heated at 333 K for 5 h. After cooling, the solvent was removed completely under vacuum. The residue was then dissolved in $CHCl_3$, and filtered. Evaporation of the filtrate afforded an orange solid (yield 200 mg, 66%). Crystals of the title complex, suitable for structural analysis, were obtained by vapor diffusion of diethyl ether into an acetonitrile solution containing the solid.

### S3. Refinement

The hydrogen atoms were included in calculated positions and treated as riding: C-H = 0.93, 0.97 and 0.96 Å, for CH, $CH_2$ and $CH_3$ H-atoms, respectively, with $U_{iso}(H) = k \times U_{eq}(C)$, where k = 1.5 for $CH_3$ H-atoms and k = 1.2 for all other H-atoms.

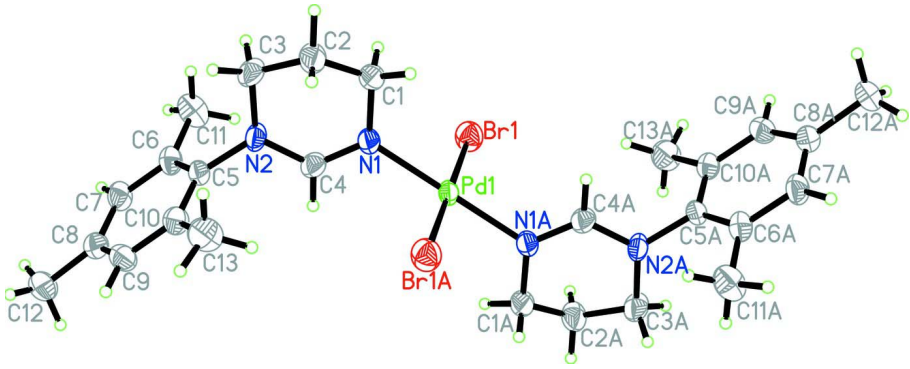


**Figure 1**

The molecular structure of the title molecule, showing 30% probability displacement ellipsoids.

## Dibromidobis[1-(2,4,6-trimethylphenyl)-1,4,5,6-tetrahydropyrimidine- $\kappa N^3$]palladium(II)

*Crystal data*

$[PdBr_2(C_{13}H_{18}N_2)_2]$
$M_r = 670.81$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 7.1348\ (14)$ Å
$b = 21.308\ (4)$ Å
$c = 8.9704\ (18)$ Å
$\beta = 94.60\ (3)°$
$V = 1359.4\ (5)$ Å$^3$
$Z = 2$
$F(000) = 672$
$D_x = 1.639$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3263 reflections
$\theta = 2.5–27.9°$
$\mu = 3.64$ mm$^{-1}$
$T = 293$ K
Prismatic, colourless
0.20 × 0.20 × 0.20 mm

*Data collection*

Rigaku CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega$ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku/MSC, 2006)
$T_{min} = 0.530$, $T_{max} = 0.530$
6809 measured reflections
2393 independent reflections
1931 reflections with $I > 2\sigma(I)$
$R_{int} = 0.034$
$\theta_{max} = 25.0°$, $\theta_{min} = 2.5°$
$h = -8\rightarrow5$
$k = -18\rightarrow25$
$l = -9\rightarrow10$

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.045$
$wR(F^2) = 0.114$
$S = 1.08$
2393 reflections
154 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.76$ e Å$^{-3}$
$\Delta\rho_{min} = -0.50$ e Å$^{-3}$

*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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Pd1 | 0.5000 | 0.5000 | 0.0000 | 0.0445 (2) |
| Br1 | 0.27385 (9) | 0.45987 (3) | −0.19525 (7) | 0.0700 (2) |
| N1 | 0.3804 (6) | 0.44800 (17) | 0.1532 (5) | 0.0502 (11) |
| N2 | 0.4027 (6) | 0.36304 (16) | 0.3218 (5) | 0.0513 (11) |
| C1 | 0.2139 (8) | 0.4701 (2) | 0.2167 (6) | 0.0595 (16) |
| H1A | 0.1044 | 0.4545 | 0.1570 | 0.071* |
| H1B | 0.2111 | 0.5156 | 0.2118 | 0.071* |
| C2 | 0.2026 (10) | 0.4506 (2) | 0.3718 (7) | 0.0722 (19) |
| H2A | 0.0783 | 0.4607 | 0.4015 | 0.087* |
| H2B | 0.2929 | 0.4747 | 0.4348 | 0.087* |
| C3 | 0.2385 (8) | 0.3831 (2) | 0.3997 (7) | 0.0659 (17) |
| H3A | 0.2622 | 0.3757 | 0.5062 | 0.079* |
| H3B | 0.1290 | 0.3589 | 0.3638 | 0.079* |
| C4 | 0.4604 (8) | 0.3983 (2) | 0.2102 (6) | 0.0534 (14) |
| H4 | 0.5700 | 0.3855 | 0.1695 | 0.064* |
| C5 | 0.5155 (7) | 0.3102 (2) | 0.3749 (6) | 0.0461 (12) |
| C6 | 0.4605 (8) | 0.2496 (2) | 0.3291 (6) | 0.0541 (14) |
| C7 | 0.5759 (8) | 0.1998 (2) | 0.3757 (6) | 0.0586 (15) |
| H7 | 0.5389 | 0.1592 | 0.3491 | 0.070* |
| C8 | 0.7456 (8) | 0.2088 (3) | 0.4614 (7) | 0.0631 (16) |
| C9 | 0.7929 (8) | 0.2699 (2) | 0.5081 (7) | 0.0628 (15) |
| H9 | 0.9027 | 0.2766 | 0.5691 | 0.075* |
| C10 | 0.6784 (8) | 0.3208 (2) | 0.4650 (7) | 0.0561 (14) |
| C11 | 0.2792 (9) | 0.2376 (3) | 0.2354 (7) | 0.0757 (18) |
| H11A | 0.1756 | 0.2527 | 0.2871 | 0.114* |
| H11B | 0.2649 | 0.1933 | 0.2180 | 0.114* |
| H11C | 0.2817 | 0.2590 | 0.1415 | 0.114* |
| C12 | 0.8751 (9) | 0.1538 (3) | 0.5026 (9) | 0.087 (2) |
| H12A | 0.9802 | 0.1548 | 0.4423 | 0.130* |
| H12B | 0.8074 | 0.1152 | 0.4855 | 0.130* |
| H12C | 0.9194 | 0.1567 | 0.6063 | 0.130* |
| C13 | 0.7335 (9) | 0.3853 (3) | 0.5220 (8) | 0.081 (2) |
| H13A | 0.7528 | 0.4123 | 0.4390 | 0.122* |
| H13B | 0.8477 | 0.3826 | 0.5861 | 0.122* |
| H13C | 0.6352 | 0.4021 | 0.5773 | 0.122* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Pd1 | 0.0507 (4) | 0.0410 (3) | 0.0427 (4) | 0.0089 (2) | 0.0098 (3) | 0.0122 (2) |
| Br1 | 0.0733 (5) | 0.0738 (4) | 0.0615 (5) | −0.0017 (3) | −0.0030 (3) | 0.0054 (3) |
| N1 | 0.054 (3) | 0.045 (2) | 0.053 (3) | 0.0112 (19) | 0.012 (2) | 0.0117 (19) |
| N2 | 0.066 (3) | 0.038 (2) | 0.052 (3) | 0.0043 (19) | 0.016 (2) | 0.0131 (18) |
| C1 | 0.060 (4) | 0.046 (3) | 0.075 (4) | 0.014 (2) | 0.023 (3) | 0.010 (3) |
| C2 | 0.084 (5) | 0.061 (3) | 0.076 (5) | 0.027 (3) | 0.034 (4) | 0.016 (3) |
| C3 | 0.078 (4) | 0.055 (3) | 0.070 (4) | 0.013 (3) | 0.040 (4) | 0.011 (3) |
| C4 | 0.056 (4) | 0.052 (3) | 0.054 (3) | 0.005 (2) | 0.020 (3) | 0.005 (2) |
| C5 | 0.048 (3) | 0.041 (3) | 0.049 (3) | 0.008 (2) | 0.006 (2) | 0.010 (2) |
| C6 | 0.068 (4) | 0.042 (3) | 0.053 (3) | 0.004 (2) | 0.005 (3) | 0.004 (2) |
| C7 | 0.071 (4) | 0.036 (3) | 0.069 (4) | 0.004 (2) | 0.006 (3) | 0.004 (2) |
| C8 | 0.058 (4) | 0.056 (3) | 0.077 (4) | 0.011 (3) | 0.017 (3) | 0.019 (3) |
| C9 | 0.049 (4) | 0.061 (3) | 0.077 (4) | 0.002 (3) | −0.006 (3) | 0.011 (3) |
| C10 | 0.057 (4) | 0.039 (3) | 0.073 (4) | −0.002 (2) | 0.005 (3) | 0.006 (2) |
| C11 | 0.085 (5) | 0.057 (3) | 0.081 (5) | 0.006 (3) | −0.017 (4) | −0.016 (3) |
| C12 | 0.070 (5) | 0.070 (4) | 0.121 (6) | 0.023 (3) | 0.014 (4) | 0.031 (4) |
| C13 | 0.073 (4) | 0.054 (3) | 0.115 (6) | −0.012 (3) | −0.009 (4) | −0.006 (3) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| Pd1—N1$^{i}$ | 2.008 (4) | C5—C6 | 1.402 (7) |
| Pd1—N1 | 2.008 (4) | C6—C7 | 1.386 (7) |
| Pd1—Br1$^{i}$ | 2.4391 (9) | C6—C11 | 1.507 (8) |
| Pd1—Br1 | 2.4391 (9) | C7—C8 | 1.394 (8) |
| N1—C4 | 1.289 (6) | C7—H7 | 0.9300 |
| N1—C1 | 1.437 (6) | C8—C9 | 1.401 (8) |
| N2—C4 | 1.343 (6) | C8—C12 | 1.519 (7) |
| N2—C5 | 1.442 (6) | C9—C10 | 1.394 (7) |
| N2—C3 | 1.474 (6) | C9—H9 | 0.9300 |
| C1—C2 | 1.461 (8) | C10—C13 | 1.508 (7) |
| C1—H1A | 0.9700 | C11—H11A | 0.9600 |
| C1—H1B | 0.9700 | C11—H11B | 0.9600 |
| C2—C3 | 1.478 (7) | C11—H11C | 0.9600 |
| C2—H2A | 0.9700 | C12—H12A | 0.9600 |
| C2—H2B | 0.9700 | C12—H12B | 0.9600 |
| C3—H3A | 0.9700 | C12—H12C | 0.9600 |
| C3—H3B | 0.9700 | C13—H13A | 0.9600 |
| C4—H4 | 0.9300 | C13—H13B | 0.9600 |
| C5—C10 | 1.380 (7) | C13—H13C | 0.9600 |
| | | | |
| N1$^{i}$—Pd1—N1 | 180.000 (1) | C6—C5—N2 | 119.1 (5) |
| N1$^{i}$—Pd1—Br1$^{i}$ | 90.30 (14) | C7—C6—C5 | 118.1 (5) |
| N1—Pd1—Br1$^{i}$ | 89.70 (14) | C7—C6—C11 | 120.0 (5) |
| N1$^{i}$—Pd1—Br1 | 89.70 (13) | C5—C6—C11 | 121.8 (5) |
| N1—Pd1—Br1 | 90.30 (14) | C6—C7—C8 | 122.0 (5) |

| | | | |
|---|---|---|---|
| Br1$^{i}$—Pd1—Br1 | 180.0 | C6—C7—H7 | 119.0 |
| C4—N1—C1 | 117.8 (4) | C8—C7—H7 | 119.0 |
| C4—N1—Pd1 | 121.7 (3) | C7—C8—C9 | 118.0 (5) |
| C1—N1—Pd1 | 120.0 (3) | C7—C8—C12 | 120.9 (5) |
| C4—N2—C5 | 119.1 (4) | C9—C8—C12 | 121.1 (6) |
| C4—N2—C3 | 119.6 (4) | C10—C9—C8 | 121.2 (6) |
| C5—N2—C3 | 120.9 (4) | C10—C9—H9 | 119.4 |
| N1—C1—C2 | 113.2 (4) | C8—C9—H9 | 119.4 |
| N1—C1—H1A | 108.9 | C5—C10—C9 | 118.9 (4) |
| C2—C1—H1A | 108.9 | C5—C10—C13 | 122.1 (5) |
| N1—C1—H1B | 108.9 | C9—C10—C13 | 118.9 (5) |
| C2—C1—H1B | 108.9 | C6—C11—H11A | 109.5 |
| H1A—C1—H1B | 107.7 | C6—C11—H11B | 109.5 |
| C1—C2—C3 | 114.5 (5) | H11A—C11—H11B | 109.5 |
| C1—C2—H2A | 108.6 | C6—C11—H11C | 109.5 |
| C3—C2—H2A | 108.6 | H11A—C11—H11C | 109.5 |
| C1—C2—H2B | 108.6 | H11B—C11—H11C | 109.5 |
| C3—C2—H2B | 108.6 | C8—C12—H12A | 109.5 |
| H2A—C2—H2B | 107.6 | C8—C12—H12B | 109.5 |
| N2—C3—C2 | 109.6 (4) | H12A—C12—H12B | 109.5 |
| N2—C3—H3A | 109.7 | C8—C12—H12C | 109.5 |
| C2—C3—H3A | 109.7 | H12A—C12—H12C | 109.5 |
| N2—C3—H3B | 109.7 | H12B—C12—H12C | 109.5 |
| C2—C3—H3B | 109.7 | C10—C13—H13A | 109.5 |
| H3A—C3—H3B | 108.2 | C10—C13—H13B | 109.5 |
| N1—C4—N2 | 127.1 (5) | H13A—C13—H13B | 109.5 |
| N1—C4—H4 | 116.5 | C10—C13—H13C | 109.5 |
| N2—C4—H4 | 116.5 | H13A—C13—H13C | 109.5 |
| C10—C5—C6 | 121.6 (4) | H13B—C13—H13C | 109.5 |
| C10—C5—N2 | 119.2 (4) | | |

| | | | |
|---|---|---|---|
| Br1$^{i}$—Pd1—N1—C4 | −73.6 (4) | C3—N2—C5—C6 | 85.6 (7) |
| Br1—Pd1—N1—C4 | 106.4 (4) | C10—C5—C6—C7 | −0.7 (8) |
| Br1$^{i}$—Pd1—N1—C1 | 98.2 (4) | N2—C5—C6—C7 | 177.0 (4) |
| Br1—Pd1—N1—C1 | −81.8 (4) | C10—C5—C6—C11 | 177.7 (5) |
| C4—N1—C1—C2 | 24.7 (8) | N2—C5—C6—C11 | −4.7 (8) |
| Pd1—N1—C1—C2 | −147.4 (4) | C5—C6—C7—C8 | −2.2 (8) |
| N1—C1—C2—C3 | −48.5 (8) | C11—C6—C7—C8 | 179.4 (5) |
| C4—N2—C3—C2 | −18.5 (8) | C6—C7—C8—C9 | 3.9 (8) |
| C5—N2—C3—C2 | 154.1 (5) | C6—C7—C8—C12 | −176.1 (5) |
| C1—C2—C3—N2 | 44.1 (8) | C7—C8—C9—C10 | −2.9 (9) |
| C1—N1—C4—N2 | 1.8 (9) | C12—C8—C9—C10 | 177.2 (5) |
| Pd1—N1—C4—N2 | 173.8 (4) | C6—C5—C10—C9 | 1.7 (8) |
| C5—N2—C4—N1 | −177.5 (5) | N2—C5—C10—C9 | −176.0 (5) |
| C3—N2—C4—N1 | −4.7 (9) | C6—C5—C10—C13 | −176.5 (5) |
| C4—N2—C5—C10 | 75.9 (7) | N2—C5—C10—C13 | 5.8 (8) |

| | | | |
|---|---|---|---|
| C3—N2—C5—C10 | −96.7 (6) | C8—C9—C10—C5 | 0.1 (8) |
| C4—N2—C5—C6 | −101.8 (6) | C8—C9—C10—C13 | 178.4 (6) |

Symmetry code: (i) −*x*+1, −*y*+1, −*z*.