

trans-Dichloridobis(4-methoxyaniline- κ N)palladium(II)

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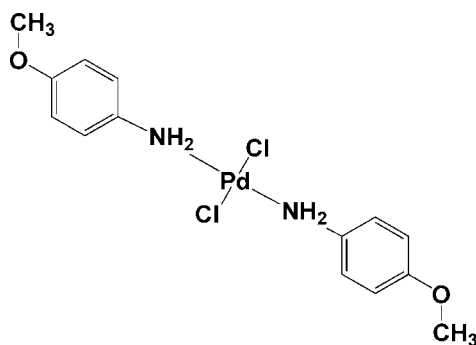
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 Key indicators: single-crystal X-ray study; $T = 173$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.023; wR factor = 0.049; data-to-parameter ratio = 14.9.

In the title compound, $[\text{PdCl}_2(\text{C}_7\text{H}_9\text{NO})_2]$, the Pd atom is situated on a crystallographic centre of inversion. The coordination environment of the Pd atom shows a slightly distorted square-planar geometry. The crystal structure exhibits weak intermolecular Pd \cdots Cl interactions, with Pd \cdots Cl distances of 3.6912 (6) Å. A chain-like arrangement of molecules realized by intermolecular N—H \cdots Cl hydrogen bonds is observed along [010].

Related literature

For catalytic activity of Pd complex compounds, see: Ojwach *et al.* (2007). For antitumoral properties of Pd compounds, see: Casas *et al.* (2008). For related structures, see: Bon *et al.* (2009); Pan *et al.* (2006).



Experimental

Crystal data

$[\text{PdCl}_2(\text{C}_7\text{H}_9\text{NO})_2]$
 $M_r = 423.60$
 Monoclinic, $P2_1/c$
 $a = 4.7333$ (1) Å
 $b = 6.0071$ (1) Å
 $c = 27.6918$ (5) Å
 $\beta = 94.806$ (1)°

$V = 784.60$ (3) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 1.53$ mm⁻¹
 $T = 173$ K
 $0.25 \times 0.08 \times 0.04$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: numerical (SADABS; Bruker, 2005)
 $T_{\min} = 0.701$, $T_{\max} = 0.941$

4837 measured reflections
 1577 independent reflections
 1326 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.024$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.049$
 $S = 1.04$
 1577 reflections
 106 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.31$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.39$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{N1}-\text{H1A}\cdots\text{Cl1}^i$	0.85 (3)	2.53 (3)	3.353 (2)	162 (2)

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT (Bruker, 2005); 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: publCIF (Westrip, 2009).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: IM2115).

References

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supporting information

Acta Cryst. (2009). E65, m673 [doi:10.1107/S1600536809018509]

***trans*-Dichloridobis(4-methoxyaniline- κ N)palladium(II)**

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S1. Comment

Coordination compounds of Pd with N-containing organic ligands attract considerable interest due to their antitumoral and catalytic activity (Casas *et al.*, 2008; Ojwach *et al.*, 2007). Similar structures with respect to the title compound differing in terms of the position of substituents at the aromatic ring were published earlier (Pan *et al.*, 2006; Bon *et al.*, 2009). The asymmetric unit of the title compound contains one-half of the molecule because Pd occupies a special position on the crystallographic centre of inversion. Pd shows a slightly distorted square-planar geometry of the coordination environment containing two chlorine atoms and two amino groups in *trans* position (Fig. 1). The crystal structure shows weak intermolecular Pd \cdots Cl interactions with Pd—Cl distances of 3.6912 (6) Å. A chain-like arrangement of molecules realized by weak N—H \cdots Cl hydrogen bonds is observed along *010* direction (Fig. 2; Table 1).

S2. Experimental

The yellow plate shaped crystals of the title compound were grown by slow evaporation of 10 ml of an ethanolic solution containing a mixture of 0.01 M H₂[PdCl₄] and 4-methoxyaniline in a 1:2 molar ratio 1:2.

S3. Refinement

H atoms bonded to N were located in a difference map and refined freely. Other H atoms were positioned geometrically and refined using a riding model with C—H = 0.98 Å for CH₃ [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$] and C—H = 0.95 Å for CH [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$]

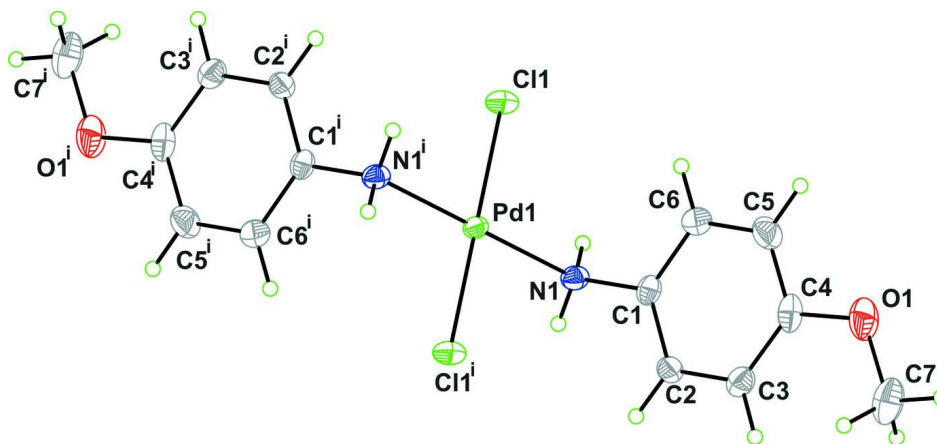
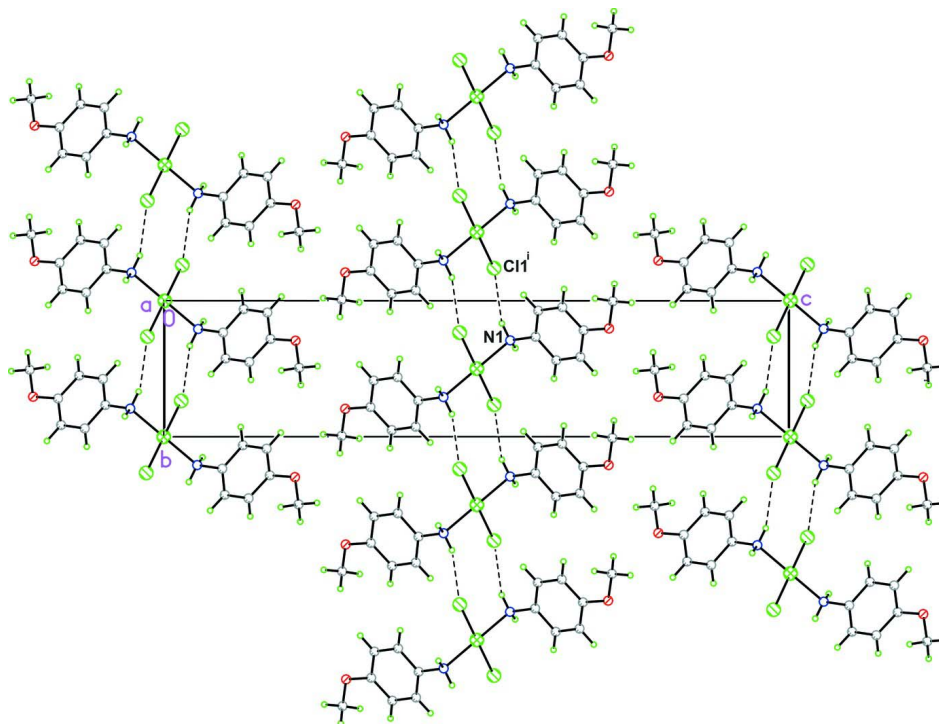


Figure 1

The title compound showing 50% probability displacement ellipsoids for the non-hydrogen atoms [Symmetry code: (i) -*x*, -*y*, -*z*].

**Figure 2**

Crystal packing of title compound, projection down the *a* axis. Dashed lines indicate hydrogen bonds [Symmetry code: (i) $x, y + 1, z$].

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Crystal data

[PdCl₂(C₇H₉NO)₂]

$M_r = 423.60$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 4.7333$ (1) Å

$b = 6.0071$ (1) Å

$c = 27.6918$ (5) Å

$\beta = 94.806$ (1)°

$V = 784.60$ (3) Å³

$Z = 2$

$F(000) = 424$

$D_x = 1.793$ Mg m⁻³

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

Cell parameters from 2257 reflections

$\theta = 3.0$ – 26.4 °

$\mu = 1.53$ mm⁻¹

$T = 173$ K

Plate, yellow

$0.25 \times 0.08 \times 0.04$ mm

Data collection

Bruker APEXII CCD

diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.26 pixels mm⁻¹

φ and ω scans

Absorption correction: numerical

(*SADABS*; Bruker, 2005)

$T_{\min} = 0.701$, $T_{\max} = 0.941$

4837 measured reflections

1577 independent reflections

1326 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.024$

$\theta_{\max} = 26.4$ °, $\theta_{\min} = 1.5$ °

$h = -2 \rightarrow 5$

$k = -7 \rightarrow 7$

$l = -34 \rightarrow 34$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.023$
 $wR(F^2) = 0.049$
 $S = 1.04$
 1577 reflections
 106 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 atoms treated by a mixture of independent
 and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0205P)^2 + 0.3835P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.31 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.39 \text{ e } \text{\AA}^{-3}$

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.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Pd1	0.0000	0.0000	0.0000	0.01570 (9)
Cl1	-0.30064 (12)	-0.26378 (11)	0.02745 (2)	0.02254 (15)
N1	-0.1173 (4)	0.2075 (4)	0.05318 (8)	0.0188 (5)
H1A	-0.162 (5)	0.332 (5)	0.0398 (9)	0.019 (7)*
H1B	-0.282 (6)	0.151 (5)	0.0615 (10)	0.029 (8)*
O1	0.6985 (4)	0.2939 (4)	0.21008 (7)	0.0347 (5)
C1	0.0967 (5)	0.2336 (4)	0.09336 (9)	0.0191 (5)
C2	0.2570 (5)	0.4248 (4)	0.09767 (9)	0.0202 (6)
H2	0.2259	0.5402	0.0744	0.024*
C3	0.4636 (5)	0.4498 (4)	0.13583 (9)	0.0231 (6)
H3	0.5742	0.5818	0.1387	0.028*
C4	0.5079 (5)	0.2813 (5)	0.16982 (9)	0.0251 (6)
C5	0.3517 (6)	0.0864 (5)	0.16452 (10)	0.0290 (6)
H5	0.3861	-0.0310	0.1872	0.035*
C6	0.1466 (6)	0.0617 (4)	0.12644 (10)	0.0256 (6)
H6	0.0401	-0.0722	0.1229	0.031*
C7	0.8612 (6)	0.4926 (6)	0.21595 (11)	0.0395 (7)
H7A	0.9714	0.5130	0.1879	0.059*
H7B	0.9903	0.4817	0.2454	0.059*
H7C	0.7340	0.6199	0.2186	0.059*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Pd1	0.01623 (13)	0.01298 (15)	0.01751 (14)	-0.00122 (12)	-0.00092 (9)	-0.00012 (13)

C11	0.0220 (3)	0.0164 (3)	0.0292 (3)	-0.0035 (3)	0.0023 (2)	0.0032 (3)
N1	0.0196 (11)	0.0148 (12)	0.0217 (12)	0.0004 (10)	-0.0008 (9)	0.0007 (10)
O1	0.0334 (10)	0.0448 (14)	0.0237 (10)	0.0059 (10)	-0.0100 (8)	-0.0007 (10)
C1	0.0191 (12)	0.0210 (14)	0.0173 (12)	0.0030 (11)	0.0029 (10)	-0.0047 (12)
C2	0.0245 (13)	0.0187 (14)	0.0177 (13)	0.0003 (11)	0.0040 (10)	0.0011 (11)
C3	0.0221 (12)	0.0239 (16)	0.0230 (14)	-0.0030 (11)	0.0009 (10)	-0.0037 (12)
C4	0.0222 (13)	0.0359 (18)	0.0171 (13)	0.0067 (13)	0.0000 (10)	-0.0053 (13)
C5	0.0380 (16)	0.0260 (15)	0.0226 (15)	0.0066 (13)	-0.0005 (12)	0.0049 (13)
C6	0.0329 (14)	0.0191 (15)	0.0245 (14)	0.0004 (12)	0.0013 (11)	-0.0004 (12)
C7	0.0320 (15)	0.055 (2)	0.0303 (15)	-0.0001 (17)	-0.0054 (12)	-0.0113 (17)

Geometric parameters (Å, °)

Pd1—N1	2.042 (2)	C2—C3	1.387 (3)
Pd1—N1 ⁱ	2.042 (2)	C2—H2	0.9500
Pd1—C11	2.3010 (6)	C3—C4	1.387 (4)
Pd1—C11 ⁱ	2.3010 (6)	C3—H3	0.9500
N1—C1	1.449 (3)	C4—C5	1.386 (4)
N1—H1A	0.85 (3)	C5—C6	1.380 (4)
N1—H1B	0.90 (3)	C5—H5	0.9500
O1—C4	1.376 (3)	C6—H6	0.9500
O1—C7	1.422 (4)	C7—H7A	0.9800
C1—C2	1.376 (4)	C7—H7B	0.9800
C1—C6	1.387 (4)	C7—H7C	0.9800
N1—Pd1—N1 ⁱ	180.00 (8)	C2—C3—C4	119.7 (2)
N1—Pd1—C11	88.23 (7)	C2—C3—H3	120.2
N1 ⁱ —Pd1—C11	91.77 (7)	C4—C3—H3	120.2
N1—Pd1—C11 ⁱ	91.77 (7)	O1—C4—C5	116.1 (3)
N1 ⁱ —Pd1—C11 ⁱ	88.23 (7)	O1—C4—C3	124.1 (3)
C11—Pd1—C11 ⁱ	180.0	C5—C4—C3	119.7 (2)
C1—N1—Pd1	113.89 (15)	C6—C5—C4	120.4 (3)
C1—N1—H1A	111.6 (18)	C6—C5—H5	119.8
Pd1—N1—H1A	107.2 (17)	C4—C5—H5	119.8
C1—N1—H1B	114.1 (17)	C5—C6—C1	119.6 (3)
Pd1—N1—H1B	104.4 (18)	C5—C6—H6	120.2
H1A—N1—H1B	105 (2)	C1—C6—H6	120.2
C4—O1—C7	116.8 (2)	O1—C7—H7A	109.5
C2—C1—C6	120.1 (2)	O1—C7—H7B	109.5
C2—C1—N1	120.1 (2)	H7A—C7—H7B	109.5
C6—C1—N1	119.7 (2)	O1—C7—H7C	109.5
C1—C2—C3	120.3 (2)	H7A—C7—H7C	109.5
C1—C2—H2	119.8	H7B—C7—H7C	109.5
C3—C2—H2	119.8		
C11—Pd1—N1—C1	105.56 (18)	C7—O1—C4—C3	-1.1 (4)
C11 ⁱ —Pd1—N1—C1	-74.44 (18)	C2—C3—C4—O1	-177.0 (2)
Pd1—N1—C1—C2	103.7 (2)	C2—C3—C4—C5	2.0 (4)

Pd1—N1—C1—C6	-73.9 (2)	O1—C4—C5—C6	177.2 (2)
C6—C1—C2—C3	-1.9 (4)	C3—C4—C5—C6	-1.9 (4)
N1—C1—C2—C3	-179.5 (2)	C4—C5—C6—C1	-0.1 (4)
C1—C2—C3—C4	-0.2 (4)	C2—C1—C6—C5	2.0 (4)
C7—O1—C4—C5	179.8 (2)	N1—C1—C6—C5	179.6 (2)

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N1—H1A \cdots Cl1 ⁱⁱ	0.85 (3)	2.53 (3)	3.353 (2)	162 (2)

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