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(*S*)-(Acetylacetonato-κ^2O,O')[2-(1-aminoethyl)-phenyl-κ^2C^1,N]palladium(II)

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The title complex, [Pd($C_8H_{10}N$)($C_5H_7O_2$)], has been prepared to be used as a building block for a co-crystallization experiment.

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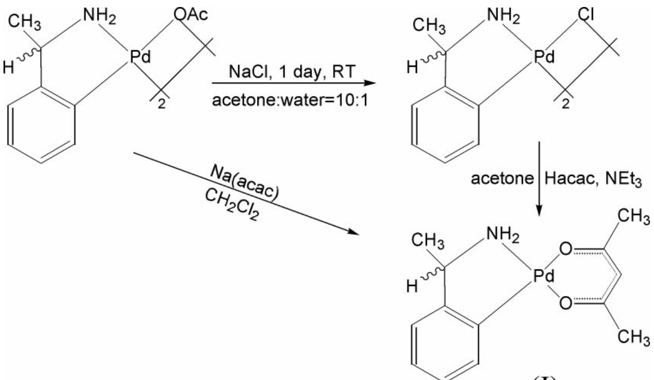
Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.003 Å
R factor = 0.020
wR factor = 0.048
Data-to-parameter ratio = 21.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Our interest in neutral inert palladium complexes appropriate for cocrystallization experiments induced us to prepare the enantiomerically pure title complex, (I).



(I)

Orthometallation results in a thermodynamically stable and kinetically inert chelate ring. A second bidentate mononegative ligand may then complete the coordination of a square-planar complex. For this purpose, we have used acetylacetone and its derivative, hexafluoroacetylacetone (Calmuschi & Englert, 2005*a*,*b*).

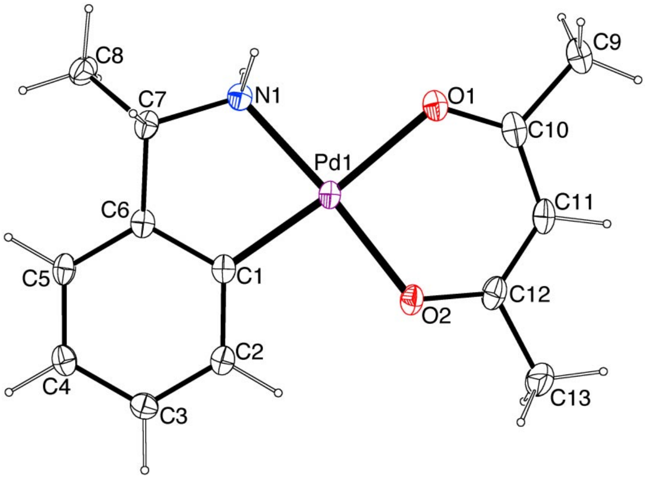


Figure 1
A plot of the molecule of (I) in the crystal structure. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown with arbitrary radii.

Single-crystal X-ray diffraction showed that crystals of the enantiomerically pure title complex and its hexafluoroacetylacetonate derivative are isomorphous in space group $P4_1$ (or $P4_3$). The metal coordination is essentially square planar. Selected bond distances and angles are listed in Table 1.

With regard to intermolecular interactions, a hydrogen bond of 2.92 Å between N1 and O1(y, $-1 - x$, $\frac{1}{4} + z$) links neighbouring molecules, forming an infinite one-dimensional chain in the c direction.

Experimental

The dinuclear intermediates in the scheme were prepared according to the procedure of Fuchita *et al.* (1997) and/or Vicente *et al.* (1997). Structural characterization of the products of the cyclopalladation reaction pathway for unsubstituted and para-substituted primary amines has already been published (Calmuschi & Englert, 2002; Calmuschi, Alesi & Englert, 2004; Calmuschi, Jonas & Englert, 2004). The monomeric complex can be prepared according to the following procedures. Method 1: a dichloromethane suspension (50 ml) containing sodium acetylacetonate (0.366 g, 3 mmol) and the chloro-bridged dimer (0.524 g, 1 mmol) were stirred at room temperature for 1 d. The sodium chloride precipitate was filtered off to leave a bright yellow solution. Concentration of the solution and addition of hexane afforded light yellow crystals in quantitative yield. Method 2: a mixture of the acetato-bridged complex (0.571 g, 1 mmol), acetylacetone (0.852 g, 8.57 mmol) and triethylamine (0.928 g, 9.05 mmol) was stirred in acetone (40 ml) for 16 h. The resulting solution was filtered and then dried *in vacuo*. The solid was suspended in water, filtered and washed with water to give a light yellow crystalline powder of the complex in quantitative yield.

Crystal data

$[Pd(C_8H_{10}N)(C_5H_7O_2)]$
$M_r = 325.68$
Tetragonal, $P4_3$
$a = 10.5872\ (11)$ Å
$c = 11.9509\ (12)$ Å
$V = 1339.6\ (2)$ Å^3
$Z = 4$
$D_x = 1.615$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 8096 reflections
$\theta = 1.9$–$28.3°$
$\mu = 1.38$ mm^{-1}
$T = 110\ (2)$ K
Prism, light yellow
0.44 × 0.12 × 0.04 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.583$, $T_{max} = 0.947$
18 582 measured reflections
3327 independent reflections
3225 reflections with $I > 2\sigma(I)$
$R_{int} = 0.028$
$\theta_{max} = 28.3°$
$h = -14 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -15 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.020$
$wR(F^2) = 0.048$
$S = 1.08$
3327 reflections
157 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -0.22$ e Å^{-3}
Absolute structure: Flack (1983), 1582 Friedel pairs
Flack parameter = −0.03 (2)

Table 1
Selected geometric parameters (Å, °).

Pd1—C1	1.970 (2)	Pd1—O1	2.1017 (17)
Pd1—O2	2.0281 (15)	O2—C12	1.280 (3)
Pd1—N1	2.0288 (19)	O1—C10	1.275 (3)
C1—Pd1—O2	92.86 (8)	C1—Pd1—O1	173.94 (9)
C1—Pd1—N1	82.28 (9)	O2—Pd1—O1	92.30 (7)
O2—Pd1—N1	172.55 (8)	N1—Pd1—O1	92.26 (10)

H atoms were placed in calculated positions and refined using a riding model, with C—H distances of 0.98 Å and N—H distances of 0.95 Å, and with $U_{iso}(H) = 1.2U_{eq}(C,N)$. Methyl groups were allowed to rotate as rigid groups. The maximum of residual electron density was close (0.86 Å) to Pd1.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97.

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