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

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The title complex, [Pd(C_8H_8N)($C_5HF_6O_2$)], has been prepared to be used as a building block for a co-crystallization experiment.

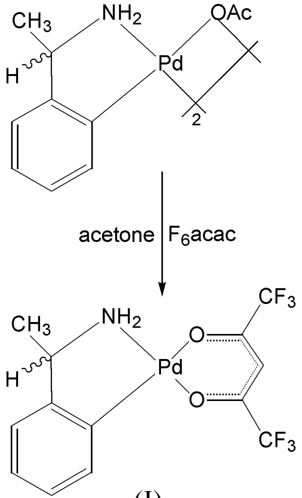
Key indicators

Single-crystal X-ray study
T = 110 K
Mean σ(C–C) = 0.007 Å
R factor = 0.037
wR factor = 0.095
Data-to-parameter ratio = 17.8

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 (Calmuschi & Englert, 2005*a*) and its derivative, hexafluoroacetylacetone (this work; also Calmuschi & Englert, 2005*b*).

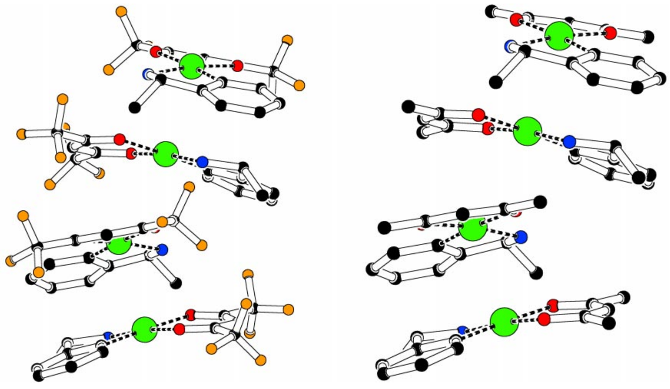


Figure 1
Packing diagrams of the unit-cell contents in (I) (left) and for its acetylacetone homologue (right), viewed along [100].

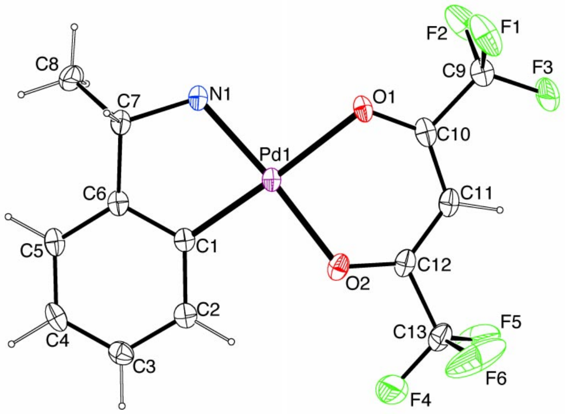


Figure 2
Plot of a molecule of (I) in the crystal structure. Displacement ellipsoids are drawn at the 30% probability level and H atoms are shown with arbitrary radii.

Single-crystal X-ray diffraction showed that crystals of the enantiomerically pure title complex and its above-mentioned homologue are isomorphous in space group $P4_1$ (or $P4_3$). Fig. 1 shows packing diagrams of both structures, viewed along [100]. The metal coordination is essentially square planar. Selected bond distances and angles are listed in Table 1. The Pd—C and C—O distances are slightly shorter in the fluorinated derivatives (this work; also Calmuschi & Englert, 2005*b*) than in the unsubstituted acetylacetonate complex (Calmuschi & Englert, 2005*a*); however, the difference is hardly significant.

With regard to intermolecular interactions, no classical hydrogen bonds were found.

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 procedure. An acetone solution (50 ml) containing hexafluoroacetylacetonate (0.445 g, 2.14 mmol) and the acetato-bridged dimer (0.571 g, 1.00 mmol) was stirred at room temperature for 16 h. The resulting solution was filtered and the filtrate was concentrated to dryness under vacuum. The resulting solid was suspended in water, filtered and washed with water to give yellow crystals in a quantitative yield.

Crystal data

$[Pd(C_8H_8N)(C_5HF_6O_2)]$
$M_r = 431.61$
Tetragonal, $P4_3$
$a = 10.951$ (5) Å
$c = 12.442$ (7) Å
$V = 1492.2$ (12) Å^3
$Z = 4$
$D_x = 1.921$ Mg m^{-3}

Mo $K\alpha$ radiation
Cell parameters from 8096 reflections
$\theta = 1.9$–$28.4°$
$\mu = 1.32$ mm^{-1}
$T = 110$ (2) K
Block, yellow
0.70 × 0.60 × 0.50 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
$T_{min} = 0.426$, $T_{max} = 0.518$
15 138 measured reflections

3715 independent reflections
3547 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 28.4°$
$h = -13 \rightarrow 14$
$k = -14 \rightarrow 14$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.095$
$S = 1.05$
3715 reflections
209 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 1.15$ e Å^{-3}
$\Delta\rho_{min} = -0.50$ e Å^{-3}
Absolute structure: Flack (1983), 1757 Friedel pairs
Flack parameter = 0.00 (4)

Table 1
Selected geometric parameters (Å, °).

Pd1—C1	1.959 (4)	Pd1—O1	2.139 (3)
Pd1—O2	2.035 (3)	O1—C10	1.261 (6)
Pd1—N1	2.035 (4)	O2—C12	1.259 (5)
C1—Pd1—O2	92.54 (15)	C1—Pd1—O1	175.99 (16)
C1—Pd1—N1	82.63 (17)	O2—Pd1—O1	90.31 (13)
O2—Pd1—N1	172.03 (16)	N1—Pd1—O1	94.22 (17)

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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