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Double cyclometallation of bis[3-(3-methoxyphenyl)-pyrazol-1-yl]methane in a bis[(acetylacetonato)-palladium(II)] complex

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.008 Å
R factor = 0.033
wR factor = 0.088
Data-to-parameter ratio = 10.2

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

The cyclometallation of aryl and pyrazolyl rings yields the title bis[(acetylacetonato)palladium(II)] complex, {μ-bis[3-(3-methoxyphenyl)pyrazol-1-yl]methane}bis[(acetylacetonato)-palladium(II)], $[Pd_2(C_5H_7O_2)_2(C_{21}H_{18}N_4O_2)]$, with five- and six-membered chelate rings. The five-membered ring PdNCCC [Pd—C 1.980 (5) Å and Pd—N 2.004 (3) Å] is planar, whereas the six-membered ring PdNNCNC [Pd—C 1.966 (5) Å and Pd—N 2.043 (4) Å] has a boat conformation. The Pd—O(acac) distances clearly demonstrate a *trans* influence, the O atoms in *trans* positions with respect to carbon forming longer Pd—O bonds than those in *trans* positions to nitrogen [Pd—O 2.097 (3) Å *versus* 2.010 (3) Å for the Pd atom in the five-membered ring and 2.054 (3) Å *versus* 1.984 (3) Å for the Pd atom in the six-membered ring].

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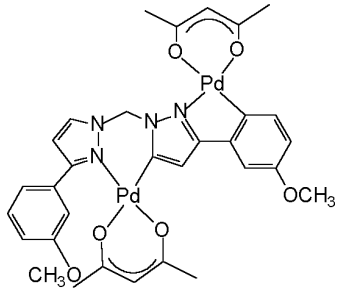

(I)

Experimental

The compound was synthesized as described by Alonso *et al.* (1994). Recrystallization from dichloromethane/hexane afforded colourless crystals suitable for X-ray analysis.

Crystal data

$[Pd_2(C_5H_7O_2)_2(C_{21}H_{18}N_4O_2)]$
M_r = 769.41
Monoclinic, $P2_1/n$
a = 14.3726 (7) Å
b = 14.6998 (11) Å
c = 15.9304 (9) Å
β = 115.156 (4)°
V = 3046.5 (3) Å^3
Z = 4
D_x = 1.678 Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 68 reflections
θ = 6.8–47.5°
μ = 9.94 mm^{-1}
T = 296 (2) K
Irregular fragment, colourless
0.35 × 0.25 × 0.15 mm

Data collection

Bruker $P4$ diffractometer
ω scans
Absorption correction: ψ scan (*XPREP*; Bruker, 2000)
T_{min} = 0.341, T_{max} = 0.779
4996 measured reflections
4011 independent reflections
3777 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 56.7°
$h = -1 \rightarrow 15$
$k = -1 \rightarrow 15$
$l = -17 \rightarrow 16$
3 standard reflections
every 97 reflections
intensity decay: none

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Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0519P)^2 + 4.3327P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$(\Delta/\sigma)_{max} = 0.006$
$wR(F^2) = 0.088$	$\Delta\rho_{max} = 1.26$ e Å^{-3}
$S = 1.07$	$\Delta\rho_{min} = -0.65$ e Å^{-3}
4011 reflections	Extinction correction: *SHELXL*97
395 parameters	Extinction coefficient: 0.00038 (4)
H-atom parameters constrained	

The H atoms were placed in idealized positions and included in the refinement in the riding model approximation with U_{iso} equal to $1.2U_{eq}$ of the carrier atom. The geometry of the diffractometer installation did not allow us to collect data with 2θ higher than 113.5°, which is a probable cause for spurious peaks in the vicinity of the heavy atoms (maximum residual density peak of 1.26 e Å^{-3} is located at 1.03 Å from the atom Pd2).

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Sheldrick, 1997*b*); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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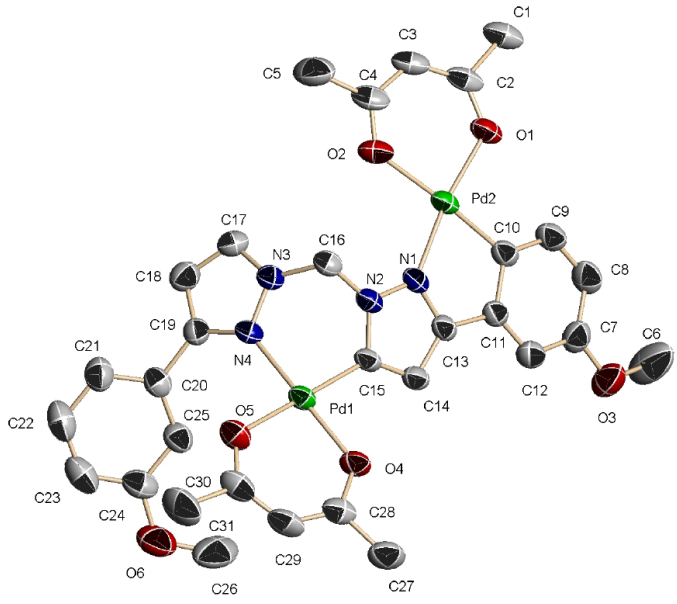

Figure 1
A view of (I) with the atomic numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

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