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#### Key indicators

Single-crystal X-ray study

$T = 298$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.013$  Å

$R$  factor = 0.050

$wR$  factor = 0.153

Data-to-parameter ratio = 15.7

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

## $\mu$ -3,3'-Bisacetylacetonato-bis[(1,10-phenanthroline)palladium(II)] bis(hexafluorophosphate) acetonitrile solvate

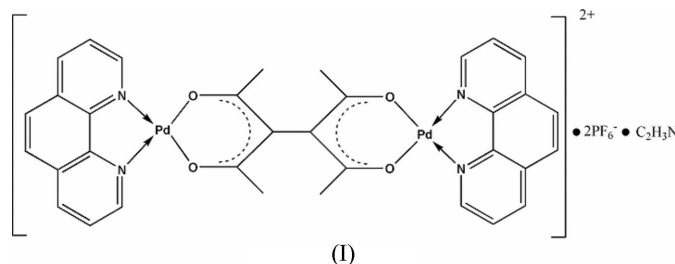
In the title complex,  $[\text{Pd}_2(\text{C}_{10}\text{H}_{12}\text{O}_4)(\text{C}_{12}\text{H}_8\text{N}_2)_2](\text{PF}_6)_2 \cdot 2\text{CH}_3\text{CN}$ , the  $\text{Pd}^{\text{II}}$  centre has a distorted *cis* square-planar geometry defined by an *O,O'*-bidentate bisacetylacetonate dianion ligand and a chelating 1,10-phenanthroline ligand. The crystal structure features electrostatic interactions between the cations and anions, intermolecular  $\pi$ - $\pi$  interactions between pairs of 1,10-phenanthroline rings, and weak hydrogen bonds involving hexafluorophosphate anions ( $\text{C}-\text{H} \cdots \text{F}$ ) and the acetonitrile solvent molecules ( $\text{C}-\text{H} \cdots \text{N}$ ).

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#### Comment

In a previous article, we reported the structure of  $\mu$ -3,3'-bisacetylacetonato-bis[(2,2'-bipyridine)palladium(II)] bis(hexafluorophosphate) acetonitrile solvate (Mei *et al.*, 2006). Here, we report the crystal structure of a new dinuclear palladium(II) complex, (I) (Fig. 1), based on 1,10-phenanthroline and bisacetylacetonate dianion ligands.

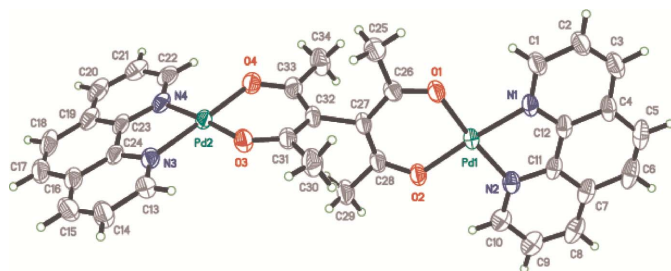


As shown in Fig. 2, the compound packs by electrostatic forces between the cations and anions,  $\pi$ - $\pi$  interactions between planar  $[\text{Pd}(\text{phen})]$  units, and weak hydrogen bonds involving hexafluorophosphate anions and acetonitrile solvent molecules. The centroid-centroid contact distance between two phenanthroline molecules is 3.356 (4) Å, and the angle between the ring-centroid vector and the normal to one of the phenanthroline ring planes is about 20°.

The non-classical hydrogen bonds that connect cations, anions and solvent acetonitrile molecules are detailed in Table 2.

#### Experimental

A mixture of (1,10-phenanthroline)dinitratopalladium(II) (41.0 mg, 0.10 mmol) and bisacetylacetonate (9.9 mg, 0.05 mmol) was dissolved in water (2 ml) and the mixture was stirred overnight. A tenfold excess of potassium hexafluorophosphate was added, which resulted in the immediate deposition of (I) as yellow microcrystals. The crystals were filtered off, washed with a minimum amount of cold water and dried under vacuum (yield 53.2 mg). Crystals appropriate



**Figure 1**  
A view of the cation of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

for data collection were obtained by the vapour diffusion of diethyl ether into a 1.0 mM solution of (I) in acetonitrile.

*Crystal data*

[Pd<sub>2</sub>(C<sub>10</sub>H<sub>12</sub>O<sub>4</sub>)(C<sub>12</sub>H<sub>8</sub>N<sub>2</sub>)<sub>2</sub>](PF<sub>6</sub>)<sub>2</sub>·  
C<sub>2</sub>H<sub>3</sub>N  
M<sub>r</sub> = 1100.40  
Orthorhombic, *Fdd2*  
a = 22.321 (5) Å  
b = 47.887 (10) Å  
c = 15.392 (3) Å  
V = 16453 (6) Å<sup>3</sup>

Z = 16  
D<sub>x</sub> = 1.777 Mg m<sup>-3</sup>  
Mo Kα radiation  
μ = 1.05 mm<sup>-1</sup>  
T = 298 (2) K  
Block, yellow  
0.45 × 0.25 × 0.20 mm

*Data collection*

Bruker SMART APEX CCD area-detector diffractometer  
φ and ω scans  
Absorption correction: multi-scan (SADABS; Bruker, 2001)  
T<sub>min</sub> = 0.740, T<sub>max</sub> = 0.810

36699 measured reflections  
8766 independent reflections  
7916 reflections with I > 2σ(I)  
R<sub>int</sub> = 0.033  
θ<sub>max</sub> = 27.0°

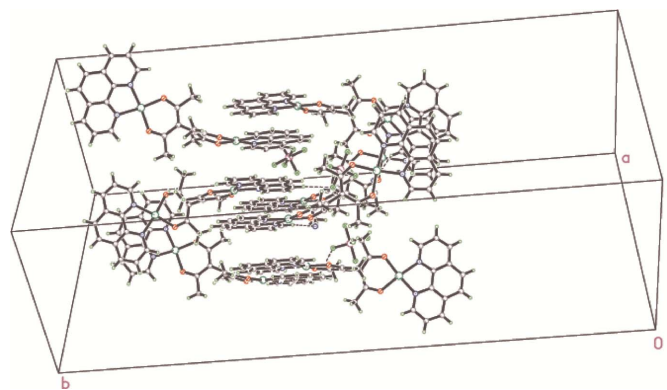
*Refinement*

Refinement on F<sup>2</sup>  
R[F<sup>2</sup> > 2σ(F<sup>2</sup>)] = 0.050  
wR(F<sup>2</sup>) = 0.153  
S = 1.02  
8766 reflections  
557 parameters  
H-atom parameters constrained

w = 1/[σ<sup>2</sup>(F<sub>o</sub><sup>2</sup>) + (0.1556P)<sup>2</sup> + 1.928P]  
where P = (F<sub>o</sub><sup>2</sup> + 2F<sub>c</sub><sup>2</sup>)/3  
(Δ/σ)<sub>max</sub> = 0.001  
Δρ<sub>max</sub> = 0.75 e Å<sup>-3</sup>  
Δρ<sub>min</sub> = -1.32 e Å<sup>-3</sup>  
Absolute structure: Flack (1983), with 4110 Friedel pairs  
Flack parameter: 0.47 (4)

**Table 1**  
Selected geometric parameters (Å, °).

Pd1—N1	2.010 (6)	Pd1—O1	1.962 (5)
Pd1—N2	2.013 (6)	Pd1—O2	1.966 (5)
Pd2—N3	2.023 (6)	Pd2—O3	1.959 (5)
Pd2—N4	1.993 (6)	Pd2—O4	1.975 (5)
N1—Pd1—N2	81.7 (3)	N3—Pd2—O4	174.6 (3)
N1—Pd1—O1	92.7 (2)	N3—Pd2—N4	81.9 (3)
N1—Pd1—O2	173.2 (2)	N4—Pd2—O3	174.6 (3)
N2—Pd1—O1	174.3 (2)	N4—Pd2—O4	93.1 (3)
N2—Pd1—O2	92.2 (2)	O1—Pd1—O2	93.5 (2)
N3—Pd2—O3	92.7 (2)	O3—Pd2—O4	92.3 (2)



**Figure 2**  
The packing of (I). Hydrogen bonds are shown as dashed lines.

**Table 2**  
Hydrogen-bond geometry (Å, °).

D—H...A	D—H	H...A	D...A	D—H...A
C3—H3A...F5 <sup>i</sup>	0.93	2.47	3.380 (12)	166
C8—H8A...F9 <sup>ii</sup>	0.93	2.20	3.102 (13)	163
C15—H15A...F3 <sup>iii</sup>	0.93	2.48	3.031 (14)	118
C20—H20A...F10 <sup>iv</sup>	0.93	2.45	3.162 (16)	133
C22—H22A...N5 <sup>v</sup>	0.93	2.56	3.375 (15)	146

Symmetry codes: (i)  $-x + \frac{1}{4}, y - \frac{1}{4}, z - \frac{1}{4}$ ; (ii)  $-x - \frac{1}{4}, y - \frac{1}{4}, z + \frac{1}{4}$ ; (iii)  $x - \frac{1}{4}, -y + \frac{3}{4}, z + \frac{1}{4}$ ; (iv)  $x - \frac{1}{4}, -y + \frac{3}{4}, z - \frac{3}{4}$ ; (v)  $-x, -y + \frac{1}{2}, z - \frac{1}{2}$ .

The value of the Flack (Flack, 1983) parameter indicates inversion twinning. The aromatic H atoms were constrained to an ideal geometry, with C—H distances of 0.93 Å and with  $U_{iso}(H) = 1.2U_{eq}(C)$ . The methyl H atoms of the bisacetylacetonate dianion were also constrained to an ideal geometry, with C—H distances of 0.96 Å and with  $U_{iso}(H) = 1.5U_{eq}(C)$ . The deepest hole in the final difference Fourier map is located 0.17 Å from Pd2.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT-Plus (Bruker, 2001); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: PLATON (Spek, 2003); software used to prepare material for publication: PLATON.

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**References**

Bruker (2001). SMART (Version 5.628), SAINT-Plus (Version 6.45) and SADABS (Version 2.10). Bruker AXS Inc., Madison, Wisconsin, USA.  
Flack, H. D. (1983). *Acta Cryst.* **A39**, 876–881.  
Mei, G.-Q., Huang, K.-L., Huang, H.-P. & Li, Y.-Z. (2006). *Acta Cryst.* **E62**, m2368–m2370.  
Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.  
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.