

$D-H \cdots A$	$D \cdots A$	$D-H \cdots A$	$D \cdots A$
O(1)—H(1) \cdots N(3 ⁱⁱ)	2.832 (4)	N(4)—H(3) \cdots O(3 ⁱ)	2.819 (5)
O(1)—H(2) \cdots O(3 ⁱⁱⁱ)	3.042 (4)	N(1)—H(4) \cdots O(4 ^{iv})	3.031 (5)

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

All H atoms were located on difference electron density maps and included as fixed isotropic scatterers (O—H 0.98–1.08, N—H 1.02–1.11 and C—H 0.91–1.15 Å), with *B* values 1.2 times those of the attached atoms at the time of their inclusion.

Data collection: *MSCI/AFSC Diffractometer Control Software* (Molecular Structure Corporation, 1988). Cell refinement: *MSCI/AFSC Diffractometer Control Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1991). Program(s) used to solve structure: *MITHRIL* (Gilmore, 1983). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including intermolecular distances involving both H and non-H atoms, and special angles involving H atoms, and least-squares-planes data have been deposited with the IUCr (Reference: BK1184). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Second Polymorph of 2,2'-Bipyridyl-(methyl)(phenyl)palladium(II)

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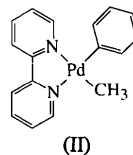
(Received 17 October 1995; accepted 30 October 1995)

Abstract

The crystal structure of a second polymorph of [Pd(CH₃)(C₆H₅)(C₁₀H₈N₂)], containing four crystallographically independent molecules, is presented and compared with the structure of the previously reported polymorph [Markies *et al.* (1994). *J. Organomet. Chem.* **482**, 191–199] containing only one crystallographically independent molecule. In both polymorphs molecules lie in infinite stacks, surrounded by six similar stacks; the main difference between the two polymorphs is about 23° in the tilt angle of the coordination plane with reference to the stacking direction.

Comment

Preliminary attempts to obtain crystals of the title compound yielded needles [modification (II)] with a large monoclinic unit cell and four molecules per asymmetric unit. Subsequent attempts under essentially the same crystallization conditions yielded better quality monoclinic plates [modification (I)] with one molecule in the asymmetric unit (Markies *et al.*, 1991, 1994). Although less accurate than the previously reported structure [modification (I)], the crystal structure of modification (II) is relevant in view of the current interest in polymorphs (Gavezzotti, 1994; Dunitz & Bernstein, 1995). Polymorphs provide information on conformational flexibility and are relevant for *ab initio* crystal structure predictions (Gavezzotti, 1994).



The four crystallographically independent molecules in (II) (Fig. 1) have very similar conformations that closely resemble the conformation found in (I). The main difference between the five molecules may be

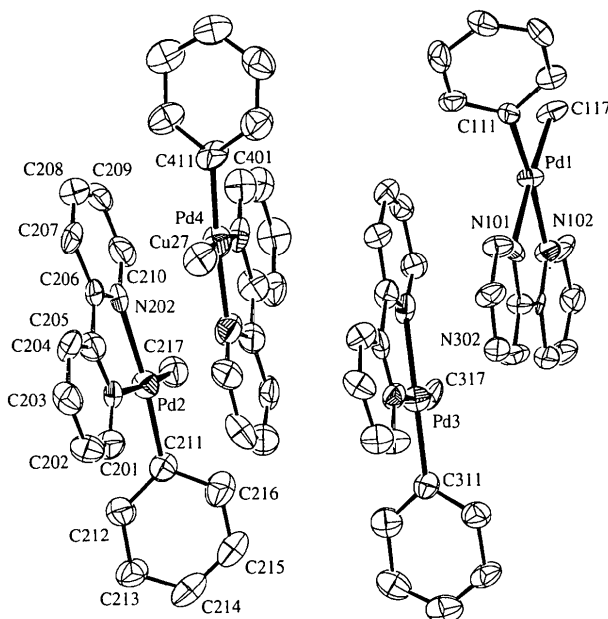


Fig. 1. Atomic displacement ellipsoid plot (30% probability level) of the four crystallographically independent molecules. H atoms have been omitted.

quantified by the dihedral angle of the phenyl ring with the coordination plane of Pd [78.7 (2)° for (I) and 70.7 (10), 65.7 (11), 75.3 (11) and 75.7 (12)° for (II)].

Molecules in (II) form infinite stacks running in the [101] direction (Fig. 2), with an interlayer distance of approximately 3.5 Å. The coordination planes are approximately perpendicular to the stacking direction with an angle ranging from 78.7 (8) to 85.9 (9)°. Each stack is surrounded by six similar stacks. A similar pattern is found in (I) where [100] is the stacking direction, the main difference being that the coordination planes are now significantly tilted [*i.e.* 58.9 (1)°] with reference to the stacking direction.

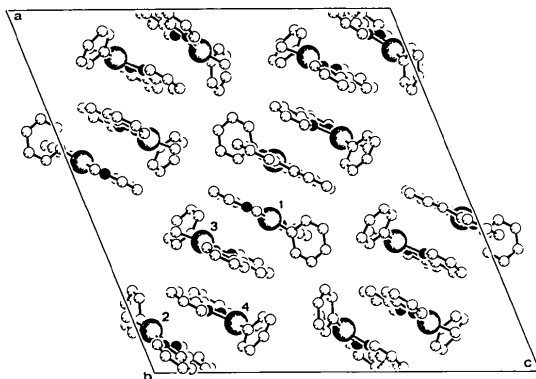


Fig. 2. Projection of the structure down the *b* axis illustrating the one-dimensional stacking of the molecules. The numbers relate to the Pd atoms.

The current structure has a higher packing coefficient (Kitaigorodskii, 1961) [69.9% for (II) and 68.7% for (I)].

Experimental

Preparation and crystallization of the title compound are described by Markies *et al.* (1994). Crystals for the diffraction experiment were obtained from acetone/pentane at 250 K.

Crystal data

[Pd(CH₃)(C₆H₅)(C₁₀H₈N₂)]
 $M_r = 354.75$
 Monoclinic
 $P2_1/c$
 $a = 25.303$ (3) Å
 $b = 9.953$ (4) Å
 $c = 25.071$ (3) Å
 $\beta = 112.08$ (1)°
 $V = 5851$ (3) Å³
 $Z = 16$

Mo $K\alpha$ radiation
 $\lambda = 0.71073$ Å
 Cell parameters from 25 reflections
 $\theta = 5.8$ – 11.9 °
 $\mu = 1.26$ mm^{−1}
 $T = 300$ K
 Needle
 $0.75 \times 0.05 \times 0.05$ mm
 Yellow

$D_x = 1.611$ Mg m^{−3}
 D_m not measured

Data collection

Enraf–Nonius CAD-4T
 rotating-anode diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 10817 measured reflections
 6631 independent reflections
 2362 observed reflections
 $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.07$
 $\theta_{\text{max}} = 21.49$ °
 $h = -26 \rightarrow 0$
 $k = -10 \rightarrow 0$
 $l = -23 \rightarrow 25$
 3 standard reflections
 frequency: 60 min
 intensity decay: 8%

Refinement

Refinement on F^2
 $R(F) = 0.069$
 $wR(F^2) = 0.19$
 $S = 1.023$
 6631 reflections
 725 parameters
 H-atom parameters not refined
 $w = 1/[\sigma^2(F_o^2) + 135.3909P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\text{max}} = -0.068$
 $\Delta\rho_{\text{max}} = 0.756$ e Å^{−3}
 $\Delta\rho_{\text{min}} = -0.555$ e Å^{−3}
 Extinction correction: none
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

	$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>U</i> _{eq}
Pd1	0.41922 (8)	0.2028 (2)	0.46274 (8)	0.0532 (8)
N101	0.4027 (8)	0.0082 (16)	0.4861 (7)	0.062 (6)
N102	0.4528 (8)	0.0773 (16)	0.4161 (8)	0.060 (7)
C101	0.3798 (10)	−0.0217 (19)	0.5238 (9)	0.062 (8)
C102	0.3735 (10)	−0.151 (2)	0.5404 (10)	0.070 (9)
C103	0.3904 (10)	−0.2525 (19)	0.5143 (10)	0.068 (8)
C104	0.4159 (11)	−0.2261 (18)	0.4748 (10)	0.074 (8)
C105	0.4201 (11)	−0.0946 (19)	0.4620 (10)	0.061 (4)
C106	0.4490 (9)	−0.0539 (19)	0.4234 (9)	0.062 (7)

C107	0.4666 (10)	−0.1499 (19)	0.3918 (10)	0.070 (8)	Pd2—N201	2.06 (2)	Pd4—N401	2.12 (2)
C108	0.4901 (11)	−0.101 (2)	0.3545 (11)	0.081 (9)	Pd2—N202	2.13 (2)	Pd4—N402	2.07 (2)
C109	0.4965 (10)	0.033 (2)	0.3485 (10)	0.074 (8)	Pd2—C211	1.94 (2)	Pd4—C411	1.97 (2)
C110	0.4748 (10)	0.120 (2)	0.3790 (10)	0.071 (8)	Pd2—C217	2.01 (3)	Pd4—C417	2.07 (3)
C111	0.3828 (9)	0.304 (2)	0.5054 (9)	0.057 (5)	N101—Pd1—N102	76.8 (7)	N301—Pd3—N302	76.3 (7)
C112	0.4140 (9)	0.376 (2)	0.5548 (9)	0.060 (7)	N101—Pd1—C111	98.0 (8)	N301—Pd3—C311	102.1 (9)
C113	0.3917 (9)	0.445 (2)	0.5905 (9)	0.068 (8)	N101—Pd1—C117	174.6 (9)	N301—Pd3—C317	174.3 (8)
C114	0.3340 (10)	0.444 (2)	0.5743 (10)	0.070 (8)	N102—Pd1—C111	174.1 (8)	N302—Pd3—C311	178.3 (8)
C115	0.3002 (9)	0.378 (3)	0.5252 (11)	0.080 (9)	N102—Pd1—C117	98.0 (8)	N302—Pd3—C317	98.6 (8)
C116	0.3246 (9)	0.309 (2)	0.4924 (9)	0.062 (7)	C111—Pd1—C117	87.2 (9)	C311—Pd3—C317	83.1 (9)
C117	0.4407 (10)	0.384 (2)	0.4373 (10)	0.064 (9)	N201—Pd2—N202	76.6 (7)	N401—Pd4—N402	77.2 (8)
Pd2	0.11468 (9)	0.8133 (2)	0.03670 (9)	0.0695 (11)	N201—Pd2—C211	97.8 (9)	N401—Pd4—C411	95.5 (9)
N201	0.0718 (8)	0.7341 (17)	0.0851 (8)	0.072 (6)	N201—Pd2—C217	176.6 (10)	N401—Pd4—C417	172.6 (10)
N202	0.0742 (8)	0.9837 (18)	0.0557 (9)	0.076 (7)	N202—Pd2—C211	173.7 (10)	N402—Pd4—C411	172.3 (10)
C201	0.0700 (11)	0.605 (2)	0.0992 (11)	0.078 (9)	N202—Pd2—C217	100.6 (10)	N402—Pd4—C417	95.8 (10)
C202	0.0411 (12)	0.558 (2)	0.1318 (11)	0.084 (10)	C211—Pd2—C217	84.8 (11)	C411—Pd4—C417	91.4 (11)
C203	0.0120 (11)	0.648 (2)	0.1513 (11)	0.086 (9)				
C204	0.0116 (10)	0.783 (2)	0.1371 (10)	0.080 (8)				
C205	0.0414 (11)	0.819 (2)	0.1045 (11)	0.073 (8)				
C206	0.0423 (10)	0.959 (2)	0.0860 (10)	0.078 (8)				
C207	0.0106 (10)	1.061 (2)	0.1002 (10)	0.081 (8)				
C208	0.0127 (11)	1.186 (2)	0.0791 (12)	0.096 (10)				
C209	0.0455 (11)	1.212 (2)	0.0474 (11)	0.092 (9)				
C210	0.0767 (11)	1.107 (2)	0.0376 (11)	0.089 (8)				
C211	0.1448 (11)	0.646 (2)	0.0199 (10)	0.070 (7)				
C212	0.1103 (10)	0.543 (3)	−0.0133 (12)	0.099 (8)				
C213	0.1297 (11)	0.417 (2)	−0.0229 (11)	0.089 (9)				
C214	0.1876 (12)	0.397 (3)	−0.0015 (13)	0.100 (10)				
C215	0.2240 (10)	0.493 (3)	0.0299 (13)	0.113 (12)				
C216	0.2030 (10)	0.615 (3)	0.0400 (11)	0.097 (9)				
C217	0.1526 (11)	0.898 (3)	−0.0126 (11)	0.081 (9)				
Pd3	0.35718 (9)	0.7702 (2)	0.26472 (8)	0.0593 (9)				
N301	0.3221 (8)	0.7175 (17)	0.3258 (8)	0.060 (6)				
N302	0.3324 (7)	0.9567 (16)	0.2885 (8)	0.061 (6)				
C301	0.3204 (11)	0.595 (2)	0.3467 (10)	0.075 (8)				
C302	0.2965 (11)	0.563 (2)	0.3856 (10)	0.078 (9)				
C303	0.2697 (11)	0.665 (2)	0.4024 (10)	0.075 (9)				
C304	0.2705 (10)	0.795 (2)	0.3820 (9)	0.066 (8)				
C305	0.2984 (9)	0.8161 (19)	0.3461 (9)	0.060 (7)				
C306	0.3046 (10)	0.9517 (19)	0.3247 (10)	0.063 (8)				
C307	0.2860 (10)	1.067 (2)	0.3442 (10)	0.069 (8)				
C308	0.2953 (10)	1.189 (2)	0.3229 (10)	0.073 (8)				
C309	0.3201 (10)	1.193 (2)	0.2834 (10)	0.073 (8)				
C310	0.3415 (10)	1.075 (2)	0.2699 (10)	0.067 (8)				
C311	0.3803 (10)	0.592 (2)	0.2444 (11)	0.062 (7)				
C312	0.3511 (10)	0.521 (2)	0.1943 (10)	0.078 (8)				
C313	0.3671 (12)	0.393 (3)	0.1812 (10)	0.088 (10)				
C314	0.4154 (12)	0.338 (2)	0.2206 (12)	0.089 (10)				
C315	0.4466 (10)	0.404 (2)	0.2697 (11)	0.075 (9)				
C316	0.4285 (10)	0.526 (2)	0.2820 (9)	0.074 (8)				
C317	0.3848 (9)	0.835 (2)	0.2062 (10)	0.063 (7)				
Pd4	0.13337 (10)	0.0169 (3)	0.26446 (10)	0.0814 (12)				
N401	0.1672 (8)	0.0911 (19)	0.2047 (9)	0.080 (7)				
N402	0.1592 (9)	−0.1565 (19)	0.2355 (9)	0.084 (7)				
C401	0.1674 (11)	0.217 (2)	0.1874 (10)	0.084 (9)				
C402	0.1929 (11)	0.259 (2)	0.1510 (11)	0.087 (10)				
C403	0.2180 (12)	0.164 (3)	0.1299 (11)	0.087 (10)				
C404	0.2193 (10)	0.029 (2)	0.1466 (10)	0.080 (8)				
C405	0.1911 (11)	−0.001 (2)	0.1820 (10)	0.079 (8)				
C406	0.1906 (11)	−0.139 (2)	0.2030 (11)	0.082 (8)				
C407	0.2169 (10)	−0.245 (2)	0.1868 (11)	0.088 (8)				
C408	0.2116 (12)	−0.371 (2)	0.2055 (12)	0.101 (10)				
C409	0.1813 (12)	−0.391 (2)	0.2403 (12)	0.101 (10)				
C410	0.1582 (11)	−0.278 (3)	0.2561 (10)	0.095 (9)				
C411	0.1157 (11)	0.195 (2)	0.2881 (11)	0.086 (7)				
C412	0.0691 (11)	0.274 (3)	0.2567 (9)	0.094 (9)				
C413	0.0597 (11)	0.405 (3)	0.2714 (12)	0.098 (10)				
C414	0.0976 (13)	0.457 (2)	0.3222 (12)	0.092 (10)				
C415	0.1422 (11)	0.383 (3)	0.3569 (11)	0.097 (10)				
C416	0.1491 (10)	0.253 (3)	0.3412 (11)	0.095 (9)				
C417	0.1061 (11)	−0.078 (3)	0.3229 (11)	0.091 (9)				

Table 2. Selected geometric parameters (Å, °)

Pd1—N101	2.110 (17)	Pd3—N301	2.10 (2)
Pd1—N102	2.097 (19)	Pd3—N302	2.115 (17)
Pd1—C111	1.94 (2)	Pd3—C311	1.99 (2)
Pd1—C117	2.05 (2)	Pd3—C317	1.96 (2)

No higher lattice symmetry than for space group $P2_1/c$ was indicated by the program *LEPAGE* (Spek, 1988). All non-H atoms were refined with anisotropic displacement parameters. H atoms were taken into account at calculated positions and refined with fixed geometry, riding on their carrier atoms and with a fixed isotropic displacement parameter (1.2 and 1.5 times the value of the equivalent isotropic displacement parameter of their sp^2 and sp^3 carrier atoms, respectively). Similarity restraints were used on the bipyridyl and phenyl moieties in view of the relatively weak data set. The atomic displacement parameters of C106 are somewhat unusual [main axis components $U_1 = 0.005$ (10), $U_2 = 0.043$ (10), $U_3 = 0.136$ (10) Å²] but probably a refinement artifact due to the relatively small reflection data-to-parameter ratio of 6631/725. No missed symmetry (*MISSYM*; Le Page, 1987) nor solvent accessible voids were detected by procedures implemented in *PLATON* (Spek, 1990, 1994).

Data collection: locally modified CAD-4 software. Cell refinement: *SET4* (de Boer & Duisenberg, 1984). Data reduction: *HELENA* (Spek, 1993). Program(s) used to solve structure: *SIR* (Altomare, Cascarano, Giacovazzo & Guagliardi, 1993). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLATON* (Spek, 1990). Software used to prepare material for publication: *PLATON*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1331). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Di- μ -phosphinato-bis[(1,10-phenanthroline)copper(II)] Nitrate, $[\text{Cu}_2(\text{H}_2\text{PO}_2)_2(\text{phen})_2](\text{NO}_3)_2$

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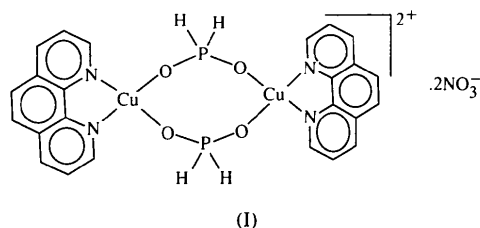
Abstract

The structure of blue $[\text{Cu}_2(\text{H}_2\text{PO}_2)_2(\text{C}_{12}\text{H}_8\text{N}_2)_2](\text{NO}_3)_2$ consists of dimeric $[\text{Cu}(\text{H}_2\text{PO}_2)(\text{phen})]$ subunits (phen is 1,10-phenanthroline) joined by two phosphinato bridging ligands, with each metal center organized in a distorted planar arrangement. These joined subunits exist as a discrete cation, with the equivalent of two nitrates as counterions. The subunits have a four-coordinate distorted square-planar arrangement of N and O atoms from the 1,10-phenanthroline and phosphinato ligands, with the fifth and sixth positions of the Cu center occupied by neighboring O atoms of the nitrate counterions. The coordination stereochemistry around each Cu is best described as tetragonal.

Comment

The existence of compounds such as $[\text{Cu}(\text{NCS})_2(\text{phen})]$ (Parker & Breneman, 1993) demonstrates that the oxidation potential of copper(II) is reduced by the presence of 1,10-phenanthroline, so that ligands that would normally be oxidized by copper(II) can coexist as part of a stable complex. The phosphinato ligand, H_2PO_2^- , was chosen as a possible bridging ligand for copper(II) complexes. The complex, $[\text{Cu}_2(\text{H}_2\text{PO}_2)_2(\text{phen})_2](\text{NO}_3)_2$, (I), exists as discrete pairs of $[\text{Cu}(\text{H}_2\text{PO}_2)(\text{phen})]$ subunits linked by the phosphinato ligands to form a cation with a 2+ charge. Two nitrate ions serve as the counterions

in this compound. Two related structures with phosphinato ligands bridging Mn^{II} atoms have been determined. The complexes $[\text{Mn}(\text{H}_2\text{PO}_2)_2(\text{bipy})]_n$ (Weakley, 1978*a*), where bipy is bipyridine, and $[\text{Mn}(\text{H}_2\text{PO}_2)_2(\text{phen})]_n$ (Weakley, 1978*b*) have structures that are bridged end-to-end through the O-atom ends of the two phosphinato ligands (H_2PO_2^-).



An ORTEP (Johnson, 1965) plot of the title complex is shown in Fig. 1, with the numbering system indicated. A stereoscopic plot of the unit cell is shown in Fig. 2. The cation, $[\text{Cu}_2(\text{H}_2\text{PO}_2)_2(\text{phen})_2]^{2+}$, consists of discrete pairs of $[\text{Cu}(\text{H}_2\text{PO}_2)(\text{phen})]$ subunits which are bridged by the phosphinato ligands in a symmetrical end-to-end fashion. The subunits have a four-coordinate distorted square-planar arrangement of N atoms from the 1,10-phenanthroline and O atoms from the phosphinato ligands about the central Cu atom. The cation interacts through the fifth and sixth positions on the Cu centers

