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A *cis*-Dichloro(1,10-phenanthroline)palladium(II) Complex

BY FRANK R. FRONCZEK, ISHENKUMBA KAHWA, SHIJIE LU, GEORGE R. NEWKOME, MARIO A. OLLINO, WILLIAM D. PITTS, AMNARD SITTATTRAKUL, JU-CHUN WANG AND STEVEN F. WATKINS

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803–1804, *USA*

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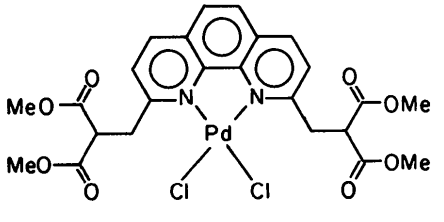
Abstract. *cis*-{2,9-Bis[2,2-bis(methoxycarbonyl)ethyl]-1,10-phenanthroline}dichloropalladium(II), $[PdCl_2(C_{24}H_{24}N_2O_8)]$, $M_r = 645{\cdot}77$, triclinic, $P\bar{1}$, $a = 8{\cdot}408$ (1), $b = 10{\cdot}222$ (1), $c = 15{\cdot}402$ (2) Å, $\alpha = 96{\cdot}45$ (1), $\beta = 96{\cdot}26$ (1), $\gamma = 100{\cdot}56$ (1)°, $V = 1281{\cdot}6$ (6) Å^3, $Z = 2$, $D_x = 1{\cdot}673$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 9{\cdot}7$ cm^{-1}, $F(000) = 652$, $T = 295$ (1) K, $R = 0{\cdot}032$ for 5327 reflections with $I > 3{\cdot}0\sigma(I)$. The 'square-planar' coordination about Pd is pyramidal, with Pd 0·196 (1) Å above the basal plane of two Cl and two N atoms. The five-membered metallocycle is in the envelope conformation, with Pd in the flap [dihedral angle 32·3 (2)°]. The phenanthroline is distorted into a crescent with limbs deviating from the mean plane by as much as 0·262 (3) Å.

Experimental. The compound (I) was synthesized as reported (Newkome, Puckett, Kiefer, Gupta, Fronczek, Pantaleo, McClure, Simpson & Deutsch, 1985) and recrystallized from dichloromethane/cyclohexane. An orange, prismatic crystal was mounted with epoxy on a glass fiber in random orientation. Details of data collection and structural refinement are given in Table 1.



(I)

The structure was solved using the Patterson heavy-atom method which revealed the position of Pd. The remaining atoms were located in successive difference Fourier syntheses. H-atom coordinates were calculated assuming ideal geometry and were included in the refinement, constrained to ride the C atoms to which they are bonded; in addition, all H-atom isotropic temperature factors were set to 1·3 times B_{eq} of each attached C.

Table 1. *Experimental details*

Crystal	Orange, prismatic, 0·24 × 0·36 × 0·44 mm
Instrument	Enraf–Nonius CAD-4 diffractometer
Monochromator	Incident beam, graphite
Unit cell	25 reflections, $26{\cdot}0 < 2\theta < 27{\cdot}9°$
Mode	ω–2θ
Standards	400, 030, 003
Corrections	Background, Lorentz, polarization, empirical absorption (0·897–1·000 on I)
2θ range	2·0–56·0°
hkl ranges	$h = 0$ to 11 $k = -13$ to 13 $l = -20$ to 19
Reflections	6320 total 6157 unique 5327 with $I > 3{\cdot}0\sigma(I)$
Solution	Patterson method
Function minimized	$\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$
Weights	$4F_o^2\mathrm{Lp}^2/[S^2(C+R^2B)+(0{\cdot}020F_o^2)^2]$, S = scan rate, C = integrated count, R = scan time/background time, B = background count
Parameters refined	334
R, wR, R(all)	0·032, 0·046, 0·043
Goodness of fit	2·14
Maximum shift/e.s.d.	0·01
$\Delta\rho$	0·85 (7), −0·39 (7) e Å^{-3}

The structure was refined in full-matrix least squares with Enraf–Nonius *SDP* (Frenz, 1978), where the function minimized was $\sum w(\lvert F_o\rvert - \lvert F_c\rvert)^2$ and the weight w is defined as $4F_o^2\sigma^2(F_o^2)$. The final cycle of refinement included 334 variable parameters and converged to $R = 0{\cdot}032$. Atomic scattering factors, including those for anomalous dispersion, were taken from *International Tables for X-ray Crystallography* (1974).

Final positional and equivalent isotropic thermal parameters are given in Table 2, and selected bond

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lengths and bond angles are shown in Table 3.* Fig. 1 shows the molecule and the atomic numbering scheme.

* Lists of structure factors, anisotropic thermal parameters, bond lengths, bond angles, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44691 (66 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Positional and equivalent isotropic thermal parameters and their e.s.d.'s*

	x	y	z	B_{eq}(Å^2)
Pd	0·09965 (2)	0·25119 (2)	0·32035 (1)	2·575 (3)
Cl(1)	0·14700 (9)	0·07170 (6)	0·38929 (5)	3·92 (1)
Cl(2)	0·36018 (9)	0·27349 (8)	0·28648 (6)	4·75 (2)
O1	0·0904 (3)	0·1365 (3)	−0·0174 (2)	5·99 (6)
O2	0·2884 (3)	0·3156 (3)	−0·0064 (2)	6·24 (7)
O3	−0·1790 (3)	0·3288 (3)	0·1147 (2)	6·44 (7)
O4	−0·1159 (4)	0·3395 (4)	−0·0184 (2)	7·56 (8)
O5	−0·3648 (3)	0·0890 (2)	0·2045 (2)	4·56 (5)
O6	−0·6053 (3)	−0·0491 (2)	0·1991 (2)	5·16 (5)
O7	−0·5115 (3)	−0·2564 (3)	0·3592 (2)	5·92 (6)
O8	−0·3659 (3)	−0·2257 (2)	0·2489 (2)	4·75 (5)
N1	0·0670 (3)	0·4304 (2)	0·2802 (1)	2·79 (4)
N2	−0·1162 (3)	0·2668 (2)	0·3691 (1)	2·77 (4)
C1	0·1321 (3)	0·5039 (3)	0·2215 (2)	3·14 (5)
C2	0·1357 (4)	0·6438 (3)	0·2299 (2)	4·00 (6)
C3	0·0727 (4)	0·7057 (3)	0·2974 (2)	4·21 (6)
C4	−0·0133 (3)	0·6276 (3)	0·3532 (2)	3·41 (5)
C5	−0·0151 (3)	0·4893 (2)	0·3411 (2)	2·89 (4)
C6	−0·1144 (3)	0·4013 (2)	0·3876 (2)	2·83 (4)
C7	−0·2135 (3)	0·4536 (3)	0·4452 (2)	3·30 (5)
C8	−0·3193 (4)	0·3603 (3)	0·4839 (2)	3·95 (6)
C9	−0·3288 (4)	0·2258 (3)	0·4597 (2)	3·87 (6)
C10	−0·2278 (3)	0·1803 (3)	0·4004 (2)	3·16 (5)
C11	−0·1030 (4)	0·6799 (3)	0·4180 (2)	4·02 (6)
C12	−0·2008 (4)	0·5968 (3)	0·4611 (2)	3·98 (5)
C13	−0·2471 (3)	0·0320 (3)	0·3718 (2)	3·36 (5)
C14	−0·4205 (3)	−0·0319 (3)	0·3262 (2)	3·39 (5)
C15	0·1982 (3)	0·4394 (3)	0·1443 (2)	3·36 (5)
C16	0·0873 (3)	0·3115 (3)	0·0945 (2)	3·13 (5)
C17	0·1681 (4)	0·2588 (3)	0·0177 (2)	3·88 (6)
C18	−0·0793 (4)	0·3310 (3)	0·0573 (2)	3·69 (6)
C19	0·1494 (6)	0·0762 (5)	−0·0947 (3)	8·0 (1)
C20	−0·3451 (4)	0·3399 (4)	0·0841 (3)	6·2 (1)
C21	−0·4559 (4)	0·0121 (3)	0·2364 (2)	3·55 (5)
C22	−0·4403 (3)	−0·1839 (3)	0·3154 (2)	3·53 (5)
C23	−0·3866 (5)	−0·3686 (3)	0·2242 (3)	5·18 (8)
C24	−0·6571 (6)	−0·0117 (5)	0·1145 (3)	7·7 (1)

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation:

$$\tfrac{4}{3}[a^2B_{11} + b^2B_{22} + c^2B_{33} + abB_{12}\cos\gamma + acB_{13}\cos\beta + bcB_{23}\cos\alpha].$$

Table 3. *Selected bond distances* (Å) *and angles* (°)

Numbers in parentheses are e.s.d.'s in the least-significant digits.

Pd	Cl(1)	2·2947 (5)		N1	C1	1·338 (2)	
Pd	Cl(2)	2·2836 (6)		N1	C5	1·373 (2)	
Pd	N1	2·053 (1)		N2	C6	1·369 (2)	
Pd	N2	2·064 (2)		N2	C10	1·336 (2)	
Cl(1)	Pd	Cl(2)	87·22 (2)	Pd	N1	C1	133·4 (1)
Cl(1)	Pd	N1	170·03 (4)	Pd	N1	C5	106·6 (1)
Cl(1)	Pd	N2	96·36 (4)	C1	N1	C5	118·6 (2)
Cl(2)	Pd	N1	94·03 (5)	Pd	N2	C6	106·4 (1)
Cl(2)	Pd	N2	168·07 (4)	Pd	N2	C10	133·5 (1)
N1	Pd	N2	80·51 (6)	C6	N2	C10	118·4 (2)

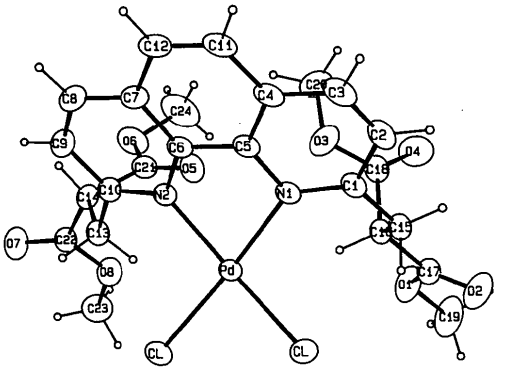

Fig. 1. Single molecule, 30% ellipsoids (Johnson, 1965).

Related literature. Synthesis of title compound and structure of related Pd complexes: Newkome *et al.* (1985); structure of analogous bipyridyl complex with similar distortions: Newkome, Fronczek, Gupta, Puckett, Pantaleo & Kiefer (1982); structure of related Zn^{II} complex: Preston & Kennard (1969*a*); structure of related Cu^{II} complex: Preston & Kennard (1969*b*); structures of related Au^{III} complexes: Robinson & Sinn (1975); structures of related Ni^{II} complexes: Butcher & Sinn (1977), Butcher, O'Connor & Sinn (1979); structure of related Pd^{II} complex: Newkome, Kiefer, Frere, Onishi, Gupta & Fronczek (1986).

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