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trans-Dichlorobis(metronidazole)-palladium(II), $[PdCl_2(C_6H_9N_3O_3)_2]$

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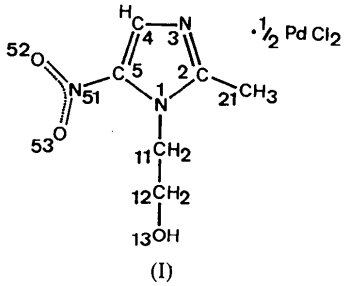
Abstract

The title complex, dichlorobis(2-methyl-5-nitroimidazole-1-ethanol-N^3)palladium(II), is *trans* square planar. The imidazole ring forms an angle of 88.8 (3)° with the square plane around the Pd atom, and an angle of 3.9 (4)° with the nitro group.

Comment

Nitroimidazoles are generally known as antiprotozoic and radiosensitizing drugs (Edwards, 1981). The structure of the title compound (I) has been determined to examine the influence of the coordination on the geometry of the heterocycle.



(I)

On the basis of a physicochemical study, Bales *et al.* (1983) concluded that the coordination of $Pt^{II}Cl_2$ to the basic imidazole was responsible for a withdrawal of electron density from the imidazole ring; however, they did not observe any change in the geometric properties of the metronidazole molecule. The structural analysis of the title compound is more appropriate for the evaluation of the effect of coordination since: (i) in the title compound the metronidazole scattering is less overpowered because of the lighter heavy atom, and (ii) the present structural model fits the measurements considerably better (R = 3.1 instead of 4.6%). However, the coordination effects, evaluated as the imidazole ring-angle differences between the title compound and the uncomplexed metronidazole (Blaton, Peeters & De Ranter, 1979), are, from the statistical point of view, still unobservable.

Since the bond angles at Pd do not deviate significantly from 90°, $PdCl_2$(metronidazole)$_2$ is square planar, as expected from the electron population of the 4*d* orbital of Pd^{II}. The complex has the Cl atoms and the metronidazole ligands *trans* to one another (Fig. 1).

The dihedral angle between the plane of the imidazole ring and the square plane around Pd is 88.8 (3)°. The dihedral angle between the plane of the imidazole ring and that of the nitro group is 3.9 (4)° [4.3 (1)° in the uncomplexed form (Blaton *et al.*, 1979)].

Each molecule forms a hydrogen bond with a symmetrically related neighbour [O13—H13···Cl^i: O13···Cl^i = 3.185 (2), H13···Cl^i = 2.48 (3) Å, O13—H13···Cl^i = 158 (4)°; symmetry code: (i) $-x+1, -y, -z$].

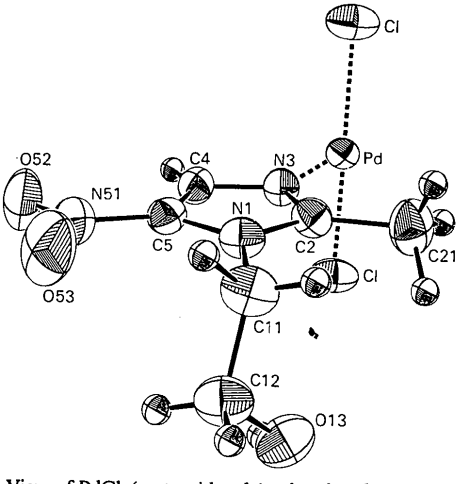


Fig. 1. View of $PdCl_2$(metronidazole)$_2$ showing the conformation.

Experimental

Crystal data

$[PdCl_2(C_6H_9N_3O_3)_2]$
M_r = 519.62
Monoclinic
$P2_1/a$

Cell parameters from 18 reflections
θ = 9–11°
μ = 1.266 mm^{-1}

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a = 7.134 (3) Å
b = 20.971 (6) Å
c = 7.635 (2) Å
β = 122.28 (1)°
V = 965.9 (5) Å^3
Z = 2
D_x = 1.787 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å

T = 293 K
Bar
0.27 × 0.12 × 0.12 mm
Yellow
Crystal source: solution of K_2PdCl_4 and metronidazole (SPECIA, Paris) in acetone/water (Bales *et al.*, 1983)

Data collection

Stoe Stadi-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: Gaussian
T_{min} = 0.70, T_{max} = 0.85
4345 measured reflections
2203 independent reflections
1508 observed reflections [$F > 4\sigma(F)$]

R_{int} = 0.021
θ_{max} = 27.5°
$h = -9 \rightarrow 9$
$k = -27 \rightarrow 0$
$l = -9 \rightarrow 9$
3 standard reflections
frequency: 120 min
intensity variation: 0.4%

Refinement

Refinement on F
R = 0.031
wR = 0.034
S = 1.252
1508 reflections
160 parameters
All H-atom parameters refined
$w = 4F^2/[\sigma^2(F^2) + (0.02F^2)^2]$
$(\Delta/\sigma)_{max}$ = 0.02

$\Delta\rho_{max}$ = 0.51 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1) (Pd, Cl, O, N, C) and Stewart, Davidson & Simpson (1965) (H)

Data collection: *DIF*4 (Stoe & Cie, 1988). Cell refinement: *DIF*4. Data reduction: *DREAM* (Blessing, 1987). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP-Plus* (Frenz, 1985). Molecular graphics: *FRODO* (Jones, 1978).

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Pd	0	0	0	0.02975 (5)
Cl	0.2548 (2)	−0.06635 (5)	−0.0014 (1)	0.0548 (2)
N1	0.1885 (4)	0.1476 (1)	−0.2367 (4)	0.0371 (6)
C2	0.0648 (5)	0.0964 (2)	−0.2546 (4)	0.0360 (7)
N3	0.1267 (4)	0.0737 (1)	−0.0680 (3)	0.0331 (5)
C4	0.2954 (5)	0.1114 (2)	0.0741 (5)	0.0366 (7)
C5	0.3353 (5)	0.1566 (2)	−0.0271 (5)	0.0377 (7)
C11	0.1796 (5)	0.1804 (2)	−0.4128 (5)	0.0471 (8)
C12	0.3682 (6)	0.1604 (2)	−0.4365 (5)	0.0521 (9)
O13	0.3669 (5)	0.0938 (1)	−0.4684 (4)	0.0584 (7)
C21	−0.1204 (6)	0.0698 (2)	−0.4510 (5)	0.056 (1)
N51	0.4900 (5)	0.2073 (2)	0.0637 (5)	0.0528 (7)
O52	0.6084 (5)	0.2076 (2)	0.2542 (5)	0.0726 (9)
O53	0.4972 (5)	0.2473 (2)	−0.0489 (5)	0.087 (1)

Table 2. *Selected geometric parameters* (Å, °)

Pd—Cl	2.293 (1)	N3—C4	1.362 (3)
Pd—N3	1.994 (2)	C4—C5	1.345 (6)
N1—C2	1.349 (4)	C5—N51	1.418 (5)
N1—C5	1.379 (3)	C11—C12	1.509 (6)
N1—C11	1.481 (5)	C12—O13	1.416 (4)
C2—N3	1.334 (4)	N51—O52	1.231 (4)
C2—C21	1.479 (4)	N51—O53	1.221 (6)
Cl—Pd—N3	89.8 (1)	N3—C4—C5	108.6 (2)
C5—N1—C11	128.8 (3)	N1—C5—C4	107.9 (3)
C2—N1—C11	124.6 (2)	C4—C5—N51	126.5 (3)
C2—N1—C5	106.0 (2)	N1—C5—N51	125.4 (3)
N1—C2—C21	125.7 (3)	N1—C11—C12	111.7 (2)
N1—C2—N3	110.4 (2)	C11—C12—O13	111.7 (3)
N3—C2—C21	123.7 (3)	C5—N51—O53	119.0 (3)
Pd—N3—C2	128.1 (2)	C5—N51—O52	116.4 (3)
C2—N3—C4	106.9 (2)	O52—N51—O53	124.5 (3)
Pd—N3—C4	124.9 (1)		

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71454 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GR1010]

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Zinc(II) Bis(phosphoglycolate) Dihydrate and Calcium Bis(phosphoglycolate) Dihydrate

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Abstract

Crystals of zinc(II) bis(phosphoglycolate) dihydrate, $Zn^{2+}.2C_2H_4O_6P^-.2H_2O$, are almost isomorphous with crystals of zinc(II) bis(phosphoenolpyruvate) dihydrate. The Zn atoms occupy centres of symmetry and are six-coordinate (through two water molecules and four phos-