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(1,1-Cyclobutanedicarboxylato-*O,O'*)-bis(trimethylphosphine-*P*)platinum(II) Monohydrate

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Abstract

The coordination around platinum is conventional square planar. The cyclobutane ring and the platinum coordination sphere are almost perpendicular to each other. The lattice contains water molecules though the crystals were grown from a cyclohexanol solution. Atom O(5) of the water molecule forms hydrogen bonds with the carboxylato O atoms O(4) and O(3[i]), respectively; the structure of the compound consists of one-dimensional polymeric chains.

Comment

The structure determination of the title compound (I) was undertaken in order to compare the structure with those of diammine analogues. Both the platinum coordination sphere and the cyclobutane ring are almost planar, with maximum displacements out of the least-squares plane of 0.111 Å for O(1) and 0.09 Å for C(5). The dihedral angle between the platinum coordination sphere and the cyclobutane ring is 88.66°. The mean distance of Pt—P(1) and Pt—P(2) is 2.222 (2) Å. The P(1)—Pt—P(2) angle is 95.13 (6)°. The mean distance of Pt—O(1) and Pt—O(2) is 2.056 (5) Å. The O(1)—Pt—O(2) angle is 86.3 (2)°. The C(4)—C(3)—C(6) and C(1)—C(3)—C(2) angles in the 1,1-cyclobutanedicarboxylate group are 88.5 (5) and 110.4 (5)°, respectively. The observed O(1)—Pt—O(2) angle of the title compound is smaller than those found in the diammine analogues [Pt(NH₃)₂(cbdca)] [89.9 (4)°; Neidle, Ismail & Sadler (1980)], where H₂cbdca is 1,1-cyclobutanedicarboxylic acid, and [Pt(*R,R*-dach)(cbdca)].H₂O [89.2 (4)°; Bitha *et*

al. (1990)], where *R,R*-dach is (1*R*,2*R*)-1,2-diaminocyclohexane. The mean Pt—O distances in these molecules are 2.029 (9) {[Pt(NH₃)₂(cbdca)]} and 2.006 (10) Å {[Pt(*R,R*-dach)(cbdca)].H₂O}. The corresponding C(4)—C(3)—C(6) and C(1)—C(3)—C(2) angles in the 1,1-cyclobutanedicarboxylato ligands are 89.2 (12) and 107.4 (8)° in [Pt(NH₃)₂(cbdca)] and 86.3 (11) and 115.9 (12)° in [Pt(*R,R*-dach)(cbdca)].H₂O, respectively.
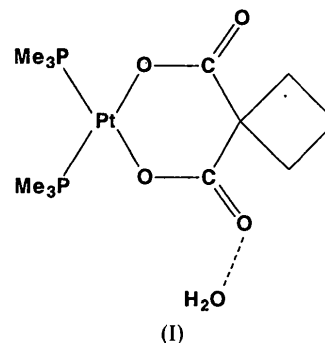

(I)

Fig. 1. A view of the unit cell. Hydrogen-bonding interactions between the water of crystallization and the carboxylato O atoms are shown as broken lines. Symmetry code: (ii) *x*, *y*, *z* − 1.
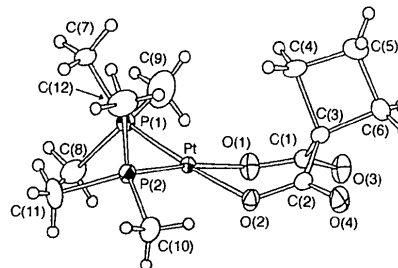

Fig. 2. *ORTEP*II (Johnson, 1976) view of the molecule with atomic labeling except for H atoms. The water of crystallization has been omitted for clarity. Thermal ellipsoids are drawn at the 30% probability level for non-H atoms.

Experimental

Crystal data

[Pt(C$_6$H$_6$O$_4$)(C$_3$H$_9$P)$_2$].H$_2$O
M_r = 507.37
Triclinic
$P\bar{1}$
a = 9.752 (2) Å
b = 11.680 (2) Å
c = 9.203 (2) Å
α = 92.43 (2)°
β = 115.30 (1)°
γ = 110.13 (1)°
V = 867.3 (3) Å^3
Z = 2
D_x = 1.943 Mg m^{-3}
D_m = 1.93 Mg m^{-3}
Density measured by flotation in 1,2-dibromoethane/hexane

Mo $K\alpha$ radiation
λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 19.9–20.0°
μ = 8.367 mm^{-1}
T = 296 K
Block
0.30 × 0.25 × 0.20 mm
Colorless
Crystal source: the compound was prepared as reported elsewhere (Miyamoto, Suzuki & Ichida, 1992a,b)

Data collection

Rigaku AFC-5R diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical using DIFABS (Walker & Stuart, 1983)
T_{min} = 0.79, T_{max} = 1.42
6611 measured reflections
6293 independent reflections
5068 observed reflections
[$F > 3.0\sigma(F)$]

θ_{max} = 32.6°
h = 0 → 14
k = −17 → 16
l = −13 → 12
3 standard reflections monitored every 147 reflections intensity variation: 19.40%

Refinement

Refinement on F
Final R = 0.041
wR = 0.044
S = 1.57
5068 reflections
182 parameters
H-atom parameters not refined

w = 1/$\sigma^2(F_o)$
$(\Delta/\sigma)_{max}$ = 0.01
$\Delta\rho_{max}$ = 2.84 e Å^{-3}
$\Delta\rho_{min}$ = −2.27 e Å^{-3}
Atomic scattering factors from International Tables for X-ray Crystallography (1974, Vol. IV)

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

$$U_{eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Pt	0.07786 (3)	0.20086 (2)	0.04675 (3)	0.02349 (9)
P(1)	−0.1070 (2)	0.2251 (1)	−0.1835 (2)	0.0317 (5)
P(2)	−0.0812 (2)	0.1613 (2)	0.1686 (2)	0.0319 (5)
O(1)	0.2380 (6)	0.2243 (5)	−0.0501 (6)	0.042 (1)
O(2)	0.2608 (5)	0.1887 (4)	0.2583 (6)	0.038 (1)
O(3)	0.4927 (6)	0.2755 (5)	−0.0129 (7)	0.051 (3)
O(4)	0.5207 (6)	0.2579 (5)	0.4497 (6)	0.052 (3)
O(5)	0.3778 (8)	0.1232 (6)	0.6673 (8)	0.066 (3)
C(1)	0.3971 (8)	0.2790 (6)	0.0394 (8)	0.032 (3)
C(2)	0.4154 (7)	0.2606 (5)	0.3171 (8)	0.030 (3)
C(3)	0.4639 (7)	0.3504 (5)	0.2134 (8)	0.029 (3)
C(4)	0.415 (1)	0.4646 (6)	0.222 (1)	0.041 (3)
C(5)	0.598 (1)	0.5482 (7)	0.332 (1)	0.058 (4)
C(6)	0.6477 (8)	0.4433 (6)	0.301 (1)	0.044 (3)
C(7)	−0.1987 (9)	0.3297 (6)	−0.1558 (9)	0.042 (3)
C(8)	−0.277 (1)	0.0866 (7)	−0.329 (1)	0.058 (3)
C(9)	−0.009 (1)	0.299 (1)	−0.301 (1)	0.080 (5)
C(10)	−0.020 (1)	0.0759 (8)	0.326 (1)	0.048 (3)
C(11)	−0.2993 (9)	0.0681 (9)	0.048 (1)	0.061 (4)
C(12)	−0.066 (1)	0.303 (1)	0.275 (1)	0.065 (4)

Table 2. Geometric parameters (Å, °)

Pt—P(1)	2.227 (2)	O(2)—C(2)	1.290 (7)
Pt—P(2)	2.216 (2)	O(3)—C(1)	1.229 (8)
Pt—O(1)	2.056 (5)	O(4)—C(2)	1.224 (7)
Pt—O(2)	2.055 (4)	C(1)—C(3)	1.518 (9)
P(1)—C(7)	1.810 (7)	C(2)—C(3)	1.537 (9)
P(1)—C(8)	1.798 (7)	C(3)—C(4)	1.573 (8)
P(1)—C(9)	1.798 (9)	C(3)—C(6)	1.547 (9)
P(2)—C(10)	1.801 (7)	C(4)—C(5)	1.53 (1)
P(2)—C(11)	1.797 (8)	C(5)—C(6)	1.52 (1)
P(2)—C(12)	1.813 (9)	O(3)···O(5^i)	2.907 (8)
O(1)—C(1)	1.294 (8)	O(4)···O(5)	3.093 (9)
P(1)—Pt—P(2)	95.13 (6)	C(7)—P(1)—C(8)	105.6 (3)
Pt—O(1)—C(1)	122.5 (4)	C(1)—C(3)—C(4)	114.0 (5)
P(1)—Pt—O(1)	90.9 (1)	C(7)—P(1)—C(9)	102.3 (5)
Pt—O(2)—C(2)	123.2 (4)	C(1)—C(3)—C(6)	116.5 (5)
P(1)—Pt—O(2)	176.1 (1)	C(8)—P(1)—C(9)	103.6 (5)
O(1)—C(1)—O(3)	122.1 (6)	C(2)—C(3)—C(4)	111.8 (5)
P(2)—Pt—O(1)	173.1 (1)	Pt—P(2)—C(10)	111.6 (3)
O(1)—C(1)—C(3)	117.7 (5)	C(2)—C(3)—C(6)	114.1 (5)
P(2)—Pt—O(2)	87.9 (1)	Pt—P(2)—C(11)	119.2 (3)
O(3)—C(1)—C(3)	120.3 (6)	C(4)—C(3)—C(6)	88.5 (5)
O(1)—Pt—O(2)	86.3 (2)	Pt—P(2)—C(12)	112.2 (3)
O(2)—C(2)—O(4)	121.9 (6)	C(3)—C(4)—C(5)	88.9 (5)
Pt—P(1)—C(7)	115.8 (2)	C(10)—P(2)—C(11)	102.1 (4)
O(2)—C(2)—C(3)	117.3 (5)	C(4)—C(5)—C(6)	91.0 (5)
Pt—P(1)—C(8)	117.7 (3)	C(10)—P(2)—C(12)	104.9 (4)
O(4)—C(2)—C(3)	120.7 (6)	C(3)—C(6)—C(5)	90.2 (5)
Pt—P(1)—C(9)	110.0 (3)	C(11)—P(2)—C(12)	105.4 (5)
C(1)—C(3)—C(2)	110.4 (5)		
Pt—O(1)—C(1)—O(3)	168.3 (5)	P(2)—Pt—O(1)—C(1)	−64 (1)
O(2)—Pt—P(1)—C(7)	−89 (2)	O(3)—C(1)—C(3)—C(6)	15.3 (8)
Pt—O(1)—C(1)—C(3)	−12.5 (7)	P(2)—Pt—O(2)—C(2)	−141.9 (5)
O(2)—Pt—P(1)—C(8)	144 (2)	O(4)—C(2)—C(3)—C(1)	126.7 (6)
Pt—O(2)—C(2)—O(4)	174.5 (5)	O(1)—Pt—P(1)—C(7)	−133.5 (3)
O(2)—Pt—P(1)—C(9)	26 (2)	O(4)—C(2)—C(3)—C(4)	−105.3 (7)
Pt—O(2)—C(2)—C(3)	−5.9 (8)	O(1)—Pt—P(1)—C(8)	100.2 (4)
O(2)—Pt—P(2)—C(10)	−25.3 (3)	O(4)—C(2)—C(3)—C(6)	−6.8 (9)
P(1)—Pt—P(2)—C(10)	157.2 (3)	O(1)—Pt—P(1)—C(9)	−18.1 (5)
O(2)—Pt—P(2)—C(11)	−144.1 (4)	C(1)—C(3)—C(4)—C(5)	−127.3 (5)
P(1)—Pt—P(2)—C(11)	38.4 (4)	O(1)—Pt—P(2)—C(10)	6 (1)
O(2)—Pt—P(2)—C(12)	92.1 (4)	C(1)—C(3)—C(6)—C(5)	125.1 (6)
P(1)—Pt—P(2)—C(12)	−85.4 (4)	O(1)—Pt—P(2)—C(11)	−112 (1)
O(2)—Pt—O(1)—C(1)	−31.6 (5)	C(2)—C(3)—C(4)—C(5)	106.7 (6)
P(1)—Pt—O(1)—C(1)	145.6 (5)	O(1)—Pt—P(2)—C(12)	124 (1)
O(2)—C(2)—C(3)—C(1)	−53.0 (7)	C(2)—C(3)—C(6)—C(5)	−104.4 (6)
P(1)—Pt—O(2)—C(2)	−2 (3)	O(1)—Pt—O(2)—C(2)	41.7 (5)
O(2)—C(2)—C(3)—C(4)	75.0 (7)	C(3)—C(4)—C(5)—C(6)	8.9 (6)
P(2)—Pt—P(1)—C(7)	49.9 (3)	O(1)—C(1)—C(3)—C(2)	63.8 (7)
O(2)—C(2)—C(3)—C(6)	173.6 (5)	C(3)—C(6)—C(5)—C(4)	−9.1 (6)
P(2)—Pt—P(1)—C(8)	−76.4 (4)	O(1)—C(1)—C(3)—C(4)	−63.0 (7)
O(3)—C(1)—C(3)—C(2)	−116.9 (6)	C(4)—C(3)—C(6)—C(5)	8.8 (6)
P(2)—Pt—P(1)—C(9)	165.2 (5)	O(1)—C(1)—C(3)—C(6)	−164.0 (6)
O(3)—C(1)—C(3)—C(4)	116.3 (7)	C(5)—C(4)—C(3)—C(6)	−8.8 (6)

Symmetry code: (i) $x, y, z - 1$.

The X-ray diffraction intensities were collected on a Rigaku AFC-5R diffractometer at the Research Center for Spectrochemistry, Faculty of Science, The University of Tokyo. The data were collected with a scan speed of 8.0° min^{-1} (in ω). Decay correction was applied. The structure was solved using the Patterson heavy-atom method which revealed the position of the Pt atom. The remaining non-H atoms were subsequently located in successive difference Fourier maps. All non-H atoms were refined anisotropically. The positions of H atoms were calculated with fixed bond length (C—H = 0.95 Å). All calculations were performed using the TEXSAN crystallographic software package of the Molecular Structure Corporation (1985) and carried

out on a DEC MicroVAX II at the Research Center for Spectrochemistry. Molecular graphics were prepared using *ORTEP*II (Johnson, 1976) and *PLUTO* (Motherwell & Clegg, 1978).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, complete geometry and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55987 (43 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1035]

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(Oxalato-*O,O'*)bis(trimethylphosphine)-platinum(II)

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Abstract

The coordination around platinum is conventional square-planar. The mean distance of Pt—O(1) and Pt—O(2) is 2.065 (6) Å. The mean distance of Pt—P(1) and Pt—P(2) is 2.216 (2) Å. The O(1)—Pt—O(2) angle is 81.4 (3)°. The P(1)—Pt—P(2) angle is 97.27 (9)°.
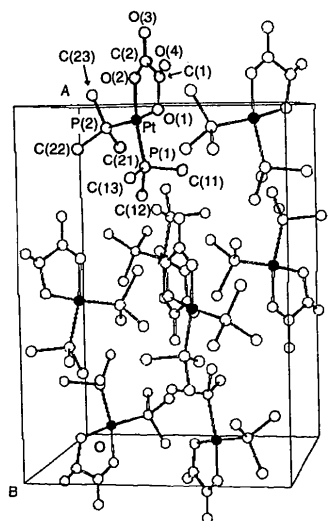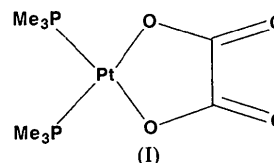
Fig. 1. A view of the unit cell with atomic numbering. H atoms have been omitted for clarity.

Comment

The structure determination of the title compound (I) was undertaken in order to compare the structure with that of the diammine analogue. The compound was prepared as reported elsewhere (Miyamoto, Suzuki & Ichida, 1992*a,b*). The O(1)—Pt—O(2) angle is small as a result of the chelating oxalate group. The mean distance of Pt—O(1) and Pt—O(2) is larger than that of diammine analogues, due to the strong *trans* influence of the phosphorus donor ligand. The O—Pt—O angle of the title compound is similar to those found in the diammine analogue and a series of Pt-oxalate complexes (Kobayashi *et al.*, 1982). In the crystal of [Pt(NH₃)₂(OCO)₂] (Rochon, Melanson, Macquet, Bélanger-Gariépy & Beauchamp, 1985), the O—Pt—O angle is 82.0 (5)° and the mean Pt—O distance is 2.01 (1) Å.

(I)

Experimental

Crystal data

[Pt(C₂O₄)(C₃H₉P)₂]	Mo *Kα* radiation
M_r = 435.27	λ = 0.71069 Å
Orthorhombic	Cell parameters from 25
Pbca	reflections
a = 16.662 (2) Å	θ = 19.8–20.0°
b = 12.990 (2) Å	μ = 10.945 mm⁻¹
c = 12.226 (2) Å	T = 297 K
V = 2646 (1) Å³	Block
Z = 8	0.38 × 0.35 × 0.35 mm
D_x = 2.185 Mg m⁻³	Colorless
D_m = 2.18 Mg m⁻³	