

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

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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)

YOSHITSUGU SUZUKI, T. KEN MIYAMOTO
AND HIKARU ICHIDA

Department of Chemistry, Faculty of Science, The University of Tokyo, Tokyo 113, Japan

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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)°.

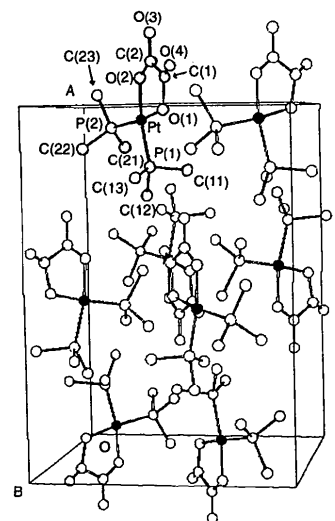
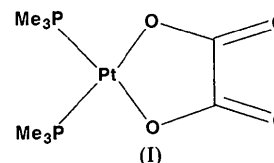


Fig. 1. A view of the unit cell with atomic numbering. H atoms have been omitted for clarity.

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, 1992a,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.



Experimental

Crystal data

[Pt(C₂O₄)(C₃H₉P)₂]

M_r = 435.27

Orthorhombic

Pbca

a = 16.662 (2) Å

b = 12.990 (2) Å

c = 12.226 (2) Å

V = 2646 (1) Å³

Z = 8

D_x = 2.185 Mg m⁻³

D_m = 2.18 Mg m⁻³

Mo Kα radiation

λ = 0.71069 Å

Cell parameters from 25 reflections

θ = 19.8–20.0°

μ = 10.945 mm⁻¹

T = 297 K

Block

0.38 × 0.35 × 0.35 mm

Colorless

Data collection

Rigaku AFC-5R diffractometer	$\theta_{\max} = 30.1^\circ$
$\omega/2\theta$ scans	$h = 0 \rightarrow 23$
Absorption correction: empirical	$k = 0 \rightarrow 18$
$T_{\min} = 0.45$, $T_{\max} = 1.00$	$l = -17 \rightarrow 0$
4335 measured reflections	3 standard reflections
4335 independent reflections	monitored every 150 reflections
2389 observed reflections	intensity variation: none
$[F > 3.0\sigma(F)]$	

Refinement

Refinement on F^2	$w = 1/\sigma^2(F_o)$
Final $R = 0.040$	$(\Delta/\sigma)_{\max} = 0.05$
$wR = 0.044$	$\Delta\rho_{\max} = 2.23 \text{ e } \text{Å}^{-3}$
$S = 1.68$	$\Delta\rho_{\min} = -3.42 \text{ e } \text{Å}^{-3}$
2389 reflections	Atomic scattering factors
136 parameters	from <i>International Tables</i>
H-atom parameters not refined	for <i>X-ray Crystallography</i>
	(1974, Vol. IV)

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

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Pt(1)	0.04136 (2)	0.09912 (3)	0.12733 (3)	0.0255 (1)
P(1)	0.0633 (1)	-0.0050 (2)	0.2673 (2)	0.029 (1)
P(2)	0.1689 (1)	0.1315 (2)	0.0877 (2)	0.028 (1)
O(1)	-0.0814 (4)	0.0858 (5)	0.1439 (5)	0.038 (4)
O(2)	0.0071 (4)	0.2064 (5)	0.0116 (6)	0.042 (4)
O(3)	-0.1000 (4)	0.2991 (6)	-0.0290 (6)	0.057 (5)
O(4)	-0.1930 (4)	0.1523 (7)	0.0770 (7)	0.065 (5)
C(1)	-0.1207 (6)	0.1506 (8)	0.0824 (9)	0.039 (5)
C(2)	-0.0695 (6)	0.2264 (8)	0.0169 (8)	0.038 (5)
C(11)	-0.0302 (6)	-0.044 (1)	0.327 (1)	0.073 (9)
C(12)	0.1148 (8)	-0.1246 (8)	0.243 (1)	0.060 (8)
C(13)	0.1138 (8)	0.054 (1)	0.3812 (9)	0.058 (8)
C(21)	0.1975 (6)	0.2564 (9)	0.136 (1)	0.056 (8)
C(22)	0.2508 (6)	0.051 (1)	0.129 (1)	0.058 (6)
C(23)	0.1810 (7)	0.140 (1)	-0.0577 (8)	0.051 (6)

Table 2. Selected bond lengths (Å) and angles ($^\circ$)

Pt(1)—P(1)	2.212 (2)	P(2)—C(22)	1.79 (1)
Pt(1)—P(2)	2.219 (2)	P(2)—C(23)	1.79 (1)
Pt(1)—O(1)	2.064 (6)	O(1)—C(1)	1.30 (1)
Pt(1)—O(2)	2.066 (6)	O(2)—C(2)	1.30 (1)
P(1)—C(11)	1.79 (1)	O(3)—C(2)	1.21 (1)
P(1)—C(12)	1.80 (1)	O(4)—C(1)	1.21 (1)
P(1)—C(13)	1.80 (1)	C(1)—C(2)	1.53 (1)
P(2)—C(21)	1.79 (1)		
P(1)—Pt(1)—P(2)	97.27 (9)	Pt(1)—P(2)—C(22)	123.8 (4)
P(1)—Pt(1)—O(1)	92.1 (2)	Pt(1)—P(2)—C(23)	109.6 (4)
P(1)—Pt(1)—O(2)	171.1 (2)	C(21)—P(2)—C(22)	103.4 (6)
P(2)—Pt(1)—O(1)	170.6 (2)	C(21)—P(2)—C(23)	104.1 (6)
P(2)—Pt(1)—O(2)	89.3 (2)	C(22)—P(2)—C(23)	103.2 (6)
O(1)—Pt(1)—O(2)	81.4 (3)	Pt(1)—O(1)—C(1)	112.7 (6)
Pt(1)—P(1)—C(11)	110.0 (4)	Pt(1)—O(2)—C(2)	111.7 (6)
Pt(1)—P(1)—C(12)	118.8 (4)	O(1)—C(1)—O(4)	123 (1)
Pt(1)—P(1)—C(13)	114.4 (4)	O(1)—C(1)—C(2)	116.0 (8)
C(11)—P(1)—C(12)	103.8 (7)	O(4)—C(1)—C(2)	121 (1)
C(11)—P(1)—C(13)	102.3 (7)	O(2)—C(2)—O(3)	123 (1)
C(12)—P(1)—C(13)	105.9 (6)	O(2)—C(2)—C(1)	116.3 (8)
Pt(1)—P(2)—C(21)	110.8 (4)	O(3)—C(2)—C(1)	120.8 (9)

The compound was prepared as reported elsewhere (Miyamoto, Suzuki & Ichida, 1992a,b). The density was measured by flotation in a 1,2-dibromoethane/hexane mixture.

The X-ray diffraction intensities were collected at the Research Center for Spectrochemistry, Faculty of Science, The University of Tokyo. The data were collected with a scan speed of $8.0^\circ \text{ min}^{-1}$ (in ω). Lorentz-polarization and absorption corrections were applied. The Pt atom was located by direct methods. Remaining non-H atoms were revealed by subsequent least-squares refinements and difference Fourier maps. All non-H atoms were refined anisotropically. The positions of H atoms were calculated geometrically. All calculations were performed using the *TEXSAN* (Molecular Structure Corporation, 1985) crystallographic software package and carried out on a DEC MicroVAX II at the Research Center for Spectrochemistry.

This work was supported in part by a Grant-in-Aid for Scientific Research (No. 04453043) from the Ministry of Education, Science, and Culture of Japan, to which our thanks are due.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55979 (22 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1038]

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[SP-4-2]-Dinitratobis(trimethylphosphine)-platinum(II)

YOSHITSUGU SUZUKI, T. KEN MIYAMOTO*
AND HIKARU ICHIDA

Department of Chemistry, Faculty of Science, The University of Tokyo, Tokyo 113, Japan

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Abstract

The coordination around platinum is conventional square-planar. The mean Pt—O and Pt—P distances are 2.125 (7) and 2.231 (3) Å , respectively. The P(1)—Pt—P(2) and