

**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 ( $\text{Å}^2$ )

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

	x	y	z	$U_{\text{eq}}$
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 ( $\text{Å}$ ) 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  $\omega$ ). 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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*Acta Cryst.* (1993). **C49**, 1318–1320

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

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(Received 11 November 1992; accepted 22 December 1992)

**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)  $\text{Å}$ , respectively. The P(1)—Pt—P(2) and

O(1)—Pt—O(4) angles are 94.9 (1) and 82.3 (3)°, respectively. Coordination appears to cause some distortion of the nitrate group. The N—O(coordinated) bond distances [N(1)—O(1), 1.31 (1); N(2)—O(4), 1.29 (1) Å] appear to be larger than the other N—O distances [1.20 (1)–1.25 (1) Å] and the bond angles O(2)—N(1)—O(3) [124 (1)°] and O(5)—N(2)—O(6) [123 (1)°] appear to be larger than the other O—N—O angles [117 (1)–119 (1)°]. The nitrate groups lie on opposite sides of the ligand plane.

### Comment

The structure of the title compound was determined in order to compare it with that of the diammine analogue. The compound was prepared according to the literature method (Trovo *et al.*, 1990). The observed O(1)—Pt—O(4) angle of the title compound is smaller than that found in the corresponding diammine analogue, *cis*-[Pt(NO<sub>3</sub>)<sub>2</sub>(NH<sub>3</sub>)<sub>2</sub>] [88.8 (5)°; Lippert, Lock, Rosenberg & Zvagulis (1977)]. The mean Pt—O distance in the title compound is longer than that found in *cis*-[Pt(NO<sub>3</sub>)<sub>2</sub>(NH<sub>3</sub>)<sub>2</sub>] [2.01 (1) Å] owing to the strong *trans* influence of the phosphorous donor ligand. The nitrate groups found in the diammine analogue lie on the same side of the ligand plane and in the crystal the N—O(coordinated) bond distances [1.28 (2)–1.30 (2) Å] appear to be larger than the other N—O distances [1.19 (2)–1.24 (2) Å].

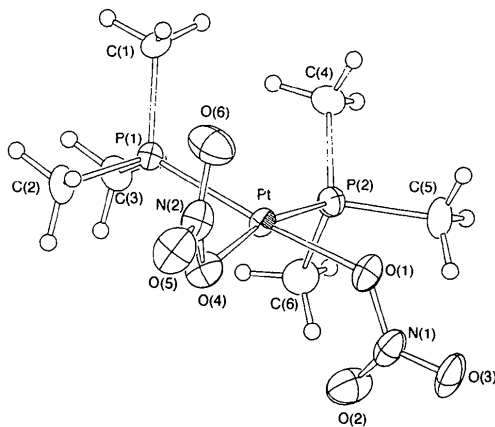


Fig. 1. ORTEP (Johnson, 1976) view of the molecule with atomic labeling except for H atoms. Thermal ellipsoids are drawn at the 30% probability level for non-H atoms.

### Experimental

#### Crystal data

[Pt(NO<sub>3</sub>)<sub>2</sub>(C<sub>3</sub>H<sub>9</sub>P)<sub>2</sub>]  
M<sub>r</sub> = 471.26

D<sub>m</sub> = 2.22 Mg m<sup>-3</sup>  
Mo K $\alpha$  radiation

Monoclinic  
P2<sub>1</sub>/c  
a = 8.514 (1) Å  
b = 14.760 (1) Å  
c = 11.360 (2) Å  
 $\beta$  = 98.79 (1)°  
V = 1410.8 (3) Å<sup>3</sup>  
Z = 4  
D<sub>x</sub> = 2.218 Mg m<sup>-3</sup>

#### Data collection

Rigaku AFC-5R diffractometer  
 $\omega/2\theta$  scans  
Absorption correction: empirical  
T<sub>min</sub> = 0.43, T<sub>max</sub> = 1.00  
5557 measured reflections  
5268 independent reflections  
2548 observed reflections  
[F > 3.0 $\sigma$ (F)]

#### Refinement

Refinement on F  
Final R = 0.044  
wR = 0.045  
S = 1.38  
2548 reflections  
154 parameters  
H-atom parameters not refined

$\lambda$  = 0.71069 Å  
Cell parameters from 25 reflections  
 $\theta$  = 15.9–17.2°  
 $\mu$  = 10.286 mm<sup>-1</sup>  
T = 296 K  
Prism  
0.25 × 0.18 × 0.12 mm  
Colorless

R<sub>int</sub> = 0.037  
 $\theta_{max}$  = 32.6°  
h = 0 → 12  
k = 0 → 21  
l = -16 → 16  
3 standard reflections monitored every 147 reflections  
intensity variation: 14.90%

w = 1/ $\sigma^2$ (F<sub>o</sub>)  
( $\Delta/\sigma$ )<sub>max</sub> = 0.02  
 $\Delta\rho_{max}$  = 2.31 e Å<sup>-3</sup>  
 $\Delta\rho_{min}$  = -2.16 e Å<sup>-3</sup>  
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (Å<sup>2</sup>)

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

	x	y	z	U <sub>eq</sub>
Pt	0.19616 (4)	0.24386 (2)	0.29590 (3)	0.0339 (1)
P(1)	0.3872 (3)	0.3095 (2)	0.4227 (2)	0.037 (1)
P(2)	0.0918 (3)	0.3740 (2)	0.2191 (3)	0.039 (1)
O(1)	0.045 (1)	0.1682 (5)	0.1678 (7)	0.052 (4)
O(2)	-0.149 (1)	0.1956 (6)	0.266 (1)	0.080 (6)
O(3)	-0.189 (1)	0.1163 (5)	0.1064 (8)	0.071 (5)
O(4)	0.244 (1)	0.1130 (5)	0.3744 (8)	0.054 (5)
O(5)	0.375 (1)	-0.0105 (5)	0.376 (1)	0.073 (5)
O(6)	0.457 (1)	0.1066 (7)	0.289 (1)	0.085 (6)
N(1)	-0.104 (1)	0.1597 (6)	0.181 (1)	0.053 (5)
N(2)	0.361 (1)	0.0673 (6)	0.345 (1)	0.054 (5)
C(1)	0.560 (1)	0.3396 (8)	0.355 (1)	0.053 (6)
C(2)	0.467 (1)	0.2353 (8)	0.543 (1)	0.057 (6)
C(3)	0.336 (1)	0.4094 (7)	0.501 (1)	0.052 (6)
C(4)	0.223 (1)	0.4626 (7)	0.182 (1)	0.051 (6)
C(5)	-0.036 (1)	0.3582 (8)	0.079 (1)	0.058 (6)
C(6)	-0.036 (1)	0.4247 (7)	0.316 (1)	0.054 (6)

Table 2. Selected bond lengths (Å), angles (°) and torsion angles (°)

Pt—P(1)	2.224 (3)	P(2)—C(5)	1.80 (1)
Pt—P(2)	2.237 (3)	P(2)—C(6)	1.82 (1)
Pt—O(1)	2.110 (7)	O(1)—N(1)	1.31 (1)
Pt—O(4)	2.139 (7)	O(2)—N(1)	1.22 (1)
P(1)—C(1)	1.81 (1)	O(3)—N(1)	1.21 (1)
P(1)—C(2)	1.80 (1)	O(4)—N(2)	1.29 (1)
P(1)—C(3)	1.81 (1)	O(5)—N(2)	1.20 (1)
P(2)—C(4)	1.81 (1)	O(6)—N(2)	1.25 (1)

Pt(1)—Pt—P(2)	94.9 (1)	Pt—P(2)—C(5)	112.6 (4)
P(1)—Pt—O(1)	170.6 (2)	Pt—P(2)—C(6)	110.7 (4)
P(1)—Pt—O(4)	92.9 (2)	C(4)—P(2)—C(5)	101.7 (6)
P(2)—Pt—O(1)	91.2 (2)	C(4)—P(2)—C(6)	106.8 (5)
P(2)—Pt—O(4)	167.5 (2)	C(5)—P(2)—C(6)	104.5 (6)
O(1)—Pt—O(4)	82.3 (3)	Pt—O(1)—N(1)	118.3 (7)
Pt—P(1)—C(1)	113.1 (4)	Pt—O(4)—N(2)	117.8 (7)
Pt—P(1)—C(2)	112.3 (4)	O(1)—N(1)—O(2)	119 (1)
Pt—P(1)—C(3)	117.9 (4)	O(1)—N(1)—O(3)	117 (1)
C(1)—P(1)—C(2)	103.5 (6)	O(2)—N(1)—O(3)	124 (1)
C(1)—P(1)—C(3)	106.3 (5)	O(4)—N(2)—O(5)	118 (1)
C(2)—P(1)—C(3)	102.3 (5)	O(4)—N(2)—O(6)	118.7 (9)
Pt—P(2)—C(4)	119.3 (4)	O(5)—N(2)—O(6)	123 (1)
Pt—O(1)—N(1)—O(2)	0 (1)	O(1)—Pt—P(1)—C(1)	48 (1)
Pt—O(1)—N(1)—O(3)	179.8 (7)	O(1)—Pt—P(1)—C(2)	-69 (1)
Pt—O(4)—N(2)—O(5)	-170.4 (8)	O(1)—Pt—P(1)—C(3)	173 (1)
Pt—O(4)—N(2)—O(6)	12 (1)	O(1)—Pt—P(2)—C(4)	-128.8 (5)
P(1)—Pt—P(2)—C(4)	44.1 (5)	O(1)—Pt—P(2)—C(5)	-9.9 (5)
P(1)—Pt—P(2)—C(5)	163.1 (5)	O(1)—Pt—P(2)—C(6)	106.8 (5)
P(1)—Pt—P(2)—C(6)	-80.3 (4)	O(1)—Pt—O(4)—N(2)	91.3 (8)
P(1)—Pt—O(1)—N(1)	153 (1)	O(4)—Pt—P(1)—C(1)	106.7 (5)
P(1)—Pt—O(4)—N(2)	-80.6 (8)	O(4)—Pt—P(1)—C(2)	-10.1 (5)
P(2)—Pt—P(1)—C(1)	-83.1 (4)	O(4)—Pt—P(1)—C(3)	-128.6 (5)
P(2)—Pt—P(1)—C(2)	160.2 (5)	O(4)—Pt—P(2)—C(4)	173 (1)
P(2)—Pt—P(1)—C(3)	41.6 (4)	O(4)—Pt—P(2)—C(5)	-68 (1)
P(2)—Pt—O(1)—N(1)	-75.7 (7)	O(4)—Pt—P(2)—C(6)	48 (1)
P(2)—Pt—O(4)—N(2)	150.7 (8)	O(4)—Pt—O(1)—N(1)	93.6 (7)

Trovo, G., Bandoli, G., Casellato, U., Corain, B., Nicolini, M. & Longato, B. (1990). *Inorg. Chem.* **29**, 4616–4621.

*Acta Cryst.* (1993). **C49**, 1320–1322

## $\mu$ -Hydroxo Diplatinum(II) Complex Containing Bridging Cyclometallated 1,3-Bis(diphenylphosphino)propane as the Tetrafluoroborate Salt

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(Received 16 September 1992; accepted 4 January 1993)

### Abstract

The structure of the title compound bis- $\mu$ -(2-[(diphenylphosphino)propyl](phenyl)phosphino)phenyl-1 $\kappa$ C<sup>1</sup>:2 $\kappa^2$ P,P';1 $\kappa^2$ P,P':2 $\kappa$ C<sup>1</sup>- $\mu$ -hydroxo-diplatinum(II) tetrafluoroborate bis(dichloromethane) solvate is reported as one of the few examples of a derivative of the parent dimers [Pt-*o*-C<sub>6</sub>H<sub>4</sub>(Ph)P(CH<sub>2</sub>)<sub>n</sub>PPh<sub>2</sub>]<sub>2</sub> where *n* = 2 or 3. Such dimers are linked by two bridging *ortho*-metallated phenyls attached to a coordinated P atom. The hydroxo group symmetrically bridges the two Pt atoms increasing their separation to 2.867 Å.

### Comment

The title complex was isolated as the result of an *in situ* hydrolysis of the product of reaction of the dimer [Pt- $\mu$ -{*o*-C<sub>6</sub>H<sub>4</sub>(Ph)PCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>PPh<sub>2</sub>}]<sub>2</sub> with silver tetrafluoroborate. NMR evidence of the mixture suggested that a silver-platinum complex was initially formed (Dekker, Elsevier, Poelsma, Vrieze, van Leeuwen, Smeets & Spek, 1992), which slowly changed to the bridging hydroxo species.

Few examples containing these *ortho*-metallated complexes have been characterized crystallographically – particularly in the series where the chelating phosphine has a propane backbone. The framework of the parent dimer is retained, although the changes in the P—Pt—Pt angles reflect the orientation demands of the bridging hydroxo

Colorless prism crystals were obtained by slow evaporation of a dichloromethane solution of the title compound. The density was measured by flotation in a diiodomethane/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° min<sup>-1</sup> (in  $\omega$ ). 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 geometrically. All calculations were performed using the TEXSAN (Molecular Structure Corporation, 1985) crystallographic software package and carried out on a DEC MicroVAX II computer at the Research Center for Spectrochemistry. Molecular graphics: ORTEPII (Johnson, 1976).

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 55983 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS1039]

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