Acta Cryst. (1976). B32, 1596

trans-Bis(dimethylphenylphosphine)bis-(3-*Z*-methoxy-1-propenyl)platinum(II)

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(*Received* 26 *January* 1976; *accepted* 9 *February* 1976)

Abstract. $[Pt(PMe_2Ph)_2(CH{:}CHCH_2OMe)_2]$, $C_{24}H_{36}O_2P_2Pt$, monoclinic, $P2_1/a$, $a=23{\cdot}26$ (2), $b=6{\cdot}16$ (1), $c=9{\cdot}28$ (1) Å, $\beta=107{\cdot}5$ (2)°, $Z=2$, $D_x=1{\cdot}61$ g cm^{-3}, $V=1268$ Å^3. The compound, which was obtained by hydrazine reduction of the corresponding acetylide, is shown to have the *Z* stereochemistry, with crystallographic symmetry $\bar{1}$.

Introduction. Measurements were made on a Nonius CAD-4 diffractometer using monochromatized Cu $K\alpha$ radiation. Intensities were measured in the θ–2θ scan mode using a scintillation counter and pulse-height discrimination, and the 1652 independent reflexions which were significantly above background were used in the structure determination. The structure was solved by the heavy-atom method and refined by full-matrix least squares. Minimizing $\sum w(F_o-|F_c|)^2$, with $w=1/(1+0{\cdot}002F_o^2)$, adjustment of coordinates, anisotropic temperature factors for Pt and P, and isotropic temperature factors for C and O, reduced *R* to 6·89%. Atomic scattering factors, including $\Delta f'$ and $\Delta f''$ were taken from *International Tables for X-ray Crystallography* (1974). The atomic coordinates and vibration parameters with their e.s.d.'s are given in Table 1.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31678 (11 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Fractional coordinates and vibration parameters* (Å$^2\times10^3$) *and their e.s.d.'s*

The temperature factors are in the form $\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{23}klb^*c^*+2U_{31}lhc^*a^*+2U_{12}hka^*b^*)]$ or $\exp[-2\pi^2U_{iso}(2\sin\theta/\lambda)^2]$.

	x	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
Pt	0	0	0	21·2 (4)	46·0 (4)	31·6 (4)	−1·1 (6)	5·7 (3)	−2·0 (5)
P	0·0840 (2)	−0·1727 (7)	0·1470 (4)	28 (2)	47 (2)	39 (2)	1 (2)	5 (2)	1 (2)

Table 1 (*cont.*)

	x	*y*	*z*	U_{iso}
C(1)	0·0122 (7)	−0·152 (3)	−0·189 (2)	52 (4)
C(2)	0·0398 (8)	−0·085 (3)	−0·291 (2)	52 (4)
C(3)	0·0668 (8)	0·134 (3)	−0·289 (2)	57 (4)
O(4)	0·1261 (6)	0·104 (2)	−0·303 (2)	65 (3)
C(5)	0·1541 (9)	0·315 (4)	−0·300 (3)	78 (6)
C(6)	0·0826 (8)	−0·275 (3)	0·332 (2)	63 (5)
C(7)	0·1075 (9)	−0·416 (3)	0·066 (2)	61 (4)
C(8)	0·1505 (6)	−0·005 (3)	0·191 (2)	41 (3)
C(9)	0·1779 (8)	0·036 (3)	0·080 (2)	52 (4)
C(10)	0·2296 (9)	0·169 (4)	0·109 (2)	68 (5)
C(11)	0·2525 (9)	0·267 (3)	0·251 (2)	68 (5)
C(12)	0·2251 (9)	0·230 (3)	0·361 (2)	68 (5)
C(13)	0·1729 (8)	0·095 (3)	0·331 (2)	56 (4)

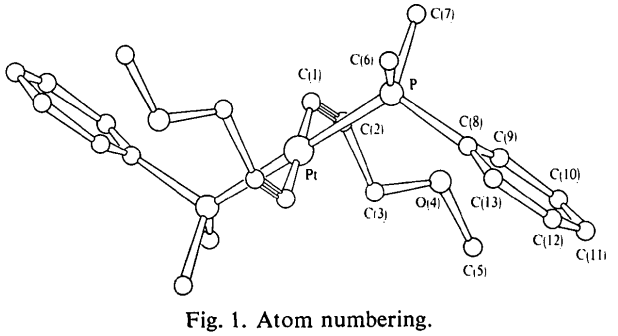

Fig. 1. Atom numbering.

Table 2. *Bond lengths* (Å) *and angles* (°) *with their e.s.d.'s*

Pt—P	2·282 (4)	P—Pt—C(1)	89·2 (4)
Pt—C(1)	2·08 (2)	Pt—C(1)–C(2)	132 (1)
C(1)–C(2)	1·35 (3)	C(1)–C(2)–C(3)	124 (2)
C(2)–C(3)	1·49 (3)	C(2)–C(3)–O(4)	107 (1)
C(3)–O(4)	1·43 (2)	C(3)–O(4)–C(5)	109 (1)
O(4)–C(5)	1·45 (3)		
P—C(6)	1·84 (2)	Pt—P—C(6)	117·8 (6)
P—C(7)	1·83 (2)	Pt—P—C(7)	116·3 (6)
P—C(8)	1·80 (2)	Pt—P—C(8)	113·0 (5)

C–C (benzene ring) 1·38–1·43 (3)

Discussion. The molecular structure and atom numbering are shown in Fig. 1 and bond lengths and angles are given in Table 2. The compound is obtained by the hydrazine reduction of *trans*-$[Pt(PMe_2Ph)_2(C{:}CCH_2OMe)_2]$ (Empsall, Shaw & Stringer, 1975) and this analysis establishes that *cis* addition occurs, giving the *Z* stereochemistry shown. The chain of atoms from C(3) through Pt to C(3′) is planar, with individual atom displacements of less than one standard deviation. The atoms Pt, P, P′, C(1), C(1′) are necessarily coplanar because of the molecular $\bar{1}$ symmetry, and the dihedral angle between these two planes is 83·1°. The differing sizes of Pt and methylene are reflected in the two angles at the olefin

group; the Pt–C(1)–C(2) angle of 132 (1)° gives a Pt···C(2) contact of 3·15 Å, while the C(1)–C(2)–C(3) angle of 124 (2)° gives a C(1)···C(3) distance of 2·50 Å. These angles may also be influenced by the interaction between Pt and C(3), which are separated by 3·58 Å, with a calculated Pt···H–C(3) contact of 2·99 Å.

We are grateful to Dr B. Sheldrick of the Biophysics Department, University of Leeds, for use of the diffractometer and to the Science Research Council for a studentship (to K.H.P.O'F.).

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Acta Cryst. (1976). B**32**, 1597

1-Oxo-2-oxa-3,3-dimethylpent-4-ene-1,4-diylbis(triphenylphosphine)platinum(II)

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(*Received* 26 *January* 1976; *accepted* 9 *February* 1976)

Abstract. [Pt(CO.OCMe$_2$C:CH$_2$)(PPh$_3$)$_2$], $C_{42}H_{38}O_2P_2Pt$, monoclinic, $P2_1/c$, $a=12{\cdot}298$ (2), $b=11{\cdot}038$ (3), $c=27{\cdot}207$ (3) Å, $\beta=102{\cdot}66$ (1)°, $D_m=1{\cdot}53$, $D_x=1{\cdot}533$ g cm^{-3}, $Z=4$, $V=3603$ Å^3. The compound, obtained by carbonylation of a hydroxyacetylene complex, is shown to have a 'platinalactone' structure with a five-membered chelate ring and exocyclic oxo and methylene groups adjacent to the metal.

Introduction. Measurements were made on a Nonius CAD-4 diffractometer using monochromatized Cu $K\alpha_1$ radiation ($\lambda=1{\cdot}54051$ Å). The cell dimensions and their e.s.d.'s were obtained by a least-squares fit of sin θ values for 25 reflexions centred using the program *SETANG*. Intensities were recorded in the θ–2θ scan mode using a scintillation counter and pulse-height discrimination. A control reflexion, monitored every 50 reflexions, had fallen in intensity by 16% at the end of data collection, and the measured reflexions were scaled accordingly. The structure determination used the 3732 independent reflexions with $\theta<70°$ and $I>3\sigma(I)$, where $I=P-2(B_1+B_2)$ and $\sigma^2(I)=P+4(B_1+B_2)+(0{\cdot}06I)^2$.

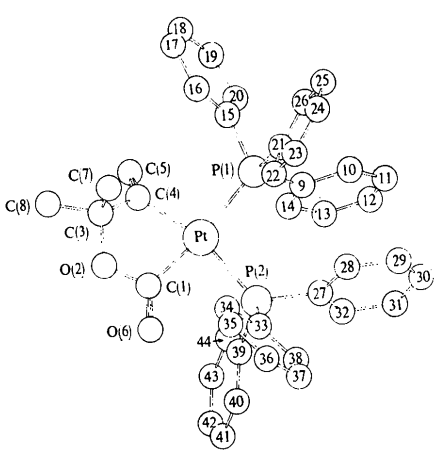

Fig. 1. Atom numbering.

The structure was solved by the heavy-atom method and refined by full-matrix least squares using the X-RAY programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). Atomic coordinates, anisotropic temperature factors for Pt and P, and isotropic temperature factors for O and C were refined; the phenyl H atoms were included in calculated positions, assigned the temperature factors of the C atoms to which they are attached, but were not refined. Atomic scattering factors, including $\Delta f'$ and $\Delta f''$, were taken from *International Tables for X-ray Crystallography* (1974). Minimization of $w(F_o-|F_c|)^2$, with weights derived from the expression for $\sigma^2(I)$ given above, gave a final R of 6·21%. The atomic coordinates and vibration parameters with their e.s.d.'s are given in Table 1.*

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Table 1. *Fractional coordinates* ($\times 10^4$) *and vibration parameters* (Å$^2\times 10^3$) *and their e.s.d.'s*

The temperature factors are in the form $\exp[-2\pi^2(U_{11}h^2a^{*2}+U_{22}k^2b^{*2}+U_{33}l^2c^{*2}+2U_{23}klb^*c^*+2U_{31}lhc^*a^*+2U_{12}hka^*b^*)]$ or $\exp[-2\pi^2U_{iso}(2\sin\theta/\lambda)^2]$.

	x	y	z	U_{11}	U_{22}	U_{33}	U_{23}	U_{31}	U_{12}
Pt	1286·4 (5)	3164·6 (5)	3553·1 (2)	41·6 (2)	36·2 (3)	41·6 (3)	8·0 (4)	17·3 (2)	10·9 (3)
P(1)	2718 (3)	3742 (3)	3163 (1)	45 (2)	38 (2)	41 (2)	3 (2)	18 (2)	6 (2)
P(2)	2343 (3)	2504 (3)	4330 (1)	44 (2)	40 (2)	46 (2)	7 (2)	19 (2)	10 (2)