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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: BK1214). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## *trans*-Di- $\mu$ -chloro-bis[chloro(trimethylphosphine-*P*)platinum(II)]

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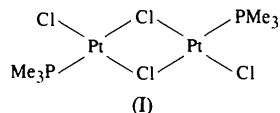
### Abstract

The title compound, [Pt<sub>2</sub>Cl<sub>4</sub>(C<sub>3</sub>H<sub>9</sub>P)<sub>2</sub>], is shown to have a *trans* conformation.

### Comment

Although the title compound, (I), has been used extensively in the synthesis of mononuclear (trimethyl-

phosphine)platinum compounds *via* cleavage of the bridging Pt—Cl bonds, the structure has not been reported previously. Previous structure analyses of related dimers (Black, Mais & Owston, 1969; Simms, Shang, Lu, Youngs & Ibers, 1987; Blake, Gould, Marr, Rankin & Schröder, 1989) have shown them to be centrosymmetric, necessitating a *trans* configuration, now shown to be a feature of the structure of [Pt<sub>2</sub>( $\mu$ -Cl)<sub>2</sub>Cl<sub>2</sub>{P(CH<sub>3</sub>)<sub>3</sub>}<sub>2</sub>].



The relative *trans* influences of the chloride and trimethylphosphine ligands would suggest this to be the thermodynamically favourable isomer and result in some asymmetry in the Pt<sub>2</sub>( $\mu$ -Cl)<sub>2</sub> unit; the Pt— $\mu$ -Cl distance *trans* to Pt—P is 2.423 (3) Å and that *trans* to Pt—Cl is 2.309 (3) Å. The orientation of the PMe<sub>3</sub> group is such that the C2 atom is within 0.038 Å of the plane defined by atoms Cl1, Cl2, Pt and P1. The resulting steric interference is presumably the reason for the observed widening of the Cl1—Pt1—P1 angle to 95.10(10)° (Black, Mais & Owston, 1969).

Details of the synthesis and NMR data (<sup>31</sup>P{<sup>1</sup>H} and <sup>195</sup>Pt{<sup>1</sup>H}) for the title compound and related chloro-bridged dimers have already been presented previously (Boag & Ravetz, 1995).

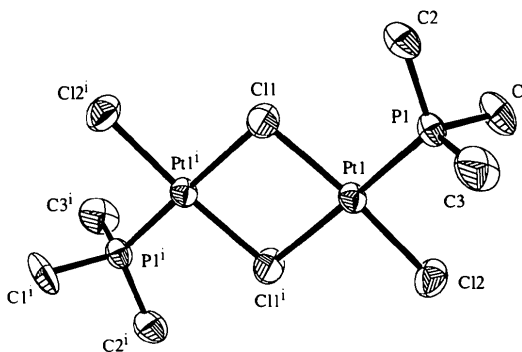


Fig. 1. The molecular structure of [Pt<sub>2</sub>( $\mu$ -Cl)<sub>2</sub>Cl<sub>2</sub>{P(CH<sub>3</sub>)<sub>3</sub>}<sub>2</sub>]. Displacement ellipsoids are drawn at the 50% probability level.

### Experimental

The crystal of the title complex used for analysis was grown by slow evaporation of a saturated CDCl<sub>3</sub> solution of [Pt<sub>2</sub>( $\mu$ -Cl)<sub>2</sub>Cl<sub>2</sub>{P(CH<sub>3</sub>)<sub>3</sub>}<sub>2</sub>].

#### Crystal data

[Pt<sub>2</sub>Cl<sub>4</sub>(C<sub>3</sub>H<sub>9</sub>P)<sub>2</sub>]  
M<sub>r</sub> = 684.12

Mo K $\alpha$  radiation  
 $\lambda$  = 0.71073 Å

Monoclinic  
 $P2_1/n$   
 $a = 6.5940 (10) \text{ \AA}$   
 $b = 10.763 (2) \text{ \AA}$   
 $c = 11.649 (2) \text{ \AA}$   
 $\beta = 105.360 (10)^\circ$   
 $V = 797.2 (2) \text{ \AA}^3$   
 $Z = 2$   
 $D_x = 2.850 \text{ Mg m}^{-3}$   
 $D_m$  not measured

#### Data collection

Nicolet  $R3m/V$  diffractometer with locally modified  $\psi$  box  
 Profile data from  $\theta/2\theta$  scans  
 Absorption correction: semi-empirical via  $\psi$  scan (SHELXTL-Plus; Sheldrick, 1987)  
 $T_{\min} = 0.261$ ,  $T_{\max} = 1.000$   
 2668 measured reflections  
 1936 independent reflections

#### Refinement

Refinement on  $F^2$   
 $R(F) = 0.0426$   
 $wR(F^2) = 0.1131$   
 $S = 1.032$   
 1936 reflections  
 67 parameters  
 Only coordinates of H atoms refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0618P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$

Cell parameters from 25 reflections  
 $\theta = 15\text{--}30^\circ$   
 $\mu = 18.367 \text{ mm}^{-1}$   
 $T = 293 (2) \text{ K}$   
 Rhombohedral block  
 $0.40 \times 0.15 \times 0.05 \text{ mm}$   
 Yellow

1479 observed reflections  
 $[I > 2\sigma(I)]$   
 $R_{\text{int}} = 0.0990$   
 $\theta_{\text{max}} = 28.07^\circ$   
 $h = -1 \rightarrow 8$   
 $k = -14 \rightarrow 1$   
 $l = -15 \rightarrow 15$   
 3 standard reflections monitored every 97 reflections  
 intensity decay: none

$(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 2.14 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -1.62 \text{ e \AA}^{-3}$   
 Extinction correction: none  
 Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{\text{eq}}$
Pt1	0.11762 (6)	0.10541 (4)	0.11383 (3)	0.0335 (2)
Cl1	-0.1500 (5)	-0.0395 (4)	0.0794 (3)	0.0600 (9)
Cl2	0.3836 (5)	0.2455 (4)	0.1314 (3)	0.0578 (8)
P1	0.0863 (4)	0.1609 (3)	0.2905 (2)	0.0359 (6)
C1	0.040 (2)	0.3240 (12)	0.3008 (14)	0.064 (4)
C2	-0.1192 (18)	0.0845 (14)	0.3373 (12)	0.057 (3)
C3	0.318 (2)	0.1269 (17)	0.4093 (11)	0.067 (4)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Pt1—P1	2.205 (3)	Pt1—Cl1	2.309 (3)
Pt1—Cl2	2.281 (3)	Pt1—Cl1'	2.423 (3)
P1—Pt1—Cl12	89.81 (11)	Cl1—Pt1—Cl1'	83.81 (10)
P1—Pt1—Cl1	95.10 (10)	Pt1—Cl1—Pt1'	96.19 (10)
Cl2—Pt1—Cl1'	91.28 (11)		

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

Data collection: *P3* software. Cell refinement: *P3* software. Data reduction: *XDISK* (Siemens, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1987). Software used to prepare material for publication: *SHELXL93*.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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### A Mixed-Valence Tetranuclear Vanadium(IV,V) Complex, $[\text{V}_4\text{O}_4(\mu\text{-OEt})_2(\mu\text{-O})_2(\text{OEt})_4(\text{phen})_2]$

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#### Abstract

The mixed-valence tetranuclear title complex, di- $\mu$ -ethoxo-1:2 $\kappa^4$ O-tetraethoxo-3 $\kappa^2$ O,4 $\kappa^2$ O-di- $\mu$ -oxo-1:3 $\kappa^2$ O;-2:4 $\kappa^2$ O-tetraoxo-1 $\kappa$ O,2 $\kappa$ O,3 $\kappa$ O,4 $\kappa$ O-bis(1,10-phenanthroline)-1 $\kappa^2$ N,N';2 $\kappa^2$ N,N'-tetravanadium,  $[\text{V}_4\text{O}_6(\text{C}_2\text{H}_5\text{O})_6(\text{C}_{12}\text{H}_8\text{N}_2)_2]$ , was prepared and the crystal structure analysis showed it to have an alkoxo-bridged dinuclear oxovanadium core. The complex has a crystallographic inversion centre.

#### Comment

The coordination chemistry of polynuclear vanadium complexes has received considerable attention in recent