

P1—Pt—Pt <sup>i</sup>	91.5 (2)	C41—P2—Pt <sup>i</sup>	112.4 (6)
P2 <sup>ii</sup> —Pt—Pt <sup>ii</sup>	97.1 (2)	C51 <sup>i</sup> —P3—C51	107.0 (6)
P1—Pt—Pt <sup>ii</sup>	150.0 (2)	C51—P3—Au	111.8 (6)
Pt <sup>i</sup> —Pt—Pt <sup>ii</sup>	60.0	P2—C1—P1	109.3 (12)
P2 <sup>ii</sup> —Pt—Au	106.3 (2)	C12—C11—P1	116.5 (9)
P1—Pt—Au	115.2 (2)	C16—C11—P1	123.3 (9)
Pt <sup>i</sup> —Pt—Au	61.09 (2)	C22—C21—P1	118.5 (10)
C21—P1—C11	106.2 (8)	C26—C21—P1	121.5 (10)
C21—P1—C1	102.7 (10)	C32—C31—P2	117.7 (9)
C11—P1—C1	103.8 (10)	C36—C31—P2	121.7 (9)
C21—P1—Pt	122.6 (7)	C42—C41—P2	119.6 (9)
C11—P1—Pt	111.3 (6)	C46—C41—P2	120.2 (9)
C1—P1—Pt	108.5 (8)	C52—C51—P3	118.2 (9)
C1—P2—C31	102.5 (10)	C56—C51—P3	121.6 (9)
Au—P3—C51—C52	31.3 (9)	C1—P2—Pt <sup>i</sup> —Pt	12.8 (8)
Pt—P1—C1—P2	53.6 (13)	P2—Pt <sup>i</sup> —Pt—P1	13.4 (2)
P1—C1—P2—Pt <sup>i</sup>	-40.9 (13)	Pt <sup>i</sup> —Pt—P1—C1	-37.1 (8)

Symmetry codes: (i)  $z, x, y$ ; (ii)  $y, z, x$ .

Data collection and cell refinement were performed with *CAD-4 EXPRESS* (Enraf-Nonius, 1992). *GX* (Mallinson & Muir, 1985) was used for data reduction. The structure was solved by Patterson and Fourier methods (*SHELXS86*; Sheldrick, 1985). Refinement was performed on  $F^2$  for unique reflections with  $I > 3\sigma(I)$ .  $wR$  factors and all goodnesses of fit  $S$  are based on  $F^2$ , conventional  $R$  factors are based on  $F$ . Preliminary studies at 296 K established that at ambient temperature (1) crystallizes in an  $F$ -centred cubic cell, space group  $Fd\bar{3}$ , with  $a = 40.69$  (1) Å. Using 890 unique observed reflections [ $I > 3\sigma(I)$ ] the structure was refined to  $R = 0.057$ . This refinement revealed a cation structure indistinguishable from that reported here, but it accounted for only 16 of the 32 anions necessary for charge balance. Accordingly, the crystal was cooled to 130 K (Oxford Cryosystems Cryostream Cooler; Cosier & Glazer, 1986) and a second data set (unique reflections only) was collected after it had been established that no change in structure occurred on cooling. This data set allowed the remaining anions to be located. In the final calculations anisotropic displacement parameters were refined only for Pt, Au and P atoms; the five phenyl rings were constrained to be rigid hexagons of side 1.39 Å. H atoms rode on the parent C atoms with the constraints  $C-H = 0.95$  Å and  $U(H) = 1.2U_{eq}(C)$ . The disorder of the six F atoms attached to P21 was accounted for by assigning fixed site occupancies of 3/8, 3/8 and 1/4 for F21, F22 and F23, respectively. The *GX* package was used for most of the refinement calculations, but the final cycles of refinement and CIF preparation were performed with *SHELXL93* (Sheldrick, 1993). Molecular graphics were prepared using *ORTEP* (Johnson, 1965).

We wish to thank EPSRC for equipment grant H24280 and Professor R. J. Puddephatt for crystals and stimulating collaboration.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry, along with figures of the anions, have been deposited with the IUCr (Reference: BM1016). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1996). **C52**, 297–300

## Dichloro(ethylenediaminetetraacetic acid- $N,N'$ )platinum(II)–Water (1/6)

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(Received 20 February 1995; accepted 7 July 1995)

## Abstract

The Pt atom of the title compound,  $[\text{PtCl}_2(\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_8)] \cdot 6\text{H}_2\text{O}$ , sits on a twofold axis and exhibits square-planar coordination geometry. Two  $\text{Cl}^-$  ions are bound to the Pt atom, as are the two N atoms of ethylenediaminetetraacetic acid. The distances and angles are typical.

## Comment

Ethylenediaminetetraacetic acid ( $\text{H}_4\text{edta}$ ) is widely recognized as a metal complexing agent and generally binds to metals in either a penta- or hexadentate fashion (Anderegg, 1987). However, in 1956  $\text{H}_4\text{edta}$  was first reported to bind to  $\text{Pt}^{\text{II}}$  and  $\text{Pd}^{\text{II}}$  in a bidentate fashion, and  $\text{H}_2\text{edta}^{2-}$  was reported to bind to these same metal ions in a tetradentate fashion (Busch & Bailar, 1956). These types of complexes have continued to attract the interest of chemists, as evidenced by the fact that various  $\text{Pt}^{\text{II}}$  and  $\text{Pd}^{\text{II}}$  edta complexes have been the subject of numerous NMR studies (Erickson, McDonald, Howie & Clow, 1968; Smith & Sawyer, 1969; Appleton, Hall

& Williams, 1982; Basallote, Vilaplana & Gonzalez-Vilchez, 1987). However, there has only been a single crystallographic study. The crystal structure of the Pd<sup>II</sup> complex [PdCl<sub>2</sub>(H<sub>4</sub>edta)]·5H<sub>2</sub>O was determined using film data, and the Pt<sup>II</sup> complex was reported to be isomorphous and probably isostructural (Robinson & Kennard, 1967, 1970). We report herein the structure of [PtCl<sub>2</sub>(H<sub>4</sub>edta)]·6H<sub>2</sub>O determined from diffractometer data.

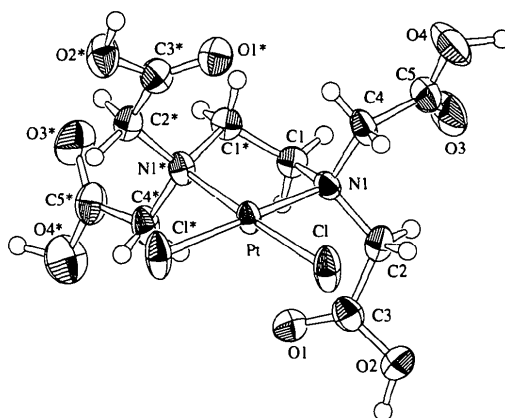
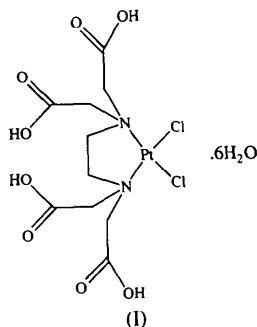


Fig. 1. Perspective view of [PtCl<sub>2</sub>(H<sub>4</sub>edta)] with the atomic numbering. The ellipsoids are drawn at the 50% probability level.

be converted to the right-handed indexing system by negating the values of *k*. We examined the present structure both in *P*4<sub>1</sub>2<sub>1</sub>2 and *P*4<sub>3</sub>2<sub>1</sub>2 and conclude that the latter gives the correct stereochemistry for the Pt compound. The *R* value changes from 0.0511 to 0.0416 (*wR* from 0.0666 to 0.0527) for the two space groups, respectively.

Both the Pd complex (Robinson & Kennard, 1967, 1970) and the Pt complex (Busch & Bailar, 1956) have been reported to contain five water molecules. However, the hydrogen-bonding distances in the present compound with six water molecules are all reasonable (the closest approach for each water molecule is *ca* 2.65 Å), giving us no reason to question the occupancy of these water molecules, except for the previous reports. We subsequently tested models for the water using two O atoms at 0.75 occupancy (for O6 and O7), analogous to the Pd complex (Robinson & Kennard, 1967, 1970). *R* increased slightly. Consequently, we refined the populations of all three water molecules. *R* decreased very slightly (to 0.0413, *wR* = 0.0524) giving final occupancies of 1.13 (5), 1.04 (5) and 1.06 (6) for O5, O6, and O7, respectively. We conclude that the X-ray data are most consistent with the hexahydrate formulation as given in the title, and the data reported are for unit occupancies of O5–O7.

### Experimental

Na<sub>2</sub>H<sub>2</sub>edta·2H<sub>2</sub>O (0.1345 g, 0.361 mmol) and KOH (0.0405 g, 0.723 mmol) were dissolved in 1 ml of water. K<sub>2</sub>PtCl<sub>4</sub> (0.150 g, 0.361 mmol) was added. After stirring for two hours, the solution became cloudy. More KOH (0.0405 g, 0.723 mmol) was added to give a clear orange solution. This solution was evaporated to dryness giving a glassy orange solid. This solid was redissolved in hot water, followed by addition of concentrated aqueous HCl. This produced yellow microcrystals. These were recrystallized from hot concentrated HCl (Whalen, 1994).

The structure consists of a Pt<sup>2+</sup> ion with approximately square-planar coordination to two Cl<sup>-</sup> ions and two N atoms of H<sub>4</sub>edta. The Pt<sup>2+</sup> ion lies on a twofold axis which relates the Cl and N atoms (Fig. 1). The Pt—Cl distance falls within the typical range for Cl *trans* to N: 2.288 (8) Å in Pt(ethylenediamine)Cl<sub>2</sub> (Iball, MacDougall & Scrimgeour, 1975) and 2.303 (4) and 2.317 (4) Å in Pt(*N,N*-dimethylethylenediamine)Cl<sub>2</sub> (Melanson, de la Chevrotiere & Rochon, 1987). The Pt—N distance also falls within the typical range for similar bonds: 2.08 (3) Å in Pt(ethylenediamine)Cl<sub>2</sub> (Iball *et al.*, 1975) and 2.039 (14) and 2.067 (12) Å in Pt(*N,N*-dimethylethylenediamine)Cl<sub>2</sub> (Melanson *et al.*, 1987). The distances within the edta moiety are typical (Kennard, 1967; Shimoi *et al.*, 1980; Stephens, 1969; Smith & Hoard, 1959; Richards, Pederson, Silverton & Hoard, 1964) and are indistinguishable from the values found for [PdCl<sub>2</sub>(H<sub>4</sub>edta)]·5H<sub>2</sub>O (Robinson & Kennard, 1967, 1970) with the exception of the C=O bonds. In the present Pt complex there are pronounced differences between the C—OH bonds and the C=O bonds. Similar differences are observed for metal complexes that contain one or two protonated carboxylate arms in addition to the deprotonated (coordinated) arms (Smith & Hoard, 1959; Kennard, 1967; Stephens, 1969). In the Pd complex, the C—OH and C=O distances are more similar [1.31 (3) and 1.33 (3) Å for C—OH; 1.24 (3) and 1.25 (3) Å for C=O (Robinson & Kennard, 1967, 1970)].

The Pd complex has been reported (Robinson & Kennard, 1967, 1970) in the space group *P*4<sub>1</sub>2<sub>1</sub>2. While there was no report of any stereochemical determination, it was noted that the systematic absences also match the space group *P*4<sub>3</sub>2<sub>1</sub>2, and that the reflections could

**Crystal data**

[PtCl<sub>2</sub>(C<sub>10</sub>H<sub>16</sub>N<sub>2</sub>O<sub>8</sub>)]·6H<sub>2</sub>O  
*M<sub>r</sub>* = 666.33  
 Tetragonal  
*P*4<sub>3</sub>2<sub>1</sub>2  
*a* = 10.220 (1) Å  
*c* = 20.973 (2) Å  
*V* = 2190.6 (6) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 2.020 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda = 0.7107$  Å  
 Cell parameters from 25 reflections  
 $\theta = 12.62$ – $13.71$ °  
 $\mu = 6.782$  mm<sup>-1</sup>  
*T* = 294.2 K  
 Octahedron  
 0.60 × 0.50 × 0.20 mm  
 Yellow

**Data collection**

AFC-7R diffractometer  
 $\omega/2\theta$  scans  
 Absorption correction:  
 $\psi$  scan (TEXSAN;  
 Molecular Structure  
 Corporation, 1985)  
*T<sub>min</sub>* = 0.385, *T<sub>max</sub>* =  
 0.993  
 1545 measured reflections  
 1545 independent reflections  
 1333 observed reflections  
 [*I* > 3σ(*I*)]

$\theta_{\max} = 27.48$ °  
 $h = 0 \rightarrow 13$   
 $k = 0 \rightarrow 9$   
 $l = 0 \rightarrow 27$   
 3 standard reflections  
 monitored every 150  
 reflections  
 intensity decay: 1.19%

**Refinement**

Refinement on *F*<sup>2</sup>  
*R* = 0.0416  
*wR* = 0.0527  
*S* = 1.989  
 1333 reflections  
 133 parameters  
 H-atom parameters not  
 refined  
 $w = 4F^2/\sigma^2(F^2)$  (see below)  
 $(\Delta/\sigma)_{\max} = 0.039$

$\Delta\rho_{\max} = 2.25$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -2.58$  e Å<sup>-3</sup>  
 Extinction correction:  
 Zachariasen (1967) type  
 2 Gaussian isotropic  
 Extinction coefficient:  
 $1.53(13) \times 10^{-6}$   
 Atomic scattering factors  
 from *International Tables*  
 for *X-ray Crystallography*  
 (1974, Vol. IV)

O3—C5	1.23 (2)	C2—C3	1.50 (2)
O4—C5	1.30 (2)	C4—C5	1.53 (2)
Cl—Pt—Cl <sup>i</sup>	90.5 (1)	N1—C1—C1 <sup>i</sup>	109.6 (7)
Cl—Pt—N1	91.9 (2)	N1—C2—C3	114.9 (9)
Cl—Pt—N1 <sup>i</sup>	177.0 (3)	O1—C3—O2	124 (1)
N1—Pt—N1 <sup>i</sup>	85.8 (5)	O1—C3—C2	128 (1)
Pt—N1—C1	106.5 (6)	O2—C3—C2	107 (1)
Pt—N1—C2	113.0 (6)	N1—C4—C5	117 (1)
Pt—N1—C4	106.5 (6)	O3—C5—O4	125 (1)
C1—N1—C2	109.8 (8)	O3—C5—C4	125 (1)
C1—N1—C4	112.0 (8)	O4—C5—C4	109 (1)
C2—N1—C4	109.0 (8)		

Symmetry code: (i) 1 - *y*, 1 - *x*, - $\frac{1}{2}$  - *z*.

Water H atoms were not located, and those belonging to H<sub>2</sub>edta were placed in calculated positions. In the weighting scheme,  $\sigma^2(F^2) = S^2(C + R^2B) + (0.040F^2)^2/Lp^2$  where *S* = scan rate, *C* = total integrated peak count, *R* = ratio of scan to background counting times, *B* = total background count, and *Lp* = Lorentz-polarization factor. The residual electron density is associated with the Pt atom.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1992). Cell refinement: *MSC/AFC Diffractometer Control Software*. Data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1985). Program(s) used to solve structure: *SAPI91* (Fan, 1991). Program(s) used to refine structure: *TEXSAN LS*. Software used to prepare material for publication: *TEXSAN FINISH*.

We are grateful to the Jacob A. and Frieda M. Hunkele Charitable Trust for partial support of this work, and to the Kresge Foundation for providing the funds for the purchase of the diffractometer used in this work.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1271). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

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

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U<sub>eq</sub></i>
Pt	-0.12774 (3)	1 - <i>x</i>	-1/4	0.03032 (9)
C1	0.0718 (3)	1.1030 (3)	-0.2994 (2)	0.0532 (8)
O1	-0.2792 (9)	1.1161 (9)	-0.3828 (4)	0.048 (2)
O2	-0.1291 (9)	1.1745 (10)	-0.4556 (4)	0.055 (3)
O3	-0.141 (1)	1.5809 (9)	-0.3343 (5)	0.067 (3)
O4	0.011 (1)	1.611 (1)	-0.2588 (5)	0.079 (4)
O5	-0.222 (2)	0.990 (2)	-0.5284 (6)	0.135 (7)
O6	0.083 (2)	1.805 (1)	-0.3356 (9)	0.177 (9)
O7	0.160 (2)	1.228 (3)	-0.133 (1)	0.19 (1)
N1	-0.1603 (9)	1.3057 (8)	-0.2951 (4)	0.031 (2)
C1	-0.3029 (10)	1.3403 (10)	-0.2834 (5)	0.033 (3)
C2	-0.134 (1)	1.301 (1)	-0.3659 (5)	0.038 (3)
C3	-0.191 (1)	1.185 (1)	-0.3998 (5)	0.041 (3)
C4	-0.070 (1)	1.401 (1)	-0.2652 (5)	0.042 (3)
C5	-0.072 (1)	1.542 (1)	-0.2907 (6)	0.050 (4)

Table 2. *Selected geometric parameters (Å, °)*

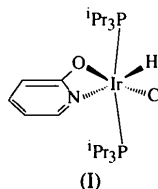
Pt—Cl	2.301 (3)	N1—C1	1.52 (1)
Pt—N1	2.077 (9)	N1—C2	1.51 (1)
O1—C3	1.20 (2)	N1—C4	1.48 (1)
O2—C3	1.33 (1)	C1—C1 <sup>i</sup>	1.50 (2)

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ligand on the metal was strongly implicated by an IR absorption at 2236 cm<sup>-1</sup> and a <sup>1</sup>H NMR resonance at exceptionally high field, δ -25.47 p.p.m. (Muetterties, 1971). However, because neither the coordination mode of the heterocycle nor the stereochemistry about the Ir center were clear from spectral data, an X-ray structure determination was undertaken.



*Acta Cryst.* (1996). **C52**, 300–302

### Chlorohydrido[2(1H)-pyridonato-*N,O*]bis[tris(1-methylethyl)phosphine]iridium(III)

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(Received 5 May 1994; accepted 8 March 1995)

#### Abstract

The title compound, [IrCl(H)(C<sub>5</sub>H<sub>4</sub>NO)(C<sub>9</sub>H<sub>21</sub>P)<sub>2</sub>], is a mononuclear complex of chelated 2(1H)-pyridone exhibiting distorted octahedral geometry, in which the phosphine ligands tilt away from the N atom towards the assumed hydrido ligand.

#### Comment

2(1H)-Pyridonato ligands commonly bridge two metals (Cotton, Fanwick, Niswander & Sekutowski, 1978; Rodman & Mann, 1988; Sherlock, Cowie, Singleton & de V. Steyn, 1989). A more unusual coordination mode involves *N,O*-chelation (Clegg, Berry & Garner, 1980; Rodman & Mann, 1985; Lahuerta, Latorre, Sanaú, Cotton & Schwotzer, 1988; Morrison, Palmer & Tocher, 1988; Leeaphon, Fanwick & Walton, 1991; Steed & Tocher, 1992). A structurally uncharacterized complex was reported to contain both *N,O*-chelating and mono-coordinate pyridonato ligands (Lahuerta, Latorre, Sanaú, Cotton & Schwotzer, 1988). We envisaged that mononuclear complexes of Ir with 2(1H)-pyridone would be formed with a sterically hindered ligand environment. Thus, 2(1H)-pyridone was combined with two moles of <sup>i</sup>Pr<sub>3</sub>P and 0.5 moles of the Ir<sup>I</sup> derivative [(C<sub>8</sub>H<sub>14</sub>)<sub>2</sub>Ir(μ-Cl)]<sub>2</sub> to provide a diamagnetic oxidative addition product whose IR and <sup>1</sup>H NMR spectral properties clearly indicated that an isomer of the title compound, (I), had been formed. In particular, the presence of a hydride

Initial refinement indicated coordination of five non-H atoms to Ir in a distorted octahedral arrangement. However, because IR and NMR spectral evidence point to the presence of a hydrido ligand on the metal, an unsuccessful attempt was made to determine the hydride atom position. The largest Fourier difference peak prior to the final refinement was designated as the missing hydride and its positional and isothermal parameters were allowed to refine. Although the refined hydride position corresponded approximately to the sixth octahedral site in direction, the Ir—H bond distance of 1.18 (12) Å was well outside that of other metal—hydride bond distances established by neutron diffraction (see, for example, Garlaschelli, Khan, Bau, Longoni & Koetzle, 1985). Therefore, we did not consider the hydride position to be accurately determined; a situation that has been encountered by others (Lavin, Holt & Crabtree, 1989; Luo, Schulte & Crabtree, 1990). We subsequently removed the proposed hydride atom and performed the final refinement of the parameters given here.

The most prominent distortions of the complex (shown in Fig. 1) from idealized octahedral geometry include the acute N—Ir—O bond angle of 62.4(3)°, which is an inescapable consequence of chelation of the heterocycle. Furthermore, tilting of the bulky phosphine ligands away from the N atom of the heterocycle and toward the assumed hydride is revealed by large P—Ir—N angles [95.4(2) and 97.8(2)°] and a small P—Ir—P angle [165.4(1)°]. In contrast, the P—Ir—Cl and P—Ir—O angles are near the ideal octahedral value. The N—Ir—O angles are within the normal range shown by the aforementioned *N,O*-chelate complexes and tilting of phosphine ligands towards a hydride has been observed previously (Luo, Schulte & Crabtree, 1990). Among *N,O*-chelate complexes, (I) exhibits unexceptional Ir—Cl and Ir—O bond lengths. The Ir—N distance [2.214(7) Å], however, somewhat exceeds the largest value [2.166(6) Å; Steed & Tocher, 1992] found for this class of complex. The high *trans* influence of the hydrido ligand may be responsible for this lengthening of the Ir—N bond (Lavin, Holt & Crabtree, 1989).