

*Acta Cryst.* (1993). **C49**, 2066–2067

## Structure of Pentaamminechloroiridium(III) Tetrachloroplatinate(II)

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(Received 25 September 1992; accepted 19 May 1993)

### Abstract

The crystal is built up of nearly octahedral  $\text{Ir}(\text{NH}_3)_5\text{Cl}^{2+}$  cations and planar  $\text{PtCl}_4^{2-}$  anions, each of *m* symmetry. The coordination of these ions is 6/6. They are stabilized by electrostatic interactions and there are hydrogen bonds ( $\text{N}-\text{H}\cdots\text{Cl}$ ) which take part in the packing of the structure to form a three-dimensional network.

### Comment

The title compound is one of a series of inorganic platinum–iridium complexes (Garnier, 1993), which was presented as a model for the preparation of reforming catalysts (Michelot, Ouali, Blais, Guérin & Kappenstein, 1988). Our structure determination confirms and refines that proposed by Michelot *et al.* (1988). We used the published coordinates but we grew single crystals by slow diffusion of  $\text{Ir}(\text{NH}_3)_5\text{Cl}_2$  and  $\text{K}_2\text{PtCl}_4$  in a tetramethoxysilane gel.

The complex crystallizes with nearly octahedral  $\text{Ir}(\text{NH}_3)_5\text{Cl}^{2+}$  cations and planar  $\text{PtCl}_4^{2-}$  anions, each of *m* symmetry. The coordination of these ions is 6/6 with interionic distances of 4.72, 4.90, 5.68 ( $\times 2$ ) and 6.15 Å ( $\times 2$ ). The Pt–Cl bond lengths in related structures containing the  $\text{PtCl}_4^{2-}$  ion are in the range 2.293 (1)–2.310 (2) Å (Britten & Lock, 1979; Mais, Owston & Wood, 1972; Ohba, Sato, Saito, Ohshima & Harada, 1983; Delafontaine, Toffoli, Khodadad & Rodier, 1988) which compares favourably with our average value of 2.304 (12) Å. The longer Pt–Cl bond length (Pt–Cl3) is not involved in hydrogen bonding. The Ir atom exhibits a more distorted octahedral conformation than in  $\text{Ir}(\text{NH}_3)_5\text{Cl}_2$  (Hambley & Lay, 1986), but the distortion is comparable with that in  $[\text{Ir}^{\text{III}}(\text{NH}_3)_5\text{Cl}]_2[\text{Pt}^{\text{IV}}\text{Cl}_6]\text{Cl}_2$  (Garnier, 1993). The structure possesses an extended hydrogen-bonding network involving the ammino H atoms and the chlorides (Cl1, Cl2 but not Cl3) of the  $\text{PtCl}_4$  entities:  $\text{N}2-\text{H}23\cdots\text{Cl}1^{\text{ii}} = 3.399$  (5) Å, 158 (5)°;  $\text{N}1-\text{H}12\cdots\text{Cl}1^{\text{ii}} = 3.316$  (7) Å, 142 (4)°;  $\text{N}3-\text{H}32\cdots\text{Cl}1^{\text{iv}}$

$= 3.429$  (6) Å, 162 (3)°;  $\text{N}3-\text{H}33\cdots\text{Cl}2^{\text{iv}} = 3.260$  (6) Å, 110 (3)°. Intramolecular hydrogen bonds are also possible:  $\text{N}2-\text{H}21\cdots\text{N}2^{\text{iii}} = 2.967$  (8) Å, 108 (4)°;  $\text{N}3-\text{H}31\cdots\text{N}3^{\text{iii}} = 2.977$  (8) Å, 92 (3)°;  $\text{N}2-\text{H}21\cdots\text{Cl}4^{\text{i}} = 3.148$  (6) Å, 92 (3)°;  $\text{N}3-\text{H}31\cdots\text{Cl}4^{\text{i}} = 3.135$  (6) Å, 133 (4)°; with the equivalent positions (i)  $x, y, z$ ; (ii)  $\frac{1}{2} - x, -y, -\frac{1}{2} + z$ ; (iii)  $x, \frac{1}{2} - y, z$ ; (iv)  $-x, -y, 1 - z$ .

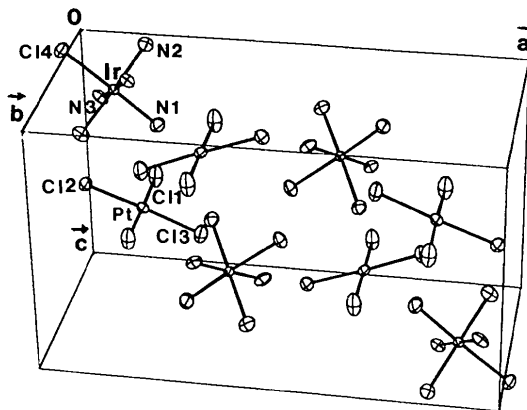


Fig. 1. A MACORTEP (Michalowicz & André, 1991) view of the title compound.

### Experimental

#### Crystal data

$[\text{IrCl}(\text{NH}_3)_5][\text{PtCl}_4]$

$M_r = 649.73$

Orthorhombic

*Pnma*

$a = 16.563$  (2) Å

$b = 8.111$  (1) Å

$c = 9.458$  (2) Å

$V = 1270.7$  (3) Å<sup>3</sup>

$Z = 4$

$D_x = 3.396$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation

$\lambda = 0.71069$  Å

Cell parameters from 25 reflections

$\theta = 9.45-15.35^\circ$

$\mu = 22.57$  mm<sup>-1</sup>

$T = 294$  K

Prismatic

$0.170 \times 0.100 \times 0.094$  mm

Red

#### Data collection

CAD-4L diffractometer

$\omega/2\theta$  scans [width (0.80 + 0.35tan $\theta$ )°]

Absorption correction:

empirical (Walker & Sturart, 1983; Uguzzoli, 1987)

$T_{\text{min}} = 0.789$  ( $\varphi$ ), 0.982

( $\theta$ ),  $T_{\text{max}} = 1.398$  ( $\varphi$ ), 1.085 ( $\theta$ )

6183 measured reflections

2485 independent reflections

1884 observed reflections

$[F > 6\sigma(F)]$

$R_{\text{int}} = 0.0341$

$\theta_{\text{max}} = 35^\circ$

$h = -26 \rightarrow 26$

$k = 0 \rightarrow 13$

$l = 0 \rightarrow 15$

3 standard reflections

monitored every 100

reflections

intensity variation:  $< 2\sigma(I)$

#### Refinement

Refinement on  $F$

$R = 0.0282$

$wR = 0.0302$

$S = 1.537$

$(\Delta/\sigma)_{\text{max}} = 0.059$

$\Delta\rho_{\text{max}} = 1.91$  e Å<sup>-3</sup>

$\Delta\rho_{\text{min}} = -2.17$  e Å<sup>-3</sup>

Extinction correction: none

1884 reflections  
88 parameters  
Only H-atom  $U$ 's refined  
 $w = 1.1433/\sigma^2(F)$

Atomic scattering factors  
from *International Tables  
for X-ray Crystallography*  
(1974, Vol. IV)

Sheldrick, G. M. (1976). *SHELX76 Program for Crystal Structure Determination*. Univ. of Cambridge, England.  
Sheldrick, G. M. (1986). *SHELXS86. Program for the Solution of Crystal Structures*. Univ. of Göttingen, Germany.  
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Walker, N. & Stuart, D. (1983). *Acta Cryst.* **A39**, 158–166.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters ( $\text{\AA}^2$ ) with e.s.d.'s (Schomaker & Marsh, 1983) in parentheses*

$$B_{\text{eq}} = (8\pi^2/3)(U_{11} + U_{22} + U_{33}).$$

	$x$	$y$	$z$	$B_{\text{eq}}$
Pt	0.15462 (2)	0.25	0.65042 (3)	1.426 (5)
Ir	0.10152 (2)	0.25	0.14085 (3)	1.078 (4)
Cl1	0.15298 (9)	-0.0330 (2)	0.6463 (3)	3.35 (4)
Cl2	0.0243 (1)	0.25	0.5671 (3)	1.99 (3)
Cl3	0.2844 (1)	0.25	0.7451 (3)	2.79 (5)
Cl4	-0.0105 (1)	0.25	-0.0140 (3)	2.13 (3)
N1	0.1964 (5)	0.25	0.2829 (9)	2.67 (2)
N2	0.1562 (3)	0.0671 (7)	0.0173 (6)	2.05 (8)
N3	0.0464 (3)	0.0665 (7)	0.2601 (6)	2.18 (9)

Table 2. *Geometric parameters ( $\text{\AA}$ ,  $^\circ$ )*

Pt—Cl1	2.296 (2)	Ir—N1	2.067 (8)
Pt—Cl2	2.298 (2)	Ir—N2	2.094 (6)
Pt—Cl3	2.328 (3)	Ir—N3	2.080 (6)
Ir—Cl4	2.364 (2)		
N2—Ir—Cl4	89.6 (2)	N1—Ir—N3	88.9 (2)
N3—Ir—Cl4	89.5 (2)	Cl1—Pt—Cl2	89.03 (4)
N2—Ir—N3	89.2 (2)	Cl1—Pt—Cl3	91.00 (4)
N1—Ir—Cl4	177.7 (2)	Cl2—Pt—Cl3	177.45 (9)
N1—Ir—N2	92.0 (2)		

Program used to solve structure: *SHELXS86* (Sheldrick, 1985).  
Program used to refine structure: *SHELX76* (Sheldrick, 1976).  
Program used for complete geometry: *PARST* (Nardelli, 1983).  
Chemical analysis: Ir/Pt = 1 (quantitative electron microprobe).  
H atoms refined isotropically (common  $B_{\text{iso}}$ ) with constrained geometry [*DFIX* parameter in *SHELX76*:  $d(\text{N—H}) = 1.01 \text{\AA}$ ].  
Considering  $\Delta\rho_{\text{min}}$  and  $\Delta\rho_{\text{max}}$ , no attempt was undertaken to refine the positional parameters of each H atom.

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 71342 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1026]

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*Acta Cryst.* (1993). **C49**, 2067–2070

## Structure of (Nitrato-*O*)(nitrato-*O,O'*)-tris(pyridazine-*N*)copper(II)

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(Received 19 January 1993; accepted 18 May 1993)

## Abstract

The coordination polyhedron about the copper(II) atom in  $\text{Cu}(\text{pdz})_3(\text{NO}_3)_2$ , where pdz is pyridazine, is a distorted octahedron consisting of three N atoms from the pyridazine ligands, one O atom from the monodentate nitrate group and two O atoms from the unsymmetrically bidentate nitrate group.

## Comment

Studies on pyridine complexes of divalent metal nitrates having the general formula  $M(\text{py})_3(\text{NO}_3)_2$ , where  $M$  is Co, Ni, Cu, Zn or Cd, have been reported by several workers (Choca, Ferraro & Nakamoto, 1972; Cameron, Taylor & Nuttall, 1971; Ouellette & Haendler, 1969; Biagetti & Haendler, 1966; Frank & Rogers, 1966; Rosenthal & Drago, 1965). With the exception of the zinc complex (Cameron, Taylor & Nuttall, 1971), however, their molecular geometries have not been determined unequivocally. Indeed, some workers have even expressed doubt as to the actual existence of  $\text{Cu}(\text{py})_3(\text{NO}_3)_2$  (Biagetti, Bottjer & Haendler, 1966). The lack of definitive structural assignment for these compounds arises from the ability of the nitrate group to function as an ionic species (Procter & Stephens, 1969), a monodentate ligand (Cameron, Taylor & Nuttall, 1972; Scavnicar & Matkovic, 1969), a chelating ligand (Santoro, Mighell & Reimann, 1970; Thompson, Lee & Gabe, 1988) or a bridging ligand (Thompson, Lee & Gabe, 1988; Wallwork & Addison, 1965). In the latter case, more than one bridging mode is possible. Furthermore,