METAL-ORGANIC COMPOUNDS

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Bis(2-chloro-1*H*-imidazol-3-ium) Hexachloroplatinate(IV), $(C_3H_4ClN_2)_2[PtCl_6]$

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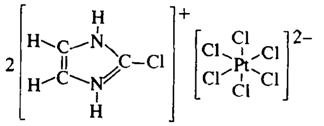
Abstract

The title salt, with a centrally located Pt atom, is comprised of $(C_3H_4ClN_2)^+$ and $[PtCl_6]^{2-}$ ions linked by N···Cl hydrogen bonds of 3.208 (8) Å. The $[PtCl_6]^{2-}$ anions form rows with intermolecular Pt—Cl···Cl—Pt contact distances of 3.681 (2) Å.

Comment

The structures of dichlorobis(2-chloroimidazole)copper(II), dibromobis(2-chloroimidazole)copper(II) and 2-chloroimidazolium aquatrichlorocuprate(II) have been described (Valle *et al.*, 1991, 1993; Valle & Ettorre, 1992). The structure of $(C_3H_4ClN_2)_2[PtCl_6]$, (I), consists of discrete $(C_3H_4ClN_2)^+$ and $[PtCl_6]^{2-}$ ions. Bond lengths at the Pt atom can be compared with that of 2.314 (1) Å in K_2PtCl_6 [(II); Ohba & Saito, 1984] and 2.323 (4) Å in $(H_3O)_2[PtCl_6]$ [(III); Rau *et al.*, 1995]. Each $[PtCl_6]^{2-}$ ion is linked to two cations through N3···Cl12 hydrogen bonds of 3.208 (8) Å. Further interactions between N1 or N3 and coordinated Cl atoms are much weaker, the N···Cl distances being *ca* 3.4 Å or greater. Accordingly, the IR absorption spectrum of (I) displays a band at 3300 cm^{-1}, attributable to the N1–H stretch, superposed on a broad complex band with a maximum at 3170 cm^{-1}, attributable to the N3–H stretch.



(I)

The Pt atom lies on a special position of $2/m$ symmetry, and the Cl12 atom and the atoms of the $(C_3H_4ClN_2)^+$ cations lie on the mirror plane at $z = \frac{1}{2}$. The $[PtCl_6]^{2-}$ anions form rows along the z axis, with intermolecular Cl11···Cl11(x, y, $-z$) contacts of 3.681 (2) Å. Each Cl11 atom is involved in one of these contacts, the Pt—Cl···Cl angle being 135.1 (3)°. It can be noted that intermolecular Cl···Cl contacts of 3.617 (1) and 3.680 (6) Å, respectively, are observed for compounds (II) and (III), with the Pt—Cl···Cl angles being 135°. There are also Cl···Cl contacts in (I) between the $(C_3H_4ClN_2)^+$ and $[PtCl_6]^{2-}$ ions. The contact distances are Cl11···Cl2($-\frac{1}{2}-x$, $\frac{1}{2}+y$, $\frac{1}{2}-z$) 3.540 (3), Cl11···Cl2($-\frac{1}{2}+x$, $-\frac{1}{2}-y$, $-\frac{1}{2}+z$) 3.709 (3) and Cl12···Cl2 3.790 (3) Å.

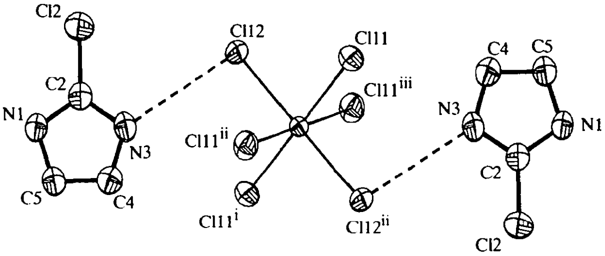


Fig. 1. *ORTEP*II (Johnson, 1976) plot of the stoichiometric unit. Displacement ellipsoids are drawn at the 50% probability level. [Symmetry codes: (i) $-x$, $-y$, $1-z$; (ii) $-x$, $-y$, z; (iii) x, y, $1-z$.]

Experimental

Crystals of (I) were obtained by slow evaporation of a mixture of $C_3H_3ClN_2$ and H_2PtCl_6 (molar ratio 2:1) in concentrated HCl.

Crystal data

$(C_3H_4ClN_2)_2[PtCl_6]$
$M_r = 614.85$
Orthorhombic
Pnnm
$a = 7.596\,(1)$ Å
$b = 15.530\,(2)$ Å
$c = 6.966\,(1)$ Å
$V = 821.8\,(2)$ Å^3
$Z = 2$
$D_x = 2.485$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 7$–$12°$
$\mu = 9.827$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.20 × 0.14 × 0.14 mm
Orange

Data collection

Philips PW1100 diffractometer
θ–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.191$, $T_{\max} = 0.253$
1076 measured reflections
1076 independent reflections

918 reflections with $I > 2\sigma(I)$
$\theta_{\max} = 28°$
$h = 0 \rightarrow 10$
$k = 0 \rightarrow 20$
$l = 0 \rightarrow 9$
3 standard reflections every 50 reflections
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.106$
$S = 1.283$
1076 reflections
60 parameters
Only H-atom U's refined
$w = 1/[\sigma^2(F_o^2) + (0.0673P)^2]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.015$
$\Delta\rho_{\max} = 1.82$ e Å^{-3} (< 1.3 Å from Pt)
$\Delta\rho_{\min} = -3.05$ e Å^{-3} (< 1.3 Å from Pt)
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Pt	0	0	1/2	0.0260 (2)
Cl11	−0.2011 (2)	0.03781 (8)	0.2642 (2)	0.0410 (3)
Cl12	−0.1139 (2)	−0.13918 (11)	1/2	0.0385 (4)
Cl2	0.0512 (4)	−0.3695 (2)	1/2	0.0610 (7)
N1	0.3991 (9)	−0.3520 (4)	1/2	0.046 (2)
C5	0.5245 (11)	−0.2872 (8)	1/2	0.047 (2)
C2	0.2438 (10)	−0.3162 (5)	1/2	0.037 (2)
N3	0.2619 (10)	−0.2335 (5)	1/2	0.057 (2)
C4	0.4368 (13)	−0.2125 (6)	1/2	0.046 (2)

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

Pt—Cl11	2.319 (1)	N1—C5	1.385 (13)
Pt—Cl12	2.328 (2)	C5—C4	1.338 (14)
Cl2—C2	1.681 (8)	C2—N3	1.292 (10)
N1—C2	1.305 (10)	N3—C4	1.368 (12)
Cl11—Pt—Cl11$^{\rm i}$	89.80 (7)	N3—C2—Cl2	125.6 (7)
Cl11—Pt—Cl12	89.44 (5)	N1—C2—Cl2	125.2 (6)
C2—N1—C5	108.2 (7)	C2—N3—C4	109.9 (7)
C4—C5—N1	106.7 (8)	C5—C4—N3	106.1 (8)
N3—C2—N1	109.1 (8)		

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

Data collection: local program (Zanotti, 1983). Cell refinement: local program. Data reduction: local program. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93 and *PARST*83 (Nardelli, 1983).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1347). Services for accessing these data are described at the back of the journal.

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Bis(acetylacetonato-*O*,*O*′)(η^4-*trans*-2,4-hexadiene)ruthenium(II) 1/6-Ethanol Solvate

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Abstract

The title compound, $[\mathrm{Ru(C_5H_7O_2)_2(C_6H_{10})}].1/6\mathrm{C_2H_6O}$, has a pseudo-octahedrally coordinated Ru$^{\rm II}$ center, where two coordination sites are occupied by a diene ligand. The diene ligand exhibits a η^4-*trans*-diene coordination mode. The compound crystallizes in space group $R\bar{3}$, incorporating disordered ethanol in cylindrical voids.

Comment

Current interest in transition metal diene complexes arises from their potential use as synthetic intermediates in the preparation of non-trivial products that are not readily accessible by conventional organic synthesis methods. The relationship between conformation and reactivity patterns of diene ligands attached to metal centers has been studied for many years. In fact, it has been demonstrated that there exist distinct reactivity profiles between a conjugated diene coordinated in a *cis versus* a *trans* conformation (Yasuda & Nakamura, 1987; Nakamura *et al.*, 1983).

While structural studies of metal complexes containing η^4-*cis*-dienes are extensive, structural data concerning η^4-*trans*-diene coordination are limited (Erker *et al.*, 1980, 1985; Kai *et al.*, 1982; Hunter *et al.*, 1985; Meléndez *et al.*, 1988, 1996; Ernst *et al.*, 1991; Sugaya