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Structure of [RuI($C_{10}H_8N_2$)($C_{15}H_{11}N_3$)]PF_6.0·5C_7H_8

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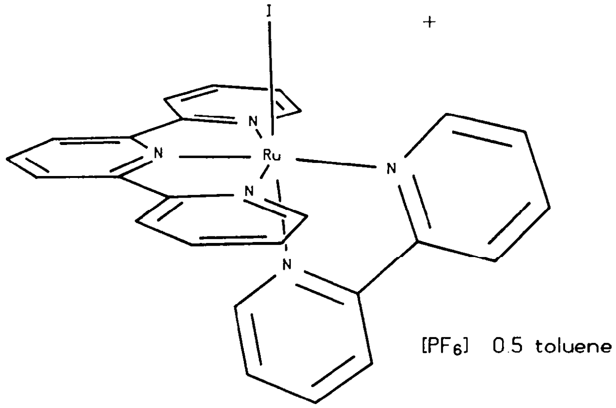
(*Received* 11 *September* 1989; *accepted* 14 *December* 1989)

Abstract. 2,2′-Bipyridyl(iodo)(2,2′:6′,2″-terpyridyl)-ruthenium(II) hexafluorophosphate 0·5 toluene solvate (1), $C_{28\cdot5}H_{23}F_6IN_5PRu$, $M_r = 808{\cdot}51$, monoclinic, $P2_1/n$, $a = 14{\cdot}482$ (3), $b = 12{\cdot}841$ (3), $c = 16{\cdot}100$ (3) Å, $\beta = 94{\cdot}92$ (2)°, $V = 2983$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}80$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}71073$ Å, $\mu = 16{\cdot}56$ cm^{-1}, $F(000) = 1580$, $T = 294$ K, $R = 0{\cdot}0367$ for 3056 observed reflections. The Ru—I distance is 2·711 (1) Å; Ru—N distances range from 1·944 (6) to 2·092 (5) Å. Bite angles are 79·1 (2) and 80·0 (2)° for the terpyridine ligand, and 77·7 (2)° for the bipyridine ligand. Ligand geometries are indicative of considerable strain associated with metal coordination. In addition to the ruthenium cation and a hexafluorophosphate anion, the asymmetric unit contains a toluene solvent molecule which is disordered about an inversion center ($\frac{1}{2}$,0,0).

Experimental. Title compound prepared by heating an ethanol solution containing one equivalent of [Ru($C_{15}H_{11}N_3$)(CF_3SO_3)$_3$], 0·9 equivalents of 2,2′-bipyridine and two equivalents of NH_4I at reflux for 3 h. Addition of saturated KPF_6 solution resulted in a brown precipitate which was collected and chromatographed on N-alumina using an acetonitrile/toluene solution. Deep-purple parallelepiped crystal of (1) obtained by slow evaporation of an acetonitrile/toluene solution, dimensions 0·35 × 0·47 × 0·52 mm. Data collected at room temperature, graphite-monochromated Mo $K\alpha$ radiation ($\lambda = 0{\cdot}71073$ Å), Nicolet $R3m/V$ diffractometer, $\omega/2\theta$ scans of 2–15° min^{-1}, $2\theta_{max} = 45°$, $h = 0$ to 15, $k = 0$ to 13, $l = -17$ to 17, 4317 measurements, 3916 unique ($R_{int} = 0{\cdot}013$), 3056 observed [$I > 3\sigma(I)$]. Unit-cell dimensions determined by least-squares fit to settings for 32 reflections ($16 < 2\theta < 29°$). Empirical absorption correction ($\mu = 16{\cdot}56$ cm^{-1}), transmission factors 0·84–1·00; secondary-extinction correction [$\eta = 0{\cdot}00012$ (3)]; three standards monitored (± 1%), 37·7 h of X-ray exposure.



(1)

Solved by direct methods; full-matrix least-squares refinement on F, $R = 0{\cdot}0367$, $wR = 0{\cdot}0470$, $S = 1{\cdot}33$, 374 variables including positional and anisotropic thermal parameters for non-hydrogen atoms, hydrogen atoms of the cation in idealized positions [d(C—H) = 0·96 Å] with isotropic group thermal parameter [$U_{iso} = 0{\cdot}085$ (6) Å^2], $(\Delta/\sigma)_{max} = 0{\cdot}01$, function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 1/[\sigma^2(F_o) + 0{\cdot}0005|F_o|^2]$.

Final difference map peaks ranged from $-0{\cdot}53$ to 0·82 e Å^{-3}; largest peaks located near the hexafluorophosphate anion and disordered solvent molecule. Computer programs (Sheldrick, 1986), scattering fac-

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0108-2701/90/061105-03$03.00

Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* (Å^2 × 10^3) *for* $[\mathrm{RuI}(\mathrm{C_{10}H_8N_2})(\mathrm{C_{15}H_{11}N_3})]\mathrm{PF_6}.0{\cdot}5\mathrm{C_7H_8}$

	x	*y*	*z*	U_{eq}*
I(1)	2030 (1)	375 (1)	1689 (1)	55 (1)
Ru(1)	1402 (1)	2350 (1)	1449 (1)	41 (1)
P(1)	2264 (2)	892 (2)	6352 (2)	80 (1)
F(1)	2973 (6)	1128 (11)	7048 (6)	230 (7)
F(2)	2643 (7)	−149 (7)	6206 (9)	236 (7)
F(3)	1533 (6)	521 (7)	6923 (5)	165 (4)
F(4)	1830 (6)	2008 (7)	6493 (7)	187 (5)
F(5)	2928 (6)	1371 (8)	5776 (6)	190 (5)
F(6)	1536 (6)	795 (10)	5626 (5)	200 (6)
N(1)	1083 (4)	2407 (4)	2671 (4)	50 (2)
N(2)	150 (4)	1791 (4)	1346 (4)	49 (2)
N(3)	1239 (4)	2087 (4)	187 (3)	49 (2)
N(4)	1068 (4)	3880 (4)	1278 (3)	45 (2)
N(5)	2706 (4)	3057 (5)	1467 (3)	46 (2)
C(1)	1623 (6)	2744 (6)	3326 (5)	63 (3)
C(2)	1334 (9)	2706 (7)	4127 (6)	87 (4)
C(3)	462 (9)	2332 (8)	4239 (6)	89 (4)
C(4)	−87 (7)	1996 (7)	3553 (6)	83 (4)
C(5)	218 (6)	2034 (6)	2787 (5)	60 (3)
C(6)	−308 (5)	1700 (6)	2023 (5)	57 (3)
C(7)	−1220 (6)	1322 (7)	1930 (7)	82 (4)
C(8)	−1596 (6)	1038 (7)	1161 (9)	91 (5)
C(9)	−1108 (6)	1106 (7)	476 (7)	75 (4)
C(10)	−219 (5)	1500 (5)	574 (5)	54 (3)
C(11)	408 (5)	1645 (5)	−72 (5)	54 (3)
C(12)	192 (6)	1381 (6)	−898 (5)	69 (3)
C(13)	809 (8)	1571 (8)	−1462 (6)	88 (4)
C(14)	1638 (8)	2025 (8)	−1219 (6)	90 (4)
C(15)	1840 (6)	2278 (7)	−388 (5)	68 (3)
C(16)	207 (6)	4261 (6)	1269 (5)	63 (3)
C(17)	.17 (7)	5288 (7)	1146 (6)	83 (4)
C(18)	712 (8)	5949 (7)	1020 (6)	94 (5)
C(19)	1597 (7)	5565 (7)	1012 (6)	80 (4)
C(20)	1755 (5)	4531 (6)	1160 (4)	51 (3)
C(21)	2680 (5)	4059 (6)	1231 (4)	51 (3)
C(22)	3479 (6)	4594 (7)	1078 (5)	69 (3)
C(23)	4310 (6)	4114 (8)	1193 (6)	77 (4)
C(24)	4331 (5)	3113 (8)	1467 (5)	71 (3)
C(25)	3531 (5)	2585 (7)	1597 (5)	65 (3)
C(26)	6106 (17)	−388 (18)	510 (15)	106 (7)
C(27)	5661 (14)	−97 (15)	1237 (12)	182 (7)
C(28)	4776 (16)	151 (17)	1047 (14)	95 (6)
C(29)	4313 (10)	271 (10)	305 (9)	118 (4)
C(30)	4781 (21)	−35 (22)	−431 (17)	127 (9)

* Equivalent isotropic *U* defined as one third of the trace of the orthogonalized U_{ij} tensor. [Atoms C(26)–C(30) model the disordered toluene molecule and were refined isotropically; atoms C(26), C(28) and C(30) are half-occupancy atoms.]

tors (Cromer & Waber, 1974), real and imaginary anomalous-dispersion corrections (Cromer, 1974).

Final atomic coordinates are listed in Table 1,* selected bond distances and angles are given in Table 2, Fig. 1 gives the atom-numbering scheme for the cation.

Table 2. *Selected bond distances* (Å) *and angles* (°) *for* $[\mathrm{RuI}(\mathrm{C_{10}H_8N_2})(\mathrm{C_{15}H_{11}N_3})]\mathrm{PF_6}.0{\cdot}5\mathrm{C_7H_8}$

I(1)—Ru(1)	2·711 (1)	Ru(1)—N(1)	2·061 (6)
Ru(1)—N(2)	1·944 (6)	Ru(1)—N(3)	2·053 (6)
Ru(1)—N(4)	2·036 (6)	Ru(1)—N(5)	2·092 (5)
N(1)—C(1)	1·329 (10)	N(1)—C(5)	1·370 (10)
N(2)—C(6)	1·330 (10)	N(2)—C(10)	1·362 (10)
N(3)—C(11)	1·364 (9)	N(3)—C(15)	1·346 (10)
N(4)—C(16)	1·338 (10)	N(4)—C(20)	1·326 (9)
N(5)—C(21)	1·341 (9)	N(5)—C(25)	1·340 (9)
C(5)—C(6)	1·455 (11)	C(10)—C(11)	1·450 (11)
C(20)—C(21)	1·465 (10)		
I(1)—Ru(1)—N(1)	89·8 (2)	I(1)—Ru(1)—N(2)	88·0 (2)
N(1)—Ru(1)—N(2)	79·1 (2)	I(1)—Ru(1)—N(3)	89·8 (2)
N(1)—Ru(1)—N(3)	159·2 (2)	N(2)—Ru(1)—N(3)	80·0 (2)
I(1)—Ru(1)—N(4)	174·2 (2)	N(1)—Ru(1)—N(4)	91·4 (2)
N(2)—Ru(1)—N(4)	97·8 (2)	N(3)—Ru(1)—N(4)	91·1 (2)
I(1)—Ru(1)—N(5)	96·5 (2)	N(1)—Ru(1)—N(5)	104·3 (2)
N(2)—Ru(1)—N(5)	174·3 (2)	N(3)—Ru(1)—N(5)	96·4 (2)
N(4)—Ru(1)—N(5)	77·7 (2)	Ru(1)—N(1)—C(1)	127·1 (6)
Ru(1)—N(1)—C(5)	113·6 (5)	C(1)—N(1)—C(5)	119·3 (7)
Ru(1)—N(2)—C(6)	119·3 (5)	Ru(1)—N(2)—C(10)	118·0 (5)
C(6)—N(2)—C(10)	122·7 (6)	Ru(1)—N(3)—C(11)	113·3 (5)
Ru(1)—N(3)—C(15)	128·7 (5)	C(11)—N(3)—C(15)	118·1 (6)
Ru(1)—N(4)—C(16)	124·4 (5)	Ru(1)—N(4)—C(20)	117·1 (5)
C(16)—N(4)—C(20)	118·6 (6)	Ru(1)—N(5)—C(21)	114·1 (4)
Ru(1)—N(5)—C(25)	126·7 (5)	C(21)—N(5)—C(25)	118·7 (6)
N(1)—C(5)—C(4)	120·9 (8)	N(1)—C(5)—C(6)	114·0 (7)
C(4)—C(5)—C(6)	125·1 (8)	N(2)—C(6)—C(5)	113·9 (6)
N(2)—C(6)—C(7)	118·3 (8)	C(5)—C(6)—C(7)	127·7 (8)
N(2)—C(10)—C(9)	119·6 (8)	N(2)—C(10)—C(11)	113·5 (6)
C(9)—C(10)—C(11)	127·0 (8)	N(3)—C(11)—C(10)	115·1 (6)
N(3)—C(11)—C(12)	121·1 (7)	C(10)—C(11)—C(12)	123·7 (7)
N(4)—C(20)—C(19)	121·5 (7)	N(4)—C(20)—C(21)	114·8 (6)
C(19)—C(20)—C(21)	123·7 (7)	N(5)—C(21)—C(20)	115·0 (6)
N(5)—C(21)—C(22)	121·5 (7)	C(20)—C(21)—C(22)	123·5 (7)

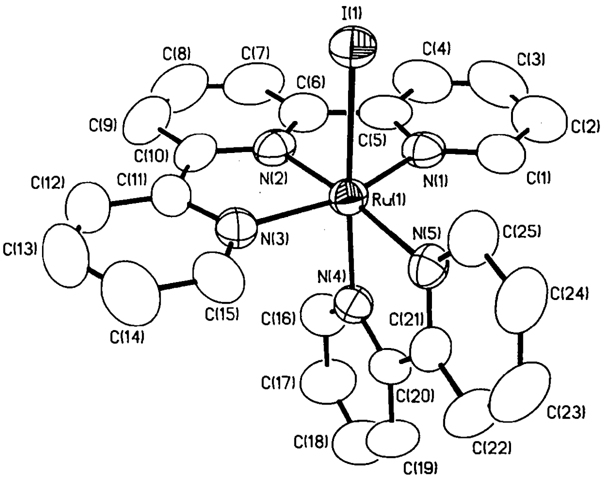

Fig. 1. Thermal ellipsoid plot (50% probability) of the cation; hydrogen atoms are omitted.

Related literature. To our knowledge, this compound represents the first structural analysis of a transition metal coordinated to both a bipyridine and a terpyridine ligand, and also is the first structural study of ruthenium coordinated to terpyridine in a tridentate fashion. In a previously studied compound, dicarbonyl(2,2′:6′,2″-terpyridyl)ruthenium(II) bromide

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* A description of the solvent disorder, a figure of the disordered molecule, tables of crystallographic details, anisotropic thermal parameters, hydrogen-atom coordinates, observed and calculated structure factors, and a complete list of bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52537 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Deacon, Patrick, Skelton, Thomas & White, 1984), the terpyridine ligand was bonded in a bidentate fashion.

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Acta Cryst. (1990). C**46**, 1107–1108

Structure of Bis(ethylenediamine)platinum(II) Dichloride

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(*Received* 4 *December* 1989; *accepted* 23 *January* 1990)

Abstract. $[Pt(C_2H_8N_2)_2]Cl_2$, $M_r = 386{\cdot}2$, triclinic, $P\bar{1}$, $a = 6{\cdot}918\ (4)$, $b = 8{\cdot}378\ (5)$ $c = 4{\cdot}951\ (2)$ Å, $\alpha = 98{\cdot}00\ (6)$, $\beta = 100{\cdot}15\ (6)$, $\gamma = 108{\cdot}57\ (6)^\circ$, $V = 261{\cdot}8\ (2)$ Å^3, $Z = 1$, $D_x = 2{\cdot}25$ Mg m^{-3}, $\lambda(\text{Ag}\ K\alpha) = 0{\cdot}56087$ Å, $\mu = 7{\cdot}5$ mm^{-1}, $F(000) = 180$, $T = 298$ K. $R = 0{\cdot}022$ for 3111 unique reflections. The ethylenediamine moieties coordinate in a square planar manner with amino N atoms attached to the Pt atom at the center of symmetry. The five-membered chelate rings adopt the *meso* form with the ligands in almost symmetric *gauche* (synclinal) conformations. The C atoms are shifted about 0·35 Å above and below the PtNN plane. The complex cations are linked two-dimensionally along the (010) plane by N—H···Cl hydrogen bonds. No short contacts are observed along **b**.

Experimental. Crystals prepared by recrystallization from an aqueous solution of the title compound (Bekaroglu, Breer, Endres, Keller & Nam Gung, 1977). The details of data collection and structure refinement are summarized in Table 1. The structure was solved by the heavy-atom method, and refined by full-matrix least squares. The H atoms were deduced clearly from difference Fourier maps, and included in the refinement. Thermal parameters were anisotropic for the non-H atoms and isotropic for the H atoms. Since two space groups, $P1$ and $P\bar{1}$, were possible, refinements were attempted for both space groups; they converged in quite similar structures with nearly the same R values. Therefore, the centrosymmetric space group $P\bar{1}$ was selected.

A projection of the structure along **c** (in which direction the cations are obliquely stacked) is shown in Fig. 1. The final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 2.† Bond lengths, angles, hydrogen bonds and torsion angles are tabulated in Table 3.

Atomic form factors and f', f'' values taken from *International Tables for X-ray Crystallography* (1974). Calculations performed on a FACOM

Table 1. *Experimental details*

Crystal	Colourless, transparent, plate like; shaped to a sphere ($d = 0{\cdot}37$ mm)
Diffractometer	Rigaku AFC-3, graphite monochromator
Scan	ω mode, width $(2{\cdot}4 + 0{\cdot}8\tan\theta)^\circ$, speed 2°min^{-1}, $2\theta < 60^\circ$
Standards	$06\bar{2}$, 054, 710, $3\bar{7}4$, $4\bar{8}3$; every 50 reflections, no significant fluctuation
Range of *hkl*	$h\ 0\rightarrow 12$, $k\ -14\rightarrow 14$, $l\ -8\rightarrow 8$
Reflections	Measured 3113 Observed 3111, $\|F_o\| > 3\sigma(\|F_o\|)$
Corrections	Lp, absorption ($\mu r = 1{\cdot}39$)
Transmission	0·15 – 0·19
Unit cell	68 reflections, $19{\cdot}5 < \theta < 21{\cdot}5^\circ$
Function minimized	$\sum w(F_o - F_c)^2$, $w = [\sigma^2(F_o) + (0{\cdot}015\|F_o\|)^2]^{-1}$
Parameters refined	66
R, wR, S	0·022, 0·028, 1·04
Max. Δ/σ	0·04
Min. and max. $\Delta\rho$	$-1{\cdot}8$ and $3{\cdot}0$ e Å^{-3} near the Pt atom ($-0{\cdot}5$ and $0{\cdot}4$ e Å^{-3} elsewhere)

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† Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52624 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/90/061107-02$03.00