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Tetra-μ-acetato-*O*:*O*′-bis(quinoline-*N*)diruthenium(II,III) hexafluorophosphate quinoline solvate

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Key indicators

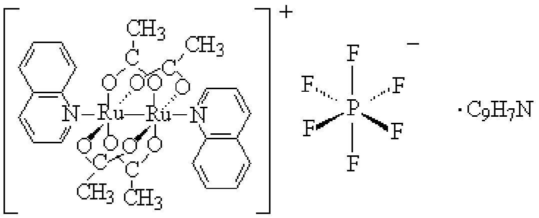
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.012 Å
Disorder in solvent or counterion
R factor = 0.043
wR factor = 0.131
Data-to-parameter ratio = 11.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title complex, $[Ru_2(O_2CCH_3)_4(C_9H_7N)_2]PF_6\cdot C_9H_7N$, shows a binuclear cage structure having an inversion center. There are weak intramolecular C—H···O interactions between the quinoline ligands and the bridging acetate O atoms.

Comment

Only a few *N*-donor heterocycle axial diadducts of diruthenium(II,III) tetracarboxylate, $[Ru_2(O_2CR)_4(N\text{-heterocycle})_2]X$ (R = alkyl or aryl, X = counter-ion), have been structurally characterized. Most of these involve pyridine or pyridine derivatives (Cotton *et al.*, 1998; Vamvounis *et al.*, 2000) and one example of biological relevance employing 2-methylimidazole as the axial ligand (Sudha & Chakravarty, 1998). This latter complex displayed a weak intermolecular hydrogen bonding interaction between the non-bound nitrogen and an oxygen on the perchlorate counterion.



(I)

As $Ru_2(O_2CR)_4Cl$ (R = CH_3 and CH_2CH_3) complexes have shown small but significant antitumor activity (Keppler *et al.*, 1989), we sought to investigate the binding of various nucleoside bases to the diruthenium(II,III) tetracarboxylate core and focus on the structural aspects of this binding, in particular, any intramolecular hydrogen bonding. In addition to looking at nucleoside base adducts, we wanted to study similar and simpler diadducts that were soluble in non-aqueous media. Molecule (I) is one such complex. While it contains typical Ru—Ru, Ru—O and Ru—N bond lengths and angles (Table 1), it displays *trans*-oriented axially bound quinoline rings with weak intramolecular interactions between the acetate O atoms and C—H bonds of bound quinolines. Four of these are given in Table 2, the strongest of which is C12—H12···O3.

Experimental

Quinoline (0.64 mmol) was added dropwise to a 5 ml solution of $[Ru_2(O_2CCH_3)_4(H_2O)_2]PF_6$ (0.16 mmol) in 2-propanol. The solution was stirred for 5 min and the gold–brown product filtered and dried. Crystals could be grown by slow evaporation from dichloromethane.

Crystal data

$[Ru(C_2H_3O_2)_4(C_9H_7N)_2]PF_6\cdot C_9H_7N$
$M_r = 970.75$
Triclinic, $P\overline{1}$
$a = 12.180$ (7) Å
$b = 10.980$ (3) Å
$c = 7.729$ (3) Å
$\alpha = 99.75$ (1)°
$\beta = 108.70$ (2)°
$\gamma = 79.0$ (3)°
$V = 954.6$ (11) Å^3
$Z = 1$
$D_x = 1.689$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 39.5$–40.0°
$\mu = 7.54$ mm^{-1}
$T = 293$ (2) K
Block, red–brown
0.55 × 0.35 × 0.30 mm

Data collection

Rigaku AFC-5*R* diffractometer
ω–2θ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min} = 0.446$, $T_{\max} = 1.000$
3253 measured reflections
3089 independent reflections
2555 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.078$
$\theta_{\max} = 64.9°$
$h = 0 \rightarrow 14$
$k = -12 \rightarrow 12$
$l = -8 \rightarrow 8$
3 standard reflections every 150 reflections intensity decay: 8.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.131$
$S = 1.05$
3089 reflections
260 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0655P)^2 + 3.3237P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 1.10$ e Å^{-3}
$\Delta\rho_{\min} = -0.71$ e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0019 (3)

Table 1
Selected geometric parameters (Å, °).

Ru1—O2	2.020 (5)	Ru1—O4	2.021 (5)
Ru1—O3	2.020 (5)	Ru1—Ru1$^{\rm i}$	2.2816 (17)
Ru1—O1	2.020 (5)	Ru1—N1	2.323 (5)
O2—Ru1—O3	89.5 (2)	Ru1$^{\rm i}$—Ru1—N1	178.24 (13)
O3—Ru1—O1	178.2 (4)	C1—O1—Ru1	119.8 (4)
O3—Ru1—O4	92.0 (2)	C1$^{\rm i}$—O3—Ru1	119.8 (4)
O2—Ru1—Ru1$^{\rm i}$	89.6 (2)	C5—N1—C13	118.4 (5)
O1—Ru1—Ru1$^{\rm i}$	89.4 (2)	C5—N1—Ru1	114.6 (5)
O2—Ru1—N1	90.5 (3)	C13—N1—Ru1	126.9 (4)
O1—Ru1—N1	88.9 (2)		

Symmetry code: (i) $1 - x, -y, 1 - z$.

Table 2
Hydrogen-bonding geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
C5—H5$\cdots$O1	0.93	2.71	3.150 (7)	110
C5—H5$\cdots$O2	0.93	2.79	3.261 (8)	112
C12—H12$\cdots$O3	0.93	2.48	3.199 (9)	135
C12—H12$\cdots$O4	0.93	2.61	3.218 (8)	124

H atoms were allowed to ride on the heavy atoms to which they were bonded with $U_{\rm iso}$ equal to $1.2U_{\rm eq}$ of the heavy atom ($1.5U_{\rm eq}$ for methyl H atoms). The PF_6^- anion was found to be disordered and each fluoride ligand was allowed to occupy two positions, each with an occupancy of 0.5, and with equal atomic displacement parameters for each *A*/*B* pair. The fluoride A/B distances were restrained to 0.80 (2) Å and the P—F distances to 1.58 (2) Å. The incorporated solvent molecule of quinoline was also found to be disordered. The N atom of the solvent was allowed to occupy each of the four possible positions equally, each with an occupancy of 0.25. The bonds across the top of the solvent rings were made equal in length, while the edge bonds (those not involving nitrogen), were restrained to 1.39 (1) Å. Similarly, in the coordinated quinoline molecule the C—C bond lengths were restrained to 1.39 (1) Å. The highest peaks and deepest holes in the final difference map were located at distances less than 1.1 Å from the heavy metal atom.

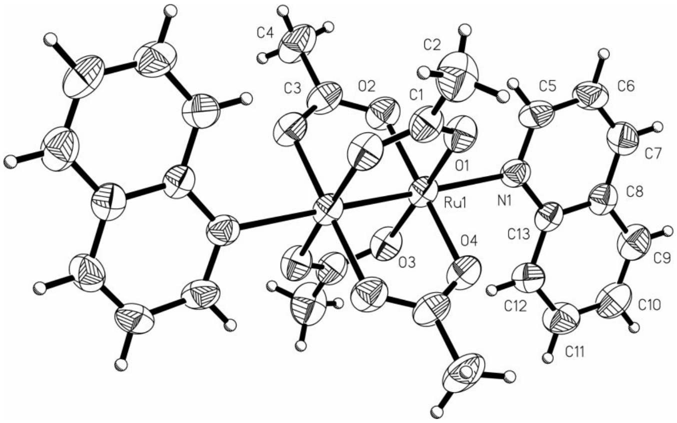


Figure 1
The structure of (I) with displacement ellipsoids drawn at the 50% probability level. The quinoline molecule of solvation and the heaxafluorophosphate counterion have been omitted.

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1994); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN for Windows* (Molecular Structure Corporation, 1997–1999); program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *TEXSAN for Windows*; software used to prepare material for publication: *SHELXL*97.

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