

Tetrakis(μ_2 -anilinopyridinato- $\kappa^2 N:N'$)aqua-diruthenium(II,III) hexafluoroantimonate diethyl ether solvate

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Key indicators

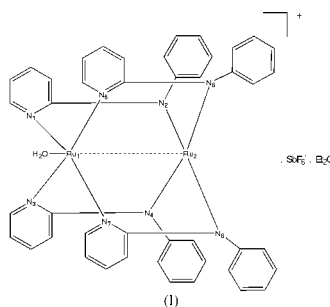
Single-crystal X-ray study
 $T = 293\text{ K}$
 Mean $\sigma(\text{C}-\text{C}) = 0.018\text{ \AA}$
 H-atom completeness 96%
 R factor = 0.086
 wR factor = 0.199
 Data-to-parameter ratio = 13.9

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

The title complex, $[\text{Ru}_2(\text{C}_{11}\text{H}_9\text{N}_2)_4(\text{H}_2\text{O})][\text{SbF}_6] \cdot \text{C}_2\text{H}_{10}\text{O}$, has a diruthenium(II,III) unit bridged by four PhNpy ligands. There is an axial aqua ligand on the ruthenium(III) atom. The $\text{Ru} \cdots \text{Ru}$ distance is $2.288(1)\text{ \AA}$.

Comment

In the chemistry of multiple-bonded diruthenium complexes, a variety of diruthenium compounds have been reported (Cotton & Walton, 1985; Tooze *et al.*, 1984; Chakravarty & Cotton, 1975) many years ago. In most cases, spectroscopic and electrochemical studies on the diruthenium complexes show that modification of the axial ligand usually results in a dramatic shift of the visible band energy and the redox potentials. These complexes of $\text{Ru}^{\text{II}}-\text{Ru}^{\text{III}}$ are quite different from those of other dimetallic species. The cationic species $[\text{Ru}_2(\mu_2\text{-PhNpy})_4 \cdot \text{H}_2\text{O}]^+$ consists of a diruthenium core held together by four PhNpy ligands. The ruthenium(III) atom that is bonded to pyridine N atoms has an axial H_2O , while the axial site on the other ruthenium(II) atom is vacant. The molecular structure of $\text{Ru}_2\text{Cl}(\text{PhNpy})_4$ has already been reported (Chakravarty *et al.*, 1985). The two molecules are very similar in structure, except that the $\text{Ru}-\text{Cl}$ bond in $\text{Ru}_2\text{Cl}(\text{PhNpy})_4$ is replaced by $\text{Ru}-\text{OH}_2$. This change has a small effect on the $\text{Ru}-\text{Ru}$ distance, which changes from $2.275(3)\text{ \AA}$ in $\text{Ru}_2\text{Cl}(\text{PhNpy})_4$ to $2.288(1)\text{ \AA}$ in $[\text{Ru}_2(\text{ap})_4(\text{H}_2\text{O})](\text{SbF}_6)$. The average $\text{Ru}^{\text{II}}-\text{N}$ distance is $2.014(6)\text{ \AA}$, but $\text{Ru}^{\text{III}}-\text{N}$ is $2.056(8)\text{ \AA}$ in the title compound, the $\text{O1}-\text{Ru1}-\text{Ru2}$ angle being $178.80(18)^\circ$.



Experimental

The title compound was synthesized by the reaction of $\text{Ru}_2\text{Cl}(\text{PhNpy})_4$ with a small excess of AgSbF_6 in methanol-dichloromethane in the dark for 12 h. The solution changed from blue to deep red. After filtration, the filtrate was evaporated under vacuum to leave a red residue, which was dissolved in dichloromethane. Well shaped crystals suitable for X-ray diffraction measurement were grown by layering diethyl ether onto the solution.

Crystal data

[Ru₂(C₁₁H₉N₂)₄(H₂O)]·
[SbF₆]₂·C₄H₁₀O
M_r = 1206.83
Monoclinic, P2₁/n
a = 10.1385 (1) Å
b = 22.5901 (5) Å
c = 21.0672 (5) Å
β = 94.860 (1)°
V = 4807.67 (16) Å³
Z = 4

D_x = 1.667 Mg m⁻³
Mo Kα radiation
Cell parameters from 5202
reflections
θ = 1.3–22.5°
μ = 1.25 mm⁻¹
T = 293 (2) K
Block, deep red
0.72 × 0.46 × 0.30 mm

Data collection

Siemens SMART CCD
diffractometer
φ and ω scans
Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)
T_{min} = 0.433, T_{max} = 0.687
15139 measured reflections

8405 independent reflections
5834 reflections with I > 2σ(I)
R_{int} = 0.048
θ_{max} = 25.1°
h = -11 → 11
k = -26 → 24
l = -25 → 14

Refinement

Refinement on F²
R[F² > 2σ(F²)] = 0.086
wR(F²) = 0.199
S = 1.17
8405 reflections
604 parameters
H atoms constrained

w = 1/[σ²(F_o²) + (0.0478P)²
+ 41.132P]
where P = (F_o² + 2F_c²)/3
(Δ/σ)_{max} = 0.001
Δρ_{max} = 0.89 e Å⁻³
Δρ_{min} = -0.75 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Ru1—O1	2.012 (6)	Sb—F4	1.719 (17)
Ru1—N7	2.048 (8)	Sb—F3	1.781 (18)
Ru1—N3	2.054 (8)	Sb—F6	1.820 (17)
Ru1—N1	2.056 (8)	C44—C45	1.369 (17)
Ru1—N5	2.067 (9)	C45—C46	1.366 (15)
Ru1—Ru2	2.2882 (11)	C52—C53	1.422 (15)
Ru2—N8	2.007 (8)	C83—C84	1.357 (19)
Ru2—N6	2.022 (8)	C85—C86	1.385 (16)
O1—Ru1—N7	91.3 (3)	C52—N6—Ru2	120.5 (7)
N7—Ru1—N3	87.9 (3)	C76—N8—C81	109.5
N3—Ru1—N1	90.3 (3)	N1—C12—C13	123.5 (11)
N1—Ru1—N5	91.4 (3)	C14—C15—C16	119.1 (11)
N7—Ru1—Ru2	89.8 (2)	C23—C22—C21	120.1 (11)
N8—Ru2—N2	179.2 (3)	C43—C42—C41	121.0 (11)
N2—Ru2—N4	89.6 (3)	C46—C45—C44	120.8 (11)
N4—Ru2—Ru1	89.5 (2)	C54—C53—C52	120.9 (13)
F4—Sb—F5	91.6 (10)	C64—C63—C62	120.2 (14)
F4—Sb—F2	83.8 (11)	C74—C75—C76	117.9 (12)
F4—Sb—F6	167.6 (12)	C83—C82—C81	119.6 (12)
C12—N1—Ru1	121.9 (7)	C84—C85—C86	120.4 (13)
C36—N4—Ru2	120.8 (6)		

The H atoms were positioned geometrically (C—H bond length fixed at 0.96 Å), assigned isotropic displacement parameters and allowed to ride on their parent C atoms before the final cycle of least-squares refinement.

Data collection: SMART (Siemens, 1994); cell refinement: SMART and SAINT (Siemens, 1996); data reduction: XPREP in SHELXTL

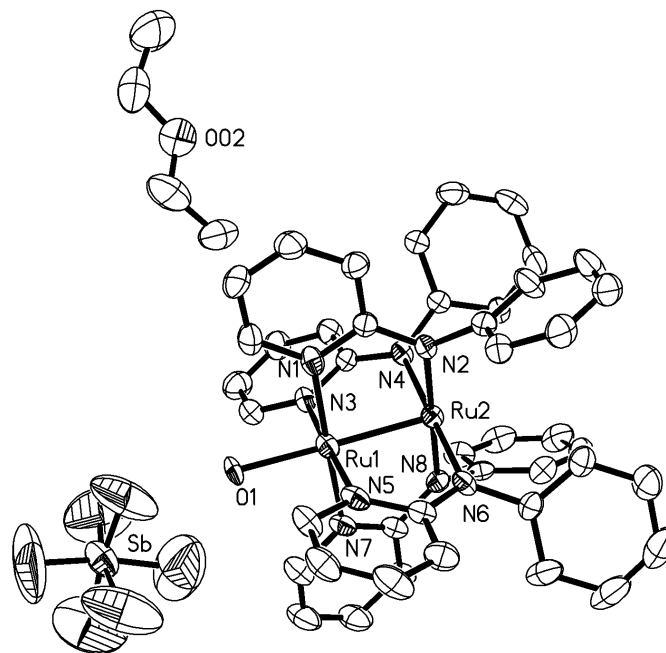


Figure 1

A view of the structure of (I), with partial atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted for clarity.

(Bruker, 1997); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL.

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