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Tetraamminehydroxonitrosylruthenium(III) tetranitropalladate(II) monohydrate

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Key indicators

Single-crystal X-ray study
T = 150 K
Mean σ(O–N) = 0.003 Å
R factor = 0.018
wR factor = 0.045
Data-to-parameter ratio = 12.8

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

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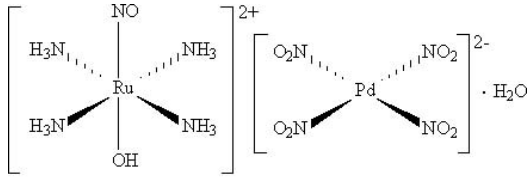
The asymmetric unit of the title compound, $[Ru(OH)(NH_3)_4(NO)][Pd(NO_2)_4]\cdot H_2O$, consists of one complex $[Ru(NO)(NH_3)_4OH]^{2+}$ cation, two half $[Pd(NO_2)_4]^{2-}$ complex anions, each anion located on an inversion centre, and a water molecule of crystallization. The crystal structure exhibits an extensive hydrogen-bonding network.

Comment

Double complex salts containing various platinum metals in the structure of complex cations and anions are among the most promising precursors for obtaining intercalates, cermets and catalysts.



(I)

The structure of title compound, (I), consists of $[Ru(NO)(NH_3)_4OH]^{2+}$ complex cations, two crystallographically independent $[Pd(NO_2)_4]^{2-}$ complex anions and solvent water molecules.

The coordination sphere of the ruthenium centre consists of a hydroxo O atom, four ammine N atoms and one nitroso N atom, forming a practically undistorted octahedron. Complex anions exist in almost ideal square-planar geometry.

The geometric characteristics of the complex cation compare well with those found in earlier studies of $[Ru(NO)(OH)(NH_3)_4][Os(NO)(NO_2)_4(OH)]\cdot 0.5H_2O$ (Salomov *et al.*, 1984), $[Ru(NO)(OH)(NH_3)_4](ReO_4)_2$ (Minacheva *et al.*, 2001), $[Ru(NO)(OH)(NH_3)_4](CrO_4)\cdot H_2O$ (Kokunova *et al.*, 1999) and $[Ru(NO)(NH_3)_4(OH)]Cl_2$ (Bottomley, 1974), with an average distance of 2.106 (5) Å for the Ru—N(NH_3) bond distance in the equatorial plane of the complex. The geometry of the complex anion is the expected square plane of such $M_2[Pd(NO_2)_4]$ complexes (*M* = Ag, K and Na; Gromilov *et al.*, 2003), with an average Pd—N(NO_2) distance of 2.032 [14] Å. The closest contact of the nitroso O atom, O(NO)···O(NO_2), is 2.995 (3) Å. The complex cations and anions are linked to each other and to the water molecules by N—H···O and O—H···O hydrogen bonds. The closest distance between the metal atoms is 5.1720 (3) Å for Ru···Ru and 5.2672 (2) Å for Ru···Pd.

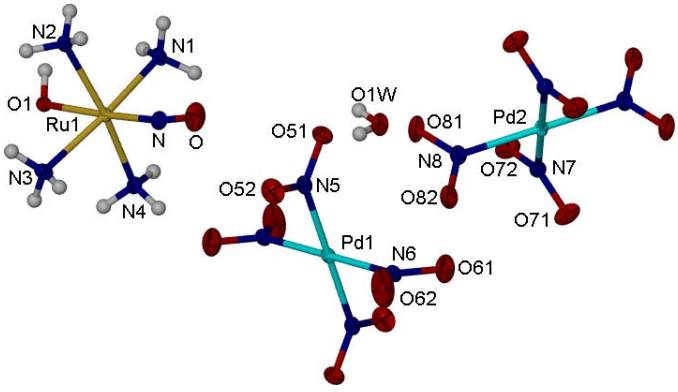


Figure 1
Displacement ellipsoid plot, drawn at the 50% probability level. H atoms are shown as small spheres of arbitrary radii (*BS*; Ozawa & Kang, 2004). In the Pd1 anion, unlabelled atoms are related to labelled atoms by the symmetry code $(1-x, 1-y, 1-z)$. In the Pd2 anion, unlabelled atoms are related to labelled atoms by the symmetry code $(1-x, -y, -z)$.

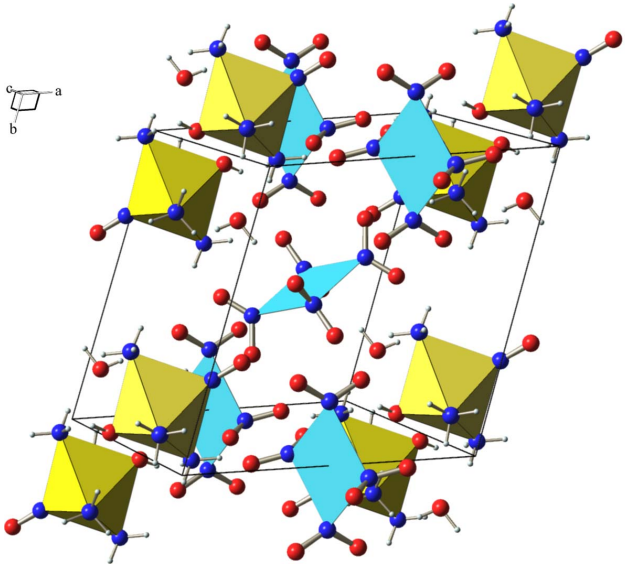


Figure 2
A perspective packing diagram of $[Ru(NO)(NH_3)_4OH][Pd(NO_2)_4]\cdot H_2O$.

Experimental

The title compound was obtained (yield ∼50%) by mixing equal volumes (11.5 ml) of solutions of $[Ru(NO)(NH_3)_4OH]Cl_2$ and $K_2[Pd(NO_2)_4]$, having the same concentration (0.17 mol l^{-1}). Crystals suitable for X-ray diffraction analysis were obtained from the filtrate with residual concentration of the complex (0.047 mol l^{-1}).

Crystal data

$[Ru(OH)(NH_3)_4(NO)]$-$[Pd(NO_2)_4]\cdot H_2O$
M_r = 524.68
Triclinic, $P\overline{1}$
a = 9.0710 (3) Å
b = 10.0861 (5) Å
c = 10.1864 (3) Å
α = 100.147 (1)°
β = 115.390 (1)°
γ = 108.728 (1)°
V = 741.78 (5) Å^3
Z = 2
D_x = 2.349 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 4128 reflections
θ = 2.4–32.5°
μ = 2.30 mm^{-1}
T = 150 (2) K
Prism, light yellow
0.21 × 0.19 × 0.12 mm

Data collection

Bruker–Nonius X8APEX CCD area-detector diffractometer
φ scans
Absorption correction: multi-scan (*SADABS*; Blessing, 1995)
T_{min} = 0.643, T_{max} = 0.770
4633 measured reflections
2756 independent reflections
2535 reflections with $I > 2\sigma(I)$
R_{int} = 0.013
θ_{max} = 25.7°
$h = -11 \rightarrow 10$
$k = -12 \rightarrow 9$
$l = -12 \rightarrow 12$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.045
S = 1.07
2756 reflections
215 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0239P)^2 + 0.2798P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.47 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Ru1—N	1.742 (2)	N5—O52	1.228 (3)
N—O	1.155 (3)	N6—O61	1.241 (3)
Ru1—O1	1.9618 (15)	N6—O62	1.170 (3)
Ru1—N1	2.1062 (19)	Pd2—N7	2.040 (2)
Ru1—N2	2.0995 (18)	Pd2—N8	2.020 (2)
Ru1—N3	2.1095 (19)	N7—O71	1.231 (3)
Ru1—N4	2.1091 (19)	N7—O72	1.212 (3)
Pd1—N5	2.018 (2)	N8—O81	1.226 (3)
Pd1—N6	2.049 (2)	N8—O82	1.241 (3)
N5—O51	1.245 (3)		
Ru1—N—O	171.8 (2)	N1—Ru1—N2	90.39 (8)
N—Ru1—O1	176.99 (8)	N1—Ru1—N4	90.49 (8)
O1—Ru1—N1	89.19 (7)	N2—Ru1—N3	89.00 (8)
O1—Ru1—N2	84.90 (7)	N3—Ru1—N4	89.08 (8)
O1—Ru1—N3	84.35 (7)	N5—Pd1—N6	89.53 (8)
O1—Ru1—N4	85.85 (7)	N7—Pd2—N8	90.52 (8)
N—Ru1—N1	89.84 (9)	O51—N5—O52	120.1 (2)
N—Ru1—N2	92.27 (9)	O61—N6—O62	121.9 (2)
N—Ru1—N3	96.62 (9)	O71—N7—O72	121.9 (2)
N—Ru1—N4	97.00 (8)	O81—N8—O82	119.35 (19)

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1···O81^{i}	0.84	2.55	3.319 (3)	153
O1—H1···O71^{ii}	0.84	2.58	3.025 (2)	115
N1—H1A···O51^{i}	0.91	2.18	3.064 (3)	165
N1—H1B···O52^{iii}	0.91	2.08	2.936 (3)	157
N1—H1C···O82^{iii}	0.91	2.23	3.075 (3)	154
N2—H2A···O72^{iv}	0.91	2.39	2.990 (3)	123
N2—H2B···O1W^{iv}	0.91	2.07	2.974 (3)	170
N2—H2B···O1W^{iv}	0.91	2.07	2.974 (3)	170
N2—H2C···O81^{i}	0.91	2.25	3.085 (3)	152
N2—H2C···O51^{i}	0.91	2.51	3.020 (3)	116
N3—H3B···O1^{v}	0.91	2.14	2.992 (3)	156
N3—H3B···O71^{vi}	0.91	2.54	3.048 (3)	116
N3—H3C···O1W^{iv}	0.91	2.22	3.105 (3)	165
N4—H4A···O62^{iii}	0.91	2.29	3.154 (3)	158
N4—H4B···O1^{v}	0.91	2.06	2.934 (3)	161
N4—H4C···O61^{vi}	0.91	2.19	3.069 (3)	161
O1W—H2···O82^{iii}	0.72 (3)	2.15 (4)	2.873 (3)	177 (4)
O1W—H3···O51	0.85 (4)	2.00 (4)	2.833 (3)	164 (3)

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $x-1, y+1, z$; (iii) $x-1, y, z$; (iv) $x, y+1, z$; (v) $-x, -y+2, -z+1$; (vi) $-x+1, -y+1, -z+1$.

The water H atoms were found in a difference Fourier map and refined freely, while the other H atoms (N—H = 0.91 Å and O—H = 0.84 Å) were placed in calculated positions and refined riding on their parent atoms [U_{iso}(H) = 1.2U_{eq}(O) and 1.5U_{eq}(N)]. The maximum electron-density peak is located 0.98 Å from an N atom.

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2004); program(s) used to refine structure: *SHELXTL*; molecular graphics: *Balls & Sticks* (*BS*) (Ozawa & Kang, 2004); software used to prepare material for publication: *SHELXTL*.

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