

Tetrakis(3-aminopyridine- κN^1)dioxorhenium(V)
iodide 3-aminopyridine solvate dihydrate

Andrzej Kochel

Faculty of Chemistry, University of Wrocław,
14 Joliot-Curie St, 50-383 Wrocław, PolandCorrespondence e-mail:
andrzej@wchuwr.chem.uni.wroc.pl

Key indicators

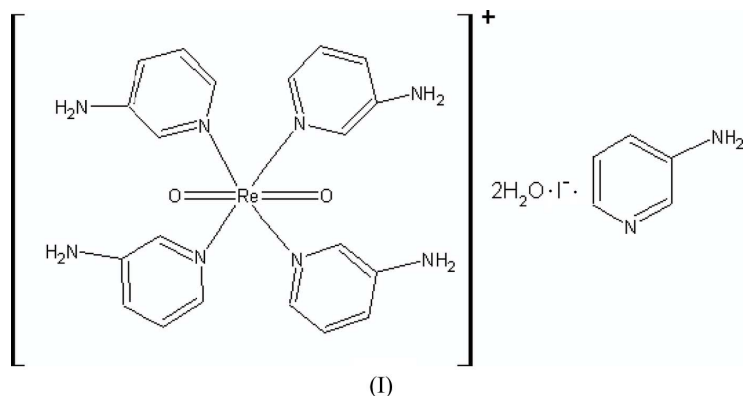
Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(C-C) = 0.006$ Å
 R factor = 0.038
 wR factor = 0.077
Data-to-parameter ratio = 30.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The asymmetric unit of the title compound, $[\text{ReO}_2(\text{C}_5\text{H}_6\text{N}_2)_4]\text{I}\cdot\text{C}_5\text{H}_6\text{N}_2\cdot 2\text{H}_2\text{O}$, contains half each of two tetrakis(3-aminopyridine)dioxorhenium(V) cations, an I^- anion, an uncoordinated 3-aminopyridine molecule and two water molecules. Each cation has a centre of inversion at the Re atom. The crystal structure is stabilized by $\text{O}-\text{H}\cdots\text{I}$, $\text{O}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{O}$, $\text{N}-\text{H}\cdots\text{I}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Received 6 June 2006
Accepted 26 June 2006

Comment

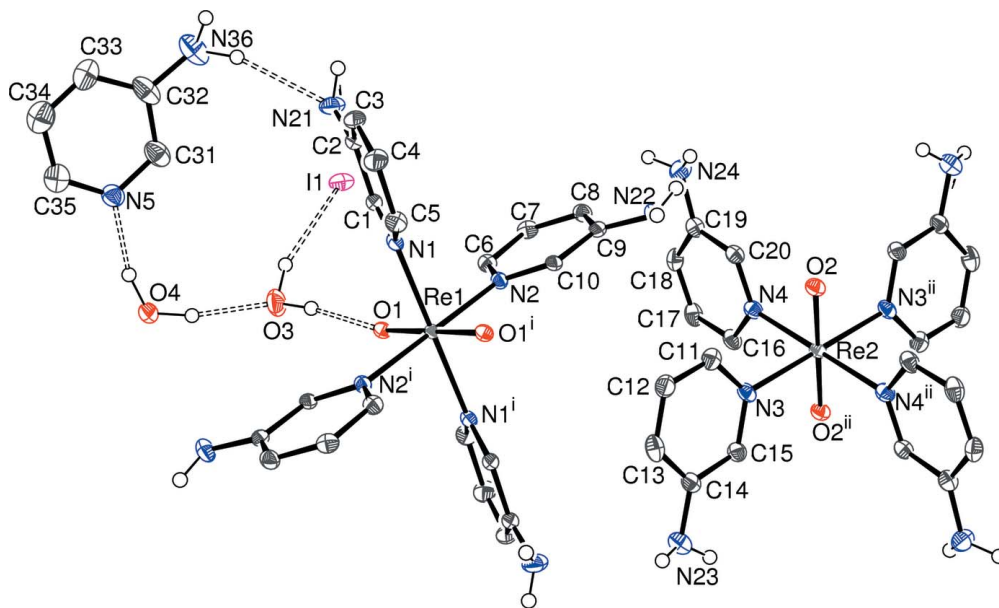
The asymmetric unit of the title compound, (I), (Fig. 1) contains half each of two independent $[\text{ReO}_2(\text{C}_5\text{H}_6\text{N}_2)_4]^+$ cations, an I^- anion, an uncoordinated 3-aminopyridine molecule and two water molecules. Each cation has a centre of inversion at the Re atom. The geometry around both Re atoms is octahedral with the four N atoms of the 3-aminopyridine molecules and the Re atom defining the equatorial plane. The Re—O distances (Table 1) are similar to those found in other oxo rhenium(V)-containing compounds (Lock & Turner, 1978; Ram & Hupp, 1991; Luck & O'Neill, 2001).



The water molecules, I^- anions and 3-aminopyridine molecules form intermolecular hydrogen bonds (Table 2), giving a three-dimensional structure. The shortest $\text{Re}\cdots\text{Re}$ distances in (I) are 6.889 (3) Å for $\text{Re1}\cdots\text{Re1}^i$ [symmetry code: (i) $x - 1, y, z$] and 9.232 (5) Å for $\text{Re1}\cdots\text{Re2}$.

Experimental

A mixture of $(\text{NH}_4)_2\text{ReI}_6$ (0.36 g; Watt & Thompson, 1963), 3-aminopyridine (0.40 g) and a solution of HI (1 ml) in water (50 ml) was stirred for 5 h at a temperature of 333 K. The color of the reaction mixture changed from dark violet–red to yellow–brown. After reaction, the mixture was cooled and the first crystalline product was obtained. X-ray quality crystals of (I) were obtained by recrystallization of the crude product from water at room temperature.

**Figure 1**

View of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms and C-bound H atoms omitted for clarity). Hydrogen bonds are indicated by dashed lines. [Symmetry codes: (i) $-x, 1 - y, 1 - z$; (ii) $-1 - x, -y, 1 - z$.]

Crystal data

[ReO₂(C₅H₆N₂)₄]₂I·C₅H₆N₂·2H₂O
 $M_r = 851.72$
 Triclinic, $P\bar{1}$
 $a = 9.4860$ (6) Å
 $b = 13.2425$ (8) Å
 $c = 13.4771$ (8) Å
 $\alpha = 81.447$ (5)°
 $\beta = 70.786$ (5)°
 $\gamma = 72.502$ (5)°
 $V = 1522.36$ (16) Å³
 $Z = 2$
 $D_x = 1.858$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 5.05$ mm⁻¹
 $T = 100$ (2) K
 Prism, red
 $0.08 \times 0.08 \times 0.04$ mm

Data collection

Kuma KM4-CCD diffractometer
 ω scans
 Absorption correction: numerical
 (*CrysAlis RED*; Oxford
 Diffraction, 2004)
 $T_{\min} = 0.681, T_{\max} = 0.826$
 27143 measured reflections
 12818 independent reflections
 8799 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.043$
 $\theta_{\text{max}} = 36.6^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.077$
 $S = 0.93$
 12818 reflections
 427 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0336P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 4.35$ e Å⁻³
 $\Delta\rho_{\text{min}} = -2.18$ e Å⁻³

Table 1

Selected bond lengths (Å).

Re1—O1	1.761 (2)	Re2—O2	1.771 (2)
Re1—N2	2.144 (3)	Re2—N4	2.145 (3)
Re1—N1	2.151 (3)	Re2—N3	2.160 (3)

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O3—H1A...I1	0.98 (3)	2.62 (4)	3.558 (3)	162 (5)
O3—H2A...O1	0.98 (4)	1.81 (5)	2.775 (3)	167 (6)
O4—H3A...N5	0.97 (4)	1.85 (4)	2.801 (5)	164 (4)
O4—H4A...O3	0.96 (5)	1.89 (4)	2.835 (4)	168 (4)
N22—H6A...O4 ⁱ	0.88 (4)	2.13 (4)	2.996 (4)	167 (4)
N21—H8A...I1 ⁱⁱ	0.82 (5)	2.93 (6)	3.717 (4)	162 (5)
N24—H10A...O4 ⁱ	0.88 (6)	2.12 (6)	2.999 (5)	174 (5)
N22—H11A...O2 ⁱⁱⁱ	0.81 (5)	2.34 (5)	3.099 (5)	156 (5)
N21—H12A...I1	0.88 (6)	2.97 (6)	3.825 (4)	163 (4)
N36—H13A...N21	0.98 (4)	2.37 (5)	3.320 (6)	163 (4)
N36—H14A...I1 ⁱⁱ	0.99 (4)	3.00 (4)	3.772 (4)	135 (3)
C6—H6...O1	0.94	2.39	2.918 (4)	114
C10—H10...O1 ^{iv}	0.94	2.34	2.886 (4)	115
C15—H15...O2 ^v	0.95	2.44	2.955 (5)	113
C16—H16...O2 ^v	0.95	2.36	2.894 (5)	114
C20—H20...O2	0.95	2.36	2.902 (4)	115

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

The O- and N-bound H atoms were located in difference maps and their positions and $U_{\text{iso}}(\text{H})$ values were freely refined. The C-bound H atoms were positioned geometrically ($\text{C—H} = 0.95$ Å) and refined as riding with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$. The highest difference peak is 0.88 Å from I1 and the deepest difference hole is 0.55 Å from I1.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2004); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2004); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXL97*.

References

Bruker (1999). *SHELXTL*. Bruker AXS Inc., Madison, Wisconsin, USA.

- Lock, C. J. L. & Turner, G. (1978). *Acta Cryst.* **B34**, 923–927.
- Luck, R. L. & O'Neill, R. S. (2001). *Polyhedron*, **20**, 773–782.
- Oxford Diffraction (2004). *CrysAlis CCD* and *CrysAlis RED*. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
- Ram, M. S. & Hupp, J. T. (1991). *Inorg. Chem.* **30**, 130–133.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Watt, G. W. & Thompson, R. J. (1963). *Inorg. Synth.* **7**, 190–192.