

2-Amino-5-nitropyridinium tetraoxido-rhenate(VII) monohydrate

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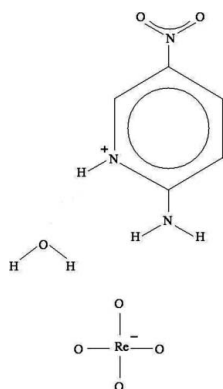
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(C-C) = 0.004$ Å; R factor = 0.015; wR factor = 0.033; data-to-parameter ratio = 15.0.

All the residues of the title compound, $(C_5H_6N_3O_2)[ReO_4] \cdot H_2O$, are located on general crystallographic positions. The 2-amino-5-nitropyridinium cation has a typical planar conformation with one of the nitro O atoms -0.058 (5) Å out of plane; the amine H atoms are also a little out of the main ring plane towards the opposite side of the aforementioned O atom [by 0.02 (4) and 0.04 (4) Å]. The perrhenate anion is nearly ideally tetrahedral. Three distinct $N-H \cdots O$ hydrogen bonds give rise to $C(8)$ zigzag chains running along [100]. $R_4^4(12)$ rings involving the two hydrogen bonds in which the water molecules interact with the perrhenate anions are also present.

Related literature

For the structural analyses of related 2-amino-5-nitropyridium salts and their potential application as non-linear optical materials, see: Masse & Zyss (1991); Puig-Molina *et al.* (1998); Aakeröy *et al.* (1998); Pecaut *et al.* (1993).



Experimental

Crystal data

$(C_5H_6N_3O_2)[ReO_4] \cdot H_2O$
 $M_r = 408.34$
 Monoclinic, $P2_1/c$
 $a = 9.6914$ (3) Å
 $b = 9.1357$ (3) Å
 $c = 13.4636$ (4) Å
 $\beta = 120.120$ (2)°

$V = 1031.08$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 11.81$ mm⁻¹
 $T = 295$ K
 $0.10 \times 0.06 \times 0.05$ mm

Data collection

Bruker APEXII diffractometer
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1996)
 $T_{min} = 0.33$, $T_{max} = 0.55$

30757 measured reflections
 2374 independent reflections
 2175 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.015$
 $wR(F^2) = 0.033$
 $S = 1.05$
 2374 reflections
 158 parameters
 5 restraints

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{max} = 0.88$ e Å⁻³
 $\Delta\rho_{min} = -0.72$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$N1-H1 \cdots O3$	0.86	1.93	2.738 (3)	157
$N1-H1 \cdots O6^i$	0.86	2.63	3.049 (4)	112
$N2-H2A \cdots O3$	0.89 (3)	2.33 (3)	3.030 (4)	135 (3)
$N2-H2A \cdots O4^{ii}$	0.89 (3)	2.65 (3)	3.259 (4)	127 (3)
$N2-H2B \cdots O5$	0.89 (3)	1.98 (3)	2.846 (3)	164 (3)
$O3-H3A \cdots O7^{iii}$	0.89 (3)	1.93 (3)	2.802 (4)	167 (3)
$O3-H3B \cdots O4^{ii}$	0.89 (3)	2.13 (3)	2.862 (4)	139 (3)

Symmetry codes: (i) $x - 1, -y + \frac{1}{2}, z - \frac{1}{2}$; (ii) $-x + 1, -y, -z$; (iii) $x - 1, y, z$.

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEPII (Johnson, 1976); software used to prepare material for publication: SHELXL97 and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZZ2177).

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supplementary materials

Acta Cryst. (2009). E65, m1000 [doi:10.1107/S1600536809029365]

2-Amino-5-nitropyridinium tetraoxidorhenate(VII) monohydrate

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Experimental

Crystals of the title compound were obtained by slow evaporation from a water solution of analytical grade reagents on a 1:1 molar ratio. The reagents used were 2-amino-5-nitropyridine (99.5%) and a perrhenic acid solution (65–75% water 99.5%), both purchased from Aldrich.

Refinement

The structure was solved by direct methods using *SHELXS97*. All H atoms were first located on a difference Fourier map as a check for data quality; those bonded to C and N aromatic atoms were then placed at idealized positions and refined as riding [C—H=0.93 Å, N—H=0.86 Å, $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{C})$ and $U_{\text{iso}}(\text{H})=1.2U_{\text{eq}}(\text{N})$]. The coordinates of the amine H atoms were then refined only with the restraint that the N—H distance would be 0.89 Å within 0.2 Å. The positions of the H atoms belonging to the water molecule were also restrained so that the intramolecular O—H distance would be 0.89 Å and the H—H distance 1.40 Å, both within 0.02 Å, thus enforcing an acceptable geometry of the water molecule [$U_{\text{iso}}(\text{H})=1.5U_{\text{eq}}(\text{O})$].

Examination of the crystal structure with *PLATON* (Spek, 2009) showed that there are no solvent-accessible voids in the crystal lattice.

Figures

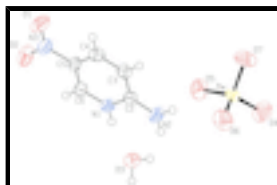


Fig. 1. *ORTEP* (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% level.

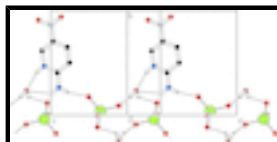


Fig. 2. Zigzag chain running along the *a* axis. Small ring motifs made of a perrhenate anion and two water molecules are included in the chain. Hydrogen bonds are indicated with dashed lines.

2-Amino-5-nitropyridinium tetraoxidorhenate(VII) monohydrate

Crystal data

(C₅H₆N₃O₂)[ReO₄]·H₂O

$M_r = 408.34$

Monoclinic, *P*2₁/*c*

Hall symbol: -*P* 2ybc

$F_{000} = 760$

$D_x = 2.631 \text{ Mg m}^{-3}$

Mo *K*α radiation, $\lambda = 0.71073 \text{ Å}$

Cell parameters from 9920 reflections

supplementary materials

$a = 9.6914 (3) \text{ \AA}$	$\theta = 2.4\text{--}32.3^\circ$
$b = 9.1357 (3) \text{ \AA}$	$\mu = 11.81 \text{ mm}^{-1}$
$c = 13.4636 (4) \text{ \AA}$	$T = 295 \text{ K}$
$\beta = 120.120 (2)^\circ$	Prism, translucent colourless
$V = 1031.08 (6) \text{ \AA}^3$	$0.10 \times 0.06 \times 0.05 \text{ mm}$
$Z = 4$	

Data collection

Bruker APEXII diffractometer	2374 independent reflections
Radiation source: fine-focus sealed tube	2175 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{\text{int}} = 0.030$
$T = 295 \text{ K}$	$\theta_{\text{max}} = 27.5^\circ$
φ and ω scans	$\theta_{\text{min}} = 2.4^\circ$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -12 \rightarrow 12$
$T_{\text{min}} = 0.33, T_{\text{max}} = 0.55$	$k = -11 \rightarrow 11$
30757 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.015$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.033$	$w = 1/[\sigma^2(F_o^2) + (0.0122P)^2 + 1.4451P]$
$S = 1.05$	where $P = (F_o^2 + 2F_c^2)/3$
2374 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
158 parameters	$\Delta\rho_{\text{max}} = 0.88 \text{ e \AA}^{-3}$
5 restraints	$\Delta\rho_{\text{min}} = -0.72 \text{ e \AA}^{-3}$
Primary atom site location: structure-invariant direct methods	Extinction correction: SHELXL97 (Sheldrick, 2008), $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
	Extinction coefficient: 0.00774 (16)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.2958 (3)	0.4684 (3)	0.0719 (2)	0.0338 (5)
H1	0.2145	0.4119	0.0493	0.041*
C2	0.4404 (3)	0.4063 (3)	0.1067 (2)	0.0315 (6)
N2	0.4512 (3)	0.2632 (3)	0.1047 (3)	0.0434 (6)
H2A	0.367 (3)	0.204 (3)	0.080 (3)	0.052*
H2B	0.543 (2)	0.219 (4)	0.124 (3)	0.052*
C3	0.5736 (3)	0.4998 (3)	0.1440 (3)	0.0352 (6)
H3	0.6741	0.4605	0.1684	0.042*
C4	0.5549 (3)	0.6463 (3)	0.1442 (2)	0.0362 (6)
H4	0.6420	0.7086	0.1688	0.043*
C5	0.4020 (3)	0.7029 (3)	0.1067 (2)	0.0332 (6)
N3	0.3785 (4)	0.8592 (3)	0.1068 (2)	0.0437 (6)
O1	0.4946 (4)	0.9377 (3)	0.1387 (3)	0.0717 (9)
O2	0.2459 (3)	0.9055 (3)	0.0781 (3)	0.0632 (7)
C6	0.2737 (3)	0.6133 (3)	0.0712 (2)	0.0336 (6)
H6	0.1728	0.6517	0.0469	0.040*
O3	0.1012 (3)	0.2314 (2)	0.0301 (2)	0.0538 (6)
H3A	0.083 (5)	0.174 (3)	0.076 (2)	0.081*
H3B	0.079 (5)	0.174 (3)	-0.030 (2)	0.081*
Re	0.905465 (12)	0.037384 (13)	0.215124 (10)	0.03195 (6)
O4	0.8222 (3)	-0.1153 (3)	0.1338 (2)	0.0680 (7)
O5	0.7677 (3)	0.1758 (3)	0.1707 (3)	0.0632 (7)
O6	0.9713 (4)	-0.0024 (3)	0.3551 (2)	0.0674 (8)
O7	1.0618 (3)	0.0893 (3)	0.1987 (3)	0.0664 (8)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0264 (11)	0.0290 (12)	0.0452 (15)	-0.0007 (9)	0.0174 (11)	0.0031 (11)
C2	0.0289 (12)	0.0319 (14)	0.0329 (15)	0.0032 (11)	0.0150 (11)	0.0054 (12)
N2	0.0375 (13)	0.0281 (13)	0.0627 (18)	0.0052 (11)	0.0237 (14)	0.0057 (13)
C3	0.0262 (13)	0.0400 (16)	0.0369 (16)	0.0004 (11)	0.0142 (12)	0.0010 (13)
C4	0.0312 (13)	0.0411 (17)	0.0341 (16)	-0.0090 (12)	0.0147 (12)	-0.0035 (13)
C5	0.0417 (15)	0.0277 (14)	0.0296 (15)	0.0020 (12)	0.0175 (12)	0.0023 (12)
N3	0.0597 (17)	0.0299 (13)	0.0415 (15)	0.0009 (12)	0.0254 (13)	0.0003 (11)
O1	0.080 (2)	0.0345 (13)	0.108 (3)	-0.0181 (13)	0.0530 (19)	-0.0083 (14)
O2	0.0674 (17)	0.0398 (14)	0.0761 (19)	0.0152 (13)	0.0313 (15)	0.0003 (13)
C6	0.0320 (13)	0.0329 (15)	0.0351 (15)	0.0054 (12)	0.0161 (12)	0.0046 (13)
O3	0.0643 (16)	0.0382 (13)	0.0750 (19)	-0.0090 (12)	0.0471 (15)	-0.0043 (13)
Re	0.02646 (7)	0.03566 (8)	0.03418 (8)	0.00296 (4)	0.01554 (5)	0.00215 (5)
O4	0.0773 (19)	0.0537 (16)	0.0667 (18)	-0.0151 (14)	0.0315 (15)	-0.0156 (14)
O5	0.0447 (13)	0.0583 (16)	0.0843 (19)	0.0226 (12)	0.0307 (13)	0.0131 (15)
O6	0.085 (2)	0.0780 (19)	0.0394 (15)	0.0150 (16)	0.0313 (14)	0.0090 (13)
O7	0.0398 (13)	0.083 (2)	0.086 (2)	0.0057 (13)	0.0389 (14)	0.0225 (17)

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Geometric parameters (\AA , $^\circ$)

N1—C6	1.341 (4)	C5—C6	1.360 (4)
N1—C2	1.358 (3)	C5—N3	1.446 (4)
N1—H1	0.8600	N3—O1	1.216 (4)
C2—N2	1.313 (4)	N3—O2	1.217 (4)
C2—C3	1.413 (4)	C6—H6	0.9300
N2—H2A	0.89 (3)	O3—H3A	0.89 (3)
N2—H2B	0.89 (3)	O3—H3B	0.89 (3)
C3—C4	1.351 (4)	Re—O6	1.698 (3)
C3—H3	0.9300	Re—O7	1.704 (2)
C4—C5	1.402 (4)	Re—O4	1.705 (3)
C4—H4	0.9300	Re—O5	1.714 (2)
C6—N1—C2	123.2 (2)	C6—C5—N3	118.6 (3)
C6—N1—H1	118.4	C4—C5—N3	120.1 (3)
C2—N1—H1	118.4	O1—N3—O2	123.3 (3)
N2—C2—N1	119.2 (3)	O1—N3—C5	117.7 (3)
N2—C2—C3	122.8 (3)	O2—N3—C5	119.0 (3)
N1—C2—C3	118.0 (3)	N1—C6—C5	118.5 (2)
C2—N2—H2A	123 (2)	N1—C6—H6	120.7
C2—N2—H2B	122 (2)	C5—C6—H6	120.7
H2A—N2—H2B	115 (3)	H3A—O3—H3B	104 (3)
C4—C3—C2	120.0 (3)	O6—Re—O7	109.89 (15)
C4—C3—H3	120.0	O6—Re—O4	109.31 (15)
C2—C3—H3	120.0	O7—Re—O4	108.01 (15)
C3—C4—C5	118.9 (3)	O6—Re—O5	109.77 (14)
C3—C4—H4	120.6	O7—Re—O5	109.69 (13)
C5—C4—H4	120.6	O4—Re—O5	110.15 (14)
C6—C5—C4	121.3 (3)		
C6—N1—C2—N2	179.9 (3)	C4—C5—N3—O1	1.0 (4)
C6—N1—C2—C3	0.1 (4)	C6—C5—N3—O2	1.9 (4)
N2—C2—C3—C4	-179.9 (3)	C4—C5—N3—O2	-177.2 (3)
N1—C2—C3—C4	-0.1 (4)	C2—N1—C6—C5	0.1 (4)
C2—C3—C4—C5	-0.2 (4)	C4—C5—C6—N1	-0.4 (4)
C3—C4—C5—C6	0.4 (4)	N3—C5—C6—N1	-179.5 (3)
C3—C4—C5—N3	179.5 (3)	N1—C2—N2—H2A	1(3)
C6—C5—N3—O1	-179.9 (3)	C3—C2—N2—H2B	-3(3)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N1—H1 \cdots O3	0.86	1.93	2.738 (3)	157
N1—H1 \cdots O6 ⁱ	0.86	2.63	3.049 (4)	112
N2—H2A \cdots O3	0.89 (3)	2.33 (3)	3.030 (4)	135 (3)
N2—H2A \cdots O4 ⁱⁱ	0.89 (3)	2.65 (3)	3.259 (4)	127 (3)
N2—H2B \cdots O5	0.89 (3)	1.98 (3)	2.846 (3)	164 (3)
O3—H3A \cdots O7 ⁱⁱⁱ	0.89 (3)	1.93 (3)	2.802 (4)	167 (3)

O3—H3B \cdots O4ⁱⁱ 0.89 (3) 2.13 (3) 2.862 (4) 139 (3)
 Symmetry codes: (i) $x-1, -y+1/2, z-1/2$; (ii) $-x+1, -y, -z$; (iii) $x-1, y, z$.

Fig. 1

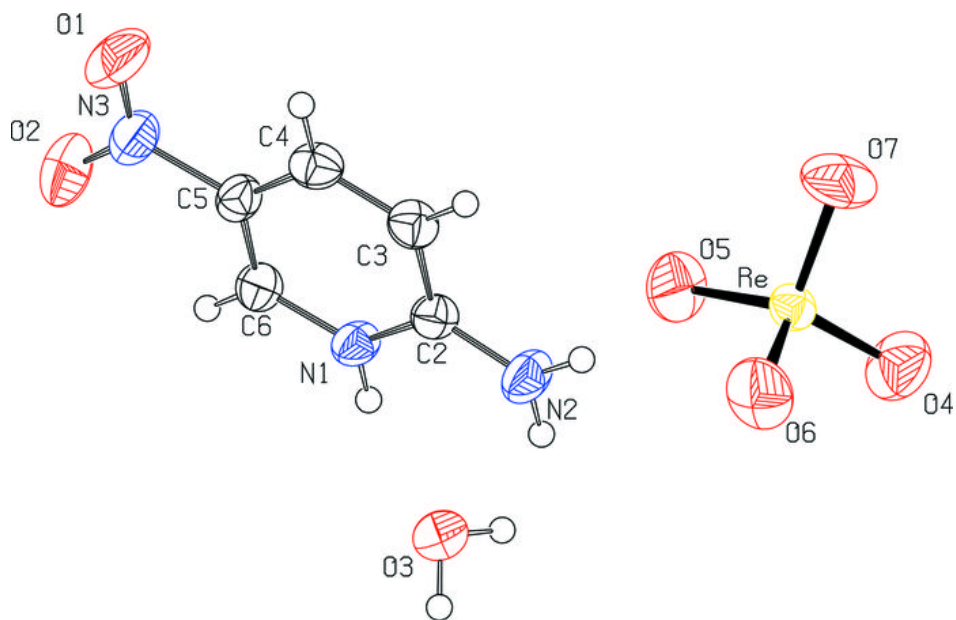


Fig. 2

