

2,2':6',2''-Terpyridinium perrhenate

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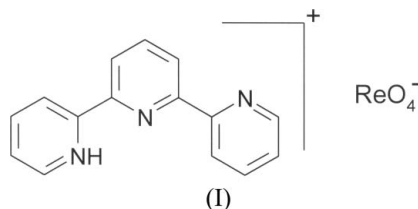
Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.015$ Å
 R factor = 0.061
 wR factor = 0.156
Data-to-parameter ratio = 16.4For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

In the title compound, $(\text{C}_{15}\text{H}_{12}\text{N}_3)[\text{ReO}_4]$, the cations exhibit a *cis-trans* conformation of the pyridine rings. The crystal structure is stabilized by a three-dimensional network of $\text{C}-\text{H}\cdots\text{O}$ hydrogen-bond interactions.

Comment

The title compound is the product of the hydrolysis of $(\text{NH}_4)_2\text{ReI}_6$ in the presence of 2,2':6',2''-terpyridine. The structure consists of terpyridinium cations (terpyH⁺) protonated at atom N1 and perrhenate anions (Fig. 1). The pyridine rings of the cation show a *cis-trans* conformation closely resembling that observed in $[\text{terpyH}](\text{CF}_3\text{SO})_3$ (Hergold-Brundić *et al.*, 1996). In this conformation, atom H1A is involved in a bifurcated hydrogen-bond interaction with atoms N2 and O1 as acceptors (Table 2). The Re—O bond lengths in the perrhenate anion (Table 1) are in agreement with the literature data (Herrman *et al.*, 1990; Kochel, 2005). In the crystal structure, cations and anions are packed in stacks extending along [010] linked by $\text{C}-\text{H}\cdots\text{O}$ hydrogen interactions (Table 2).



Experimental

A mixture of $(\text{NH}_4)_2\text{ReI}_6$ (0.02 g) and 2,2':6',2''-terpyridine (0.04 g) was dissolved in water (50 ml) and heated at 340 K for 10 h. The solution changed colour from black to light brown. After completion of the reaction, the solution was left to evaporate slowly at room temperature. After 5 d, yellow crystals of the title compound were obtained (yield 60%). The compound is stable in air, and dissolves easily in hot water, methanol and ethanol. IR (KBr, cm^{-1}): ν 3090, 1620, 1604, 1581, 1562, 1528, 1455, 1427, 1407, 1298, 1063, 920, 904, 874, 1455, 1427, 1407, 1298, 1063, 920, 904, 874, 774, 736, 648, 620, 398, 340, 320, 298, 257, 238, 191, 135, 108, 85, 71. Analysis calculated for $\text{C}_{15}\text{H}_{12}\text{N}_3\text{O}_4\text{Re}$: C 37.18, H 2.49, N 8.67, O 13.20, Re 26.00%; found: C 33.34, H 2.01, N 9.10%.

Crystal data

$(\text{C}_{15}\text{H}_{12}\text{N}_3)[\text{ReO}_4]$
 $M_r = 484.48$
Triclinic, $P\bar{1}$
 $a = 6.112$ (4) Å
 $b = 10.796$ (2) Å
 $c = 11.806$ (2) Å
 $\alpha = 76.18$ (3) $^\circ$
 $\beta = 87.12$ (3) $^\circ$
 $\gamma = 74.16$ (3) $^\circ$
 $V = 727.7$ (5) Å 3

$Z = 2$
 $D_x = 2.211$ Mg m $^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 3150
reflections
 $\theta = 3.5\text{--}28.4$ $^\circ$
 $\mu = 8.38$ mm $^{-1}$
 $T = 100$ (2) K
Needle, yellow
 $0.12 \times 0.05 \times 0.05$ mm

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Data collection

Kuma KM-4 CCD diffractometer
 ω scans
 Absorption correction: numerical
 (CrysAlis RED in KM-4 CCD
 Software; Oxford Diffraction,
 2004)
 $T_{\min} = 0.567$, $T_{\max} = 0.897$
 10097 measured reflections

3403 independent reflections
 2853 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.124$
 $\theta_{\text{max}} = 28.4^\circ$
 $h = -8 \rightarrow 7$
 $k = -14 \rightarrow 14$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.157$
 $S = 1.02$
 3403 reflections
 208 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.1028P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 3.10 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\text{min}} = -3.64 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Re1—O3	1.715 (8)	Re1—O2	1.727 (7)
Re1—O1	1.723 (7)	Re1—O4	1.728 (7)
O3—Re1—O1	109.0 (4)	O3—Re1—O4	109.9 (4)
O3—Re1—O2	108.7 (4)	O1—Re1—O4	110.3 (4)
O1—Re1—O2	110.3 (4)	O2—Re1—O4	108.6 (4)

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
N1—H1A \cdots N2	0.86	2.26	2.645 (11)	108
N1—H1A \cdots O1 ⁱ	0.86	2.07	2.763 (12)	137
C1—H1 \cdots O4 ⁱⁱ	0.93	2.41	3.258 (14)	151
C3—H3 \cdots O2 ⁱⁱⁱ	0.93	2.46	3.185 (13)	134
C4—H4 \cdots O2 ^{iv}	0.93	2.49	3.399 (13)	166
C7—H7 \cdots O2 ^v	0.93	2.60	3.505 (13)	166

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

All H atoms were placed in calculated positions ($C-H = 0.93 \text{ Å}$ and $N-H = 0.86 \text{ Å}$) and refined using a riding model, with $U_{\text{iso}}(H) = 1.2U_{\text{eq}}(C,N)$. The highest peak and deepest hole in the final difference Fourier synthesis are located 0.95 and 0.84 Å, respectively, from atom Re1. The relatively small size and poor diffraction quality of the crystals selected for the X-ray analysis could account for the rather high R_{int} value.

Data collection: *KM-4 CCD Software* (Oxford Diffraction, 2004); cell refinement: *KM-4 CCD Software*; data reduction: *KM-4 CCD Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

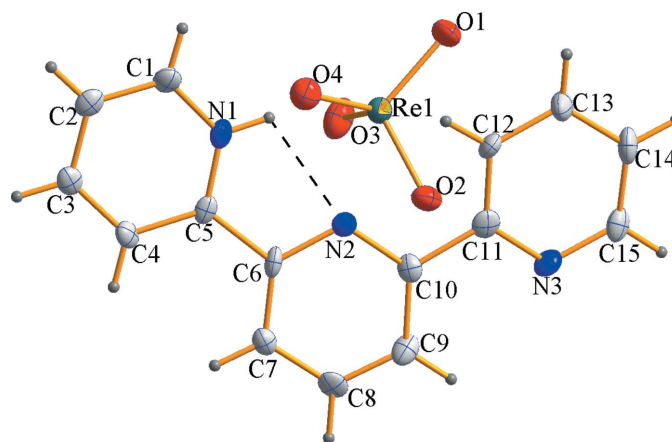


Figure 1

Molecular structure of the title compound, showing the intramolecular N—H \cdots N hydrogen bond (dashed line). Displacement ellipsoids are drawn at the 50% probability level.

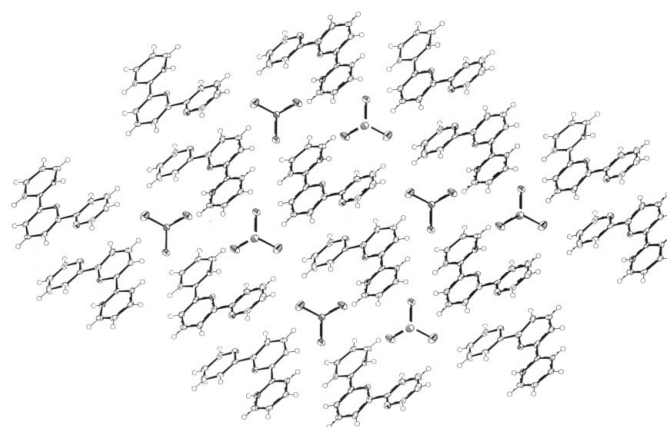


Figure 2

Crystal packing of the title compound viewed along the [010] direction. Displacement ellipsoids are drawn at the 50% probability level.

References

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