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4,7-Phenanthrolin-4-ium perrhenate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.026
wR factor = 0.060
Data-to-parameter ratio = 15.9

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

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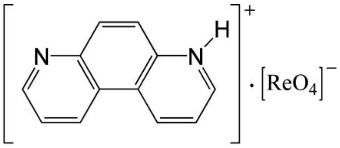
In the title complex, $(C_{12}H_9N_2)[ReO_4]$, the ions are connected together *via* hydrogen bonds. The crystal structure comprises alternating layers of cations and anions aligned along the *b*-axis direction.

Comment

High oxidation state organorhenium compounds are receiving increasing attention owing to their importance as catalysts or as catalyst precursors, particularly for olefin metathesis (Romao *et al.*, 1997). In spite of these investigations, there are few studies on perrhenate structures. In the present paper, we report the crystal structure of a new perrhenate complex, (I) (Fig. 1 and Table 1).



(I)

Strong N—H–O hydrogen bonds connect anions and cations in the crystal structure. In addition, other non-classical hydrogen bonds are also observed, as detailed in Table 2. Although the 4,7-phenanthrolin-4-ium cations are parallel to

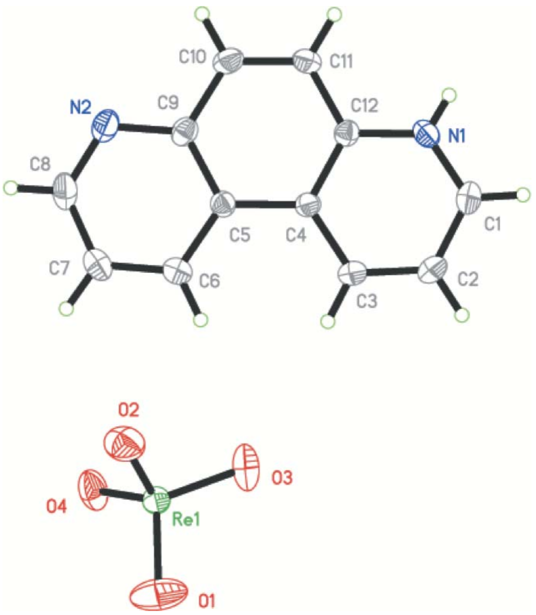


Figure 1
View of the components of (I), showing 30% probability displacement ellipsoids and the atom-numbering scheme.

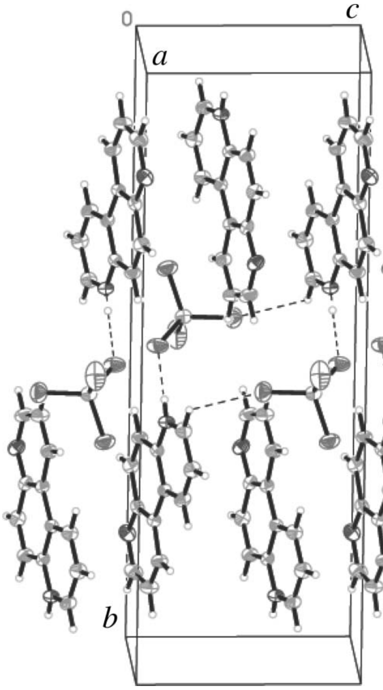


Figure 2
The packing of (I), viewed approximately down the *a* axis. Hydrogen bonds are shown as dashed lines.

each other, with a dihedral angle 2.02 (2)°, there are no close π–π interactions between them. The crystal structure comprises alternating layers of cations and anions, as represented in Fig. 2.

Experimental

A mixture of 4,7-phenanthroline (90.0 mg, 0.5 mmol) and potassium perrhenate (145.0 mg, 0.5 mmol) was dissolved in tetrahydrofuran (20 ml) under nitrogen. The reaction mixture was stirred overnight at room temperature. The solvent was removed *in vacuo* and the product (I) was obtained as a pale-yellow solid (204.7 mg, 95%). Crystals suitable for data collection were obtained by slow evaporation of an acetone solution of (I). ^{1}H NMR (400 MHz, [D$_6$]-acetone): δ 8.21 (2H, *dd*, J = 4.9 and 8.4 Hz, phen-H$_{3,8}$), 8.55 (2H, *s*, phen-H$_{5,6}$), 9.29 (2H, *d*, J = 4.8 Hz, phen-H$_{4,7}$), 9.73 (2H, *d*, J = 8.5 Hz, phen-H$_{2,9}$).

Crystal data

$(C_{12}H_9N_2)[ReO_4]$
$M_r = 431.41$
Monoclinic, $P2_1/c$
$a = 7.8374$ (16) Å
$b = 20.840$ (4) Å
$c = 7.5280$ (15) Å
$\beta = 92.95$ (3)°
$V = 1227.9$ (4) Å^3
$Z = 4$
$D_x = 2.334$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 9.91$ mm^{-1}
$T = 293$ (2) K
Block, yellow
0.22 × 0.10 × 0.08 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{\min} = 0.32$, $T_{\max} = 0.45$
11638 measured reflections
2815 independent reflections
2267 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.031$
$\theta_{\max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.060$
$S = 1.08$
2815 reflections
177 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0193P)^2 + 1.693P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 0.78$ e Å^{-3}
$\Delta\rho_{\min} = -1.09$ e Å^{-3}
Extinction correction: *SHELXL97*
Extinction coefficient: 0.00182 (17)

Table 1
Selected geometric parameters (Å, °).

Re1—O1	1.696 (5)	Re1—O3	1.704 (4)
Re1—O2	1.708 (4)	Re1—O4	1.728 (4)
O1—Re1—O2	108.3 (2)	O2—Re1—O3	108.3 (2)
O1—Re1—O3	112.1 (3)	O2—Re1—O4	109.5 (2)
O1—Re1—O4	110.3 (3)	O3—Re1—O4	108.3 (2)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H9···O4$^{\rm i}$	0.77 (5)	1.98 (5)	2.746 (6)	168 (5)
C1—H1···O1$^{\rm ii}$	0.93	2.41	3.254 (7)	151
C1—H1···O2$^{\rm iii}$	0.93	2.55	3.024 (7)	112
C2—H2···N2$^{\rm iv}$	0.93	2.44	3.350 (7)	166
C10—H10···O3$^{\rm v}$	0.93	2.50	3.252 (7)	138

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

The imino H atom was located in a difference Fourier map and refined independently. The aromatic H atoms were constrained to an ideal geometry, with C—H = 0.93 Å and $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The deepest hole in the final Fourier difference map was located 0.65 Å from atom Re1..

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2001); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *PLATON*.

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