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Bis[4,4′-dimethyl-2,2′-bipyridinium(+)] tetraoxorhenate(VII) triiodide

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The asymmetric unit of the title compound, $(C_{12}H_{13}N_2)_2[ReO_4]I_3$, contains two 4,4′-dimethyl-2,2′-bipyridinium cations, one ReO_4^- anion and two half-triiodide anions, each anion lying on a centre of symmetry. The crystal packing is stabilized by N—H⋯O, C—H⋯O and C—H⋯I interactions.

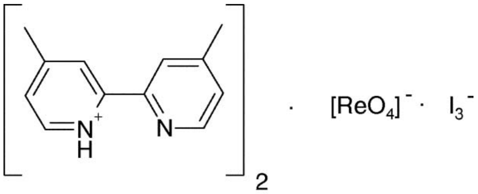
Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.005 Å
R factor = 0.026
wR factor = 0.059
Data-to-parameter ratio = 20.0

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

Comment

The title compound, (I) (Fig. 1), contains a network of 2,2′-bipyridinium cations, $[Re^{VII}O_4]^-$ perrhenate anions and I_3^- triiodide anions.



(I)

The Re—O bond lengths in the perrhenate anion (Table 1) are similar to those found for the same species in other molecular compounds (Herrmann *et al.*, 1990). There are two half-triiodide ions in the asymmetric unit of (I). In each case, the complete ion is generated by inversion symmetry, which constrains it to be symmetric and linear. The I—I distances in (I) (Table 1) are similar to those found in related compounds (Schmidt-Brucken & Abram, 2001)

N—H⋯O hydrogen bonds and C—H⋯O and C—H⋯I interactions are present in (I) (Table 2), and help to stabilize the crystal packing. The ions are arranged in alternating layers (Fig. 2). The first consists of 4,4′-dimethyl-2,2′-bipyridinium cations and $[ReO_4]^-$ anions, while the other consists of I_3^- ions. There are also π–π interactions between the pyridine rings [centroid-to-centroid distances range from 3.75 (2) to 3.52 (2) Å].

Experimental

A mixture of $(NH_4)_2ReI_6$ (0.019 g) and 4,4′-dimethyl-2,2′-bipyridine (0.040 g) was dissolved in water (50 ml) containing 46% HI solution (2 ml). The mixture was heated at 320 K for 5 h. The solution changed color from black to violet. After the reaction, the solution was left at room temperature and the water slowly evaporated. After 3 d, the solution became colorless and red crystals of (I) were deposited in 57% yield. Compound (I) is stable in air and dissolves easily in CH_2Cl_2, acetone and pyridine. IR (KBr, ν cm^{-1}) 3442, 2922, 1628,1597, 1515, 1446, 1380, 1348, 1302, 1250, 1225, 1215, 1106, 1021, 993, 920, 889, 825, 720, 510, 444, 417, 519, 510, 470, 444, 346, 326, 306, 286, 249, 135, 118, 71. Elemental analysis calculated for

$C_{24}H_{26}I_3N_4O_4Re$: C 28.78, H 2.61, N 5.59%, found: C 27.50, H 2.32, N 4.95%.

Crystal data

$(C_{12}H_{13}N_2)_2[ReO_4]I_3$
$M_r = 1001.40$
Triclinic, $P\bar{1}$
$a = 9.667$ (2) Å
$b = 12.096$ (2) Å
$c = 14.185$ (3) Å
$\alpha = 66.41$ (3)°
$\beta = 85.10$ (3)°
$\gamma = 74.07$ (3)°
$V = 1461.1$ (5) Å^3

$Z = 2$
$D_x = 2.276$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 5240 reflections
$\theta = 3.0$–25.0°
$\mu = 7.36$ mm^{-1}
$T = 100$ (2) K
Block, red
0.30 × 0.15 × 0.10 mm

Data collection

KM-4/CCD diffractometer
ω scans
Absorption correction: numerical (*CrysAlis RED*; Oxford Diffraction, 2004
$T_{\min} = 0.378$, $T_{\max} = 0.595$
19623 measured reflections

6808 independent reflections
5964 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.049$
$\theta_{\max} = 28.5°$
$h = -12 \rightarrow 12$
$k = -16 \rightarrow 13$
$l = -18 \rightarrow 18$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.026$
$wR(F^2) = 0.059$
$S = 1.01$
6808 reflections
340 parameters

H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0325P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.001$
$\Delta\rho_{\max} = 1.61$ e Å^{-3}
$\Delta\rho_{\min} = -1.09$ e Å^{-3}

Table 1
Selected bond lengths (Å).

Re1—O4	1.723 (3)	Re1—O3	1.741 (3)
Re1—O1	1.727 (3)	I1—I2	2.9369 (8)
Re1—O2	1.730 (3)	I3—I4	2.9224 (8)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1A···O1	0.99 (5)	1.79 (5)	2.714 (4)	154 (5)
N4—H2A···O3$^{\rm i}$	0.80 (5)	2.09 (5)	2.748 (4)	140 (5)
C4—H4···O4$^{\rm ii}$	0.95	2.48	3.183 (5)	130
C23—H23···O2$^{\rm iii}$	0.95	2.46	3.199 (5)	133
C26—H26···O2$^{\rm iii}$	0.95	2.58	3.429 (5)	148
C30—H30A···O3$^{\rm iii}$	0.98	2.57	3.466 (6)	151
C20—H20···I2$^{\rm iv}$	0.95	3.05	3.998 (4)	169

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

The positions of the N-bound H atoms were refined freely along with their isotropic displacement parameters. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.95–0.98 Å and with $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C). The highest peak and deepest hole are located 1.31 Å from atom N1 and 0.88 Å from Re1, respectively.

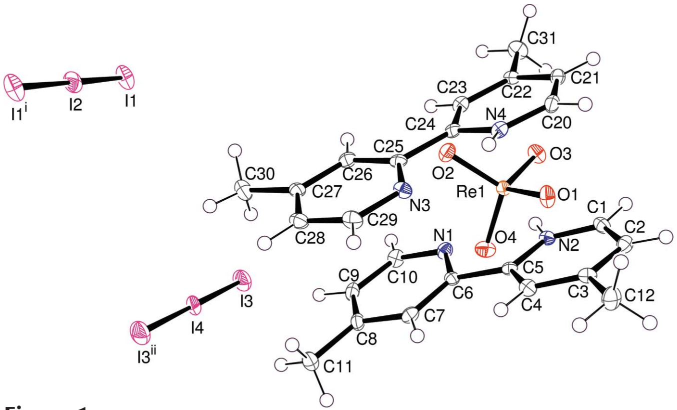


Figure 1
View of the components of (I), showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). [Symmetry codes: (i) $-x, 2-y, -z$; (ii) $1-x, 1-y, -z$.]

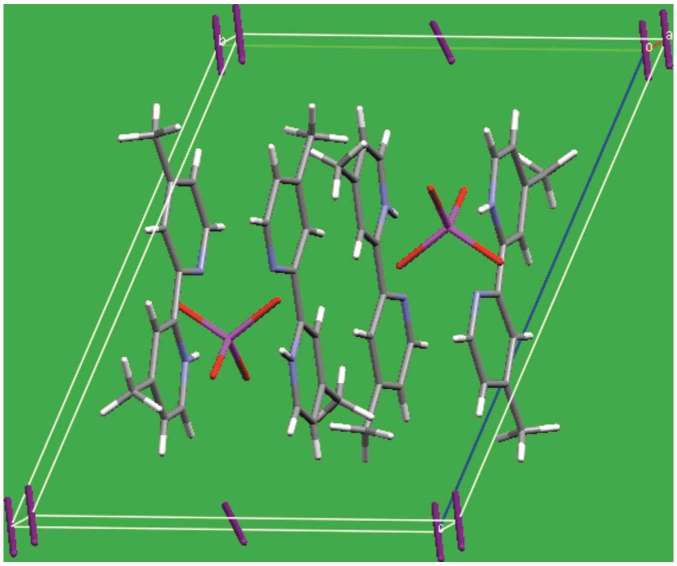


Figure 2
The unit-cell contents of (I).

Data collection: *KM4CCD* (Oxford Diffraction, 2004); cell refinement: *KM4CCD*; data reduction: *KM4CCD*; 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*.

References

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Herrmann, W. A., Kuchler, J. G., Kiprof, O. & Riede, J. (1990). *J. Organomet. Chem.* **395**, 55–67.
Oxford Diffraction (2004). *CrysAlis RED* (Version 1.171.13) and *KM4CCD*. Oxford Diffraction Ltd, Abingdon, Oxfordshire, England.
Schmidt-Brucken, B. & Abram, U. (2001). *Z. Anorg. Allg. Chem.* **627**, 7–8.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.