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2,2′-Bipyrimidinium hexachlororhenate(IV) dihydrate

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.028
wR factor = 0.057
Data-to-parameter ratio = 35.0

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

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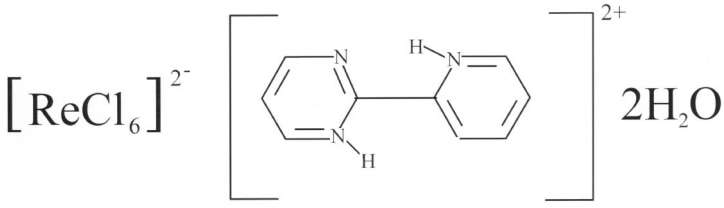
In the title compound, $(C_8H_8N_4)[ReCl_6]\cdot 2H_2O$, the Re atom occupies an inversion centre and is octahedrally coordinated by chloride anions. The cation also has an inversion centre. Various N—H···O, O—H···Cl and C—H···Cl interactions help to stabilize the crystal packing.

Comment

The title complex, (I) (Fig. 1), is a salt-like rhenium(IV)-containing hydrate. The Re^{IV} cation (electron configuration $5d^3$) occupies an inversion centre and bonds to six chloride ions in a fairly regular octahedral geometry (Table 1). The Re—Cl bond lengths are similar to those found in other hexachlororhenates (Figgis *et al.*, 1961; Kochel, 2004). The 2,2′-bipyrimidinium cation has an inversion centre at the mid-point of the C—C bond linking the two rings, and shows no unusual features.



(I)

In the crystal structure of (I), various types of hydrogen bonds (Table 2) link the cations, anions and water molecules

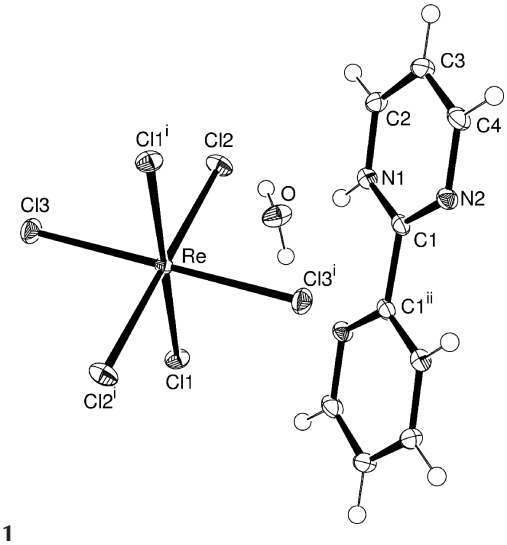


Figure 1
View of the components of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). [Symmetry codes: (i) $-x, -y, -z$; (ii) $1-x, -y, 1-z$.]

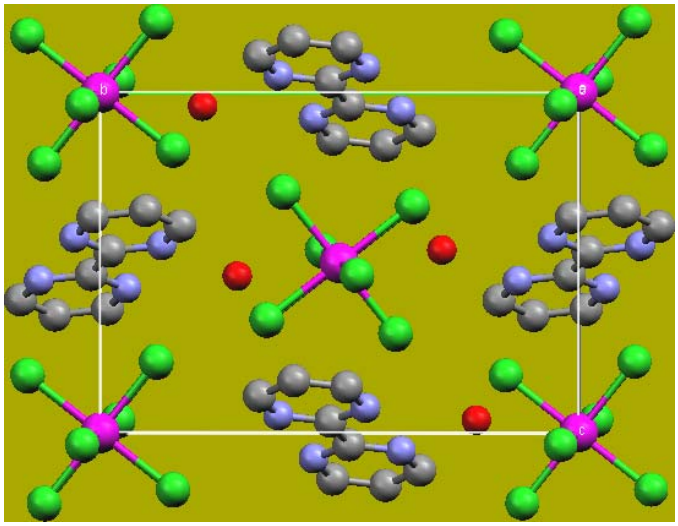


Figure 2
The crystal packing in (I), with H atoms omitted for clarity.

together. In the crystal packing, two characteristic types of arrangement of molecules may be discerned: in the [010] and [001] directions, a layered arrangement (alternating anions and cations) is observed, while in the [100] direction, anions arrange themselves into columns inside troughs formed of cations and water molecules, and the whole is surrounded by a network of hydrogen bonds. The shortest intermetallic distances in (I) are Re···Re^{i} = 7.941 (2) Å and Re···Re^{ii} = 7.825 (2) Å [symmetry codes: (i) $x-1, y, z$; (ii) $-x, \frac{1}{2}+y, -\frac{1}{2}-z$].

Experimental

$(NH_4)_2ReCl_6$ (0.1 g) was dissolved in water (20 ml) with concentrated HCl (2 ml) and the mixture was heated under reflux at 340 K. After 30 min, 2,2′-bipyrimidine (0.15 g) dissolved in water (50 ml) was added. The mixture was heated for a further 3 h. After cooling, the yellow precipitate was filtered off and washed with ethanol and diethyl ether. Crystals of (I) suitable for X-ray study were obtained by slow evaporation of an aqueous solution of the yellow precipitate. IR (KBr, ν cm^{-1}): 3446, 3068, 2576, 2034, 1617, 1562, 1508, 1427, 1404, 1338, 1281, 1130, 1056, 995, 984, 826, 736, 735, 670, 451, 561, 458, 301, 169, 126. Analysis calculated for $C_8H_{12}Cl_6N_4O_2Re$: C 16.14, H 2.03, N 9.41, O 5.37, Cl 35.74, Re 31.29%; found: C 15.12, H 1.90, N 9.15, Cl 35.30%.

Crystal data

$(C_8H_8N_4)[ReCl_6]\cdot 2H_2O$
M_r = 595.13
Monoclinic, $P2_1/c$
a = 7.941 (2) Å
b = 12.199 (2) Å
c = 9.804 (2) Å
β = 118.18 (3)°
V = 837.2 (3) Å^3
Z = 2
D_x = 2.361 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2760 reflections
θ = 3.2–36.5°
μ = 8.22 mm^{-1}
T = 100 K
Plate, yellow
0.12 × 0.10 × 0.05 mm

Data collection

Kuma KM4/CCD diffractometer
ω scans
Absorption correction: numerical (*CrysAlis RED*in *KM4CCD Software*; Oxford Diffraction, 2004)
T_{min} = 0.567, T_{max} = 0.897
14616 measured reflections
3711 independent reflections
2860 reflections with $I > 2\sigma(I)$
R_{int} = 0.049
θ_{max} = 36.5°
$h = -13 \rightarrow 12$
$k = -20 \rightarrow 16$
$l = -14 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.057
S = 1.07
3711 reflections
106 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0255P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 1.75 e Å^{-3}
$\Delta\rho_{min}$ = −2.24 e Å^{-3}

Table 1
Selected bond lengths (Å).

Re—Cl1	2.3681 (9)	Re—Cl3	2.3601 (8)
Re—Cl2	2.3553 (8)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1···O	0.82 (5)	1.83 (5)	2.627 (4)	165 (5)
N1—H1···N2^{i}	0.82 (5)	2.40 (4)	2.749 (4)	107 (4)
O—H1W···Cl1^{ii}	0.83 (5)	2.48 (5)	3.183 (3)	144 (4)
O—H1W···Cl1^{iii}	0.83 (5)	2.77 (5)	3.340 (3)	127 (4)
O—H2W···Cl3^{iv}	0.82 (6)	2.72 (6)	3.340 (3)	133 (4)
C2—H2···Cl1^{iii}	0.93	2.81	3.670 (4)	154
C4—H4···Cl2^{v}	0.93	2.74	3.595 (4)	153

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

H atoms bonded to C atoms were placed in calculated positions, with C—H = 0.93 Å, and refined as riding with the constraint U_{iso}(H) = 1.2U_{eq}(carrier) applied. H atoms associated with N and O atoms were located in difference maps and then freely refined. The highest peak and deepest hole are located 0.88 Å from atom Cl1 and 0.73 Å from Re, respectively.

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

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