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Bis(2-Amino-6-methylpyridinium) aqua-tetrabromidooxidorhenate(V) bromide

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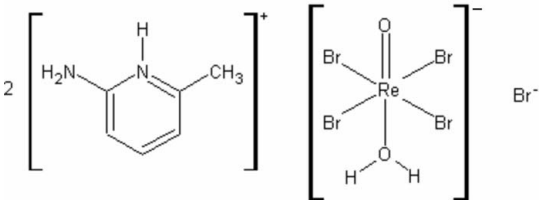
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.008 Å; R factor = 0.039; wR factor = 0.069; data-to-parameter ratio = 42.3.

The title compound, $(C_6H_9N_2)_2\cdot[ReO(H_2O)Br_4]\cdot Br$, is a molecular salt incorporating rhenium(V)-containing oxoanions. The oxoanion shows a distorted *trans*-ReO_2Br_4 geometry. In the crystal structure, the component species are connected *via* N—H···Br and O—H···Br interactions.

Related literature

For related structures, see: Abram *et al.* (1996); Chiozzone *et al.* (2006); Kochel (2007). For synthetic background, see: Watt & Thompson (1963).



Experimental

Crystal data

$(C_6H_9N_2)_2\cdot[ReO(H_2O)Br_4]\cdot Br$
M_r = 838.07
Monoclinic, $P2_1/c$
a = 11.996 (2) Å
b = 12.154 (3) Å
c = 18.206 (3) Å
β = 125.43 (2)°
V = 2162.9 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 14.87 mm^{-1}
T = 100 (2) K
0.08 × 0.08 × 0.07 mm

Data collection

Kuma KM4 CCD diffractometer
Absorption correction: numerical (*CrysAlis RED*; Oxford Diffraction, 2003)
T_{min} = 0.340, T_{max} = 0.563
40507 measured reflections
10288 independent reflections
5873 reflections with $I > 2\sigma(I)$
R_{int} = 0.080

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.039
$wR(F^2)$ = 0.069
S = 0.92
10288 reflections
243 parameters
8 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 2.13 e Å^{-3}
$\Delta\rho_{min}$ = −2.32 e Å^{-3}

Table 1
Selected bond lengths (Å).

Re1—O2	1.667 (3)	Re1—Br2	2.5141 (6)
Re1—O1	2.206 (3)	Re1—Br1	2.5223 (5)
Re1—Br3	2.5117 (6)	Re1—Br4	2.5254 (6)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···Br5	0.92 (3)	2.54 (5)	3.354 (4)	148 (5)
O1—H1W···Br5^{i}	0.95 (4)	2.25 (4)	3.176 (3)	165 (4)
N2—H2N···Br1^{ii}	0.92 (5)	2.76 (5)	3.573 (5)	148 (4)
N2—H2N···Br4^{ii}	0.92 (5)	2.91 (5)	3.424 (6)	117 (4)
O1—H2W···Br5^{iii}	0.95 (3)	2.28 (3)	3.225 (4)	170 (4)
N2—H3N···Br5	0.92 (2)	2.75 (4)	3.514 (4)	141 (3)
N3—H4N···Br5^{iv}	0.92 (5)	2.42 (5)	3.273 (4)	154 (4)
N4—H5N···Br2^{v}	0.90 (5)	2.83 (5)	3.592 (6)	143 (4)
N4—H6N···Br4^{vi}	0.92 (4)	2.79 (5)	3.647 (5)	155 (5)

Symmetry codes: (i) $-x, -y+2, -z+1$; (ii) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (iii) $x-1, y, z-1$; (iv) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (v) $x+1, -y+\frac{3}{2}, z+\frac{1}{2}$; (vi) $-x, -y+1, -z+1$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2003); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2003); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); *XP* (Bruker, 1999); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2535).

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supplementary materials

Acta Cryst. (2007). E**63**, m2533 [**doi:10.1107/S1600536807044133**]

Bis(2-Amino-6-methylpyridinium) aquatetrabromidooxidorhenate(V) bromide

A. Kochel

Comment

This paper reports the crystal structure of a molecular salt containing the $[Re^{V}O(H_2O)Br_4]^{-}$ anion. All atoms lie on general positions (Fig. 1). The $[ReO(H_2O)Br_4]^{-}$ anion in (I) shows a distorted *trans*-ReO_2Br_4 octahedral geometry about the central Re(V) ion (Table 1). The water molecule is weakly coordinated to rhenium at the base of octahedron with a Re—O distance of 2.206 (3) Å. The Re—Br bond lengths in (I) agree with those in related crystal structures (Abram *et al.*, 1996; Chiozzone *et al.*, 2006; Kochel, 2007).

In the crystal, the component species interact *via* N—H···Br and O—H···Br hydrogen bonds (Table 2), resulting in alternating layers of anions and cations perpendicular to [010] The shortest Re···Re distances in (I) are 6.819 (3) Å for Re1···Re1^{i} (i = $-x$, $-y$ + 2, $-z$ + 1) and 8.546 (4) Å for Re1···Re2^{i} (i = $-x$ − 1, $-y$ + 2, $-z$).

Experimental

$(NH_4)_2ReBr_6$ was obtained by the method of Watt & Thompson (1963). A mixture of $(NH_4)_2ReBr_6$ (0.33 g) and 2-amino-6-methylpyridine (0.80 g) was dissolved in 50- ml of ethanol solution, which was heated at 323 K for 10 h followed by slow cooling. After the reaction the mixture colour was yellow–orange. The solution was filtered and the filtrate was left standing for evaporation. After five days, orange plates of (I) appeared. Anal. Calc for $C_{12}H_{20}Br_5N_4O_2Re$: C 17.19, H 2.57, N 6.68%; found C 16.34, H 2.30, N 6.54%. IR (KBr): 1195(*versus*), 1200(*s*), 990(*m*), 950(*s*), 1054(*m*), 980(*s*), 873(*m*), 810(*s*), 730(*m*), 310(*s*), 174(*m*), 159(*m*).

Refinement

The N– and O-bound H atoms were located in difference maps and their positions were freely refined with U_{iso}(H) = 1.2U_{eq}(carrier). The C-bound H atoms were placed in idealized positions (C—H = 0.93–0.96 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl C).

The highest difference peak is 0.59Å from Re1. The deepest difference hole is 0.68Å from Re1.

Figures

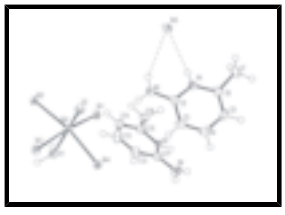

Fig. 1. The molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms).

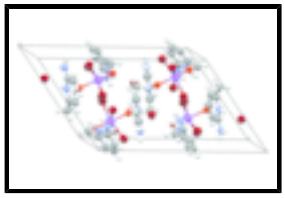

Fig. 2. The crystal packing in (I).

Bis(2-Amino-6-methylpyridinium) aquatetrabromidooxidorhenate(V) bromide

Crystal data

$(C_6H_9N_2)_2{\cdot}[ReO(H_2O)Br_4]{\cdot}Br$	F_{000} = 1544
M_r = 838.07	D_x = 2.574 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2ybc	Cell parameters from 5614 reflections
a = 11.996 (2) Å	θ = 2.7–36.7º
b = 12.154 (3) Å	μ = 14.87 mm^{-1}
c = 18.206 (3) Å	T = 100 (2) K
β = 125.43 (2)º	Prism, orange
V = 2162.9 (7) Å^3	0.08 × 0.08 × 0.07 mm
Z = 4	

Data collection

Kuma KM4 CCD diffractometer	10288 independent reflections
Radiation source: fine-focus sealed tube	5873 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.080
T = 100(2) K	θ_{max} = 36.7º
ω scans	θ_{min} = 2.7º
Absorption correction: numerical (CrysAlis [CrysAlis RED?]; Oxford Diffraction, 2003)	h = −19→19
T_{min} = 0.340, T_{max} = 0.563	k = −18→20
40507 measured reflections	l = −30→24

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.039	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2)$ = 0.069	$w = 1/[\sigma^2(F_o^2) + (0.0236P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.92	$(\Delta/\sigma)_{max}$ = 0.001
10288 reflections	$\Delta\rho_{max}$ = 2.13 e Å^{-3}

243 parameters
8 restraints
Primary atom site location: structure-invariant direct methods

$\Delta\rho_{min} = -2.32$ e Å^{-3}
Extinction correction: none

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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.4220 (4)	0.6746 (3)	0.9044 (2)	0.0208 (7)
H1N	0.409 (5)	0.7471 (18)	0.911 (3)	0.025*
N2	0.1884 (4)	0.6673 (3)	0.8315 (3)	0.0324 (9)
H2N	0.104 (3)	0.634 (4)	0.797 (3)	0.039*
H3N	0.193 (5)	0.7408 (18)	0.844 (3)	0.039*
C2	0.3048 (4)	0.6156 (3)	0.8599 (3)	0.0229 (9)
C3	0.3142 (5)	0.5033 (3)	0.8462 (3)	0.0271 (10)
H3	0.2366	0.4591	0.8173	0.033*
C4	0.4377 (5)	0.4602 (4)	0.8757 (3)	0.0310 (11)
H4	0.4440	0.3861	0.8658	0.037*
C5	0.5566 (5)	0.5257 (4)	0.9212 (3)	0.0318 (11)
H5	0.6406	0.4953	0.9406	0.038*
C6	0.5479 (4)	0.6329 (4)	0.9365 (3)	0.0261 (9)
C7	0.6656 (5)	0.7116 (5)	0.9858 (4)	0.0397 (13)
H7A	0.6761	0.7364	1.0396	0.060*
H7B	0.6482	0.7736	0.9478	0.060*
H7C	0.7478	0.6753	1.0016	0.060*
N3	0.1990 (3)	0.6251 (3)	0.5887 (2)	0.0182 (7)
H4N	0.265 (3)	0.621 (4)	0.578 (3)	0.022*
N4	0.2592 (4)	0.4446 (3)	0.6379 (3)	0.0278 (8)
H5N	0.334 (3)	0.457 (4)	0.639 (3)	0.033*
H6N	0.239 (5)	0.379 (2)	0.653 (3)	0.033*
C11	0.1736 (4)	0.5308 (3)	0.6153 (3)	0.0205 (8)
C12	0.0562 (5)	0.5275 (4)	0.6155 (3)	0.0275 (10)
H12	0.0324	0.4632	0.6311	0.033*
C13	−0.0216 (5)	0.6198 (4)	0.5926 (3)	0.0321 (11)
H13	−0.0980	0.6187	0.5938	0.039*
C14	0.0113 (5)	0.7163 (4)	0.5672 (3)	0.0288 (10)
H14	−0.0421	0.7791	0.5527	0.035*

C15	0.1211 (4)	0.7179 (3)	0.5638 (3)	0.0212 (8)
C16	0.1635 (4)	0.8130 (3)	0.5331 (3)	0.0250 (9)
H16A	0.1512	0.7943	0.4776	0.038*
H16B	0.2581	0.8296	0.5781	0.038*
H16C	0.1086	0.8760	0.5239	0.038*
Re1	−0.237586 (16)	0.926025 (12)	0.281334 (11)	0.01677 (4)
Br1	−0.06351 (4)	1.03636 (3)	0.27687 (3)	0.02292 (9)
Br2	−0.38484 (4)	1.09524 (3)	0.23272 (3)	0.02134 (9)
Br3	−0.44110 (4)	0.81224 (3)	0.23999 (3)	0.02152 (9)
Br4	−0.12108 (4)	0.75203 (3)	0.28299 (3)	0.02382 (9)
O1	−0.3328 (3)	0.9120 (2)	0.1355 (2)	0.0197 (6)
O2	−0.1646 (3)	0.9336 (2)	0.3918 (2)	0.0248 (6)
Br5	0.34325 (4)	0.88956 (3)	0.98187 (3)	0.02261 (9)
H1W	−0.324 (5)	0.978 (2)	0.111 (3)	0.027*
H2W	−0.426 (2)	0.897 (4)	0.088 (2)	0.027*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0184 (17)	0.0190 (16)	0.027 (2)	−0.0032 (14)	0.0139 (16)	−0.0034 (14)
N2	0.0181 (19)	0.029 (2)	0.041 (3)	−0.0060 (17)	0.0119 (19)	−0.0156 (18)
C2	0.024 (2)	0.025 (2)	0.020 (2)	−0.0060 (18)	0.0128 (19)	−0.0040 (17)
C3	0.035 (3)	0.023 (2)	0.029 (3)	−0.0080 (19)	0.021 (2)	−0.0075 (18)
C4	0.045 (3)	0.022 (2)	0.026 (2)	0.008 (2)	0.021 (2)	−0.0002 (18)
C5	0.026 (2)	0.039 (3)	0.029 (3)	0.014 (2)	0.015 (2)	−0.001 (2)
C6	0.021 (2)	0.037 (2)	0.024 (2)	−0.0022 (19)	0.015 (2)	−0.0027 (19)
C7	0.021 (2)	0.054 (3)	0.047 (3)	−0.012 (2)	0.021 (2)	−0.015 (3)
N3	0.0143 (16)	0.0228 (17)	0.0180 (17)	0.0015 (14)	0.0096 (15)	−0.0032 (13)
N4	0.031 (2)	0.0208 (19)	0.035 (2)	0.0061 (17)	0.0214 (19)	0.0049 (16)
C11	0.024 (2)	0.0206 (19)	0.014 (2)	−0.0007 (17)	0.0095 (18)	0.0008 (15)
C12	0.024 (2)	0.034 (2)	0.028 (3)	−0.0007 (19)	0.017 (2)	0.0030 (19)
C13	0.022 (2)	0.045 (3)	0.037 (3)	0.004 (2)	0.021 (2)	0.006 (2)
C14	0.021 (2)	0.029 (2)	0.034 (3)	0.0073 (19)	0.015 (2)	0.004 (2)
C15	0.022 (2)	0.0206 (19)	0.018 (2)	0.0020 (17)	0.0097 (18)	0.0008 (16)
C16	0.022 (2)	0.0187 (19)	0.027 (2)	0.0003 (17)	0.010 (2)	0.0014 (17)
Re1	0.01591 (7)	0.01429 (7)	0.01882 (8)	0.00079 (6)	0.00933 (6)	0.00002 (6)
Br1	0.0185 (2)	0.01779 (18)	0.0319 (2)	−0.00143 (16)	0.01433 (19)	−0.00067 (16)
Br2	0.0206 (2)	0.01803 (18)	0.0250 (2)	0.00517 (15)	0.01295 (18)	0.00171 (15)
Br3	0.0198 (2)	0.02158 (19)	0.0243 (2)	−0.00228 (16)	0.01344 (18)	0.00220 (16)
Br4	0.0198 (2)	0.01426 (18)	0.0349 (3)	0.00186 (15)	0.01442 (19)	−0.00040 (16)
O1	0.0256 (15)	0.0159 (13)	0.0201 (15)	0.0004 (12)	0.0147 (13)	0.0013 (11)
O2	0.0258 (15)	0.0221 (14)	0.0218 (16)	0.0022 (13)	0.0111 (13)	0.0022 (12)
Br5	0.0247 (2)	0.02478 (19)	0.0205 (2)	−0.00570 (17)	0.01438 (18)	−0.00151 (16)

Geometric parameters (Å, °)

N1—C2	1.352 (5)	N4—H5N	0.903 (19)
N1—C6	1.363 (5)	N4—H6N	0.920 (19)
N1—H1N	0.913 (19)	C11—C12	1.411 (6)

N2—C2	1.331 (6)	C12—C13	1.361 (6)
N2—H2N	0.922 (19)	C12—H12	0.9300
N2—H3N	0.915 (19)	C13—C14	1.399 (6)
C2—C3	1.404 (6)	C13—H13	0.9300
C3—C4	1.354 (6)	C14—C15	1.354 (6)
C3—H3	0.9300	C14—H14	0.9300
C4—C5	1.410 (7)	C15—C16	1.496 (6)
C4—H4	0.9300	C16—H16A	0.9600
C5—C6	1.349 (6)	C16—H16B	0.9600
C5—H5	0.9300	C16—H16C	0.9600
C6—C7	1.498 (6)	Re1—O2	1.667 (3)
C7—H7A	0.9600	Re1—O1	2.206 (3)
C7—H7B	0.9600	Re1—Br3	2.5117 (6)
C7—H7C	0.9600	Re1—Br2	2.5141 (6)
N3—C11	1.347 (5)	Re1—Br1	2.5223 (5)
N3—C15	1.363 (5)	Re1—Br4	2.5254 (6)
N3—H4N	0.920 (19)	O1—H1W	0.948 (19)
N4—C11	1.354 (5)	O1—H2W	0.955 (19)
C2—N1—C6	124.7 (4)	C13—C12—C11	118.9 (4)
C2—N1—H1N	113 (3)	C13—C12—H12	120.6
C6—N1—H1N	122 (3)	C11—C12—H12	120.6
C2—N2—H2N	124 (3)	C12—C13—C14	121.2 (4)
C2—N2—H3N	118 (3)	C12—C13—H13	119.4
H2N—N2—H3N	118 (4)	C14—C13—H13	119.4
N2—C2—N1	118.4 (4)	C15—C14—C13	119.7 (4)
N2—C2—C3	124.3 (4)	C15—C14—H14	120.2
N1—C2—C3	117.3 (4)	C13—C14—H14	120.2
C4—C3—C2	119.2 (4)	C14—C15—N3	118.1 (4)
C4—C3—H3	120.4	C14—C15—C16	125.4 (4)
C2—C3—H3	120.4	N3—C15—C16	116.5 (4)
C3—C4—C5	121.1 (4)	C15—C16—H16A	109.5
C3—C4—H4	119.5	C15—C16—H16B	109.5
C5—C4—H4	119.5	H16A—C16—H16B	109.5
C6—C5—C4	119.6 (4)	C15—C16—H16C	109.5
C6—C5—H5	120.2	H16A—C16—H16C	109.5
C4—C5—H5	120.2	H16B—C16—H16C	109.5
C5—C6—N1	118.1 (4)	O2—Re1—O1	178.68 (12)
C5—C6—C7	125.6 (4)	O2—Re1—Br3	97.54 (10)
N1—C6—C7	116.4 (4)	O1—Re1—Br3	82.06 (8)
C6—C7—H7A	109.5	O2—Re1—Br2	98.64 (10)
C6—C7—H7B	109.5	O1—Re1—Br2	82.62 (7)
H7A—C7—H7B	109.5	Br3—Re1—Br2	88.99 (2)
C6—C7—H7C	109.5	O2—Re1—Br1	98.28 (10)
H7A—C7—H7C	109.5	O1—Re1—Br1	82.12 (8)
H7B—C7—H7C	109.5	Br3—Re1—Br1	164.174 (16)
C11—N3—C15	124.7 (3)	Br2—Re1—Br1	88.83 (2)
C11—N3—H4N	116 (3)	O2—Re1—Br4	97.56 (10)
C15—N3—H4N	119 (3)	O1—Re1—Br4	81.18 (7)
C11—N4—H5N	116 (3)	Br3—Re1—Br4	88.71 (2)

C11—N4—H6N	118 (3)	Br2—Re1—Br4	163.793 (16)
H5N—N4—H6N	125 (4)	Br1—Re1—Br4	89.02 (2)
N3—C11—N4	118.5 (4)	Re1—O1—H1W	112 (3)
N3—C11—C12	117.4 (4)	Re1—O1—H2W	129 (3)
N4—C11—C12	124.1 (4)	H1W—O1—H2W	97 (4)
C6—N1—C2—N2	−179.4 (4)	C15—N3—C11—N4	179.7 (4)
C6—N1—C2—C3	0.2 (6)	C15—N3—C11—C12	1.5 (6)
N2—C2—C3—C4	178.0 (4)	N3—C11—C12—C13	−2.6 (6)
N1—C2—C3—C4	−1.6 (6)	N4—C11—C12—C13	179.2 (4)
C2—C3—C4—C5	1.2 (7)	C11—C12—C13—C14	1.4 (7)
C3—C4—C5—C6	0.6 (7)	C12—C13—C14—C15	1.1 (7)
C4—C5—C6—N1	−2.0 (7)	C13—C14—C15—N3	−2.2 (7)
C4—C5—C6—C7	178.3 (5)	C13—C14—C15—C16	176.5 (4)
C2—N1—C6—C5	1.6 (6)	C11—N3—C15—C14	1.0 (6)
C2—N1—C6—C7	−178.7 (4)	C11—N3—C15—C16	−177.8 (4)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···Br5	0.92 (3)	2.54 (5)	3.354 (4)	148 (5)
O1—H1W···Br5^{i}	0.95 (4)	2.25 (4)	3.176 (3)	165 (4)
N2—H2N···Br1^{ii}	0.92 (5)	2.76 (5)	3.573 (5)	148 (4)
N2—H2N···Br4^{ii}	0.92 (5)	2.91 (5)	3.424 (6)	117 (4)
O1—H2W···Br5^{iii}	0.95 (3)	2.28 (3)	3.225 (4)	170 (4)
N2—H3N···Br5	0.92 (2)	2.75 (4)	3.514 (4)	141 (3)
N3—H4N···Br5^{iv}	0.92 (5)	2.42 (5)	3.273 (4)	154 (4)
N4—H5N···Br2^{v}	0.90 (5)	2.83 (5)	3.592 (6)	143 (4)
N4—H6N···Br4^{vi}	0.92 (4)	2.79 (5)	3.647 (5)	155 (5)

Symmetry codes: (i) −*x*, −*y*+2, −*z*+1; (ii) *x*, −*y*+3/2, *z*+1/2; (iii) *x*−1, *y*, *z*−1; (iv) *x*, −*y*+3/2, *z*−1/2; (v) *x*+1, −*y*+3/2, *z*+1/2; (vi) −*x*, −*y*+1, −*z*+1.

Fig. 1

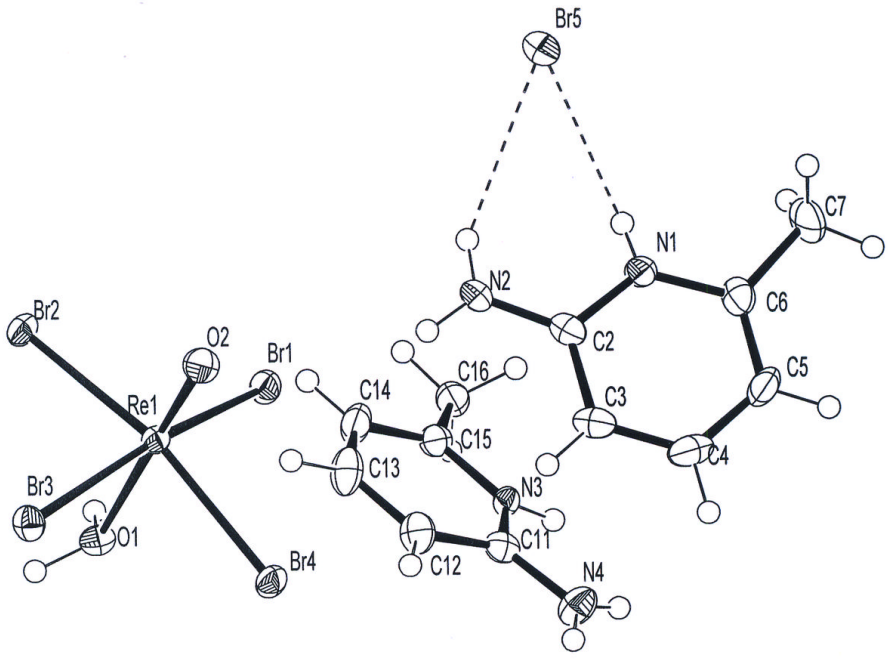
Br5
C7
N1
N2
C6
Br2
O2
Br1
C2
C16
C5
C14
C3
Re1
C15
C4
C13
Br3
N3
O1
C11
Br4
C12
N4

Fig. 2