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Tetrakis(2,2′:6′,2″-terpyridinium) hexabromidorhenate(IV) bis[aquatetrabromidooxidorhenate(V)] tetrabromide dihydrate

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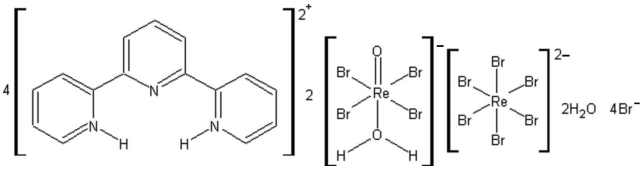
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.009 Å; R factor = 0.036; wR factor = 0.062; data-to-parameter ratio = 20.7.

The title compound, $(C_{15}H_{13}N_3)_4[ReBr_6][ReBr_4O(H_2O)]_2Br_4\cdot 2H_2O$, is a mixed-valence molecular salt containing rhenium(IV) (as centrosymmetric $[ReBr_6]^{2-}$ anions) and rhenium(V) (as *trans*-$[ReO(H_2O)Br_4]^{-}$ oxidoanions). The Re—O and Re—Br distances are consistent with the different oxidation states of Re in the anions. A complex hydrogen-bonding network helps to establish the crystal packing.

Related literature

For related literature, see: Abram *et al.* (1996); Chiozzone *et al.* (2006); Kochel (2007); Rose *et al.* (1996); Preetz & Struess (1998); Watt & Thompson (1963).



Experimental

Crystal data

$(C_{15}H_{13}N_3)_4[ReBr_6]$-$[ReBr_4O(H_2O)]_2Br_4\cdot 2H_2O$
M_r = 3042.03
Triclinic, $P\overline{1}$
a = 10.3458 (6) Å
b = 10.3563 (6) Å
c = 19.0582 (14) Å
α = 81.361 (6)°
β = 79.041 (6)°
γ = 79.987 (5)°
V = 1959.7 (2) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 13.85 mm^{-1}
T = 100 (2) K
0.10 × 0.10 × 0.05 mm

Data collection

Oxford Diffraction KM-4-CCD diffractometer
Absorption correction: numerical (*CrysAlis RED*; Oxford Diffraction, 2003)
T_{min} = 0.456, T_{max} = 0.794
24231 measured reflections
9275 independent reflections
5991 reflections with $I > 2\sigma(I)$
R_{int} = 0.056

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.036
$wR(F^2)$ = 0.062
S = 0.94
9275 reflections
448 parameters
4 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.94 e Å^{-3}

Table 1
Selected bond lengths (Å).

Re1—Br3	2.5044 (6)	Re2—Br7	2.5108 (7)
Re1—Br2	2.5099 (6)	Re2—Br4	2.5123 (7)
Re1—Br1	2.5179 (6)	Re2—Br5	2.5182 (7)
Re2—O2	1.663 (4)	Re2—Br6	2.5239 (7)
Re2—O1	2.246 (4)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1*N*···Br8^{i}	0.86	2.47	3.228 (6)	146
O1—H1*W*···Br8^{ii}	0.89	2.46	3.310 (4)	157
O1—H2*W*···O3*A*^{ii}	0.88	1.83	2.710 (5)	166
N3—H3*N*···Br8^{i}	0.85	2.48	3.236 (5)	147
O3*A*—H3*W*···Br5^{iii}	0.88	2.68	3.517 (4)	157
N4—H4*N*···Br9^{iii}	0.86	2.51	3.247 (6)	143
O3*A*—H4*W*···Br9	0.89	2.45	3.338 (4)	171
N6—H6*N*···Br9^{iii}	0.85	2.48	3.245 (5)	147
C9—H9···Br3^{i}	0.93	2.76	3.664 (6)	162
C15—H15···Br9^{i}	0.92	2.81	3.598 (6)	142
C22—H22···O3*A*	0.92	2.58	3.504 (8)	170
C23—H23···Br7^{iv}	0.92	2.88	3.810 (7)	172
C29—H29···Br4^{v}	0.93	2.85	3.738 (6)	158
C35—H35···Br8^{iii}	0.92	2.73	3.474 (6)	137

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

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) and *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: HB2433).

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supplementary materials

Acta Cryst. (2007). E**63**, m1968 [**doi:10.1107/S1600536807029923**]

Tetrakis(2,2':6',2''-terpyridinium) hexabromidorhenate(IV) bis[aquatetrabromidooxidorhenate(V)] tetrabromide dihydrate

A. Kochel

Comment

This paper reports the crystal structure of the title mixed valence rhenium compound, (I). One of rhenium atoms lies on a centre of symmetry and other one in general position.

Compound (I) (Fig. 1) and some of its derivatives show interesting magnetic properties (Rose *et al.*, 1996). Several papers describing the crystal structures of bromorhenates are available in the literature (Preetz & Struess, 1988, Chiozzone *et al.*, 2006) but surprisingly the crystal structure of (I) has not been determined yet.

The $[ReO(H_2O)Br_4]^-$ units in (I) shows a distorted octahedral geometry about the Re(V) site defined by the terminal oxo ligand, the *trans* aqua ligand, and four bromide ions (Table 1). The Re—Br bond lengths in (I) agree with those in related crystal structures (Kochel, 2007; Abram *et al.*, 1996).

In the crystal packing of (I), layers of rhenate anions and terpyridinium cations could be distinguished. However, the anion layer is additionally filled with the water molecules and bromide anions. The crystal structure is consolidated by a complex hydrogen bond system including various C—H···O links (Table 2).

Experimental

$(NH_4)2ReBr_6$ was obtained by the method of Watt & Thompson (1963). A mixture of $(NH_4)2ReBr_6$ (0.46 g) and the terpy lignad (0.50 g) was dissolved in 50-ml of 1:1 *v*/*v* water-ethanol solution. Then it was sealed under nitrogen and heated for 15 h to 423 K. After the reaction the mixture colour was yellow orange. The solution was diluted with ethanol and left standing for evaporation. After five days orange plates of (I) appeared. Anal. Calc. C23.69 H 1.98 N 5.52 /% found C 21.40 H 1.20 N 5.14 /%.

Refinement

The water H atoms were located in a difference map and refined as riding in their as-found relative positions with U_{iso}(H) = 1.2U_{eq}(O). The other H atoms were placed in idealized positions (N—H = 0.86 Å, C—H = 0.93 Å) and refined as riding with U_{iso}(H) = 1.2U_{eq}(carrier).

Figures

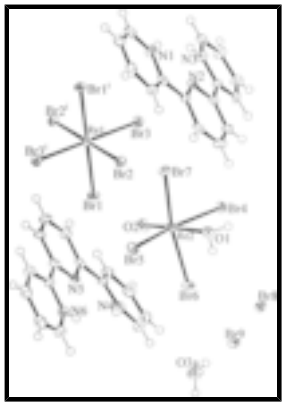

Fig. 1. The molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). Symmetry code: (i) −*x*, 1 − *y*, 1 − *z*.

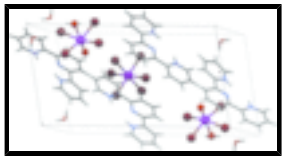

Fig. 2. The crystal packing in (I).

Tetrakis(2,2':6',2''-terpyridinium) hexabromidorhenate(IV) bis[aquatetrabromidooxidorhenate(V)] tetrabromide dihydrate

Crystal data

$(C_{15}H_{13}N_3)_4[ReBr_6][ReBr_4O(H_2O)]_2Br_4·2H_2O$	$Z = 1$
$M_r = 3042.03$	$F_{000} = 1407$
Triclinic, $P\overline{1}$	$D_x = 2.578$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 10.3458$ (6) Å	Cell parameters from 5311 reflections
$b = 10.3563$ (6) Å	$\theta = 2.7$–28.6º
$c = 19.0582$ (14) Å	$\mu = 13.85$ mm^{-1}
$\alpha = 81.361$ (6)º	$T = 100$ (2) K
$\beta = 79.041$ (6)º	Prism, orange
$\gamma = 79.987$ (5)º	0.10 × 0.10 × 0.05 mm
$V = 1959.7$ (2) Å^3	

Data collection

Oxford Diffraction KM-4-CCD diffractometer	9275 independent reflections
Radiation source: fine-focus sealed tube	5991 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.056$
$T = 100$(2) K	$\theta_{max} = 28.6$º
ω scans	$\theta_{min} = 2.7$º
Absorption correction: numerical (CrysAlis RED; Oxford Diffraction, 2003)	$h = -13 \rightarrow 13$

T_{min} = 0.456, T_{max} = 0.794	k = −13→13
24231 measured reflections	l = −25→25

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.036	H-atom parameters constrained
$wR(F^2)$ = 0.062	$w = 1/[\sigma^2(F_o^2) + (0.0215P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 0.94	$(\Delta/\sigma)_{max} < 0.001$
9275 reflections	$\Delta\rho_{max}$ = 1.24 e Å^{-3}
448 parameters	$\Delta\rho_{min}$ = −0.94 e Å^{-3}
4 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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 $F^2 > 2$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	0.1742 (5)	0.0591 (5)	0.6381 (3)	0.0177 (12)
H1N	0.1977	0.1054	0.6661	0.021*
N2	0.3703 (5)	0.2058 (5)	0.6009 (3)	0.0173 (12)
N3	0.3926 (5)	0.3432 (5)	0.7045 (3)	0.0186 (12)
H3N	0.3393	0.2863	0.7089	0.022*
C1	0.0747 (7)	−0.0095 (6)	0.6643 (4)	0.0261 (16)
H1	0.0315	−0.0047	0.7116	0.031*
C2	0.0346 (6)	−0.0877 (6)	0.6222 (3)	0.0210 (15)
H2	−0.0351	−0.1356	0.6403	0.025*
C3	0.1013 (6)	−0.0925 (6)	0.5524 (4)	0.0255 (16)
H3	0.0786	−0.1473	0.5237	0.031*
C4	0.2019 (6)	−0.0166 (6)	0.5246 (3)	0.0193 (14)
H4	0.2430	−0.0165	0.4768	0.023*
C5	0.2408 (6)	0.0599 (5)	0.5696 (3)	0.0155 (14)
C6	0.3483 (6)	0.1416 (5)	0.5493 (3)	0.0145 (13)

C7	0.4232 (6)	0.1521 (6)	0.4791 (3)	0.0211 (15)
H7	0.4062	0.1074	0.4439	0.025*
C8	0.5220 (7)	0.2300 (6)	0.4644 (3)	0.0215 (15)
H8	0.5728	0.2386	0.4186	0.026*
C9	0.5459 (6)	0.2957 (6)	0.5175 (3)	0.0190 (14)
H9	0.6139	0.3472	0.5084	0.023*
C10	0.4666 (6)	0.2831 (6)	0.5845 (3)	0.0160 (14)
C11	0.4774 (6)	0.3597 (6)	0.6420 (3)	0.0168 (14)
C12	0.5655 (6)	0.4496 (6)	0.6356 (3)	0.0206 (15)
H12	0.6271	0.4617	0.5937	0.025*
C13	0.5607 (6)	0.5214 (6)	0.6924 (3)	0.0201 (15)
H13	0.6187	0.5824	0.6883	0.024*
C14	0.4699 (6)	0.5021 (6)	0.7550 (3)	0.0214 (15)
H14	0.4654	0.5506	0.7929	0.026*
C15	0.3863 (6)	0.4102 (6)	0.7604 (3)	0.0206 (15)
H15	0.3259	0.3945	0.8024	0.025*
N4	0.2439 (5)	0.5363 (5)	0.1040 (3)	0.0184 (12)
H4N	0.2335	0.4798	0.0775	0.022*
N5	0.0414 (5)	0.3972 (5)	0.1317 (2)	0.0158 (11)
N6	0.0141 (5)	0.2830 (5)	0.0195 (3)	0.0218 (13)
H6N	0.0713	0.3348	0.0182	0.026*
C21	0.3407 (6)	0.6082 (6)	0.0809 (4)	0.0235 (15)
H21	0.3943	0.5978	0.0364	0.028*
C22	0.3632 (7)	0.6978 (6)	0.1214 (4)	0.0251 (16)
H22	0.4321	0.7476	0.1055	0.030*
C23	0.2800 (7)	0.7120 (6)	0.1872 (4)	0.0245 (16)
H23	0.2921	0.7732	0.2155	0.029*
C24	0.1791 (6)	0.6352 (6)	0.2107 (3)	0.0222 (15)
H24	0.1249	0.6432	0.2552	0.027*
C25	0.1599 (6)	0.5473 (6)	0.1676 (3)	0.0182 (14)
C26	0.0557 (6)	0.4606 (6)	0.1854 (3)	0.0173 (14)
C27	−0.0240 (6)	0.4503 (6)	0.2539 (3)	0.0203 (15)
H27	−0.0135	0.4973	0.2898	0.024*
C28	−0.1191 (6)	0.3664 (6)	0.2652 (3)	0.0244 (16)
H28	−0.1721	0.3543	0.3103	0.029*
C29	−0.1359 (6)	0.3010 (6)	0.2104 (3)	0.0203 (15)
H29	−0.2008	0.2462	0.2175	0.024*
C30	−0.0533 (6)	0.3188 (6)	0.1443 (3)	0.0186 (14)
C31	−0.0661 (6)	0.2550 (6)	0.0832 (3)	0.0182 (14)
C32	−0.1550 (6)	0.1664 (6)	0.0841 (4)	0.0265 (16)
H32	−0.2101	0.1411	0.1267	0.032*
C33	−0.1602 (6)	0.1163 (6)	0.0211 (4)	0.0243 (16)
H33	−0.2198	0.0584	0.0216	0.029*
C34	−0.0770 (6)	0.1524 (6)	−0.0426 (4)	0.0256 (16)
H34	−0.0807	0.1205	−0.0851	0.031*
C35	0.0101 (6)	0.2361 (6)	−0.0406 (3)	0.0220 (15)
H35	0.0682	0.2601	−0.0822	0.026*
Re2	0.38002 (2)	0.12190 (2)	0.218448 (13)	0.01424 (7)
Br4	0.56622 (6)	0.13304 (6)	0.28386 (3)	0.01760 (14)

Br5	0.23701 (6)	0.05495 (6)	0.14103 (3)	0.02149 (15)
Br6	0.49427 (7)	0.24963 (6)	0.10751 (3)	0.02226 (15)
Br7	0.30660 (6)	−0.05229 (6)	0.31752 (3)	0.01964 (15)
O1	0.5235 (4)	−0.0491 (4)	0.1763 (2)	0.0189 (10)
H1W	0.6001	−0.0612	0.1932	0.023*
H2W	0.5545	−0.0555	0.1300	0.023*
O2	0.2755 (4)	0.2512 (4)	0.2478 (2)	0.0202 (10)
Re1	0.0000	0.5000	0.5000	0.01419 (9)
Br1	0.07213 (6)	0.68849 (6)	0.40966 (3)	0.01884 (15)
Br2	0.14274 (6)	0.34007 (6)	0.42001 (3)	0.01800 (14)
Br3	0.19123 (6)	0.50460 (6)	0.56235 (3)	0.02026 (15)
O3A	0.6108 (4)	0.8904 (4)	0.0401 (2)	0.0269 (11)
H3W	0.6256	0.9008	−0.0073	0.032*
H4W	0.6422	0.8045	0.0481	0.032*
Br8	0.82755 (6)	0.83705 (6)	0.21020 (3)	0.01960 (15)
Br9	0.73144 (6)	0.56873 (6)	0.05088 (3)	0.01989 (15)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.022 (3)	0.015 (3)	0.020 (3)	−0.003 (2)	−0.005 (2)	−0.012 (2)
N2	0.019 (3)	0.012 (3)	0.019 (3)	0.001 (2)	−0.004 (2)	0.004 (2)
N3	0.023 (3)	0.016 (3)	0.018 (3)	−0.004 (2)	−0.003 (2)	−0.004 (2)
C1	0.028 (4)	0.029 (4)	0.020 (4)	0.003 (3)	−0.010 (3)	−0.001 (3)
C2	0.022 (4)	0.016 (3)	0.025 (4)	−0.002 (3)	−0.006 (3)	0.000 (3)
C3	0.021 (4)	0.024 (4)	0.033 (4)	0.004 (3)	−0.006 (3)	−0.011 (3)
C4	0.020 (4)	0.025 (4)	0.015 (3)	0.001 (3)	−0.005 (3)	−0.010 (3)
C5	0.022 (4)	0.009 (3)	0.016 (3)	0.003 (3)	−0.007 (3)	−0.003 (3)
C6	0.016 (3)	0.009 (3)	0.017 (3)	0.004 (2)	−0.002 (3)	−0.003 (3)
C7	0.025 (4)	0.021 (4)	0.016 (3)	0.007 (3)	−0.009 (3)	−0.004 (3)
C8	0.030 (4)	0.021 (4)	0.013 (3)	−0.004 (3)	−0.002 (3)	−0.005 (3)
C9	0.020 (4)	0.020 (3)	0.016 (3)	−0.002 (3)	0.000 (3)	−0.002 (3)
C10	0.021 (4)	0.013 (3)	0.014 (3)	−0.001 (3)	−0.008 (3)	0.000 (3)
C11	0.016 (3)	0.017 (3)	0.018 (3)	0.001 (3)	−0.002 (3)	−0.005 (3)
C12	0.009 (3)	0.026 (4)	0.025 (4)	−0.002 (3)	−0.001 (3)	−0.001 (3)
C13	0.024 (4)	0.017 (3)	0.022 (4)	−0.008 (3)	−0.004 (3)	−0.005 (3)
C14	0.026 (4)	0.015 (3)	0.025 (4)	−0.005 (3)	−0.002 (3)	−0.010 (3)
C15	0.021 (4)	0.021 (4)	0.020 (4)	0.000 (3)	−0.007 (3)	−0.002 (3)
N4	0.020 (3)	0.017 (3)	0.018 (3)	−0.002 (2)	−0.001 (2)	−0.003 (2)
N5	0.018 (3)	0.015 (3)	0.012 (3)	0.002 (2)	−0.002 (2)	0.002 (2)
N6	0.021 (3)	0.023 (3)	0.023 (3)	−0.008 (2)	−0.004 (3)	0.000 (3)
C21	0.021 (4)	0.024 (4)	0.026 (4)	−0.006 (3)	0.001 (3)	−0.007 (3)
C22	0.022 (4)	0.020 (4)	0.035 (4)	−0.010 (3)	−0.006 (3)	0.003 (3)
C23	0.032 (4)	0.011 (3)	0.033 (4)	−0.001 (3)	−0.015 (3)	−0.002 (3)
C24	0.023 (4)	0.024 (4)	0.017 (3)	−0.001 (3)	−0.003 (3)	−0.001 (3)
C25	0.022 (4)	0.017 (3)	0.016 (3)	−0.004 (3)	−0.008 (3)	0.003 (3)
C26	0.019 (4)	0.015 (3)	0.018 (3)	0.002 (3)	−0.008 (3)	0.000 (3)
C27	0.024 (4)	0.020 (4)	0.017 (3)	−0.001 (3)	−0.005 (3)	−0.007 (3)

C28	0.027 (4)	0.022 (4)	0.016 (3)	0.003 (3)	0.007 (3)	0.002 (3)
C29	0.026 (4)	0.016 (3)	0.018 (3)	−0.006 (3)	−0.006 (3)	0.008 (3)
C30	0.019 (4)	0.013 (3)	0.023 (4)	−0.003 (3)	−0.004 (3)	0.002 (3)
C31	0.019 (4)	0.021 (3)	0.016 (3)	−0.005 (3)	−0.004 (3)	0.000 (3)
C32	0.022 (4)	0.031 (4)	0.024 (4)	−0.004 (3)	−0.005 (3)	0.003 (3)
C33	0.026 (4)	0.022 (4)	0.029 (4)	−0.003 (3)	−0.012 (3)	−0.007 (3)
C34	0.027 (4)	0.022 (4)	0.031 (4)	−0.004 (3)	−0.012 (3)	−0.003 (3)
C35	0.023 (4)	0.025 (4)	0.017 (3)	0.002 (3)	−0.001 (3)	−0.010 (3)
Re2	0.01566 (15)	0.01453 (14)	0.01316 (14)	−0.00219 (11)	−0.00329 (11)	−0.00269 (11)
Br4	0.0190 (4)	0.0191 (3)	0.0172 (3)	−0.0046 (3)	−0.0060 (3)	−0.0042 (3)
Br5	0.0228 (4)	0.0224 (4)	0.0227 (4)	−0.0037 (3)	−0.0104 (3)	−0.0050 (3)
Br6	0.0269 (4)	0.0208 (4)	0.0179 (3)	−0.0054 (3)	−0.0030 (3)	0.0028 (3)
Br7	0.0213 (4)	0.0215 (4)	0.0153 (3)	−0.0064 (3)	0.0005 (3)	−0.0007 (3)
O1	0.021 (2)	0.017 (2)	0.019 (2)	−0.0025 (18)	−0.0044 (19)	−0.0049 (19)
O2	0.023 (2)	0.018 (2)	0.021 (2)	0.0033 (18)	−0.009 (2)	−0.0073 (19)
Re1	0.0168 (2)	0.01280 (19)	0.01210 (19)	−0.00249 (15)	−0.00024 (16)	−0.00106 (15)
Br1	0.0234 (4)	0.0151 (3)	0.0161 (3)	−0.0048 (3)	0.0003 (3)	0.0017 (3)
Br2	0.0175 (3)	0.0175 (3)	0.0183 (3)	−0.0012 (3)	−0.0004 (3)	−0.0051 (3)
Br3	0.0178 (3)	0.0245 (4)	0.0190 (3)	−0.0031 (3)	−0.0045 (3)	−0.0027 (3)
O3A	0.038 (3)	0.023 (3)	0.019 (2)	−0.004 (2)	0.003 (2)	−0.010 (2)
Br8	0.0198 (3)	0.0238 (4)	0.0159 (3)	−0.0051 (3)	−0.0010 (3)	−0.0052 (3)
Br9	0.0225 (4)	0.0212 (4)	0.0155 (3)	−0.0050 (3)	0.0005 (3)	−0.0033 (3)

Geometric parameters (Å, °)

N1—C1	1.329 (8)	C21—C22	1.368 (8)
N1—C5	1.354 (7)	C21—H21	0.9300
N1—H1N	0.8600	C22—C23	1.391 (9)
N2—C6	1.340 (7)	C22—H22	0.9300
N2—C10	1.348 (7)	C23—C24	1.388 (8)
N3—C15	1.341 (7)	C23—H23	0.9299
N3—C11	1.345 (7)	C24—C25	1.376 (8)
N3—H3N	0.8600	C24—H24	0.9301
C1—C2	1.382 (8)	C25—C26	1.479 (8)
C1—H1	0.9298	C26—C27	1.405 (8)
C2—C3	1.380 (9)	C27—C28	1.389 (8)
C2—H2	0.9300	C27—H27	0.9300
C3—C4	1.390 (8)	C28—C29	1.380 (8)
C3—H3	0.9301	C28—H28	0.9300
C4—C5	1.405 (8)	C29—C30	1.388 (8)
C4—H4	0.9300	C29—H29	0.9299
C5—C6	1.472 (8)	C30—C31	1.460 (8)
C6—C7	1.412 (8)	C31—C32	1.404 (8)
C7—C8	1.372 (8)	C32—C33	1.391 (8)
C7—H7	0.9299	C32—H32	0.9300
C8—C9	1.383 (8)	C33—C34	1.390 (9)
C8—H8	0.9301	C33—H33	0.9299
C9—C10	1.382 (8)	C34—C35	1.365 (8)
C9—H9	0.9300	C34—H34	0.9299

C10—C11	1.476 (8)	C35—H35	0.9300
C11—C12	1.390 (8)	Re1—Br3^{i}	2.5043 (6)
C12—C13	1.390 (8)	Re1—Br3	2.5044 (6)
C12—H12	0.9301	Re1—Br2	2.5099 (6)
C13—C14	1.384 (8)	Re1—Br2^{i}	2.5100 (6)
C13—H13	0.9300	Re1—Br1	2.5179 (6)
C14—C15	1.374 (8)	Re1—Br1^{i}	2.5180 (6)
C14—H14	0.9299	Re2—O2	1.663 (4)
C15—H15	0.9299	Re2—O1	2.246 (4)
N4—C21	1.321 (7)	Re2—Br7	2.5108 (7)
N4—C25	1.359 (7)	Re2—Br4	2.5123 (7)
N4—H4N	0.8600	Re2—Br5	2.5182 (7)
N5—C26	1.340 (7)	Re2—Br6	2.5239 (7)
N5—C30	1.343 (7)	O1—H1W	0.8929
N6—C35	1.318 (7)	O1—H2W	0.8879
N6—C31	1.357 (7)	O3A—H3W	0.8787
N6—H6N	0.8600	O3A—H4W	0.8932
C1—N1—C5	123.5 (5)	C25—C24—H24	120.3
C1—N1—H1N	118.2	C23—C24—H24	120.2
C5—N1—H1N	118.2	N4—C25—C24	118.2 (6)
C6—N2—C10	118.4 (5)	N4—C25—C26	116.5 (5)
C15—N3—C11	124.0 (5)	C24—C25—C26	125.3 (6)
C15—N3—H3N	118.0	N5—C26—C27	123.4 (6)
C11—N3—H3N	118.0	N5—C26—C25	115.5 (5)
N1—C1—C2	120.7 (6)	C27—C26—C25	121.1 (6)
N1—C1—H1	119.6	C28—C27—C26	116.7 (6)
C2—C1—H1	119.6	C28—C27—H27	121.7
C3—C2—C1	118.0 (6)	C26—C27—H27	121.6
C3—C2—H2	121.0	C29—C28—C27	120.7 (6)
C1—C2—H2	121.0	C29—C28—H28	119.7
C2—C3—C4	120.8 (6)	C27—C28—H28	119.6
C2—C3—H3	119.6	C28—C29—C30	118.2 (6)
C4—C3—H3	119.6	C28—C29—H29	120.9
C3—C4—C5	119.2 (6)	C30—C29—H29	120.8
C3—C4—H4	120.4	N5—C30—C29	122.8 (6)
C5—C4—H4	120.4	N5—C30—C31	115.5 (5)
N1—C5—C4	117.6 (6)	C29—C30—C31	121.7 (6)
N1—C5—C6	116.5 (5)	N6—C31—C32	116.3 (6)
C4—C5—C6	125.9 (6)	N6—C31—C30	117.9 (5)
N2—C6—C7	122.0 (6)	C32—C31—C30	125.7 (6)
N2—C6—C5	116.3 (5)	C33—C32—C31	119.8 (6)
C7—C6—C5	121.7 (6)	C33—C32—H32	120.1
C8—C7—C6	118.2 (6)	C31—C32—H32	120.1
C8—C7—H7	120.9	C34—C33—C32	120.4 (6)
C6—C7—H7	120.9	C34—C33—H33	119.8
C7—C8—C9	120.1 (6)	C32—C33—H33	119.8
C7—C8—H8	119.9	C35—C34—C33	117.8 (6)
C9—C8—H8	120.0	C35—C34—H34	121.1

C10—C9—C8	118.5 (6)	C33—C34—H34	121.1
C10—C9—H9	120.7	N6—C35—C34	121.2 (6)
C8—C9—H9	120.8	N6—C35—H35	119.4
N2—C10—C9	122.7 (6)	C34—C35—H35	119.4
N2—C10—C11	115.4 (5)	O2—Re2—O1	178.43 (18)
C9—C10—C11	121.8 (5)	O2—Re2—Br7	98.52 (14)
N3—C11—C12	117.9 (6)	O1—Re2—Br7	83.05 (10)
N3—C11—C10	117.1 (5)	O2—Re2—Br4	98.19 (13)
C12—C11—C10	125.0 (6)	O1—Re2—Br4	81.84 (10)
C11—C12—C13	119.5 (6)	Br7—Re2—Br4	87.55 (2)
C11—C12—H12	120.2	O2—Re2—Br5	97.92 (13)
C13—C12—H12	120.3	O1—Re2—Br5	82.07 (10)
C14—C13—C12	120.1 (6)	Br7—Re2—Br5	89.73 (2)
C14—C13—H13	119.9	Br4—Re2—Br5	163.89 (2)
C12—C13—H13	119.9	O2—Re2—Br6	95.66 (14)
C15—C14—C13	119.0 (6)	O1—Re2—Br6	82.77 (10)
C15—C14—H14	120.5	Br7—Re2—Br6	165.81 (2)
C13—C14—H14	120.5	Br4—Re2—Br6	90.20 (2)
N3—C15—C14	119.5 (6)	Br5—Re2—Br6	88.55 (2)
N3—C15—H15	120.3	Re2—O1—H1W	112.3
C14—C15—H15	120.3	Re2—O1—H2W	124.6
C21—N4—C25	123.1 (5)	H1W—O1—H2W	99.9
C21—N4—H4N	118.4	$Br3^{i}$—Re1—Br3	180.0
C25—N4—H4N	118.4	$Br3^{i}$—Re1—Br2	89.34 (2)
C26—N5—C30	118.1 (5)	Br3—Re1—Br2	90.66 (2)
C35—N6—C31	124.4 (5)	$Br3^{i}$—Re1—$Br2^{i}$	90.66 (2)
C35—N6—H6N	117.8	Br3—Re1—$Br2^{i}$	89.34 (2)
C31—N6—H6N	117.8	Br2—Re1—$Br2^{i}$	180.0
N4—C21—C22	120.9 (6)	$Br3^{i}$—Re1—Br1	90.53 (2)
N4—C21—H21	119.5	Br3—Re1—Br1	89.47 (2)
C22—C21—H21	119.6	Br2—Re1—Br1	89.44 (2)
C21—C22—C23	118.0 (6)	$Br2^{i}$—Re1—Br1	90.56 (2)
C21—C22—H22	121.0	$Br3^{i}$—Re1—$Br1^{i}$	89.47 (2)
C23—C22—H22	121.0	Br3—Re1—$Br1^{i}$	90.53 (2)
C24—C23—C22	120.3 (6)	Br2—Re1—$Br1^{i}$	90.56 (2)
C24—C23—H23	119.9	$Br2^{i}$—Re1—$Br1^{i}$	89.44 (2)
C22—C23—H23	119.9	Br1—Re1—$Br1^{i}$	180.0
C25—C24—C23	119.5 (6)	H3W—O3A—H4W	99.5
C5—N1—C1—C2	1.3 (9)	C25—N4—C21—C22	1.2 (9)
N1—C1—C2—C3	0.2 (9)	N4—C21—C22—C23	−0.8 (9)
C1—C2—C3—C4	−2.6 (9)	C21—C22—C23—C24	1.0 (9)
C2—C3—C4—C5	3.6 (9)	C22—C23—C24—C25	−1.6 (9)
C1—N1—C5—C4	−0.3 (8)	C21—N4—C25—C24	−1.7 (9)
C1—N1—C5—C6	179.8 (5)	C21—N4—C25—C26	179.4 (6)
C3—C4—C5—N1	−2.1 (8)	C23—C24—C25—N4	1.9 (9)
C3—C4—C5—C6	177.7 (6)	C23—C24—C25—C26	−179.3 (6)

C10—N2—C6—C7	0.4 (8)	C30—N5—C26—C27	−0.6 (9)
C10—N2—C6—C5	−179.2 (5)	C30—N5—C26—C25	−178.9 (5)
N1—C5—C6—N2	0.6 (7)	N4—C25—C26—N5	−10.8 (8)
C4—C5—C6—N2	−179.2 (5)	C24—C25—C26—N5	170.4 (5)
N1—C5—C6—C7	−178.9 (5)	N4—C25—C26—C27	170.9 (5)
C4—C5—C6—C7	1.2 (9)	C24—C25—C26—C27	−7.9 (9)
N2—C6—C7—C8	0.6 (9)	N5—C26—C27—C28	1.8 (9)
C5—C6—C7—C8	−179.9 (5)	C25—C26—C27—C28	180.0 (5)
C6—C7—C8—C9	−0.1 (9)	C26—C27—C28—C29	−2.2 (9)
C7—C8—C9—C10	−1.4 (9)	C27—C28—C29—C30	1.5 (9)
C6—N2—C10—C9	−1.9 (8)	C26—N5—C30—C29	−0.2 (9)
C6—N2—C10—C11	174.9 (5)	C26—N5—C30—C31	178.5 (5)
C8—C9—C10—N2	2.4 (9)	C28—C29—C30—N5	−0.3 (9)
C8—C9—C10—C11	−174.2 (5)	C28—C29—C30—C31	−178.9 (5)
C15—N3—C11—C12	1.4 (9)	C35—N6—C31—C32	2.2 (9)
C15—N3—C11—C10	−176.9 (5)	C35—N6—C31—C30	−177.6 (6)
N2—C10—C11—N3	1.1 (8)	N5—C30—C31—N6	−2.1 (8)
C9—C10—C11—N3	177.9 (5)	C29—C30—C31—N6	176.7 (5)
N2—C10—C11—C12	−177.1 (6)	N5—C30—C31—C32	178.1 (6)
C9—C10—C11—C12	−0.2 (9)	C29—C30—C31—C32	−3.1 (10)
N3—C11—C12—C13	−1.8 (9)	N6—C31—C32—C33	−2.2 (9)
C10—C11—C12—C13	176.3 (5)	C30—C31—C32—C33	177.7 (6)
C11—C12—C13—C14	0.8 (9)	C31—C32—C33—C34	0.7 (10)
C12—C13—C14—C15	0.8 (9)	C32—C33—C34—C35	1.0 (9)
C11—N3—C15—C14	0.2 (9)	C31—N6—C35—C34	−0.7 (10)
C13—C14—C15—N3	−1.3 (9)	C33—C34—C35—N6	−1.0 (9)

Symmetry codes: (i) −*x*, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···Br8^{ii}	0.86	2.47	3.228 (6)	146
O1—H1W···Br8^{iii}	0.89	2.46	3.310 (4)	157
O1—H2W···O3A^{iii}	0.88	1.83	2.710 (5)	166
N3—H3N···Br8^{ii}	0.85	2.48	3.236 (5)	147
O3A—H3W···Br5^{iv}	0.88	2.68	3.517 (4)	157
N4—H4N···Br9^{iv}	0.86	2.51	3.247 (6)	143
O3A—H4W···Br9	0.89	2.45	3.338 (4)	171
N6—H6N···Br9^{iv}	0.85	2.48	3.245 (5)	147
C9—H9···Br3^{ii}	0.93	2.76	3.664 (6)	162
C15—H15···Br9^{ii}	0.92	2.81	3.598 (6)	142
C22—H22···O3A	0.92	2.58	3.504 (8)	170
C23—H23···Br7^{v}	0.92	2.88	3.810 (7)	172
C29—H29···Br4^{vi}	0.93	2.85	3.738 (6)	158
C35—H35···Br8^{iv}	0.92	2.73	3.474 (6)	137

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

Fig. 1

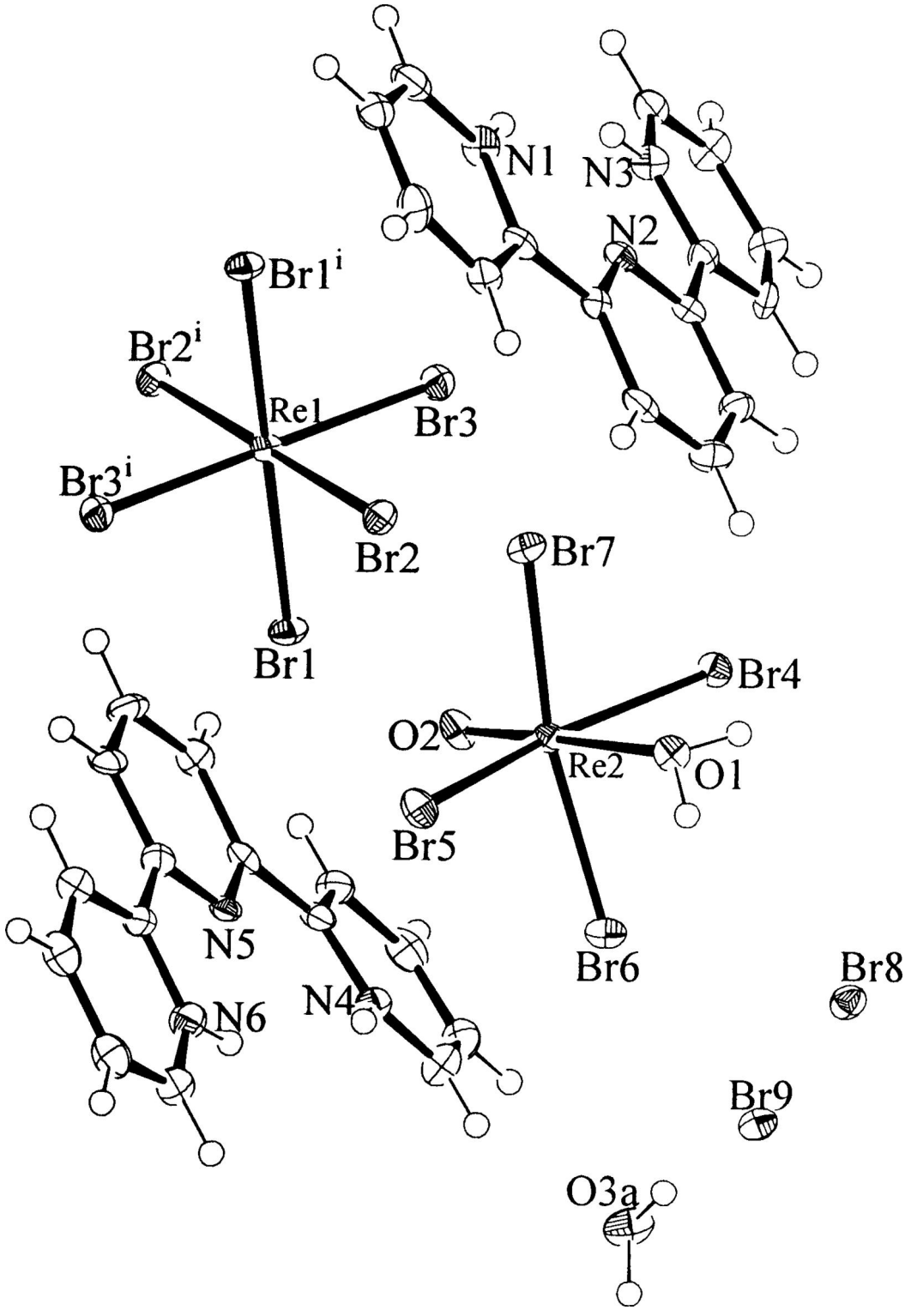
N1
N3
N2
Br1i
Br2i
Re1
Br3
Br3i
Br2
Br1
Br7
Br4
O2
Re2
O1
Br5
N5
Br6
Br8
N6
N4
Br9
O3a

Fig. 2