

Aquatricarbonyl(4-carboxypyridine-2-carboxylato- κ^2N,O^2)rhenium(I)

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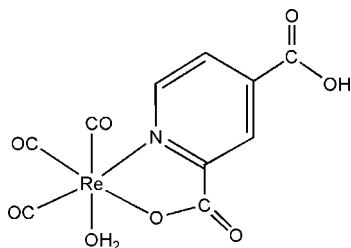
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(C-C) = 0.007$ Å; R factor = 0.027; wR factor = 0.065; data-to-parameter ratio = 15.7.

There are two molecules with similar bond dimensions in the asymmetric unit of the title complex, $[Re(C_7H_4NO_4)(CO)_3(H_2O)]$. The metal centre is coordinated facially by three carbonyl groups, is chelated by a 4-carboxypyridine-2-carboxylate ligand and is also coordinated by a water molecule. $O-H \cdots O$ hydrogen bonds give rise to a three-dimensional network.

Related literature

For the monoclinic polymorph of the title compound, see: Mundwiler *et al.* (2004). For related structures, see: Kemp (2006); Roodt *et al.* (2003); Schutte *et al.* (2007); Wang *et al.* (2003); Alvarez *et al.* (2007). For the synthesis of the precursor, see: Alberto *et al.* (1996);



Experimental

Crystal data

 $[Re(C_7H_4NO_4)(CO)_3(H_2O)]$
 $M_r = 454.37$

 Triclinic, $P\bar{1}$
 $a = 9.5024$ (11) Å

 $b = 12.4254$ (16) Å

 $c = 12.4889$ (16) Å

 $\alpha = 101.799$ (4)°

 $\beta = 107.943$ (4)°

 $\gamma = 111.346$ (4)°

 $V = 1220.4$ (3) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 10.00$ mm⁻¹
 $T = 100$ (2) K

 $0.27 \times 0.17 \times 0.05$ mm

Data collection

Bruker APEXII diffractometer

Absorption correction: multi-scan

(SADABS; Bruker, 2004)

 $T_{\min} = 0.140$, $T_{\max} = 0.605$

15096 measured reflections

5848 independent reflections

 4869 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.037$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.026$
 $wR(F^2) = 0.065$
 $S = 1.05$

5848 reflections

373 parameters

7 restraints

H atoms treated by a mixture of independent and constrained refinement

 $\Delta\rho_{\text{max}} = 1.21$ e Å⁻³
 $\Delta\rho_{\text{min}} = -1.80$ e Å⁻³
Table 1

Selected geometric parameters (Å, °).

N1—Re1	2.180 (4)	C2—Re1	1.906 (5)
O5—Re1	2.153 (3)	C3—Re1	1.885 (6)
O15—Re2	2.148 (3)	C13—Re2	1.883 (5)
N2—Re2	2.166 (4)	O14—Re2	2.153 (3)
C11—Re2	1.892 (5)	O4—Re1	2.170 (3)
C12—Re2	1.947 (5)	Re1—C1	1.915 (5)
C11—Re2—C12	89.52 (19)	O15—Re2—N2	74.98 (12)
C11—Re2—O15	170.52 (16)	C2—Re1—O5	98.77 (15)
C12—Re2—O15	98.45 (16)	C1—Re1—O4	95.49 (17)
C11—Re2—O14	95.63 (16)	O5—Re1—O4	80.73 (12)
C12—Re2—O14	96.35 (17)	O5—Re1—N1	74.77 (12)
O15—Re2—O14	78.48 (12)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O7—H7 ⁱ ···O15 ⁱ	0.82	1.81	2.595 (4)	160
O4—H4B ⁱ ···O16 ⁱⁱ	0.85 (5)	2.51 (5)	2.920 (5)	111 (4)
O4—H4B ⁱ ···O8 ⁱⁱⁱ	0.85 (5)	2.01 (3)	2.780 (5)	150 (5)
O4—H4B ⁱ ···O16 ⁱⁱ	0.85 (5)	2.51 (5)	2.920 (5)	111 (4)
O14—H14B ⁱ ···O6	0.85 (5)	1.84 (2)	2.671 (5)	169 (5)
O14—H14A ⁱ ···O18 ⁱⁱⁱ	0.840 (18)	1.87 (2)	2.674 (4)	161 (5)

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

Data collection: APEX2 (Bruker, 2005); cell refinement: SAINT-Plus (Bruker, 2004); data reduction: SAINT-Plus; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: DIAMOND (Brandenburg & Putz, 2005) and ORTEP-3 (Farrugia, 1999); software used to prepare material for publication: SHELXL97.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2479).

References

- Alberto, R., Schibli, R. & Schubiger, P. A. (1996). *Polyhedron*, **15**, 1079–1083.
- Alvarez, C. M., Garcia-Rodriguez, R. & 3554.
- Brandenberg, K. & Putz, H. (2005). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.
- Bruker (2004). *SAINT-Plus* (including *XPREP*) and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bruker (2005). *APEX2*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
- Kemp, G. (2006). PhD thesis, University of Johannesburg, South Africa.
- Mundwiler, S., Kundig, M., Ortner, K. & Alberto, R. (2004). *Dalton Trans.* pp. 1320–1328.
- Roodt, A., Otto, S. & Steyl, G. (2003). *Coord. Chem. Rev.* **245**, 121–129.
- Schutte, M., Visser, H. G. & Steyl, G. (2007). *Acta Cryst. E* **63**, m3195–m3196.
- Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.
- Wang, W., Spingler, B. & Alberto, R. (2003). *Inorg. Chim. Acta*, **355**, 386–391.

supplementary materials

Acta Cryst. (2008). E64, m1226-m1227 [doi:10.1107/S160053680802761X]

Aquatricarbonyl(4-carboxypyridine-2-carboxylato- κ^2N,O^2)rhenium(I)

M. Schutte and H. G. Visser

Comment

The title compound, $\text{Re}(\text{C}_{10}\text{H}_6\text{NO}_8)$, is one of many Re(I)-tricarbonyl complexes currently under investigation in the field of radiopharmacology. One polymorph of the title compound was reported earlier [Mundwiler *et al.*, (2004)].

The Re(I) core is coordinated by three facial carbonyl groups, one pyridine-2-carboxylato-4-carboxylic acid ligand and a water molecule. A slightly distorted octahedral geometry around the Re(I) metal centre is observed, possibly due to the effect of the small bite angles of $74.78(14)^\circ$ and $74.98(12)^\circ$ respectively for the two pyridine-2,4-dicarboxylic acid units. The Re—OH₂ bond distances of $2.153(4)$ Å and $2.170(4)$ Å compare well with related structures [Mundwiler *et al.*, (2004) and Kemp, (2006)] of $2.198(5)$ Å and $2.192(4)$ Å. The Re—CO distances are well within the normal range, $1.883(6)$ Å to $1.947(6)$ Å. The crystal structure shows a range of hydrogen bonding of the types OH—O and CH—O thereby forming a 3D polymeric network, with DA distances ranging from $2.595(4)$ Å to $3.426(5)$ Å.

Experimental

$[\text{NEt}_4]_2[\text{Re}(\text{CO})_3\text{Br}_3]$ (300 mg, 0.389 mmol), as prepared by Alberto *et al.* (1996) was stirred in 40 ml of water at pH 2.2 for 20 minutes until dissolved. AgNO_3 (198 mg, 1.167 mmol) was added to the solution and stirred for 24 h at room temperature. The precipitate, AgBr , was filtered off and weighed (0.220 g). 2,4-Pyridinedicarboxylic acid (65 mg, 0.389 mmol) was added to the filtrate as a solid and stirred for 36 h. The solution turned bright yellow with a light yellow precipitate. The product was filtered off, dried and weighed. Crystals were obtained by slow evaporation of the filtrate. (Yield: 0.240 g, 68%).

Refinement

The aromatic H atoms were placed in geometrically idealized positions and constrained to ride on its parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The highest electron density lies within 1.14 Å from Re1. The hydrogen atoms of the coordinated water molecules were determined from a difference Fourier map and their positional parameters freely refined with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$.

Figures

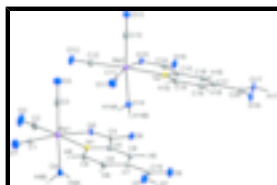


Fig. 1. Representation of the title compound, showing the numbering scheme and displacement ellipsoids (50% probability).

Aquatricarbonyl(4-carboxypyridine-2-carboxylato- κ^2N,O^2)rhenium(I)

Crystal data

[Re(C ₇ H ₄ NO ₄)(CO) ₃ (H ₂ O)]	$Z = 4$
$M_r = 454.37$	$F_{000} = 848.0$
Triclinic, $P\bar{1}$	$D_x = 2.473 \text{ Mg m}^{-3}$
Hall symbol: -P 1	Mo $K\alpha$ radiation
$a = 9.5024 (11) \text{ \AA}$	$\lambda = 0.71073 \text{ \AA}$
$b = 12.4254 (16) \text{ \AA}$	Cell parameters from 7140 reflections
$c = 12.4889 (16) \text{ \AA}$	$\theta = 2.8\text{--}28.3^\circ$
$\alpha = 101.799 (4)^\circ$	$\mu = 10.00 \text{ mm}^{-1}$
$\beta = 107.943 (4)^\circ$	$T = 100 (2) \text{ K}$
$\gamma = 111.346 (4)^\circ$	Plate, yellow
$V = 1220.4 (3) \text{ \AA}^3$	$0.27 \times 0.17 \times 0.05 \text{ mm}$

Data collection

Bruker APEX diffractometer	$R_{\text{int}} = 0.037$
φ and ω scans	$\theta_{\text{max}} = 28.3^\circ$
Absorption correction: multi-scan (SADABS; Bruker, 2004)	$\theta_{\text{min}} = 1.8^\circ$
$T_{\text{min}} = 0.140$, $T_{\text{max}} = 0.605$	$h = -12 \rightarrow 12$
15096 measured reflections	$k = -16 \rightarrow 15$
5048 independent reflections	$l = -16 \rightarrow 16$
4869 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	7 restraints
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.027$	$w = 1/[\sigma^2(F_o^2) + (0.0251P)^2]$
$wR(F^2) = 0.065$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{\text{max}} = 0.015$
5848 reflections	$\Delta\rho_{\text{max}} = 1.21 \text{ e \AA}^{-3}$
373 parameters	$\Delta\rho_{\text{min}} = -1.80 \text{ e \AA}^{-3}$
	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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
N1	0.3078 (4)	0.2654 (3)	0.5400 (3)	0.0095 (8)
O5	0.1030 (4)	0.1933 (3)	0.3130 (2)	0.0113 (7)
O6	0.2391 (4)	0.1072 (3)	0.2433 (3)	0.0152 (7)
C4	0.2193 (6)	0.1635 (4)	0.3289 (4)	0.0116 (9)
O15	0.0841 (4)	0.2152 (3)	-0.1450 (3)	0.0112 (7)
C5	0.3392 (6)	0.1998 (4)	0.4572 (4)	0.0102 (9)
O16	0.0735 (4)	0.0827 (3)	-0.3002 (3)	0.0160 (7)
C15	0.3019 (5)	0.1585 (4)	-0.1099 (4)	0.0097 (9)
C14	0.1407 (6)	0.1487 (4)	-0.1934 (4)	0.0118 (10)
C18	0.5968 (5)	0.1949 (4)	0.0518 (4)	0.0116 (9)
H18	0.6981	0.2089	0.1084	0.014*
C17	0.5336 (6)	0.1168 (4)	-0.0662 (4)	0.0115 (9)
C16	0.3816 (5)	0.0981 (4)	-0.1491 (4)	0.0106 (9)
H16	0.3358	0.0459	-0.2288	0.013*
C8	0.5409 (6)	0.2756 (4)	0.6944 (4)	0.0127 (10)
H8	0.6081	0.3019	0.7761	0.015*
C6	0.4696 (6)	0.1716 (4)	0.4875 (4)	0.0129 (10)
H6	0.4884	0.1278	0.4282	0.015*
C9	0.4082 (5)	0.3013 (4)	0.6568 (4)	0.0122 (10)
H9	0.3874	0.3453	0.7146	0.015*
C7	0.5727 (5)	0.2098 (4)	0.6085 (4)	0.0105 (9)
C20	0.6310 (6)	0.0551 (4)	-0.0994 (4)	0.0131 (10)
C19	0.5084 (5)	0.2516 (4)	0.0845 (4)	0.0118 (10)
H19	0.5507	0.3031	0.1641	0.014*
N2	0.3633 (4)	0.2346 (3)	0.0051 (3)	0.0092 (8)
O8	0.7441 (4)	0.1233 (3)	0.5666 (3)	0.0200 (8)
O7	0.8100 (4)	0.2290 (3)	0.7589 (3)	0.0190 (8)
H7	0.8878	0.2118	0.773	0.029*
C10	0.7178 (5)	0.1827 (4)	0.6423 (4)	0.0134 (10)
O18	0.7570 (4)	0.0669 (3)	-0.0231 (3)	0.0174 (8)
O17	0.5674 (4)	-0.0113 (3)	-0.2139 (3)	0.0176 (8)
H17	0.6264	-0.0424	-0.2258	0.026*
O11	0.4169 (4)	0.4220 (3)	0.3181 (3)	0.0206 (8)
O12	-0.0486 (5)	0.3985 (3)	0.0753 (3)	0.0290 (9)
C11	0.3339 (6)	0.3833 (4)	0.2154 (4)	0.0129 (10)
C12	0.0473 (6)	0.3683 (4)	0.0652 (4)	0.0165 (10)
O2	-0.2361 (5)	0.3030 (4)	0.3410 (3)	0.0296 (10)
C2	-0.1119 (6)	0.3005 (4)	0.3895 (4)	0.0178 (11)
C3	0.2069 (6)	0.4532 (5)	0.4629 (4)	0.0211 (12)
O3	0.2698 (5)	0.5484 (3)	0.4535 (3)	0.0283 (9)
C13	0.3283 (6)	0.4712 (5)	0.0407 (4)	0.0162 (10)
O13	0.4026 (4)	0.5690 (3)	0.0395 (3)	0.0209 (8)
O14	0.0791 (4)	0.1290 (3)	0.0380 (3)	0.0128 (7)

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O4	-0.0362 (4)	0.1106 (3)	0.4661 (3)	0.0191 (8)
H4A	-0.070 (6)	0.046 (3)	0.408 (3)	0.029*
H4B	-0.092 (6)	0.097 (4)	0.508 (4)	0.029*
H14B	0.123 (5)	0.112 (5)	0.098 (3)	0.029*
H14A	-0.025 (2)	0.093 (4)	0.010 (4)	0.029*
Re2	0.20999 (2)	0.315032 (16)	0.046124 (15)	0.00973 (6)
Re1	0.09158 (2)	0.296297 (16)	0.468442 (15)	0.01020 (6)
C1	0.1002 (6)	0.3789 (5)	0.6194 (4)	0.0165 (10)
O1	0.1138 (5)	0.4303 (3)	0.7126 (3)	0.0272 (9)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0096 (18)	0.013 (2)	0.0088 (18)	0.0082 (16)	0.0031 (15)	0.0057 (16)
O5	0.0128 (16)	0.0157 (18)	0.0070 (15)	0.0087 (14)	0.0052 (13)	0.0014 (13)
O6	0.0183 (17)	0.0185 (19)	0.0109 (16)	0.0125 (15)	0.0057 (14)	0.0022 (14)
C4	0.016 (2)	0.013 (2)	0.013 (2)	0.010 (2)	0.0097 (19)	0.0047 (19)
O15	0.0142 (16)	0.0144 (17)	0.0063 (14)	0.0101 (14)	0.0035 (13)	0.0007 (13)
C5	0.012 (2)	0.008 (2)	0.006 (2)	0.0038 (18)	0.0014 (17)	-0.0007 (17)
O16	0.0185 (18)	0.0204 (19)	0.0076 (15)	0.0131 (15)	0.0024 (13)	-0.0010 (14)
C15	0.012 (2)	0.008 (2)	0.009 (2)	0.0047 (18)	0.0040 (18)	0.0034 (18)
C14	0.012 (2)	0.016 (3)	0.010 (2)	0.008 (2)	0.0044 (18)	0.0060 (19)
C18	0.006 (2)	0.017 (3)	0.009 (2)	0.0058 (19)	0.0008 (17)	0.0032 (19)
C17	0.015 (2)	0.011 (2)	0.014 (2)	0.0092 (19)	0.0085 (19)	0.0059 (19)
C16	0.011 (2)	0.010 (2)	0.008 (2)	0.0039 (19)	0.0039 (17)	0.0016 (18)
C8	0.012 (2)	0.016 (2)	0.006 (2)	0.006 (2)	0.0017 (17)	0.0023 (18)
C6	0.013 (2)	0.010 (2)	0.013 (2)	0.0028 (19)	0.0073 (19)	0.0026 (19)
C9	0.013 (2)	0.015 (2)	0.006 (2)	0.008 (2)	0.0025 (18)	-0.0010 (18)
C7	0.005 (2)	0.014 (2)	0.011 (2)	0.0053 (18)	0.0015 (17)	0.0029 (19)
C20	0.012 (2)	0.014 (2)	0.016 (2)	0.008 (2)	0.0073 (19)	0.005 (2)
C19	0.010 (2)	0.014 (2)	0.008 (2)	0.0056 (19)	0.0030 (18)	0.0001 (18)
N2	0.0081 (18)	0.010 (2)	0.0085 (18)	0.0053 (16)	0.0021 (14)	0.0014 (15)
O8	0.0175 (18)	0.027 (2)	0.0152 (17)	0.0143 (16)	0.0046 (14)	0.0022 (15)
O7	0.0150 (17)	0.029 (2)	0.0117 (16)	0.0145 (16)	0.0008 (14)	0.0025 (15)
C10	0.006 (2)	0.017 (3)	0.016 (2)	0.0058 (19)	0.0029 (18)	0.006 (2)
O18	0.0116 (17)	0.026 (2)	0.0170 (17)	0.0135 (16)	0.0056 (14)	0.0040 (15)
O17	0.0187 (18)	0.025 (2)	0.0133 (17)	0.0179 (16)	0.0065 (14)	0.0014 (15)
O11	0.0230 (19)	0.021 (2)	0.0082 (16)	0.0068 (16)	0.0034 (14)	-0.0011 (15)
O12	0.028 (2)	0.028 (2)	0.041 (2)	0.0201 (19)	0.0204 (19)	0.0099 (19)
C11	0.013 (2)	0.012 (2)	0.019 (2)	0.009 (2)	0.0114 (19)	0.005 (2)
C12	0.018 (3)	0.011 (2)	0.018 (2)	0.006 (2)	0.007 (2)	0.002 (2)
O2	0.026 (2)	0.044 (3)	0.0178 (18)	0.027 (2)	0.0004 (16)	0.0021 (18)
C2	0.026 (3)	0.019 (3)	0.010 (2)	0.012 (2)	0.010 (2)	0.002 (2)
C3	0.022 (3)	0.020 (3)	0.018 (3)	0.012 (2)	0.007 (2)	-0.002 (2)
O3	0.040 (2)	0.020 (2)	0.033 (2)	0.0141 (19)	0.0231 (19)	0.0118 (18)
C13	0.018 (2)	0.016 (3)	0.012 (2)	0.010 (2)	0.0043 (19)	0.000 (2)
O13	0.029 (2)	0.0149 (19)	0.0237 (19)	0.0093 (16)	0.0168 (16)	0.0079 (15)
O14	0.0086 (16)	0.0141 (18)	0.0141 (17)	0.0046 (14)	0.0031 (13)	0.0054 (14)

O4	0.025 (2)	0.0138 (19)	0.0214 (19)	0.0088 (16)	0.0164 (16)	0.0019 (15)
Re2	0.00971 (10)	0.01041 (10)	0.00854 (9)	0.00554 (8)	0.00342 (7)	0.00128 (7)
Re1	0.01226 (10)	0.01220 (11)	0.00847 (9)	0.00860 (8)	0.00437 (7)	0.00248 (8)
C1	0.016 (2)	0.017 (3)	0.023 (3)	0.011 (2)	0.009 (2)	0.012 (2)
O1	0.039 (2)	0.028 (2)	0.0169 (19)	0.0175 (19)	0.0158 (17)	0.0020 (17)

Geometric parameters (Å, °)

N1—C9	1.343 (5)	C7—C10	1.493 (6)
N1—C5	1.357 (5)	C20—O18	1.215 (5)
N1—Re1	2.180 (4)	C20—O17	1.309 (5)
O5—C4	1.261 (5)	C19—N2	1.337 (5)
O5—Re1	2.153 (3)	C19—H19	0.93
O6—C4	1.258 (5)	N2—Re2	2.166 (4)
C4—O6	1.258 (5)	O8—C10	1.223 (5)
C4—C5	1.508 (6)	O7—C10	1.313 (5)
O15—C14	1.287 (5)	O11—C11	1.161 (5)
O15—Re2	2.148 (3)	O12—C12	1.135 (6)
C5—C6	1.372 (6)	C11—Re2	1.892 (5)
O16—C14	1.227 (5)	C12—Re2	1.947 (5)
C15—N2	1.351 (5)	O2—C2	1.164 (6)
C15—C16	1.368 (6)	C2—Re1	1.906 (5)
C15—C14	1.508 (6)	C3—O3	1.165 (6)
C18—C19	1.376 (6)	C3—Re1	1.885 (6)
C18—C17	1.385 (6)	C13—O13	1.169 (6)
C18—H18	0.93	C13—Re2	1.883 (5)
C17—C16	1.395 (6)	O14—Re2	2.153 (3)
C17—C20	1.496 (6)	O14—H14B	0.85 (4)
C16—H16	0.93	O14—H14A	0.85 (4)
C8—C9	1.379 (6)	O4—Re1	2.170 (3)
C8—C7	1.390 (6)	O4—H4A	0.85 (5)
C8—H8	0.93	O4—H4B	0.85 (5)
C6—C7	1.388 (6)	Re1—C1	1.915 (5)
C6—H6	0.93	C1—O1	1.151 (5)
C9—H9	0.93		
C9—N1—C5	117.6 (4)	C15—N2—Re2	115.9 (3)
C9—N1—Re1	126.4 (3)	C10—O7—H7	109.5
C5—N1—Re1	115.9 (3)	O8—C10—O7	125.2 (4)
C4—O5—Re1	118.5 (3)	O8—C10—C7	121.8 (4)
O6—C4—O5	122.9 (4)	O7—C10—C7	113.0 (4)
O6—C4—O5	122.9 (4)	C20—O17—H17	109.5
O6—C4—C5	119.9 (4)	O11—C11—Re2	175.5 (4)
O6—C4—C5	119.9 (4)	O12—C12—Re2	179.4 (5)
O5—C4—C5	117.2 (4)	O2—C2—Re1	179.9 (5)
C14—O15—Re2	118.6 (3)	O3—C3—Re1	176.0 (5)
N1—C5—C6	123.1 (4)	O13—C13—Re2	178.8 (4)
N1—C5—C4	113.5 (4)	Re2—O14—H14B	116 (3)
C6—C5—C4	123.3 (4)	Re2—O14—H14A	121 (3)
N2—C15—C16	122.9 (4)	H14B—O14—H14A	112 (3)

supplementary materials

N2—C15—C14	114.7 (4)	Re1—O4—H4A	124 (3)
C16—C15—C14	122.3 (4)	Re1—O4—H4B	121 (3)
O16—C14—O15	124.0 (4)	H4A—O4—H4B	110 (3)
O16—C14—C15	120.6 (4)	C13—Re2—C11	88.2 (2)
O15—C14—C15	115.4 (4)	C13—Re2—C12	87.6 (2)
C19—C18—C17	119.4 (4)	C11—Re2—C12	89.52 (19)
C19—C18—H18	120.3	C13—Re2—O15	97.19 (16)
C17—C18—H18	120.3	C11—Re2—O15	170.52 (16)
C18—C17—C16	118.9 (4)	C12—Re2—O15	98.45 (16)
C18—C17—C20	118.5 (4)	C13—Re2—O14	174.48 (16)
C16—C17—C20	122.6 (4)	C11—Re2—O14	95.63 (16)
C15—C16—C17	118.2 (4)	C12—Re2—O14	96.35 (17)
C15—C16—H16	120.9	O15—Re2—O14	78.48 (12)
C17—C16—H16	120.9	C13—Re2—N2	96.92 (17)
C9—C8—C7	119.0 (4)	C11—Re2—N2	96.70 (16)
C9—C8—H8	120.5	C12—Re2—N2	172.39 (16)
C7—C8—H8	120.5	O15—Re2—N2	74.98 (12)
C5—C6—C7	118.6 (4)	O14—Re2—N2	78.73 (13)
C5—C6—H6	120.7	C3—Re1—C2	88.6 (2)
C7—C6—H6	120.7	C3—Re1—C1	88.1 (2)
N1—C9—C8	122.7 (4)	C2—Re1—C1	88.6 (2)
N1—C9—H9	118.7	C3—Re1—O5	95.56 (17)
C8—C9—H9	118.7	C2—Re1—O5	98.77 (15)
C6—C7—C8	119.0 (4)	C1—Re1—O5	171.85 (16)
C6—C7—C10	119.0 (4)	C3—Re1—O4	176.21 (16)
C8—C7—C10	122.0 (4)	C2—Re1—O4	92.71 (18)
O18—C20—O17	124.8 (4)	C1—Re1—O4	95.49 (17)
O18—C20—C17	120.7 (4)	O5—Re1—O4	80.73 (12)
O17—C20—C17	114.5 (4)	C3—Re1—N1	96.75 (18)
N2—C19—C18	121.8 (4)	C2—Re1—N1	171.93 (16)
N2—C19—H19	119.1	C1—Re1—N1	97.60 (17)
C18—C19—H19	119.1	O5—Re1—N1	74.77 (12)
C19—N2—C15	118.7 (4)	O4—Re1—N1	81.58 (14)
C19—N2—Re2	125.3 (3)	O1—C1—Re1	176.6 (4)

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O7—H7 \cdots O15 ⁱ	0.82	1.81	2.595 (4)	160
O4—H4B \cdots O16 ⁱⁱ	0.85 (5)	2.51 (5)	2.920 (5)	111 (4)
O4—H4B \cdots O8 ⁱⁱⁱ	0.85 (5)	2.01 (3)	2.780 (5)	150 (5)
O4—H4B \cdots O16 ⁱⁱ	0.85 (5)	2.51 (5)	2.920 (5)	111 (4)
O14—H14B \cdots O6	0.85 (5)	1.84 (2)	2.671 (5)	169 (5)
O14—H14A \cdots O18 ⁱⁱⁱ	0.840 (18)	1.87 (2)	2.674 (4)	161 (5)
O17—H17 \cdots O6 ^{iv}	0.82	1.78	2.602 (4)	177
C8—H8 \cdots O13 ^v	0.93	2.58	3.253 (5)	130
C6—H6 \cdots O17 ^{iv}	0.93	2.55	3.426 (5)	156

C19—H19...O2^{vi}

0.93

2.5

3.131 (5)

126

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

Fig. 1

