Crystal structure of *fac*-tricarbonylchloridobis(4-hydroxypyridine)rhenium(I)–pyridin-4(1*H*)-one (1/1)

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The asymmetric unit of the title compound, $[ReCl(C_5H_5NO)_2(CO)_3]\cdot C_5H_5NO$, contains one molecule of the complex *fac*-[ReCl(4-pyOH)$_2$(CO)$_3$] (where 4-pyOH represents 4-hydroxypyridine) and one molecule of pyridin-4(1*H*)-one (4-HpyO). In the molecule of the complex, the Re atom is coordinated to two N atoms of the two 4-pyOH ligands, three carbonyl C atoms, in a facial configuration, and the Cl atom. The resulting geometry is slightly distorted octahedral. In the crystal structure, both fragments are associated by hydrogen bonds; two 4-HpyO molecules bridge between two molecules of the complex using the O=C group as acceptor for two different HO– groups of coordinated 4-pyOH from two neighbouring metal complexes. The resulting square arrangements are extented into infinite chains by hydrogen bonds involving the N—H groups of the 4-HpyO molecule and the chloride ligands. The chains are further stabilized by π-stacking interactions.

1. Chemical context

The structural stability of the *fac*-{Re^I(CO)$_3$} fragment and its trend to form sixfold coordinated octahedral complexes make it a suitable candidate for the construction of self-assambled metallomacrocycles, with some of them showing interesting properties (Slone *et al.*, 1998; Sun & Lees, 2002). Bipyridine (and pyrazine) based ligands are usually chosen to obtain square or rectangular metallocycles, [Re$_4$(*L*)$_4$(CO)$_{12}$] (*L* is the bridging ligand) with internal diameters of 5–9 nm. In the present work, we present the structure of a rhenium complex, where the square architecture is achieved by a coordinative Re—*L* link (where *L* is 4-hydroxypyridine) and by hydrogen-bonding interactions involving a 4-pyridone molecule (a tautomer of 4-hydroxypyridine *L*).
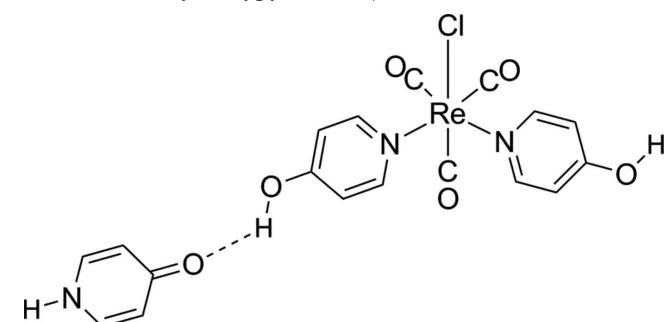
2. Structural commentary

The crystal structure consists of molecules of *fac*-[ReCl(4-pyOH)$_2$(CO)$_3$] (where 4-pyOH represents 4-hydroxypyridine) and pyridin-4(1*H*)-one (4-HpyO) in a 1:1 ratio (Fig. 1). Both

https://doi.org/10.1107/S2056989017013512 **1551**
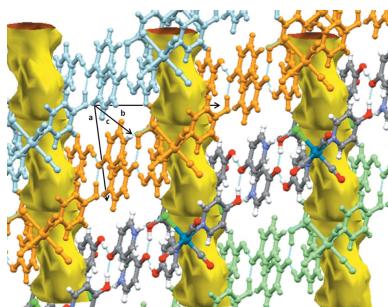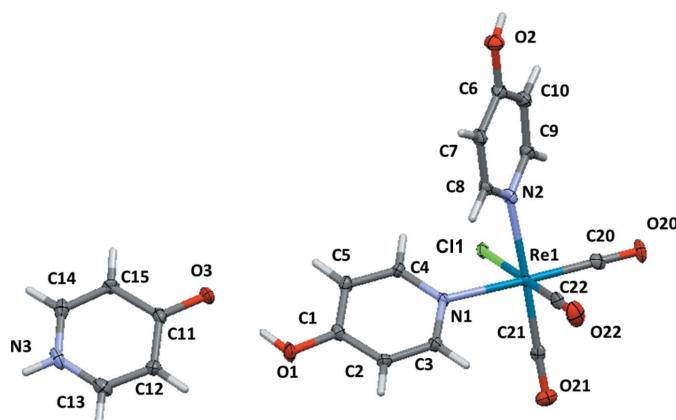
Figure 1
The molecular structure of the title compound, with displacement ellipsoids drawn at the 50% probability level.

Table 1
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1—H1···O3	0.94 (8)	1.62 (8)	2.556 (5)	175 (7)
O2—H2···O3^i	1.01 (7)	1.57 (7)	2.569 (5)	169 (6)
N3—H3A···Cl1^{ii}	0.97 (7)	2.32 (7)	3.218 (5)	152 (5)
C9—H9···O22^{iii}	0.95	2.56	3.317 (7)	137
C3—H3···Cl1^{iv}	0.95	2.89	3.580 (5)	131
C14—H14···O21^v	0.95	2.62	3.264 (7)	126

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

molecules are associated through hydrogen bonding (see below). The existence of the pyridone form instead of hydroxypyridine is confirmed by the C—O bond distance, subtantially shorthened in 4-HpyO [C11—O3 = 1.293 (5) Å] with respect to the coordinated 4-pyOH [O1—C1 = 1.335 (5) Å and O2—C6 = 1.339 (5) Å], indicating the presence of a double C=O bond in 4-HpyO. The C—C bond lengths involving the carbonyl group [C11—C12 = 1.425 (6) Å and C11—C15 = 1.432 (6) Å] are elongated with respect to those observed in the 4-pyOH fragments [for instance, C1—C2 = 1.404 (6) Å and C1—C5 = 1.395 (6) Å]. The C—N bond lengths are also longer than their typical values in pyridines or pyridinium cations. These parameters are close to those found in the crystal structure of the free (uncoordinated) 4-pyridone (Jones, 2001; Tyl et al., 2008) or to those involved in hydrogen bonding (Campos-Gaxiola et al. 2014; Staun & Oliver, 2012; 2015).

The molecular structure of fac-[ReCl(4-pyOH)$_2$(CO)$_3$] is similar to other tricarbonylrhenium(I) complexes with two pyridine-based ligands (Abel & Wilkinson, 1959; Farrell et al., 2016). The coordination polyhedron around the Re atom can be described as slightly distorted octahedral (all angles are close to 90 or 180°), formed by coordination of the two N atoms of the two 4-pyOH ligands (N1 and N2), by the three carbonyl C atoms, in a facial configuration, and the chloride ligand. Both Re—N bond lengths [2.208 (4) and 2.210 (4) Å] are statistically equivalent. Nevertheless, the Re—Cl bond in

the present compound [2.4986 (10) Å] is longer that those found in pyridine derivatives described recently by Farrell et al. (2016), with an average value of 2.4649 (4) Å. This fact is likely due to the hydrogen-bonding interaction involving the chloride and the N—H group of a neighbouring 4-pyridone since this interaction is absent in those structures.

3. Supramolecular features

The molecular association in the crystal is strongly directed by hydrogen bonding (Table 1). Two 4-pyridone molecules bridge between two fac-[ReCl(4-pyOH)$_2$(CO)$_3$] using the ketone O=C group as the hydrogen-bonding acceptor to two different HO– groups, forming $R_4^2(28)$ rings centred at the g Wyckoff site (Fig. 2). The N—H group of the pyridone unit also establishes hydrogen-bond interactions, with the chloride group, yielding a new centrosymmetric ring $R_4^4(28)$ (at the f Wickoff site). Although the centroid-to-centroid distance between the pyridone and hydroxypyridone is rather long (3.791 Å), some distances between the atoms and centroids of the rings [C4···N3^{vi} = 3.231 Å, C4···C14^{vi} = 3.470 Å, C5···C14^{vi} = 3.478 Å and C5···Ci^{vi} = 3.365 Å; symmetry code: (vi) $1-x, 2-y, 1-z$; see Fig. 2] suggest a (slipped) π-stacking interaction. Both intermolecular interactions work to form infinite chains, as represented in Fig. 2, which are further supported by weak C—H···O and C—H···Cl interactions (the most representative ones are included in Table 1). The formation of the $R_4^2(28)$ rings yields a small channel-like void of ca 103 Å^3 per unit cell, as shown in Fig. 3. No substantial electron density is found in the channels (ca 4 electrons per void based on a PLATON/SQUEEZE analysis (Spek, 2009, 2015).
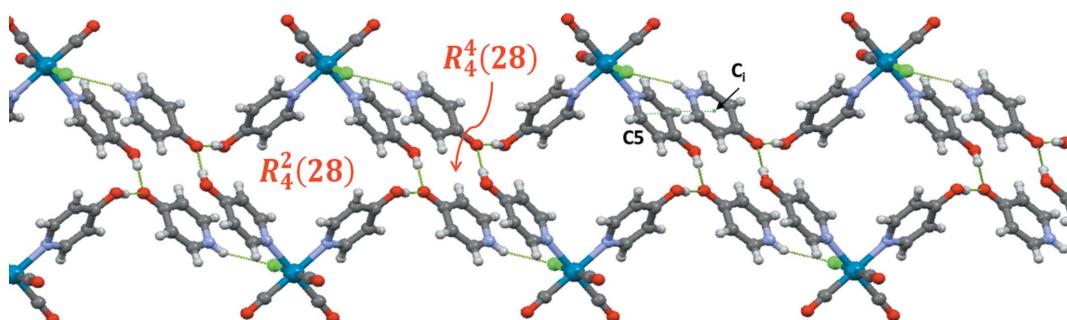

Figure 2
Representation of the formation of chains by hydrogen-bonding and π-stacking in the crystal structure.
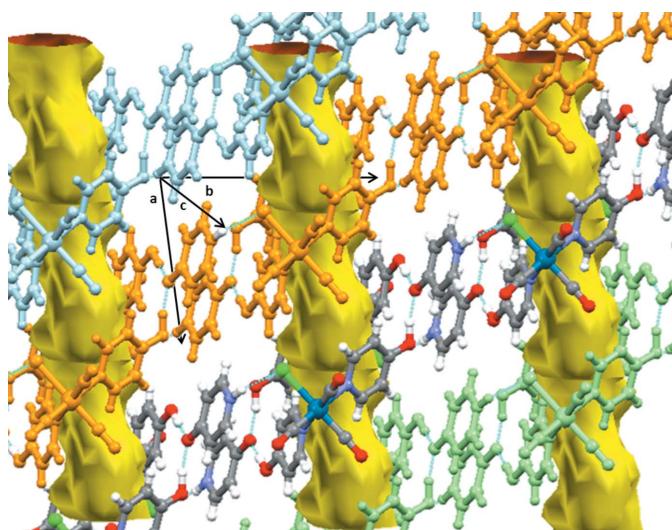
Figure 3
Association of the chains and formation of the empty channels in the crystal structure.

4. Database survey

The structures of several complexes with the metal centre *fac*-tricarbonylrhenium(I) and pyridine-based ligands have been reported (Abel & Wilkinson, 1959; Farrell *et al.*, 2016). The pyridine fragment can be part of a bridging ligand between different metal centres to form tetranuclear complexes as reported by Levine *et al.* (2009). When ligands based on 4,4′-bipyridine are chosen, square (Slone *et al.*, 1996; Bera *et al.*, 2004; Sun *et al.*, 2002) or rectangular (Dinolfo & Hupp, 2004; Gupta *et al.*, 2011; Lu *et al.*, 2012; Nagarajaprakash *et al.*, 2014; Orsa *et al.*, 2007) homo- or heteronuclear complexes are isolated. Applications of these compounds as sensors (Keefe *et al.*, 2000), luminescent materials (Slone *et al.*, 1996) or cytotoxic agents (Orsa *et al.*, 2007) have been also reported.

5. Synthesis and crystallization

The complex *fac*-[ReCl(4-pyOH)$_2$(CO)$_3$] was obtained by refluxing for 90 min a mixture of 4-hydroxypyridine (29 mg, 0.31 mmol) and [ReCl(CH$_3$CN)$_2$(CO)$_3$] in chloroform–methanol (1:1 *v/v*, 10 ml). The solution was concentrated (to half of initial volume), diethyl ether was added and the mixture cooled to 277 K. Finally, the solid was filtered off and vacuum dried on CaCl$_2$ (yield: 81%, 30 mg; m.p. 418–421 K). Analysis, calculated for C$_{13}$H$_{10}$ClN$_2$O$_5$Re: C 31.5, H 2.0, N 5.6%; found: C 31.9, H 1.9, N 5.5%. MS–ESI [*m/z* (%)]: 461 (100) [*M* – Cl]$^+$. IR (ATR, cm^{-1}): 2016 (*m*), 1865 (*b, s*), ν(CO).

Single crystals of the title compound (too few for elemental analysis or meaningful estimation of the yield) were obtained from solutions of *fac*-[ReCl(CO)$_3$(4-pyOH)$_2$] in CHCl$_3$:-CH$_2$Cl$_2$:ether (1:1:1) stored at 253 K (several days).

Table 2
Experimental details.

Crystal data	
Chemical formula	[ReCl(C$_5$H$_5$NO)$_2$(CO)$_3$]··C$_5$H$_5$NO
M_r	590.98
Crystal system, space group	Triclinic, $P\bar{1}$
Temperature (K)	100
a, b, c (Å)	7.5235 (13), 11.717 (2), 13.644 (2)
α, β, γ (°)	66.694 (4), 78.757 (4), 81.374 (4)
V (Å^3)	1079.9 (3)
Z	2
Radiation type	Mo $K\alpha$
μ (mm^{-1})	5.79
Crystal size (mm)	0.36 × 0.35 × 0.04
Data collection	
Diffractometer	Bruker D8 Venture Photon 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.352, 0.647
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	28225, 4476, 4312
R_{int}	0.041
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.630
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.029, 0.077, 1.35
No. of reflections	4476
No. of parameters	272
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.63, −1.03

Computer programs: *APEX3* (Bruker, 2014), *SAINT* (Bruker, 2014), *SHELXS2013* (Sheldrick, 2015*a*), *SHELXL2013* (Sheldrick, 2015*b*).

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms on O and N atoms were located *via* difference Fourier analyses and refined with $U_{iso}(H) = 1.5U_{eq}(O)$ and $1.2U_{eq}(N)$. Other H atoms were included at calculated sites and allowed to ride on their carrier atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

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Crystal structure of *fac*-tricarbonylchloridobis(4-hydroxypyridine)rhenium(I)–pyridin-4(1*H*)-one (1/1)

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Computing details

Data collection: *APEX3* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL2013* (Sheldrick, 2015b).

fac-Tricarbonylchloridobis(4-hydroxypyridine)rhenium(I)–pyridin-4(1_H_)-one (1/1)

Crystal data
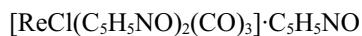
$[ReCl(C_5H_5NO)_2(CO)_3]\cdot C_5H_5NO$	$F(000) = 568$
$M_r = 590.98$	$D_x = 1.818$ Mg m^{-3}
Triclinic, $P\bar{1}$	Melting point: 145 K
$a = 7.5235$ (13) Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 11.717$ (2) Å	Cell parameters from 9858 reflections
$c = 13.644$ (2) Å	$\theta = 3.3–26.6°$
$\alpha = 66.694$ (4)°	$\mu = 5.79$ mm^{-1}
$\beta = 78.757$ (4)°	$T = 100$ K
$\gamma = 81.374$ (4)°	Plate, yellow
$V = 1079.9$ (3) Å^3	$0.36 \times 0.35 \times 0.04$ mm
$Z = 2$	

Data collection

Bruker D8 Venture Photon 100 CMOS diffractometer	4476 independent reflections
	4312 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.041$
Absorption correction: multi-scan (SADABS; Krause *et al.*, 2015)	$\theta_{max} = 26.6°$, $\theta_{min} = 2.8°$
$T_{min} = 0.352$, $T_{max} = 0.647$	$h = -9 \rightarrow 9$
28225 measured reflections	$k = -14 \rightarrow 14$
	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.029$	Hydrogen site location: mixed
$wR(F^2) = 0.077$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.35$	
4476 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0178P)^2 + 5.8451P]$ where $P = (F_o^2 + 2F_c^2)/3$
272 parameters	
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 1.63$ e Å^{-3}
	$\Delta\rho_{min} = -1.03$ e Å^{-3}

Extinction correction: SHELXL2013
(Sheldrick, 2015b),
$Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0119 (9)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Re1	0.86373 (2)	0.70746 (2)	0.07222 (2)	0.01123 (10)
Cl1	1.09377 (15)	0.82393 (10)	0.09609 (9)	0.0135 (2)
O1	0.3391 (5)	0.8384 (4)	0.4444 (3)	0.0208 (8)
H1	0.394 (10)	0.843 (6)	0.499 (6)	0.031*
O2	1.1814 (5)	0.2036 (3)	0.4087 (3)	0.0229 (8)
O3	0.4725 (5)	0.8444 (3)	0.6006 (3)	0.0203 (8)
H2	1.319 (10)	0.196 (6)	0.399 (6)	0.030*
O20	1.1184 (5)	0.6629 (4)	−0.1173 (3)	0.0232 (8)
O21	0.6852 (5)	0.9364 (4)	−0.0929 (3)	0.0243 (8)
O22	0.5800 (6)	0.5598 (4)	0.0526 (4)	0.0299 (9)
N1	0.6881 (5)	0.7397 (4)	0.2102 (3)	0.0129 (8)
N2	0.9814 (5)	0.5408 (4)	0.1954 (3)	0.0138 (8)
N3	0.1310 (6)	0.9766 (4)	0.8090 (4)	0.0225 (9)
H3A	0.056 (9)	1.011 (6)	0.859 (6)	0.027*
C1	0.4552 (7)	0.8016 (4)	0.3733 (4)	0.0154 (9)
C2	0.3875 (7)	0.7932 (5)	0.2880 (4)	0.0165 (10)
H2A	0.2607	0.8072	0.2848	0.020*
C3	0.5057 (7)	0.7646 (4)	0.2088 (4)	0.0151 (9)
H3	0.4577	0.7621	0.1503	0.018*
C4	0.7506 (7)	0.7409 (4)	0.2958 (4)	0.0140 (9)
H4	0.8767	0.7204	0.3000	0.017*
C5	0.6415 (7)	0.7705 (5)	0.3779 (4)	0.0166 (10)
H5	0.6922	0.7697	0.4368	0.020*
C6	1.1246 (7)	0.3148 (4)	0.3390 (4)	0.0169 (10)
C7	0.9392 (7)	0.3537 (5)	0.3529 (4)	0.0187 (10)
H7	0.8585	0.3038	0.4120	0.022*
C8	0.8757 (7)	0.4642 (4)	0.2803 (4)	0.0149 (9)
H8	0.7493	0.4884	0.2904	0.018*
C9	1.1622 (7)	0.5051 (5)	0.1839 (4)	0.0170 (10)
H9	1.2407	0.5590	0.1264	0.020*
C10	1.2371 (7)	0.3934 (5)	0.2526 (4)	0.0191 (10)
H10	1.3638	0.3706	0.2408	0.023*
C11	0.3637 (7)	0.8866 (4)	0.6664 (4)	0.0159 (9)
C12	0.1806 (7)	0.9321 (5)	0.6507 (4)	0.0192 (10)
H12	0.1357	0.9326	0.5901	0.023*

C13	0.0699 (7)	0.9751 (5)	0.7227 (4)	0.0206 (10)
H13	−0.0523	1.0045	0.7120	0.025*
C14	0.3048 (8)	0.9366 (5)	0.8259 (4)	0.0202 (10)
H14	0.3458	0.9412	0.8857	0.024*
C15	0.4216 (6)	0.8902 (4)	0.7590 (4)	0.0149 (9)
H15	0.5419	0.8601	0.7736	0.018*
C20	1.0245 (7)	0.6793 (4)	−0.0466 (4)	0.0175 (10)
C21	0.7553 (6)	0.8525 (4)	−0.0305 (4)	0.0148 (9)
C22	0.6898 (7)	0.6144 (5)	0.0594 (4)	0.0197 (10)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.01149 (13)	0.01337 (13)	0.01111 (13)	0.00016 (7)	−0.00161 (7)	−0.00747 (8)
Cl1	0.0141 (5)	0.0153 (5)	0.0144 (5)	−0.0007 (4)	−0.0023 (4)	−0.0091 (4)
O1	0.0169 (18)	0.032 (2)	0.0186 (18)	0.0003 (15)	0.0016 (15)	−0.0177 (16)
O2	0.024 (2)	0.0183 (18)	0.0187 (18)	0.0032 (15)	−0.0025 (15)	−0.0015 (15)
O3	0.0179 (18)	0.0264 (19)	0.0191 (18)	0.0058 (15)	−0.0019 (14)	−0.0144 (15)
O20	0.026 (2)	0.0278 (19)	0.0184 (18)	−0.0017 (16)	0.0063 (16)	−0.0159 (16)
O21	0.025 (2)	0.0226 (19)	0.0214 (19)	0.0016 (16)	−0.0089 (16)	−0.0037 (16)
O22	0.024 (2)	0.037 (2)	0.040 (2)	−0.0070 (17)	−0.0022 (18)	−0.026 (2)
N1	0.0115 (19)	0.0162 (19)	0.0112 (18)	0.0018 (15)	0.0003 (15)	−0.0072 (15)
N2	0.0117 (19)	0.0133 (18)	0.016 (2)	−0.0008 (15)	0.0001 (16)	−0.0066 (16)
N3	0.024 (2)	0.020 (2)	0.023 (2)	−0.0028 (18)	0.0078 (19)	−0.0120 (18)
C1	0.014 (2)	0.017 (2)	0.015 (2)	−0.0020 (18)	0.0015 (18)	−0.0073 (18)
C2	0.016 (2)	0.020 (2)	0.017 (2)	0.0035 (18)	−0.0061 (19)	−0.0097 (19)
C3	0.015 (2)	0.017 (2)	0.016 (2)	−0.0007 (18)	−0.0037 (18)	−0.0090 (19)
C4	0.017 (2)	0.016 (2)	0.012 (2)	0.0009 (18)	−0.0043 (18)	−0.0074 (18)
C5	0.018 (2)	0.019 (2)	0.016 (2)	−0.0022 (19)	−0.0033 (19)	−0.0102 (19)
C6	0.020 (2)	0.014 (2)	0.017 (2)	0.0006 (18)	−0.0036 (19)	−0.0059 (19)
C7	0.019 (2)	0.016 (2)	0.017 (2)	0.0008 (19)	0.002 (2)	−0.0053 (19)
C8	0.013 (2)	0.018 (2)	0.015 (2)	−0.0006 (18)	0.0007 (18)	−0.0089 (19)
C9	0.015 (2)	0.020 (2)	0.016 (2)	−0.0006 (18)	−0.0006 (19)	−0.0077 (19)
C10	0.015 (2)	0.021 (2)	0.020 (2)	0.0025 (19)	−0.002 (2)	−0.008 (2)
C11	0.021 (2)	0.014 (2)	0.013 (2)	0.0001 (18)	−0.0003 (19)	−0.0074 (18)
C12	0.022 (3)	0.019 (2)	0.017 (2)	0.005 (2)	−0.005 (2)	−0.009 (2)
C13	0.018 (2)	0.018 (2)	0.027 (3)	0.0001 (19)	−0.001 (2)	−0.012 (2)
C14	0.027 (3)	0.021 (2)	0.017 (2)	−0.007 (2)	0.000 (2)	−0.011 (2)
C15	0.011 (2)	0.016 (2)	0.022 (2)	−0.0007 (17)	−0.0068 (19)	−0.0106 (19)
C20	0.021 (3)	0.011 (2)	0.024 (3)	0.0000 (18)	−0.008 (2)	−0.0085 (19)
C21	0.010 (2)	0.019 (2)	0.017 (2)	−0.0061 (18)	0.0021 (18)	−0.0086 (19)
C22	0.014 (2)	0.027 (3)	0.016 (2)	0.005 (2)	0.0001 (19)	−0.011 (2)

Geometric parameters (Å, º)

Re1—C22	1.898 (6)	C2—C3	1.376 (7)
Re1—C21	1.914 (5)	C2—H2A	0.9500
Re1—C20	1.933 (5)	C3—H3	0.9500

Re1—N1	2.208 (4)	C4—C5	1.383 (7)
Re1—N2	2.210 (4)	C4—H4	0.9500
Re1—Cl1	2.4987 (11)	C5—H5	0.9500
O1—C1	1.333 (6)	C6—C10	1.393 (7)
O1—H1	0.94 (8)	C6—C7	1.401 (7)
O2—C6	1.341 (6)	C7—C8	1.367 (7)
O2—H2	1.01 (7)	C7—H7	0.9500
O3—C11	1.289 (6)	C8—H8	0.9500
O20—C20	1.143 (7)	C9—C10	1.387 (7)
O21—C21	1.151 (6)	C9—H9	0.9500
O22—C22	1.155 (7)	C10—H10	0.9500
N1—C4	1.348 (6)	C11—C12	1.426 (7)
N1—C3	1.362 (6)	C11—C15	1.433 (7)
N2—C8	1.346 (6)	C12—C13	1.363 (7)
N2—C9	1.360 (6)	C12—H12	0.9500
N3—C13	1.353 (7)	C13—H13	0.9500
N3—C14	1.353 (7)	C14—C15	1.355 (7)
N3—H3A	0.97 (7)	C14—H14	0.9500
C1—C2	1.401 (7)	C15—H15	0.9500
C1—C5	1.402 (7)		
C22—Re1—C21	87.9 (2)	C5—C4—H4	118.2
C22—Re1—C20	89.6 (2)	C4—C5—C1	119.2 (4)
C21—Re1—C20	88.6 (2)	C4—C5—H5	120.4
C22—Re1—N1	91.96 (19)	C1—C5—H5	120.4
C21—Re1—N1	92.49 (18)	O2—C6—C10	124.3 (5)
C20—Re1—N1	178.11 (17)	O2—C6—C7	117.8 (5)
C22—Re1—N2	91.80 (19)	C10—C6—C7	117.8 (4)
C21—Re1—N2	177.97 (17)	C8—C7—C6	119.2 (5)
C20—Re1—N2	93.45 (18)	C8—C7—H7	120.4
N1—Re1—N2	85.51 (15)	C6—C7—H7	120.4
C22—Re1—Cl1	177.81 (16)	N2—C8—C7	124.0 (5)
C21—Re1—Cl1	94.05 (14)	N2—C8—H8	118.0
C20—Re1—Cl1	91.33 (15)	C7—C8—H8	118.0
N1—Re1—Cl1	87.03 (11)	N2—C9—C10	122.8 (5)
N2—Re1—Cl1	86.19 (11)	N2—C9—H9	118.6
C1—O1—H1	113 (4)	C10—C9—H9	118.6
C6—O2—H2	109 (4)	C9—C10—C6	119.2 (5)
C4—N1—C3	116.7 (4)	C9—C10—H10	120.4
C4—N1—Re1	124.0 (3)	C6—C10—H10	120.4
C3—N1—Re1	119.2 (3)	O3—C11—C12	122.0 (5)
C8—N2—C9	116.8 (4)	O3—C11—C15	121.4 (5)
C8—N2—Re1	121.6 (3)	C12—C11—C15	116.6 (4)
C9—N2—Re1	121.4 (3)	C13—C12—C11	120.0 (5)
C13—N3—C14	120.9 (5)	C13—C12—H12	120.0
C13—N3—H3A	123 (4)	C11—C12—H12	120.0
C14—N3—H3A	116 (4)	N3—C13—C12	121.1 (5)
O1—C1—C2	118.1 (4)	N3—C13—H13	119.4

O1—C1—C5	124.5 (5)	C12—C13—H13	119.4
C2—C1—C5	117.4 (4)	N3—C14—C15	121.3 (5)
C3—C2—C1	119.6 (5)	N3—C14—H14	119.4
C3—C2—H2A	120.2	C15—C14—H14	119.4
C1—C2—H2A	120.2	C14—C15—C11	120.0 (5)
N1—C3—C2	123.2 (4)	C14—C15—H15	120.0
N1—C3—H3	118.4	C11—C15—H15	120.0
C2—C3—H3	118.4	O20—C20—Re1	179.5 (4)
N1—C4—C5	123.7 (5)	O21—C21—Re1	177.0 (4)
N1—C4—H4	118.2	O22—C22—Re1	178.1 (4)

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1—H1⋯O3	0.94 (8)	1.62 (8)	2.556 (5)	175 (7)
O2—H2⋯O3[i]	1.01 (7)	1.57 (7)	2.569 (5)	169 (6)
N3—H3A⋯Cl1[ii]	0.97 (7)	2.32 (7)	3.218 (5)	152 (5)
C9—H9⋯O22[iii]	0.95	2.56	3.317 (7)	137
C3—H3⋯Cl1[iv]	0.95	2.89	3.580 (5)	131
C14—H14⋯O21[v]	0.95	2.62	3.264 (7)	126

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