

Crystal structures of a manganese(I) and a rhenium(I) complex of a bipyridine ligand with a non-coordinating benzoic acid moiety

Sheri Lense,^{a*} Ilia A. Guzei,^{b*} Jessica Andersen^a and Kong Choua Thao^a^aUniversity of Wisconsin Oshkosh Department of Chemistry, 800 Algoma Blvd., Oshkosh, WI 54902, USA, and^bDepartment of Chemistry, University of Wisconsin-Madison, 1101 University Ave., Madison, WI, 53706, USA.

*Correspondence e-mail: lenses@uwosh.edu, iguzei@chem.wisc.edu

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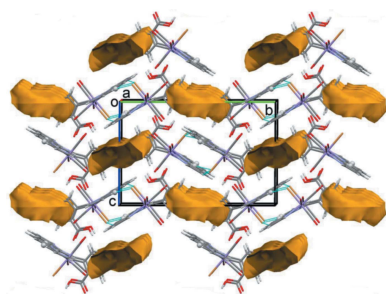
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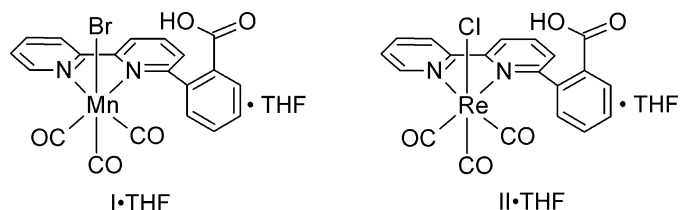
The structures of two facially coordinated Group VII metal complexes are reported, namely: *fac*-bromido[2-(2,2'-bipyridin-6-yl)benzoic acid- κ^2N,N']tricarbonylmanganese(I) tetrahydrofuran monosolvate, $[\text{MnBr}(\text{C}_{17}\text{H}_{12}\text{N}_2\text{O}_2)(\text{CO})_3]\cdot\text{C}_4\text{H}_8\text{O}$, **I**, and *fac*-[2-(2,2'-bipyridin-6-yl)benzoic acid- κ^2N,N']tricarbonylchloridorhenium(I) tetrahydrofuran monosolvate, $[\text{ReCl}(\text{C}_{17}\text{H}_{12}\text{N}_2\text{O}_2)(\text{CO})_3]\cdot\text{C}_4\text{H}_8\text{O}$, **II**. In both complexes, the metal ion is coordinated by three carbonyl ligands, a halide ion, and a 2-(2,2'-bipyridin-6-yl)benzoic acid ligand, in a distorted octahedral geometry. In manganese complex **I**, the tetrahydrofuran (THF) solvent molecule could not be refined due to disorder. The benzoic acid fragment is also disordered over two positions, such that the carboxylic acid group is either positioned near to the bromide ligand or to the axial carbonyl ligand. In the crystal of **I**, the complex molecules are linked by a pair of $\text{C}-\text{H}\cdots\text{Br}$ hydrogen bonds, forming inversion dimers that stack up the *a*-axis direction. In the rhenium complex **II**, there is hydrogen bonding between the benzoic acid moiety and a disordered co-crystallized THF molecule. In the crystal, the molecules are linked by $\text{C}-\text{H}\cdots\text{Cl}$ hydrogen bonds, forming layers parallel to (100) separated by layers of THF solvent molecules.

1. Chemical context

Crystal structures of *fac*- $[\text{M}(2,2'\text{-bipyridyl})(\text{CO})_3\text{X}]^{n+}$ ($M = \text{Mn}^{\text{I}}$ or Re^{I} , $X = \text{monoanionic ligand}$, $n = 0$ or $X = \text{neutral ligand}$, $n = 1$) complexes have been reported for complexes bearing many different bipyridyl derivatives. Among the numerous examples are structures reported by Chen *et al.* (2005), Gerlits & Coppens (2001), and Horn *et al.* (1987). Complexes of the type *fac*- $[\text{Re}(2,2'\text{-bipyridyl})(\text{CO})_3\text{X}]^{n+}$ and *fac*- $[\text{Mn}(2,2'\text{-bipyridyl})(\text{CO})_3\text{X}]^{n+}$, are of particular interest as selective catalysts for the reduction of CO_2 to CO (Bourrez *et al.*, 2011; Hawecker *et al.*, 1986; Smieja *et al.*, 2013; Sampson *et al.*, 2014; Machan *et al.*, 2014; Smieja & Kubiak, 2010). The addition of weak Brønsted acids such as water or methanol are necessary for the catalytic turnover of Mn complexes (Smieja *et al.*, 2013) and they also significantly increase the catalytic rate of Re complexes (Smieja *et al.*, 2012). Moreover, the use of bipyridyl ligands in these complexes containing phenolic functional groups positioned near the CO_2 binding site, which can act as intramolecular proton donors, have been shown to enhance catalytic performance. *fac*-Tricarbonylbromido[2-(2,2'-bipyridin-6-yl- κ^2N,N')phenol]manganese(I) showed enhanced catalytic activity for the reduction of CO_2 to CO



compared to *fac*-tricarbonylbromido(2,2'-bipyridine)-manganese(I) (Agarwal *et al.*, 2015). *fac*-Tricarbonylbromido[2-(4-phenyl-2,2'-bipyridin-6-yl)- κ^2N,N']benzene-1,3-diol]-manganese(I) was found to electrocatalytically reduce CO₂ to a mixture of CO and formic acid in the absence of external Brønsted acids (Franco *et al.*, 2014). In the presence of external Brønsted acids, selectivity for formate *versus* CO was found to depend on acid strength (Franco *et al.*, 2017).



Herein, we report on the syntheses and structural characterizations of two new complexes of the type *fac*-[*M*(2,2'-bipyridyl)(CO)₃X]^{*n*+}, *viz.* *fac*-bromido[2-(2,2'-bipyridin-6-yl)benzoic acid- κ^2N,N']tricarbonylmanganese(I) tetrahydrofuran monosolvate, **I**, and *fac*-[2-(2,2'-bipyridin-6-yl)benzoic acid- κ^2N,N']tricarbonylchloridorhenium(I) tetrahydrofuran monosolvate, **II**, in which the bipyridyl ligand contains a different type of intramolecular proton donor positioned near the CO₂ binding site. These complexes are the first reported examples of a bipyridyl ligand containing a 2-(2,2'-bipyridin-6-yl)benzoic acid backbone in which the benzoic acid moiety remains protonated and does not coordinate to the metal.

2. Structural commentary

The molecular structures of compounds **I** and **II** are illustrated in Figs. 1 and 2, respectively. Both compounds crystallize as tetrahydrofuran (THF) monosolvates, THF having been used for the recrystallization of both compounds. The metal atoms exhibit distorted octahedral geometries and contain primary

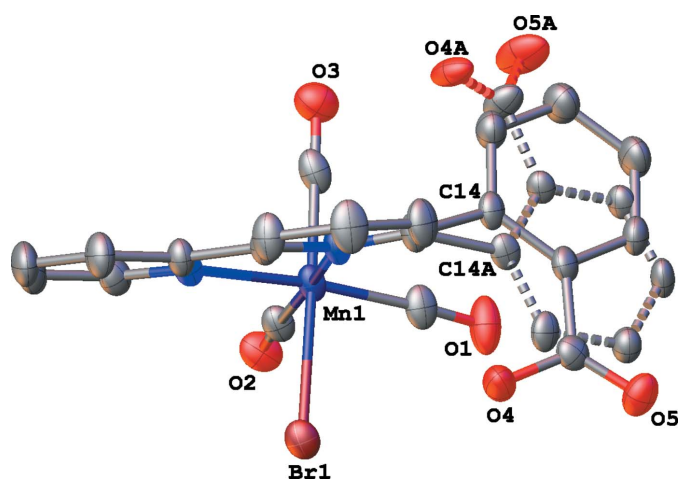


Figure 1
The molecular structure of compound **I**, with partial atom labeling and 50% probability displacement ellipsoids. Both disorder components of the benzoic acid group are shown (the *minor* one with dashed lines), and H atoms have been omitted for clarity.

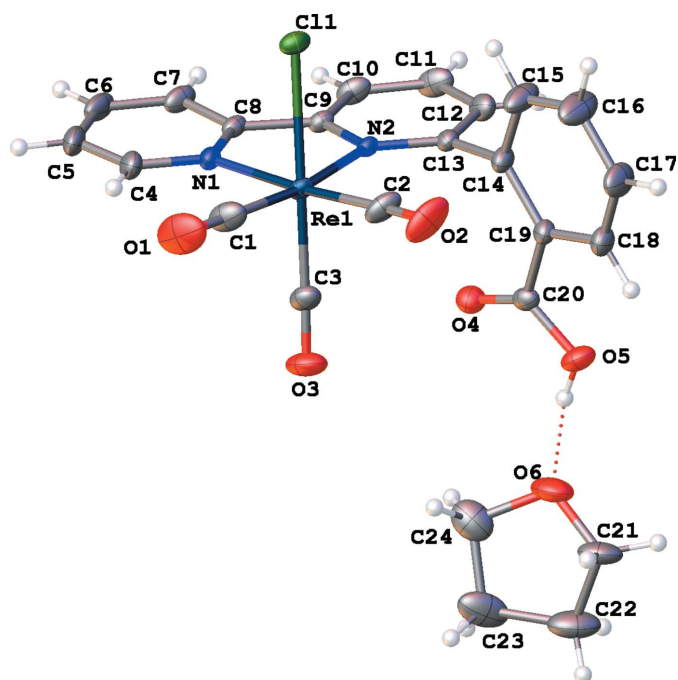


Figure 2
A molecular structure of compound **II**, with atom labeling and 50% probability ellipsoids. The two minor solvent disorder components have been omitted for clarity

coordination spheres similar to those of other *fac*-[Re(α -diimine)(CO)₃Cl] and *fac*-[Mn(α -diimine)(CO)₃Br] complexes; including *fac*-tricarbonylchlorido(4,4'-dihydroxy-2,2'-bipyridine)rhenium(I) (**III**; Manbeck *et al.*, 2015), *fac*-tricarbonyliodido(2,2'-bipyridine)manganese(I) (**IV**; Stor *et al.*, 1995), and *fac*-tricarbonylbromido[2-(2,2'-bipyridin-6-yl)- κ^2N,N']-phenol]manganese(I) (**V**; Agarwal *et al.*, 2015). The metal–ligand bond distances are similar to those previously reported for complexes of this type, for *e.g.*, in **I** the Mn–N bond

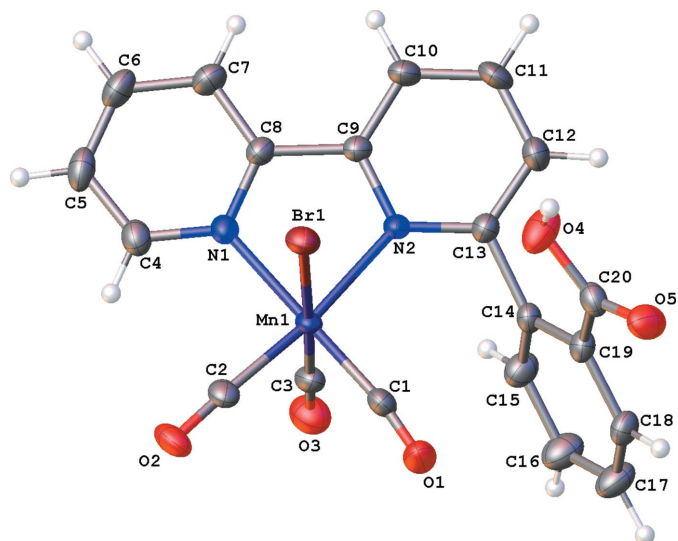


Figure 3
The molecular structure of compound **I**, with atom labeling and showing the position of the major component of the disordered benzoic acid group. Displacement ellipsoids are drawn at the 50% probability level.

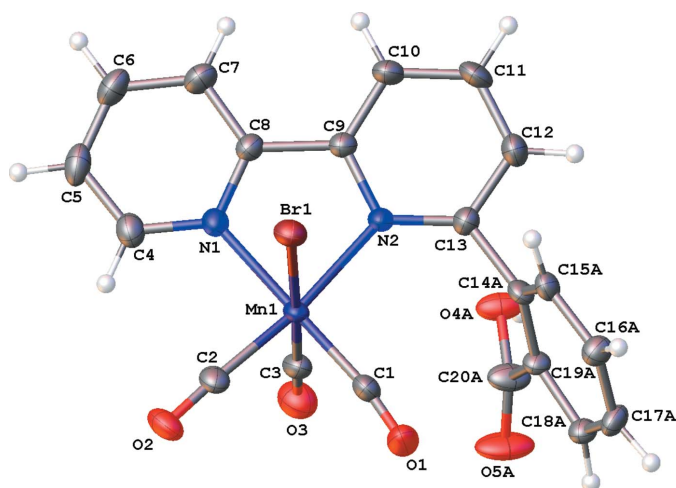


Figure 4
The molecular structure of compound **I**, with atom labeling and showing the position of the minor component of the disordered benzoic acid group. Displacement ellipsoids are drawn at the 50% probability level.

distances are 2.029 (2) and 2.082 (2) Å, while in **V** the Mn–N bond distances are 2.0347 (8) and 2.091 (1) Å.

In **I**, the benzoic acid fragment is disordered over two positions (Fig. 1). In the major component, the carboxylic acid group is positioned near the bromide ligand (see Fig. 3), whereas in the minor component the benzoic acid fragment is rotated such that the carboxylic acid group is positioned near the axial carbonyl ligand (see Fig. 4). In **II**, the benzoic acid fragment is not disordered, and the carboxylic acid group is positioned near the axial carbonyl ligand (Fig. 2).

Molecules with similar motifs, in which a benzoic acid is bound to a pyridyl ring in the *ortho* position (Charris-Molina *et al.*, 2017) or to a phenyl ring in the *ortho* position (Dobson & Gerkin, 1998), have been structurally characterized. Compared to the torsion angles between the benzoic acid fragment and the pyridyl or phenyl rings in these structures, the benzoic acid fragment and the pyridyl ring in **I** and **II** are closer to being perpendicular to each other, with the N2–C13–C14–C19 torsion angle being -116.4 (3)° in the major component of **I** and 100.55 (19)° in **II**. In the minor component of **I**, the N2–C13–C14A–C19A torsion angle is 85.7 (8)°. In contrast, for the structures reported by Charris-Molina *et al.* (2017) the analogous torsion angles are 52.6 (4), -40.5 (3), -51.5 (5) and 48.8 (3)°. In the structure of biphenyl-2-carboxylic acid itself (Dobson & Gerkin, 1998), the analogous torsion angles of the four molecules of the asymmetric unit are -46.7 (4), -52.3 (4), 48.2 (4) and 52.3 (4)°. Smaller absolute values of the torsion angles for **I** and **II** would result in closer contacts between the atoms of the benzoic acid fragment and the ancillary ligands around the metal, which may explain the more perpendicular torsion angles found in **I** and **II**.

3. Supramolecular features

In compound **I**, the THF solvate molecule is disordered over several positions and probably forms intermolecular hydrogen

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C10–H10 \cdots Br1 ⁱ	0.95	2.87	3.685 (3)	144

Symmetry code: (i) $-x + 1, -y + 1, -z + 1$.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5–H5 \cdots O6	0.90 (3)	1.74 (4)	2.615 (4)	164 (3)
O5–H5 \cdots O6A	0.90 (3)	1.68 (4)	2.516 (15)	154 (3)
O5–H5 \cdots O6B	0.90 (3)	1.83 (4)	2.642 (9)	150 (3)
C5–H5A \cdots Cl1 ⁱ	0.95	2.80	3.371 (2)	120
C10–H10 \cdots Cl1 ⁱⁱ	0.95	2.70	3.552 (2)	149
C12–H12 \cdots Cl1 ⁱⁱⁱ	0.95	2.76	3.524 (2)	138

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

bonds. In the crystal, complex molecules are linked by pairs of C–H \cdots Br hydrogen bonds, forming inversion dimers (Table 1). A view of the crystal packing is given in Fig. 5 and shows the voids occupied by the disordered THF solvent molecules.

In compound **II**, there is hydrogen bonding between the benzoic acid group and the oxygen atom of the disordered THF molecule (Table 2). In the crystal, the complex molecules are linked by C–H \cdots Cl hydrogen bonds, forming layers lying parallel to the *bc* plane (Table 2 and Fig. 6), which are separated by layers of THF solvent molecules.

4. Database survey

A search of the Cambridge Structural Database (Version 5.39, last update February 2018; Groom *et al.*, 2016) for complexes

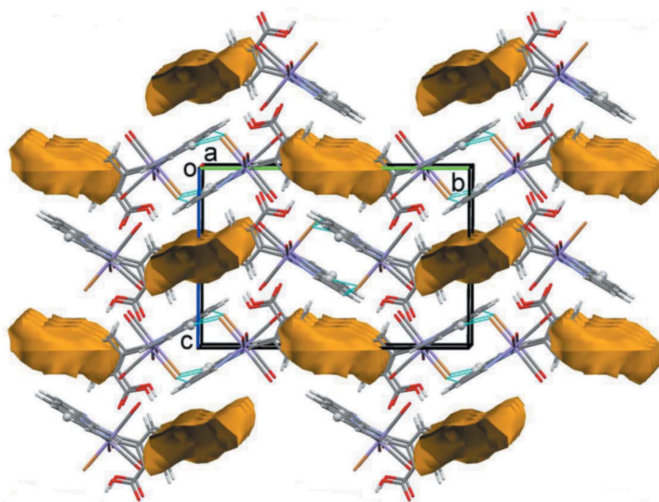


Figure 5
A view along the *a* axis of the crystal packing of compound **I**. The C–H \cdots Br hydrogen bonds (Table 1) are shown as dashed lines and the regions occupied by the disordered THF solvent molecules as yellow/brown cavities (Mercury; Macrae *et al.*, 2008). Only the major component of the disordered benzoic acid group is shown.

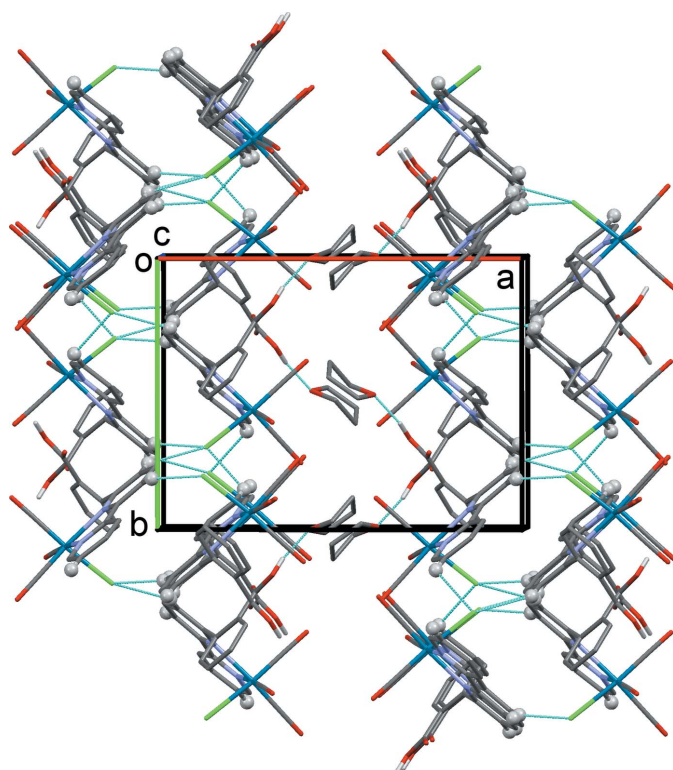


Figure 6
A view along the *c* axis of the crystal packing of compound **II**. The O–H...O and C–H...Cl hydrogen bonds are shown as dashed lines (Table 2). Only the major component of the disordered THF molecule is shown and H atoms not involved in these interactions have been omitted for clarity.

containing a polypyridine ligand with an *o*-benzoate substituent in the 6-position of the polypyridine moiety gave two hits, *viz.* [2-(2,2':4',2''-terpyridin-6'-yl- κ^2N^1, N^1)benzoato- κO]manganese(II) trihydrate (CSD refcode MEWBAT; Liu, 2013) and {[dimethyl(phenyl)silyl]acetato}-[*N*-(3,5-di-*t*-butylphenyl)-2-[6-[3,5-di-*t*-butyl-2-({[tris(pentafluorophenyl)- λ^5 -boranyl]oxy)carbonyl]phenyl]pyridin-2-yl]quinolin-8-amine]-scandium toluene solvate (RIPILOT; LeBlanc *et al.*, 2014). Unlike in **I** and **II**, the benzoate substituent in these complexes is deprotonated and coordinates to the metal forming a seven-membered chelate ring. The reaction conditions and the low oxidation number of the metals in **I** and **II** would be expected to disfavor deprotonation of the benzoic acid substituent and its coordination to the metal.

5. Electrochemistry

In order to determine whether **I** and **II** could act as pre-catalysts for the reduction of CO₂, cyclic voltammetry experiments were performed. These studies were conducted in acetonitrile containing 1 mM **I** or **II** and 0.1 M tetrabutylammonium hexafluorophosphate using a glassy carbon working electrode, a platinum wire auxiliary electrode, and an Ag/Ag⁺ non-aqueous reference electrode. Ferrocene was used as an internal standard. In order to determine whether a catalytic current enhancement was observed in the presence of

the substrate, the current response was measured under an inert gas atmosphere, after bubbling CO₂ through the solution, and after bubbling CO₂ through the solution in the presence of an external Brønsted acid (5% water by volume). In the presence of CO₂ and water, similar complexes have shown a catalytic current enhancement at the potential at which the complexes undergo a second one-electron reduction (Bourrez *et al.*, 2011; Agarwal *et al.*, 2015; Smieja & Kubiak, 2010). Interestingly, neither complex presented here showed electrocatalytic activity for the reduction of CO₂ at or near this potential, even in the presence of an external Brønsted acid (5% water by volume). In order to probe whether catalysis was inhibited specifically by the intramolecular nature of the benzoic acid substituent, the cyclic voltammetry of *fac*-[Mn(2,2'-bipyridyl)(CO)₃Br] was performed in the presence of CO₂, 5% water, and up to 50 molar equivalents of benzoic acid. Even in the presence of 50 molar equivalents of benzoic acid, the current enhancement was similar to that in the presence of only CO₂ and 5% water (Bourrez *et al.*, 2011), indicating that it is the presence of the benzoic acid substituent in an intramolecular fashion that inhibits catalysis.

6. Synthesis and crystallization

Toluene, ethanol, and acetonitrile used in syntheses were degassed by sparging with N₂. THF, hexane, and pentane were dried over molecular sieves and degassed using the freeze-pump-thaw method when used for recrystallization. All other reagents and solvents were purchased commercially and used as received. Metallated complexes were manipulated and stored in the dark to minimize exposure to light.

Synthesis of methyl 2-(2,2'-bipyridin-6-yl)benzoate: The reagents 6-bromo-2,2'-bipyridine (0.500 g, 2.13 mmol) and 2-methoxycarbonylphenylboronic acid, pinacol ester (0.715 g, 2.73 mmol) and the catalyst tetrakis(triphenylphosphine)palladium(0) (0.11 g, 0.095 mmol) were placed in a Kjedahl-shaped Schlenk flask. The flask was then evacuated and refilled with nitrogen three times, ending with the flask under nitrogen. Toluene (26 ml), ethanol (2.6 ml), and 2 M aqueous K₂CO₃ (2.1 ml) were added to the flask, which was then heated at 368 K under nitrogen under stirring for 41 h. The reaction mixture was cooled to room temperature, and then saturated aqueous ammonium chloride (26 ml) and deionized water (26 ml) were added to the reaction flask. The product mixture was then extracted with dichloromethane three times (42 ml, 30 ml, 25 ml). The combined organic layers were dried over magnesium sulfate and then filtered. The solvent was removed under vacuum. The product was purified by column chromatography using silica gel 60 as the solid phase and diethyl ether as the eluant (*R_f* = 0.60). (yield 0.395 g, 63.9%) ¹H NMR (270 MHz, CDCl₃) (ppm): δ 8.69 (*d*, 1H, *J* = 4.7 Hz), δ 8.51–8.48 (*m*, 2H), δ 7.95–7.84 (*m*, 2H), δ 7.77 (*dd*, 1H, *J* = 7.5 Hz, *J* = 1.3 Hz), δ 7.69–7.45 (*m*, 4H), δ 7.34 (poorly resolved multiplet, 1H), δ 3.53 (*s*, 3H). MS (ES-API): found *m/z* = 291.1 [*M* + H]⁺; {C₁₈H₁₅N₂O₂}⁺ requires 291.1.

Synthesis of 2-(2,2'-bipyridin-6-yl)benzoic acid: Water (7.7 ml) containing 0.77 g (19 mmol) of dissolved sodium

Table 3
Experimental details.

	(I)	(II)
Crystal data		
Chemical formula	[MnBr(C ₁₇ H ₁₂ N ₂ O ₂)(CO) ₃] \cdot C ₄ H ₈ O	[ReCl(C ₁₇ H ₁₂ N ₂ O ₂)(CO) ₃] \cdot C ₄ H ₈ O
M_r	495.17	654.07
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$
Temperature (K)	100	100
a, b, c (Å)	10.525 (3), 18.187 (5), 12.459 (4)	15.462 (4), 11.370 (3), 13.370 (3)
β (°)	105.928 (13)	98.023 (10)
V (Å ³)	2293.2 (12)	2327.4 (10)
Z	4	4
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm ⁻¹)	2.35	5.38
Crystal size (mm)	0.21 \times 0.04 \times 0.03	0.28 \times 0.24 \times 0.22
Data collection		
Diffractometer	Bruker SMART APEXIII area detector	Bruker SMART APEXIII area detector
Absorption correction	Multi-scan (SADABS; Krause <i>et al.</i> , 2015)	Analytical (SADABS; Krause <i>et al.</i> , 2015)
T_{\min} , T_{\max}	0.673, 0.802	0.285, 0.526
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	51901, 6992, 5393	70522, 7944, 7361
R_{int}	0.048	0.027
$(\sin \theta/\lambda)_{\text{max}}$ (Å ⁻¹)	0.716	0.742
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.041, 0.104, 1.01	0.016, 0.039, 1.03
No. of reflections	6992	7944
No. of parameters	346	342
No. of restraints	149	27
H-atom treatment	H-atom parameters constrained	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	0.59, -0.77	1.63, -1.64

Computer programs: APEX3 and SAINT-Plus (Bruker, 2015), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b), OLEX2 (Dolomanov *et al.*, 2009), Mercury (Macrae *et al.*, 2008) and PLATON (Spek, 2009).

hydroxide was added to methyl 2-(2,2'-bipyridin-6-yl)benzoate (0.175 g, 0.60 mmol). The reaction mixture was refluxed for 3 h under stirring. The reaction was then cooled to room temperature. Aqueous hydrochloric acid (2 *N*) was added dropwise until the pH was approximately 4. The white precipitate that appeared was collected on a Büchner funnel by vacuum filtration. The precipitate was washed with 4 ml deionized water and then dried *in vacuo* (yield 0.167 g, 100%). ¹H NMR (270 MHz, D₂O containing 1 *M* NaOH) (ppm): δ 8.84 (*d*, 1H, $J = 4.6$ Hz), δ 8.46 (*d*, 1H, $J = 8.1$ Hz), δ 8.21–8.29 (*m*, 3H), δ 7.88–9.95 (*m*, 2H), δ 7.71–7.81 (*m*, 4H).

Synthesis of compound I: Bromopentacarbonylmanganese(I) (0.0525 g, 0.191 mmol) and 2-(2,2'-bipyridin-6-yl)benzoic acid (0.0500 g, 0.181 mmol) were placed in a Schlenk flask, which was then evacuated and refilled with nitrogen three times, ending with the flask under nitrogen. Acetonitrile (9.4 ml) was added to the flask, which was then covered with aluminium foil. The reaction was heated to 333 K and stirred under a nitrogen atmosphere for 12 h. The reaction was cooled to room temperature and the solvent then removed under vacuum. The crude product was dissolved in a minimal amount of THF and recrystallized by slow diffusion of hexane into the solution. These recrystallization conditions performed on a smaller scale produced the yellow needle-like crystals used for X-ray crystallographic analysis. IR ν_{CO} (KBr pellet, cm⁻¹): 2019(*s*), 1933(*s*), 1912(*s*).

Synthesis of compound II: Pentacarbonylchlororhenium(I) (0.0273 g, 0.0755 mmol) and 2-(2,2'-bipyridin-6-yl)benzoic acid (0.0207 g, 0.0749 mmol) were placed in a Schlenk flask, which was then evacuated and refilled with nitrogen three times, ending with the flask under nitrogen. Acetonitrile (3.5 ml) was added to the flask, which was then covered with aluminium foil. The reaction was heated to 333 K and stirred under a nitrogen atmosphere for 12 h. The reaction was cooled to room temperature and the solvent then removed under vacuum. The crude product was dissolved in a minimal amount of THF and recrystallized by slow diffusion of pentane into the solution. These recrystallization conditions performed on a smaller scale produced the yellow block-like crystals used for X-ray crystallographic analysis. IR ν_{CO} (KBr pellet, cm⁻¹): 2072(*s*), 1977(*s*), 1936(*s*).

7. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. For both compounds the C-bound H atoms were included in idealized positions and allowed to ride on the parent atoms: C—H = 0.95–0.99 Å with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. In compound **II**, the carboxylic H atom was located in a difference-Fourier map and freely refined.

In compound **I**, the benzoic acid fragment is disordered over two positions with the major component contribution of

74.8 (3)%. The disordered fragment was refined with restraints. The solvent THF molecule is disordered over at least three positions. Bond-length restraints were applied to model the molecules but the resulting isotropic displacement coefficients suggested they were mobile. In addition, the refinement was computationally unstable. Finally the option SQUEEZE of the program *PLATON* (Spek, 2015) was used to correct the diffraction data for diffuse scattering effects and to identify the solvate molecule. *PLATON* calculated the upper limit of volume that can be occupied by the solvent to be 475 Å³, or 21% of the unit-cell volume. The program calculated 153 electrons in the unit cell for the diffuse species. This closely corresponds to four molecules of THF (160 electrons) per unit cell, or one THF molecule per Mn^I complex (**I**). Their formula mass and unit-cell characteristics were not taken into account during refinement. It is very likely that this solvate molecule, which is disordered over several positions, could form hydrogen bonds.

In compound **II**, the THF molecule is disordered over three positions in a 0.672 (3):0.202 (2):0.126 (3) ratio. The disordered molecules were refined with restraints and constraints (Guzei, 2014).

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supporting information

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Crystal structures of a manganese(I) and a rhenium(I) complex of a bipyridine ligand with a non-coordinating benzoic acid moiety

Sheri Lense, Ilia A. Guzei, Jessica Andersen and Kong Choua Thao

Computing details

For both structures, data collection: *APEX3* (Bruker, 2015); cell refinement: *SAINTE-Plus* (Bruker, 2015); data reduction: *SAINTE-Plus* (Bruker, 2015); program(s) used to solve structure: *SHELXT* (Sheldrick, 2015a); program(s) used to refine structure: *SHELXL* (Sheldrick, 2015b); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009) and *Mercury* (Macrae *et al.*, 2008); software used to prepare material for publication: *OLEX2* (Dolomanov *et al.*, 2009) and *PLATON* (Spek, 2009).

fac-Bromido[2-(2,2'-bipyridin-6-yl)benzoic acid- κ^2N,N']tricarbonylmanganese(I) tetrahydrofuran monosolvate (I)

Crystal data

[MnBr(C₁₇H₁₂N₂O₂)(CO)₃] \cdot C₄H₈O

$M_r = 495.17$

Monoclinic, *P2₁/n*

$a = 10.525$ (3) Å

$b = 18.187$ (5) Å

$c = 12.459$ (4) Å

$\beta = 105.928$ (13)°

$V = 2293.2$ (12) Å³

$Z = 4$

$F(000) = 984$

$D_x = 1.434$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9894 reflections

$\theta = 2.2$ – 28.9 °

$\mu = 2.35$ mm⁻¹

$T = 100$ K

Needle, yellow

$0.21 \times 0.04 \times 0.03$ mm

Data collection

Bruker SMART APEXIII area detector
diffractometer

Radiation source: microfocus sealed X-ray tube,
Incoatec I μ s

Mirror optics monochromator

Detector resolution: 7.9 pixels mm⁻¹

0.5 ° ω and 0.5 ° ϕ scans

Absorption correction: multi-scan
(SADABS; Krause *et al.*, 2015)

$T_{\min} = 0.673$, $T_{\max} = 0.802$

51901 measured reflections

6992 independent reflections

5393 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.048$

$\theta_{\max} = 30.6$ °, $\theta_{\min} = 2.0$ °

$h = -15 \rightarrow 15$

$k = -26 \rightarrow 25$

$l = -17 \rightarrow 17$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.041$

$wR(F^2) = 0.104$

$S = 1.01$

6992 reflections

346 parameters

149 restraints

Primary atom site location: dual

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0415P)^2 + 3.4283P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 0.59 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -0.76 \text{ e } \text{Å}^{-3}$$

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 (Å²)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Br1	0.81210 (3)	0.58497 (2)	0.67241 (2)	0.03094 (8)	
Mn1	0.79215 (3)	0.67459 (2)	0.51236 (3)	0.02312 (9)	
O1	0.8538 (2)	0.79491 (12)	0.6780 (2)	0.0465 (6)	
O2	1.08314 (18)	0.67387 (11)	0.57136 (18)	0.0358 (4)	
O3	0.7716 (2)	0.77396 (12)	0.32364 (19)	0.0422 (5)	
N1	0.7657 (2)	0.58451 (10)	0.41253 (18)	0.0250 (4)	
N2	0.58835 (19)	0.66090 (10)	0.47642 (18)	0.0231 (4)	
C1	0.8213 (2)	0.74942 (14)	0.6135 (2)	0.0312 (6)	
C2	0.9700 (2)	0.67300 (13)	0.5454 (2)	0.0276 (5)	
C3	0.7791 (2)	0.73615 (14)	0.3956 (3)	0.0319 (6)	
C4	0.8611 (3)	0.55066 (14)	0.3774 (2)	0.0320 (6)	
H4	0.9461	0.5727	0.3938	0.038*	
C5	0.8408 (3)	0.48547 (15)	0.3187 (2)	0.0365 (6)	
H5	0.9105	0.4628	0.2960	0.044*	
C6	0.7168 (3)	0.45389 (15)	0.2939 (3)	0.0399 (7)	
H6	0.7000	0.4089	0.2538	0.048*	
C7	0.6172 (3)	0.48852 (15)	0.3280 (3)	0.0361 (6)	
H7	0.5313	0.4676	0.3113	0.043*	
C8	0.6446 (2)	0.55402 (13)	0.3867 (2)	0.0265 (5)	
C9	0.5448 (2)	0.59597 (12)	0.4244 (2)	0.0247 (5)	
C10	0.4168 (3)	0.57149 (15)	0.4078 (3)	0.0352 (6)	
H10	0.3897	0.5259	0.3714	0.042*	
C11	0.3283 (3)	0.61409 (16)	0.4449 (3)	0.0415 (7)	
H11	0.2408	0.5973	0.4370	0.050*	
C12	0.3689 (3)	0.68058 (15)	0.4929 (3)	0.0365 (6)	
H12	0.3088	0.7114	0.5166	0.044*	
C13	0.4982 (2)	0.70278 (13)	0.5069 (2)	0.0276 (5)	
O4	0.5438 (3)	0.69265 (14)	0.7265 (2)	0.0426 (7)	0.748 (3)
H4A	0.5453	0.6705	0.7860	0.064*	0.748 (3)
O5	0.5472 (3)	0.79126 (15)	0.8336 (2)	0.0375 (7)	0.748 (3)
C14	0.5363 (3)	0.78182 (19)	0.5386 (3)	0.0255 (7)	0.748 (3)
C15	0.5461 (5)	0.8292 (2)	0.4543 (4)	0.0335 (10)	0.748 (3)
H15	0.5330	0.8108	0.3807	0.040*	0.748 (3)
C16	0.5748 (5)	0.9035 (2)	0.4753 (4)	0.0411 (9)	0.748 (3)
H16	0.5847	0.9351	0.4174	0.049*	0.748 (3)
C17	0.5889 (4)	0.9309 (2)	0.5823 (4)	0.0393 (9)	0.748 (3)

H17	0.6067	0.9816	0.5975	0.047*	0.748 (3)
C18	0.5768 (4)	0.8846 (2)	0.6650 (4)	0.0300 (9)	0.748 (3)
H18	0.5873	0.9037	0.7379	0.036*	0.748 (3)
C19	0.5495 (3)	0.80940 (17)	0.6456 (3)	0.0260 (7)	0.748 (3)
C20	0.5467 (5)	0.7646 (2)	0.7440 (4)	0.0277 (8)	0.748 (3)
O4A	0.4563 (8)	0.8099 (4)	0.3602 (6)	0.0343 (19)	0.252 (3)
H4AA	0.4104	0.8358	0.3080	0.051*	0.252 (3)
O5A	0.5901 (11)	0.9062 (5)	0.3988 (8)	0.053 (3)	0.252 (3)
C14A	0.5335 (9)	0.7683 (5)	0.5909 (9)	0.024 (2)	0.252 (3)
C15A	0.5520 (14)	0.7576 (7)	0.7034 (10)	0.027 (3)	0.252 (3)
H15A	0.5446	0.7098	0.7319	0.033*	0.252 (3)
C16A	0.5818 (10)	0.8179 (5)	0.7748 (8)	0.028 (2)	0.252 (3)
H16A	0.5914	0.8127	0.8525	0.034*	0.252 (3)
C17A	0.5970 (11)	0.8861 (5)	0.7292 (9)	0.029 (2)	0.252 (3)
H17A	0.6121	0.9277	0.7772	0.035*	0.252 (3)
C18A	0.5916 (11)	0.8967 (6)	0.6210 (9)	0.024 (2)	0.252 (3)
H18A	0.6125	0.9429	0.5947	0.029*	0.252 (3)
C19A	0.5536 (8)	0.8360 (4)	0.5484 (7)	0.0224 (18)	0.252 (3)
C20A	0.5423 (17)	0.8528 (7)	0.4324 (10)	0.034 (3)	0.252 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Br1	0.02879 (13)	0.02479 (12)	0.04266 (16)	-0.00564 (9)	0.01555 (11)	0.00050 (10)
Mn1	0.02055 (17)	0.01701 (15)	0.0360 (2)	-0.00424 (12)	0.01480 (15)	-0.00188 (14)
O1	0.0328 (10)	0.0387 (11)	0.0734 (16)	-0.0104 (9)	0.0234 (11)	-0.0271 (11)
O2	0.0229 (9)	0.0385 (10)	0.0490 (12)	-0.0039 (7)	0.0148 (8)	0.0071 (9)
O3	0.0390 (11)	0.0416 (11)	0.0484 (13)	-0.0009 (9)	0.0160 (10)	0.0083 (10)
N1	0.0253 (10)	0.0208 (9)	0.0327 (11)	-0.0001 (7)	0.0144 (9)	0.0007 (8)
N2	0.0218 (9)	0.0171 (8)	0.0340 (11)	-0.0034 (7)	0.0139 (8)	-0.0027 (8)
C1	0.0220 (11)	0.0286 (12)	0.0481 (16)	-0.0055 (9)	0.0183 (11)	-0.0071 (11)
C2	0.0281 (12)	0.0219 (10)	0.0378 (14)	-0.0031 (9)	0.0172 (11)	0.0031 (10)
C3	0.0239 (12)	0.0224 (11)	0.0532 (17)	-0.0046 (9)	0.0173 (12)	-0.0046 (11)
C4	0.0303 (13)	0.0305 (12)	0.0402 (15)	0.0029 (10)	0.0182 (12)	0.0009 (11)
C5	0.0420 (15)	0.0334 (13)	0.0394 (15)	0.0132 (11)	0.0200 (13)	-0.0015 (11)
C6	0.0484 (17)	0.0271 (13)	0.0469 (17)	0.0033 (12)	0.0178 (14)	-0.0119 (12)
C7	0.0363 (14)	0.0267 (12)	0.0473 (17)	-0.0057 (10)	0.0151 (13)	-0.0132 (11)
C8	0.0287 (12)	0.0197 (10)	0.0347 (13)	-0.0025 (9)	0.0150 (10)	-0.0049 (9)
C9	0.0247 (11)	0.0197 (10)	0.0330 (13)	-0.0044 (8)	0.0134 (10)	-0.0038 (9)
C10	0.0296 (13)	0.0276 (12)	0.0539 (18)	-0.0117 (10)	0.0206 (13)	-0.0121 (12)
C11	0.0260 (13)	0.0384 (14)	0.068 (2)	-0.0138 (11)	0.0261 (14)	-0.0121 (14)
C12	0.0284 (13)	0.0325 (13)	0.0559 (18)	-0.0017 (10)	0.0237 (13)	-0.0102 (12)
C13	0.0257 (11)	0.0217 (10)	0.0387 (14)	-0.0012 (9)	0.0143 (10)	-0.0060 (10)
O4	0.068 (2)	0.0295 (13)	0.0364 (15)	0.0106 (12)	0.0239 (15)	0.0043 (11)
O5	0.0448 (16)	0.0388 (14)	0.0356 (15)	-0.0106 (12)	0.0223 (12)	-0.0067 (11)
C14	0.0217 (15)	0.0238 (16)	0.0346 (19)	0.0003 (12)	0.0137 (15)	-0.0061 (14)
C15	0.041 (2)	0.026 (2)	0.036 (2)	-0.0005 (17)	0.0158 (19)	-0.0044 (16)
C16	0.056 (3)	0.0269 (17)	0.043 (2)	-0.0034 (16)	0.0183 (19)	-0.0004 (15)

C17	0.055 (3)	0.0223 (17)	0.044 (2)	-0.0020 (16)	0.0189 (19)	-0.0034 (16)
C18	0.034 (2)	0.0257 (17)	0.034 (2)	0.0016 (14)	0.016 (2)	-0.0077 (17)
C19	0.0243 (15)	0.0233 (14)	0.0327 (17)	0.0036 (11)	0.0117 (13)	-0.0038 (13)
C20	0.0226 (17)	0.0299 (18)	0.034 (2)	0.0031 (13)	0.0132 (18)	0.0016 (16)
O4A	0.047 (4)	0.030 (3)	0.021 (3)	-0.016 (3)	0.002 (3)	0.004 (3)
O5A	0.076 (7)	0.043 (4)	0.038 (5)	-0.033 (4)	0.015 (4)	0.007 (4)
C14A	0.022 (4)	0.022 (4)	0.035 (5)	-0.004 (3)	0.017 (4)	0.000 (3)
C15A	0.023 (5)	0.028 (4)	0.036 (5)	-0.003 (3)	0.017 (4)	-0.007 (4)
C16A	0.033 (5)	0.024 (4)	0.031 (4)	-0.002 (3)	0.014 (4)	-0.003 (3)
C17A	0.039 (6)	0.019 (4)	0.037 (5)	0.001 (3)	0.021 (5)	-0.006 (3)
C18A	0.027 (5)	0.017 (4)	0.031 (5)	-0.003 (3)	0.014 (4)	-0.001 (3)
C19A	0.025 (4)	0.018 (3)	0.029 (4)	-0.004 (3)	0.014 (3)	0.000 (3)
C20A	0.046 (7)	0.033 (6)	0.026 (4)	-0.017 (5)	0.015 (4)	0.001 (3)

Geometric parameters (Å, °)

Br1—Mn1	2.5391 (7)	C13—C14A	1.562 (10)
Mn1—N1	2.029 (2)	O4—H4A	0.8400
Mn1—N2	2.082 (2)	O4—C20	1.326 (5)
Mn1—C1	1.822 (3)	O5—C20	1.215 (5)
Mn1—C2	1.803 (3)	C14—C15	1.384 (6)
Mn1—C3	1.810 (3)	C14—C19	1.395 (5)
O1—C1	1.139 (3)	C15—H15	0.9500
O2—C2	1.145 (3)	C15—C16	1.394 (5)
O3—C3	1.116 (3)	C16—H16	0.9500
N1—C4	1.349 (3)	C16—C17	1.392 (6)
N1—C8	1.345 (3)	C17—H17	0.9500
N2—C9	1.364 (3)	C17—C18	1.364 (6)
N2—C13	1.350 (3)	C18—H18	0.9500
C4—H4	0.9500	C18—C19	1.405 (5)
C4—C5	1.378 (4)	C19—C20	1.479 (5)
C5—H5	0.9500	O4A—H4AA	0.8400
C5—C6	1.381 (4)	O4A—C20A	1.338 (13)
C6—H6	0.9500	O5A—C20A	1.220 (12)
C6—C7	1.386 (4)	C14A—C15A	1.375 (13)
C7—H7	0.9500	C14A—C19A	1.378 (10)
C7—C8	1.386 (3)	C15A—H15A	0.9500
C8—C9	1.476 (3)	C15A—C16A	1.393 (12)
C9—C10	1.379 (3)	C16A—H16A	0.9500
C10—H10	0.9500	C16A—C17A	1.391 (12)
C10—C11	1.384 (4)	C17A—H17A	0.9500
C11—H11	0.9500	C17A—C18A	1.346 (12)
C11—C12	1.365 (4)	C18A—H18A	0.9500
C12—H12	0.9500	C18A—C19A	1.414 (11)
C12—C13	1.384 (4)	C19A—C20A	1.449 (12)
C13—C14	1.515 (4)		
N1—Mn1—Br1	86.05 (6)	C13—C12—H12	120.3

N1—Mn1—N2	79.15 (8)	N2—C13—C12	122.9 (2)
N2—Mn1—Br1	87.16 (6)	N2—C13—C14	117.0 (2)
C1—Mn1—Br1	88.54 (9)	N2—C13—C14A	124.3 (4)
C1—Mn1—N1	174.43 (11)	C12—C13—C14	119.3 (2)
C1—Mn1—N2	101.88 (9)	C12—C13—C14A	110.6 (4)
C2—Mn1—Br1	87.32 (8)	C20—O4—H4A	109.5
C2—Mn1—N1	94.95 (10)	C15—C14—C13	117.5 (3)
C2—Mn1—N2	172.18 (9)	C15—C14—C19	119.5 (3)
C2—Mn1—C1	83.50 (11)	C19—C14—C13	122.7 (3)
C3—Mn1—Br1	178.20 (8)	C14—C15—H15	119.4
C3—Mn1—N1	92.27 (11)	C14—C15—C16	121.3 (4)
C3—Mn1—N2	93.14 (10)	C16—C15—H15	119.4
C3—Mn1—C1	93.13 (13)	C15—C16—H16	120.4
C3—Mn1—C2	92.21 (11)	C15—C16—C17	119.2 (4)
C4—N1—Mn1	125.32 (18)	C17—C16—H16	120.4
C8—N1—Mn1	116.16 (16)	C16—C17—H17	120.2
C8—N1—C4	118.3 (2)	C18—C17—C16	119.7 (4)
C9—N2—Mn1	113.44 (15)	C18—C17—H17	120.2
C13—N2—Mn1	129.34 (16)	C17—C18—H18	119.1
C13—N2—C9	116.8 (2)	C17—C18—C19	121.9 (4)
O1—C1—Mn1	172.5 (2)	C19—C18—H18	119.1
O2—C2—Mn1	176.5 (2)	C14—C19—C18	118.5 (4)
O3—C3—Mn1	179.7 (3)	C14—C19—C20	125.1 (3)
N1—C4—H4	118.5	C18—C19—C20	116.3 (4)
N1—C4—C5	122.9 (3)	O4—C20—C19	114.3 (4)
C5—C4—H4	118.5	O5—C20—O4	122.6 (4)
C4—C5—H5	120.8	O5—C20—C19	123.1 (4)
C4—C5—C6	118.5 (2)	C20A—O4A—H4AA	109.5
C6—C5—H5	120.8	C15A—C14A—C13	121.1 (8)
C5—C6—H6	120.3	C15A—C14A—C19A	121.6 (9)
C5—C6—C7	119.3 (3)	C19A—C14A—C13	117.2 (8)
C7—C6—H6	120.3	C14A—C15A—H15A	120.5
C6—C7—H7	120.4	C14A—C15A—C16A	118.9 (11)
C6—C7—C8	119.1 (3)	C16A—C15A—H15A	120.5
C8—C7—H7	120.4	C15A—C16A—H16A	120.9
N1—C8—C7	121.8 (2)	C17A—C16A—C15A	118.2 (9)
N1—C8—C9	114.9 (2)	C17A—C16A—H16A	120.9
C7—C8—C9	123.3 (2)	C16A—C17A—H17A	118.1
N2—C9—C8	115.0 (2)	C18A—C17A—C16A	123.9 (9)
N2—C9—C10	122.5 (2)	C18A—C17A—H17A	118.1
C10—C9—C8	122.5 (2)	C17A—C18A—H18A	121.4
C9—C10—H10	120.4	C17A—C18A—C19A	117.1 (10)
C9—C10—C11	119.2 (2)	C19A—C18A—H18A	121.4
C11—C10—H10	120.4	C14A—C19A—C18A	119.8 (9)
C10—C11—H11	120.5	C14A—C19A—C20A	126.3 (9)
C12—C11—C10	118.9 (2)	C18A—C19A—C20A	113.8 (8)
C12—C11—H11	120.5	O4A—C20A—C19A	113.7 (9)
C11—C12—H12	120.3	O5A—C20A—O4A	120.0 (10)

C11—C12—C13	119.5 (2)	O5A—C20A—C19A	125.6 (11)
Mn1—N1—C4—C5	173.4 (2)	C12—C13—C14—C19	73.9 (4)
Mn1—N1—C8—C7	-173.9 (2)	C12—C13—C14A—C15A	72.9 (10)
Mn1—N1—C8—C9	6.8 (3)	C12—C13—C14A—C19A	-111.1 (7)
Mn1—N2—C9—C8	-9.8 (3)	C13—N2—C9—C8	176.6 (2)
Mn1—N2—C9—C10	170.4 (2)	C13—N2—C9—C10	-3.2 (4)
Mn1—N2—C13—C12	-168.8 (2)	C13—C14—C15—C16	177.1 (4)
Mn1—N2—C13—C14	21.9 (4)	C13—C14—C19—C18	-176.0 (3)
Mn1—N2—C13—C14A	-7.7 (6)	C13—C14—C19—C20	8.8 (5)
N1—C4—C5—C6	0.7 (4)	C13—C14A—C15A—C16A	-179.2 (9)
N1—C8—C9—N2	2.3 (3)	C13—C14A—C19A—C18A	-177.2 (8)
N1—C8—C9—C10	-178.0 (3)	C13—C14A—C19A—C20A	1.5 (15)
N2—C9—C10—C11	0.2 (5)	C14—C15—C16—C17	-2.5 (7)
N2—C13—C14—C15	69.6 (4)	C14—C19—C20—O4	6.6 (6)
N2—C13—C14—C19	-116.4 (3)	C14—C19—C20—O5	-173.5 (4)
N2—C13—C14A—C15A	-90.2 (10)	C15—C14—C19—C18	-2.1 (5)
N2—C13—C14A—C19A	85.7 (8)	C15—C14—C19—C20	-177.3 (4)
C4—N1—C8—C7	1.6 (4)	C15—C16—C17—C18	1.2 (7)
C4—N1—C8—C9	-177.7 (2)	C16—C17—C18—C19	-0.5 (7)
C4—C5—C6—C7	0.2 (5)	C17—C18—C19—C14	0.9 (6)
C5—C6—C7—C8	-0.2 (5)	C17—C18—C19—C20	176.6 (4)
C6—C7—C8—N1	-0.7 (4)	C18—C19—C20—O4	-168.7 (4)
C6—C7—C8—C9	178.5 (3)	C18—C19—C20—O5	11.2 (6)
C7—C8—C9—N2	-177.1 (3)	C19—C14—C15—C16	2.9 (7)
C7—C8—C9—C10	2.7 (4)	C14A—C15A—C16A—C17A	-2.7 (17)
C8—N1—C4—C5	-1.7 (4)	C14A—C19A—C20A—O4A	29 (2)
C8—C9—C10—C11	-179.6 (3)	C14A—C19A—C20A—O5A	-160.9 (15)
C9—N2—C13—C12	3.6 (4)	C15A—C14A—C19A—C18A	-1.3 (15)
C9—N2—C13—C14	-165.7 (3)	C15A—C14A—C19A—C20A	177.4 (13)
C9—N2—C13—C14A	164.7 (5)	C15A—C16A—C17A—C18A	-3.7 (17)
C9—C10—C11—C12	2.6 (5)	C16A—C17A—C18A—C19A	7.3 (17)
C10—C11—C12—C13	-2.2 (5)	C17A—C18A—C19A—C14A	-4.8 (15)
C11—C12—C13—N2	-1.0 (5)	C17A—C18A—C19A—C20A	176.4 (12)
C11—C12—C13—C14	168.0 (3)	C18A—C19A—C20A—O4A	-152.6 (12)
C11—C12—C13—C14A	-164.4 (5)	C18A—C19A—C20A—O5A	18 (2)
C12—C13—C14—C15	-100.1 (4)	C19A—C14A—C15A—C16A	5.0 (17)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
C10—H10...Br1 ⁱ	0.95	2.87	3.685 (3)	144

Symmetry code: (i) $-x+1, -y+1, -z+1$.

fac-[2-(2,2'-Bipyridin-6-yl)benzoic acid- κ^2N,N']tricarbonylchloridorhenium(I) tetrahydrofuran monosolvate (II)*Crystal data*[ReCl(C₁₇H₁₂N₂O₂)(CO)₃] \cdot C₄H₈O $M_r = 654.07$ Monoclinic, $P2_1/c$ $a = 15.462$ (4) Å $b = 11.370$ (3) Å $c = 13.370$ (3) Å $\beta = 98.023$ (10)° $V = 2327.4$ (10) Å³ $Z = 4$ $F(000) = 1272$ $D_x = 1.867$ Mg m⁻³Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 9101 reflections

 $\theta = 2.6$ – 31.8 ° $\mu = 5.38$ mm⁻¹ $T = 100$ K

Block, yellow

 $0.28 \times 0.24 \times 0.22$ mm*Data collection*Bruker SMART APEXIII area detector
diffractometerRadiation source: microfocus sealed X-ray tube,
Incoatec I μ s

Mirror optics monochromator

Detector resolution: 7.9 pixels mm⁻¹0.5° ω and 0.5° ϕ scansAbsorption correction: analytical
(SADABS; Krause *et al.*, 2015) $T_{\min} = 0.285$, $T_{\max} = 0.526$

70522 measured reflections

7944 independent reflections

7361 reflections with $I > 2\sigma(I)$ $R_{\text{int}} = 0.027$ $\theta_{\max} = 31.9$ °, $\theta_{\min} = 1.3$ ° $h = -22 \rightarrow 22$ $k = -16 \rightarrow 16$ $l = -19 \rightarrow 19$ *Refinement*Refinement on F^2

Least-squares matrix: full

 $R[F^2 > 2\sigma(F^2)] = 0.016$ $wR(F^2) = 0.039$ $S = 1.03$

7944 reflections

342 parameters

27 restraints

Primary atom site location: dual

Hydrogen site location: mixed

H atoms treated by a mixture of independent
and constrained refinement $w = 1/[\sigma^2(F_o^2) + (0.0182P)^2 + 2.0257P]$ where $P = (F_o^2 + 2F_c^2)/3$ $(\Delta/\sigma)_{\max} = 0.003$ $\Delta\rho_{\max} = 1.63$ e Å⁻³ $\Delta\rho_{\min} = -1.64$ e Å⁻³*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 (Å²)*

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Re1	0.25443 (2)	0.56361 (2)	0.41243 (2)	0.01643 (2)	
Cl1	0.12745 (2)	0.69931 (3)	0.37941 (3)	0.02075 (7)	
O1	0.38542 (11)	0.75829 (16)	0.48236 (17)	0.0571 (5)	
O2	0.31136 (12)	0.60610 (14)	0.20431 (13)	0.0469 (4)	
O3	0.40094 (9)	0.38300 (14)	0.46361 (13)	0.0395 (3)	
O4	0.25774 (8)	0.21703 (11)	0.31781 (9)	0.0263 (2)	
O5	0.31281 (9)	0.16759 (12)	0.17689 (11)	0.0295 (3)	
H5	0.339 (2)	0.111 (3)	0.218 (2)	0.064 (9)*	
N1	0.20926 (9)	0.54664 (12)	0.55735 (10)	0.0189 (2)	

N2	0.15082 (8)	0.42971 (10)	0.38837 (9)	0.0141 (2)	
C1	0.33623 (12)	0.68554 (17)	0.45436 (17)	0.0336 (4)	
C2	0.28734 (13)	0.58568 (16)	0.28011 (16)	0.0295 (4)	
C3	0.34552 (11)	0.44986 (15)	0.44347 (15)	0.0260 (3)	
C4	0.24129 (12)	0.60914 (17)	0.63982 (14)	0.0298 (4)	
H4	0.2891	0.6606	0.6359	0.036*	
C5	0.20740 (15)	0.6013 (2)	0.72977 (15)	0.0386 (5)	
H5A	0.2321	0.6456	0.7870	0.046*	
C6	0.13710 (14)	0.5282 (2)	0.73522 (13)	0.0365 (5)	
H6	0.1123	0.5219	0.7961	0.044*	
C7	0.10326 (12)	0.46437 (17)	0.65100 (13)	0.0271 (3)	
H7	0.0542	0.4145	0.6530	0.033*	
C8	0.14155 (10)	0.47363 (13)	0.56311 (11)	0.0178 (3)	
C9	0.11021 (9)	0.40700 (13)	0.47062 (11)	0.0163 (2)	
C10	0.04153 (11)	0.32791 (14)	0.46733 (14)	0.0256 (3)	
H10	0.0159	0.3116	0.5264	0.031*	
C11	0.01085 (12)	0.27318 (16)	0.37729 (17)	0.0313 (4)	
H11	-0.0374	0.2207	0.3729	0.038*	
C12	0.05092 (11)	0.29564 (15)	0.29444 (14)	0.0272 (3)	
H12	0.0306	0.2587	0.2318	0.033*	
C13	0.12151 (10)	0.37260 (13)	0.30166 (11)	0.0185 (3)	
C14	0.15673 (11)	0.39961 (15)	0.20576 (12)	0.0236 (3)	
C15	0.11602 (16)	0.4894 (2)	0.14585 (15)	0.0413 (5)	
H15	0.0723	0.5356	0.1704	0.050*	
C16	0.13830 (19)	0.5124 (2)	0.05091 (16)	0.0514 (7)	
H16	0.1110	0.5752	0.0116	0.062*	
C17	0.20068 (16)	0.4436 (2)	0.01340 (14)	0.0395 (5)	
H17	0.2160	0.4589	-0.0518	0.047*	
C18	0.24041 (12)	0.35285 (17)	0.07105 (12)	0.0274 (3)	
H18	0.2825	0.3052	0.0448	0.033*	
C19	0.21945 (10)	0.33025 (14)	0.16769 (12)	0.0206 (3)	
C20	0.26432 (10)	0.23343 (14)	0.23004 (13)	0.0204 (3)	
O6	0.4105 (3)	0.0039 (5)	0.2725 (3)	0.0444 (9)	0.672 (3)
C21	0.4761 (3)	-0.0296 (4)	0.2093 (3)	0.0412 (10)	0.672 (3)
H21A	0.5111	0.0390	0.1931	0.049*	0.672 (3)
H21B	0.4492	-0.0674	0.1458	0.049*	0.672 (3)
C22	0.5303 (3)	-0.1156 (4)	0.2774 (4)	0.0535 (11)	0.672 (3)
H22A	0.5900	-0.1222	0.2591	0.064*	0.672 (3)
H22B	0.5029	-0.1945	0.2737	0.064*	0.672 (3)
C23	0.5321 (2)	-0.0610 (4)	0.3845 (3)	0.0523 (11)	0.672 (3)
H23A	0.5304	-0.1234	0.4358	0.063*	0.672 (3)
H23B	0.5856	-0.0134	0.4028	0.063*	0.672 (3)
C24	0.4518 (3)	0.0153 (4)	0.3778 (3)	0.0467 (9)	0.672 (3)
H24A	0.4122	-0.0126	0.4249	0.056*	0.672 (3)
H24B	0.4678	0.0981	0.3940	0.056*	0.672 (3)
O6A	0.4080 (11)	-0.0019 (17)	0.2447 (7)	0.0339 (13)	0.202 (3)
C24A	0.4408 (8)	-0.0269 (14)	0.3488 (8)	0.0339 (13)	0.202 (3)
H24C	0.3953	-0.0664	0.3821	0.041*	0.202 (3)

H24D	0.4579	0.0469	0.3857	0.041*	0.202 (3)
C23A	0.5209 (7)	-0.1082 (11)	0.3488 (8)	0.0339 (13)	0.202 (3)
H23C	0.5763	-0.0639	0.3632	0.041*	0.202 (3)
H23D	0.5207	-0.1731	0.3981	0.041*	0.202 (3)
C22A	0.5060 (8)	-0.1537 (11)	0.2397 (8)	0.0339 (13)	0.202 (3)
H22C	0.5610	-0.1829	0.2184	0.041*	0.202 (3)
H22D	0.4617	-0.2171	0.2311	0.041*	0.202 (3)
C21A	0.4733 (14)	-0.0426 (17)	0.1815 (10)	0.0339 (13)	0.202 (3)
H21C	0.5206	0.0156	0.1796	0.041*	0.202 (3)
H21D	0.4461	-0.0610	0.1117	0.041*	0.202 (3)
O6B	0.4356 (8)	0.0219 (9)	0.2593 (10)	0.0298 (18)*	0.126 (3)
C21B	0.4661 (11)	-0.0418 (14)	0.1775 (7)	0.0298 (18)*	0.126 (3)
H21E	0.4967	0.0119	0.1359	0.036*	0.126 (3)
H21F	0.4164	-0.0783	0.1338	0.036*	0.126 (3)
C22B	0.5288 (9)	-0.1366 (12)	0.2266 (9)	0.0298 (18)*	0.126 (3)
H22E	0.5892	-0.1061	0.2424	0.036*	0.126 (3)
H22F	0.5288	-0.2068	0.1828	0.036*	0.126 (3)
C23B	0.4892 (9)	-0.1641 (9)	0.3233 (9)	0.0298 (18)*	0.126 (3)
H23E	0.4393	-0.2192	0.3098	0.036*	0.126 (3)
H23F	0.5333	-0.1975	0.3765	0.036*	0.126 (3)
C24B	0.4593 (9)	-0.0416 (11)	0.3526 (7)	0.0298 (18)*	0.126 (3)
H24E	0.4085	-0.0480	0.3899	0.036*	0.126 (3)
H24F	0.5071	-0.0007	0.3959	0.036*	0.126 (3)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.01365 (3)	0.01547 (3)	0.02146 (3)	-0.00104 (2)	0.00704 (2)	-0.00100 (2)
Cl1	0.02042 (15)	0.01814 (15)	0.02561 (17)	0.00341 (12)	0.00999 (12)	0.00509 (13)
O1	0.0344 (8)	0.0381 (9)	0.0991 (15)	-0.0196 (7)	0.0102 (9)	-0.0154 (9)
O2	0.0676 (11)	0.0336 (8)	0.0500 (9)	-0.0017 (7)	0.0453 (9)	0.0034 (7)
O3	0.0222 (6)	0.0359 (8)	0.0597 (10)	0.0097 (6)	0.0029 (6)	-0.0023 (7)
O4	0.0279 (6)	0.0249 (6)	0.0258 (6)	0.0039 (5)	0.0025 (5)	0.0017 (5)
O5	0.0250 (6)	0.0258 (6)	0.0395 (7)	0.0063 (5)	0.0105 (5)	-0.0027 (5)
N1	0.0201 (6)	0.0206 (6)	0.0159 (5)	0.0042 (5)	0.0022 (4)	-0.0032 (4)
N2	0.0152 (5)	0.0137 (5)	0.0142 (5)	0.0023 (4)	0.0046 (4)	0.0000 (4)
C1	0.0223 (8)	0.0249 (8)	0.0550 (12)	-0.0057 (7)	0.0106 (7)	-0.0059 (8)
C2	0.0330 (9)	0.0223 (7)	0.0385 (10)	0.0010 (6)	0.0234 (8)	0.0013 (7)
C3	0.0171 (7)	0.0261 (8)	0.0355 (9)	-0.0004 (6)	0.0062 (6)	-0.0043 (6)
C4	0.0297 (8)	0.0322 (9)	0.0258 (8)	0.0064 (7)	-0.0017 (6)	-0.0135 (7)
C5	0.0413 (11)	0.0496 (12)	0.0227 (8)	0.0188 (9)	-0.0034 (7)	-0.0165 (8)
C6	0.0434 (11)	0.0513 (12)	0.0160 (7)	0.0246 (10)	0.0085 (7)	0.0002 (7)
C7	0.0334 (9)	0.0310 (8)	0.0196 (7)	0.0122 (7)	0.0128 (6)	0.0070 (6)
C8	0.0215 (6)	0.0177 (6)	0.0151 (6)	0.0060 (5)	0.0061 (5)	0.0019 (5)
C9	0.0169 (6)	0.0148 (6)	0.0185 (6)	0.0019 (5)	0.0069 (5)	0.0013 (5)
C10	0.0230 (7)	0.0198 (7)	0.0370 (9)	-0.0019 (6)	0.0148 (6)	0.0016 (6)
C11	0.0215 (7)	0.0208 (7)	0.0525 (11)	-0.0063 (6)	0.0084 (7)	-0.0063 (7)
C12	0.0235 (7)	0.0229 (7)	0.0339 (9)	-0.0003 (6)	-0.0002 (6)	-0.0115 (6)

C13	0.0188 (6)	0.0177 (6)	0.0185 (6)	0.0043 (5)	0.0015 (5)	-0.0038 (5)
C14	0.0287 (8)	0.0259 (7)	0.0159 (6)	0.0078 (6)	0.0021 (6)	-0.0033 (6)
C15	0.0574 (13)	0.0442 (11)	0.0242 (9)	0.0312 (10)	0.0117 (8)	0.0077 (8)
C16	0.0722 (17)	0.0596 (15)	0.0237 (9)	0.0390 (14)	0.0119 (10)	0.0149 (9)
C17	0.0532 (13)	0.0509 (13)	0.0155 (7)	0.0185 (10)	0.0084 (8)	0.0037 (7)
C18	0.0319 (8)	0.0321 (9)	0.0182 (7)	0.0052 (7)	0.0037 (6)	-0.0051 (6)
C19	0.0226 (7)	0.0208 (7)	0.0176 (6)	0.0031 (5)	0.0004 (5)	-0.0046 (5)
C20	0.0148 (6)	0.0188 (6)	0.0273 (7)	-0.0012 (5)	0.0013 (5)	-0.0033 (6)
O6	0.0276 (13)	0.0409 (15)	0.061 (2)	0.0138 (11)	-0.0072 (19)	0.001 (2)
C21	0.0222 (14)	0.0421 (19)	0.058 (3)	0.0132 (13)	0.001 (2)	0.009 (2)
C22	0.0369 (19)	0.053 (2)	0.071 (3)	0.0202 (18)	0.010 (2)	0.022 (2)
C23	0.0303 (16)	0.080 (3)	0.049 (2)	0.0020 (18)	0.0129 (16)	0.028 (2)
C24	0.046 (2)	0.044 (2)	0.048 (2)	-0.0090 (17)	0.0000 (17)	0.0119 (17)
O6A	0.032 (3)	0.049 (3)	0.020 (2)	0.007 (2)	0.0013 (19)	0.008 (2)
C24A	0.032 (3)	0.049 (3)	0.020 (2)	0.007 (2)	0.0013 (19)	0.008 (2)
C23A	0.032 (3)	0.049 (3)	0.020 (2)	0.007 (2)	0.0013 (19)	0.008 (2)
C22A	0.032 (3)	0.049 (3)	0.020 (2)	0.007 (2)	0.0013 (19)	0.008 (2)
C21A	0.032 (3)	0.049 (3)	0.020 (2)	0.007 (2)	0.0013 (19)	0.008 (2)

Geometric parameters (Å, °)

Re1—C11	2.4875 (6)	C18—C19	1.399 (2)
Re1—N1	2.1580 (14)	C19—C20	1.492 (2)
Re1—N2	2.2008 (13)	O6—C21	1.458 (5)
Re1—C1	1.9074 (19)	O6—C24	1.469 (5)
Re1—C2	1.9244 (19)	C21—H21A	0.9900
Re1—C3	1.9146 (18)	C21—H21B	0.9900
O1—C1	1.150 (2)	C21—C22	1.508 (5)
O2—C2	1.151 (2)	C22—H22A	0.9900
O3—C3	1.149 (2)	C22—H22B	0.9900
O4—C20	1.206 (2)	C22—C23	1.558 (7)
O5—H5	0.90 (3)	C23—H23A	0.9900
O5—C20	1.333 (2)	C23—H23B	0.9900
N1—C4	1.347 (2)	C23—C24	1.507 (5)
N1—C8	1.347 (2)	C24—H24A	0.9900
N2—C9	1.3648 (18)	C24—H24B	0.9900
N2—C13	1.3506 (19)	O6A—C24A	1.441 (11)
C4—H4	0.9500	O6A—C21A	1.478 (13)
C4—C5	1.380 (3)	C24A—H24C	0.9900
C5—H5A	0.9500	C24A—H24D	0.9900
C5—C6	1.378 (4)	C24A—C23A	1.546 (12)
C6—H6	0.9500	C23A—H23C	0.9900
C6—C7	1.380 (3)	C23A—H23D	0.9900
C7—H7	0.9500	C23A—C22A	1.534 (13)
C7—C8	1.392 (2)	C22A—H22C	0.9900
C8—C9	1.473 (2)	C22A—H22D	0.9900
C9—C10	1.388 (2)	C22A—C21A	1.533 (13)
C10—H10	0.9500	C21A—H21C	0.9900

C10—C11	1.379 (3)	C21A—H21D	0.9900
C11—H11	0.9500	O6B—C21B	1.4432
C11—C12	1.367 (3)	O6B—C24B	1.4435
C12—H12	0.9500	C21B—H21E	0.9900
C12—C13	1.392 (2)	C21B—H21F	0.9900
C13—C14	1.493 (2)	C21B—C22B	1.5350
C14—C15	1.393 (2)	C22B—H22E	0.9900
C14—C19	1.400 (2)	C22B—H22F	0.9900
C15—H15	0.9500	C22B—C23B	1.5390
C15—C16	1.386 (3)	C23B—H23E	0.9900
C16—H16	0.9500	C23B—H23F	0.9900
C16—C17	1.389 (3)	C23B—C24B	1.5350
C17—H17	0.9500	C24B—H24E	0.9900
C17—C18	1.380 (3)	C24B—H24F	0.9900
C18—H18	0.9500		
N1—Re1—C11	82.45 (4)	C21—O6—C24	109.5 (3)
N1—Re1—N2	75.48 (5)	O6—C21—H21A	111.6
N2—Re1—C11	82.11 (4)	O6—C21—H21B	111.6
C1—Re1—C11	94.36 (6)	O6—C21—C22	101.1 (3)
C1—Re1—N1	94.91 (8)	H21A—C21—H21B	109.4
C1—Re1—N2	170.08 (7)	C22—C21—H21A	111.6
C1—Re1—C2	85.75 (9)	C22—C21—H21B	111.6
C1—Re1—C3	89.41 (8)	C21—C22—H22A	111.1
C2—Re1—C11	93.41 (6)	C21—C22—H22B	111.1
C2—Re1—N1	175.84 (7)	C21—C22—C23	103.5 (3)
C2—Re1—N2	103.67 (7)	H22A—C22—H22B	109.0
C3—Re1—C11	174.86 (5)	C23—C22—H22A	111.1
C3—Re1—N1	93.76 (7)	C23—C22—H22B	111.1
C3—Re1—N2	93.61 (6)	C22—C23—H23A	110.7
C3—Re1—C2	90.36 (8)	C22—C23—H23B	110.7
C20—O5—H5	109 (2)	H23A—C23—H23B	108.8
C4—N1—Re1	124.05 (13)	C24—C23—C22	105.4 (3)
C8—N1—Re1	117.26 (10)	C24—C23—H23A	110.7
C8—N1—C4	118.58 (15)	C24—C23—H23B	110.7
C9—N2—Re1	114.86 (10)	O6—C24—C23	104.5 (4)
C13—N2—Re1	127.46 (10)	O6—C24—H24A	110.8
C13—N2—C9	117.63 (13)	O6—C24—H24B	110.8
O1—C1—Re1	178.1 (2)	C23—C24—H24A	110.8
O2—C2—Re1	174.45 (19)	C23—C24—H24B	110.8
O3—C3—Re1	178.65 (18)	H24A—C24—H24B	108.9
N1—C4—H4	118.7	C24A—O6A—C21A	108.4 (10)
N1—C4—C5	122.6 (2)	O6A—C24A—H24C	110.3
C5—C4—H4	118.7	O6A—C24A—H24D	110.3
C4—C5—H5A	120.6	O6A—C24A—C23A	106.9 (9)
C6—C5—C4	118.87 (18)	H24C—C24A—H24D	108.6
C6—C5—H5A	120.6	C23A—C24A—H24C	110.3
C5—C6—H6	120.5	C23A—C24A—H24D	110.3

C5—C6—C7	119.08 (17)	C24A—C23A—H23C	111.6
C7—C6—H6	120.5	C24A—C23A—H23D	111.6
C6—C7—H7	120.3	H23C—C23A—H23D	109.4
C6—C7—C8	119.45 (19)	C22A—C23A—C24A	100.9 (8)
C8—C7—H7	120.3	C22A—C23A—H23C	111.6
N1—C8—C7	121.38 (15)	C22A—C23A—H23D	111.6
N1—C8—C9	115.86 (13)	C23A—C22A—H22C	111.5
C7—C8—C9	122.76 (15)	C23A—C22A—H22D	111.5
N2—C9—C8	116.40 (13)	H22C—C22A—H22D	109.3
N2—C9—C10	122.22 (14)	C21A—C22A—C23A	101.5 (9)
C10—C9—C8	121.36 (14)	C21A—C22A—H22C	111.5
C9—C10—H10	120.4	C21A—C22A—H22D	111.5
C11—C10—C9	119.24 (16)	O6A—C21A—C22A	99.9 (11)
C11—C10—H10	120.4	O6A—C21A—H21C	111.8
C10—C11—H11	120.5	O6A—C21A—H21D	111.8
C12—C11—C10	118.92 (16)	C22A—C21A—H21C	111.8
C12—C11—H11	120.5	C22A—C21A—H21D	111.8
C11—C12—H12	120.0	H21C—C21A—H21D	109.5
C11—C12—C13	120.07 (16)	C21B—O6B—C24B	109.5
C13—C12—H12	120.0	O6B—C21B—H21E	110.4
N2—C13—C12	121.85 (15)	O6B—C21B—H21F	110.4
N2—C13—C14	121.29 (14)	O6B—C21B—C22B	106.4
C12—C13—C14	116.49 (14)	H21E—C21B—H21F	108.6
C15—C14—C13	117.06 (15)	C22B—C21B—H21E	110.4
C15—C14—C19	118.97 (16)	C22B—C21B—H21F	110.4
C19—C14—C13	123.40 (15)	C21B—C22B—H22E	111.5
C14—C15—H15	119.5	C21B—C22B—H22F	111.5
C16—C15—C14	121.01 (18)	C21B—C22B—C23B	101.5
C16—C15—H15	119.5	H22E—C22B—H22F	109.3
C15—C16—H16	120.1	C23B—C22B—H22E	111.5
C15—C16—C17	119.87 (19)	C23B—C22B—H22F	111.5
C17—C16—H16	120.1	C22B—C23B—H23E	111.5
C16—C17—H17	120.1	C22B—C23B—H23F	111.5
C18—C17—C16	119.80 (18)	H23E—C23B—H23F	109.3
C18—C17—H17	120.1	C24B—C23B—C22B	101.5
C17—C18—H18	119.6	C24B—C23B—H23E	111.5
C17—C18—C19	120.72 (17)	C24B—C23B—H23F	111.5
C19—C18—H18	119.6	O6B—C24B—C23B	106.4
C14—C19—C20	120.28 (14)	O6B—C24B—H24E	110.4
C18—C19—C14	119.60 (15)	O6B—C24B—H24F	110.4
C18—C19—C20	120.12 (15)	C23B—C24B—H24E	110.4
O4—C20—O5	124.18 (16)	C23B—C24B—H24F	110.4
O4—C20—C19	124.17 (15)	H24E—C24B—H24F	108.6
O5—C20—C19	111.64 (14)		
Re1—N1—C4—C5	175.99 (14)	C13—N2—C9—C10	0.4 (2)
Re1—N1—C8—C7	-174.60 (12)	C13—C14—C15—C16	-173.3 (2)
Re1—N1—C8—C9	4.26 (17)	C13—C14—C19—C18	171.61 (16)

Re1—N2—C9—C8	-0.54 (16)	C13—C14—C19—C20	-8.9 (2)
Re1—N2—C9—C10	177.80 (12)	C14—C15—C16—C17	1.5 (4)
Re1—N2—C13—C12	-175.22 (11)	C14—C19—C20—O4	-8.5 (2)
Re1—N2—C13—C14	-2.4 (2)	C14—C19—C20—O5	171.69 (15)
N1—C4—C5—C6	-1.1 (3)	C15—C14—C19—C18	0.5 (3)
N1—C8—C9—N2	-2.41 (19)	C15—C14—C19—C20	179.93 (18)
N1—C8—C9—C10	179.23 (14)	C15—C16—C17—C18	-0.2 (4)
N2—C9—C10—C11	-2.4 (2)	C16—C17—C18—C19	-0.9 (4)
N2—C13—C14—C15	-88.2 (2)	C17—C18—C19—C14	0.8 (3)
N2—C13—C14—C19	100.55 (19)	C17—C18—C19—C20	-178.70 (18)
C4—N1—C8—C7	1.7 (2)	C18—C19—C20—O4	170.91 (16)
C4—N1—C8—C9	-179.42 (14)	C18—C19—C20—O5	-8.9 (2)
C4—C5—C6—C7	0.6 (3)	C19—C14—C15—C16	-1.6 (4)
C5—C6—C7—C8	1.0 (3)	O6—C21—C22—C23	-37.5 (4)
C6—C7—C8—N1	-2.2 (2)	C21—O6—C24—C23	-24.1 (5)
C6—C7—C8—C9	179.01 (15)	C21—C22—C23—C24	24.2 (4)
C7—C8—C9—N2	176.43 (14)	C22—C23—C24—O6	-1.0 (5)
C7—C8—C9—C10	-1.9 (2)	C24—O6—C21—C22	39.3 (5)
C8—N1—C4—C5	-0.1 (3)	O6A—C24A—C23A—C22A	-18.3 (16)
C8—C9—C10—C11	175.90 (15)	C24A—O6A—C21A—C22A	36.3 (19)
C9—N2—C13—C12	1.8 (2)	C24A—C23A—C22A—C21A	39.9 (14)
C9—N2—C13—C14	174.62 (13)	C23A—C22A—C21A—O6A	-46.9 (16)
C9—C10—C11—C12	2.1 (3)	C21A—O6A—C24A—C23A	-11.4 (19)
C10—C11—C12—C13	0.0 (3)	O6B—C21B—C22B—C23B	31.5
C11—C12—C13—N2	-2.1 (2)	C21B—O6B—C24B—C23B	-12.4
C11—C12—C13—C14	-175.19 (16)	C21B—C22B—C23B—C24B	-37.4
C12—C13—C14—C15	85.0 (2)	C22B—C23B—C24B—O6B	31.5
C12—C13—C14—C19	-86.3 (2)	C24B—O6B—C21B—C22B	-12.3
C13—N2—C9—C8	-177.94 (13)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5 \cdots O6	0.90 (3)	1.74 (4)	2.615 (4)	164 (3)
O5—H5 \cdots O6A	0.90 (3)	1.68 (4)	2.516 (15)	154 (3)
O5—H5 \cdots O6B	0.90 (3)	1.83 (4)	2.642 (9)	150 (3)
C5—H5A \cdots C11 ⁱ	0.95	2.80	3.371 (2)	120
C10—H10 \cdots C11 ⁱⁱ	0.95	2.70	3.552 (2)	149
C12—H12 \cdots C11 ⁱⁱⁱ	0.95	2.76	3.524 (2)	138

Symmetry codes: (i) $x, -y+3/2, z+1/2$; (ii) $-x, -y+1, -z+1$; (iii) $-x, y-1/2, -z+1/2$.