research communications

The crystal structures of tetrakis(*μ-n*-butyrato-*κ²O:O′*)bis[bromidorhenium(III)] and tetrakis-(*μ-n*-butyrato-*κ²O:O′*)bis[chloridorhenium(III)] acetonitrile disolvate

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The title complexes, $[Re_2Br_2(O_2CC_3H_7)_4]$, (**1**), and $[Re_2(O_2CC_3H_7)_4Cl_2]\cdot 2CH_3CN$, (**2**), both exhibit paddlewheel structures with four carboxylate ligands bridging two Re^{III} atoms. The Re—Re distances are 2.2325 (2) and 2.2299 (3) Å, indicating quadruple bonds between the Re^{III} atoms in each complex. Both complexes contain an inversion center at the mid-point of the Re—Re bond. The Re—Br bond [2.6712 (3) Å] in (**1**) is 0.1656 (6) Å longer than the Re—Cl distance [2.5056 (5) Å] of (**2**). In (**2**), the N atom of each co-crystallized acetonitrile solvent molecule is nearly equidistant between and in close contact with two carboxylate C atoms.

1. Chemical context

The first compound discovered to contain a metal–metal quadruple bond was $K_2Re_2Cl_8\cdot 2H_2O$ (Cotton & Harris, 1965); since then numerous other quadruply bonded complexes have been isolated (Cotton *et al.*, 2005). Dirhenium quadruply bonded complexes are of interest due to their ability to act as molecular building blocks for the formation of molecular triangles and other multiple-metal arrays in which electronic coupling and delocalization between metal sites can be explored (Bera, Angaridis *et al.* 2001; Bera, Smucker *et al.*, 2001; Vega *et al.*, 2002). The title complexes are of the structural type classified as paddlewheel complexes, where the four carboxylate ligands bridge the two metal atoms, creating a paddlewheel appearance. A variety of these dirhenium(III) tetracarboxylate complexes were synthesized by Cotton *et al.* (1966) and in subsequent years the crystal structures of $[Re_2Cl_2(O_2CCH_3)_4]$, $[Re_2Cl_2(O_2CC_6H_5)_4]$ (Bennett *et al.*, 1968), $[Re_2(ReO_4)_2(O_2CC_3H_7)_4]$ (Calvo *et al.*, 1970), $[Re_2X_2\{O_2CC(CH_3)_3\}_4]$, where X = Cl or Br (Collins *et al.*, 1979), $[Re_2Cl_2(O_2CCH_3)_4]$ (Koz'min *et al.*, 1980), and $[Re_2Cl_2(O_2CC_3H_7)_4]$ (Thomson *et al.*, 2014) have been reported. For additional dirhenium tetracarboxylate structures, see: Shtemenko *et al.* (2001), Cotton *et al.* (1997), and Vega *et al.* (2002). This communication reports and compares the structures of $[Re_2Br_2(O_2CC_3H_7)_4]$, (**1**), and $[Re_2(O_2CC_3H_7)_4Cl_2]\cdot 2CH_3CN$, (**2**).

2. Structural commentary

Both of the title dirhenium metal complexes are located on crystallographic inversion centers that coincide with the midpoint of the Re—Re bonds. The short Re—Re bond

lengths of 2.2325 (2) and 2.2299 (3) Å, in (**1**) and (**2**), respectively, are indicative of quadruple bonds (Tables 1 and 2). The four butyrate groups bridge the two Re^{III} metal atoms in both cases, forming the anticipated paddlewheel structures (Figs. 1 and 2).


(1)

(2)
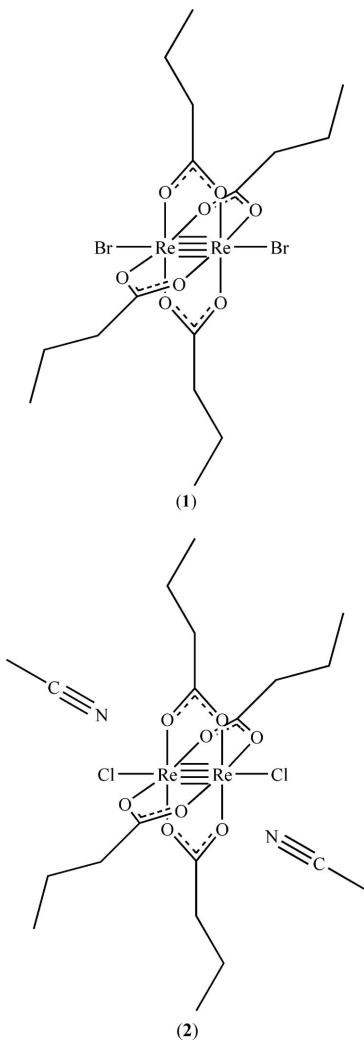
The asymmetric unit of (**2**) also contains one co-crystallized acetonitrile solvent molecule in a general position, thus giving rise to twice that in the formula unit.

The $X-Re-Re-X$ bonds in (**1**) and (**2**) are nearly linear, as can be seen in the $Re-Re-Br$ [175.018 (7)°] and $Re-Re-Cl$ [178.254 (11)°] bond angles, and are comparable to those observed in similar compounds (Collins *et al.*, 1979; Thomson *et al.*, 2014). The $Re-Cl$ bond length [2.5056 (5) Å] of (**2**) is similar to those of the previously published analog without co-crystallized acetonitrile (Thomson *et al.*, 2014), $[Re_2Cl_2(O_2CC(CH_3)_3)_4]$, and $[Re_2Cl_2(O_2CC_6H_5)_4]\cdot2CHCl_3$ (Bennett *et al.*, 1968; Collins *et al.*, 1979). The $Re-Br$ bond length [2.6712 (3) Å] of (**1**) is slightly longer than the $Re-Br$ bond [2.603 (1) Å] found in $[Re_2Br_2(O_2CC(CH_3)_3)_4]$ (Collins *et al.*, 1979). The $Re-Br$ and $Re-Cl$ distances of (**1**) and (**2**) differ by 0.1656 (6) Å and those of Cotton and coworkers differ by 0.126 (3), both of which are consistent with the difference in covalent radii of Cl and Br (0.15 Å).
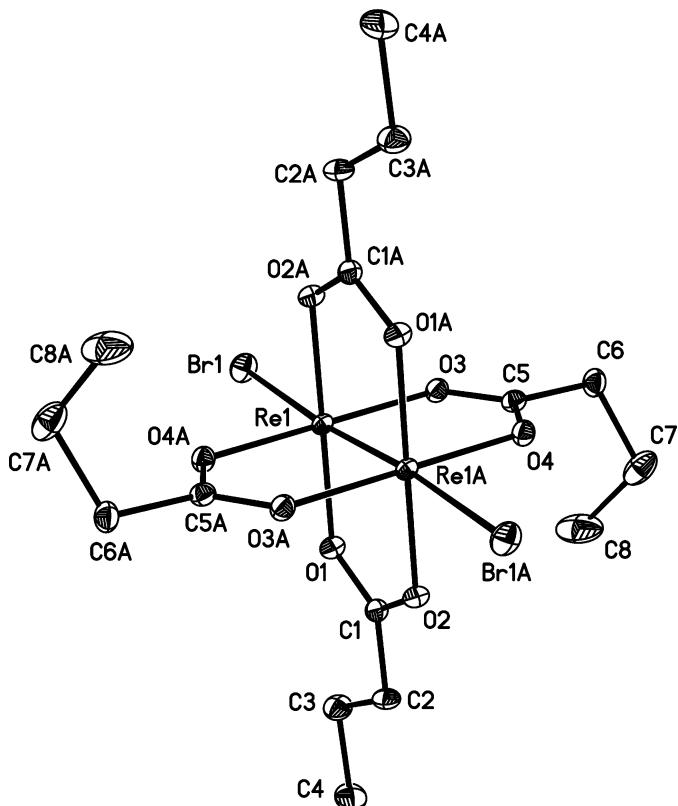
Figure 1
The molecular structure of title compound (**1**), with displacement ellipsoids drawn at the 50% probability level. The symmetry-equivalent half is generated by operator $(-x + 1, -y + 1, -z + 1)$.
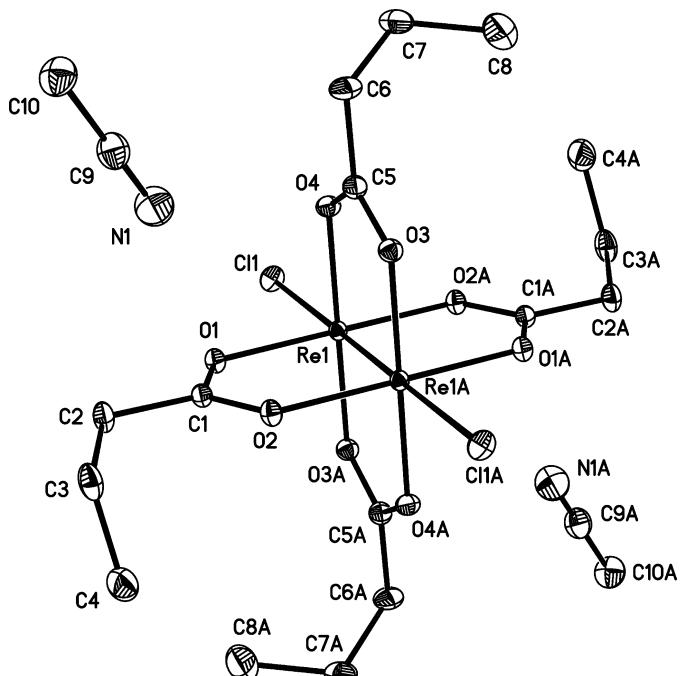

Figure 2
The molecular structure of title compound (**2**), with displacement ellipsoids drawn at the 50% probability level. The symmetry-equivalent half is generated by operator $(-x, -y, -z + 1)$.

Table 1
Selected geometric parameters (Å, °) for (**1**).

Re1—O4[i]	2.0102 (15)	Re1—O3	2.0295 (14)
Re1—O2[i]	2.0159 (14)	Re1—Re1[i]	2.2325 (2)
Re1—O1	2.0225 (15)	Re1—Br1	2.6712 (3)
O4[i]—Re1—O2[i]	89.22 (6)	O1—Re1—O3	89.64 (6)
O4[i]—Re1—O1	90.42 (6)	Re1[i]—Re1—Br1	175.018 (7)
O2[i]—Re1—O3	90.72 (6)		
C1—C2—C3—C4	−177.54 (19)	C5—C6—C7—C8	56.0 (3)

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

Table 2
Selected geometric parameters (Å, °) for (**2**).

Re1—O1	2.0216 (12)	Re1—O4	2.0255 (12)
Re1—O2[i]	2.0217 (12)	Re1—Re1[i]	2.2299 (3)
Re1—O3[i]	2.0238 (12)	Re1—Cl1	2.5056 (5)
O1—Re1—O3[i]	89.75 (5)	O2[i]—Re1—O4	89.75 (5)
O2[i]—Re1—O3[i]	90.13 (5)	Re1[i]—Re1—Cl1	178.254 (11)
O1—Re1—O4	90.37 (5)		
C1—C2—C3—C4	−70.2 (2)	C5—C6—C7—C8	−67.9 (2)

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

The structure of (**1**) is isotypic with the chlorido analog published by Thomson *et al.* (2014). Inspection of the torsion angles of the hydrocarbon chains reveals the possible effect of the co-crystallization of solvent in $[Re_2Cl_2(O_2CC_3H_7)_4]$. In compound (**2**), the C1—C2—C3—C4 torsion angle is −70.2 (2)°, comparable to −67.9 (2)° for C5—C6—C7—C8 (Fig. 1). In the structure of $[Re_2Cl_2(O_2CC_3H_7)_4]$ without co-crystallizing solvent, the torsion angles vary more [C1—C2—C3—C4 = −55.2 (5) and C5—C6—C7—C8, 179.5 (4)°]
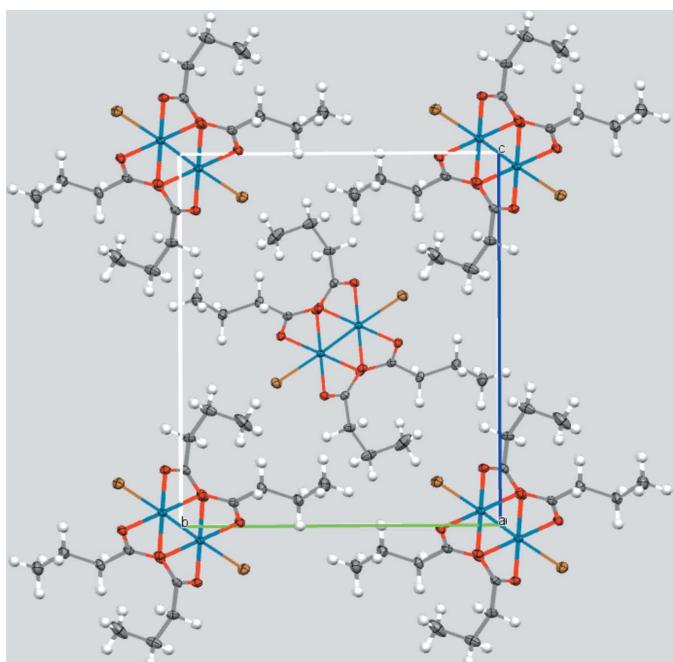
Figure 3
The packing arrangements of (**1**).
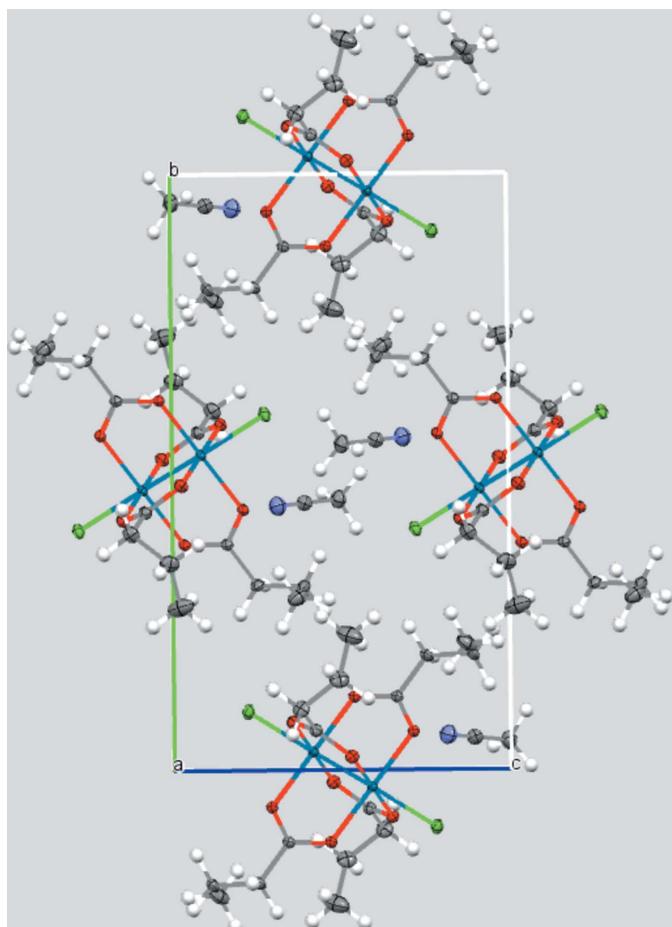
Figure 4
The packing arrangements of (**2**).

(Thomson *et al.*, 2014), similar to those observed in (**1**) (Table 1).

3. Supramolecular features

Packing arrangements are shown in Figs. 3 and 4. In (**2**) nitrogen atom N1 of the co-crystallized acetonitrile solvent molecule is located at distances of 3.197 (3) and 3.216 (3) Å from the carboxylate carbon atoms C1 and C5, respectively. This is just within the sum of the van der Waals radii of 3.25 Å (Bondi, 1964), and suggests the presence of a weak electrostatic interaction between the solvent and dirhenium species.

4. Database survey

There are 145 structures in the Cambridge Structural Database to date (CSD, Version 5.36, update No. 3, May 2015; Groom & Allen, 2014) that have explicitly defined Re—Re quadruple bonds. However, this appears to be an inconsistent denotation, as many other structures that contain quadruple bonds are not presented as such. For instance only six of the eleven carboxylate paddlewheel complexes in the CSD (to date) have their Re—Re bonds defined as quadruple. Thus a better way to search appears to be by bond length. There are

Table 3
Experimental details.

	(1)	(2)
Crystal data		
Chemical formula	$[Re_2Br_2(C_4H_7O_2)_4]$	$[Re_2(C_4H_7O_2)_4Cl_2]\cdot2C_2H_3N$
M_r	880.60	873.79
Crystal system, space group	Monoclinic, $P2_1/n$	Monoclinic, $P2_1/c$
Temperature (K)	100	100
a, b, c (Å)	6.6833 (5), 12.2817 (10), 14.6134 (12)	8.5589 (13), 17.097 (3), 10.0494 (15)
β (°)	100.5380 (16)	105.830 (3)
V (Å^3)	1179.27 (16)	1414.8 (4)
Z	2	2
Radiation type	Mo $K\alpha$	Mo $K\alpha$
μ (mm^{-1})	13.68	8.78
Crystal size (mm)	$0.36 \times 0.16 \times 0.12$	$0.36 \times 0.34 \times 0.12$
Data collection		
Diffractometer	Bruker SMART APEXII CCD platform	Bruker *SMART* APEXII CCD platform
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2014)	Multi-scan (*SADABS*; Sheldrick, 2014)
T_{min}, T_{max}	0.161, 0.440	0.187, 0.440
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	43018, 6464, 5503	51215, 7730, 6874
R_{int}	0.039	0.038
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.875	0.876
Refinement		
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.021, 0.047, 1.04	0.021, 0.040, 1.15
No. of reflections	6464	7730
No. of parameters	129	157
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å^{-3})	2.11, −1.56	1.42, −1.61

Computer programs: *APEX2* and *SAINT* (Bruker, 2014), *SIR2011* (Burla *et al.*, 2012), *SHELXL2014* (Sheldrick, 2015) and *SHELXTL* (Sheldrick, 2008).

298 entries with Re—Re bond lengths $\leq$ 2.29 Å. The only examples of defined quadruple bonds greater than this (excluding obviously disordered structures) are two dirhenium structures with bridging hydride ligands (CSD refcodes BIBLED and BIBLIH; Green *et al.*, 1982) and two with bridging di-*p*-tolylformamidine ligands (CSD refcodes KOZFUA and KOZGEL; Cotton & Ren, 1992).

5. Synthesis and crystallization

The title compounds were previously synthesized *via* microwave irradiation and fully characterized by elemental analysis and UV–Vis and IR spectroscopies (Reed *et al.*, 2015). For crystallization each compound was dissolved in acetonitrile and a few drops of diethyl ether were added to the acetonitrile solution which produced seed crystals. Slow evaporation of the solvent at room temperature in a glovebox produced single crystals suitable for X-ray diffraction.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. H atoms were placed geometrically and treated as riding atoms: methylene, C—H = 0.99 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$ and methyl, C—H = 0.98 Å, with $U_{iso}(H) = 1.5U_{eq}(C)$.

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supporting information

The crystal structures of tetrakis(*μ-n*-butyrato-*κ²O:O′*)bis[bromidorhenium(III)] and tetrakis(*μ-n*-butyrato-*κ²O:O′*)bis[chloridorhenium(III)] acetonitrile disolvate

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Computing details

For both compounds, data collection: *APEX2* (Bruker, 2014); cell refinement: *SAINT* (Bruker, 2014); data reduction: *SAINT* (Bruker, 2014); program(s) used to solve structure: *SIR2011* (Burla *et al.*, 2012); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

(1) Tetrakis(*μ-n*-butyrato-*κ²O:O′*)bis[bromidorhenium(III)]

Crystal data

[Re$_2$Br$_2$(C$_4$H$_7$O$_2$)$_4$]	$F(000) = 816$
$M_r = 880.60$	$D_x = 2.480$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 6.6833$ (5) Å	Cell parameters from 3849 reflections
$b = 12.2817$ (10) Å	$\theta = 3.3$–38.2°
$c = 14.6134$ (12) Å	$\mu = 13.68$ mm^{-1}
$\beta = 100.5380$ (16)°	$T = 100$ K
$V = 1179.27$ (16) Å^3	Needle, orange
$Z = 2$	$0.36 \times 0.16 \times 0.12$ mm

Data collection

Bruker SMART APEXII CCD platform diffractometer	6464 independent reflections
	5503 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.039$
ω scans	$\theta_{max} = 38.5°$, $\theta_{min} = 2.2°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2014)	$h = -11 \rightarrow 11$
	$k = -21 \rightarrow 21$
$T_{min} = 0.161$, $T_{max} = 0.440$	$l = -25 \rightarrow 25$
43018 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.021$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.047$	
$S = 1.04$	H-atom parameters constrained
6464 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0202P)^2 + 0.9723P]$ where $P = (F_o^2 + 2F_c^2)/3$
129 parameters	
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 2.11$ e Å^{-3}
	$\Delta\rho_{min} = -1.55$ e Å^{-3}

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 ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
Re1	0.41766 (2)	0.44073 (2)	0.53764 (2)	0.00983 (2)
Br1	0.19163 (3)	0.30257 (2)	0.61673 (2)	0.01786 (4)
O1	0.5665 (2)	0.31578 (12)	0.48971 (10)	0.0133 (2)
O2	0.7293 (2)	0.43464 (12)	0.41390 (10)	0.0128 (2)
O3	0.1966 (2)	0.43002 (12)	0.42259 (10)	0.0130 (2)
O4	0.3633 (2)	0.54841 (12)	0.34843 (10)	0.0131 (2)
C1	0.6954 (3)	0.33636 (16)	0.43620 (13)	0.0122 (3)
C2	0.8048 (3)	0.24669 (16)	0.39774 (14)	0.0146 (3)
H2A	0.9528	0.2611	0.4142	0.018*
H2B	0.7672	0.2486	0.3290	0.018*
C3	0.7644 (4)	0.13262 (17)	0.43061 (16)	0.0182 (4)
H3A	0.6184	0.1147	0.4112	0.022*
H3B	0.7982	0.1293	0.4994	0.022*
C4	0.8936 (4)	0.05015 (19)	0.38890 (18)	0.0239 (5)
H4A	0.8633	−0.0234	0.4084	0.036*
H4B	1.0381	0.0661	0.4107	0.036*
H4C	0.8622	0.0549	0.3208	0.036*
C5	0.2113 (3)	0.48493 (16)	0.34960 (13)	0.0124 (3)
C6	0.0567 (3)	0.47160 (19)	0.26301 (14)	0.0165 (4)
H6A	0.0077	0.5441	0.2391	0.020*
H6B	−0.0611	0.4302	0.2771	0.020*
C7	0.1485 (4)	0.4111 (2)	0.18869 (17)	0.0251 (5)
H7A	0.0414	0.3999	0.1330	0.030*
H7B	0.2565	0.4566	0.1701	0.030*
C8	0.2376 (4)	0.3019 (3)	0.2226 (2)	0.0373 (7)
H8A	0.2849	0.2640	0.1715	0.056*
H8B	0.1331	0.2579	0.2443	0.056*
H8C	0.3524	0.3130	0.2740	0.056*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.01032 (3)	0.01015 (3)	0.00965 (3)	−0.00067 (2)	0.00351 (2)	−0.00002 (2)
Br1	0.01861 (9)	0.01864 (9)	0.01763 (9)	−0.00465 (7)	0.00674 (7)	0.00246 (7)
O1	0.0140 (6)	0.0128 (6)	0.0137 (6)	0.0002 (5)	0.0045 (5)	−0.0012 (5)
O2	0.0124 (6)	0.0132 (6)	0.0139 (6)	0.0008 (5)	0.0050 (5)	−0.0011 (5)
O3	0.0125 (6)	0.0153 (6)	0.0115 (6)	−0.0009 (5)	0.0029 (4)	−0.0002 (5)
O4	0.0140 (6)	0.0141 (6)	0.0115 (6)	−0.0006 (5)	0.0028 (5)	0.0014 (5)
C1	0.0113 (7)	0.0139 (8)	0.0112 (7)	0.0004 (6)	0.0018 (6)	−0.0007 (6)

C2	0.0143 (8)	0.0129 (8)	0.0174 (8)	0.0029 (6)	0.0049 (7)	−0.0030 (6)
C3	0.0219 (10)	0.0139 (8)	0.0198 (9)	0.0030 (7)	0.0060 (7)	−0.0017 (7)
C4	0.0274 (12)	0.0185 (10)	0.0258 (11)	0.0081 (8)	0.0048 (9)	−0.0023 (8)
C5	0.0122 (8)	0.0126 (8)	0.0125 (7)	0.0002 (6)	0.0030 (6)	−0.0020 (6)
C6	0.0162 (9)	0.0210 (9)	0.0114 (8)	−0.0008 (7)	0.0002 (6)	−0.0004 (7)
C7	0.0222 (11)	0.0361 (13)	0.0183 (9)	−0.0110 (10)	0.0068 (8)	−0.0097 (9)
C8	0.0262 (13)	0.0418 (17)	0.0430 (16)	0.0033 (12)	0.0035 (11)	−0.0264 (13)

Geometric parameters (Å, º)

Re1—O4^i	2.0102 (15)	C3—C4	1.528 (3)
Re1—O2^i	2.0159 (14)	C3—H3A	0.9900
Re1—O1	2.0225 (15)	C3—H3B	0.9900
Re1—O3	2.0295 (14)	C4—H4A	0.9800
Re1—Re1^i	2.2325 (2)	C4—H4B	0.9800
Re1—Br1	2.6712 (3)	C4—H4C	0.9800
O1—C1	1.290 (2)	C5—C6	1.489 (3)
O2—C1	1.281 (2)	C6—C7	1.533 (3)
O2—Re1^i	2.0159 (14)	C6—H6A	0.9900
O3—C5	1.281 (2)	C6—H6B	0.9900
O4—C5	1.283 (2)	C7—C8	1.514 (4)
O4—Re1^i	2.0102 (14)	C7—H7A	0.9900
C1—C2	1.488 (3)	C7—H7B	0.9900
C2—C3	1.521 (3)	C8—H8A	0.9800
C2—H2A	0.9900	C8—H8B	0.9800
C2—H2B	0.9900	C8—H8C	0.9800
O4^i—Re1—O2^i	89.22 (6)	C2—C3—H3B	109.7
O4^i—Re1—O1	90.42 (6)	C4—C3—H3B	109.7
O2^i—Re1—O1	179.64 (6)	H3A—C3—H3B	108.2
O4^i—Re1—O3	179.91 (6)	C3—C4—H4A	109.5
O2^i—Re1—O3	90.72 (6)	C3—C4—H4B	109.5
O1—Re1—O3	89.64 (6)	H4A—C4—H4B	109.5
O4^i—Re1—Re1^i	90.86 (4)	C3—C4—H4C	109.5
O2^i—Re1—Re1^i	89.64 (4)	H4A—C4—H4C	109.5
O1—Re1—Re1^i	90.35 (4)	H4B—C4—H4C	109.5
O3—Re1—Re1^i	89.08 (4)	O3—C5—O4	120.85 (18)
O4^i—Re1—Br1	93.87 (4)	O3—C5—C6	120.16 (18)
O2^i—Re1—Br1	88.85 (4)	O4—C5—C6	118.92 (18)
O1—Re1—Br1	91.19 (4)	C5—C6—C7	110.48 (19)
O3—Re1—Br1	86.19 (4)	C5—C6—H6A	109.6
Re1^i—Re1—Br1	175.018 (7)	C7—C6—H6A	109.6
C1—O1—Re1	119.12 (13)	C5—C6—H6B	109.6
C1—O2—Re1^i	120.39 (13)	C7—C6—H6B	109.6
C5—O3—Re1	120.03 (13)	H6A—C6—H6B	108.1
C5—O4—Re1^i	119.18 (13)	C8—C7—C6	112.4 (2)
O2—C1—O1	120.51 (18)	C8—C7—H7A	109.1
O2—C1—C2	118.63 (18)	C6—C7—H7A	109.1

O1—C1—C2	120.85 (18)	C8—C7—H7B	109.1
C1—C2—C3	115.75 (18)	C6—C7—H7B	109.1
C1—C2—H2A	108.3	H7A—C7—H7B	107.8
C3—C2—H2A	108.3	C7—C8—H8A	109.5
C1—C2—H2B	108.3	C7—C8—H8B	109.5
C3—C2—H2B	108.3	H8A—C8—H8B	109.5
H2A—C2—H2B	107.4	C7—C8—H8C	109.5
C2—C3—C4	109.79 (19)	H8A—C8—H8C	109.5
C2—C3—H3A	109.7	H8B—C8—H8C	109.5
C4—C3—H3A	109.7		
Re1^i—O2—C1—O1	0.2 (2)	Re1—O3—C5—O4	−1.1 (3)
Re1^i—O2—C1—C2	179.10 (13)	Re1—O3—C5—C6	175.86 (14)
Re1—O1—C1—O2	0.2 (2)	Re1^i—O4—C5—O3	1.0 (3)
Re1—O1—C1—C2	−178.66 (14)	Re1^i—O4—C5—C6	−175.98 (14)
O2—C1—C2—C3	177.34 (18)	O3—C5—C6—C7	−108.8 (2)
O1—C1—C2—C3	−3.7 (3)	O4—C5—C6—C7	68.2 (3)
C1—C2—C3—C4	−177.54 (19)	C5—C6—C7—C8	56.0 (3)

Symmetry code: (i) −x+1, −y+1, −z+1.

(2) Tetrakis(μ-n-butyrato-$\kappa^2 O$:O')bis[chloridorhenium(III)]

Crystal data
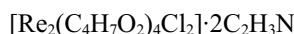
[Re$_2$(C$_4$H$_7$O$_2$)$_4$Cl$_2$]·2C$_2$H$_3$N	$F(000) = 832$
$M_r = 873.79$	$D_x = 2.051$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 8.5589$ (13) Å	Cell parameters from 3912 reflections
$b = 17.097$ (3) Å	$\theta = 2.4$–38.3°
$c = 10.0494$ (15) Å	$\mu = 8.78$ mm^{-1}
$\beta = 105.830$ (3)°	$T = 100$ K
$V = 1414.8$ (4) Å^3	Plate, orange
$Z = 2$	$0.36 \times 0.34 \times 0.12$ mm

Data collection

Bruker SMART APEXII CCD platform diffractometer	7730 independent reflections
	6874 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.038$
ω scans	$\theta_{max} = 38.5°$, $\theta_{min} = 2.4°$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2014)	$h = -14 \rightarrow 14$
	$k = -29 \rightarrow 29$
$T_{min} = 0.187$, $T_{max} = 0.440$	$l = -17 \rightarrow 17$
51215 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.021$	Secondary atom site location: difference Fourier map
$wR(F^2) = 0.040$	
$S = 1.15$	Hydrogen site location: inferred from neighbouring sites
7730 reflections	
157 parameters	H-atom parameters constrained
0 restraints	

$w = 1/[\sigma^2(F_o^2) + (0.0071P)^2 + 1.4405P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 1.42$ e Å^{-3}
$\Delta\rho_{min} = -1.61$ e Å^{-3}

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 (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$
Re1	0.03406 (2)	0.02939 (2)	0.41289 (2)	0.00845 (2)
Cl1	0.11816 (5)	0.09708 (2)	0.22230 (4)	0.01465 (7)
O1	0.11825 (15)	0.12189 (7)	0.53706 (12)	0.0114 (2)
O2	0.05072 (15)	0.06310 (7)	0.71113 (12)	0.0116 (2)
O3	0.18769 (15)	−0.08019 (7)	0.64715 (13)	0.0119 (2)
O4	0.25576 (15)	−0.02169 (7)	0.47284 (13)	0.0125 (2)
C1	0.1121 (2)	0.12144 (9)	0.66290 (17)	0.0110 (3)
C2	0.1786 (2)	0.18996 (10)	0.75268 (19)	0.0155 (3)
H2A	0.1391	0.2384	0.7004	0.019*
H2B	0.2984	0.1894	0.7726	0.019*
C3	0.1342 (3)	0.19286 (11)	0.88930 (19)	0.0190 (3)
H3A	0.1606	0.1419	0.9367	0.023*
H3B	0.2011	0.2333	0.9491	0.023*
C4	−0.0440 (3)	0.21089 (13)	0.8716 (3)	0.0272 (4)
H4A	−0.0650	0.2134	0.9626	0.041*
H4B	−0.1109	0.1697	0.8162	0.041*
H4C	−0.0711	0.2613	0.8244	0.041*
C5	0.2915 (2)	−0.06622 (10)	0.57910 (18)	0.0128 (3)
C6	0.4563 (2)	−0.10219 (12)	0.6230 (2)	0.0185 (3)
H6A	0.4641	−0.1352	0.7055	0.022*
H6B	0.5385	−0.0602	0.6497	0.022*
C7	0.4947 (2)	−0.15213 (12)	0.5097 (2)	0.0226 (4)
H7A	0.4768	−0.1204	0.4245	0.027*
H7B	0.6109	−0.1670	0.5391	0.027*
C8	0.3925 (3)	−0.22572 (15)	0.4773 (3)	0.0392 (6)
H8A	0.4236	−0.2555	0.4053	0.059*
H8B	0.2775	−0.2114	0.4447	0.059*
H8C	0.4104	−0.2577	0.5610	0.059*
N1	0.4670 (3)	0.05726 (13)	0.8138 (2)	0.0299 (4)
C9	0.5906 (3)	0.05264 (13)	0.8933 (2)	0.0221 (4)
C10	0.7485 (3)	0.04674 (14)	0.9950 (2)	0.0255 (4)
H10A	0.7727	0.0959	1.0466	0.038*
H10B	0.8324	0.0365	0.9475	0.038*
H10C	0.7465	0.0038	1.0591	0.038*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.00949 (2)	0.00778 (2)	0.00844 (2)	−0.00046 (2)	0.00305 (2)	0.00032 (2)
Cl1	0.01616 (16)	0.01644 (16)	0.01207 (15)	−0.00189 (13)	0.00506 (13)	0.00287 (13)
O1	0.0142 (5)	0.0097 (5)	0.0105 (5)	−0.0014 (4)	0.0037 (4)	−0.0004 (4)
O2	0.0149 (5)	0.0103 (5)	0.0097 (5)	−0.0016 (4)	0.0035 (4)	−0.0003 (4)
O3	0.0118 (5)	0.0115 (5)	0.0122 (5)	0.0009 (4)	0.0032 (4)	0.0012 (4)
O4	0.0112 (5)	0.0129 (5)	0.0140 (5)	0.0013 (4)	0.0048 (4)	0.0016 (4)
C1	0.0118 (6)	0.0094 (6)	0.0117 (6)	−0.0007 (5)	0.0031 (5)	−0.0006 (5)
C2	0.0198 (8)	0.0115 (6)	0.0151 (7)	−0.0042 (6)	0.0049 (6)	−0.0036 (6)
C3	0.0290 (9)	0.0148 (7)	0.0136 (7)	−0.0054 (7)	0.0067 (7)	−0.0046 (6)
C4	0.0316 (11)	0.0234 (9)	0.0325 (11)	−0.0057 (8)	0.0185 (9)	−0.0097 (8)
C5	0.0118 (6)	0.0123 (6)	0.0136 (7)	0.0008 (5)	0.0022 (5)	0.0002 (5)
C6	0.0114 (7)	0.0213 (8)	0.0217 (8)	0.0047 (6)	0.0027 (6)	0.0018 (7)
C7	0.0172 (8)	0.0225 (9)	0.0296 (10)	0.0056 (7)	0.0092 (7)	0.0004 (8)
C8	0.0315 (12)	0.0256 (11)	0.0572 (18)	0.0014 (9)	0.0066 (12)	−0.0116 (11)
N1	0.0290 (10)	0.0304 (10)	0.0270 (9)	0.0018 (8)	0.0018 (8)	0.0006 (8)
C9	0.0257 (9)	0.0199 (8)	0.0200 (8)	−0.0010 (7)	0.0050 (7)	0.0002 (7)
C10	0.0235 (9)	0.0288 (10)	0.0210 (9)	−0.0006 (8)	0.0005 (8)	0.0001 (8)

Geometric parameters (Å, º)

Re1—O1	2.0216 (12)	C4—H4A	0.9800
Re1—O2i	2.0217 (12)	C4—H4B	0.9800
Re1—O3i	2.0238 (12)	C4—H4C	0.9800
Re1—O4	2.0255 (12)	C5—C6	1.490 (2)
Re1—Re1i	2.2299 (3)	C6—C7	1.529 (3)
Re1—Cl1	2.5056 (5)	C6—H6A	0.9900
O1—C1	1.280 (2)	C6—H6B	0.9900
O2—C1	1.282 (2)	C7—C8	1.516 (3)
O2—Re1i	2.0217 (12)	C7—H7A	0.9900
O3—C5	1.283 (2)	C7—H7B	0.9900
O3—Re1i	2.0238 (12)	C8—H8A	0.9800
O4—C5	1.279 (2)	C8—H8B	0.9800
C1—C2	1.493 (2)	C8—H8C	0.9800
C2—C3	1.522 (3)	N1—C9	1.141 (3)
C2—H2A	0.9900	C9—C10	1.459 (3)
C2—H2B	0.9900	C10—H10A	0.9800
C3—C4	1.518 (3)	C10—H10B	0.9800
C3—H3A	0.9900	C10—H10C	0.9800
C3—H3B	0.9900		
O1—Re1—O2i	179.84 (5)	C3—C4—H4A	109.5
O1—Re1—O3i	89.75 (5)	C3—C4—H4B	109.5
O2i—Re1—O3i	90.13 (5)	H4A—C4—H4B	109.5
O1—Re1—O4	90.37 (5)	C3—C4—H4C	109.5
O2i—Re1—O4	89.75 (5)	H4A—C4—H4C	109.5

O3^i—Re1—O4	179.87 (5)	H4B—C4—H4C	109.5
O1—Re1—Re1^i	89.63 (4)	O4—C5—O3	120.86 (15)
O2^i—Re1—Re1^i	90.27 (4)	O4—C5—C6	118.98 (16)
O3^i—Re1—Re1^i	90.15 (4)	O3—C5—C6	120.16 (16)
O4—Re1—Re1^i	89.80 (4)	C5—C6—C7	112.87 (16)
O1—Re1—Cl1	88.98 (4)	C5—C6—H6A	109.0
O2^i—Re1—Cl1	91.13 (4)	C7—C6—H6A	109.0
O3^i—Re1—Cl1	90.89 (4)	C5—C6—H6B	109.0
O4—Re1—Cl1	89.16 (4)	C7—C6—H6B	109.0
Re1^i—Re1—Cl1	178.254 (11)	H6A—C6—H6B	107.8
C1—O1—Re1	120.09 (10)	C8—C7—C6	113.24 (19)
C1—O2—Re1^i	119.38 (11)	C8—C7—H7A	108.9
C5—O3—Re1^i	119.40 (11)	C6—C7—H7A	108.9
C5—O4—Re1	119.78 (11)	C8—C7—H7B	108.9
O1—C1—O2	120.63 (15)	C6—C7—H7B	108.9
O1—C1—C2	118.72 (15)	H7A—C7—H7B	107.7
O2—C1—C2	120.64 (15)	C7—C8—H8A	109.5
C1—C2—C3	115.01 (15)	C7—C8—H8B	109.5
C1—C2—H2A	108.5	H8A—C8—H8B	109.5
C3—C2—H2A	108.5	C7—C8—H8C	109.5
C1—C2—H2B	108.5	H8A—C8—H8C	109.5
C3—C2—H2B	108.5	H8B—C8—H8C	109.5
H2A—C2—H2B	107.5	N1—C9—C10	180.0 (3)
C4—C3—C2	113.00 (17)	C9—C10—H10A	109.5
C4—C3—H3A	109.0	C9—C10—H10B	109.5
C2—C3—H3A	109.0	H10A—C10—H10B	109.5
C4—C3—H3B	109.0	C9—C10—H10C	109.5
C2—C3—H3B	109.0	H10A—C10—H10C	109.5
H3A—C3—H3B	107.8	H10B—C10—H10C	109.5
Re1—O1—C1—O2	−1.0 (2)	Re1—O4—C5—O3	1.2 (2)
Re1—O1—C1—C2	178.70 (12)	Re1—O4—C5—C6	−178.97 (12)
Re1^i—O2—C1—O1	0.8 (2)	Re1^i—O3—C5—O4	−1.1 (2)
Re1^i—O2—C1—C2	−178.84 (12)	Re1^i—O3—C5—C6	179.11 (12)
O1—C1—C2—C3	167.38 (16)	O4—C5—C6—C7	−58.1 (2)
O2—C1—C2—C3	−12.9 (2)	O3—C5—C6—C7	121.71 (19)
C1—C2—C3—C4	−70.2 (2)	C5—C6—C7—C8	−67.9 (2)

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