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Aquatricarbonyl(3,5,7-tribromotropolonato)rhenium(I) methanol solvate

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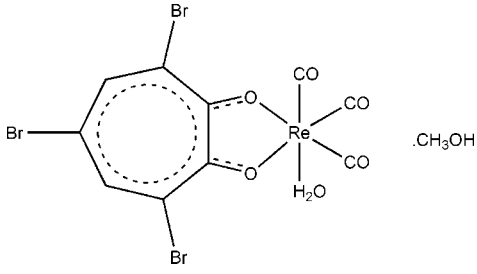
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.009 Å; R factor = 0.033; wR factor = 0.079; data-to-parameter ratio = 17.4.

The title complex, $[Re(C_7H_2Br_3O_2)(CO)_3(H_2O)]\cdot CH_3OH$, crystallized as a neutral Re^I compound and one methanol solvent molecule in the asymmetric unit. The metal centre is coordinated facially by three carbonyl groups. The bidentate tribromotropolanate ligand and a water molecule complete the distorted octahedral coordination around the central metal. Intermolecular Br···O [3.226 (5) Å] and Br···Br [3.590 (2) Å] contacts are observed between adjacent molecules. These contacts, together with an array of O—H···O, O—H···Br and C—H···O hydrogen bonds, complete a three-dimensional polymeric network formed between the methanol solvent and the complex.

Related literature

For a smiliar tribromotropolonato Re^I structure, see: Schutte *et al.* (2007). For other related structures, see: Kemp (2006); Roodt *et al.* (2003); Wang *et al.* (2003); Alvarez *et al.* (2007); Brasey *et al.* (2004); Gibson *et al.* (1999); Bochkova *et al.* (1987); Cheng *et al.* (1988); Mundwiler *et al.* (2004). For the synthesis of the precursor, see: Alberto *et al.* (1996). For synthesis of the tribromotropolone ligand, see: Steyl & Roodt (2006).



Experimental

Crystal data

$[Re(C_7H_2Br_3O_2)(CO)_3(H_2O)]\cdot CH_4O$
M_r = 678.1
Triclinic, $P\overline{1}$
a = 9.090 (5) Å
b = 9.379 (5) Å
c = 10.010 (5) Å
α = 109.569 (5)°
β = 94.285 (5)°
γ = 102.133 (5)°
V = 776.3 (7) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 15.58 mm^{-1}
T = 100 (2) K
0.19 × 0.06 × 0.03 mm

Data collection

Bruker APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
T_{min} = 0.150, T_{max} = 0.626
8673 measured reflections
3599 independent reflections
3018 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.079
S = 1.05
3599 reflections
207 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 2.40 e Å^{-3}
$\Delta\rho_{min}$ = −2.11 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Re01—C1	1.882 (7)	Re01—O4	2.123 (5)
Re01—C3	1.897 (6)	Re01—O5	2.146 (4)
Re01—C2	1.899 (7)	Re01—O6	2.170 (5)
O4—Re01—O5	74.07 (16)	O5—Re01—O6	79.17 (18)
O4—Re01—O6	78.93 (19)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O6—H6B···Br1^{i}	1.06 (8)	2.68 (8)	3.421 (6)	127 (5)
O6—H6B···O5^{i}	1.06 (8)	1.86 (8)	2.825 (7)	149 (6)
C15—H15···O2^{ii}	0.93	2.5	3.409 (8)	166
O7—H7···O1^{iii}	0.82	2.39	2.986 (7)	130
O6—H6A···O7	0.99 (8)	1.69 (8)	2.665 (7)	167 (7)

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

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

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: KJ2103).

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supporting information

Acta Cryst. (2008). E**64**, m1610–m1611 [doi:10.1107/S1600536808038737]

Aquatricarbonyl(3,5,7-tribromotropolonato)rhenium(I) methanol solvate

Marietjie Schutte, Hendrik G. Visser and Andreas Roodt

S1. Comment

This structure forms part of an ongoing investigation of the structural and kinetic behaviour of *fac*-$Re(CO)_3$ compounds (Schutte *et al.*, 2007; Roodt *et al.*, 2003). The title complex crystallized as a neutral Re^{I} compound and one methanol solvate molecule in the assymetric unit. The Re—CO bond distances are well within the normal range. The Re—O bond distances compare well with the analogous bromido complex (Schutte *et al.*, 2007) and other related structures (Alvarez *et al.*, 2007; Brasey *et al.*, 2004; Gibson *et al.*, 1999; Bochkova *et al.*, 1987; Cheng *et al.*, 1988; Wang *et al.*, 2003). The Re—OH_2 distance is also comparable to that of related structures (Mundwiler *et al.*, 2004; Kemp, 2006). The small bite angle O4—Re01—O5 might be the reason for the slightly distorted octahedral geometry around the Re^{I} metal centre.

Interesting intermolecular Br···O and Br···Br contacts are observed between adjacent molecules with distances of 3.226 (5) Å between Br1 and O3 and 3.590 (2) Å between Br2 and Br2 of the next molecule. These contacts together with an array of O—H···O, O—H···Br and C—H···O hydrogen bonds (see Table 2), complete a complex three-dimensional polymeric network.

S2. Experimental

$[NEt_4]_2[Re(CO)_3Br_3]$ was prepared as described by Alberto *et al.* (1996). 300 mg (0.3894 mmole) of $[NEt_4]_2[Re(CO)_3Br_3]$ was dissolved in 10 ml of H_2O at pH 2.2 and stirred for 30 minutes (until dissolved). $AgNO_3$ (198 mg, 1.167 mmol) was added to the solution and stirred for 24 h at room temperature. AgBr was formed as a grey precipitate and was filtered off and weighed (0.220 g). Tribromotroplone [151 mg, 0.4514 mmol for synthesis see Steyl & Roodt (2006)] in 2 ml of methanol was added the solution and stirred for 40 h at room temperature. The filtrate was left to stand for a few days and orange plate-like crystals suitable for X-ray diffraction were collected.

S3. Refinement

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

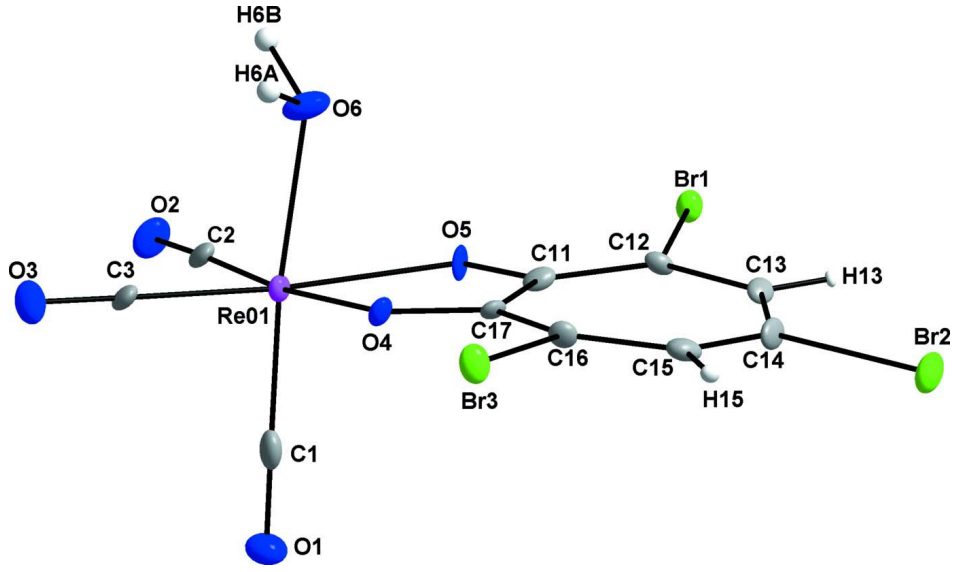

Figure 1

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

Aquatricarbonyl(3,5,7-tribromotropolonato)rhenium(I) methanol solvate

Crystal data

$[Re(C_7H_2Br_3O_2)(CO)_3(H_2O)]\cdot CH_4O$
$M_r = 678.1$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 9.090\ (5)$ Å
$b = 9.379\ (5)$ Å
$c = 10.010\ (5)$ Å
$\alpha = 109.569\ (5)°$
$\beta = 94.285\ (5)°$
$\gamma = 102.133\ (5)°$
$V = 776.3\ (7)$ Å^3
$Z = 2$
$F(000) = 620$
$D_x = 2.901$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 3145 reflections
$\theta = 2.2–28.2°$
$\mu = 15.58$ mm^{-1}
$T = 100$ K
Plate, orange
0.19 × 0.06 × 0.03 mm

Data collection

Bruker APEX diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.150$, $T_{max} = 0.626$
8673 measured reflections
3599 independent reflections
3018 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.2°$
$h = -8\rightarrow12$
$k = -11\rightarrow12$
$l = -13\rightarrow10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.079$
$S = 1.05$
3599 reflections
207 parameters
0 restraints
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0385P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 2.41$ e Å^{-3}
$\Delta\rho_{min} = -2.11$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Re01	0.54228 (3)	0.51098 (3)	0.75340 (3)	0.00853 (8)
Br1	0.46576 (7)	0.05513 (7)	0.28520 (6)	0.01185 (14)
O4	0.3428 (5)	0.3587 (5)	0.7724 (4)	0.0107 (9)
C11	0.3852 (7)	0.1903 (7)	0.5557 (7)	0.0098 (13)
C2	0.7128 (8)	0.6389 (7)	0.7149 (7)	0.0141 (8)
C15	0.1120 (7)	−0.0432 (8)	0.6174 (7)	0.0130 (13)
H15	0.0347	−0.093	0.6543	0.016 (19)*
C3	0.5502 (8)	0.6908 (8)	0.9164 (7)	0.0141 (8)
C17	0.2988 (7)	0.2213 (7)	0.6772 (6)	0.0079 (12)
O5	0.4976 (5)	0.3042 (5)	0.5642 (4)	0.0096 (9)
C16	0.1735 (7)	0.1137 (7)	0.6939 (6)	0.0100 (13)
C12	0.3491 (7)	0.0514 (7)	0.4343 (6)	0.0084 (12)
C13	0.2524 (7)	−0.0921 (7)	0.4077 (7)	0.0101 (13)
H13	0.2554	−0.1698	0.3217	0.012 (18)*
C14	0.1514 (7)	−0.1368 (7)	0.4909 (7)	0.0135 (13)
Br2	0.04885 (8)	−0.35228 (8)	0.42285 (7)	0.01741 (15)
Br3	0.08290 (7)	0.19489 (8)	0.85829 (7)	0.01438 (15)
O2	0.8201 (5)	0.7193 (5)	0.6960 (5)	0.0181 (11)
O3	0.5530 (6)	0.8006 (6)	1.0157 (5)	0.0191 (11)
O1	0.7678 (6)	0.4158 (6)	0.9272 (5)	0.0221 (11)
C1	0.6804 (8)	0.4526 (8)	0.8620 (7)	0.0153 (14)
O7	0.1793 (6)	0.6493 (6)	0.8032 (5)	0.0186 (11)
H7	0.1438	0.5913	0.8449	0.028*
C4	0.1962 (8)	0.8155 (7)	0.8974 (7)	0.0141 (8)
H4C	0.2448	0.833	0.992	0.021*
H4A	0.0974	0.8365	0.9024	0.021*
H4B	0.2572	0.8837	0.8583	0.021*
O6	0.3692 (6)	0.5548 (6)	0.6216 (5)	0.0214 (11)
H6A	0.295 (9)	0.599 (9)	0.681 (8)	0.032*
H6B	0.406 (9)	0.640 (9)	0.575 (8)	0.032*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re01	0.00886 (14)	0.00633 (14)	0.00918 (13)	−0.00064 (10)	0.00140 (9)	0.00280 (10)
Br1	0.0139 (3)	0.0099 (3)	0.0110 (3)	0.0015 (3)	0.0039 (2)	0.0033 (2)
O4	0.016 (3)	0.006 (2)	0.010 (2)	0.001 (2)	0.0039 (18)	0.0042 (18)
C11	0.007 (3)	0.012 (3)	0.015 (3)	0.005 (3)	0.004 (2)	0.008 (3)
C2	0.024 (2)	0.0071 (19)	0.0122 (17)	0.0075 (18)	0.0005 (15)	0.0033 (15)

C15	0.010 (3)	0.017 (4)	0.014 (3)	0.002 (3)	−0.002 (2)	0.009 (3)
C3	0.024 (2)	0.0071 (19)	0.0122 (17)	0.0075 (18)	0.0005 (15)	0.0033 (15)
C17	0.010 (3)	0.007 (3)	0.010 (3)	0.001 (3)	0.000 (2)	0.006 (2)
O5	0.010 (2)	0.004 (2)	0.011 (2)	−0.0030 (19)	0.0036 (17)	−0.0002 (17)
C16	0.011 (3)	0.012 (3)	0.008 (3)	0.002 (3)	0.003 (2)	0.005 (3)
C12	0.006 (3)	0.013 (3)	0.007 (3)	0.001 (3)	0.001 (2)	0.005 (2)
C13	0.004 (3)	0.010 (3)	0.014 (3)	0.000 (3)	−0.002 (2)	0.004 (3)
C14	0.010 (3)	0.007 (3)	0.019 (3)	−0.004 (3)	−0.004 (3)	0.004 (3)
Br2	0.0181 (4)	0.0091 (3)	0.0225 (3)	−0.0015 (3)	0.0040 (3)	0.0052 (3)
Br3	0.0123 (3)	0.0144 (3)	0.0140 (3)	−0.0005 (3)	0.0057 (3)	0.0036 (3)
O2	0.016 (3)	0.015 (3)	0.025 (3)	0.001 (2)	0.010 (2)	0.011 (2)
O3	0.017 (3)	0.017 (3)	0.018 (2)	0.005 (2)	0.003 (2)	−0.001 (2)
O1	0.022 (3)	0.025 (3)	0.020 (3)	0.008 (2)	−0.001 (2)	0.010 (2)
C1	0.020 (4)	0.009 (3)	0.012 (3)	0.000 (3)	0.003 (3)	−0.001 (3)
O7	0.025 (3)	0.021 (3)	0.022 (3)	0.013 (2)	0.008 (2)	0.018 (2)
C4	0.024 (2)	0.0071 (19)	0.0122 (17)	0.0075 (18)	0.0005 (15)	0.0033 (15)
O6	0.025 (3)	0.025 (3)	0.025 (3)	0.013 (3)	0.008 (2)	0.018 (2)

Geometric parameters (Å, º)

Re01—C1	1.882 (7)	C3—O3	1.162 (8)
Re01—C3	1.897 (6)	C17—C16	1.415 (8)
Re01—C2	1.899 (7)	C16—Br3	1.895 (6)
Re01—O4	2.123 (5)	C12—C13	1.372 (9)
Re01—O5	2.146 (4)	C13—C14	1.378 (9)
Re01—O6	2.170 (5)	C13—H13	0.93
Br1—C12	1.899 (6)	C14—Br2	1.900 (6)
O4—C17	1.278 (7)	O1—C1	1.168 (8)
C11—O5	1.289 (7)	O7—C4	1.495 (8)
C11—C12	1.408 (9)	O7—H7	0.82
C11—C17	1.477 (8)	C4—H4C	0.96
C2—O2	1.171 (8)	C4—H4A	0.96
C15—C16	1.379 (9)	C4—H4B	0.96
C15—C14	1.398 (9)	O6—H6A	0.99 (8)
C15—H15	0.93	O6—H6B	1.06 (8)
C1—Re01—C3	89.5 (3)	C16—C17—C11	125.5 (6)
C1—Re01—C2	87.8 (3)	C11—O5—Re01	117.1 (4)
C3—Re01—C2	85.0 (3)	C15—C16—C17	131.3 (6)
C1—Re01—O4	96.2 (2)	C15—C16—Br3	113.9 (5)
C3—Re01—O4	99.6 (2)	C17—C16—Br3	114.6 (4)
C2—Re01—O4	173.9 (2)	C13—C12—C11	131.5 (6)
C1—Re01—O5	96.7 (2)	C13—C12—Br1	113.1 (4)
C3—Re01—O5	171.5 (2)	C11—C12—Br1	115.2 (5)
C2—Re01—O5	100.9 (2)	C12—C13—C14	128.9 (6)
O4—Re01—O5	74.07 (16)	C12—C13—H13	115.6
C1—Re01—O6	174.3 (3)	C14—C13—H13	115.6
C3—Re01—O6	94.2 (2)	C13—C14—C15	128.3 (6)

C2—Re01—O6	96.8 (2)	C13—C14—Br2	115.9 (5)
O4—Re01—O6	78.93 (19)	C15—C14—Br2	115.8 (5)
O5—Re01—O6	79.17 (18)	O1—C1—Re01	178.7 (6)
C17—O4—Re01	118.0 (4)	C4—O7—H7	109.5
O5—C11—C12	120.1 (5)	O7—C4—H4C	109.5
O5—C11—C17	115.0 (5)	O7—C4—H4A	109.5
C12—C11—C17	124.9 (6)	H4C—C4—H4A	109.5
O2—C2—Re01	177.7 (6)	O7—C4—H4B	109.5
C16—C15—C14	128.0 (6)	H4C—C4—H4B	109.5
C16—C15—H15	116	H4A—C4—H4B	109.5
C14—C15—H15	116	Re01—O6—H6A	110 (4)
O3—C3—Re01	179.1 (6)	Re01—O6—H6B	117 (4)
O4—C17—C16	119.0 (5)	H6A—O6—H6B	102 (6)
O4—C17—C11	115.4 (6)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O6—H6*B*···Br1^{i}	1.06 (8)	2.68 (8)	3.421 (6)	127 (5)
O6—H6*B*···O5^{i}	1.06 (8)	1.86 (8)	2.825 (7)	149 (6)
C15—H15···O2^{ii}	0.93	2.5	3.409 (8)	166
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Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1; (ii) *x*−1, *y*−1, *z*; (iii) −*x*+1, −*y*+1, −*z*+2.