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

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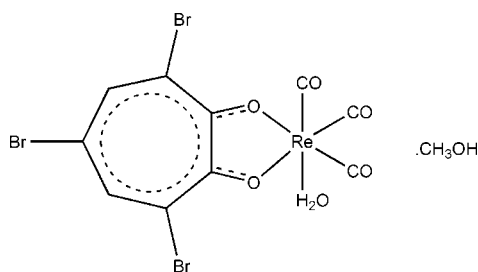
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Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.009$ Å; R factor = 0.033; wR factor = 0.079; data-to-parameter ratio = 17.4.

The title complex, $[\text{Re}(\text{C}_7\text{H}_2\text{Br}_3\text{O}_2)(\text{CO})_3(\text{H}_2\text{O})]\cdot\text{CH}_3\text{OH}$, 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 tribromotropolonato ligand and a water molecule complete the distorted octahedral coordination around the central metal. Intermolecular $\text{Br}\cdots\text{O}$ [3.226 (5) Å] and $\text{Br}\cdots\text{Br}$ [3.590 (2) Å] contacts are observed between adjacent molecules. These contacts, together with an array of $\text{O}-\text{H}\cdots\text{O}$, $\text{O}-\text{H}\cdots\text{Br}$ and $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds, complete a three-dimensional polymeric network formed between the methanol solvent and the complex.

Related literature

For a similar 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

$[\text{Re}(\text{C}_7\text{H}_2\text{Br}_3\text{O}_2)(\text{CO})_3(\text{H}_2\text{O})]\cdot\text{CH}_3\text{OH}$	$\beta = 94.285 (5)^\circ$
$M_r = 678.1$	$\gamma = 102.133 (5)^\circ$
Triclinic, $P\bar{1}$	$V = 776.3 (7) \text{ \AA}^3$
$a = 9.090 (5) \text{ \AA}$	$Z = 2$
$b = 9.379 (5) \text{ \AA}$	Mo $K\alpha$ radiation
$c = 10.010 (5) \text{ \AA}$	$\mu = 15.58 \text{ mm}^{-1}$
$\alpha = 109.569 (5)^\circ$	$T = 100 (2) \text{ K}$
	$0.19 \times 0.06 \times 0.03 \text{ mm}$

Data collection

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

Refinement

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

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, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
O6—H6B \cdots Br1 ⁱ	1.06 (8)	2.68 (8)	3.421 (6)	127 (5)
O6—H6B \cdots O5 ⁱ	1.06 (8)	1.86 (8)	2.825 (7)	149 (6)
C15—H15 \cdots O2 ⁱⁱ	0.93	2.5	3.409 (8)	166
O7—H7 \cdots O1 ⁱⁱⁱ	0.82	2.39	2.986 (7)	130
O6—H6A \cdots 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 (Brandenburg & Putz, 2005) and ORTEP-3 (Farrugia, 1999); software used to prepare material for publication: SHELXL97.

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

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supplementary materials

Acta Cryst. (2008). E64, m1610-m1611 [doi:10.1107/S1600536808038737]

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

M. Schutte, H. G. Visser and A. Roodt

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 asymmetric 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—OH₂ distance is also comparable to that of related structures (Mundwiler *et al.*, 2004; Kemp, 2006). The small bite angle O4—ReO1—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.

Experimental

[NEt₄]₂[Re(CO)₃Br₃] was prepared as described by Alberto *et al.* (1996). 300 mg (0.3894 mmole) of [NEt₄]₂[Re(CO)₃Br₃] was dissolved in 10 ml of H₂O at pH 2.2 and stirred for 30 minutes (until dissolved). AgNO₃ (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 to 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.

Refinement

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

Figures

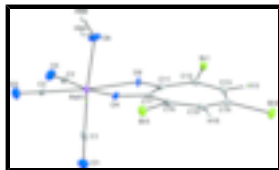


Fig. 1. Representation of the title compound, showing the numbering scheme and displacement ellipsoids (50% probability).

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

Crystal data

$[\text{Re}(\text{C}_7\text{H}_2\text{Br}_3\text{O}_2)(\text{CO})_3(\text{H}_2\text{O})] \cdot \text{CH}_4\text{O}$

$M_r = 678.1$

Triclinic, $P\bar{1}$

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) Å³

$Z = 2$

$F_{000} = 620$

$D_x = 2.901$ 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

$T = 100$ (2) K

Plate, orange

$0.19 \times 0.06 \times 0.03$ 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 $I > 2\sigma(I)$

$R_{\text{int}} = 0.035$

$\theta_{\max} = 28.3$ °

$\theta_{\min} = 2.2$ °

$h = -8 \rightarrow 12$

$k = -11 \rightarrow 12$

$l = -13 \rightarrow 10$

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

H atoms treated by a mixture of
independent and constrained refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0385P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} < 0.001$$

$$\Delta\rho_{\max} = 2.41 \text{ e } \text{Å}^{-3}$$

$$\Delta\rho_{\min} = -2.11 \text{ e } \text{Å}^{-3}$$

Extinction correction: none

207 parameters

Special details

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

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
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 (\AA^2)

	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)

supplementary materials

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, °)

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, °)

<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>
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) $-x+1, -y+1, -z+2$.

