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Bromido-1κBr-tricarbonyl-2$\kappa^3 C$-(2η^5-cyclopentadienyl)molybdenum(I)-tungsten(I)(*W—Mo*)

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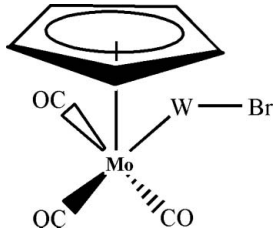
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.017 Å; R factor = 0.031; wR factor = 0.069; data-to-parameter ratio = 21.0.

The title compound, $[WMoBr(C_5H_5)(CO)_3]$, is built up from a pseudo-square-pyramidal piano-stool coordination around the Mo atom, the important geometry being Mo—W = 2.6872 (7) Å, W—Br = 2.5591 (9) Å and Mo—W—Br = 158.35 (3)°.

Related literature

For related literature, see Albright *et al.* (1978); Bueno & Churchill (1981); Changamu *et al.* (2006); Friedrich *et al.* (2004).



Experimental

Crystal data

$[WMoBr(C_5H_5)(CO)_3]$
M_r = 508.82
Tetragonal, $P\bar{4}2_1c$
a = 11.9375 (9) Å
c = 15.546 (2) Å
V = 2215.4 (4) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 15.09 mm^{-1}
T = 100 (2) K
0.11 × 0.10 × 0.07 mm

Data collection

Bruker APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.251, T_{max} = 0.347
13298 measured reflections
2673 independent reflections
2497 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.069
S = 1.02
2673 reflections
127 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.72 e Å^{-3}
Absolute structure: Flack (1983), 1118 Friedel pairs
Flack parameter: 0.00 (1)

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: DN2343).

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supporting information

Acta Cryst. (2008). E**64**, m780 [doi:10.1107/S1600536808012828]

Bromido-1κBr-tricarbonyl-2$\kappa^3 C$-(2η^5-cyclopentadienyl)molybdenum(I)tungsten(I)(*W—Mo*)

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S1. Comment

The compound **I** was a by-product of a study on the functionalization of paraffins using transition metals. The functionalized compounds have potential applications in catalysis and organic syntheses (Changamu *et al.*, 2006). The compound **I** is similar to the reported structure of (η^5-$C_5H_5(CO)_3$MoHgCl (Bueno *et al.*, 1981), Albright *et al.* (1978). The bond distances of W—Mo, 2.6872 (7) Å and W—Br, 2.5591 (9) Å are comparable to Hg—Mo, 2.693 (30) Å and Hg—Cl, 2.437 (8) Å respectively. The slight difference between the bond lenghts involving the halides could be attributed to the difference in electronegativity and hence basicity between bromine and chlorine.The coordination around Mo is a pseudo-square pyramidal piano stool arrangement.(Fig. 1)

S2. Experimental

The compound **I** was prepared according to a reported procedure (Friedrich *et al.*, 2004) and crystals were grown by slow evaporation of a mixture of dichloromethane and hexane at 263 K.

S3. Refinement

Hydrogen atoms were treated as riding on their parent C atoms with C–H = 0.95 Å and U_{iso}(H) = 1.2 U_{eq}(C).

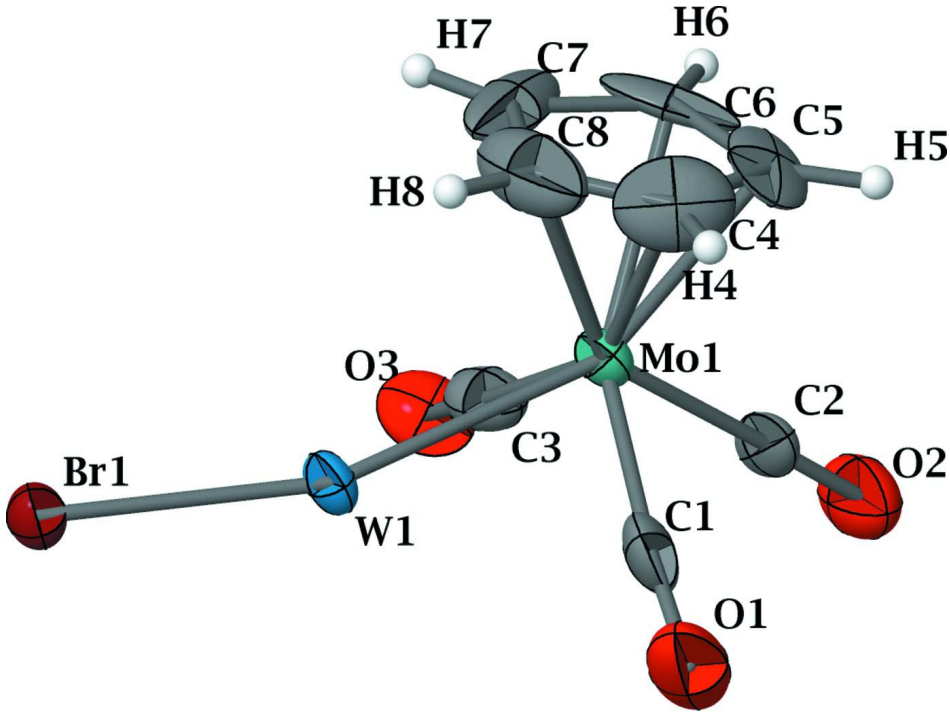

Figure 1

Molecular structure of the title complex showing the atom numbering scheme. Ellipsoids are drawn at the 50% probability level.

Bromido-1κBr-tricarbonyl-2κ^3C-(2η^5- cyclopentadienyl)molybdenum(I)tungsten(I)(W—Mo)

Crystal data

$[WMoBr(C_5H_5)(CO)_3]$
M_r = 508.82
Tetragonal, $P\bar{4}2_1c$
Hall symbol: P -4 2n
a = 11.9375 (9) Å
c = 15.546 (2) Å
V = 2215.4 (4) Å^3
Z = 8
$F(000)$ = 1824
D_x = 3.051 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 2238 reflections
θ = 2.2–25.5°
μ = 15.09 mm^{-1}
T = 100 K
Block, yellow
0.11 × 0.10 × 0.07 mm

Data collection

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Radiation source: fine-focus sealed tube
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ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.251, T_{max} = 0.347
13298 measured reflections
2673 independent reflections
2497 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
θ_{max} = 28.3°, θ_{min} = 2.2°
h = −15→14
k = −8→15
l = −20→18

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.069$
$S = 1.02$
2673 reflections
127 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0238P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 1.31$ e Å^{-3}
$\Delta\rho_{min} = -0.72$ e Å^{-3}
Absolute structure: Flack (1983), 1118 Friedel pairs
Absolute structure parameter: 0.00 (1)

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.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
W1	0.64476 (3)	0.39974 (3)	0.89630 (2)	0.01742 (9)
Mo1	0.73518 (6)	0.20182 (6)	0.85204 (5)	0.01833 (16)
Br1	0.62054 (7)	0.61253 (7)	0.90359 (5)	0.01974 (17)
O1	0.5124 (6)	0.1402 (6)	0.9503 (5)	0.0480 (19)
O2	0.6348 (5)	0.0161 (6)	0.7340 (4)	0.0402 (17)
O3	0.6948 (5)	0.3305 (6)	0.6794 (4)	0.0330 (16)
C1	0.5937 (8)	0.1680 (7)	0.9122 (6)	0.031 (2)
C2	0.6724 (7)	0.0833 (7)	0.7766 (6)	0.026 (2)
C3	0.7057 (7)	0.2868 (8)	0.7450 (6)	0.027 (2)
C4	0.8467 (10)	0.1507 (11)	0.9698 (7)	0.047 (3)
H4	0.8151	0.1245	1.0222	0.057*
C5	0.8729 (8)	0.0831 (8)	0.9024 (7)	0.038 (2)
H5	0.8622	0.0043	0.8996	0.046*
C6	0.9203 (7)	0.1535 (11)	0.8354 (6)	0.044 (3)
H6	0.9468	0.1308	0.7805	0.052*
C7	0.9181 (9)	0.2684 (10)	0.8715 (9)	0.059 (4)
H7	0.9431	0.3359	0.8455	0.071*
C8	0.8694 (10)	0.2533 (11)	0.9549 (7)	0.052 (3)
H8	0.8557	0.3126	0.9943	0.063*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
W1	0.01888 (17)	0.01447 (16)	0.01889 (15)	0.00532 (12)	0.00265 (14)	−0.00152 (14)

Mo1	0.0158 (3)	0.0170 (3)	0.0222 (3)	0.0033 (3)	0.0015 (3)	−0.0020 (3)
Br1	0.0214 (4)	0.0168 (4)	0.0209 (4)	0.0015 (3)	0.0032 (3)	0.0010 (3)
O1	0.036 (4)	0.032 (4)	0.076 (5)	0.006 (3)	0.029 (4)	0.011 (4)
O2	0.034 (4)	0.033 (4)	0.054 (4)	0.006 (3)	−0.008 (3)	−0.016 (3)
O3	0.035 (4)	0.045 (4)	0.019 (3)	0.007 (3)	−0.005 (3)	0.002 (3)
C1	0.032 (5)	0.015 (4)	0.045 (6)	0.009 (4)	0.004 (5)	0.003 (4)
C2	0.020 (5)	0.020 (5)	0.037 (5)	0.003 (4)	0.006 (4)	−0.012 (4)
C3	0.020 (5)	0.033 (5)	0.029 (5)	0.002 (4)	−0.004 (4)	−0.008 (4)
C4	0.042 (6)	0.069 (8)	0.030 (5)	−0.002 (7)	0.000 (5)	0.002 (6)
C5	0.033 (5)	0.030 (5)	0.053 (6)	0.015 (4)	−0.025 (5)	0.004 (5)
C6	0.016 (5)	0.091 (9)	0.024 (5)	0.028 (5)	−0.007 (4)	−0.007 (5)
C7	0.025 (6)	0.043 (7)	0.110 (11)	−0.014 (5)	−0.036 (6)	0.048 (7)
C8	0.037 (7)	0.058 (8)	0.061 (7)	0.015 (6)	−0.016 (5)	−0.035 (7)

Geometric parameters (Å, º)

W1—Br1	2.5591 (9)	O3—C3	1.152 (12)
W1—Mo1	2.6872 (7)	C4—C8	1.276 (17)
Mo1—C1	1.972 (10)	C4—C5	1.359 (15)
Mo1—C3	1.980 (10)	C4—H4	0.9500
Mo1—C2	1.985 (8)	C5—C6	1.453 (15)
Mo1—C6	2.298 (8)	C5—H5	0.9500
Mo1—C5	2.307 (8)	C6—C7	1.482 (17)
Mo1—C7	2.344 (10)	C6—H6	0.9500
Mo1—C4	2.345 (12)	C7—C8	1.433 (16)
Mo1—C8	2.346 (10)	C7—H7	0.9500
O1—C1	1.184 (11)	C8—H8	0.9500
O2—C2	1.132 (10)		
Br1—W1—Mo1	158.35 (3)	C4—Mo1—W1	104.9 (3)
C1—Mo1—C3	110.5 (4)	C8—Mo1—W1	82.5 (3)
C1—Mo1—C2	79.1 (4)	O1—C1—Mo1	175.0 (8)
C3—Mo1—C2	78.5 (4)	O2—C2—Mo1	178.9 (8)
C1—Mo1—C6	145.6 (4)	O3—C3—Mo1	174.3 (8)
C3—Mo1—C6	101.8 (4)	C8—C4—C5	112.4 (11)
C2—Mo1—C6	96.8 (4)	C8—C4—Mo1	74.3 (7)
C1—Mo1—C5	108.9 (4)	C5—C4—Mo1	71.5 (6)
C3—Mo1—C5	136.6 (4)	C8—C4—H4	123.8
C2—Mo1—C5	91.8 (4)	C5—C4—H4	123.8
C6—Mo1—C5	36.8 (4)	Mo1—C4—H4	121.9
C1—Mo1—C7	143.7 (4)	C4—C5—C6	107.4 (10)
C3—Mo1—C7	95.8 (4)	C4—C5—Mo1	74.5 (6)
C2—Mo1—C7	132.1 (4)	C6—C5—Mo1	71.3 (5)
C6—Mo1—C7	37.2 (4)	C4—C5—H5	126.3
C5—Mo1—C7	60.0 (4)	C6—C5—H5	126.3
C1—Mo1—C4	93.6 (4)	Mo1—C5—H5	119.8
C3—Mo1—C4	152.9 (4)	C5—C6—C7	104.9 (9)
C2—Mo1—C4	119.4 (4)	C5—C6—Mo1	71.9 (5)

C6—Mo1—C4	58.5 (4)	C7—C6—Mo1	73.0 (5)
C5—Mo1—C4	34.0 (4)	C5—C6—H6	127.6
C7—Mo1—C4	57.2 (4)	C7—C6—H6	127.6
C1—Mo1—C8	108.4 (4)	Mo1—C6—H6	119.6
C3—Mo1—C8	124.1 (4)	C8—C7—C6	103.5 (9)
C2—Mo1—C8	148.0 (4)	C8—C7—Mo1	72.3 (6)
C6—Mo1—C8	59.1 (4)	C6—C7—Mo1	69.7 (5)
C5—Mo1—C8	56.1 (4)	C8—C7—H7	128.2
C7—Mo1—C8	35.6 (4)	C6—C7—H7	128.2
C4—Mo1—C8	31.6 (4)	Mo1—C7—H7	121.7
C1—Mo1—W1	73.4 (2)	C4—C8—C7	111.8 (10)
C3—Mo1—W1	72.1 (3)	C4—C8—Mo1	74.2 (7)
C2—Mo1—W1	128.8 (3)	C7—C8—Mo1	72.1 (6)
C6—Mo1—W1	129.5 (3)	C4—C8—H8	124.1
C5—Mo1—W1	137.7 (3)	C7—C8—H8	124.1
C7—Mo1—W1	92.5 (3)	Mo1—C8—H8	121.1

Br1—W1—Mo1—C1	−172.7 (3)	C8—Mo1—C6—C5	73.6 (7)
Br1—W1—Mo1—C3	−54.0 (3)	W1—Mo1—C6—C5	119.9 (6)
Br1—W1—Mo1—C2	−112.2 (3)	C1—Mo1—C6—C7	−115.9 (9)
Br1—W1—Mo1—C6	36.8 (3)	C3—Mo1—C6—C7	83.9 (7)
Br1—W1—Mo1—C5	87.2 (4)	C2—Mo1—C6—C7	163.6 (6)
Br1—W1—Mo1—C7	41.3 (3)	C5—Mo1—C6—C7	−112.4 (8)
Br1—W1—Mo1—C4	97.9 (3)	C4—Mo1—C6—C7	−76.0 (7)
Br1—W1—Mo1—C8	75.5 (3)	C8—Mo1—C6—C7	−38.9 (6)
C1—Mo1—C4—C8	−120.1 (8)	W1—Mo1—C6—C7	7.4 (7)
C3—Mo1—C4—C8	33.5 (13)	C5—C6—C7—C8	−0.4 (9)
C2—Mo1—C4—C8	160.4 (7)	Mo1—C6—C7—C8	65.0 (7)
C6—Mo1—C4—C8	81.2 (8)	C5—C6—C7—Mo1	−65.4 (6)
C5—Mo1—C4—C8	120.8 (11)	C1—Mo1—C7—C8	8.5 (10)
C7—Mo1—C4—C8	36.9 (7)	C3—Mo1—C7—C8	145.7 (7)
W1—Mo1—C4—C8	−46.3 (8)	C2—Mo1—C7—C8	−134.5 (8)
C1—Mo1—C4—C5	119.1 (7)	C6—Mo1—C7—C8	−112.3 (8)
C3—Mo1—C4—C5	−87.3 (11)	C5—Mo1—C7—C8	−72.6 (7)
C2—Mo1—C4—C5	39.6 (8)	C4—Mo1—C7—C8	−32.7 (6)
C6—Mo1—C4—C5	−39.6 (7)	W1—Mo1—C7—C8	73.4 (7)
C7—Mo1—C4—C5	−83.8 (8)	C1—Mo1—C7—C6	120.8 (8)
C8—Mo1—C4—C5	−120.8 (11)	C3—Mo1—C7—C6	−102.0 (6)
W1—Mo1—C4—C5	−167.1 (6)	C2—Mo1—C7—C6	−22.2 (8)
C8—C4—C5—C6	0.7 (13)	C5—Mo1—C7—C6	39.7 (6)
Mo1—C4—C5—C6	64.2 (6)	C4—Mo1—C7—C6	79.6 (7)
C8—C4—C5—Mo1	−63.4 (10)	C8—Mo1—C7—C6	112.3 (8)
C1—Mo1—C5—C4	−67.1 (8)	W1—Mo1—C7—C6	−174.3 (6)
C3—Mo1—C5—C4	138.6 (7)	C5—C4—C8—C7	−1.0 (14)
C2—Mo1—C5—C4	−146.2 (7)	Mo1—C4—C8—C7	−62.8 (8)
C6—Mo1—C5—C4	115.0 (10)	C5—C4—C8—Mo1	61.8 (9)
C7—Mo1—C5—C4	74.7 (8)	C6—C7—C8—C4	0.9 (12)
C8—Mo1—C5—C4	32.8 (7)	Mo1—C7—C8—C4	64.1 (9)

W1—Mo1—C5—C4	18.7 (9)	C6—C7—C8—Mo1	−63.2 (6)
C1—Mo1—C5—C6	177.9 (6)	C1—Mo1—C8—C4	65.5 (8)
C3—Mo1—C5—C6	23.6 (8)	C3—Mo1—C8—C4	−162.4 (7)
C2—Mo1—C5—C6	98.8 (6)	C2—Mo1—C8—C4	−33.5 (12)
C7—Mo1—C5—C6	−40.2 (6)	C6—Mo1—C8—C4	−79.1 (8)
C4—Mo1—C5—C6	−115.0 (10)	C5—Mo1—C8—C4	−35.3 (7)
C8—Mo1—C5—C6	−82.2 (7)	C7—Mo1—C8—C4	−119.8 (10)
W1—Mo1—C5—C6	−96.2 (7)	W1—Mo1—C8—C4	135.2 (7)
C4—C5—C6—C7	−0.1 (10)	C1—Mo1—C8—C7	−174.7 (7)
Mo1—C5—C6—C7	66.2 (6)	C3—Mo1—C8—C7	−42.6 (8)
C4—C5—C6—Mo1	−66.3 (7)	C2—Mo1—C8—C7	86.3 (11)
C1—Mo1—C6—C5	−3.5 (10)	C6—Mo1—C8—C7	40.7 (6)
C3—Mo1—C6—C5	−163.7 (6)	C5—Mo1—C8—C7	84.5 (7)
C2—Mo1—C6—C5	−84.0 (6)	C4—Mo1—C8—C7	119.8 (10)
C7—Mo1—C6—C5	112.4 (8)	W1—Mo1—C8—C7	−105.0 (7)
C4—Mo1—C6—C5	36.5 (6)		