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fac-Bromidotricarbonyl[2-(diisopropylphosphanyl)benzaldehyde-κ^2O,P]rhenium(I)

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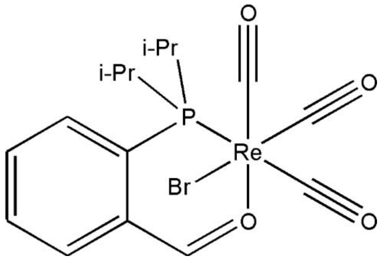
Key indicators: single-crystal X-ray study; T = 200 K; mean σ(C–C) = 0.005 Å; R factor = 0.018; wR factor = 0.045; data-to-parameter ratio = 20.1.

The structure of the title complex, $[\mathrm{ReBr}(\mathrm{C}_{13}\mathrm{H}_{19}\mathrm{OP})(\mathrm{CO})_3]$, displays a facial coordination of the three CO ligands and a κ^2O,P coordination mode of the 2-diisopropylphosphinobenzaldehyde ligands. The Re—C bond distance for the CO ligand *trans* to the P atom is, due to its *trans* influence, elongated to 1.943 (3) Å, showing that this CO ligand is more weakly bound to the Re centre than the other two.

Related literature

For the structures of halo-*fac*-tricarbonyl-[κ^2O,P-(ligand)]-rhenium(I) complexes with ligands based on 2-diphenylphosphinobenzaldehyde or 2-diphenylphosphinobenzoic acid derivatives, see: Correia *et al.* (2001); Chen *et al.* (2001); Palma *et al.* (2004).



Experimental

Crystal data

$[\mathrm{ReBr}(\mathrm{C}_{13}\mathrm{H}_{19}\mathrm{OP})(\mathrm{CO})_3]$
M_r = 572.39
Monoclinic, $P2_1/c$
a = 10.750 (2) Å
b = 15.194 (3) Å
c = 13.699 (3) Å
β = 125.29 (3)°
V = 1826.4 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 8.94 mm^{-1}
T = 200 K
0.43 × 0.33 × 0.31 mm

Data collection

Siemens SMART 1000 CCD diffractometer
Absorption correction: numerical *SADABS* (Bruker, 1997) $T_{\min}$ = 0.631, $T_{\max}$ = 1
18487 measured reflections
4436 independent reflections
4095 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.045
S = 1.11
4436 reflections
221 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{\max}$ = 0.51 e Å^{-3}
$\Delta\rho_{\min}$ = −1.04 e Å^{-3}

Table 1
Selected bond lengths (Å).

Re1—C14	1.901 (3)	Re1—O1	2.1739 (18)
Re1—C15	1.943 (3)	Re1—P1	2.4655 (13)
Re1—C16	1.915 (3)	Re1—Br1	2.6116 (6)

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XPMA* (Zsolnai, 1996), *ORTEP* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2010).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: VN2046).

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supplementary materials

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fac-Bromidotricarbonyl[2-(diisopropylphosphanyl)benzaldehyde-κ^2*O*,*P*]rhenium(I)

Christos Apostolidis, Martin Ahlmann and Olaf Walter

Experimental

The title compound was obtained from the reaction of 371.0 mg (1.38 mmol) 2-diisopropylphosphinobenzaldehyde-dimethylacetale and 561.5 mg (1.38 mmol) bromo-pentacarbonyl-rhenium(I) in refluxing thf (30 ml) after recrystallization from diethylether in 93% yield (736 mg). Single crystals of the title compound were grown in an NMR tube with $CDCl_3$ as the solvant.

Refinement

The position of the H atom located at the aldehyde carbon atom was localized and refined together with its isotropic displacement parameter. The positions of all other H atoms were calculated at geometrical positions according to the hybridization of the atoms they are bound to. The isotropic *U* values of the hydrogen atoms were refined group-wisely.

Computing details

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINT* (Bruker, 1997); data reduction: *SAINT* (Bruker, 1997); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: XPMA (Zsolnai, 1996), *ORTEP* (Farrugia, 1997); software used to prepare material for publication: *publCIF* (Westrip, 2010).

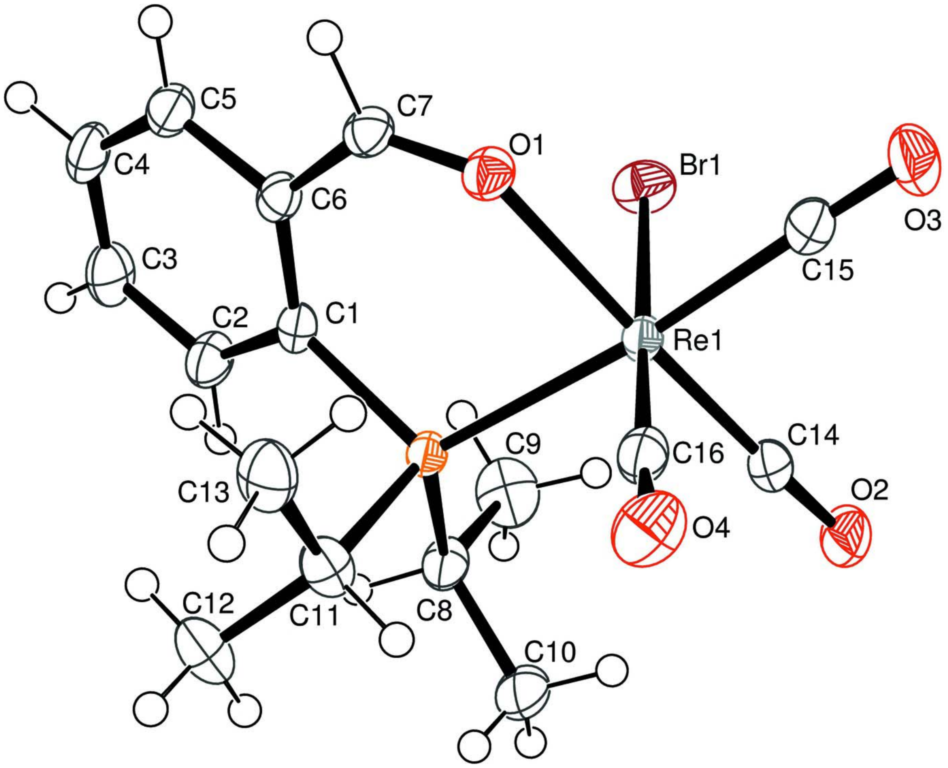

Figure 1

Molecular structure of the title compound with displacement ellipsoids at the 50% probability level.

fac-Bromidotricarbonyl[2-(diisopropylphosphanyl)benzaldehyde- $\kappa^2 O,P$]rhenium(I)

Crystal data

$[ReBr(C_{13}H_{19}OP)(CO)_3]$
$M_r = 572.39$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 10.750$ (2) Å
$b = 15.194$ (3) Å
$c = 13.699$ (3) Å
$\beta = 125.29$ (3)°
$V = 1826.4$ (9) Å^3
$Z = 4$
$F(000) = 1088$
$D_x = 2.082$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 165 reflections
$\theta = 2.3$–28.3°
$\mu = 8.94$ mm^{-1}
$T = 200$ K
Cube, orange
0.43 × 0.33 × 0.31 mm

Data collection

Siemens SMART 1000 CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8 pixels mm^{-1}
ω scans
Absorption correction: numerical *SADABS* (Bruker, 1997)
$T_{min} = 0.631$, $T_{max} = 1$
18487 measured reflections
4436 independent reflections
4095 reflections with $I > 2\sigma(I)$
$R_{int} = 0.027$
$\theta_{max} = 28.3°$, $\theta_{min} = 2.3°$
$h = -14 \rightarrow 14$
$k = -19 \rightarrow 19$
$l = -17 \rightarrow 17$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.018$
$wR(F^2) = 0.045$
$S = 1.11$
4436 reflections
221 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0228P)^2 + 0.987P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.51$ e Å^{-3}
$\Delta\rho_{min} = -1.04$ e Å^{-3}

Special details

Experimental. Spectroscopic data: ^{1}H{^{31}P} NMR (CDCl$_3$): $\delta = 9.71$, s, 1H, CHO; 7.99, dd, $^3J_{HH} = 7.5$ Hz, $^4J_{HH} = 1.2$ Hz, 1H, CH(arom); 7.89, dt, $^3J_{HH} = 7.5$ Hz, $^4J_{HH} = 1.2$ Hz, 1H, CH(arom); 7.78, dt, $^3J_{HH} = 7.5$ Hz, $^4J_{HH} = 1.2$ Hz, 1H, CH(arom); 7.72, dd, $^3J_{HH} = 7.5$ Hz, $^4J_{HH} = 1.2$ Hz, 1H, CH(arom); 2.94, hept, $^3J_{HH} = 7.0$ Hz, 1H, CH(Me)$_2$; 2.55, hept, $^3J_{HH} = 6.9$ Hz, 1H, CH(Me)$_2$; 1.38, d, $^3J_{HH} = 7.0$ Hz, 3H, CH$_3$; 1.31, d, $^3J_{HH} = 7.0$ Hz, 3H, CH$_3$; 1.03, d, $^3J_{HH} = 7.0$ Hz, 3H, CH$_3$; 0.97, d, $^3J_{HH} = 6.9$ Hz, 3H, CH$_3$; ^{31}P{^{1}H} NMR (CDCl$_3$): $\delta = 19.7$, s; ^{13}C{^{31}P}{^{1}H} NMR (CDCl$_3$): $\delta = 200.6$; 196.2; 190.3; 141.0; 136.0; 132.5; 131.6; 27.9; 23.3; 18.9; 18.2; 17.2; 16.2; IR ν_{CO} [cm^{-1}]: 2041, 1949, 1905, 1634; UV vis (CHCl$_3$): 414 nm (ε 2142); 261 nm (ε 9780); UV vis (MeCN): 391 nm (ε 1794); 312 nm (sh); 257 nm (ε 7952); 214 nm (ε 27000)

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. The data of the structure have been deposited at the CCDC with the reference number 894003 (Allen, 2002).

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

	x	y	z	U_{iso}*/U_{eq}
Re1	0.096757 (10)	0.651316 (6)	0.822326 (9)	0.01984 (4)
Br1	0.09257 (4)	0.70831 (2)	1.00026 (3)	0.03331 (7)
P1	−0.17759 (7)	0.68270 (4)	0.68063 (6)	0.01901 (13)
O1	0.0136 (2)	0.53062 (12)	0.85118 (17)	0.0246 (4)
O2	0.2119 (2)	0.83034 (14)	0.8052 (2)	0.0381 (5)
O3	0.4316 (2)	0.59455 (16)	1.0029 (2)	0.0446 (6)
O4	0.1170 (3)	0.57547 (15)	0.6249 (2)	0.0387 (5)
C1	−0.2889 (3)	0.63407 (17)	0.7300 (2)	0.0217 (5)
C2	−0.4274 (3)	0.6705 (2)	0.6956 (3)	0.0294 (6)
H2	−0.4596	0.7228	0.6525	0.035 (4)*
C3	−0.5192 (3)	0.6307 (2)	0.7243 (3)	0.0343 (7)
H3	−0.6104	0.6571	0.7013	0.035 (4)*
C4	−0.4759 (3)	0.5528 (2)	0.7861 (3)	0.0320 (6)
H4	−0.5383	0.5258	0.8038	0.035 (4)*
C5	−0.3387 (3)	0.51476 (19)	0.8221 (2)	0.0275 (6)
H5	−0.3092	0.4618	0.8638	0.035 (4)*
C6	−0.2436 (3)	0.55548 (17)	0.7962 (2)	0.0220 (5)

C7	−0.1008 (3)	0.50735 (17)	0.8444 (2)	0.0231 (5)
C8	−0.2409 (3)	0.79816 (17)	0.6455 (3)	0.0273 (6)
H8	−0.3526	0.7981	0.5939	0.038 (9)*
C9	−0.1909 (4)	0.8520 (2)	0.7555 (3)	0.0427 (8)
H9A	−0.0820	0.8576	0.8046	0.049 (4)*
H9B	−0.2227	0.8231	0.8000	0.049 (4)*
H9C	−0.2365	0.9093	0.7316	0.049 (4)*
C10	−0.1865 (4)	0.8395 (2)	0.5746 (3)	0.0390 (8)
H10A	−0.2189	0.8998	0.5572	0.049 (4)*
H10B	−0.2292	0.8077	0.5011	0.049 (4)*
H10C	−0.0773	0.8368	0.6209	0.049 (4)*
C11	−0.2622 (3)	0.6290 (2)	0.5325 (3)	0.0288 (6)
H11	−0.2026	0.6485	0.5029	0.030 (9)*
C12	−0.4282 (4)	0.6553 (2)	0.4382 (3)	0.0429 (8)
H12A	−0.4655	0.6244	0.3650	0.048 (4)*
H12B	−0.4338	0.7176	0.4242	0.048 (4)*
H12C	−0.4892	0.6404	0.4663	0.048 (4)*
C13	−0.2484 (4)	0.5296 (2)	0.5436 (3)	0.0357 (7)
H13A	−0.3162	0.5073	0.5620	0.048 (4)*
H13B	−0.1455	0.5138	0.6063	0.048 (4)*
H13C	−0.2749	0.5047	0.4694	0.048 (4)*
C14	0.1677 (3)	0.76221 (19)	0.8091 (2)	0.0262 (6)
C15	0.3070 (3)	0.6152 (2)	0.9370 (3)	0.0296 (6)
C16	0.1055 (3)	0.60419 (18)	0.6968 (3)	0.0256 (6)
H7	−0.101 (3)	0.4472 (19)	0.876 (2)	0.015 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.01570 (6)	0.01934 (6)	0.02291 (6)	−0.00064 (3)	0.01024 (4)	0.00179 (4)
Br1	0.04358 (17)	0.02932 (15)	0.02861 (14)	−0.00256 (12)	0.02177 (13)	−0.00259 (12)
P1	0.0162 (3)	0.0179 (3)	0.0219 (3)	−0.0005 (2)	0.0105 (2)	0.0015 (3)
O1	0.0244 (9)	0.0195 (9)	0.0297 (10)	0.0009 (7)	0.0155 (8)	0.0024 (8)
O2	0.0290 (11)	0.0302 (11)	0.0431 (13)	−0.0090 (9)	0.0139 (10)	0.0058 (10)
O3	0.0185 (10)	0.0465 (14)	0.0523 (14)	0.0029 (9)	0.0110 (10)	0.0147 (11)
O4	0.0445 (13)	0.0394 (13)	0.0460 (13)	−0.0008 (10)	0.0342 (11)	−0.0043 (10)
C1	0.0184 (12)	0.0227 (13)	0.0240 (13)	−0.0042 (10)	0.0122 (10)	−0.0025 (10)
C2	0.0242 (14)	0.0278 (15)	0.0385 (16)	−0.0006 (11)	0.0194 (12)	0.0019 (12)
C3	0.0220 (14)	0.0438 (18)	0.0406 (17)	−0.0017 (12)	0.0201 (13)	−0.0037 (14)
C4	0.0296 (15)	0.0386 (17)	0.0346 (15)	−0.0143 (13)	0.0225 (13)	−0.0074 (13)
C5	0.0296 (14)	0.0285 (14)	0.0261 (13)	−0.0083 (11)	0.0172 (11)	−0.0033 (11)
C6	0.0238 (12)	0.0200 (13)	0.0227 (12)	−0.0051 (10)	0.0136 (10)	−0.0037 (10)
C7	0.0265 (13)	0.0186 (13)	0.0242 (13)	−0.0033 (10)	0.0147 (11)	−0.0013 (10)
C8	0.0219 (13)	0.0190 (13)	0.0375 (15)	0.0028 (10)	0.0151 (12)	0.0069 (11)
C9	0.0440 (19)	0.0245 (16)	0.054 (2)	0.0056 (13)	0.0251 (17)	−0.0042 (14)
C10	0.0322 (16)	0.0329 (17)	0.0437 (19)	−0.0016 (12)	0.0171 (14)	0.0165 (14)
C11	0.0243 (13)	0.0342 (15)	0.0253 (14)	0.0002 (11)	0.0127 (11)	−0.0040 (12)
C12	0.0294 (16)	0.053 (2)	0.0275 (16)	−0.0033 (14)	0.0055 (13)	−0.0007 (14)
C13	0.0361 (16)	0.0339 (17)	0.0338 (16)	−0.0083 (13)	0.0184 (13)	−0.0114 (13)
C14	0.0182 (12)	0.0294 (15)	0.0268 (14)	0.0000 (10)	0.0106 (10)	0.0037 (11)

C15	0.0241 (14)	0.0283 (15)	0.0338 (15)	−0.0038 (11)	0.0152 (12)	0.0044 (12)
C16	0.0210 (12)	0.0241 (14)	0.0316 (14)	0.0002 (10)	0.0151 (11)	0.0033 (11)

Geometric parameters (Å, º)

Re1—C14	1.901 (3)	C5—H5	0.9300
Re1—C15	1.943 (3)	C6—C7	1.468 (4)
Re1—C16	1.915 (3)	C7—H7	1.01 (3)
Re1—O1	2.1739 (18)	C8—C9	1.514 (5)
Re1—P1	2.4655 (13)	C8—C10	1.528 (4)
Re1—Br1	2.6116 (6)	C8—H8	0.9800
P1—C1	1.835 (3)	C9—H9A	0.9600
P1—C8	1.842 (3)	C9—H9B	0.9600
P1—C11	1.865 (3)	C9—H9C	0.9600
O1—C7	1.231 (3)	C10—H10A	0.9600
O2—C14	1.153 (3)	C10—H10B	0.9600
O3—C15	1.144 (3)	C10—H10C	0.9600
O4—C16	1.148 (3)	C11—C13	1.517 (4)
C1—C2	1.392 (4)	C11—C12	1.533 (4)
C1—C6	1.406 (4)	C11—H11	0.9800
C2—C3	1.393 (4)	C12—H12A	0.9600
C2—H2	0.9300	C12—H12B	0.9600
C3—C4	1.371 (5)	C12—H12C	0.9600
C3—H3	0.9300	C13—H13A	0.9600
C4—C5	1.383 (4)	C13—H13B	0.9600
C4—H4	0.9300	C13—H13C	0.9600
C5—C6	1.405 (3)		
C14—Re1—C16	90.87 (12)	O1—C7—H7	118.4 (15)
C14—Re1—C15	89.06 (12)	C6—C7—H7	112.5 (15)
C16—Re1—C15	89.00 (12)	C9—C8—C10	111.4 (3)
C14—Re1—O1	174.15 (10)	C9—C8—P1	112.9 (2)
C16—Re1—O1	94.97 (9)	C10—C8—P1	109.7 (2)
C15—Re1—O1	91.35 (10)	C9—C8—H8	107.5
C14—Re1—P1	96.71 (8)	C10—C8—H8	107.5
C16—Re1—P1	91.28 (8)	P1—C8—H8	107.5
C15—Re1—P1	174.22 (8)	C8—C9—H9A	109.5
O1—Re1—P1	82.87 (5)	C8—C9—H9B	109.5
C14—Re1—Br1	91.10 (9)	H9A—C9—H9B	109.5
C16—Re1—Br1	177.00 (8)	C8—C9—H9C	109.5
C15—Re1—Br1	88.77 (10)	H9A—C9—H9C	109.5
O1—Re1—Br1	83.07 (5)	H9B—C9—H9C	109.5
P1—Re1—Br1	90.74 (3)	C8—C10—H10A	109.5
C1—P1—C8	104.98 (12)	C8—C10—H10B	109.5
C1—P1—C11	102.31 (13)	H10A—C10—H10B	109.5
C8—P1—C11	104.94 (14)	C8—C10—H10C	109.5
C1—P1—Re1	111.71 (9)	H10A—C10—H10C	109.5
C8—P1—Re1	118.89 (9)	H10B—C10—H10C	109.5
C11—P1—Re1	112.42 (10)	C13—C11—C12	110.0 (3)
C7—O1—Re1	136.60 (18)	C13—C11—P1	111.6 (2)

C2—C1—C6	117.4 (2)	C12—C11—P1	113.6 (2)
C2—C1—P1	120.9 (2)	C13—C11—H11	107.1
C6—C1—P1	121.56 (19)	C12—C11—H11	107.1
C1—C2—C3	121.7 (3)	P1—C11—H11	107.1
C1—C2—H2	119.1	C11—C12—H12A	109.5
C3—C2—H2	119.1	C11—C12—H12B	109.5
C4—C3—C2	120.4 (3)	H12A—C12—H12B	109.5
C4—C3—H3	119.8	C11—C12—H12C	109.5
C2—C3—H3	119.8	H12A—C12—H12C	109.5
C3—C4—C5	119.6 (3)	H12B—C12—H12C	109.5
C3—C4—H4	120.2	C11—C13—H13A	109.5
C5—C4—H4	120.2	C11—C13—H13B	109.5
C4—C5—C6	120.5 (3)	H13A—C13—H13B	109.5
C4—C5—H5	119.7	C11—C13—H13C	109.5
C6—C5—H5	119.7	H13A—C13—H13C	109.5
C5—C6—C1	120.4 (2)	H13B—C13—H13C	109.5
C5—C6—C7	113.0 (2)	O2—C14—Re1	177.4 (3)
C1—C6—C7	126.6 (2)	O3—C15—Re1	178.6 (3)
O1—C7—C6	129.1 (2)	O4—C16—Re1	177.2 (2)