

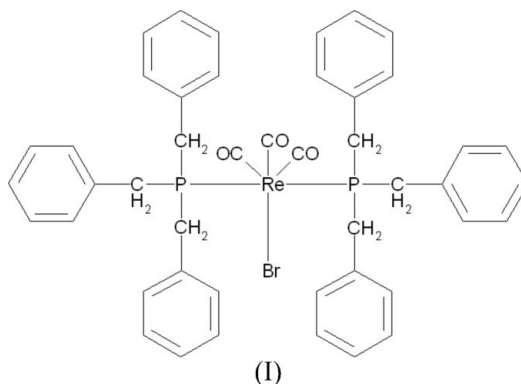
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Key indicators

Single-crystal X-ray study
 $T = 150$ K
Mean $\sigma(\text{C}-\text{C}) = 0.004$ Å
 R factor = 0.020
 wR factor = 0.049
Data-to-parameter ratio = 14.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.*fac,cis*-Bromidotricarbonylbis(tribenzylphosphine)-rhenium(I)Crystals of the title compound, *fac,cis*-[ReBr(C₂₁H₂₁P)₂(CO)₃], were obtained by recrystallization of a sample from CHCl₃ layered with hexane. The geometry about the d^6 Re^I centre is (distorted) octahedral with the three CO ligands *fac* and the two tribenzylphosphine ligands *cis*. In accordance with the *trans* influence, the Re—C bond *trans* to Br is significantly shorter than those *trans* to the organophosphine ligands.Received 9 February 2007
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Comment

In recent reports, we have described the synthesis and spectroscopic characterization of a series of tricarbonylbis(triorganophosphine)halides of Mn^I and Re^I, together with structural studies on selected compounds: *mer,trans*-[MnBr(CO)₃{P(C₆H₄Cl-4)₃}₂], *fac,cis*-[MnBr(CO)₃(dppe)], *fac,cis*-[MnBr(CO)₃(dppf)], *fac,cis*-[ReBr(CO)₃{P(C₆H₄OMe-4)₂}₂] and *fac,cis*-[ReBr(CO)₃(dppf)] (Beckett *et al.*, 2003), and *fac,cis*-[MnBr_{0.3}Cl_{0.7}(CO)₃(dppp)] (Light *et al.*, 2004). The synthesis and spectroscopic characterization of *fac,cis*-[ReBr(CO)₃{P(CH₂C₆H₅)₃}₂], (I), was described in an earlier publication, but crystals suitable for X-ray diffraction were unavailable at the time. We can now report a crystallographic study of (I).The molecular structure of (I) is shown in Fig. 1. The overall *fac,cis* geometry about an octahedral d^6 Re^I centre, as was indicated by earlier spectroscopic (IR and NMR) evidence, is confirmed by this crystallographic study. The octahedral geometry is considerably distorted [*cis* angles range from 81.055 (14) to 98.32 (2)°, with P1—Re1—P2 the largest, and *trans* angles range from 169.03 (8) to 173.81 (8)°], but bond lengths and angles are well within previously reported ranges for related compounds (Beckett *et al.*, 2003; Carballo *et al.*, 2001; Gibson *et al.* 2001). The Re1—C1 bond *trans* to Br is significantly shorter than the Re1—C2 and Re1—C3 bonds *trans* to P.

Experimental

fac,cis-[ReBr(CO)₃{P(CH₂C₆H₅)₃]₂, (I), was prepared by adapting a standard literature method (Angelici *et al.*, 1963) and its physical and spectroscopic properties have been reported previously (Beckett *et al.*, 2003). Orange single crystals suitable for X-ray diffraction studies were obtained by slow diffusion of hexane (layered) into a chloroform solution of (I) at 279 K.

Crystal data

[ReBr(C₂₁H₂₁P)₂(CO)₃] $\gamma = 92.164 (6)^\circ$
 $M_r = 958.84$ $V = 1969.6 (3) \text{ \AA}^3$
 Triclinic, $P\bar{1}$ $Z = 2$
 $a = 10.158 (1) \text{ \AA}$ Mo $K\alpha$ radiation
 $b = 10.373 (1) \text{ \AA}$ $\mu = 4.22 \text{ mm}^{-1}$
 $c = 19.289 (2) \text{ \AA}$ $T = 150 (2) \text{ K}$
 $\alpha = 103.046 (5)^\circ$ $0.10 \times 0.08 \times 0.08 \text{ mm}$
 $\beta = 94.822 (6)^\circ$

Data collection

Bruker–Nonius KappaCCD diffractometer 32577 measured reflections
 Absorption correction: multi-scan (SORTAV; Blessing, 1995) 6638 independent reflections
 $T_{\min} = 0.664, T_{\max} = 0.714$ 6477 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.020$ 469 parameters
 $wR(F^2) = 0.049$ H-atom parameters constrained
 $S = 1.07$ $\Delta\rho_{\text{max}} = 0.69 \text{ e \AA}^{-3}$
 6638 reflections $\Delta\rho_{\text{min}} = -1.09 \text{ e \AA}^{-3}$

Table 1

Selected bond angles ($^\circ$).

C2–Re1–C3	93.80 (11)	C1–Re1–P1	90.37 (9)
C2–Re1–C1	85.52 (11)	P2–Re1–P1	98.32 (2)
C3–Re1–C1	86.38 (12)	C2–Re1–Br1	173.81 (8)
C2–Re1–P2	92.78 (8)	C3–Re1–Br1	88.40 (8)
C3–Re1–P2	85.19 (8)	C1–Re1–Br1	88.85 (9)
C1–Re1–P2	171.28 (9)	P2–Re1–Br1	93.160 (18)
C2–Re1–P1	96.39 (8)	P1–Re1–Br1	81.055 (18)
C3–Re1–P1	169.03 (8)		

All H atoms were placed in idealized positions (C–H = 0.95–0.99 Å) and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The deepest residual electron-density hole is located 0.93 Å from atom Re1.

Data collection: COLLECT (Hooft, 1998); cell refinement: DENZO (Otwinowski & Minor, 1997) and COLLECT; data reduc-

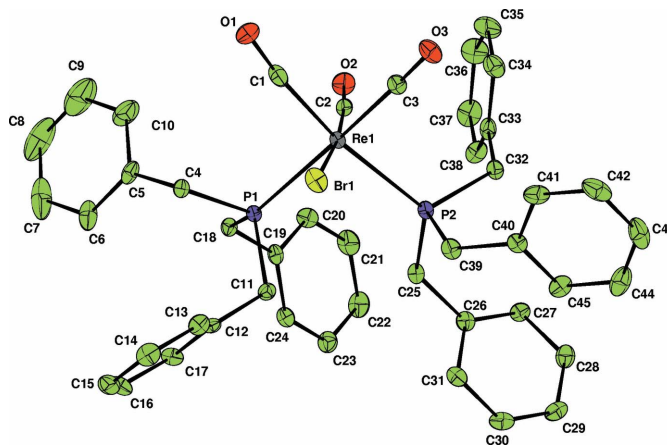


Figure 1

The molecular structure of (I), showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 35% probability level and H atoms have been omitted for clarity

tion: DENZO and COLLECT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: CAMERON (Watkin *et al.*, 1993); software used to prepare material for publication: WinGX (Farrugia, 1999).

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