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Bis(μ-diphenylphosphido)bis{*trans*-tricarbonyl-bis[1-(4-ethoxybutoxy)pentylidene]rhenium}

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The geometric centre of the title molecule, $Re_2(\mu\text{-}PPh_2)_2(CO)_6[ax\text{-}C(^nBu)(OBuOEt)]_2$ or $[Re_2(C_{11}H_{22}O_2)_2(C_{12}H_{10}P)_2(CO)_6]$, lies on a crystallographic inversion centre, realising a *trans*-position of the two *Z*-configurated carbene ligands.

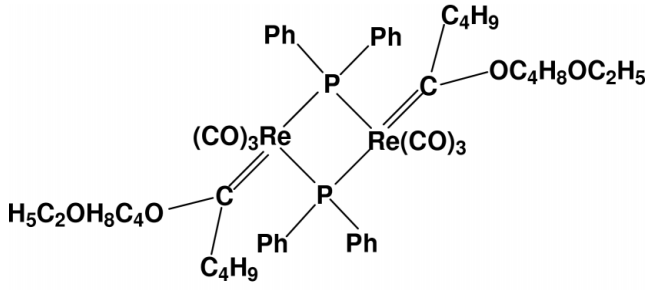
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.009 Å
R factor = 0.028
wR factor = 0.066
Data-to-parameter ratio = 18.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

Recently, we reported the preparation and characterization of Fischer-type carbene complexes $Re_2(\mu\text{-}PPh_2)_2(CO)_{8-n}(ax\text{-}C(R)OR')_n$ (n = 1,2; R, R' = organic residue) (Haupt *et al.*, 1998). The title compound, (I), is another member of this family.



(I)

The centre of the molecule lies on a crystallographic inversion centre. As a consequence, the Re_2P_2 ring is planar and the ax-carbene ligands at both Re atoms are in an *anti* position. Additionally, each rhenium is attached to three CO groups and the two common bridging μ-P atoms. The resulting coordination geometry is a slightly distorted octahedron. The CO and carbene ligands show ecliptic arrangement along the Re···Re vector, with torsion angles C—Re···Re—C in the range 0.3–0.6°. The orientation of the carbene ligand along the Re═C bond shows a *Z* configuration, as is already known from the other mentioned compounds of this type, and the Re···Re—C4—O4 torsion angle of 3.4 (2)° indicates the orientation of the C—O bond parallel to the Re···Re vector. The position of both the ^{n}Bu, as well as the $OC_4H_8OC_2H_5$-chain of the carbene group, avoids intramolecular steric crowding with the phenyl and carbonyl ligands. The shortest non-bonding intramolecular distance is 1.984 (1) Å for H31*A*···H38*B* and the shortest non-bonding intermolecular interaction is 2.430 (1) Å for H36*A*···H40*A*($x-1$, y, z), both from the two branches of the carbene ligands. The Re═C carbene bond length is 2.105 (4) Å and the Re—P bonds are

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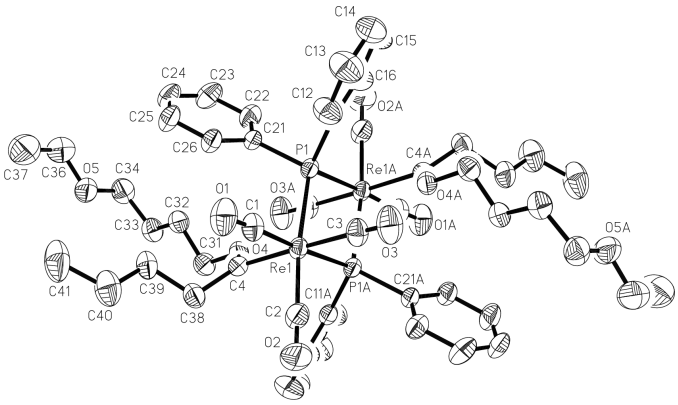

Figure 1
The molecular structure with H atoms omitted. Displacement ellipsoids are drawn at the 40% probability level.

2.5328 (15) and 2.5355 (14) Å (to P1 and P1′, respectively). These values compare well with those known from the related compounds $Re_2(\mu\text{-}PPh_2)_2(CO)_6$[ax-C(Ph)OEt]$_2$ or $Re_2(\mu\text{-}PPh_2)_2(CO)_6$[ax-C(nBu)OMe][ax-C(Ph)OMe] with Re═C in the range 2.08–2.131 Å and Re—P in the range 2.520–2.540 Å (Haupt *et al.*, 1998).

Experimental

Reaction of *trans*-$Li_2[Re_2(\mu\text{-}PPh_2)_2(CO)_6\{C(Bu)O\}_2]$ with CF_3SO_3Et (molar ratio 1:2) in $CHCl_3$/pentane solution at room temperature. The synthesis of the precursor Li complex salt has been described elsewhere (Haupt *et al.*, 1998).

Crystal data

$[Re_2(C_{11}H_{22}O_2)_2(C_{12}H_{10}P)_2(CO)_6]$
M_r = 1283.37
Triclinic, $P\overline{1}$
a = 10.294 (6) Å
b = 12.102 (5) Å
c = 12.230 (6) Å
α = 69.86 (2)°
β = 86.58 (2)°
γ = 66.39 (2)°
V = 1305.3 (11) Å^3
Z = 1
D_x = 1.633 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 8–15°
μ = 4.75 mm^{-1}
T = 293 (2) K
Prism, yellow
0.49 × 0.41 × 0.20 mm

Data collection

Bruker *P*4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.150, T_{max} = 0.409
5891 measured reflections
5615 independent reflections
4907 reflections with $I > 2\sigma(I)$
R_{int} = 0.026
θ_{max} = 27.6°
$h = -13 \rightarrow 13$
$k = -14 \rightarrow 15$
$l = 0 \rightarrow 15$
2 standard reflections every 400 reflections intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.066
S = 1.04
5615 reflections
299 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0371P)^2 + 0.5044P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.00 e Å^{-3}
$\Delta\rho_{min}$ = −0.87 e Å^{-3}
Extinction correction: *SHELXL*97
Extinction coefficient: 0.0081 (4)

Table 1
Selected geometric parameters (Å, °).

Re1—C4	2.105 (4)	C4—O4	1.304 (5)
Re1—P1^{i}	2.5328 (15)	C4—C38	1.514 (6)
Re1—P1	2.5355 (14)		
P1^{i}—Re1—P1	75.40 (4)	O4—C4—Re1	119.5 (3)
Re1^{i}—P1—Re1	104.60 (4)		

Symmetry code: (i) $2 - x, -y, 1 - z$.

Due to a generator failure during data collection about 400 intensities out of about 6000 were not recorded. The largest peak of residual electron density is close to a heavy atom, 0.85 Å from Re1. H atoms were refined at calculated positions riding on the parent C atoms with isotropic displacement parameters $U_{eq}(H) = 1.2U_{iso}(C)$.

Data collection: *XSCANS* (Bruker, 1996); cell refinement: *XSCANS*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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