Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

Heptacarbonyl(*α*-ethoxybenzylidene)di-*μ*-diethylphosphidodirhenium

Ulrich Flörke* and Dina Petters

Fachbereich Chemie und Chemietechnik,
Universität Paderborn, Warburgerstr. 100,
D-33098 Paderborn, Germany

Correspondence e-mail:
uf@chemie.uni-paderborn.de

Key indicators

Single-crystal X-ray study
T = 200 K
Mean σ(C–C) = 0.011 Å
R factor = 0.038
wR factor = 0.096
Data-to-parameter ratio = 20.2

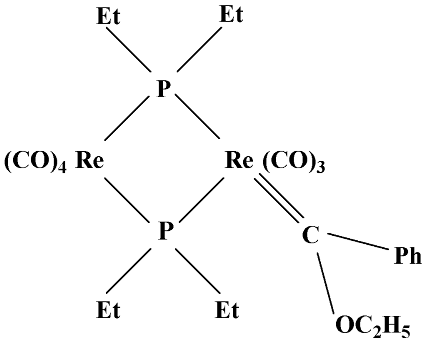
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, $[\mathrm{Re}_2(\mu\text{-PEt}_2)_2(\mathrm{CO})_7\{\text{ax–C(Ph)OEt}\}]$ or $[\mathrm{Re}_2(\mathrm{C}_9\mathrm{H}_{10}\mathrm{O})(\mathrm{C}_4\mathrm{H}_{10}\mathrm{P})_2(\mathrm{CO})_7]$, contains a carbene ligand with a Z configuration and a non-planar central $\mathrm{Re}_2\mathrm{P}_2$ ring.

Received 28 January 2002
Accepted 4 February 2002
Online 8 February 2002

Comment

The reaction of $\mathrm{Re}_2(\mu\text{-P}R_2)_2(\mathrm{CO})_8$ with nucleophilic lithium organyls LiR' leads to the salts $\mathrm{Li}(\mathrm{Re}_2(\mu\text{-P}R_2)_2(\mathrm{CO})_7\{\text{ax-C}(R')\mathrm{O}\}$ which then can be converted to Fischer-type carbene complexes (Haupt *et al.*, 1998*a*). The title compound, (I), with R = ethyl and R' = phenyl, is an axial monocarbene complex with a $\mathrm{Re}_2\mathrm{P}_2$ ring and slightly distorted octahedral coordination at both Re atoms. The C(Ph)OEt carbene group with a Z configuration is axially attached to Re2 with a Re═C double-bond length of 2.098 (7) Å. The orientation of the ReCOEt group is parallel to the Re···Re vector with a Re···Re—C8—O8 torsion angle of −3.2 (6)°. The Re1—P bonds of 2.5154 (17) and 2.5122 (19) Å for P1 and P2, respectively, are slightly longer than those for Re2—P of 2.5024 (18) and 2.5023 (17) Å, and reflect the unsymmetrical monosubstitution of this compound. These distances are, on average, slightly shorter than the Re—P bonds of the related mono- and dicarbene complexes which have μ-PPh_2-bridging ligands instead (Haupt *et al.*, 1998*a*). The average values for the enclosed ring angles are Re—P—Re = 104.35 (6)° and P—Re—P = 75.43 (6)°. The $\mathrm{Re}_2\mathrm{P}_2$ ring is folded, with an ReP_2/$\mathrm{P}_2\mathrm{Re}$ dihedral angle of 6.2 (1)°. This is a well known effect due to an intramolecular balance of unsymmetrical or sterically unfavourable substituted carbonyl complexes of this type (Haupt *et al.*, 1998*a*,*b*; Flörke & Petters, 2001). The C—Re—Re—C torsion angles range from 0.2 (4) to 8.8 (4)° (absolute values), in accordance with the ecliptic carbonyl-ligand arrangements.



(I)

DOI: 10.1107/S1600536802002258

Experimental

The title compound was obtained by reaction of Li(Re$_2$(μ-PEt$_2$)$_2$(CO)$_7${C(Ph)O}} with OEt$_3$BF$_4$ in CHCl$_3$ solution at room temperature. The synthesis of the precursor Li salt has been described earlier (Haupt *et al.*, 1998*a*).

Crystal data

[Re$_2$(C$_9$H$_{10}$O)(C$_4$H$_{10}$P)$_2$(CO)$_7$]
M_r = 880.82
Triclinic, $P\overline{1}$
a = 8.425 (2) Å
b = 10.504 (1) Å
c = 17.674 (4) Å
α = 73.04 (1)°
β = 78.82 (1)°
γ = 88.59 (1)°
V = 1466.8 (5) Å^3

Z = 2
D_x = 1.994 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 29 reflections
θ = 7.8–23.7°
μ = 8.40 mm^{-1}
T = 200 (2) K
Needle, colourless
0.38 × 0.15 × 0.06 mm

Data collection

Bruker *P*4 diffractometer
ω scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{\min}$ = 0.083, $T_{\max}$ = 0.608
8026 measured reflections
6665 independent reflections
5086 reflections with $I > 2\sigma(I)$

$R_{\rm int}$ = 0.023
$\theta_{\max}$ = 27.5°
$h = -10 \rightarrow 1$
$k = -13 \rightarrow 13$
$l = -22 \rightarrow 22$
3 standard reflections every 397 reflections intensity decay: 1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.096
S = 0.98
6665 reflections
330 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0575P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.94 e Å^{-3}
$\Delta\rho_{\min}$ = −0.82 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Re1—P2	2.5122 (19)	Re2—P1	2.5024 (18)
Re1—P1	2.5154 (17)	C8—O8	1.300 (8)
Re2—C8	2.098 (7)	C8—C11	1.517 (9)
Re2—P2	2.5023 (17)	O8—C9	1.495 (9)
P2—Re1—P1	75.23 (5)	Re2—P2—Re1	104.40 (6)
P2—Re2—P1	75.63 (6)	O8—C8—Re2	123.6 (5)
Re2—P1—Re1	104.30 (6)		

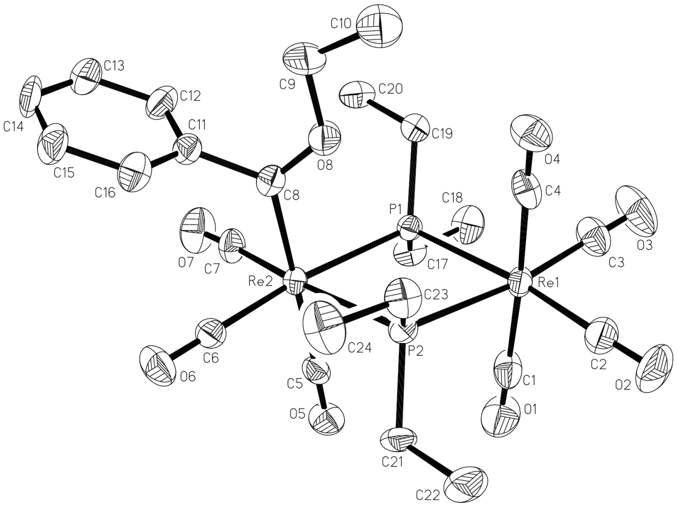


Figure 1
The molecular structure of (I) with H atoms omitted. Displacement ellipsoids are drawn at the 50% probability level.

H atoms were placed at calculated positions, riding on the attached C atoms, with isotropic displacement parameters $U_{\rm iso}$(H) = 1.2$U_{\rm eq}$(C) [1.5$U_{\rm eq}$(C) for methyl groups].

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*.

References

Bruker (1996). *XSCANS*. Version 2.21. Bruker AXS Inc., Madison, Wisconsin, USA.

Bruker (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.

Flörke, U. & Petters, D. (2001). *Acta Cryst.* E**57**, m237–m238.

Haupt, H.-J., Petters, D. & Flörke, U. (1998*a*). *J. Organomet. Chem.* **558**, 81–89.

Haupt, H.-J., Petters, D. & Flörke, U. (1998*b*). *J. Organomet. Chem.* **553**, 497–501.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.