Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Heptacarbonyl-$\kappa^3C,2\kappa^4C$-{μ-dicyclohexyl-[1(η^5)-cyclopentadienyl]phosphine-2κP}-molybdenumrhenium(*Mo–Re*)

Ulrich Flörke

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

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

Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.010 Å
R factor = 0.043
wR factor = 0.090
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.

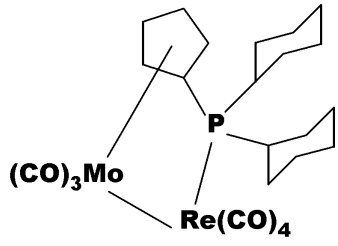
© 2001 International Union of Crystallography
Printed in Great Britain – all rights reserved

Received 16 August 2001
Accepted 7 September 2001
Online 20 September 2001

The title compound, $(CO)_4ReMo(CO)_3\{\eta^5\text{–}C_5H_4P(C_6H_{11})_2\}$ or $[MoRe(C_{17}H_{26}P)(CO)_7]$, displays one of the few known unbridged Re—Mo single bonds, with a length of 3.1307 (8) Å.

Comment

To date, only two crystal structure determinations on heterobimetallic η^5–C_5H_4–P bridged complexes have been reported. These are the manganese–molybdenum compounds $(CO)_4MnMo(CO)_3(\eta^5\text{-}C_5H_4PPh_2)$ (Casey *et al.*, 1982) and $(CO)_4MnMo(CO)_2(PPh_2Et)(\eta^5\text{-}C_5H_4PPh_2)$ (Doyle *et al.*, 1992). The related rhenium–molybdenum title complex, (I) (Fig. 1), represents one of the rare examples of an unsupported Re—Mo single bond. It has a length of 3.1307 (8) Å, which may be compared to that of 3.111 (2) Å in $(CO)_5ReMo(CO)_4CPh$ (Fischer *et al.*, 1976) and 3.172 (1) or 3.188 (1) Å in $(CO)_4LReMo(CO)_3Cp$, with L = CO or L = CN^tBu, respectively (Ingham *et al.*, 1992). According to a search of a recent release of the Cambridge Structural Database (Allen & Kennard, 1993), most ligand-bridged Re—Mo bonds cover the range from 2.842 to 3.106 Å, with the exception of three μ-H/μ-P bridged complexes, with Re—Mo single-bond lengths in the range 3.136–3.199 Å, that are even longer than the unbridged ones. The overall geometry of the title complex resembles that of the related Mn–Mo complexes mentioned above. The Mo—Re—P—X torsion angle is 8.8 (1)° (ignoring sign, X = Cp ring centroid) and the dihedral angle between the Cp ring plane and the MoRePX plane is 91.2 (2)°. Other relevant parameters are Re—P 2.4479 (17), Mo—X 2.049 (5) Å, Mo—Re—P 75.09 (4), Re—P—C31 101.10 (16) Re—Mo—X 112.2 (1) and P—X—Mo 74.7 (2)°.



(I)

Experimental

The reagents $Re_2(CO)_{10}$ (0.5 mmol), $MoCp(CO)_3)_2$ (0.5 mmol) and $HPCy_2$ (1 mmol) were dissolved in 2 ml xylene and reacted at 460 K for 12 h in a glass tube. Products were separated by thick-layer chromatography with CH_2Cl_2/hexane (1:2) as eluant. The title

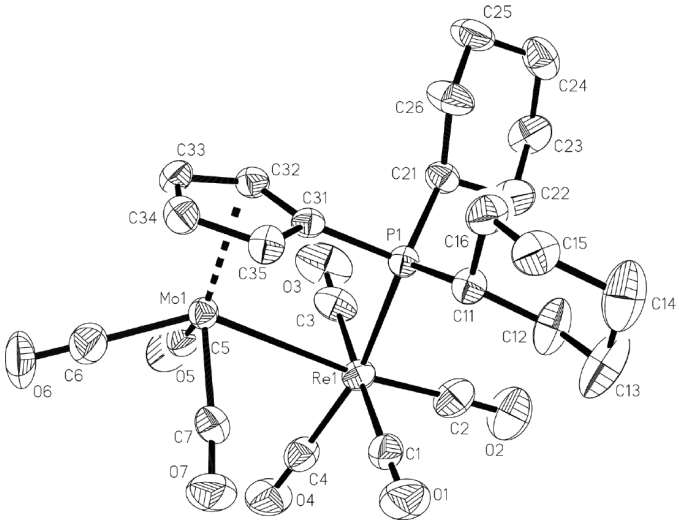


Figure 1
The molecular structure of the title compound with H atoms omitted. Displacement ellipsoids are drawn at the 50% probability level.

compound was isolated in 18% yield and recrystallized from CH_2Cl_2 solution (Lothert, 1994).

Crystal data

$[MoRe(C_{17}H_{26}P)(CO)_7]$	D_x = 1.881 Mg m^{-3}
M_r = 739.56	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 32 reflections
a = 14.173 (2) Å	θ = 7.3–18.6°
b = 11.146 (2) Å	μ = 5.21 mm^{-1}
c = 16.571 (3) Å	T = 293 (2) K
β = 93.82 (1)°	Prism, orange
V = 2611.9 (8) Å^3	0.46 × 0.29 × 0.10 mm
Z = 4	

Data collection

Bruker $P4$ diffractometer	R_{int} = 0.031
ω scans	θ_{max} = 27.6°
Absorption correction: ψ scan (North *et al.*, 1968)	$h = -18 \rightarrow 18$
T_{min} = 0.301, T_{max} = 0.994	$k = 0 \rightarrow 14$
6247 measured reflections	$l = 0 \rightarrow 21$
6042 independent reflections	3 standard reflections every 397 reflections
3964 reflections with $I > 2\sigma(I)$	intensity decay: <1%

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.043	$w = 1/[\sigma^2(F_o^2) + (0.0393P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.090	$(\Delta/\sigma)_{max}$ = 0.002
S = 0.96	$\Delta\rho_{max}$ = 0.71 e Å^{-3}
6042 reflections	$\Delta\rho_{min}$ = −0.74 e Å^{-3}
299 parameters	

Table 1
Selected geometric parameters (Å, °).

X = Cp ring centroid.

Re1—P1	2.4479 (17)	Mo1—X	2.049 (5)
Re1—Mo1	3.1307 (8)	P1—C31	1.811 (4)
P1—Re1—Mo1	75.09 (4)	Re1—Mo1—X	112.2 (1)

H atoms were refined at calculated positions riding on their attached 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*.

References

Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
Bruker (1996). *XSCANS*. Version 2.21. Bruker AXS Inc., Madison, Wisconsin, USA.
Bruker (1998). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
Casey, C. P., Bullock, R. M., Fultz, W. C. & Rheingold, A. L. (1982). *Organometallics*, **1**, 1591–1596.
Doyle, M. J., Duckworth, T. J., Manojlovic Muir, L., Mays, M. J., Raithby, P. R. & Robertson, F. J. (1992). *J. Chem. Soc. Dalton Trans.* pp. 2703–2714.
Fischer, E. O., Huttner, G., Lindner, T. L., Frank, A. & Kreißl, F. R. (1976). *Angew. Chem. Int. Ed. Engl.* **33**, 157–158.
Ingham, W. L., Travlos, S. D., Boeyens, J. C. A., Berry, M. & Coville, N. J. (1992). *Acta Cryst.* C**48**, 465–468.
Lothert, T. (1994). Dissertation thesis, University of Paderborn, Germany.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.