Acta Crystallographica Section E
**Structure Reports**
**Online**

ISSN 1600-5368

# *cis*-Dicarbonylbis[1,2-ethanediylbis(diphenylphosphine)-*P*,*P*′]tungsten(0) benzene sesquisolvate

**Alexander C. Filippou,† Athanassios I. Philippopoulos and Burkhard Ziemer***

Institut für Chemie, Humboldt-Universität zu Berlin, Hessische Straße 1-2, D-10115 Berlin, Germany

† Additional contact author, email: filippou@-chemie.hu-berlin.de.

Correspondence e-mail:
burkhard=ziemer@chemie.hu-berlin.de

**Key indicators**

Single-crystal X-ray study
$T$ = 180 K
Mean $\sigma$(C–C) = 0.005 Å
$R$ factor = 0.022
$wR$ factor = 0.053
Data-to-parameter ratio = 14.3

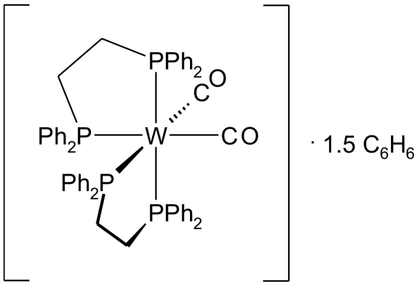
For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Received 21 May 2001
Accepted 28 June 2001
Online 13 July 2001

The title compound, *cis*-[W(CO)$_2$(dppe)$_2$] [dppe is 1,2-ethanediylbis(diphenylphosphine), $C_{26}H_{24}P_2$], was obtained as a by-product during the preparation of the dinitrogen complex *trans*-[W(CO)(N$_2$)(dppe)$_2$]. The crystal structure of the benzene sesquisolvate, *i.e.* *cis*-[W($C_{26}H_{24}P_2$)$_2$(CO)$_2$]·1.5$C_6H_6$, has been determined.

## Comment

Reaction of the germylenes Cp*Ge*X* (Cp* = C$_5$Me$_5$; *X* = Cl, Br, I) with the bis-dinitrogen complex *trans*-[W(dppe)$_2$(N$_2$)$_2$] has been recently shown to provide a convenient route to the complexes *trans*-[*X*(dppe)$_2$W≡Ge($\eta^1$–Cp*)] (*X* = Cl, Br, I) bearing a transition metal–germanium triple bond (Filippou *et al.*, 2000). In order to test the influence of a carbonyl ligand on the course of this reaction, the dinitrogen complex *trans*-[W(CO)(N$_2$)(dppe)$_2$], (2), was prepared from *trans*-[W(CO)(DMF)(dppe)$_2$] (DMF is *N,N*′-dimethylformamide), (1) (Ishida *et al.*, 1991). Yellow single crystals of the title compound, (3), as benzene sesquisolvate (1.5 $C_6H_6$), (3*a*), were obtained from the washings after isolation of complex (2).



(3*a*)

Complex (3) was first prepared by Chatt & Watson (1961); however, its molecular structure in the solid state was unknown until now. Two distorted octahedral molecules with marginally different bonding parameters and three molecules of benzene are present in the asymmetric unit of the cell. The W—P bonds *trans* to the carbonyl ligands have an average value of 2.5490 (9) Å, which is considerably larger than that of the W—P bonds *cis* to the carbonyl ligands [2.4525 (9) Å], indicating the strong *trans* influence of the CO groups.

## Experimental

Complex (1) (220 mg, 0.203 mmol) was suspended in benzene (20 ml) and the suspension stirred under an atmosphere of dry nitrogen. IR monitoring of the reaction revealed a gradual increase in intensity of

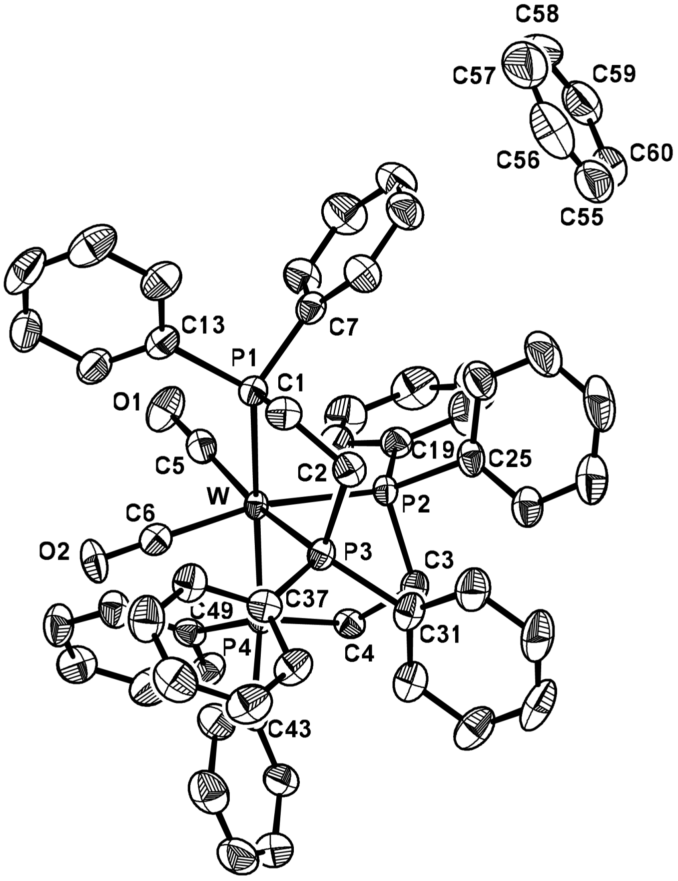


**Figure 1**
*DIAMOND* plot (Brandenburg, 1996) of one molecule (3a) and one solvate molecule showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

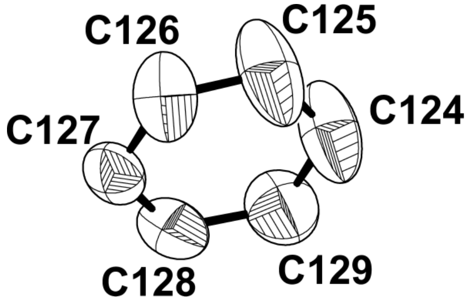


**Figure 2**
*DIAMOND* plot (Brandenburg, 1996) of one solvate molecule. H atoms have been omitted for clarity.

the $\nu(\text{N}\equiv\text{N})$ and $\nu(\text{C}\equiv\text{O})$ absorption of the product (2) at 2071 and 1801 cm$^{-1}$, respectively, and of the $\nu(\text{C}=\text{O})$ absorption of uncoordinated DMF at 1684 cm$^{-1}$. After approximately 10 h, all starting material had dissolved and the colour of the solution had changed from red to orange. Pentane (25 ml) was then added to the solution to precipitate complex (2) as an orange solid, which was washed with pentane (2 × 5 ml) and dried *in vacuo* at 253 K. The supernatant solution was combined with the pentane washings and allowed to stand for one day at ambient temperature to afford yellow crystals of (3*a*). IR of the crystals (KBr, cm$^{-1}$): 1852 (*vs*), 1778 (*vs*) [$\nu(\text{C}\equiv\text{O})$]; (THF, cm$^{-1}$): 1857 (*vs*), 1795 (s) [$\nu(\text{C}\equiv\text{O}$]; ($C_6H_6$, cm$^{-1}$): 1857(*vs*), 1795 (s) [$\nu(\text{C}\equiv\text{O})$]. During data collection the crystal was in cold $N_2$ gas of the Cryostream cooler (Oxford Cryosystems) mounted on a $\varphi$-axis diffractometer.

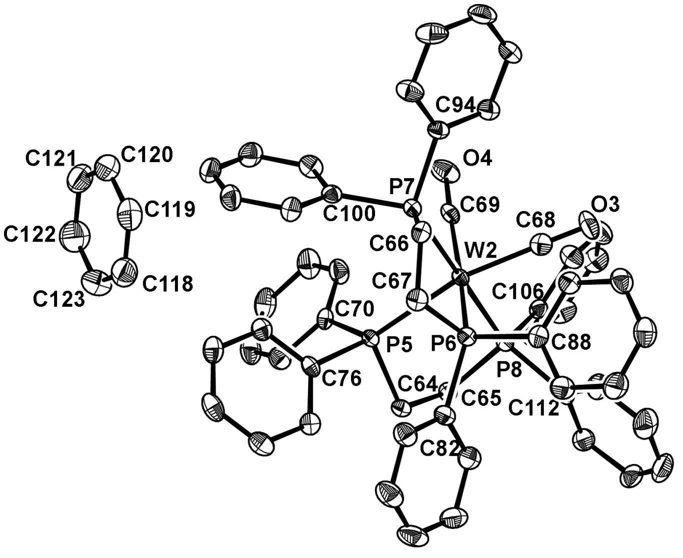


**Figure 3**
*DIAMOND* plot (Brandenburg, 1996) of the second molecule of (3a) and one solvate molecule showing 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

*Crystal data*

$[\text{W}(C_{26}H_{24}P_2)_2(\text{CO})_2]\cdot 1.5C_6H_6$
$M_r = 1153.82$
Triclinic, $P\overline{1}$
$a = 12.675$ (2) Å
$b = 18.259$ (3) Å
$c = 22.864$ (4) Å
$\alpha = 89.15$ (2)°
$\beta = 87.73$ (2)°
$\gamma = 77.31$ (2)°
$V = 5158.1$ (16) Å$^3$

$Z = 4$
$D_x = 1.486$ Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 5000 reflections
$\theta = 2.5$–25.0°
$\mu = 2.41$ mm$^{-1}$
$T = 180$ (2) K
Block, yellow
0.40 × 0.24 × 0.12 mm

*Data collection*

Stoe IPDS diffractometer
$\varphi$-oscillat., $\varphi$-incr. = 1.1°, 218 exposure scans
Absorption correction: numerical (*NUMABS*32 in *XRED*1.08; Stoe & Cie, 1997)
$T_{\min} = 0.446$, $T_{\max} = 0.761$
47 419 measured reflections

18 050 independent reflections
15173 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.034$
$\theta_{\max} = 25.5°$
$h = -15 \rightarrow 15$
$k = -22 \rightarrow 22$
$l = -27 \rightarrow 27$

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.053$
$S = 0.94$
18050 reflections
1262 parameters
H-atom parameters not refined

$w = 1/[\sigma^2(F_o^2) + (0.0344P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = 0.020$
$\Delta\rho_{\max} = 0.70$ e Å$^{-3}$
$\Delta\rho_{\min} = -1.27$ e Å$^{-3}$
Extinction correction: *SHELXL*97
Extinction coefficient: 0.00038 (5)

Data collection: *IPDS*2.87 (Stoe & Cie, 1997); cell refinement: *IPDS*2.87; data reduction: *IPDS*2.87; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 1996); software used to prepare material for publication: *SHELXL*97.

*Acta Cryst.* (2001). E**57**, m338–m340 Alexander Filippou *et al.* • $C_{54}H_{48}O_2P_4W\cdot 1.5(C_6H_6)$ **m339**

## References

Brandenburg, K. (1996). *DIAMOND*. Crystal Impact GbR, Bonn, Germany.

Chatt, J. & Watson, H. R. (1961). *J. Chem. Soc.* pp. 4980–4988.

Filippou, A. C., Philippopoulos, A. I., Portius, P. & Neumann, D. U. (2000). *Angew. Chem.* **112**, 2881–2884; *Angew. Chem. Int. Ed.* **39**, 2778–2781.

Ishida, T., Mizobe, Y., Tanase, T. & Hidai, M. (1991). *J. Organomet. Chem.* **409**, 355–365.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.

Stoe & Cie (1997). *IPDS*2.87, *XRED*1.08 and *XSTEP*32. Stoe & Cie, Darmstadt, Germany.

**m340** Alexander Filippou *et al.* • $C_{54}H_{48}O_2P_4W \cdot 1.5(C_6H_6)$ *Acta Cryst.* (2001). E**57**, m338–m340