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Pentane and tetrahydrofuran solvates of *trans*-dichlorobis[1,2-ethanediylbis(diphenylphosphine)-*P*,*P*′]-molybdenum(II)

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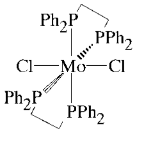
trans-[$MoCl_2$(dppe)$_2$] [dppe is 1,2-ethanediylbis(diphenylphosphine), $C_{26}H_{24}P_2$] was obtained as a side product from the reaction of *trans*-[Mo(dppe)$_2$(N_2)$_2$] with Cp*GeCl to give the germylyne complex *trans*-[Cl(dppe)$_2$Mo$\equiv$Ge(η^1-Cp*)]. The crystal structures of the hemipentane ($0.5C_5H_{12}$) and ditetrahydrofuran ($2C_4H_8O$) solvates of *trans*-[$MoCl_2$(dppe)$_2$], (III*a*) and (III*b*), respectively, have been determined.

Comment

We have shown recently that reaction of the dinitrogen complex *trans*-[W(dppe)$_2$(N_2)$_2$] (dppe is $Ph_2PCH_2CH_2PPh_2$) with the halogermylenes Cp*Ge*X* (Cp* = C_5Me_5; *X* = Cl, Br, I) affords selectively the germylyne complexes *trans*-[*X*(dppe)$_2$W$\equiv$Ge(η^1-Cp*)] (Filippou *et al.*, 2000). We wanted to extend this chemistry to related molybdenum compounds and therefore studied the reaction of *trans*-[Mo(dppe)$_2$(N_2)$_2$], (I), with Cp*GeCl in boiling toluene. Elimination of both dinitrogen ligands occurs from (I), leading to a mixture of three phosphorous-containing products. Two of these products were identified by ^1H, $^{31}P\{^1H\}$ and $^{13}C\{^1H\}$ NMR spectroscopy to be the germylyne complex *trans*-[Cl(dppe)$_2$Mo$\equiv$Ge(η^1-Cp*)], (II), and the Mo^{II} compound *trans*-[Mo(dppe)$_2Cl_2$], (III). Attempts to separate (II) from (III) by fractional crystallization led to single crystals of the less soluble title compound (III) as the hemipentane, (III*a*), and ditetrahydrofuran, (III*b*), solvates, which crystallize in different space groups (*C*2/*c* and *P*2$_1$/*c*, respectively). The known solvate *trans*-[Mo(dppe)$_2$$Cl_2$]·$CH_2Cl_2$ crystallizes in *P*2$_1$/*n* (Nardelli *et al.*, 1980). The bonding parameters of the three structures differ significantly.



·*X*

(III*a*) $X = 0.5C_5H_{12}$
(III*b*) $X = 2C_4H_8O$

Experimental

An equimolar mixture of (I) and Cp*GeCl was heated in gently boiling toluene. The colour of the solution changed from orange to dark-red. An IR spectrum of the reaction solution (2200–1500 cm^{-1}) revealed that the $\nu^1(N_2)$ absorptions of (I) at 2040 (*w*) and 1977 (*vs*) cm^{-1} had disappeared. The solution was evaporated to dryness and the residue washed with pentane. Yellow crystals of (III*a*) were obtained upon diffusion of pentane into a toluene solution of the residue at ambient temperature. Single crystals of (III*b*) were obtained from a THF solution of the residue upon diffusion of a diethyl ether/pentane mixture.

Compound (III*a*)

Crystal data

[$MoCl_2(C_{26}H_{24}P_2)_2$]·$0.5C_5H_{12}$
M_r = 999.70
Monoclinic, *C*2/*c*
a = 49.542 (11) Å
b = 10.958 (3) Å
c = 18.123 (4) Å
β = 100.03 (3)°
V = 9688 (4) Å^3
Z = 8
D_x = 1.371 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 5000 reflections
θ = 5.0–20.3°
μ = 0.550 mm^{-1}
T = 180 (2) K
Plate, yellow
0.24 × 0.20 × 0.04 mm

Data collection

Stoe IPDS diffractometer
Method: φ-oscill., φ-incr. = 1.3°, 183 exposure
Absorption correction: refdelf (*ABSCOR*; Stoe & Cie, 1997)
T_{min} = 0.879, T_{max} = 0.978
29 774 measured reflections
8494 independent reflections
2986 reflections with $I > 2\sigma(I)$
R_{int} = 0.099
θ_{max} = 25.05°
$h = -58 \rightarrow 58$
$k = -13 \rightarrow 13$
$l = -21 \rightarrow 21$
Intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.074
$wR(F^2)$ = 0.157
S = 0.801
8494 reflections
555 parameters
H-atom parameters not refined
$w = 1/[\sigma^2(F_o^2) + (0.0326P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.028
$\Delta\rho_{max}$ = 1.24 e Å^{-3}
$\Delta\rho_{min}$ = −1.09 e Å^{-3}

Compound (III*b*)

Crystal data

[$MoCl_2(C_{26}H_{24}P_2)_2$]·$2C_4H_8O$
M_r = 1107.83
Monoclinic, *P*2$_1$/*c*
a = 11.3197 (11) Å
b = 13.4052 (14) Å
c = 17.455 (2) Å
β = 96.093 (11)°
V = 2633.7 (4) Å^3
Z = 2
D_x = 1.397 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 26 reflections
θ = 13.5–16.4°
μ = 0.516 mm^{-1}
T = 180 (2) K
Block, brown
0.80 × 0.54 × 0.28 mm

Data collection

Stoe Stadi-4 diffractometer
Method: 2θ/ω scans, ratio = 1.0, width (ω) = 1.2–1.68°
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.683, T_{max} = 0.869
6801 measured reflections
4617 independent reflections
4065 reflections with $I > 2\sigma(I)$
R_{int} = 0.032
θ_{max} = 24.97°
$h = -13 \rightarrow 13$
$k = 0 \rightarrow 15$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 120 min
intensity decay: 8.6%

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Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0565P)^2 + 1.2270P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.033$	$(\Delta/\sigma)_{\max} = 0.009$
$wR(F^2) = 0.094$	$\Delta\rho_{\max} = 0.80$ e Å^{-3}
$S = 1.068$	$\Delta\rho_{\min} = -0.85$ e Å^{-3}
4617 reflections	
313 parameters	
H-atom parameters not refined	

It is remarkable that the pentane solvent molecule of (III*a*) could not be localized in the difference map until the absorption correction was carried out. The calculation was carried out with *ABSCOR* (Stoe & Cie, 1997), a modification of *DIFABS* (Walker & Stuart, 1983). In contrast to *DIFABS, ABSCOR* loads and provides F^2 instead of F values. For a better convergence in the following least-squares cycles, geometrical restraints had to be applied to the disordered solvent.

For compound (III*a*), 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: *XSTEP*-2.18 (Stoe & Cie, 1997); software used to prepare material for publication: *SHELXL*97. For compound (III*b*), data collection: *STADI*4-1.06 (Stoe & Cie, 1997); cell refinement: *STADI*4-1.06; data reduction: *XRED*-1.07 (Stoe & Cie, 1997); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *XSTEP*-2.18 (Stoe & Cie, 1997); software used to prepare material for publication: *SHELXL*97.

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