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Reactions of the sixteen electron compound $[MoBr_2(CO)_2(PPh_3)_2]$ with N- and P-donor ligands. Crystal structure of $[MoBr_2(CO)_2(dppm)(PPh_3)] \cdot 0.4CHCl_3$

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Abstract

The reactions of $[MoBr_2(CO)_2(PPh_3)_2]$ with N- and P-donor ligands [pyridine (py), NR_3 (R = H, Et, Bu^n), 2,2′-bipyridyl (bipy), PR_3 (R = H, Et), $P(OR)_3$ (R = Me, Et), $P(OCH_2)_3CCH_3$, and $Ph_2PCH_2PPh_2$ (dppm)] have been shown to give the new mixed ligand compounds $[MoBr_2(CO)_2(bipy)(PPh_3)]$, $[MoBr_2(CO)_2(PH_3)_2(PPh_3)]$, $[MoBr_2(CO)_2(dppm)(PPh_3)]$ and the cationic complex $[MoBr(CO)_2[P(OCH_2)_3CCH_3]_4]Br$. Further reaction of $[MoBr_2(CO)_2(dppm)(PPh_3)]$ with $P(OMe)_3$ leads to $[MoBr_2(CO)_2(dppm)\ P(OMe)_3]$. The structure of $[MoBr_2(CO)_2(dppm)(PPh_3)]$ has been established by X-ray diffraction. ^{31}P NMR studies on $[MoBr_2(CO)_2(dppm)L]$ (L = $P(OMe)_3$, PPh_3) have revealed fluxional behaviour in solution at room temperature.

Introduction

Seven-coordinate complexes of molybdenum(II) are well known [1]. Some six-coordinate compounds have been reported [2]. In an earlier paper [3] we described the preparation of several molybdenum(II) complexes containing anionic O- or S-donor ligands, exploiting the reactivity of the sixteen electron $[MoBr_2(CO)_2(PPh_3)_2]$ [4] to produce seven-coordinate eighteen electron complexes. In an attempt to prepare new mixed ligand complexes of molybdenum(II) we have studied the behaviour of $[MoBr_2(CO)_2(PPh_3)_2]$ towards mono- and bi-dentate N- or P-donor neutral ligands. Depending on the electronic and steric properties of the ligands different results are obtained. In some cases there is total replacement of PPh_3 by the new ligand, whereas in other cases mixed ligand complexes containing PPh_3 and another different N- or P-donor ligand are obtained.

Results and discussion

(a) Reactions with N-donor ligands. Treatment of $[MoBr_2(CO)_2(PPh_3)_2]$ with pyridine in dichloromethane at room temperature led to $[MoBr_2(CO)_2(Py)_2]$ [5], which had been previously prepared by reaction of $[MoBr_2(CO)_4]_2$ with pyridine. No mixed triphenylphosphine-pyridine species were detected in solution when various molar ratios of reactants were used.

Formation of *trans*-$[Mo(CO)_4(PPh_3)_2]$ was observed when more basic amines NR_3 ($R = H$, Et, Bu^n) were used. This effect had been observed previously [3] and seems to be associated to the hard base character of the ligands.

The reaction with 2,2′-bipyridyl gave different results depending on the molar ratio of reactants. Thus $[MoBr_2(CO)_2(bipy)(PPh_3)]$ and $[MoBr(CO)_2(bipy)_2]Br$ were obtained by use of 1/1 and 1/2 molar ratios, respectively. The ionic compound had been previously prepared [5] by treatment of $[MoBr_2(CO)_4]_2$ with 2,2′-bipyridyl. On the other hand formation of $[MoBr_2(CO)_2(bipy)(PPh_3)]$ was observed when $[MoBr_2(CO)_3(bipy)]$ was treated with PPh_3 [6].

(b) Reactions with P-donor ligands. When PH_3 was passed through a blue suspension of $[MoBr_2(CO)_2(PPh_3)_2]$ in dichloromethane at room temperature a yellow solution was quickly formed. When hexane was added while the flow of PH_3 was continued a precipitate analyzing as $[MoBr_2(CO)_2(PH_3)_2(PPh_3)]$ was obtained. However, attempts to isolate the compound by concentration of the dichloromethane solution led to regeneration of the starting compound. The equilibrium of eq. 1 was present in this reaction and so, elimination of PPh_3 was necessary for

$$\underset{\text{(blue)}}{\left[MoBr_2(CO)_2(PPh_3)_2\right]} + 2PH_3 \rightleftharpoons \underset{\text{(yellow)}}{\left[MoBr_2(CO)_2(PH_3)_2(PPh_3)\right]} + PPh_3 \qquad (1)$$

isolation of the mixed PH_3/PPh_3 compound. The latter complex reacted with PPh_3 to give the original blue complex. Total replacement of PPh_3 by PH_3 has not been observed.

The reaction with PEt_3 was different, showing the importance of the steric hindrance in attempt to generate six- or seven-coordinate compounds. Formation of only $[MoBr_2(CO)_2(PEt_3)_2]$ [7] was detected by IR spectroscopy.

Treatment of $[MoBr_2(CO)_2(PPh_3)_2]$ with $P(OMe)_3$ or $P(OEt)_3$ gave the known $[MoBr_2(CO)_2\{P(OR)_3\}_3]$ [8], but no mixed phosphine/phosphite complexes were detected. Use of $P(OCH_2)_3CCH_3$, with a cone angle significantly smaller than the previous phosphites, gave the new $[MoBr(CO)_2\{P(OCH_2)_3CCH_3\}_4]Br$ even when less than an equivalent amount of phosphite was used.

Reaction of $[MoBr_2(CO)_2(PPh_3)_2]$ with dppm in molar ratio 1/2 gave the seven-coordinate $[MoBr_2(CO)_2(dppm)_2]$ [9]. Use of 1/1 molar ratios gave the mixed $[MoBr_2(CO)_2(dppm)(PPh_3)]$. This complex reacted with $P(OMe)_3$ to give the more soluble $[MoBr_2(CO)_2(dppm)\{P(OMe)_3\}]$.

Table 1

Analytical, conductivity and IR data

Compound	ν(CO) [a] (cm^{-1})	Analysis (Found (calc.) (%)) C	H	N	Λ ($S\ cm^2\ mol^{-1}$) [b]
$[MoBr_2(CO)_2(bipy)(PPh_3)]$	1945, 1860	49.0 (49.3)	3.2 (3.2)	3.9 (3.8)	3.8
$[MoBr_2(CO)_2(PH_3)_2(PPh_3)]$	1960, 1882	37.9 (37.4)	3.4 (3.3)		3.6
$[MoBr(CO)_2\{P(OCH_2)_3CCH_3\}_4]Br$	2000, 1922	29.0 (29.2)	4.1 (4.0)		124.0
$[MoBr_2(CO)_2(dppm)(PPh_3)]$	1942, 1865	56.2 (56.4)	4.0 (3.9)		4.0
$[MoBr_2(CO)_2(dppm)\{P(OMe)_3\}]$	1955, 1878	43.5 (43.9)	3.8 (3.8)		3.5

[a] In dichloromethane solution. [b] In acetone ca. 10^{-3} *M*.

Analytical, conductivity and IR data are listed in Table 1.

(c) ^{31}P NMR *. Temperature dependence was revealed for $[MoBr_2(CO)_2(dppm)(PPh_3)]$ when $^{31}P\{^1H\}$ NMR measurements were carried out in $CDCl_3$ solution. However attempts to record NMR spectra at low temperature were unsuccessful because the compound was not sufficiently soluble. For a study of the fluxional behaviour of these seven-coordinate complexes in solution, the analogous but very soluble $[MoBr_2(CO)_2(dppm)\{P(OMe)_3\}]$ was employed. At −40 °C an AMX pattern (δ −33.0dd, +20.0dd, +140.6dd; J_{AM} 70.8 Hz, J_{MX} 29.3 Hz, J_{AX} 280.8 Hz) was found. At room temperature there is exchange between P_A and P_M which broaden their signals so much that they are not seen (near to coalescence) and the signal of P_X becomes a triplet with a broad central line (δ +140.6 ppm). The triphenylphosphine derivative showed a similar triplet (δ +32.8 ppm) at room temperature.

(d) X-Ray structure study of $[MoBr_2(CO)_2(dppm)\ (PPh_3)] \cdot 0.4\ CHCl_3$. The results are summarized in Figs. 1 and 2, and Tables 2 to 5. The coordination sphere about the central Mo atom cannot be described exactly in terms of any of the idealized seven-coordinate geometries, capped octahedron (C.O.), pentagonal bipyramid (P.B.), and capped trigonal prism (C.T.P.). This can be seen by comparing the observed L–Mo–L angles (Table 2) with those for the idealized polyhedra. The average angular deviations from the three idealized geometries are given at the bottom of Table 2 [10]. From these data it seems that the C.O. and C.T.P. (or an intermediate) geometries account for the actual structure better than the P.B. geometry. The same result is observed in other complexes of the type [M(unidentate)$_5$(bidentate)] [11], for which the C.O. geometry is found when the bidentate ligand is a five membered ring species and distortions towards a C.T.P. structure arise if the bidentate ligand is a four membered ring.

The C.T.P. geometry shown in Fig. 2 has the two bromo ligands in positions consistent with the view of Hoffmann et al. [12] that the most electronegative

(Continued on p. 304)

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* Proton decoupled, positive shifts to high frequency of external 85% H_3PO_4.

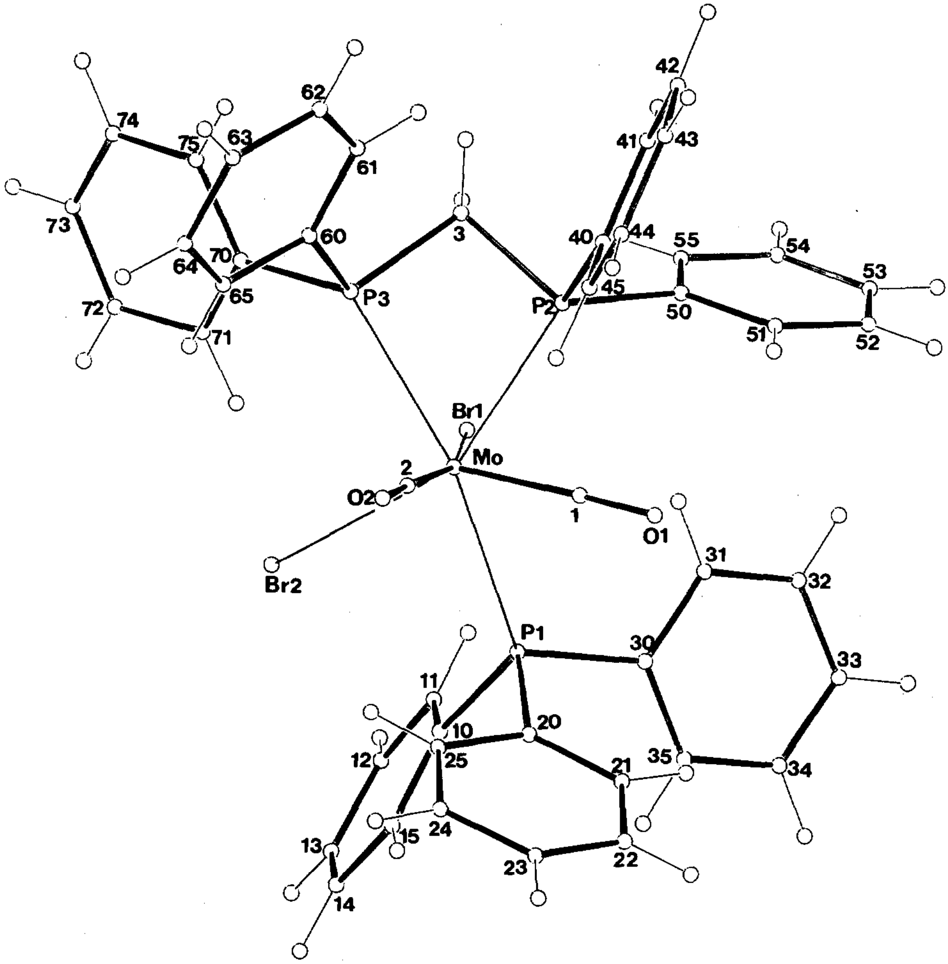
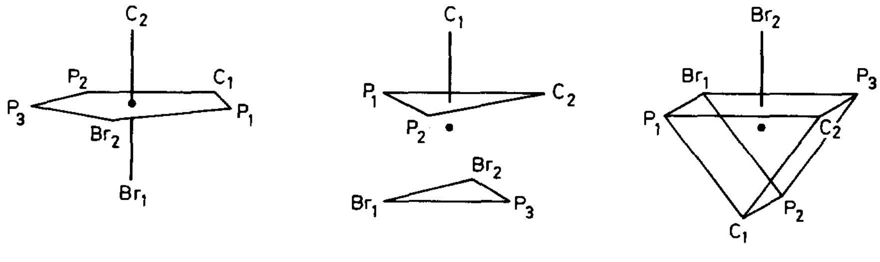


Fig. 1. The molecular structure of $[MoBr_2(CO)_2(dppm)(PPh_3)]$ with the atomic numbering scheme.



P.B. C.O. C.T.P.

Fig. 2. Arrangement of the atoms in the three idealized geometries.

Table 2

L–Mo–L bond angles (deg) grouped by reference to the idealized geometries (see Fig. 2).

	Observed	"ideal" CTP	"ideal" CO	"ideal" PB
Br(2)–Mo–Br(1)	90.4(1)	82	89.6	90
Br(2)–Mo–P(3)	87.8(1)	82	89.6	72
Br(2)–Mo–C(2)	77.9(2)	82	76.6	90
Br(2)–Mo–P(1)	79.3(1)	82	76.6	72
Br(2)–Mo–C(1)	135.2(2)	144.2	125.5	144
Br(2)–Mo–P(2)	151.1(1)	144.2	160.4	144
Br(1)–Mo–P(3)	83.1(1)	99	89.6	90
C(2)–Mo–P(1)	102.5(2)	99	112.8	90
Br(1)–Mo–P(1)	83.9(1)	78.7	76.6	90
C(2)–Mo–P(3)	87.4(2)	78.7	76.6	90
Br(1)–Mo–C(2)	165.3(2)	164	160.4	180
P(1)–Mo–P(3)	161.5(1)	164	160.4	144
C(1)–Mo–C(2)	76.5(3)	75.1	74.1	90
C(1)–Mo–P(1)	71.2(2)	75.1	74.1	72
P(2)–Mo–Br(1)	79.6(1)	75.1	76.6	90
P(2)–Mo–P(3)	64.3(1)	75.1	76.6	72
C(1)–Mo–Br(1)	118.2(2)	118.9	125.5	90
C(1)–Mo–P(3)	126.8(2)	118.9	125.5	144
P(2)–Mo–P(1)	125.8(1)	118.9	112.8	144
P(2)–Mo–C(2)	106.3(2)	118.9	112.8	90
C(1)–Mo–P(2)	72.3(2)	71.5	74.1	72
Average angular deviation		6.1%	5.7%	10.5%

Table 3

Selected geometrical parameters (Å,°)

Mo–Br(1)	2.681(1)	Mo–C(2)	1.940(7)	P(2)–C(3)	1.836(5)
Mo–Br(2)	2.665(1)	C(1)–O(1)	1.171(6)	P(2)–C(40)	1.825(6)
Mo–P(1)	2.578(2)	C(2)–O(2)	1.160(10)	P(2)–C(50)	1.813(6)
Mo–P(2)	2.503(1)	P(1)–C(10)	1.837(5)	P(3)–C(3)	1.836(5)
Mo–P(3)	2.593(2)	P(1)–C(20)	1.834(6)	P(3)–C(60)	1.831(8)
Mo–C(1)	1.927(5)	P(1)–C(30)	1.843(6)	P(3)–C(70)	1.832(6)

Br(1)–Mo–P(1)–C(10)	−60.1(2)	Mo–P(2)–C(3)–P(3)	25.5(2)
Br(1)–Mo–P(1)–C(20)	−179.3(2)	Mo–P(2)–C(40)–C(41)	−169.8(4)
Br(1)–Mo–P(1)–C(30)	60.4(2)	Mo–P(2)–C(50)–C(51)	−88.1(5)
Br(1)–Mo–P(2)–C(3)	67.6(2)	Mo–P(3)–C(3)–P(2)	−24.4(2)
Br(1)–Mo–P(2)–C(40)	177.2(2)	Mo–P(3)–C(60)–C(61)	104.8(6)
Br(1)–Mo–P(2)–C(50)	−53.6(2)	Mo–P(3)–C(70)–C(71)	2.9(8)
Br(1)–Mo–P(3)–C(3)	−62.2(2)	C(1)–P(1)–Br(2)–P(3)	31.9(1)
Br(1)–Mo–P(3)–C(60)	−174.8(3)	P(1)–Br(2)–P(3)–P(2)	−3.6(1)
Br(1)–Mo–P(3)–C(70)	50.1(3)	Br(2)–P(3)–P(2)–C(1)	−27.1(2)
Br(2)–Mo–P(1)–C(30)	152.0(2)	P(3)–P(2)–C(1)–P(1)	68.7(2)
C(1)–Mo–P(1)–C(30)	−62.2(3)	P(2)–C(1)–P(1)–Br(2)	−66.9(2)
C(2)–Mo–P(1)–C(30)	−133.1(3)	P(1)–Br(2)–P(3)–C(3)	29.6(3)
Mo–P(1)–C(30)–C(31)	−0.6(6)	Br(2)–P(3)–C(3)–P(2)	−52.2(4)
Mo–P(1)–C(20)–C(21)	−121.8(5)	P(3)–C(3)–P(2)–C(1)	12.7(5)
Mo–P(1)–C(10)–C(11)	69.9(5)	P(3)–P(2)–C(1)–P(1)	60.0(5)

Table 4

Crystal data, experimental and refinement parameters

Crystal data	
Formula	$C_{45}H_{37}P_3O_2Br_2Mo \cdot 0.4(CHCl_3)$
Crystal habit	Yellow transparent prism
Crystal size (mm)	0.33 × 0.03 × 0.06
Symmetry	Triclinic, $P\bar{1}$
Unit cell determination:	Least-squares fit from 66 reflexions ($\theta < 45$)
Unit cell dimensions (Å)	18.231(3), 11.097(2), 11.932(2)
(°)	112.07(1), 89.96(1), 82.74(2)
Packing: V(Å^3), Z	2216.1(7), 2
D_c(g cm^{-3}), M, $F(000)$	1.51, 1006.26, 1006
μ (cm^{-1})	66.79
Experimental data	
Technique	Four circle diffractometer: PW1100 Bisecting geometry Graphite oriented monochromator; Cu-K_α $\omega/2\theta$ scans, scan width 1.5° Detector apertures 1 × 1, θ_{max} 60° 1 min/reflex.
Number of reflexions:	
Measured	6601
Independent	6601
Observed	6124 [3 $\sigma(I)$ criterion]
Standard reflexions:	2 reflexions every 90 min Variation: none
Max-min transmission factors:	1.43–0.67
Solution and refinement	
Solution	Patterson
Refinement	L.s. on F_{obs} with 3 blocks
Parameters:	
Number of variables	653
Degrees of freedom	5471
Ratio of freedom	9.4
H atoms	Difference synthesis
Final shift/error	0.04
ω-scheme	Empirical as to give no trends in $\langle w\Delta^2F \rangle$ vs. $\langle F_0 \rangle$ and $\langle \sin\theta/\lambda \rangle$
Max. thermal value	U_{11}(C(13)) 0.38(4) Å^2
Final ΔF peaks	2.5 e Å^{-3}
Final R and R_w	0.059, 0.067
Computer and programs	VAX 11/750, DIFABS[17], XRAY[18]
Scattering factors	Int. Tables for X-Ray Crystallography [19]

π-donor is in the capping position and the other π-donor in the next most favorable position, the quadrilateral face.

The C.O. structural aproximation corresponds to satisfactory positions for all the atoms. The carbonyl ligands are in the capping and in the capped face positions in

Table 5. Atomic coordinates

Atom	x	y	z
Mo	0.74405(2)	0.34064(4)	0.19679(3)
Br(1)	0.79449(3)	0.19318(6)	0.32145(6)
Br(2)	0.81414(3)	0.16320(6)	−0.00489(6)
P(2)	0.74531(7)	0.51448(12)	0.40405(11)
P(1)	0.64905(7)	0.17735(12)	0.12543(11)
P(3)	0.86800(7)	0.43503(13)	0.25240(12)
C(1)	0.64579(30)	0.43693(53)	0.23899(46)
O(1)	0.58640(22)	0.49694(41)	0.25980(42)
C(2)	0.73255(31)	0.43147(59)	0.08578(49)
O(2)	0.72364(33)	0.48876(51)	0.02213(44)
C(3)	0.84616(28)	0.50050(54)	0.41632(46)
C(10)	0.68177(28)	0.00217(48)	0.04240(47)
C(11)	0.71820(36)	−0.06761(61)	0.10435(55)
C(12)	0.74046(36)	−0.20063(61)	0.05074(67)
C(13)	0.72672(38)	−0.26636(57)	−0.06934(63)
C(14)	0.69054(44)	−0.20021(66)	−0.13234(56)
C(15)	0.66777(37)	−0.06515(59)	−0.07724(52)
C(20)	0.58750(32)	0.22079(51)	0.02089(49)
C(21)	0.51097(35)	0.24949(67)	0.04168(59)
C(22)	0.46793(41)	0.28481(84)	−0.03982(79)
C(23)	0.50093(51)	0.28830(84)	−0.14361(83)
C(24)	0.57624(44)	0.25595(72)	−0.16594(69)
C(25)	0.61899(36)	0.22372(61)	−0.08228(61)
C(30)	0.58790(28)	0.16726(50)	0.24379(50)
C(31)	0.59337(32)	0.24216(54)	0.36383(52)
C(32)	0.54880(38)	0.22911(70)	0.45212(56)
C(33)	0.49789(38)	0.14282(70)	0.42018(65)
C(34)	0.49182(40)	0.06662(72)	0.29782(75)
C(35)	0.53714(36)	0.07624(60)	0.21126(57)
C(40)	0.72012(31)	0.68802(51)	0.43147(51)
C(41)	0.73341(41)	0.78053(69)	0.54393(58)
C(42)	0.71781(53)	0.91250(69)	0.56988(76)
C(43)	0.68824(53)	0.95582(69)	0.48347(89)
C(44)	0.67460(43)	0.86830(76)	0.37412(74)
C(45)	0.68974(33)	0.73455(59)	0.34536(55)
C(50)	0.69874(31)	0.50554(52)	0.53440(45)
C(51)	0.62444(34)	0.55965(60)	0.55960(53)
C(52)	0.58486(40)	0.55202(65)	0.65567(61)
C(53)	0.61887(48)	0.49127(65)	0.72605(63)
C(54)	0.69185(51)	0.43627(70)	0.70275(61)
C(55)	0.73303(37)	0.44290(62)	0.60569(52)
C(60)	0.88597(29)	0.57263(64)	0.21196(59)
C(61)	0.88244(45)	0.70067(73)	0.29246(75)
C(62)	0.89728(58)	0.79900(102)	0.25404(103)
C(63)	0.91756(54)	0.76823(111)	0.13728(112)
C(64)	0.92266(51)	0.64058(144)	0.05570(106)
C(65)	0.90553(40)	0.54315(104)	0.09300(84)
C(70)	0.95936(30)	0.33682(63)	0.23022(53)
C(71)	0.96983(58)	0.20449(94)	0.17726(96)
C(72)	1.04030(48)	0.13308(107)	0.15887(104)
C(73)	1.09922(40)	0.19656(102)	0.19925(83)
C(74)	1.09036(38)	0.33031(89)	0.25442(73)
C(75)	1.02027(36)	0.40185(79)	0.26973(64)
Cl(1) [a]	0.89209(58)	0.32603(146)	0.74630(154)
Cl(2) [a]	0.95763(150)	0.22981(285)	0.52431(277)
Cl(3) [a]	0.86265(215)	0.11901(243)	0.58822(247)

[a] Atoms with population parameter of 0.40(2).

accord with predictions based on the electronic effects of π-acceptors and with structures of several other species [11].

The bond distances (Table 3) are normal for the donor atoms involved.

The overall conformation of the complex is shown in Table 3 by means of the torsion angles. The arrangement of the substituents around each of the phosphorus angles is significantly distorted from the $C3$ symmetry as judged from the Bye, Schweizer and Dunitz model [13] without symmetrization ($d1$ 50.9°, $d2$ 43.4° and $d3$ 39.7°).

Experimental

All preparations were carried out under nitrogen by conventional Schlenk techniques. Solvents were dried and degassed before use. The compound $[MoBr_2(CO)_2(PPh_3)_2]$ [4] and the ligands PH_3 [14] and $P(OCH_2)_3CCH_3$ [15] were prepared by published methods. Other reagents were purchased from commercial sources and used as received.

Preparation of $[MoBr_2(CO)_2(bipy)(PPh_3)]$. To a suspension of $[MoBr_2(CO)_2(PPh_3)_2]$ (0.2 g, 0.24 mmol) in 20 cm^3 of dichloromethane was added 2,2′-bipyridyl (0.037 g, 0.24 mmol). After 1 h stirring the solution was filtered. Addition of hexane (ca. 20 cm^3) and concentration, afforded the complex as orange crystals. Yield 0.15 g (86%).

Preparation of $[MoBr_2(CO)_2(PH_3)_2(PPh_3)]$. A stream of PH_3 was passed through a suspension of $[MoBr_2(CO)_2(PPh_3)_2]$ (0.2 g, 0.24 mmol) in 10 cm^3 of dichloromethane. The orange-yellow solution formed was poured into a flask containing 50 cm^3 of hexane while the passage of phosphine through the mixture was maintained. The yellow precipitate was washed with hexane and dried. Yield 0.12 g (78%).

Preparation of $[MoBr(CO)_2\{P(OCH_2)_3CCH_3\}_4]Br$. To a suspension of $[MoBr_2(CO)_2(PPh_3)_2]$ (0.2 g, 0.24 mmol) in 20 cm^3 of dichloromethane was added $P(OCH_2)_3CCH_3$ (0.14 g, 0.96 mmol). After 15 min stirring the mixture was filtered. Addition of hexane (ca. 20 cm^3) and concentration gave the product as yellow crystals. Yield 0.22 g. (90%).

Preparation of $[MoBr_2(CO)_2(dppm)(PPh_3)]$. This was prepared in the way as described for the preceding compound but by use of dppm (0.092 g, 0.24 mmol) and with stirring for 2 h. Yield 0.2 g, (90%).

Preparation of $[MoBr_2(CO)_2(dppm)\ P(OMe)_3]$. To a solution of $[MoBr_2(CO)_2(dppm)(PPh_3)]$ (0.2 g, 0.21 mmol) in 20 cm^3 of dichloromethane was added trimethylphosphite (0.03 g, 0.24 mmol). Filtration, addition of hexane and concentration, gave the compound as yellow crystals. Yield 0.15 g (86%).

Crystal structure determination

Yellow crystals of $[MoBr_2(CO)_2(dppm)(PPh_3)] \cdot 0.4CHCl_3$ were grown from chloroform/hexane. Table 4 lists the details of the diffraction study. The $CHCl_3$ molecule has an occupancy of 0.40(2) and its C atom could not be located. Table 5 shows the final atomic coordinates. Lists of structure factors, thermal parameters, and the hydrogen coordinates can be obtained from the authors on request.

Crystal data. The complex crystallizes in the $P\bar{1}$ space group, with lattice constants of a 18.231(3), b 11.097(2), c 11.932(2) Å, α 112.07(1), β 89.96(1) and γ 82.74(2) and $Z = 2$ with 0.80(2) $CHCl_3$ molecules in the unit cell.

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