routine Patterson (*SHELX*76; Sheldrick, 1976) and direct methods (*MULTAN*88; Debaerdemaeker *et al.*, 1988) failed because of an inability to determine the Cr-atom positions uniquely, while the P atoms in the cations could be located easily. The structures could only be solved by a painstakingly meticulous interpretation of the Patterson maps coupled with judicious symmetry considerations. The P atoms in the cations were placed at the origin while the two anions in the unit cell of each compound were statistically distributed among the eight possible sites leading to fourfold disordering of the Cr-atom environments. Full-matrix least-squares refinements on F^2 using *SHELXL*93 (Sheldrick, 1994) with anisotropy for Cr, P and phenyl C atoms and isotropic displacement parameters for O atoms with fractional occupancies were carried out. H atoms were placed in geometrically calculated positions and were not refined. Program used for geometric calculations: *PARST* (Nardelli, 1983). Software used to prepare material for publication: *SHELXL*93. The calculations were performed using a VAX3400 computer at the Computer Centre, Indian Association for the Cultivation of Science.

The authors wish to thank Professor R. G. Bhattacharya, Jadavpur University, for providing the crystals and many helpful discussions. They are also indebted to Professors R. Bhattacharya, D. Ghosh and A. Pal, IACS, Calcutta, for magnetic susceptibility and EPR studies.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HE1008). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C50, 1404–1406

Bis[tris(tetrahydrofuran)lithium(1+)] Bis(μ-diphenylphosphanido)bis(tetracarbonylmolybdate)(2−)

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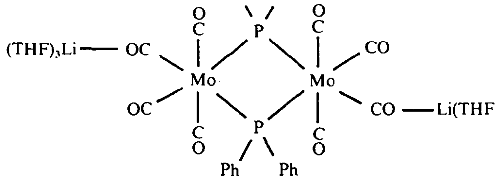
(*Received* 12 *October* 1993; *accepted* 16 *February* 1994)

Abstract

In the dianion $[Mo_2(\mu\text{-}PPh_2)_2(CO)_8]^{2-}$, two Mo centres are 4.100 (1) Å apart and are bridged asymmetrically by two diphenylphosphanido ligands. Each metal atom is also coordinated by four terminal CO ligands. There are strong interactions between the dianion and the two $[Li(thf)_3]^+$ cations (thf = C_4H_8O). The Li ions are ligated by one equatorial carbonyl O atom from each Mo centre.

Comment

Binuclear transition metal complexes $[M_2(\mu\text{-}PR_2)_2(CO)_8]$ (M = Cr, Mo, W) have been investigated extensively with respect to their synthesis, bonding, chemical and electrochemical properties (Linck & Nassimbeni, 1973; Treichel, Dean & Douglas, 1972; Keiter *et al.*, 1992). It has been demonstrated that the complex $[W_2(\mu\text{-}PPh_2)_2(CO)_8]$ undergoes two-electron reduction with cleavage of the M—M bond to give the dianion $[W_2(\mu\text{-}PPh_2)_2(CO)_8]^{2-}$ (Shyu, Calligaris, Nardin & Wojcicki, 1987). We have prepared the lithium salt of $[Mo_2(\mu\text{-}PPh_2)_2(CO)_8]^{2-}$ by reaction of $[Mo(CO)_4(Ph_2PLi)_2]$ with $SmCl_3$ in thf. Yellow crystals of $[Li(thf)_3]_2[Mo_2(\mu\text{-}PPh_2)_2(CO)_8]$, (I), suitable for X-ray analysis were obtained by cooling a saturated solution of thf. The present structure is isomorphous with its tungsten analogue



(I)

Acta Crystallographica Section C
ISSN 0108-2701 ©1994

(Shyu, Calligaris, Nardin & Wojcicki, 1987), although we have used the $P2_1/n$ setting which gives a smaller β angle.

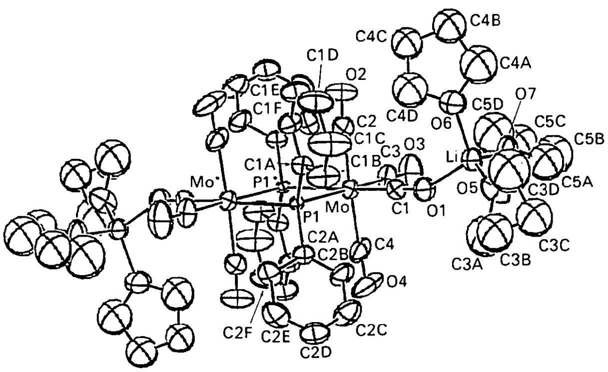


Fig. 1. The molecular structure of the title compound, with displacement ellipsoids plotted at the 35% probability level.

Experimental

Crystal data

$[\mathrm{Li}(C_4H_8O)_3]_2$-
$[\mathrm{Mo}_2(C_{12}H_{10}P)_2(CO)_8]$
$M_r = 1232.89$
Monoclinic
$P2_1/n$
$a = 11.278\ (5)$ Å
$b = 19.531\ (7)$ Å
$c = 14.204\ (2)$ Å
$\beta = 100.38\ (2)°$
$V = 3077.3\ (18)$ Å^3
$Z = 2$
$D_x = 1.330$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11\text{–}13°$
$\mu = 0.504$ mm^{-1}
$T = 293$ K
Block
0.48 × 0.42 × 0.24 mm
Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical
$T_{\min} = 0.929$, $T_{\max} = 0.999$
4695 measured reflections
4438 independent reflections

3194 observed reflections $[I > 3\sigma(I)]$
$R_{\mathrm{int}} = 0.024$
$\theta_{\max} = 23°$
$h = 0 \rightarrow 12$
$k = 0 \rightarrow 21$
$l = -15 \rightarrow 15$
3 standard reflections
frequency: 120 min
intensity variation: <2%

Refinement

Refinement on F
$R = 0.068$
$wR = 0.088$
$S = 3.918$
3194 reflections
283 parameters
H-atom parameters not refined
$w = 1/\{1 + [(F_o - 43.2)/142.7]^2\}$

$(\Delta/\sigma)_{\max} = 0.03$
$\Delta\rho_{\max} = 0.96$ e Å^{-3}
$\Delta\rho_{\min} = -0.58$ e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

B_{iso} for thf C atoms; $B_{\mathrm{eq}} = (8\pi^2/3)\sum_i\sum_j U_{ij}\, a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for others.

	x	*y*	*z*	$B_{\mathrm{iso}}/B_{\mathrm{eq}}$
Mo	−0.06721 (7)	−0.01679 (4)	0.12298 (6)	4.59 (2)
P1	−0.0938 (2)	0.0627 (1)	−0.0265 (2)	2.18 (5)
O1	−0.2560 (6)	0.0621 (4)	0.2151 (5)	7.4 (2)
O2	0.1365 (8)	0.0790 (5)	0.2349 (7)	9.6 (3)
O3	−0.027 (1)	−0.1126 (6)	0.3019 (7)	12.0 (3)
O4	−0.2781 (8)	−0.1161 (5)	0.029 (1)	13.2 (3)
O5	−0.4468 (8)	0.1561 (5)	0.2881 (6)	9.1 (2)
O6	−0.1690 (9)	0.1814 (6)	0.3530 (7)	10.3 (3)
O7	−0.2772 (8)	0.0518 (6)	0.4236 (7)	11.0 (3)
C1	−0.1857 (9)	0.0325 (5)	0.1802 (7)	5.5 (2)
C2	0.0643 (9)	0.0465 (6)	0.1916 (7)	5.8 (2)
C3	−0.039 (1)	−0.0775 (6)	0.2343 (8)	7.0 (3)
C4	−0.199 (1)	−0.0806 (6)	0.0623 (9)	7.6 (3)
C1*A*	−0.0827 (8)	0.1530 (5)	0.0099 (7)	4.8 (2)
C1*B*	0.0278 (9)	0.1812 (5)	0.0476 (8)	6.1 (3)
C1*C*	0.040 (1)	0.2490 (6)	0.080 (1)	8.0 (3)
C1*D*	−0.064 (1)	0.2897 (7)	0.078 (1)	9.9 (4)
C1*E*	−0.172 (1)	0.2619 (7)	0.045 (1)	12.7 (5)
C1*F*	−0.184 (1)	0.1926 (6)	0.011 (1)	9.2 (4)
C2*A*	−0.2472 (8)	0.0633 (5)	−0.0985 (7)	4.8 (2)
C2*B*	−0.3473 (9)	0.0467 (7)	−0.0590 (9)	7.7 (3)
C2*C*	−0.462 (1)	0.0489 (8)	−0.114 (1)	9.6 (4)
C2*D*	−0.480 (1)	0.0652 (7)	−0.207 (1)	8.4 (4)
C2*E*	−0.384 (1)	0.0818 (8)	−0.2474 (9)	8.7 (4)
C2*F*	−0.264 (1)	0.0831 (6)	−0.1933 (8)	7.0 (3)
C3*A*	−0.541 (3)	0.137 (2)	0.203 (2)	20 (1)
C3*B*	−0.608 (2)	0.209 (1)	0.190 (2)	17.1 (8)
C3*C*	−0.629 (2)	0.209 (1)	0.279 (2)	17.0 (8)
C3*D*	−0.481 (3)	0.225 (2)	0.308 (3)	24 (1)
C4*A*	−0.136 (2)	0.216 (1)	0.444 (2)	18.6 (9)
C4*B*	−0.016 (2)	0.249 (1)	0.446 (2)	14.5 (6)
C4*C*	0.022 (2)	0.230 (1)	0.357 (1)	12.8 (6)
C4*D*	−0.089 (2)	0.203 (1)	0.295 (2)	16.7 (8)
C5*A*	−0.349 (2)	0.035 (1)	0.482 (2)	17.8 (9)
C5*B*	−0.290 (3)	−0.024 (2)	0.539 (2)	21 (1)
C5*C*	−0.186 (2)	−0.040 (1)	0.512 (2)	18.0 (9)
C5*D*	−0.163 (4)	0.009 (2)	0.456 (3)	25 (1)
Li	−0.294 (2)	0.115 (1)	0.321 (1)	6.5 (5)

Table 2. *Selected geometric parameters* (Å, °)

C—C bond lengths are 1.34 (3)–1.44 (2) Å in the phenyl rings and 1.31 (5)–1.67 (4) Å in the thf rings.

Mo—P1	2.604 (3)	O3—C3	1.17 (2)
Mo—P1$^{\mathrm{i}}$	2.623 (3)	O4—C4	1.17 (1)
Mo—C1	1.95 (2)	O5—C3*A*	1.51 (3)
Mo—C2	2.04 (2)	O5—C3*D*	1.45 (4)
Mo—C3	1.96 (1)	O5—Li	1.88 (3)
Mo—C4	2.01 (1)	O6—C4*A*	1.46 (3)
P1—C1*A*	1.835 (9)	O6—C4*D*	1.40 (3)
P1—C2*A*	1.844 (9)	O6—Li	1.91 (2)
O1—C1	1.16 (1)	O7—C5*A*	1.30 (3)
O1—Li	1.93 (2)	O7—C5*D*	1.53 (4)
O2—C2	1.12 (1)	O7—Li	1.90 (2)
P1—Mo—C1	93.4 (3)	O6—Li—O7	108 (1)
P1—Mo—C2	89.7 (4)	C1—O1—Li	149.4 (9)
P1—Mo—C3	177.3 (4)	C3*A*—O5—C3*D*	102 (2)
P1—Mo—C4	93.0 (4)	C3*A*—O5—Li	125 (1)
C1—Mo—C2	89.8 (4)	C3*D*—O5—Li	128 (2)
C1—Mo—C3	89.1 (5)	C4*A*—O6—C4*D*	107 (2)
C1—Mo—C4	88.6 (5)	C4*A*—O6—Li	126 (1)
C2—Mo—C3	89.1 (4)	C4*D*—O6—Li	127 (1)
C2—Mo—C4	176.9 (5)	C5*A*—O7—C5*D*	105 (3)
C3—Mo—C4	88.2 (5)	C5*A*—O7—Li	132 (1)
P1—Mo—P1$^{\mathrm{i}}$	76.5 (2)	C5*D*—O7—Li	123 (2)
Mo—P1—Mo$^{\mathrm{i}}$	103.5 (1)	Mo—C1—O1	179.4 (7)
Mo—P1—C1*A*	110.6 (3)	Mo—C2—O2	175 (2)
Mo—P1—C2*A*	114.5 (3)	Mo—C3—O3	177 (1)
C1*A*—P1—C2*A*	99.2 (5)	Mo—C4—O4	177 (1)

O1—Li—O5	111 (1)	P1—C1*A*—C1*B*	120.3 (7)
O1—Li—O6	108 (2)	P1—C1*A*—C1*F*	121.7 (8)
O1—Li—O7	104 (2)	P1—C2*A*—C2*B*	121.6 (8)
O5—Li—O6	113 (1)	P1—C2*A*—C2*F*	119.7 (8)
O5—Li—O7	115 (1)		

Symmetry code: (i) $-x, -y, -z$.

The structure was solved by the Patterson method and refined by full-matrix least squares. Anisotropic displacement parameters were refined for all non-H atoms with the exception of 12 thf methylene C atoms. The large isotropic *B* values for these atoms (Table 1) and the large range shown by thf C—C bonds may indicate some disorder of the thf groups. All calculations were performed using the *SDP* system (Enraf–Nonius, 1985) on a MicroVAX II computer.

We thank the Hong Kong Research Grant Council and the University of Hong Kong for support.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: MU1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1406–1407

trans-Dichlorotetrapyridineruthenium(II)

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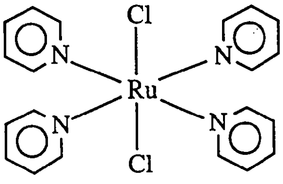
(*Received* 4 *October* 1993; *accepted* 16 *February* 1994)

Abstract

$[Ru(py)_4Cl_2]$ (where py = pyridine, C_5H_5N), crystallizes in the tetragonal space group $I4_1/acd$. Ru and Cl atoms occupy sites of 222 and 2 point symmetry, respectively. The Ru atom has slightly distorted octahedral coordination. The Cl—Ru—Cl moiety is linear, as a result of symmetry requirements.

Comment

The title compound, (I), was prepared using a modification of procedures described by Bottomley & Mukaida (1982) and Gilbert, Rose & Wilkinson (1970). Hydrated $RuCl_3$ was dissolved in 90% ethanol. To this solution excess pyridine was added and the mixture was refluxed for 1 h. After cooling, the resulting precipitate was filtered and washed with water, and then with diethyl ether (yield 42%). Orange–red crystals suitable for X-ray analysis were grown by slow evaporation of a 1:1 CH_2Cl_2/CH_3CN solution of the complex.



(I)

Since there have been relatively few structural studies of *trans*-dichlorotetraammineruthenium(II) species, we have determined the structure of the title compound. It crystallizes in $I4_1/acd$ (No. 142; origin taken at $\bar{1}$). The Ru atom lies on the special position with site symmety 222 [Wyckoff position 8(*b*)] and the Cl atom lies on a twofold axis [Wyckoff position 16(*f*)]. The pyridine ligand is in a general position and four symmetrically related pyridine ligands are bonded to each Ru atom. The Ru—Cl bond distance is comparable to those observed in other dichlororuthenium(II) complexes (Seal & Ray, 1984), but is significantly longer than those observed in $[Ru^{III}(py)_4Cl_2]^+$ (Al-Zamil *et al.*, 1982).

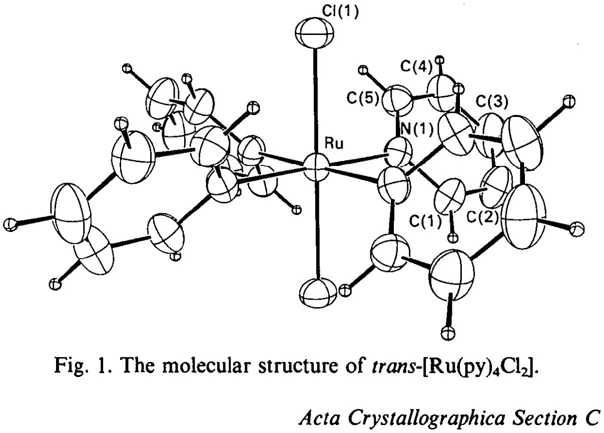


Fig. 1. The molecular structure of *trans*-$[Ru(py)_4Cl_2]$.