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Reaction of $Mo_2(O_2CMe)_2Cl_2$(dppe) with pyridine; structural characterizations of the products of $Mo_2Cl_2(OAc)_2Py_2$ and $MoOCl_2$(dppe)(Py)

Jhy-Der Chen, F. Albert Cotton* and Seong-Joo Kang

Department of Chemistry and Laboratory for Structure and Bonding, Texas A&M University, College Station, TX 77843 (USA)

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Abstract

By layering a CH_2Cl_2/pyridine solution of $Mo_2Cl_2(OAc)_2$(dppe) with n-hexane, two different kinds of crystals were obtained, both of which were characterized by X-ray crystallography. Crystal data for $Mo_2Cl_2(OAc)_2Py_2$ (**1**): $P\bar{1}$, $a=11.979(2)$, $b=13.977(3)$, $c=9.036(1)$ Å, $\alpha=103.15(1)$, $\beta=94.46(1)$, $\gamma=115.12(1)°$, $V=1307.5(4)$ Å^3, Z=2. Final residuals for **1**: $R=0.0493$, $R_w=0.0660$. Crystal data for $MoOCl_2$(dppe)(Py) (**2**): $P2_1/c$, $a=11.480(2)$, $b=17.854(2)$, $c=15.693(2)$ Å, $\beta=115.51(1)°$, $V=2982.3(7)$ Å^3, Z=4. Final residuals for **2**: $R=0.0458$, $R_w=0.0769$. The molecules of **1** were found to be linked by two Cl---Mo bridges (3.018 and 3.082 Å) into infinite chains.

Introduction

Compounds of the type $Mo_2(O_2CR)_2X_2(L)_2$ where X=Cl or Br and L represents a monodentate ligand or one half of a bidentate ligand have been reported before, beginning in 1976 [1]. In some cases X-ray crystallographic structure determinations have been reported [1–5] and in other cases they have not [6]. With the sole exception of $Mo_2(O_2CCF_3)Cl_2(C_2H_5CN)_2$ [4] these compounds have all contained phosphines as the neutral ligands, L.

In this paper we report the direct conversion of a phosphine compound, $Mo_2Cl_2(O_2CCH_3)_2$(dppe) to the pyridine compound *cis*-$Mo_2(O_2CCH_3)Cl_2(Py)_2$. The complex $MoOCl_2$(dppe)(Py) which was obtained in the same reaction will also be reported. The preparation and structure of $Mo_2(O_2CCH_3)_2Cl_2(Py)_2$ support the proposed *cis* structure of $Mo_2Cl_2(OAc)_2$(dppe).

Experimental

General procedures

All manipulations were carried out under an atmosphere of dry oxygen-free argon using Schlenk techniques, unless otherwise noted. Solvents were dried and deoxygenated by refluxing over the appropriate reagents before use. THF, n-hexane, ether and toluene were purified by distillation from sodium–potassium/benzophenone. Pyridine was distilled from CaH_2.

Preparations

Starting material

$Mo_2(OAc)_4$ was prepared according to a reported procedure [7].

$Mo_2Cl_2(OAc)_2$dppe

$(Mo)_2(OAc)_4$ (1.0 g) was placed in a flask containing a THF/toluene mixture (1:1, 50 ml), followed by the addition of Me_3SiCl (1 ml) and dppe (1.0 g). The mixture was then stirred for 20 h. The pink solid was filtered from a dark green solution, washed with THF and ether, and then dried *in vacuo*. Yield 0.78 g (45%). The red crystals of $Mo_2Cl_2(OAc)_2Py_2$ and green crystals of $MoOCl_2$(dppe)(Py) were obtained by layering a CH_2Cl_2/pyidine solution of $Mo_2Cl_2(OAc)_2$dppe with n-hexane.

X-ray crystallography

The structures of $Mo_2Cl_2(OAc)_2Py_2$ and $MoOCl_2$(dppe)(Py) were determined by a general procedure that has been fully described elsewhere [8].

$Mo_2Cl_2(OAc)_2Py_2$ (1)

A red crystal was mounted on the top of a fiber with epoxy cement. Crystal quality was confirmed

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*Author to whom correspondence should be addressed.

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by a rotation photograph. The unit cell constants were determined from 22 reflections with 2θ values in the range 44–52°. These were consistent with a triclinic system. The diffraction data were collected at 20 ± 1 °C on a Rigaku AFC5R diffractometer with graphite monochromated Cu Kα ($\lambda\alpha = 1.54$ Å) radiation. Data reduction was carried out by standard methods with the use of well-established computational procedures; crystallographic computing was done on a local area VAX cluster, employing the VAX/VMS V4.6 computer.

The ω–2θ collection method was used to scan a possible 4171 data points in the range of $4 < 2\theta < 120°$. The structure factors were obtained after Lorentz and polarization corrections. Empirical absorption corrections based on azimuthal (Ψ) scans of reflections of Eulerian angle χ near 90° were applied to the data [9]. The crystal data are given in Table 1.

The Patterson methods program in SHELXS-86 [10] led to the location of the positions of the molybdenum atoms. The remaining atoms were found in a series of alternating difference Fourier maps and least-squares refinements. All atoms were defined anisotropically, but hydrogen atoms were omitted from the model. The final residuals of the refinement were $R = 0.0493$ and $R_w = 0.0660$. Atomic positional parameters and equivalent isotropic thermal parameters are listed in Table 2.

$MoOCl_2(dppe)(Py)$ (2)

The method used to obtain the structure of this compound was similar to that for **1**; the crystal data are given in Table 1. Atomic positions and equivalent isotropic thermal parameters are listed in Table 3.

Results and discussion

The red complex $Mo_2Cl_2(OAc)_2Py_2$ (**1**) was obtained from a simple reaction in which the dppe ligand of $Mo_2Cl_2(OAc)_2(dppe)$ was replaced by two

TABLE 1. Crystal data for $Mo_2Cl_2(OAc)_2Py_2 \cdot 2CH_2Cl_2$ (**1**) and $MoOCl_2(dppe)(Py)$ (**2**)

	1	2
Formula	$Mo_2Cl_2O_4N_2C_{14}H_{16} \cdot 2CH_2Cl_2$	$MoOCl_2P_2NC_{31}H_{29}$
Formula weight	708.95	660.38
Space group	$P\bar{1}$	$P2_1/c$
a (Å)	11.979(2)	11.480(2)
b (Å)	13.977(3)	17.854(2)
c (Å)	9.036(1)	15.639(2)
α, (°)	103.15(1)	90
β (°)	94.46(1)	115.51(1)
γ (°)	115.12(1)	90
V (Å^3)	1307.5(4)	2982.3(7)
Z	2	4
D_{calc} (g/cm^3)	1.801	1.471
Crystal size (mm)	$0.40 \times 0.35 \times 0.15$	$0.42 \times 0.31 \times 0.25$
μ(Cu Kα) (cm^{-1})	140.507	65.862
Data collection instrument	Rigaku-AFC5R	Rigaku-AFC5R
Radiation monochromated in incident beam (Cu Kα, Å)	1.541838	1.541838
Orientation reflections: no., range (2θ)	22; $44.6 < 2\theta < 52.9$	25; $48.1 < 2\theta < 52.9$
Temperature (°)	20 ± 1	20 ± 1
Scan method	2θ–ω	2θ–ω
Data collection range, 2θ (°)	$4 \leqslant 2\theta \leqslant 120$	$4 \leqslant 2\theta \leqslant 120$
No. unique data, total with $F_0^2 > 3\sigma(F_0^2)$	3683, 2697	4263, 3831
No. parameters refined	289	343
Transmission factors: max., min.	1.00, 0.47	1.00, 0.56
R^a	0.0493	0.0458
R_w^b	0.0660	0.0769
Quality-of-fit indicator^c	1.451	1.656
Largest shift/e.s.d., final cycle	0.31	0.03
Largest peak (e/Å^3)	0.629	0.744

[a] $R = \Sigma||F_0|-|F_c||/\Sigma|F_0|$. [b] $R_w = [\Sigma w(|F_0|-|F_c|)^2/\Sigma\, w|F_0|^2]^{1/2}$; $w = 1/\sigma^2(|F_0|)$. [c] Quality-of-fit $= [\Sigma w(|F_0|-|F_c|)^2/(N_{observed} - N_{parameters})]^{1/2}$.

TABLE 2. Positional and isotropic equivalent thermal parameters (Å^2) and their e.s.d.s for $Mo_2Cl_2(OAc)_2Py_2 \cdot 2CH_2Cl_2$

Atom	x	y	z	B (Å^2)
Mo(1)	0.18870(6)	0.04516(5)	−0.00147(7)	3.76(2)
Mo(2)	0.36037(7)	0.03811(5)	0.05622(7)	3.94(2)
Cl(1)	0.0270(2)	0.8973(2)	0.0747(2)	4.40(5)
Cl(2)	0.6229(2)	0.0783(2)	0.1791(2)	4.85(6)
O(1)	0.2195(5)	0.1518(4)	0.2202(6)	5.2(2)
O(2)	0.7163(6)	0.8132(4)	0.0755(6)	5.2(2)
O(3)	0.5396(6)	0.8206(5)	0.0126(6)	5.5(2)
O(4)	0.3961(6)	0.1442(5)	0.2825(6)	5.4(2)
N(1)	0.8801(7)	0.0551(5)	0.2499(7)	4.4(2)
N(2)	0.3075(7)	0.9001(5)	0.1590(7)	4.6(2)
C(1)	0.3181(9)	0.1784(7)	0.3169(9)	4.8(3)
C(2)	0.343(1)	0.2527(9)	0.480(1)	7.0(3)
C(3)	0.5976(9)	0.7729(8)	0.061(1)	5.2(3)
C(4)	0.529(1)	0.6692(8)	0.105(1)	7.0(3)
C(5)	0.861(1)	0.0045(8)	0.360(1)	6.4(3)
C(6)	0.901(1)	0.0599(9)	0.516(1)	6.7(3)
C(7)	0.033(1)	0.8259(9)	0.439(1)	6.6(3)
C(8)	−0.014(1)	0.2283(9)	0.445(1)	6.6(3)
C(9)	0.9426(9)	0.1666(8)	0.294(1)	5.6(3)
C(10)	0.357(1)	0.9198(8)	0.309(1)	6.1(3)
C(11)	0.333(1)	0.8347(9)	0.379(1)	7.2(4)
C(12)	0.254(1)	0.7276(8)	0.290(1)	7.2(3)
C(13)	0.204(1)	0.7056(8)	0.136(1)	6.9(3)
C(14)	0.2334(9)	0.7931(8)	0.076(1)	5.5(3)
Cl(3)	0.8468(6)	0.4190(5)	0.3432(7)	16.5(2)
Cl(4)	0.6292(6)	0.4524(4)	0.3243(7)	18.5(3)
C(15)	0.693(1)	0.363(1)	0.288(2)	11.7(6)
Cl(5)	0.238(2)	0.4458(9)	0.274(1)	25.5(7)
Cl(6)	0.8207(9)	0.5812(6)	0.055(1)	12.0(3)
Cl(7)	0.094(1)	0.4179(6)	0.155(2)	17.0(5)
Cl(8)	0.342(1)	−0.5484(7)	0.240(1)	13.2(3)
C(16)	0.205(2)	0.370(1)	0.106(2)	12.5(6)

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as: $(4/3)[a^2\beta_{11}+b^2\beta_{22}+c^2\beta_{33}+ab(\cos\gamma)\beta_{12}+ac(\cos\beta)\beta_{13}+bc(\cos\alpha)\beta_{23}]$.

TABLE 3. Positional and isotropic equivalent thermal parameters (Å^2) and their e.s.d.s for $MoOCl_2$(dppe)(Py)

Atom	x	y	z	B (Å^2)
Mo	0.17798(3)	0.21198(2)	0.17539(2)	2.467(8)
Cl(1)	0.2693(1)	0.13122(6)	0.31403(8)	3.39(2)
Cl(2)	0.3213(1)	0.15427(8)	0.11014(8)	4.01(3)
P(1)	0.0594(1)	0.24952(6)	0.27295(8)	2.54(2)
P(2)	0.3283(1)	0.30564(7)	0.27600(8)	2.67(2)
O	0.1056(3)	0.2781(2)	0.0974(2)	3.57(8)
N	0.0403(4)	0.1229(2)	0.1042(3)	3.26(9)
C(1)	−0.0632(4)	0.3211(3)	0.2294(3)	3.0(1)
C(2)	−0.1667(5)	0.3213(3)	0.2552(4)	3.9(1)
C(3)	−0.2553(5)	0.3768(3)	0.2244(4)	4.6(1)
C(4)	−0.2430(5)	0.4335(3)	0.1694(4)	4.4(1)
C(5)	−0.1380(5)	0.4365(3)	0.1467(4)	4.3(1)
C(6)	−0.0487(5)	0.3792(3)	0.1749(4)	3.6(1)
C(7)	−0.0173(4)	0.1748(3)	0.3108(3)	3.0(1)
C(8)	0.0229(5)	0.1522(3)	0.4014(3)	3.8(1)
C(9)	−0.0362(5)	0.0906(3)	0.4251(4)	4.7(1)
C(10)	−0.1328(5)	0.0540(3)	0.3595(4)	4.8(1)
C(11)	−0.1734(5)	0.0761(3)	0.2685(4)	4.5(1)
C(12)	−0.1158(5)	0.1371(3)	0.2450(4)	4.0(1)
C(13)	0.1710(5)	0.2946(3)	0.3760(3)	3.1(1)
C(14)	0.2489(4)	0.3516(3)	0.3463(3)	3.3(1)
C(15)	0.3759(4)	0.3830(3)	0.2191(4)	3.3(1)
C(16)	0.3518(5)	0.3782(3)	0.1253(4)	4.1(1)
C(17)	0.3866(6)	0.4368(4)	0.0788(4)	5.4(2)
C(18)	0.4452(5)	0.5002(3)	0.1279(5)	5.4(2)
C(19)	0.4731(5)	0.5047(3)	0.2235(5)	5.3(2)
C(20)	0.4378(5)	0.4453(3)	0.2683(4)	4.2(1)
C(21)	0.4764(4)	0.2713(3)	0.3598(4)	3.2(1)
C(22)	0.5562(5)	0.2367(3)	0.3237(4)	4.5(1)
C(23)	0.6693(6)	0.2072(3)	0.3840(6)	5.8(2)
C(24)	0.6987(7)	0.2112(4)	0.4784(6)	6.4(2)
C(25)	0.6174(7)	0.2463(5)	0.5130(5)	7.2(2)
C(26)	0.5046(6)	0.2745(4)	0.4532(4)	5.2(2)
C(27)	0.0500(5)	0.0512(3)	0.1365(4)	4.2(1)
C(28)	−0.0427(6)	−0.0006(3)	0.0972(4)	5.2(1)
C(29)	−0.1502(6)	0.0184(4)	0.0250(4)	5.8(1)
C(30)	−0.1624(6)	0.0905(4)	−0.0090(5)	5.8(2)
C(31)	−0.0631(5)	0.1422(3)	0.0312(4)	4.4(1)

Anisotropically refined atoms are given in the form of the equivalent isotropic displacement parameter defined as: $(4/3)[a^2\beta_{11}+b^2\beta_{22}+c^2\beta_{33}+ab(\cos\gamma)\beta_{12}+ac(\cos\beta)\beta_{13}+bc(\cos\alpha)\beta_{23}]$.

pyridine ligands. The green complex $MoOCl_2$(dppe)(Py) (**2**) was obtained as a byproduct resulting from oxidation of $Mo_2Cl_2(OAc)_2$(dppe). The source of oxygen was most likely air. In the previously reported preparation [6] of $Mo_2Cl_2(OAc)_2$(dppe), the method was different, starting with $Mo_2Cl_2(OAc)_2(PPh_2)_2$. The present method gave a yield of only 45% but has the advantage of proceeding directly from $Mo_2(OAc)_4$. The compound was previously described as purple whereas our product appeared pink, perhaps because of different particle sizes.

$Mo_2Cl_2(OAc)_2(Py)_2$ (1)

Crystals of this complex conform to the space group $P\bar{1}$ with two molecules in the unit cell. Figure 1 shows the ORTEP diagrams of these two molecules which are interrelated by an inversion center. Table 4 lists some selected bond distances and angles for **1**. The molecules of **1** have the *cis* arrangement of the two carboxylate groups.

The molecules of **1** show that the Mo–O bond length is a function of the *trans* ligand within the same molecule. The average Mo–O bond distance *trans* to Cl is about 0.1 Å longer than the average Mo–O distance *trans* to the pyridine ligand, suggesting that the chloride atom lies higher in a *trans*-influence series for the Mo(II) complexes than the pyridine ligand. Combining the series, $Et_3P > Me_3CCO_2 \geqslant Cl$, which was found from two isomers of

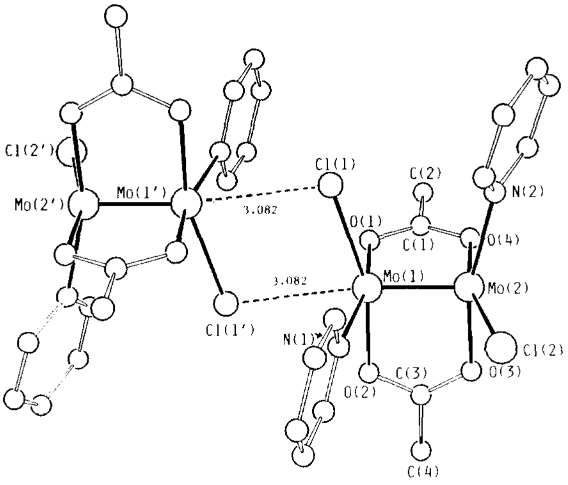


Fig. 1. ORTEP drawing for $Mo_2Cl_2(OAc)_2Py_2$, showing two molecules which are interrelated by an inversion center. The dashed lines represent the weak interactions between the molecules.

$Mo_2(O_2CCMe_3)_2Cl_2(PEt_3)_2$ [2], the series now can be written as $Et_3P > Me_3CCO_2 \geqslant Cl > Py$.

The crystal packing that leads to the formation of infinite chains is shown in Scheme 1. The molecules are linked together by weak interactions through two chlorine atoms (Mo---Cl(1) = 3.082 Å and Mo---Cl(2) = 3.018 Å). The molecules of $Mo_2(O_2CCF_3)_2(NCC_2H_5)_2Cl_2 \cdot Mo_2(O_2CCF_3)_3(C_2H_5CN)Cl$ [4] were also found to be linked by three Cl---Mo bridges (2.894–3.005 Å) and one O---Mo bridge into finite chains in which the two dinuclear units alternate.

Due to the insolubility of $Mo_2Cl_2(OAc)_2(dppe)$ in most solvents, the crystal structure was not determined, but a *cis* arrangement of the acetate groups was proposed on indirect evidence. The preparation of $Mo_2Cl_2(OAc)_2Py_2$ by direct ligand replacement provides support for the proposed structure of $Mo_2Cl_2(OAc)_2(dppe)$ given below. This kind of structure has also been proposed by Bakir and Walton for complexes of the type $Mo_2X_2(OAc)_2(PP)$ (X = Cl, Br, I; PP = diphosphine ligands) [6] and was considered to be most stable when the overall rotation geometry remains eclipsed, as seen in the cases with quadruply bonded Mo_2^{4+} species.

$Mo_2OCl_2(dppe)(Py)$ (2)

The crystals of 2 conform to the space group $P2_1/c$ with four molecules in the unit cell. Figure 2 shows the ORTEP diagram of this complex. The five ligands coordinated to the Mo atom form a highly distorted octahedron. Table 5 lists some selected bond distances and angles for 2. The two Cl atoms are *cis* to each other and the oxygen atom

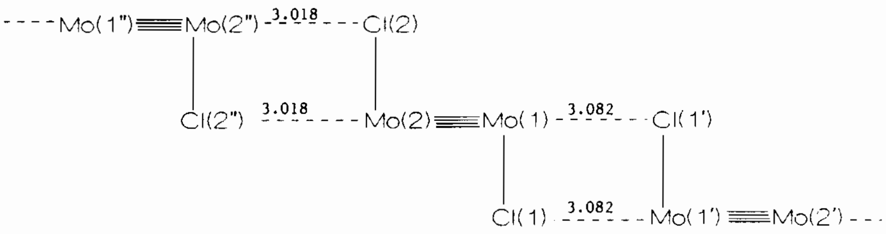
TABLE 4. Selected bond distances (Å) and angles (°) for $Mo_2Cl_2(OAc)_2Py_2 \cdot 2CH_2Cl_2$

Bond lengths (Å)	
Mo(1)–Mo(2)	2.131(1)
Mo(1)–Cl(1)	2.440(2)
Mo(1)–O(1)	2.105(5)
Mo(1)–O(2)	2.114(6)
Mo(1)–N(1)	2.232(6)
Mo(2)–Cl(2)	2.445(2)
Mo(2)–O(3)	2.094(6)
Mo(2)–O(4)	2.123(5)
Mo(2)–N(2)	2.203(8)
Bond angles (°)	
Mo(2)–Mo(1)–Cl(1)	106.42(7)
Mo(2)–Mo(1)–O(1)	91.2(2)
Mo(2)–Mo(1)–O(2)	90.9(2)
Mo(2)–Mo(1)–N(1)	102.5(2)
Cl(1)–Mo(1)–O(1)	88.6(2)
Cl(1)–Mo(1)–O(2)	162.4(2)
Cl(1)–Mo(1)–N(1)	90.6(2)
O(1)–Mo(1)–O(2)	87.9(2)
O(1)–Mo(1)–N(1)	165.9(3)
O(2)–Mo(1)–N(1)	88.6(2)
Mo(1)–Mo(2)–Cl(2)	105.25(7)
Mo(1)–Mo(2)–O(3)	91.0(2)
Mo(1)–Mo(2)–O(4)	90.6(2)
Mo(1)–Mo(2)–N(2)	104.3(2)
Cl(2)–Mo(2)–O(3)	90.6(2)
Cl(2)–Mo(2)–O(4)	164.1(2)
Cl(2)–Mo(2)–N(2)	89.4(2)
O(3)–Mo(2)–O(4)	87.7(2)
O(3)–Mo(2)–N(2)	164.2(3)
O(4)–Mo(2)–N(2)	87.9(2)
Mo(1)–O(1)–C(1)	118.1(6)
Mo(1)–O(2)–C(3)	118.1(7)
Mo(2)–O(3)–C(3)	119.3(5)
Mo(2)–O(4)–C(1)	118.3(5)
Mo(1)–N(1)–C(5)	119.5(5)
Mo(1)–N(1)–C(9)	122.5(6)
Mo(2)–N(2)–C(10)	119.5(5)
Mo(2)–N(2)–C(14)	123.1(6)

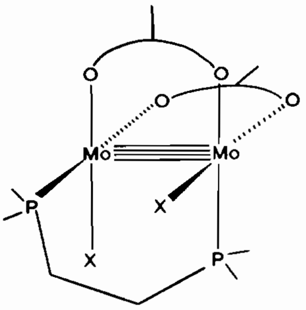
Numbers in parentheses are e.s.d.s in the least significant digits.

The two Mo–Cl bond distances, Mo–Cl(1) = 2.492(1) Å and Mo–Cl(2) = 2.458(1) Å, differ significantly, due to the different *trans* influences of P and O (O > P). The two different Mo–P distances of 2.481(1) and 2.502(1) Å indicate that the *trans* influences of Py and Cl are in the order that Py > Cl.

Complex 2 allows some interesting comparisons with $MoOCl_2(PMe_2Ph)_3$ [11]. First, the average of the P–Mo–P angles of the *cis* phosphorus atoms in $MoOCl_2(PMe_2Ph)_3$ is 94.1°, while in 2 the angle is only 80.8° due to the constraint imposed by the five-membered ring. Second, both the meridional planes of 2 are occupied by three types of atoms, while in $MoOCl_2(PMe_2Ph)_3$ one plane is occupied by three types of atoms and the other only by two types. So



Scheme 1.



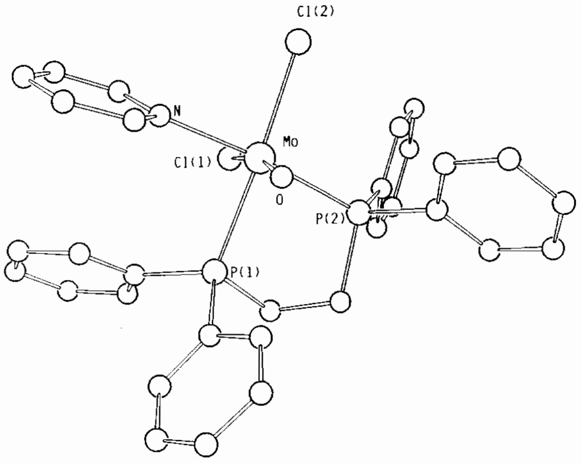


Fig. 2. ORTEP drawing for $MoOCl_2(dppe)(Py)$.

far as we know, complex **2** is the only complex of the type $MoOX_2L_3$ (X = halogen atom and L = monodentate ligand) in which such a configuration has been structurally characterized.

Supplementary material

Full tables of bond distances, bond angles anisotropic and thermal parameters (10 pages)l; and two listings of observed and calculated structure factors (35 pages) are available from author F.A.C. upon request.

TABLE 5. Selected bond distances (Å) and angles (°) for $MoOCl_2(dppe)(Py)$

Distances (Å)	
Mo–Cl(1)	2.492(1)
Mo–Cl(2)	2.458(2)
Mo–P(1)	2.481(1)
Mo–P(2)	2.502(1)
Mo–O	1.682(3)
Mo–N	2.231(4)
Angles (°)	
Cl(1)–Mo–Cl(2)	89.24(4)
Cl(1)–Mo–P(1)	76.81(4)
Cl(1)–Mo–P(2)	82.33(4)
Cl(1)–Mo–O	167.3(1)
Cl(1)–Mo–N	91.0(1)
Cl(2)–Mo–P(1)	166.04(4)
Cl(2)–Mo–P(2)	97.40(4)
Cl(2)–Mo–O	102.8(2)
Cl(2)–Mo–N	87.3(1)
P(1)–Mo–P(2)	80.81(4)
P(1)–Mo–O	91.1(1)
P(1)–Mo–N	92.9(1)
P(2)–Mo–O	91.8(1)
P(2)–Mo–N	171.7(1)
O–Mo–N	93.8(1)
Mo–P(1)–C(1)	118.6(2)
Mo–P(1)–C(7)	116.3(2)
Mo–P(1)–C(13)	107.1(2)
Mo–P(2)–C(14)	107.0(1)
Mo–P(2)–C(15)	117.3(2)
Mo–P(2)–C(21)	118.1(2)
C(14)–P(2)–C(15)	104.7(2)
Mo–N–C(27)	122.5(3)
Mo–N–C(31)	118.7(3)

Numbers in parentheses are e.s.d.s in the least significant digits.

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