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ORIGINAL PAPER

Synthesis and Structural Characterization of a Dimolybdenum Complex Bridged by the Hydroxymethylphenylphosphinate Ligand

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Abstract Hydroxymethylphenylphosphinic acid, **1** was synthesized and structurally characterized by single crystal X-ray diffraction. Ligand **1** crystallized in the monoclinic $P2_1$ space group with two molecules comprising the unit cell and cell dimensions $a = 7.5587(13)$ Å, $b = 5.9019(8)$ Å, $c = 8.808(3)$ Å and $\beta = 90.90(2)°$. Molecules were linked up by hydrogen bonding involving all three oxygen atoms of **1** resulting in 2-dimensional sheets. Reacting **1** with MoO_2Cl_2 in a mixture of ethanol and dichloromethane afforded the dinuclear molybdenum complex $Mo_2O_4Cl_2[(HOCH_2)PhPOO]_2$, **2** where the two molybdenum atoms were bridged by the phosphinate ligand. Complex **2** crystallized in the $P2_{1/C}$ space group as a dietherate with $a = 9.208(3)$ Å, $b = 14.353(4)$ Å, $c = 12.805(4)$ Å and $\beta = 98.64(3)°$ and contained terminal O and Cl atom disorder. The two crystallographically identical molybdenum centers are in distorted octahedral environments. Complexes were analyzed by IR, ^{31}P NMR and X-ray crystallography.

Keywords Hydroxymethylphenylphosphinic acid · Dioxomolybdenum · Molybdenum dimers · Hydrogen bonding

Introduction

Various organic phosphinates have been studied for their capacity to stabilize a variety of transition metals and to display a variety of coordination modes (dimers or polymeric chains) including $R_2PO_2^-$, R = Me [1–3], Ph [2, 4–15], C_6H_{11} [16], p-$CH_3OC_6H_4$ [17–19] and $R_1R_2PO_2^-$, $R_1 = Me$, $R_2 = CH_2NH_3^+$ [20, 21]. Phosphinic acids have also been investigated as flame retardant materials previously [22–25]. Hydroxymethylphenylphosphinic acid, **1**, is an asymmetric organic phosphinic acid which has three oxygen atoms capable of ligation and we anticipated that a 2-dimensional polymeric sheet may be formed through hydrogen bonding. For example, the oxygen atoms in the hydroxymethylphenylphosphinate ligand could exhibit monodentate, bidentate or tridentate bonding modes. In our lab, we have utilized phosphinates, such as diphenylphosphinic acid and dimethylphosphinic acid to prepare molybdenum tetramers with distorted Mo_4O_4 cubic cores [2, 26]. We have also previously detailed the structures and catalytic activities of monodentate complexes of the form $MO_2Cl_2L_2$ (M = Mo, W and L = various monodentate ligands) [26–30]. Better solubility and catalytic properties (specifically the utilization of H_2O_2 as a source of oxygen) of the complexes have been observed in polar and non-chlorinated solvents, such as ethanol, when polar hydroxyl groups are introduced in the ligand [27]. Therefore, we are interested in making complexes containing polar ligands, such as ligand **1**, as this may afford better solubility and catalytic properties. In this work, we describe the synthesis and characterization of dinuclear dioxo molybdenum complexes bridged by **1** displaying a bridging tridentate ligand mode. The structure of the hydroxymethylphenylphosphinic acid was also characterized by single crystal X-ray diffraction and we became aware of a recent crystallographic report

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on **1** during the preparation of this manuscript ($R = 5.67\%$) [31].

Experimental

General Method

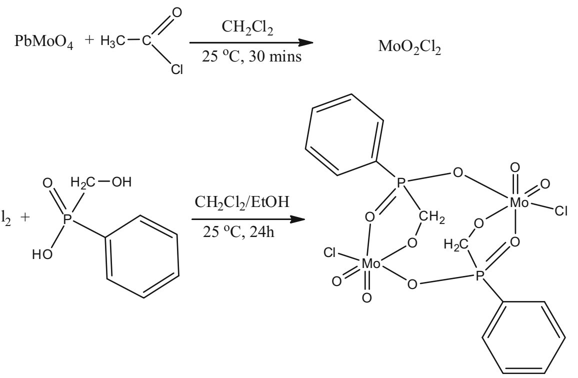
Infrared spectra were obtained on a Perkin Elmer Spectrum one FT-IR spectrometer. ^{31}P NMR data were recorded on a Varian XL-400 spectrometer. A Fisher-Johns melting point apparatus (Fisher Scientific Company) was used for the melting point determinations. Solvents were used as received from commercial suppliers. Chemicals were purchased from Aldrich and all were used as received.

Synthesis and Characterization of Hydroxymethylphenylphosphinic Acid $(HOCH_2)PhPOOH$ (**1**)

Hydroxymethylphenylphosphinic acid was prepared by modifications to the literature method for bis(hydroxymethyl)phosphinic acid [32]. A mixture of phenylphosphinic acid (7.1 g, 50 mmol), concentrated hydrochloric acid (10 mL) and formaldehyde (1.60 g) was charged in a two neck flask and warmed up to 40–45 °C until a clear solution was obtained. The solution was then refluxed at 120 °C for 30 h. The product was obtained by removing all the volatiles. 4.6 grams of **1** was afforded after re-crystallization in isopropyl alcohol. **1** was characterized by IR, ^{1}H NMR and ^{31}P NMR. Yield: 54%. M.p.: 124.5–126 °C. IR (neat, cm^{-1}) 3458(s), 2903(w), 1438(s) 1128(m, P=O) 1052(m) 967(s) 858(s) 769(m), 742(s), 689(s). ^{31}P NMR (EtOH) $\delta = 35.9$ ppm (s, 1P) relative to H_3PO_4. The ligand was also structurally characterized by single X-ray diffraction.

Synthesis and Characterization of Molybdenum Dimer $Mo_2O_4Cl_2[(HOCH_2)PhPOO]_2$ (**2**)

The MoO_2Cl_2 unit was prepared as shown in Scheme 1 following established procedures [28, 33–35]. A mixture of lead molybdate (0.693 g, 1.885 mmol), acetyl chloride and dichloromethane was stirred for 30 min at room temperature resulting in a brown solution which was obtained by filtration. The solution was then added in a dropwise manner to a solution of hydroxymethylphenylphosphinic acid (0.332 g, 1.885 mmol) in CH_2Cl_2/EtOH resulting first in an orange solution which then changed to a light yellowish-green solution after one hour of stirring. This solution was then stirred at room temperature for 24 h. The solution was concentrated under vacuum and hexane added to precipitate the yellowish-green product. The precipitate was filtered and dried under vacuum at room temperature overnight affording 0.45 g of the molybdenum dimer $Mo_2O_4Cl_2[(HOCH_2)PhPOO]_2$. Yield : 72%. M.p.: 166–168 °C. IR (neat, cm^{-1}) 3303(m), 1593(m), 1439(s), 1131 (s, P=O) 1036(w), 1011(w), 993(w), 952(s, Mo = O, sym), 913(s, Mo = O, asym), 864(w), 771(w), 736(s), 689(s). ^{31}P NMR(EtOH) $\delta = 42.1$ ppm(s, 1P) relative to H_3PO_4.



Scheme 1 Synthesis of Mo dimer $Mo_2O_4Cl_2[(HOCH_2)PhPOO]_2$ (**2**)

X-Ray Crystallography

Suitable crystals of compound **1** were obtained by allowing slow growth from a solution of **1** in isopropyl alcohol. Those of **2** were obtained by layering an ethanol solution with diethyl ether for several days. Suitable crystals of **1** and **2** were coated with epoxy resin and mounted on the tips of thin glass fibers. All measurements were obtained by using an Enraf–Nonius Turbo CAD4 X-ray diffractometer. MoKα radiation ($\lambda = 0.71073$ Å) was used for data collection. The collection of the data, solution and refinements of the structures were accomplished using established procedures [2, 36–38].

Results and Discussion

Synthesis

We have previously reported on the synthesis, characterization and catalytic properties of dioxomolybdenum and dioxotungsten complexes supported by the monodentate hydrophilic ligand hydroxymethyldiphenyl phosphine oxide [27]. We discovered that by introducing a polar hydroxymethyl group in the ligand, these complexes showed better solubility in non-chlorinated solvents, such as ethanol and may be more applicable for catalytic epoxidation reactions utilizing H_2O_2 as the source of oxygen. It was with these results in mind that we decided to explore the syntheses of molybdenum tetramers, consisting of an "Mo_4O_8" [2, 26] core, which may be stabilized with the conjugate base of ligand **1**. We prepared ligand **1** by refluxing a mixture of phenylphosphinic acid, concentrated HCl and formaldehyde for 30 h. The IR spectrum contained an absorption at 1128 cm^{-1} which may be ascribed to a P=O bond vibration in agreement with the literature values [25].

The procedure used to prepare the complex **2** is shown in Scheme 1. The "MoO_2Cl_2" unit was generated by mixing lead molybdate with acetyl chloride in dichloromethane [28, 33–35]. Treatment of equal moles of "MoO_2Cl_2" with **1** in a mixture of dichloromethane and ethanol at room temperature produces the molybdenum dimer **2** as a moisture sensitive, yellowish-green solid in 72% yield. The IR spectrum contains diagnostic bands at 952 and 913 cm^{-1} which are due to the symmetric and asymmetric stretching modes for the cis-dioxomolybdenum unit (MoO_2) [39]. A strong-intensity absorption band at 1131 cm^{-1} can be assigned to a P=O stretch which is blue shifted compared to the equivalent mode in free ligand (1128 cm^{-1}). The ^{31}P NMR spectrum of **2** in ethanol displays a single resonance at 42.1 ppm for the P atom of the coordinated ligand which is shifted downfield of the resonance for the P atom on the free ligand **1** (35.9 ppm). The magnitude of this shift, 6.2 ppm, is comparable to that observed with other related phosphonate ligands [27, 29].

Table 1 Crystal data and structure refinement details of complexes **1** and **2**

Identification code	**1**	**2**
Molecular formula	$C_7H_9O_3P$	$C_{14}H_{16}Cl_2Mo_2O_{10}P_2 \cdot 2C_4H_{10}O$
Formula weight	172.11	817.23
Crystal size (mm)	0.40 × 0.15 × 0.15	0.20 × 0.10 × 0.10
Crystal color	Colorless	Light blue
Temperature (K)	290 (2)	290 (2)
Crystal system	Monoclinic	Monoclinic
Space group	$P2_1$	$P2_{1/C}$
a (Å)	7.5587(13)	9.208(3)
b (Å)	5.9019(8)	14.353(4)
c (Å)	8.808(3)	12.805(4)
β (°)	90.90(2)	98.64(3)
V $(Å)^3$	392.88(16)	1673.1(9)
Z	2	4
Dc (Mg/m^3)	1.455	1.622
Absorption coefficient (mm^{-1})	0.302	1.057
F (000)	180	824
Scan range θ (°)	2.31–24.91	2.15–24.99
No. of reflections measured	971	2933
No. of observed data, $I > 2\sigma$ (I)	902	1968
No. of parameters	109	191
$R1$, $wR2$ $[I > 2\sigma$ $(I)]^{a,b}$	0.0217, 0.0573^{c}	0.0506, 0.1137^{d}
$R1$, $wR2$ (all data)	0.0235, 0.0606	0.1034, 0.1333
Goodness of fit on F^2	1.117	1.144

[a] $R1 = \sum||F_o| - |Fc||/\sum|F_o|$

[b] $wR2 = [\sum[w(F_o^2 - F_c^2)^2]/\sum[w(F_o^2)^2]]^{1/2}$

[c] $w = 1/[\sigma^2(F_o^2) + (0.0310P)^2 + 0.1117P]$ where $P = (F_o^2 + 2F_c^2)/3$

[d] $w = 1/[\sigma^2(Fo^2) + (0.0.0445P)^2 + 3.3894P]$ where $P = (F_o^2 + 2F_c^2)/3$

X-Ray Crystallographic Data

Crystal data and details of structural refinement are listed in Table 1. Selected bond lengths and angles of compound **1** and **2** are given in Tables 2 and 3 respectively.

A Mercury [40] drawing of ligand **1** is shown in Fig. 1. One hydroxymethylphenylphosphinic acid molecule comprises the asymmetric unit and all H atoms were refined with riding parameters. The geometry around the phosphorus atom is pseudo tetrahedral with the O–P–O and C–P–C bond angles equal to 116.30(11)° and 108.92(12)° respectively. The bond angles for O–P–C range from 104.03(9)–110.79(12)° probably due to minimization of steric hindrance with the phenyl ring. There are two significantly different P–O bond distances; a short P=O(2) and longer P–O(1)H at 1.497(2) and 1.5488(17) Å respectively. These P–O bond distances are within the range of expected values for P=O and P–OH bond lengths [41, 42]. All of the O atoms in complex **1** are involved in H-bonding as displayed in Fig. 2 with each molecule H-bonding to four adjacent molecules. Two of these interactions are related by symmetry and the three unique interactions are listed in Table 2. The different P–O bond distances results in an asymmetric hydrogen bridge [43, 44]. The net result of the H-bonds are polymeric chains ($C_2^2(10)$), located parallel to the a axis, constituting a polar middle section with the H-bonds consisting of pentatomic rings with a bifurcated H donor ($R_1^2(5)$) and a hydrophobic exterior with the protruding phenyl groups. In compound **1**, the crystal is built up from infinite 2-dimensional polymeric sheets held together by O–H···O hydrogen bonds, parallel to the *a–b* plane, as shown in Fig. 3. Finally, although complex **1** has crystallized in a chiral and polar space group, the crystal structure is not since the dipole vectors of adjacent molecules are approximately perpendicular to the polar axis but are antiparallel [45].

A Mercury [40] drawing of complex **2** is shown in Fig. 4. The structure contains disorder (89/11%) between terminal Cl and O atoms and these were refined with the Mo–Cl and Mo–O distances for all Cl and O atoms involved constrained to 2.38 and 1.67 Å respectively.

Table 2 Selected bond distances (Å), bond angles (°) and H-bonds for compound **1**

Bond distances			
P(1)–O(2)	1.497(2)	P(1)–C(1)	1.799(2)
P(1)–O(3)	1.5488(17)	O(1)–C(7)	1.412(3)
P(1)–C(7)	1.803(3)		
Bond angles			
O(2)–P(1)–O(3)	116.30(11)	O(3)–P(1)–C(1)	104.03(9)
O(2)–P(1)–C(7)	109.83(12)	C(7)–P(1)–C(1)	108.92(12)
O(3)–P(1)–C(7)	106.61(13)	C(7)–O(1)–H(1)	109.5
O(2)–P(1)–C(1)	110.79(12)	P(1)–O(3)–H(31)	109.5

D–H···A	d(D–H)	d(H···A)	d(D···A)	<(DHA)
O(1)–H(1)···O1^{a}	0.82	2.44	3.0949(12)	137.5
O(1)–H(1)···O2^{a}	0.82	2.29	3.002(3)	145.2
O(3)–H(31)···O2^{b}	0.82	1.74	2.512(3)	157.1

Hydrogen bonds for H···A < (r(A) + 2.000 Å) and < DHA > 110°

Symmetry transformations used to generate equivalent atoms: ^{a} $-x+1, y+1/2, -z+2$; ^{b} $-x, y+1/2, -z+2$

Table 3 Selected bond distances (Å) and bond angles (°) for compound **2**

Bond distances			
Mo(1)–O(2)	1.669(5)	Mo(1)–Cl(1)	2.338(2)
Mo(1)–O(3)	1.754(7)	O(1)–P(1)^{a}	1.519(5)
Mo(1)–O(31)	1.77(4)	P(1)–O(11)	1.503(5)
Mo(1)–O(11)	2.033(4)	P(1)–O(1)^{a}	1.519(5)
Mo(1)–O(1)	2.113(4)	P(1)–C(1)	1.786(6)
Mo(1)–Cl(11)	2.15(3)	P(1)–C(7)^{a}	1.815(7)
Mo(1)–O(71)	2.286(5)		
Bond angles			
O(2)–Mo(1)–O(3)	102.8(4)	O(1)–Mo(1)–O(71)	72.33(17)
O(2)–Mo(1)–O(31)	89(2)	Cl(11)–Mo(1)–O(71)	87.1(8)
O(3)–Mo(1)–O(31)	104.4(17)	O(2)–Mo(1)–Cl(1)	96.93(19)
O(2)–Mo(1)–O(11)	95.3(2)	O(3)–Mo(1)–Cl(1)	95.4(3)
O(3)–Mo(1)–O(11)	90.0(3)	O(11)–Mo(1)–Cl(1)	165.22(16)
O(31)–Mo(1)–O(11)	163.5(16)	O(1)–Mo(1)–Cl(1)	87.87(13)
O(2)–Mo(1)–O(1)	97.1(2)	Cl(11)–Mo(1)–Cl(1)	91.6(8)
O(3)–Mo(1)–O(1)	159.3(3)	O(71)–Mo(1)–Cl(1)	83.36(14)
O(31)–Mo(1)–O(1)	81.4(16)	P(1)^{a}–O(1)–Mo(1)	129.3(3)
O(11)–Mo(1)–O(1)	82.37(17)	Cl(1)–O(31)–Mo(1)	141(6)
O(2)–Mo(1)–Cl(11)	103.4(10)	O(11)–P(1)–O(1)^{a}	115.6(3)
O(31)–Mo(1)–Cl(11)	100.6(18)	O(11)–P(1)–C(1)	106.1(3)
O(11)–Mo(1)–Cl(11)	93.7(8)	O(1)^{a}–P(1)–C(1)	109.6(3)
O(1)–Mo(1)–Cl(11)	159.4(10)	O(11)–P(1)–C(7)^{a}	110.4(3)
O(2)–Mo(1)–O(71)	169.4(2)	O(1)^{a}–P(1)–C(7)^{a}	103.4(3)
O(3)–Mo(1)–O(71)	87.7(3)	C(1)–P(1)–C(7)^{a}	111.9(3)
O(31)–Mo(1)–O(71)	89.3(18)	P(1)–O(11)–Mo(1)	152.4(3)
O(11)–Mo(1)–O(71)	83.10(18)		

D–H···A	d(D–H)	d(H···A)	d(D···A)	<(DHA)
O(71)–H(71)···O(103)^{b}	0.82	1.75	2.560(7)	169.6

Hydrogen bond for H···A < (r(A) + 2.000 Å) and <DHA > 110°

Symmetry transformations used to generate equivalent atoms: ^{a} $-x+2, -y+1, -z+1$; ^{b} $-x+2, y+1/2, -z+1/2$

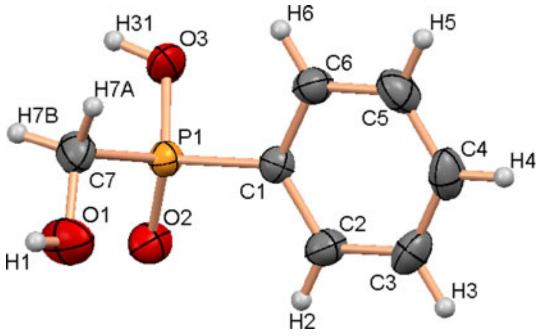


Fig. 1 Mercury [40] representation of **1** with selected atom numbering. Thermal ellipsoids are drawn at the 50% probability level

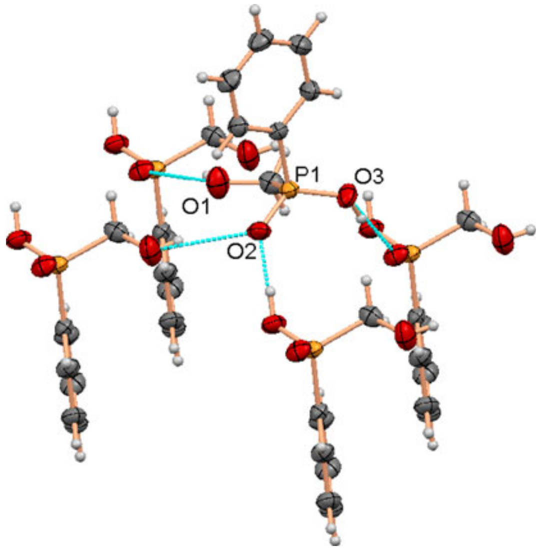


Fig. 2 The hydrogen bonding interactions (*dashed lines*) of **1**

The dinuclear molybdenum complex is positioned around an inversion point in the $P2_{1/C}$ space group and is H-bonded to two diethyl ether molecules via the H-atoms on the hydroxyl moiety. The two crystallographically identical molybdenum centers are bridged together by two tridentate hydroxymethylphenylphosphinate ligands. The molybdenum atoms, separated by 5.465(2) Å are in distorted octahedral environments in which the two terminal oxygen atoms are bonded *cis* to each other. The molybdenum centers are also each coordinated to one chloride ligand and three oxygen atoms from the two bridging hydroxymethylphenylphosphinate ligands. Each hydroxymethylphenylphosphinate ligand forms a five-membered chelating ring through the bidentate $–O=PCH_2OH$-linkage to the Mo atoms. These two oxygen atoms (O(71), O(1)) from the hydroxymethylphenylphosphinate ligand are *cis* to each other with an O(71)–Mo(1)–O(1) angle of 72.33(17)° and are located trans to the terminal oxo-ligands, The two P–O distances at P(1)–O(1) = 1.519(5) and P(1)–O(11) = 1.503(5) Å are slightly different

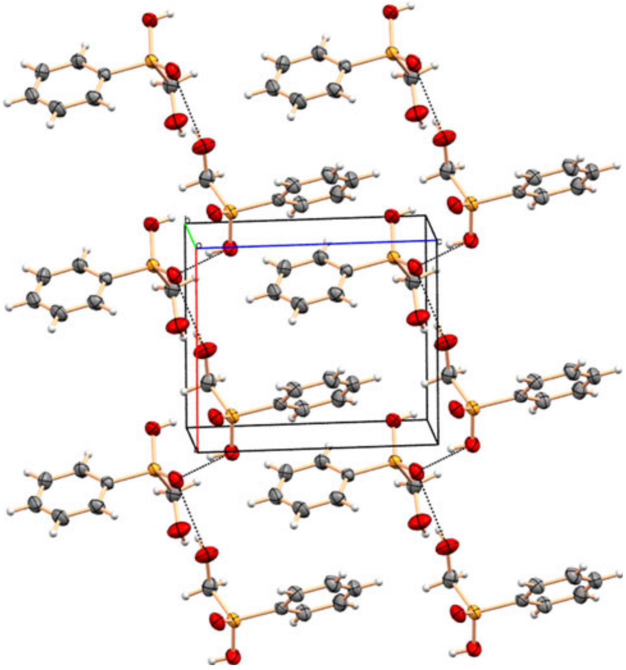


Fig. 3 The crystal packing of **1** showing the arrangement of the 2D polymer formed via hydrogen bonds (*dashed lines*)

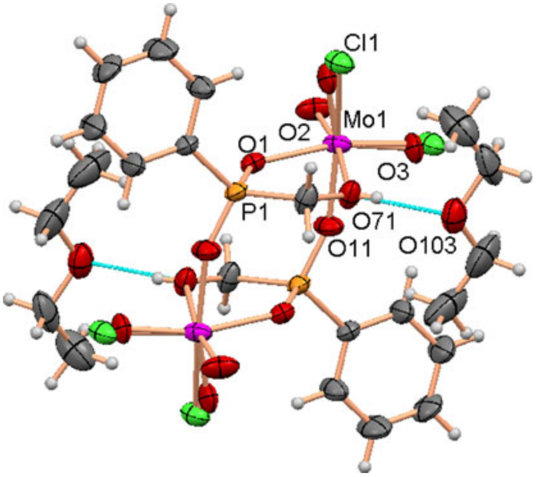


Fig. 4 Mercury [40] drawing of **2** with selected atom numbering for the major orientation. Thermal ellipsoids are drawn at the 30% probability level and the H-bonds to the ether molecules are illustrated with *dashed lines*

probably reflective of the fact that the distances between the Mo and O atoms are also significantly different with Mo(1)–O(1) = 2.113(4) and Mo(1)–O(11) = 2.033(4) Å. The fact that there is a wide variance in the P–O–Mo bond angles with Mo(1)–O(1)–P(1) = 129.3(3) and Mo(1) O(11)–P(1) = 152.4(3)° illustrated the flexibility of this kind of coordination mode given the slight differences in distances noted previously.

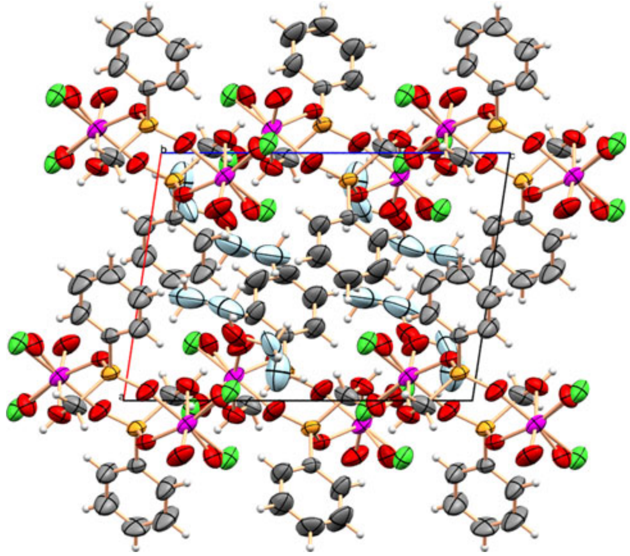


Fig. 5 Mercury [40] drawing of the crystal packing of **2** viewed down the *b* axis. The C atoms in the ether molecule are lighter in appearance

The bond distance between Mo(1) and O(2) (ordered) is 1.669(5) Å which is within the expected range of 1.66–1.76 Å [46]. The bond distances for the disordered Mo = O_{oxo} are Mo(1)–O(3) = 1.754(7) and Mo(1)–O(31) = 1.77(4) Å which are all significantly longer. The Mo–Cl bond lengths at Mo(1)–Cl(1) = 2.338(2) and Mo(1)–Cl(11) = 2.15(3) Å are in the range found for Mo–Cl_t distances in complexes featuring disorder [47]. Irregularities in these distances can be ascribed to this disorder. Interestingly enough this complex packs so that the polar bonds are located on the *bc* plane with the nonpolar phenyl ring and H-bonded diethyl ether molecules in center of the unit cell as illustrated in Fig. 5.

Conclusion

A functionalized organic phosphinic acid, **1**, was synthesized and structurally characterized. Structural examination of the ligand **1** revealed infinite 1-dimentional polymers formed via intermolecular hydrogen bonds. The dinuclear dioxomolybdenum complex **2** bridged by tridentate ligand **1** was also prepared and characterized by IR, ^{31}P NMR and single X-ray diffraction. Complex **2** contained disorder between terminal Cl and O atoms.

Supporting Information Available

Crystallographic data for the structural analysis of the compounds have been deposited at the Cambridge Crystallographic Data Center (CCDC). The CCDC numbers are 804207 for **1** and 804208 for **2**. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, United Kingdom; Fax: +44 1223 336033 or e-mail: deposit@ccdc.cam.ac.uk.

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