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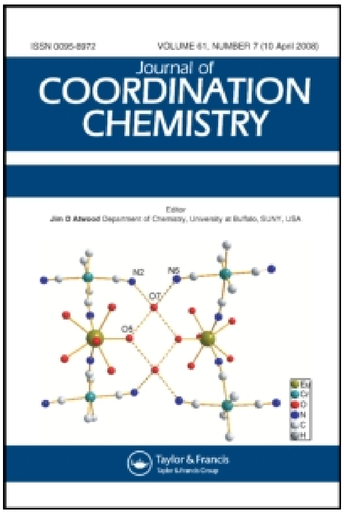

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Synthesis and structure of the first molybdenum compound of N,O,N-hydroxybis(2-pyridyl)methanolato (η^3-dpkO, OH). The structure of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2 \cdot 2$dmso

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When di-2-pyridyl ketone (dpk) was allowed to react with $[Mo(CO)_6]$ in toluene under reflux in air $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$ where η^3-dpkO,OH is N,O,N-hydroxybis(2-pyridyl)-methanolato was isolated. Infrared and ^{1}H-NMR spectral data measured on solutions of the isolated compound indicate the absence of the carbonyl groups and the presence of coordinated η^3-dpkO,OH, terminal and bridging oxo-groups and elemental analysis confirmed the formulation of the product as $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$. Crystals of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2 \cdot 2$dmso obtained from a dimethyl sulfoxide (dmso) solution of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$ are in the monoclinic $P2_1/c$ space group. X-ray structural analysis confirmed the identity of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$ and shows a dioxomolybdenum oxo-bridged dimer with two terminal oxygen atoms and one tridentate N,O,N-dpkO,OH occupying the coordination sites of the high-valent molybdenum(VI) in a pseudo-octahedral coordination geometry. The molecular packing shows parallel stacks of $[Mo(O)_2(\mu\text{-}O)\ (\eta^3\text{-dpkO,OH})]_2 \cdot 2$dmso and disclose an extensive network of non-covalent interactions within each stack.

Keywords: Molybdenum; Dioxomolybdenum; Oxo-bridged; di-2-Pyridyl ketone; X-ray; Synthesis

1. Introduction

High-valent molybdenum(VI)-oxo compounds have been extensively studied because of their rich physico-chemical properties, reactivity patterns and applications in many important chemical and biological processes that include oxygen and electron transfer in homogenous and heterogeneous catalysis [1–7]. We have been interested in the chemistry of di-2-pyridyl ketone (dpk) and its oxime and hydrazone derivatives (see scheme 1), and reported on the synthesis, characterization and structures of a series of low-valent metal compounds of di-2-pyridyl ketone derivatives, and their applications as molecular sensors for metal ions and biomolecules that include monosaccharide [8–19]. The electrochemical reactions of *fac*-$[Re(CO)_3(dpk)Cl]$ in the presence/absence of CO_2, methylchloroformate ($ClCO_2Me$) and group I and II

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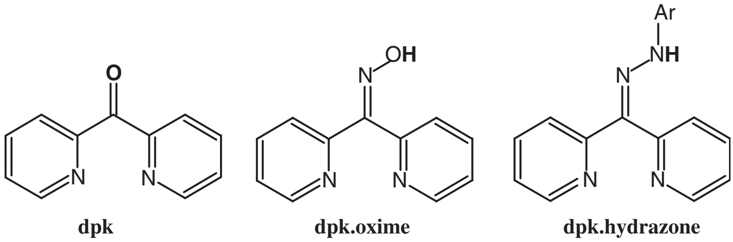


Scheme 1. Di-2-pyridylketone and its oxime and hydrazone derivatives.

metal ions revealed interesting electrochemical behavior and suggested the use of *fac*-[Re(CO)$_3$(dpk)Cl] as an electrochemical sensor for electrophiles [8, 9]. Electrochemical experiments on *fac*-[Re(CO)$_3$(dpk.oxime)Cl] showed the coordinated oxime moiety to act as an electron sink/source and that its first reduction in non-aqueous media is solvent dependent, controlled by the proton accepting ability of the solvent [10]. Optical measurements on dpknph and *fac*-[Re(CO)$_3$(dpknph)Cl] and other related compounds revealed the existence of two charge-transfer bands for the free ligand and its metal compound; that the electronic/charge transfer in *fac*-[Re(CO)$_3$(dpknph)Cl] is faster than in the case of free dpknph; and demonstrated the applicability of *fac*-[Re(CO)$_3$(dpknph)Cl] as a spectrophotometric sensor for metal ions and biomolecules [11–13]. These results prompted us to explore how slight variations in the metal center and ligand backbone may affect the physico-chemical properties of metal compounds of di-2-pyridyl ketone and its oxime and hydrazone derivatives. In the course of investigating the reaction of molybdenum(0) hexacarbonyl, [Mo(CO)$_6$], with dpk in refluxing toluene to prepare low-valent [Mo(CO)$_4$(dpk)], we encountered the formation of a high-valent molybdenum(VI)-dioxo compound of hydrated di-2-pyridyl ketone, N,O,N-hydroxybis(2-pyridyl)methanolato (η^3-dpkO,OH). This marks the first isolated molybdenum compound of a dpk derivative, and points to the facile oxidation of [Mo(CO)$_6$] and use as a synthon for the synthesis of high-valent molybdenum oxo-compounds. In this report we describe the synthesis [Mo(O)$_2$(μ-O)(η^3-dpkO,OH)]$_2$ and solid state structure of its dimethyl sulfoxide solvated derivative.

2. Experimental

2.1. *Reagents and reaction procedures*

Solvents were reagent grade and thoroughly deoxygenated prior to use. All other reagents were obtained from commercial sources and used without further purification.

2.2. *Preparation of [Mo(O)$_2$(μ-O)(η^3-dpkO,OH)]$_2$*

A mixture of [Mo(CO)$_6$] (100 mg, 0.37 mmol), dpk (100 mg, 0.54 mmol) and toluene (20 mL) was allowed to react under reflux in air for 2 h. The resulting brown precipitate was filtered off, washed with hexane, diethyl ether and dried; yield 40 mg (80%)

Table 1. Crystal data and structure refinement for $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)]_2 \cdot 2dmso$.

Empirical formula	$C_{26}H_{30}Mo_2N_4O_{11}S_2$
Formula weight	830.56
Temperature (K)	296(2)
Wavelength (Å)	0.71073
Crystal system, space group	Monoclinic, $P2_1/c$
Unit cell dimensions (Å, °)	$a = 8.8820(10)$, $\alpha = 90$ $b = 11.3560(10)$, $\beta = 93.513(10)$ $c = 16.063(2)$, $\gamma = 90$
Volume (Å^3)	1617.1(3)
Z, Calculated density (g cm^{-3})	4, 1.706
Absorption coefficient (mm^{-1})	0.967
$F(000)$	836
Theta range for data collection (°)	2.20 to 25.50
Range of h, k, l	$-1/10$, $-13/1$, $-19/19$
Reflections collected/unique	4018/3017 [$R_{int} = 0.0300$]
Completeness to theta = 25.50	100.0%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	3017/0/205
Goodness-of-fit on F^2	1.022
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0358$, $wR_2 = 0.0765$
R indices (all data)	$R_1 = 0.0565$, $wR_2 = 0.0826$
Largest diff. peak and hole (e Å^{-3})	0.351 and -0.284

(Found: C, 39.21; H, 2.89. $C_{22}H_{18}Mo_2N_4O_9$ requires C, 39.19; H, 2.69%). Infrared data (KBr disk, cm^{-1}): ν(O–H) ~3425, ν(C=N and C=C of pyridine) 1640–1600, ν(Mo=O) ~945 and ν(Mo–O) ~ 775. ^{1}H NMR (δ ppm) in dmso-d$_6$: 8.50 (d, 1H), 7.75 (t, 1H), 7.37 (t, 1H), 7.14 (d, 1H).

2.3. *Physical measurements*

Solution ^{1}H NMR spectra were recorded on a Bruker ACE 500-MHz Fourier-transform spectrometer and referenced to the residual protons in the incompletely deuterated solvent. Infrared spectra were recorded as KBr pellets on a Perkin-Elmer Spectrum 1000 FT-IR Spectrometer.

2.4. *X-ray crystallography*

Crystals of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)]_2 \cdot 2dmso$ were obtained from a dmso solution of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)]_2$ when allowed to stand for several days. A single crystal was selected and mounted on a glass fiber with epoxy cement. A Bruker AXS with Mo-Kα radiation and a graphite monochromator was used for data collection and the SHELXTL software package version 5.1 was used for structure solution [20, 21]. Cell parameters and other crystallographic information are given in table 1 along with additional details concerning data collection, and table 2 gives a list of atomic coordinates. All non-hydrogen atoms were refined with anisotropic thermal parameters.

Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{Å}^2 \times 10^3$) for $[\mathrm{Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)}]_2 \cdot 2\mathrm{dmso}$. U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

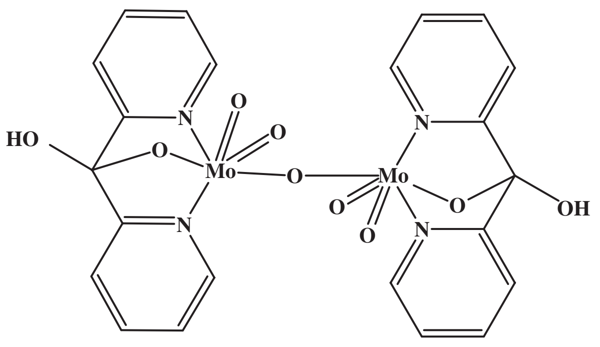
	x	y	z	U_{eq}
Mo	11(1)	1394(1)	4362(1)	30(1)
O(1)	−1587(3)	1230(2)	3728(2)	48(1)
O(2)	−431(3)	2438(2)	5065(2)	49(1)
O(3)	0	0	5000	37(1)
O(4)	1154(3)	2388(2)	3618(1)	36(1)
N(1)	2571(3)	1462(3)	4870(2)	37(1)
N(2)	1294(4)	153(3)	3424(2)	35(1)
C(11)	3167(5)	1266(4)	5642(3)	50(1)
C(12)	4700(5)	1354(4)	5833(3)	63(1)
C(13)	5631(5)	1647(4)	5210(3)	62(1)
C(14)	5016(5)	1848(4)	4415(3)	49(1)
C(15)	3468(4)	1768(3)	4275(3)	39(1)
C(1)	2597(4)	1965(4)	3435(2)	39(1)
C(21)	993(5)	−957(3)	3188(2)	44(1)
C(22)	1764(5)	−1481(4)	2558(2)	51(1)
C(23)	2830(6)	−849(4)	2173(2)	54(1)
C(24)	3164(5)	292(4)	2419(2)	45(1)
C(25)	2359(4)	746(3)	3046(2)	37(1)
S	2937(2)	5564(1)	4049(1)	64(1)
O	4016(4)	4701(3)	3704(2)	75(1)
C(2)	2392(6)	4926(6)	4987(3)	82(2)
C(3)	1219(6)	5409(5)	3455(3)	75(2)
O(5)	3297(3)	2694(3)	2892(2)	55(1)

2.5. *Analytical procedures*

Elemental microanalyses were performed by MEDAC Ltd., Department of Chemistry, Brunel University, Uxbridge, United Kingdom.

3. Results and discussion

Reaction between $[\mathrm{Mo(CO)_6}]$ and dpk in refluxing toluene in air gave $[\mathrm{Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)}]_2$ (see below). The formulation of the isolated compounds as $[\mathrm{Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)}]_2$ was based on the results of its elemental analysis and a number of spectroscopic measurements and corroborated by X-ray structural analysis of a dimethyl sulfoxide solvated crystal of $[\mathrm{Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)}]_2$ obtained when a dmso solution of $[\mathrm{Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)}]_2$ was allowed to stand at room temperature for several days. In the IR spectrum of $[\mathrm{Mo(O)_2(\mu\text{-}O)(\eta^3\text{-}dpkO,OH)}]_2$ peaks appeared in the $\nu(\mathrm{OH})$ and the combined $\nu(\mathrm{C{=}C})$ and $\nu(\mathrm{C{=}N})$ of the pyridine vibrations consistent with the coordination of dpkO,OH, and peaks in the $\nu(\mathrm{Mo{=}O})$ and $\nu(\mathrm{Mo{-}O})$ ranges in accord with the formulation of the compound. In the $\nu(\mathrm{C{\equiv}O})$ region no peaks appeared consistent with the disappearance of the carbonyl groups of $[\mathrm{Mo(CO)_6}]$.

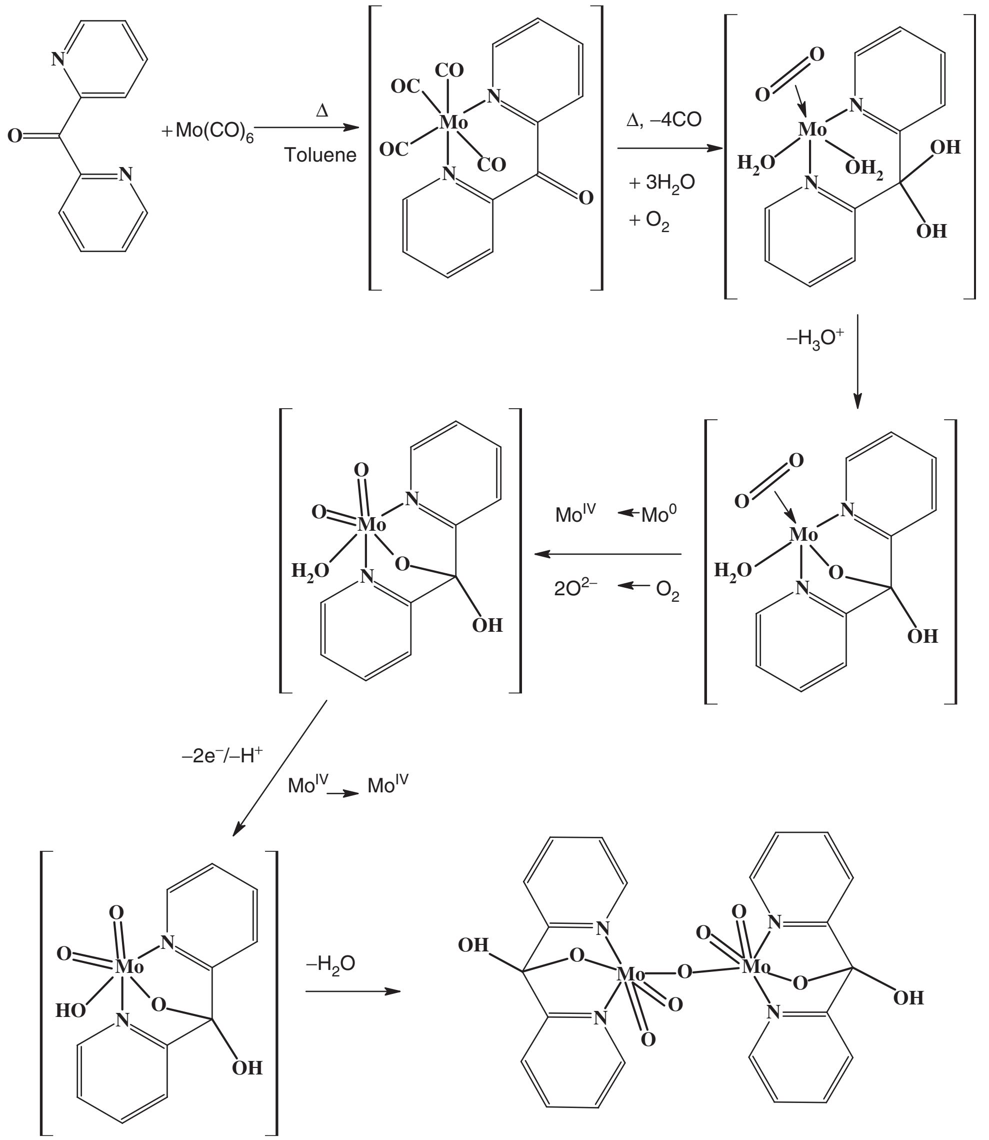


$[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$

The appearance of broad O–H stretching vibration at $3425\,cm^{-1}$ signifies intensive intermolecular hydrogen bonding in the solid state as the free hydroxyl stretching band appears sharp between $3700–3580\,cm^{-1}$ [22]. The ^{1}H-NMR spectra measured in d_6-dmso confirmed the symmetrical coordination of η^3-dpkO,OH as evident by the appearance of four peaks with the same intensity. The proton of the hydroxyl group of coordinated η^3-dpkO,OH was not observed under our experimental conditions, perhaps due to its fast exchange with the water protons of d_6-dmso.

Although detailed analysis of the conversion of low-valent molybdenum(0) hexacarbonyl to high-valent oxobridge-dioxomolybdenum(VI) of η^3-dpkO,OH remains to be explored a plausible mechanism for the conversion is shown in scheme 2. The proposed mechanism is consistent with the tridentate binding of hydrated di-2-pyridyl ketone, the oxidation of $Mo^0 \rightarrow Mo^{VI}$, the dimerization of Mo^{IV}-dioxo and is in accord with the ease of decarbonylation/oxidative decarbonylation of molybdenum-carbonyl compounds [23–25]. Work is in progress in our laboratory to isolate the proposed intermediates and utilize spectroscopic techniques to elucidate the proposed mechanism.

Single crystals of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2 \cdot 2dmso$ grown from a dmso solution of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$ are monoclinic $P2_1/c$. A view of the molecular structure of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2 \cdot 2dmso$ is shown in figure 1 and reveals the presence of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$ and dmso. The molecule possesses an inversion center that coincides with the bridging oxygen atom and solvated dmso and the total molecule is generated by replication of the asymmetric unit $[Mo(O)_2(O)(\eta^3\text{-dpkO,OH})] \cdot dmso$. The coordination about molybdenum is pseudo-octahedral with two terminal oxygen atoms, one bridging oxygen atom, and one oxygen atom, and two nitrogen atoms from the tridentate dpkO,OH occupying coordinated sites. The bridging oxygen atom (O3) and the terminal oxygen atoms adopt *fac*-stereochemistry. The axial sites are occupied by one N atom (N2) from η^3-N,O,N-dpkO,OH and one terminal oxygen atom (O2). The coordinated η^3-N,O,N-dpkO,OH anion forms a six-membered metallocyclic ring (Mo/N1/C15/C1/C25/N2) and two five-membered metallocyclic rings (Mo/Nn/Cn5/C4/O4) where $n = 1$ or 2) fused along the Mo–O4–C1 junction, with the pyridine rings bent away from chelating oxide anion. This arrangement leaves one pyridine ring in the equatorial plane and the other in the axial plane, and the oxygen atom and the hydroxy group (O5–H) of η^3-N,O,N-dpkO,OH exposed for potential intermolecular interactions.



Scheme 2. A plausable mechanism for the conversion of $[Mo(CO)_6]$ to $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]$.

Deviation from octahedral geometry about molybdenum is due to the electron–electron repulsion of the terminal oxygen atoms and adjacent groups coupled with the constraints associated with tridentate binding of η^3-N,O,N-dpkO,OH anion. This is similar to that we reported for high-valent metal-nitrido and amido compounds [26, 27]. These constraints are evident from the bond angles of coordinated atoms (see table 3). The N–Mo–N bite angle of 75.70(10)° for the coordinated η^3-N,O,N-dpkO,OH anion is of the same order as the 74.3(4)° angle reported for the five-membered Re/N/C/C/N metallocyclic ring in *fac*-$[Re(CO)_3(bipy)(OPOF_2)]$ (bipy = 2,2′-bipyridine) [28] and is

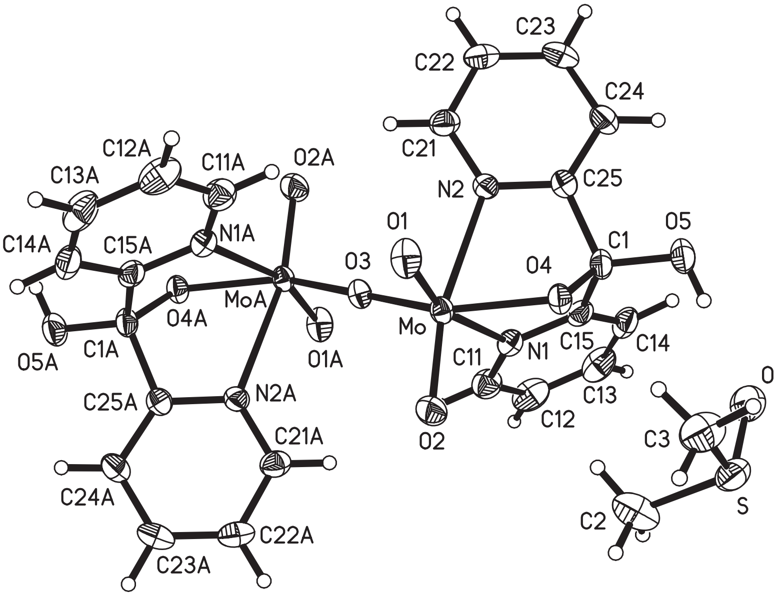


Figure 1. A view of the molecular structure of $[\mathrm{Mo(O)_2}(\mu\text{-O})(\eta^3\text{-dpkO,OH})]_2 \cdot 2\mathrm{dmso}$. The thermal ellipsoids are drawn at the 30% probability level.

Table 3. Bond lengths (Å) and angles (°) for $[\mathrm{Mo(O)_2}(\mu\text{-O})(\eta^3\text{-dpkO,OH})]_2 \cdot 2\mathrm{dmso}$.

Mo–O(2)	1.700(3)	Mo–O(1)	1.706(3)
Mo–O(3)	1.8862(3)	Mo–O(4)	1.970(2)
Mo–N(1)	2.370(3)	Mo–N(2)	2.401(3)
O(4)–C(1)	1.417(4)	N(1)–C(15)	1.328(5)
N(1)–C(11)	1.336(5)	C(11)–C(12)	1.381(6)
C(12)–C(13)	1.377(7)	C(15)–C(1)	1.531(5)
C(1)–O(5)	1.378(4)	C(1)–C(25)	1.528(5)
S–O	1.500(4)	S–C(3)	1.757(5)
S–C(2)	1.767(5)		
O(2)–Mo–O(1)	104.78(14)	O(2)–Mo–O(3)	102.40(9)
O(1)–Mo–O(3)	101.59(9)	O(2)–Mo–O(4)	98.76(12)
O(1)–Mo–O(4)	98.13(11)	O(3)–Mo–O(4)	146.15(7)
O(2)–Mo–N(1)	90.22(12)	O(1)–Mo–N(1)	162.80(12)
O(3)–Mo–N(1)	82.93(7)	O(4)–Mo–N(1)	70.81(10)
O(2)–Mo–N(2)	164.60(13)	O(1)–Mo–N(2)	88.35(12)
O(3)–Mo–N(2)	82.34(7)	O(4)–Mo–N(2)	70.96(10)
N(1)–Mo–N(2)	75.71(10)	$\mathrm{Mo^i}$–O(3)–Mo	180.000(15)
C(1)–O(4)–Mo	116.0(2)	C(15)–N(1)–C(11)	119.4(4)
C(15)–N(1)–Mo	111.4(2)	C(11)–N(1)–Mo	129.2(3)
C(25)–N(2)–C(21)	118.7(3)	C(25)–N(2)–Mo	111.4(2)
C(21)–N(2)–Mo	129.6(3)	N(1)–C(11)–C(12)	121.6(4)
C(13)–C(12)–C(11)	118.9(4)	C(14)–C(13)–C(12)	119.5(4)
C(13)–C(14)–C(15)	118.3(4)	N(1)–C(15)–C(14)	122.3(4)
N(1)–C(15)–C(1)	112.3(3)	C(14)–C(15)–C(1)	125.3(4)
O(5)–C(1)–O(4)	112.1(3)	O(5)–C(1)–C(25)	110.1(3)
O(4)–C(1)–C(25)	106.8(3)	O(5)–C(1)–C(15)	115.0(3)
O(4)–C(1)–C(15)	106.4(3)	O–S–C(3)	106.7(2)
O–S–C(2)	105.1(2)	C(3)–S–C(2)	98.5(3)

Symmetry transformations used to generate equivalent atoms: i. $-x$, $-y$, $-z+1$.

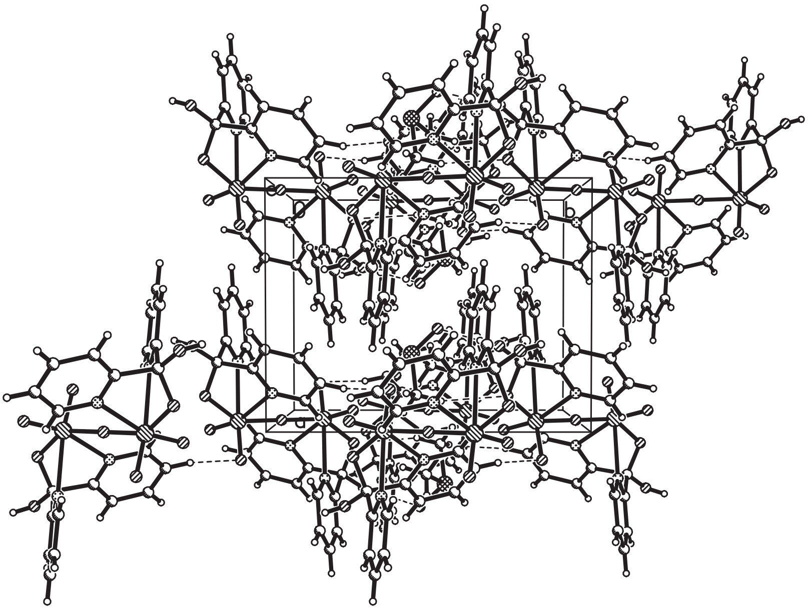


Figure 2. Packing of molecules of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2 \cdot 2dmso$. Non-covalent bonds are represented by dashed line.

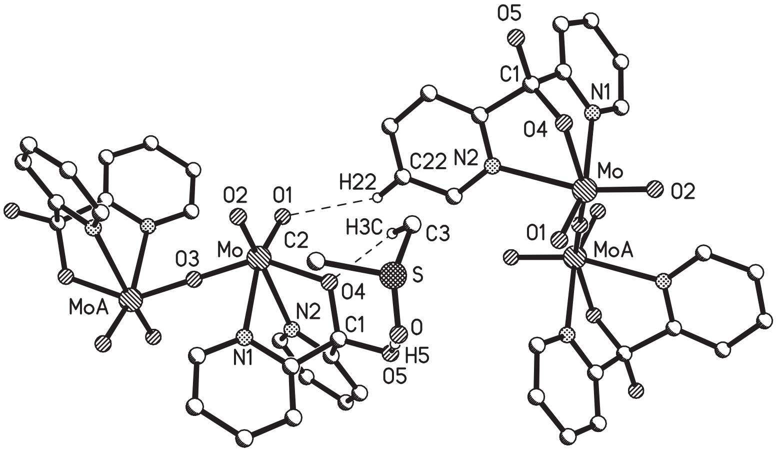


Figure 3. A view of the hydrogen bonds of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2 \cdot 2dmso$.

smaller than the 84.6(4)° reported for the six-membered (Re/N1/C15/C1/C25/N2) metallocyclic ring in $[Re(V)OCl_2(\eta^3\text{-dpkO,OH})]$ [29]. The Mo–O–Mo bond angle of 180° falls in the upper limit of Mo–O–Mo bond angles of oxo-bridged dioxomolybdenum dimers. The bond distances of coordinated atoms are similar to those reported in other oxo-bridged dioxomolybdenum dimers [30].

The molecular packing pattern of molecules of **I** discloses parallel stacks (figure 2). Within each stack there is a web of inter- and intramolecular hydrogen bonds of the type O–H···O and C–H···O involved in stabilization of the structure of (**I**) (see figure 3 and table 4). Intramolecular interaction occurs between solvated dmso

Table 4. Hydrogen bonds of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2 \cdot 2dmso$.

D–H···A	d(D–H)	d(H···A)	d(D···A)	∠(DHA)
O(5)–H(5A)···O	0.82	1.86	2.683(4)	177.7
O(5)–H(5A)···S	0.82	3.01	3.775(3)	155.5
C(22)–H(22A)···O(1)^i	0.93	2.50	3.318(5)	146.8

Symmetry transformations used to generate equivalent atoms: i. $-x$, $y-1/2$,$-z+1/2$.

and the oxide and hydroxy groups of coordinated dpk,OH and intermolecular interaction of the type O···H–C occurs between terminal equatorial oxygen atom (O1) and the hydrogen atom of C22 of the axial pyridine ring of the adjacent molybdenum dimer. The distances and angles of the non-covalent bonds are of the same order as those reported for other compounds [31]. For example, in *fac*-$Re(CO)_3(\eta^3\text{-dpkO,OH})$ hydrogen bond parameters of 0.93, 2.26, 3.183(7) Å, and 171° were observed for the soft non-classical C–H···O hydrogen bond, and parameters of 0.82, 1.86, 2.679(5) Å and 179° were observed for the classical O–H···O hydrogen bond.

Owing to their convenient synthesis, rich physical and chemical properties, potential use in non-linear optics and molecular sensing, work is in progress by us to isolate a variety of metal compounds of polypyridyl-like ligands to explore their electro-optical properties, solid state structures and potential use as catalysts and sensors.

4. Conclusion

The isolation of $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$ marks the first time a molybdenum compound of hydrated dpk η^3-N,O,NdpkO,OH is isolated. Spectroscopic measurements confirmed the absence of the carbonyl groups and revealed the presence of coordinated η^3-dpkO,OH, and terminal and bridging oxygen atoms. X-ray structural analysis on dmso-solvated $[Mo(O)_2(\mu\text{-}O)(\eta^3\text{-dpkO,OH})]_2$ confirmed the identity of the isolated compounds and show distorted octahedral coordination about molybdenum.

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