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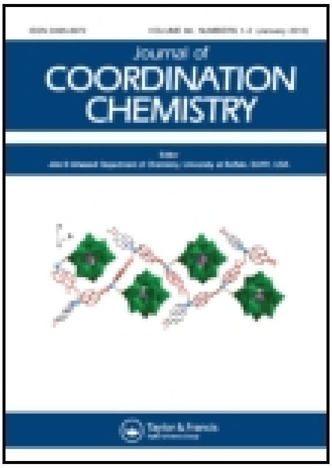

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Molybdenum(VI)-oxodiperoxo complex containing an oxazine ligand: synthesis, X-ray studies, and catalytic activity

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Molybdenum(VI)–oxodiperoxo complex containing an oxazine ligand: synthesis, X-ray studies, and catalytic activity

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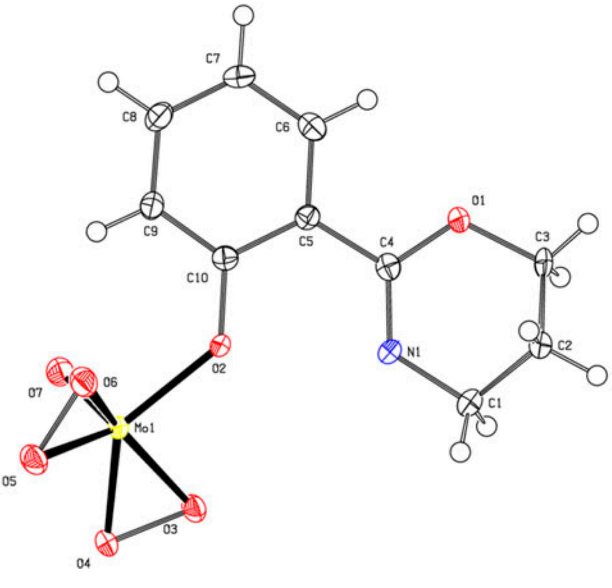

A new mononuclear molybdenum(VI)–oxodiperoxo complex $[MoO(O_2)_2(phox)]$ with a simple bidentate ligand, 2-(2′-hydroxyphenyl)-5,6-dihydro-1,3-oxazine (Hphox), has been synthesized and characterized by X-ray structure analysis, elemental analysis, infrared, and ^{1}H NMR spectroscopy. A triclinic space group *P-1* was determined by X-ray crystallography from single-crystal data of this complex. The resulting complex functioned as a facile sulfide oxidation catalyst with urea hydrogen peroxide as terminal oxidant at room temperature. The catalyst showed efficient reactivity in oxidation of sulfides giving high yield and selectivity.

Keywords: Molybdenum complex; Oxodiperoxo; Oxazine; Crystal structure; Oxidation

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1. Introduction

Selective oxidation of sulfides to sulfoxides is important for producing sulfoxides as chemical intermediates to afford useful chemicals [1–7]. The application of transition metal complexes as catalysts in organic synthesis is an exciting field of research, employed in a wide range of areas of preparative organic chemistry [8–15]. More generally, in the presence of hydrogen peroxide, metals form oxido–peroxido complexes as the basis for the synthesis of a number of fine chemicals [16–19].

The chemistry of transition metal oxido–peroxido complexes has received special attention due to their importance in industrial, pharmaceutical, and biological processes [20–22]. Coordination to a metal center activates peroxido groups towards oxidation of a variety of substrates, rendering oxido–peroxido metal complexes important as intermediates in biological and synthetic catalysis [23–29].

We recently reported a highly efficient method for oxidation reactions by using Mo(VI) oxo–peroxo complexes with oxazoline and Schiff base ligands [30, 31]. In order to study the effect of complex structure on catalytic reactivity, herein, we describe the synthesis of a new mononuclear molybdenum(VI)–oxodiperoxo complex [$MoO(O_2)_2$(phox)] ((Hphox) = 2-(2′-hydroxyphenyl)-5,6-dihydro-1,3-oxazine) and the use of this complex as a catalyst in oxidation of sulfides in the presence of urea hydrogen peroxide (UHP) as an oxidant under air at room temperature (scheme 1).

2. Experimental setup

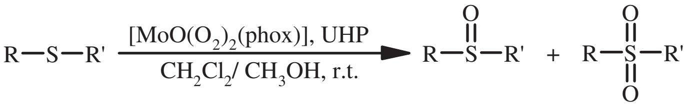
2.1. *General procedure*

Chemicals and solvents were purchased from Fluka and Merck Chemical companies. The ligand 2-(2′-hydroxyphenyl)-5,6-dihydro-1,3-oxazine (Hphox) was synthesized according to a published procedure [32].

Elemental analyses (carbon, hydrogen, and nitrogen) of the compound were obtained from a Carlo ERBA Model EA 1108 analyzer. FT-IR spectrum was obtained by using a Unicam Matson 1000 FT-IR spectrophotometer using KBr disks at room temperature. MS spectra were recorded on a Finnigan MAT95 spectrometer. The products of oxidation reactions were determined and analyzed by an HP Agilent 6890 gas chromatograph equipped with a HP-5 capillary column and a flame-ionization detector. The crystal evaluation and data collection were performed on a Bruker APEX diffractometer (Mo Kα radiation, $\lambda = 0.71073$ Å).

2.2. *Synthesis of [$MoO(O_2)_2$(phox)]*

[$MoO(O_2)_2$(phox)] was synthesized by dissolving MoO_3 (2.5 mM, 0.36 g) in H_2O_2 (30%, 5 cm^3) by stirring at room temperature. Addition of 2-(2′-hydroxyphenyl)-5,6-dihydro-1,3-oxazine (Hphox; 2.5 mM, 0.44 g) dissolved in a minimum volume of ethanol (ca. 15 cm^3)



Scheme 1. Oxidation of sulfides by the [$MoO(O_2)_2$(phox)]/UHP catalytic system.

to the above solution under stirring gave a yellow solid. The solid was filtered off, washed with water, methanol, and finally with diethyl ether, and dried in vacuum. Orange crystals of the complex were obtained with slow evaporation from an acetonitrile solution of the product. Yield 0.57 g (65%). $C_{10}H_{10}MoNO_7$ (352.13): Calcd C 34.07, H 2.84, N 3.98; found C 33.92, H 3.06, N 4.05; Selected IR frequency (KBr disk, cm^{-1}): 1644($\nu_{C=N}$), 966 ($\nu_{(Mo=O)}$) and 860 ($\nu_{(O–O)}$); ^1H NMR (500 MHz, DMSO-d^6, δ): 1.96 (q, 2H), 3.54 (d, 2H), 4.41 (t, 2H), 6.70–7.63 (m, 4H).

2.3. *General procedure for sulfide oxidation*

To a solution of sulfide (0.2 mM), chlorobenzene (0.2 mM) as internal standard and $[MoO(O_2)_2(phox)]$ (0.015 mM) in a 1 : 1 mixture of CH_3OH/CH_2Cl_2 (1 mL), 0.4 mM UHP was added as the oxidant. The mixture was stirred at room temperature and the reaction progress was monitored by GC. Assignments of products were made by comparison with authentic samples.

3. Results and discussion

3.1. *Complex characterization*

$[MoO(O_2)_2(phox)]$ was synthesized by dissolving MoO_3 (freshly precipitated) in H_2O_2 and treating the resulting solution with 2-(2′-hydroxyphenyl)-5,6-dihydro-1,3-oxazine dissolved in ethanol. The complex is air stable and was fully characterized by spectroscopy (^1H NMR and IR), elemental analysis, and X-ray crystallography. In order to study the binding of the ligand to molybdenum in the new complex, the IR spectrum of the free ligand was compared with the spectrum of the molybdenum complex. The absorption of the complex at 1644 cm^{-1} assigned to the C=N of the ligand did not shift showing that the imine nitrogen is not coordinated [30]. The $\nu_{(Mo=O)}$ and $\nu_{(O–O)}$ vibrations of the complex appear around 966 and 860 cm^{-1}, respectively [30]. The coordinating modes of the ligand were confirmed by comparing ^1H NMR patterns of the ligand and the complex. The signal appearing at $\delta = 14.2$ ppm, due to the phenolic OH of the ligand, disappears in the spectrum of the complex. The ^1H NMR spectrum of the other protons of the complex exhibits almost no changes in comparison with the ligand and all the signals in the complex were very similar to that of the ligand and appear nearly at the same positions. Each of the hydrogens in the oxazine ring and aromatic ring show distinct peaks at 1.96–4.41 and 6.70–7.63 ppm, respectively [32].

The ORTEP view of the complex with atom numbering scheme is shown in figure 1. A summary of the single-crystal X-ray structure refinement is shown in table 1, and selected bond lengths and angles for the complex are listed in table 2.

The geometry around Mo can best be described as distorted pentagonal pyramidal with the axial site occupied by oxo (O_7) (Mo–O_7 distance 1.6694(19) Å). The phenolate oxygen (O_2) with significantly longer bond length of 2.0229(19) Å and the peroxo moieties (Mo–O_3, Mo–O_4 distances 1.9850(19) and 1.9333(19) Å; Mo–O_5, Mo–O_6 distances 1.926(2) and 1.923(2) Å) define the equatorial plane. The structural parameters given in figure 1 are consistent with those reported for oxodiperoxo–Mo(VI) complexes [33, 34].

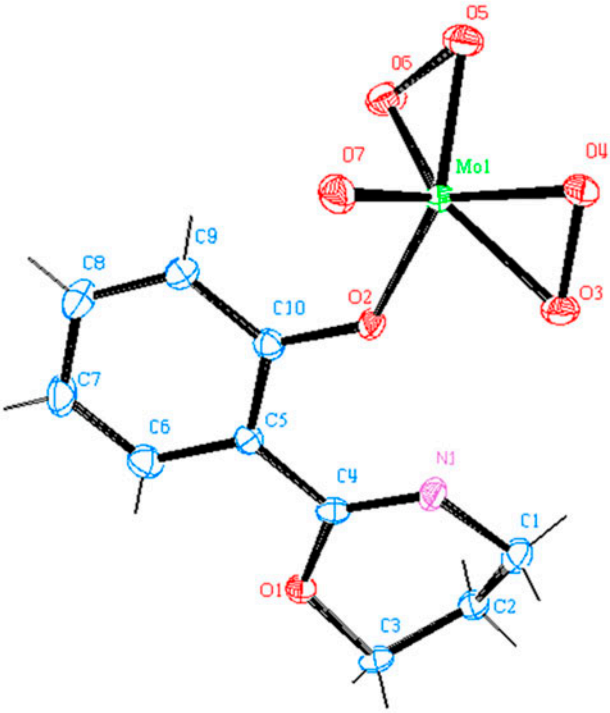


Figure 1. ORTEP diagram of [$MoO(O_2)_2$(phox)] with thermal ellipsoids drawn at a 50% probability limit.

The observation that greater stability of the oxodiperoxo–Mo(VI) complexes is attained when the two peroxo groups coordinate in the equatorial plane is consistent with our structure [35].

3.2. *Catalytic activity*

In order to evaluate the catalytic activities of [$MoO(O_2)_2$(phox)] for oxidation of sulfides, the reactions were optimized according to the oxidation of methylphenyl sulfide (MPS) through investigation of the factors that influence oxidation, such as solvent, the amount of catalyst, and the amount of UHP.

In order to understand the effect of solvent on the catalytic reactivity, a series of reactions was investigated by performing the model reaction in dichloromethane, acetonitrile, methanol, dichloroethane, and (1 : 1) mixture of CH_2Cl_2/CH_3OH. Among the solvents examined, the (1 : 1) mixture of CH_2Cl_2/CH_3OH was found to be best for this protocol (table 3). The conversion of MPS monotonously increases with the addition of catalyst from 0.0025 to 0.015 mM. When the amount of catalyst is increased to 0.02 mM, the conversion of MPS oxidation reduces from 87 to 81%. The amount of H_2O_2 could also affect the conversion and methylphenylsulfoxide selectivity (table 3). When the amount of H_2O_2 was increased from 0.1 to 0.4 mM, the conversion of MPS increased from 25 to 94%. With a further increase of H_2O_2 to 0.5 mM, the selectivity to methylphenylsulfoxide decreased from 95 to 88%.

Table 1. Crystal data and structure refinement for [MoO$(O_2)_2$(phox)].

Empirical formula	$C_{10}H_{10}MoNO_7$
Formula weight	352.13
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group	*P-1*
Unit cell dimensions	$a = 8.4403(11)$ Å
	$\alpha = 65.1050(17)°$
	$b = 8.6815(12)$ Å
	$\beta = 68.9470(18)°$
	$c = 9.5957(13)$ Å
	$\gamma = 68.6800(18)°$
Volume	576.18(13) $Å^3$
Z	2
Density (Calcd)	2.030 g cm^{-3}
Absorption coefficient	1.169 mm^{-1}
$F(0\,0\,0)$	350
Crystal size	0.240 × 0.270 × 12.000 mm
θ Range for data collection	2.41°–25.03°
Index ranges	$-10 \le h \le 10$, $-10 \le k \le 10$, $-11 \le l \le 11$
Reflections collected	4731
Independent reflections	2034 [$R_{int} = 0.0211$]
Completeness to $\theta = 28.70°$	99.9%
Absorption correction	multi-scan
Max. and min. transmission	0.7670 and 0.0250
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	2034/0/172
Goodness-of-fit on F^2	1.121
Final R indices [$I > 2\sigma(I)$]	$R_1 = 0.0220$, $wR_2 = 0.0536$
R indices (all data)	$R_1 = 0.0245$, $wR_2 = 0.0550$
Largest diff. peak and hole	0.928 and −0.315 e$Å^{-3}$

Note: $R_{int} = \Sigma|F_o^2 - F_o^2(\text{mean})|/\Sigma[F_o^2]$;
$R_1 = \Sigma||F_o| - |F_c||/\Sigma|F_o|$;
GOOF $= S = \{\Sigma[w(F_o^2 - F_c^2)^2]/(n-p)\}^{1/2}$;
$wR_2 = \{\Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]\}^{1/2}$;
$w = 1/[\sigma(F_o^2) + (aP)^2 + bP]$, where P is $[2F_c^2 + \text{Max}(F_o^2, 0)]/3$.

Table 2. Significant bond lengths (Å) and angles (°) for [MoO$(O_2)_2$(phox)].

Bond lengths (Å)		Bond angles (°)			
Mo1–O7	1.6694(19)	O7–Mo1–O6	104.59(10)	O7–Mo1–O5	103.30(9)
Mo1–O6	1.923(2)	O6–Mo1–O5	44.95(8)	O7–Mo1–O4	100.34(9)
Mo1–O5	1.926(2)	O6–Mo1–O4	130.19(8)	O5–Mo1–O4	87.75(8)
Mo1–O4	1.9333(19)	O7–Mo1–O3	107.65(9)	O6–Mo1–O3	147.72(9)
Mo1–O3	1.9850(19)	O6–Mo1–O2	88.42(8)	O5–Mo1–O2	132.50(8)
Mo1–O2	2.0229(19)	O4–Mo1–O2	130.95(8)	O3–Mo1–O2	86.69(8)
O3–O4	1.478(3)	O3–O4–Mo1	69.71(11)	O4–O3–Mo1	66.00(10)
O5–O6	1.472(3)	O5–O6–Mo1	67.65(11)	O6–O5–Mo1	67.40(11)

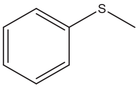
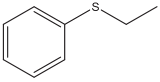
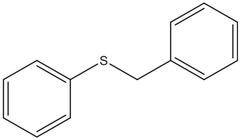
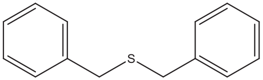
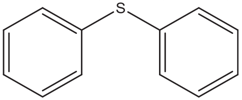
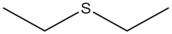
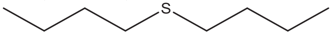
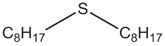
A series of structurally diverse sulfides were subjected to oxidation reaction using [MoO$(O_2)_2$(phox)] as catalyst and UHP as oxidant (table 4). The reactions were run with molar ratio of 1 : 13 : 26 for catalyst : substrate : UHP that was found to be optimum. Arylalkyl (table 4, entries 1,2), arylbenzyl (table 4, entry 3), dibenzyl (table 4, entry 4), diaryl (table 4, entry 5) and dialkyl (table 4, entries 6–8) sulfides underwent clean and selective oxidation to the corresponding sulfoxides in air, with impressive selectivity (63–100%). Very good

Table 3. The effect of various conditions in oxidation of MPS by $[MoO(O_2)_2(phox)]$.

Entry	Amount of catalyst (mM)	Amount of UHP (mM)	Solvent (1 mL)	Conversion (%)[a]	Selectivity to sulfoxide (%)[b]
1	0.01	0.2	CH_2Cl_2	Trace	–
2	0.01	0.2	CH_3OH	72	85
3	0.01	0.2	$C_2H_4Cl_2$	Trace	–
4	0.01	0.2	CH_3CN	55	75
5	0.01	0.2	CH_3OH/CH_2Cl_2	77	96
6	0.0025	0.2	CH_3OH/CH_2Cl_2	75	98
7	0.005	0.2	CH_3OH/CH_2Cl_2	71	98
8	0.015	0.2	CH_3OH/CH_2Cl_2	87	97
9	0.02	0.2	CH_3OH/CH_2Cl_2	81	89
10	0.015	0.1	CH_3OH/CH_2Cl_2	25	100
11	0.015	0.3	CH_3OH/CH_2Cl_2	90	96
12	0.015	0.4	CH_3OH/CH_2Cl_2	94	95
13	0.015	0.5	CH_3OH/CH_2Cl_2	95	88
14	–	0.4	CH_3OH/CH_2Cl_2	11	100

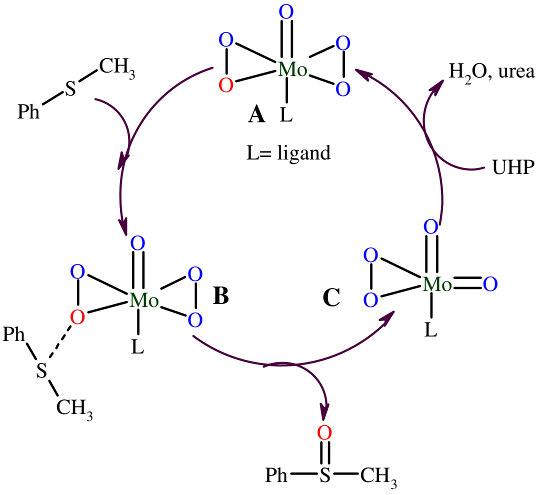
[a] The GC conversion (%) are measured relative to the starting substrate.
[b] Selectivity to sulfoxide = (sulfoxide/(sulfoxide + sulfone))% × 100.

Table 4. Oxidation of sulfides catalyzed by $[MoO(O_2)_2(phox)]$/UHP.[a]

Entry	Substrate	Conversion (%)[b] (TON)[c]	Selectivity (%)[d]
1		94(12.5)	95
2		94(12.5)	67
3		90(12)	91
4		97(12.9)	91
5		71(9.5)	63
6		100(13.3)	100
7		100(13.3)	100
8		100(13.3)	100

[a] The molar ratios for $[MoO(O_2)_2(phox)]$: substrate : oxidant are 1 : 13 : 26. The reactions were performed in (1 : 1) mixture of CH_2Cl_2/CH_3OH (1 mL) under air at room temperature within 20 min.
[b] The GC yield (%) are measured relative to the starting sulfide.
[c] TON = (mM of sulfoxide + mM of sulfone)/mM of catalyst.
[d] Selectivity to sulfoxide = (sulfoxide/(sulfoxide + sulfone))% × 100.

conversions of substrates depending on the nature of the sulfide from 71 to 100% were obtained for all cases. Aromatic and aliphatic sulfides undergo oxidation reactions more easily than diaryl substrates. The lowest conversion was obtained for diphenyl sulfide.



Scheme 2. Proposed mechanism for sulfide oxidation by the $[MoO(O_2)_2(phox)]$/UHP catalytic system.

The mechanism of oxidation in the presence of oxodiperoxo–Mo(VI) complexes is widely accepted even though detailed mechanistic investigations have led to a more refined understanding in recent years [1]. The mechanism for the catalytic oxidation of sulfides catalyzed by the oxodiperoxo–Mo(VI) complex $[MoO(O_2)_2(phox)]$ has been proposed (scheme 2) by comparison with literature reports [1]. The catalytic cycle begins with the addition of sulfide to complex **A** to form intermediate **B** that can transfer an oxygen atom to sulfide. Then, UHP can react with generated intermediate **C** to regenerate the catalyst.

4. Conclusion

The six-coordinate oxodiperoxo–Mo(VI) complex $[MoO(O_2)_2(phox)]$ has been synthesized and characterized by physico-chemical methods. Solid state studies further exemplified the distorted pentagonal pyramidal geometry around Mo. In this study, we have demonstrated the effectiveness of this complex as a catalyst for the oxidation of sulfides to their corresponding sulfoxides. Easy preparation, mild reaction conditions, high yields of the products, short reaction time, and high selectivity make this catalytic system a useful method for oxidation reactions.

Supplementary material

The CIF file of crystal structure complex $[MoO(O_2)_2(phox)]$ has been deposited with the CCDC, No. 980856. This data can be obtained free of charge via http://www.ccdc.cam.ac.uk/conts/retrieving.html, or from the Cambridge Crystallographic Data Center, 12 Union Road, Cambridge CB2 1EZ, UK; Fax: +44 1223 336 033; or E-mail: deposit@ccdc.cam.ac.uk.

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