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Synthesis and Characterization of a Dioxomolybdenum(VI) Aminohydrazone Complex and its Performance as an Efficient Homogeneous Catalyst in the Selective Oxidation of Benzylic Alcohols

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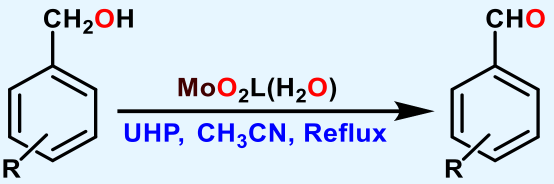


Abstract: A dioxomolybdenum(VI) complex was successfully synthesized by the reaction between [$MoO_2(acac)_2$] and a tridentate ligand obtained from the condensation of 4-aminobenzohydrazide and 3-ethoxysalicylaldehyde. The structures of synthesized products were explored through elemental analysis (CHN) and various techniques such as FT-IR, ^1H and ^{13}C NMR spectroscopies. Moreover, the complex was utilized in the selective oxidizing of benzylic alcohols to the corresponding aryl aldehydes by the use of urea hydrogen peroxide (UHP) in acetonitrile under reflux conditions. The present protocol has several advantages, including short reaction times, high product yields and simplicity in operation.

Keywords: Dioxomolybdenum(VI) complex, Tridentate Schiff base, Selective oxidation, Benzylic alcohols, Homogeneous catalyst

1. INTRODUCTION

During the past few years, much attention has been paid to the coordination chemistry of molybdenum due to their biochemical significance.[1-3] In addition to the vital role of dioxomolybdenum(VI) complexes with aroylhydrazones in biological activities, their high catalytic activities have made them more attractive in synthetic processes.[4-6] These complexes have appreciable applications as catalysts in organic transformations, specifically in the oxidation of various organic compounds. The MoO_2(VI) complexes have been extensively utilized as oxidation catalysts in the epoxidation of olefins,[7] hydroxylation of benzene,[8] oxidation of sulfides,[9] and particularly oxidation of alcohols.[10-12]

Hydrazones, a particular group of Schiff bases, have many potential applications in various biological, catalytic, optical and analytical fields.[13,14] These compounds are effective antibacterial, antifungal, antiviral, antineoplastic, and DNA photocleaving agents.[15-17] Hydrazones can adopt different modes of coordination according to the requirements of geometries and oxidation states of various metal ions to form coordination complexes.[18]

One of the most widespread reactions in organic syntheses is the oxidation of primary and secondary alcohols to the related carbonyl compounds.[19] This reaction is very important because the carbonyl compounds are essential intermediates in synthesizing different kinds of dyes, agrochemicals, perfumes, artificial flavoring and medicines.[20] In traditional methods, stoichiometric amounts of oxidants such as permanganates, chromates, etc., were utilized to oxidize alcohols.[21-23] The use of conventional reagents has many drawbacks such as the production of environmental pollutants, low yield and poor efficiency.[24] Also, these methods generate by-products and consume large amounts of oxidizing reagents.[25,26] There are several developed procedures in the literature for this transformation to overcome the problems mentioned above.[27-30]

Considering the advantages of metal complexes and our ongoing efforts to prepare the new metal complexes,[31-38] we attempted to synthesize a new dioxomolybdenum complex using a tridentate Schiff base ligand derived from 3-ethoxysalicylaldehyde and 4-aminobenzohydrazide. The prepared complex was characterized by employing conventional techniques. After successful synthesis and characterization of the target complex, its catalytic efficiency was evaluated in the selective oxidation of benzylic alcohols.

2. EXPERIMENTAL

Materials and methods

All of the materials and solvents are of analytical grade and purchased from Fluka, Merck and Sigma-Aldrich companies. The elemental composition (CHN) was determined by using Heraeus CHN-O-FLASH EA 1112 instrument. ^1H and ^{13}C NMR spectra were recorded with the help of BRUKER AVANCE 400 MHz spectrometer. Tetramethylsilane (TMS) was employed as an internal standard reference. The chemical shift values (δ) are assigned by comparing them with TMS, whose value is arbitrarily fixed as zero ppm. Infrared spectra were taken after making KBr pellets by using an IRPrestige-21 spectrophotometer (Shimadzu).

Preparation of *(E)*-4-amino-*N'*-(3-ethoxy-2-hydroxybenzylidene)benzohydrazide (H_2L) ligand

3-Ethoxysalicylaldehyde (10 mmol, 1.67 g) and 4-aminobenzohydrazide (10 mmol, 1.52 g) were separately dissolved in 50 mL hot MeOH. The solutions were mixed with continual stirring after complete dissolution and the resulting mixture was refluxed for 3 h. On completion of the reaction monitored by TLC (ethyl acetate:*n*-hexane, 3:7), the reaction mixture was cooled to room temperature. The precipitated solid product was collected with the help of filter paper and washed thrice with cold MeOH to remove the possible impurities. The Schiff base ligand was spectroscopically pure and used in the synthesis of the corresponding metal complex. Yield 83%. Anal. Calc. for $C_{16}H_{17}N_3O_3$: C, 64.20; H, 5.72; N, 14.04, Found: C, 64.11; H, 5.76; N, 13.95%. FT-IR (KBr, cm^{-1}); 3570 ($\nu_{N–H}$); 1660 ($\nu_{C=O}$); 1606 ($\nu_{C=N}$); 1477, 1591 ($\nu_{C=C}$); 1161 ($\nu_{C–O}$); 1014 ($\nu_{N–N}$). ^1H NMR (400 MHz DMSO-d_6, ppm): 1.35 [3H , (H-C8), t, ^3J = 6.8 Hz], 4.06 [2H, (H-C7), q, ^3J = 6.8 Hz], 5.86 [2H, s, (-NH_2),], 6.61 [2 H, (H-C13, H-C15), t, ^3J = 8.8 Hz], 6.83 [1H, (H-C4), t, ^3J = 8.0 Hz], 7.00 [1H, (H-C3), dd, ^3J = 8.0 Hz, ^4J = 1.2 Hz], 7.07 [1H, (H-C5), dd, ^3J = 8.0 Hz, ^4J = 1.2 Hz], 7.69 [2H, (H-C12, H-C16), d, ^3J = 8.8 Hz], 8.56 [1H, s, (-CH=N),], 11.35 [1H, s, (-NH)], 11.74 [1H, s, (-OH)]. ^{13}C NMR (100 MHz, DMSO-d_6, ppm): 14.8 (C8), 64.0 (C7) 112.6 (C13, C15), 115.0 (C3), 118.7 (C6), 118.8 (C4), 119.0 (C11), 121.3 (C5), 129.4 (C12, C16), 146.9 (C2), 147.0 (C1), 147.4 (C14), 152.5 (C9), 162.5 (C10).

Preparation of $[MoO_2L(H_2O)]$ complex

The dioxomolybdenum complex was prepared by suspending equimolar amounts of $[MoO_2(acac)_2]$ (0.5 mmol, 0.165 g) and H_2L (0.5 mmol, 0.150 g) in 50 mL of MeOH. The contents of the suspension were refluxed for roughly 3 h. After that, two-thirds of the solvent was evaporated and the solution was cooled using ice bath. The resultant solid product was then filtered off and washed with H_2O, MeOH and Et_2O, separately, and finally dried in a desiccator under an inert atmosphere. Yield 76%. Anal. Calc. for $C_{16}H_{17}MoN_3O_6$: C, 43.35; H, 3.87; N, 9.48, Found: C, 43.24; H, 3.84; N, 9.59 %. FT-IR (KBr, cm^{-1}); 1600 ($\nu_{C=N}$); 1448 ($\nu_{C=N-N=C}$); 1435, 1566 ($\nu_{C=C}$); 1257 ($\nu_{C–O}$); 1029 (ν_{N-N}); 935 ($\nu_{O=Mo=O}$) *asym*; 914 ($\nu_{O=Mo=O}$) *sym*; 569 (ν_{Mo-O}); 480 (ν_{Mo-N}). ^1H NMR (400 MHz, DMSO-d_6, ppm): 1.34 [3H, (H-C8), t, ^3J = 6.8 Hz,], 4.06 [2H, (H-C7), q, ^3J = 6.8 Hz], 5.96 [2H, s, (-NH_2),], 6.60 [2H, (H-C13, H-C15), t, ^3J = 8.8 Hz], 6.97 [1H, (H-C4), t, ^3J = 8.0 Hz], 7.18 [1H, (H-C3), dd, ^3J = 8.0 Hz, ^4J = 1.2 Hz], 7.25 [1H, (H-C5), dd, ^3J = 8.0 Hz, ^4J = 1.2 Hz], 7.69 [2H, (H-C12, H-C16), d, ^3J = 8.8 Hz], 8.76 [1H, s, (-CH=N),]. ^{13}C NMR (100 MHz, DMSO-d_6, ppm): 14.7 (C8), 64.2 (C7) 113.1 (C13, C15), 115.6 (C3), 117.8 (C6), 121.0 (C4), 121.2 (C11), 125.0 (C5), 129.9 (C12, C16), 147.5 (C2), 149.1 (C1), 152.7 (C14), 153.1 (C9), 169.4 (C10).

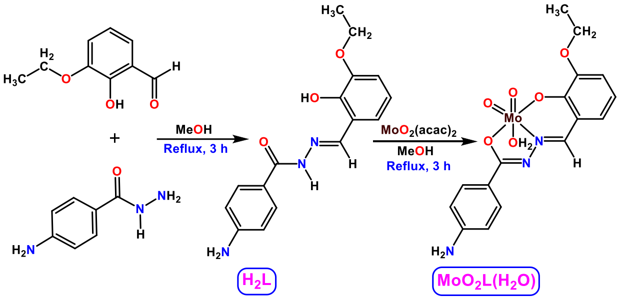
Typical method for the oxidizing of benzylic alcohols in the presence of $[MoO_2L(H_2O)]$ complex

To a solution of benzyl alcohol (1 mmol) and UHP (2 mmol) in 5 mL CH_3CN, 0.006 mmol of the catalyst was added and refluxed for the specified time as shown in Table 4. The progress of the reaction was monitored using TLC (ethyl acetate:*n*-hexane, 2:5). On completion of the reaction, the solvent was evaporated under reduced pressure to get the desired product in high yield (92 %) after chromatography using silica gel.

3. RESULTS AND DISCUSSION

Synthesis

An ONO tridentate ligand (H_2L) was synthesized through the treatment of 3-ethoxysalicylaldehyde with 4-aminobenzohydrazide in refluxed methanol. The reaction of H_2L (*(E)*-4-amino-*N'*-(3-ethoxy-2-hydroxybenzylidene)benzohydrazide) with $[MoO_2(acac)_2]$ in MeOH under reflux conditions produces the dioxomolybdenum complex $[MoO_2L(H_2O)]$ (Scheme 1).



Scheme 1. Preparation of H_2L Schiff base ligand and $[MoO_2L(H_2O)]$ complex

FT-IR spectral studies

Figure 1 shows the important FT-IR spectral absorption peaks of the H_2L and $[MoO_2L(H_2O)]$ in the form of a stacked spectrum. In the vibrational spectrum of the ligand, two significant peaks that appeared at 3570 and 1660 cm^{-1} are attributed to the stretching vibrational frequencies of the hydrazoneic (NH) and carbonyl (C=O) groups, respectively. The disappearance of these peaks in the complex spectrum indicates deprotonation and the involvement of these sites in coordination with the metal atom. A sharp peak in the spectrum of the ligand belongs to azomethine chromophore at 1606 cm^{-1} is shifted to lower wavenumber (1600 cm^{-1}) after complexation, which demonstrates the involvement of this moiety in coordination.[39,40] Another indication of donor site contribution is the shifting of stretching vibrational peak belonging to phenolic oxygen in H_2L from 1161 to 1257 cm^{-1} upon complex formation.[41,42] The stretching frequencies of the NH_2 group in the ligand at 3238 and 3350 cm^{-1} changed significantly to 3367 and 3468 cm^{-1} upon the establishment of the metal complex.

The *cis*-MoO_2 moiety gave its peculiar asymmetric and symmetric stretching peaks at 935 and 914 cm^{-1}. Moreover, stretching vibrations of Mo-N and Mo-O were observed at 480 and 569 cm^{-1}, respectively. These molybdenum-associated vibrations are in good agreement with the closely analogous compounds previously reported in the literature.[43,44] The peak appeared at 895 cm^{-1} can be attributed to the stretching vibration of Mo-OH_2 that indicates the water is coordinated to the metal center as the sixth coordination site of the complex. This is confirmed by the broadband peak visible at 2800-3500 cm^{-1}.[45]

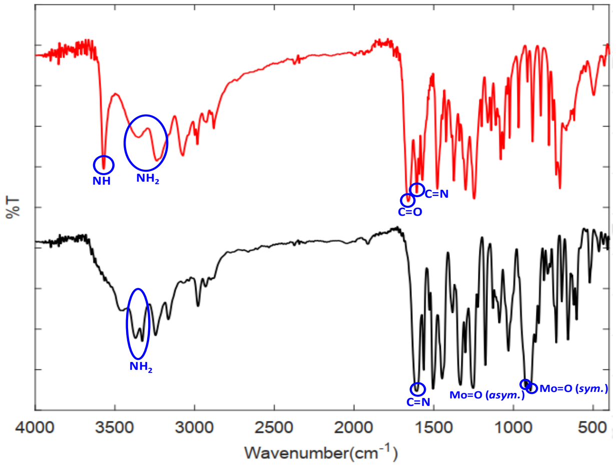


Figure 1. A stacked FT-IR spectrum of the H_2L (top) and $[MoO_2L(H_2O)]$ (bottom).

^{1}H and ^{13}C NMR spectral studies

The ^{1}H and ^{13}C NMR spectra of the synthesized ligand and its corresponding dioxomolybdenum complex are manifested in Figures 2-4 and the spectral details are given in the experimental section. In the ^{1}H NMR spectrum of the ligand, there are two singlet signals at δ = 11.74 and 11.35 ppm related to –OH (phenolic) and –NH protons, respectively. These peaks are disappeared after complexation, indicating that phenolate and enolate oxygen atoms of the ligand are involved in coordinating with the molybdenum. This also reveals the keto-imine tautomerism on complex formation. Moreover, a singlet of azomethine (–HC=N) proton at δ = 8.56 ppm, observed in the spectrum of the ligand, was shifted downfield at δ = 8.76 ppm showing the deshielding due to the decrease in the electronic density upon the coordination of azomethine nitrogen with the metal ion.

The position of aromatic protons in the complex spectrum shows a slight shifting relative to that in the ligand spectrum. A singlet of the amino proton (–NH_2) at δ = 5.86 ppm, observed in the spectrum of the ligand, was also shifted downfield at δ = 5.96 ppm showing the deshielding in the complex. The ethoxy substituent of the ligand shows a quartet at δ = 4.06 ppm and a triplet at δ = 1.35 ppm due to –OCH_2 and –CH_3 protons, respectively, with coupling constant of the same value ($^3J_{H,H}$ = 6.8 Hz). The aliphatic protons did not show any noticeable changes in their chemical shifts during complexation.

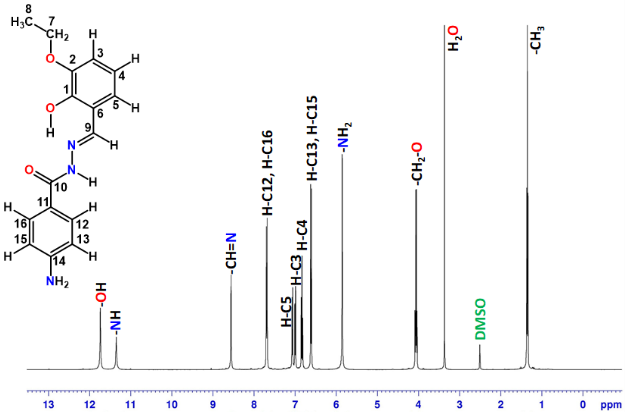


Figure 2. ^{1}H NMR spectrum of the H_2L in deuterated DMSO.

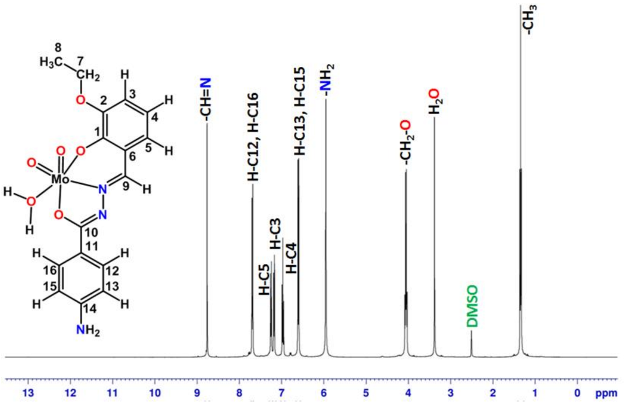


Figure 3. ^{1}H NMR spectrum of the $[MoO_2L(H_2O)]$ in deuterated DMSO.

The three important signals for the carbonyl, phenolic, and methine carbons were observed in ^{13}C NMR of the ligand at 162.5, 152.5, and 147.0 ppm, respectively. The carbon atoms present nearby of the complexing atoms (i.e., C10, C1 and C9) exhibited a significant swap in the location of their signals because of coordination, verifying the involvement of these species in complex formation. The remaining aromatic carbons of H_2L and $[MoO_2L(H_2O)]$ were observed in their particular localities according to the earlier reports. Hence, ^{13}C NMR spectral data support the conclusions derived from ^{1}H NMR spectral details.

Catalytic activity studies

After successfully synthesizing and characterizing the $[MoO_2L(H_2O)]$ complex, its catalytic efficiency was evaluated in the selective oxidation of benzylic alcohols under neutral conditions. To optimize the reaction conditions, the oxidation of benzyl alcohol was selected as a model. In order to find a suitable solvent, different polar and non-polar solvents such as methanol, ethanol, acetonitrile, acetone, carbon tetrachloride, chloroform, dichloromethane and 1,2-dichloroethane were examined by the use of urea hydrogen peroxide (UHP) in the presence of a catalytic amount of $[MoO_2L(H_2O)]$ complex. The results indicated that CH_3CN under reflux conditions gives a higher yield of benzaldehyde (Table 1).

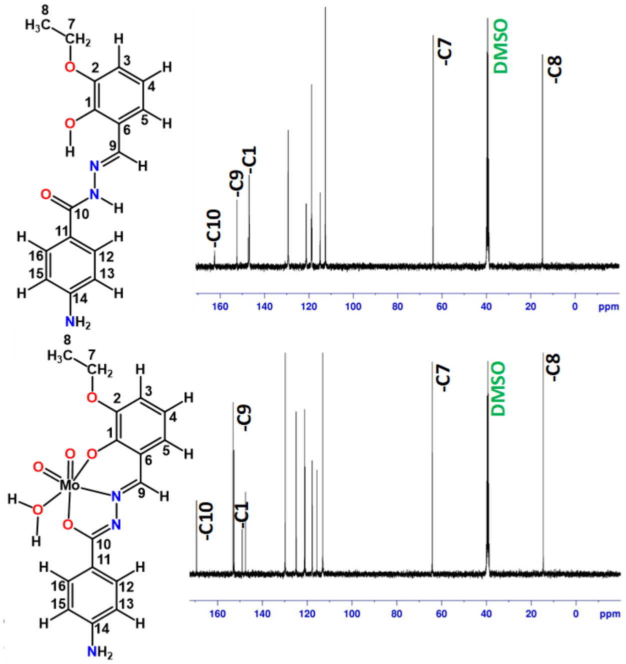


Figure 4. ^{13}C NMR spectra of the H_2L and $[MoO_2L(H_2O)]$ in deuterated DMSO.

Table 1. Effect of solvent on oxidation of benzyl alcohol catalyzed by $[MoO_2L(H_2O)]$ complex[a]

Entry	Solvent	Conditions	Yield (%)[b]	TOF (h^{-1})
1	CH_3CH_2OH	Reflux	45	37.5
2	CH_3OH	Reflux	53	44.2
3	$ClCH_2CH_2Cl$	Reflux	33	27.5
4	CH_3COCH_3	Reflux	58	48.3
5	$CHCl_3$	Reflux	26	21.7
6	CCl_4	Reflux	Trace	-
7	CH_2Cl_2	Reflux	Trace	-
8	**CH_3CN**	**Reflux**	**92**	**76.7**
9	CH_3CN	r.t.	Trace	-
10	CH_3CN	40 ºC	60	50
11	CH_3CN	60 ºC	73	60.8
12	CH_3CN	70 ºC	80	70

[a] Reaction conditions: benzyl alcohol (1 mmol), UHP (2 mmol), catalyst (0.006 mmol), solvent (5 mL), 2 h
[b] Isolated yield.

The effect of various oxidants like $NaIO_4$, H_2O_2, Bu_4NIO_4, *tert*-BuOOH and UHP was also tested in the oxidation of benzyl alcohol in the presence of $[MoO_2L(H_2L)]$ catalyst in CH_3CN under reflux conditions and the results showed that UHP is the best oxygen source in this reaction. The optimal amount of the oxidant was 2 mmol. With lower amounts, the reaction did not complete even at higher reaction times and higher amounts of the oxidant had no significant effect on the yield of produced aldehyde (Table 2). It is noteworthy that in the presence of large amounts of oxidant (3 mmol and higher) in addition to aldehyde, some carboxylic acid was also produced.

Table 2. Effect of type and amount of oxidant on oxidation of benzyl alcohol catalyzed by $[MoO_2L(H_2O)]$ complex[a]

Entry	Oxidant	Amount (mmol)	Yield (%)[b]	TOF (h^{-1})
1	No oxidant	0	0	-
2	$NaIO_4$	2	Trace	-
3	H_2O_2	2	82	68.3
4	$(Bu)_4NIO_4$	2	Trace	-
5	*tert*-BuOOH	2	55	45.8
6	**UHP**	**2**	**92**	**76.7**
7	UHP	1	48	40
8	UHP	1.5	67	55.8
9	UHP	2.5	93	77.5

[a] Reaction conditions: benzyl alcohol (1 mmol), oxidant, catalyst (0.006 mmol), CH_3CN (5 mL), reflux conditions, 2 h. [b] Isolated yield.

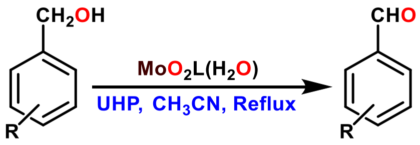
To obtain the optimal amount of the catalyst, different amounts of the catalyst were used on the oxidation reaction of benzyl alcohol using UHP in refluxed CH_3CN and it was observed that the subjected oxidation process was completed in the presence of 0.006 mmol catalyst. Using higher amounts of the catalyst did not have a substantial effect on the product yield and reaction time, and the reaction did not proceed much in the absence of the catalyst (Table 3).

Table 3. Optimization of catalyst amount on oxidation of benzyl alcohol catalyzed by $[MoO_2L(H_2O)]$ complex[a]

Entry	Catalyst amount (mmol)	Yield (%)[b]	TOF (h^{-1})
1	No catalyst	0	-
2	0.002	35	87.5
3	0.004	62	77.5
4	**0.006**	**92**	**76.7**
5	0.008	93	58.1

[a] Reaction conditions: benzyl alcohol (1 mmol), UHP (2 mmol), catalyst, CH_3CN (5 mL), reflux conditions, 2 h. [b] Isolated yield.

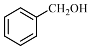
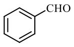
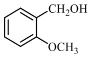
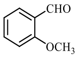
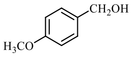
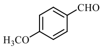
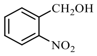
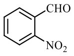
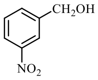
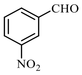
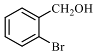
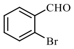
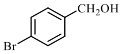
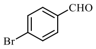
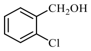
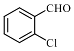
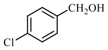
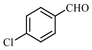
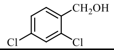
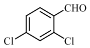
The versatility of this catalytic method was evaluated for the oxidation of a variety of benzylic alcohols using $[MoO_2L(H_2O)]$ complex as an efficient homogeneous catalyst under the optimal conditions (Scheme 2, Table 4). Various benzylic alcohols having electron-withdrawing and -donating substituents are treated with UHP to produce the desirable substituted aryl aldehydes in high isolated yields.



Scheme 2. Oxidation of benzylic alcohols using UHP in the presence of $[MoO_2L(H_2O)]$ complex

This procedure has remarkable chemoselectivity and the benzylic hydroxyl groups were oxidized in this catalytic system to the corresponding benzaldehydes in 100 % selectivity. One of the most significant advantages of the present procedure is that there is no evidence of overoxidation to the formation of carboxylic acid in all the studied substrates.

Table 4. Selective oxidation of benzylic alcohols to aryl aldehydes using UHP catalyzed by $[MoO_2L(H_2O)]$ complex[a]

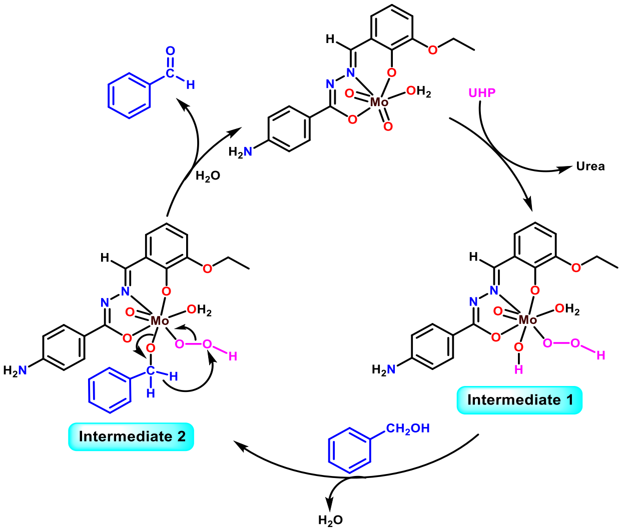
Entry	Substrate	Product[b]	Yield (%)[c]	TOF (h^{-1})
1			92	76.7
2			94	78.3
3			91	75.8
4			89	74.2
5			87	72.5
6			90	75
7			92	76.7
8			91	75.8
9			92	76.7
10			88	73.3

[a]Reaction conditions: benzylic alcohol (1 mmol), UHP (2 mmol), catalyst (0.006 mmol), CH_3CN (5 mL), reflux conditions, 2 h. [b]All products were identified by comparison of their physical and spectral data with those of authentic samples. [c]Isolated Yield.

A suggested mechanism for the oxidation of benzyl alcohol using UHP in the presence of $[MoO_2L(H_2O)]$ is depicted in Scheme 3. First, the molybdenum center of the complex is attacked by hydrogen peroxide released from UHP and produces intermediate **1**. This peroxomolybdenium intermediate after reaction with benzyl alcohol affords the intermediate **2**. In the end, by the breaking of the peroxide bond, one molecule of water is released and the Schiff base complex is regenerated to produce benzaldehyde as the desired product.

4. CONCLUSIONS

In conclusion, we have prepared an ONO-donor tridentate Schiff base ligand and its dioxomolybdenum(VI) complex. The prepared compounds were then characterized by elemental analysis, FT-IR, ^1H and ^{13}C NMR techniques. The catalytic activity of the complex was also studied in the selective oxidation of benzylic alcohols to the corresponding aryl aldehydes using UHP in acetonitrile under reflux conditions. The present procedure has many advantages, including short reaction time, high yield and excellent selectivity to produce desirable aryl aldehydes with no overoxidation to carboxylic acids.



Scheme 3. Suggested mechanism for the oxidation of benzyl alcohol with UHP catalyzed by $[MoO_2L(H_2O)]$ complex.

CONFLICTS OF INTEREST

There are no conflicts of interest to declare.

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