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Synthesis and Characterization of Glucose Derived Dioxo-molybdenum (VI) Complexes and Their Application in Sulphide Oxidation

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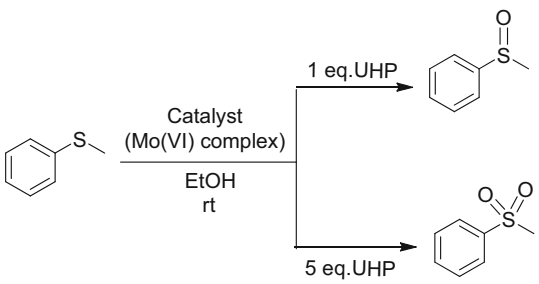
Abstract Three new molybdenum (VI) complexes of 4,6-*O*-ethylidene-*β*-D-glucopyranosylamine derived ligands has been synthesized and the same has been used in the oxidation of thioanisole along with an earlier reported analogous complex. A selective oxidation of thioanisole to methyl phenyl sulphoxide in high yield is achieved using 1:1 mixture of thioanisole and urea hydrogen peroxide (UHP) in ethanol. A longer reaction time or excess of UHP, leads to the formation of corresponding sulphone, which was confirmed using HPLC and NMR measurements.

Graphical Abstract The oxidation of thioanisole into corresponding sulphoxide and sulphone has been explored using dioxo-molybdenum (VI) complexes of 4,6-*O*-ethylidene-*β*-D-glucopyranosylamine derived Schiff base ligands.



Keywords Glucopyranosylamine · Molybdenum · Urea hydrogen peroxide · Sulphoxide · Sulphone

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1 Introduction

Biochemistry is rich with the chemistry of lighter elements (at. no. <35) [1, 2], with few exceptions and especially that for group 6 elements of the periodic table. The lightest element of this group (Cr) is mainly regarded for its toxicity inside the biological system, while tungsten (W) is found generally in thermophilic organisms [3]. Mo controls several biochemical reactions in the form of nitrogenase, nitrate reductase, DMSO reductase, xanthine oxidase etc., [4, 5]. The catalytic reactions of Mo in various molybdoenzymes inspires the inorganic chemists to explore the catalytic reactions using the complexes containing this metal ion due to its adaptable nature and accessible stable oxidation states [6]. Molybdenum complexes have been used in industrial ammoxidation of olefins [7], olefin epoxidation [8] as well as olefin metathesis [9].

Structurally characterized sugar containing molybdenum complexes are known, since more than a decade, however its use has only been explored by Zhao et al. in the epoxidation of cyclooctene and *cis*-, *trans*-*β*-methylstyrene [10]. Molybdenum complexes are well known for oxo transfer reactions like isomerization of allyl alcohols, epoxidation, phosphine oxidation, sulfide oxidation, oxidative bromination etc. [11–14], however utility of sugar containing Mo complexes are scarce. Lack of utility of sugar derived molybdenum complexes prompted us to perform the catalytic reaction using these complexes and along this line we have explored the sulfide oxidation reactions using four glucose derived molybdenum complexes. Synthesis of two ligands [15, 16] and one molybdenum complex [17] along with their crystal structures have already been reported and here we are presenting the synthesis and characterization of new two ligands (Scheme 1; H_3L3, H_3L4) and molybdenum complexes of

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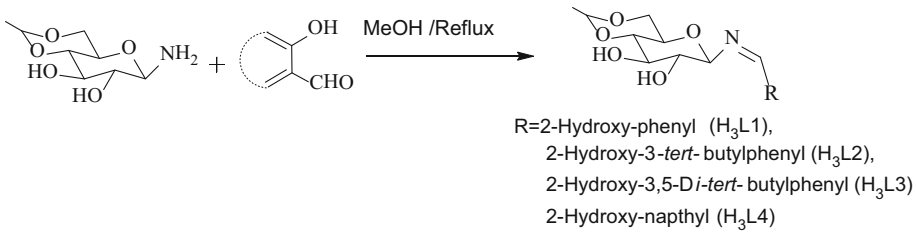
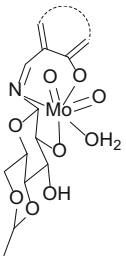
Scheme 1 Synthetic route for ligands





Ligand	Complex No.
H_3L1	1
H_3L2	2
H_3L3	3
H_3L4	4

Fig. 1 Molybdenum complexes of H_3Ln

H_3Ln ($n = 2$–4; Fig. 1). The sulfide oxidation reaction has been optimized using 4,6-*O*-ethylidene-*N*-(2-hydroxybenzylidene)-β-D-glucopyranosylamine derived molybdenum (VI) [Mo (VI)] complex and reactions of rest three molybdenum complexes were performed under optimized condition. We have achieved an isolated yield of 89 % methyl phenyl sulfoxide using (1:1) urea hydrogen peroxide (UHP) in 15 min. Longer reaction time and excess of UHP concentration leads to the conversion of sulphoxide to sulphone. Formation of both sulphoxide and sulphone have been confirmed by HPLC and NMR spectroscopy.

In conclusion, this paper deals with the synthesis and characterization of glucose derived ligands, their Mo (VI) complexes and application of latter in sulfide oxidation reactions.

2 Experimental

2.1 General

All experiments were performed under normal atmospheric conditions and room temperature. 2-Hydroxy-1-napthaldehyde and UHP were purchased from Sigma-Aldrich India, 3,5-di-*tert*-butyl-2-hydroxy benzaldehyde and Mo (VI) oxide bis(2,4-pentanedionate) [$MoO_2(acac)_2$] were purchased from Alfa Aeser. The compounds H_3L1 and H_3L2 were synthesized following the reported literature procedure [15, 16]. The rest of the chemicals along with solvents were procured from local suppliers and solvents were purified and dried following the standard methods before use.

^1H and ^{13}C NMR were recorded in DMSO-d_6 on a Bruker Avance spectrometer and data was processed using MestReNova software. The solution electronic spectra were recorded on Shimadzu UV-260 spectrophotometer and IR spectra on ABB Bomen MB 3000 FTIR machine using KBr Matrix. HRMS of organic samples and ESI-MS of complexes were recorded on Thermoscientific Q Exactive and Hewlett-Packard' HP GS/MS 5890/5972 mass spectrometer respectively.

2.2 Preparation of *N*-(3,5-di-*tert*-butyl-2-hydroxybenzilidene)-4,6-*O*-ethylidene-β-D-glucopyranosylamine (H_3L3)

This compound was synthesiszed adapting the procedure reported for H_3L2 [16], but using 4,6-*O*-ethylidene-β-D-glucopyranosylamine (2.00 g, 9.75 mmol) in ethanol (20 mL), and 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde (2.510 g, 10.7 mmol). Yield: 3.301 g (80 %); yellow solid; mp 106–108 °C; IR (KBr; cm^{-1}): 3433, 2955,1628, 1103. UV–Vis [λ_{max}; nm (ε; L cm^{-1} mol^{-1}) in DMSO]: 262 (320000), 333 (100000). ^1H NMR (DMSO-d_6 400 MHz, ppm): δ 13.64 (1H, s, Ar–OH), 8.59 (1H, s, HC=N), 7.35 (2H, s, ArH), 5.56 (1H, d, $J = 6.0$ Hz, glucose–OH), 5.37 (1H, d, $J = 5.6$ Hz, glucose–OH), 4.77 (1H, q, $J = 4.8$ Hz, ethylidene CH), 4.54 (1H, d, $J = 8.4$ Hz, glucose H-1), 4.06 (1H, m, glucose H-5), 3.57–3.40 (3H, m, glucose H-3, H-4, H-6a), 3.28 (1H, m, glucose H-6b), 3.13 (1H, m, glucose H-2), 1.39 (9H, s, ^{t}Bu),1.27 (12H, m, ^{t}Bu and ethylidene CH_3); ^{13}C NMR (101 MHz, DMSO-d_6, ppm): δ 167.22 (C=N), 157.92, 140.35, 136.10, 127.64, 127.32, 117.84, 99.09, 95.75, 80.60, 75.22, 73.73, 68.31, 67.80, 35.03, 34.33, 31.74, 29.70, 20.79; HRMS: *m/z* calcd for $(M + H)^+$ $C_{23}H_{36}NO_6$ 422.2543; found 422.2754.

2.3 Preparation of *N*-(2-hydroxynapthylidene)-4,6-*O*-ethylidene-β-D-glucopyranosylamine (H_3L4)

This compound was synthesized following the procedure reported for H_3L1 [15], but using 4,6-*O*-ethylidene-β-D-glucopyranosylamine (0.102 g, 0.5 mmol) and 2-hydroxy-1-napthaldehyde (0.094 g, 0.55 mmol) in ethanol (3 mL). Yield: 0.153 g (87 %), fluoresent yellow solid; mp: chared at 228–230 °C; IR (KBr; cm^{-1}): 3433, 3209,1643, 1095;

UV–Vis [λ_{max}; nm (ε; $Lcm^{-1}mol^{-1}$) in DMSO]: 304 (6080), 365 (2740), 403 (3820), 422 (3700). ^{1}H NMR (DMSO-d_6 400 MHz, ppm): δ 14.30 (1H, m, Ar–OH), 9.25 (1H, d, $J = 7.2$ Hz, HC=N), 8.13 (1H, d, $J = 8.8$ Hz, Ar–H), 7.85 (1H, d, $J = 9.6$ Hz, Ar–H), 7.73 (1H, d, $J = 8.0$ Hz, Ar–H), 7.50 (1H, m, Ar–H), 7.29 (1H, t, $J = 7.2$ Hz, Ar–H), 6.86 (1H, d, $J = 9.2$ Hz, Ar–H), 5.76 (1H, d, $J = 6.0$ Hz, glucose–OH), 5.47 (1H, d, $J = 5.2$ Hz, glucose–OH), 4.78 (2H, m, ethylidene CH and glucose H-1), 4.08 (1H, m, glucose H-5), 3.57–3.39 (3H, m, glucose H-3, H-4, H-6a), 3.32–3.22 (2H, m, glucose H-2, H-6b), 1.25 (3H, d, $J = 5.2$ Hz, ethylidene CH_3); ^{13}C NMR (101 MHz, DMSO-d_6, ppm): δ 173.88 (C=N), 159.77, 137.73, 134.04, 129.48, 128.66, 126.55, 123.87, 123.54, 119.54, 107.22, 99.11, 92.49, 80.32, 74.90, 73.69, 68.72, 67.64, 20.76; HRMS: *m/z* calcd for $(M + H)^+$ $C_{19}H_{22}NO_6$ 360.1447; found 360.1593.

2.4 Preparation of MoO_2(HL2) (**2**)

To a methanolic solution (5 mL) of H_3L2 (0.292 g, 0.8 mmol), $MoO_2(acac)_2$ (0.195 g, 0.6 mmol) was added and the reaction mixture was stirred at room temperature for 13 h to result in clear yellow solution. The reaction mixture was concentrated, residue was dissolved in diethyl ether (3 mL) and excess hexane was added to that while stirring to result in light yellow solid product, which was filtered and dried under vacuum.Yield: 0.234 g (76 %); yellow solid; mp >250 °C; IR (KBr; cm^{-1}): 3649, 3433, 3163, 2962, 1643, 1011, 910. UV–Vis [λ_{max}; nm (ε; $L\ cm^{-1}\ mol^{-1}$) in DMSO]: 276 (shoulder), 354.5 (355000). ^{1}H NMR (DMSO-d_6 400 MHz, ppm): δ 8.51 (1H, d, $J = 2.4$ Hz, HC=N), 7.60 (1H, d, $J = 8.8$ Hz, ArH), 7.48 (1H, dd, $J = 7.6$ Hz, 1.6 Hz, ArH), 6.91 (1H, t, $J = 7.6$ Hz, ArH), 5.62 (1H, d, $J = 5.6$ Hz, glucose–OH), 4.78–4.73 (2H, m, glucose H-1, ethylidene CH), 4.19 (1H, m, glucose H-5), 3.77–3.67 (3H, m, glucose, H-3, H-4, H-6a), 3.54 (1H, m, glucose H-6b), 3.34 (1H, glucose H-2), 1.36 (9H, s, ^{t}Bu), 1.27 (3H, d, $J = 4.8$ Hz, ethylidene CH_3); ^{13}C NMR (101 MHz, DMSO-d_6, ppm): δ 161.24 (C=N), 159.81, 139.25, 133.34, 132.49, 121.52, 119.74, 99.41, 91.32, 85.37, 81.22, 73.59, 70.03, 67.76, 35.24, 29.98, 20.71; ESI-MS: *m/z* calcd for $(M + H)^+$ $C_{19}H_{27}MoNO_9$ 510.3; found 510.9.

2.5 Preparation of MoO_2(HL3) (**3**)

This compound was prepared following the procedure adopted for complex **2**, but using (H_3L3) (0.198 g, 0.47 mmol) and $MoO_2(acac)_2$ (0.150 g, 0.46 mmol). Yield: 0.194 g (74 %); yellow solid; mp 223–225 °C; IR (KBr; cm^{-1}): 3379, 2955, 1643, 1103, 903. UV–Vis [λ_{max}; nm (ε; $Lcm^{-1}mol^{-1}$) in DMSO]: 282 (879000), 359 (198000). ^{1}H NMR (DMSO-d_6 400 MHz, ppm): δ 8.52 (1H, d, $J = 2.4$ Hz, HC=N), 7.59 (1H, d, $J = 2.8$ Hz, ArH), 7.47 (1H, d, $J = 2.4$, ArH), 5.56 (1H, d, $J = 5.6$ Hz, glucose–OH), 4.76 (1H, q, $J = 5.1$ Hz, ethylidene CH), 4.67 (1H, dd, $J = 8.8$ Hz, 2.2 Hz, glucose H-1), 4.17 (1H, m, glucose H-5), 3.70–3.62 (3H, m, glucose H-3, H-4, H-6a), 3.49 (1H, m, glucose H-6b), 3.31 (1H, glucose H-2), 1.35 (9H, s, ^{t}Bu),1.26 (12H, m, ^{t}Bu and ethylidene CH_3); ^{13}C NMR (101 MHz, DMSO-d_6, ppm): δ 160.24 (C=N), 159.22, 141.59, 138.31, 129.69, 129.59, 121.00, 99.42, 91.30, 85.30, 81.27, 77.29, 73.61, 70.00, 67.78, 35.39, 34.48, 31.66, 30.04, 20.70; ESI-MS: *m/z* calcd for $(M + H)^+$ $C_{23}H_{35}MoNO_9$ 566.4; found 566.9.

2.6 Preparation of MoO_2(HL4) (**4**)

This compound was prepared following the procedure adopted for complex **1** [17] but using H_3L4 (0.143 g, 0.4 mmol) and $MoO_2(acac)_2$ (0.097 g, 0.3 mmol) Yield: 0.120 g (80 %), flouresent yellow solid; mp:> 250 °C; IR (KBr; cm^{-1}): 3433, 1628, 1142, 1103, 895. UV–Vis [λ_{max}; nm (ε; $L\ cm^{-1}\ mol^{-1}$) in DMSO]: 313 (1349000), 380 (525000). ^{1}H NMR (DMSO-d_6 400 MHz, ppm): δ 9.30 (1H, d, $J = 2.0$ Hz, HC=N), 8.15 (1H, d, $J = 8.4$, ArH), 8.06 (1H, d, 9.2 Hz, ArH), 7.90 (1H, d, $J = 7.6$ Hz, ArH), 7.62 (1H, m, ArH), 7.43 (1H, t, $J = 7.2$ Hz, ArH), 7.16 (1H, d, $J = 8.8$ Hz, ArH), 5.62 (1H, d, $J = 5.6$ Hz, glucose–OH), 4.77 (2H, m, glucose H-1 and ethylidene CH), 4.27 (1H, m, glucose H-5), 3.82–3.69 (3H, m, glucose H-3, H-4, H-6a), 3.59 (1H, m, glucose H-6b), 3.37 (1H, glucose H-2), 1.26 (3H, d, $J = 4.8$ Hz, ethylidene CH_3); ^{13}C NMR (101 MHz, DMSO-d_6, ppm): δ 163.20 (C=N), 154.04, 136.56, 133.23, 129.47, 128.96, 128.32, 124.54, 121.99, 120.58, 111.93, 99.41, 91.48, 85.53, 81.27, 73.51, 70.02, 67.79, 20.70.

2.7 General Procedure for Selective Oxidation of Thioanisole to Methyl Phenyl Sulphoxide

To a mixture of molybdenum complexes (1–4; 0.05 mmol) and thioanisole (1 mmol) in 3 mL of ethanol, UHP (1 mmol) was added and the reaction mixture was stirred at room temperature. The progress of reaction was monitored by TLC as well as HPLC. The reaction mixture was concentrated under reduced pressure and the pure product was isolated by column chromatography using silica gel as solid support and a mixed solvent (n-hexane/ethyl acetate 70:30) as eluent.

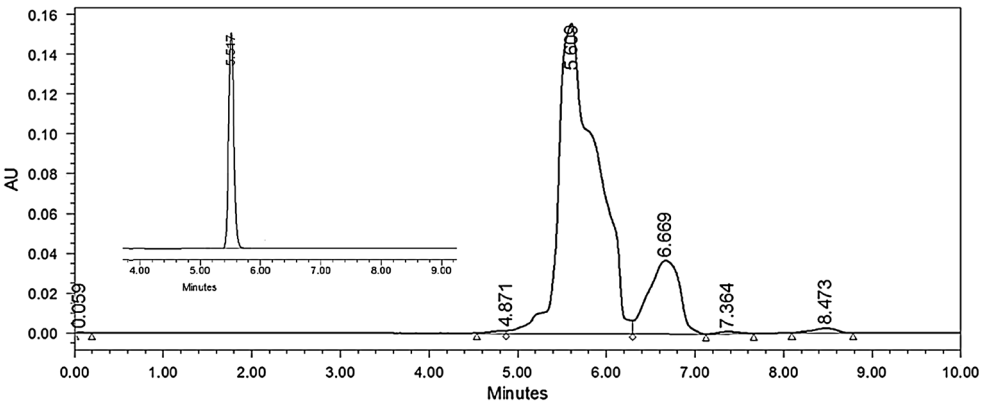


Fig. 2 HPLC spectrum of the reaction mixture set for thioanisole oxidation; *inset* represents the spectrum of isolated pure methyl phenyl sulphoxide after column chromatography

Table 1 Summary of control reaction for the oxidation of thioanisole

Entry	Catalyst 1 (mmol)	Substrate (mmol)	UHP (mmol)	Yield of sulphoxide[a] (%)	Yield of sulphone[a] (%)
1	0	1	1	0	0
2	0.05	1	0	0	0
3	0.05	1	1	89	0
4	0.05	1	2	20	50
5	0.05	1	5	0	94

Refer Fig. 1 for catalyst

[a] Isolated yield after 15 min of reaction time

Table 2 Effect of solvent on oxidation of thioanisole to methyl phenyl sulphoxide

Entry	Solvent	Time	Isolated yield (%)
1	Ethanol	15 min	89
2	Methanol	15 min	85
3	Acetonitrile	15 min	72
4	Toluene	2 h	33
5	THF	2 h	66
6	DCM	2 h	52

3 Results and Discussion

3.1 Synthesis and Characterization of Ligands and Complexes

Synthesis of H_3L3 and H_3L4 were carried out by condensing 4,6-*O*-ethylidene-*β*-D-glucopyranosylamine with 3,5-di-*tert*-butyl-2-hydroxybenzaldehyde and 2-hydroxy-1-napthaldehyde respectively. The product formation was confirmed by FTIR, UV–Visible, NMR and mass spectroscopy. FTIR spectra of H_3L3 and H_3L4 exhibited strong characteristic bands for ν_{C-O} and ν_{O-H} in the range of 1157–1011, and 3433–3209 cm^{-1} respectively. $\nu_{C=N}$ stretch for H_3L3 appeared at 1628 and that for H_3L4 at 1643 cm^{-1}. HRMS signals at *m/z* 422.2754 and 360.1593 corresponds to $(M + H)^+$ ion peaks of H_3L3 and H_3L4 respectively. ^1H and ^{13}C NMR spectrum recorded in DMSo-d_6, mentioned in the experimental section, clearly confirmed the formation of pure products H_3L3 and H_3L4. Both the ligands exhibited a coupling constant for saccharide C1–H, $^3J_{H-H} = 8.3$ Hz, supporting the presence of β-anomeric form of the sugar moiety.

Table 3 Results of methyl phenyl sulphoxide formation by different catalysts under optimized reaction conditions

Entry	Catalyst	Solvent	Time (min)	Isolated yield (%)
1	**1**	Ethanol	15	89
2	**2**	Ethanol	15	83
3	**3**	Ethanol	15	80
4	**4**	Ethanol	15	75

The catalyst number has been represented in bold to make a difference with that of the entry serial number

After confirming the ligands formation, H_3L1, H_3L2, H_3L3, and H_3L4 were treated with $MoO_2(acac)_2$ in methanol to afford the corresponding sugar bound Mo (VI) complexes. FTIR studies of all the complexes exhibited characteristic vibration bands in the range of 910–895 cm^{-1}, corresponding to $\nu_{Mo=O}$. No appreciable changes were observed for ν_{C-O} and $\nu_{C=N}$ stretching frequencies of complexes with respect to the corresponding free ligands, but a change in the ν_{O-H} region of spectra was observed. Sharp bands in the OH region of the ligands converted into broad ones after metalation reaction, indicating the increment in hydrogen bonding interactions. ^1H NMR spectra of all the molybdenum complexes exhibit the loss of phenolic, and C2-hydroxyl proton of the glucose moiety revealing the binding of metal ion with the ligand via deprotonation. In all the complexes, signal of glucose C3–OH shifted down field with respect to the free ligands. Complexation of ligands (H_3L2, H_3L3, H_3L4) with Mo (VI) also influenced the chemical shifts of the saccharide skeletal protons and all such five protons underwent downfield shift by about 0.2 ppm. Even in the molybdenum

complexes, the β-anomeric form of the ligands were retained as $^{3}J_{H-H}$ were found to be 8.9.

3.2 Selective Oxidation of Sulfides to Sulphoxide and Suphones

The structure of molybdenum complexes containing glycosylamine derived Schiff bases has been already established [10, 17], however their applications are yet to be explored. Only one report is available on catalytic epoxidation reactions [10] by *cis*-dioxo Mo (VI) complexes of sugar derived chiral Schiff base ligands. To the best of our knowledge there is no report on oxidation of sulfides to sulphoxides or sulphones using similar saccharide derived molybdenum complexes. Lack of such studies prompted us to explore the sulfide oxidation reaction using sugar derived molybdenum complexes. Along this line, firstly we optimized the conditions for the oxidation of thioanisole to methyl phenyl sulphoxide and methyl phenyl sulphone using UHP and complex 1. Ethanolic solution of complex (1), thioanisole and UHP in 0.05:1:1 ratio was stirred at room temperature and progress of reaction was monitored by TLC as well as HPLC. Building of peaks at 5.5 min in the HPLC spectrum supported the formation of methyl phenyl suphoxide at the cost of decrement of peak at 6.6 min, which corresponds to thioanisole (Fig. 2). The pure methyl phenyl suphoxide was isolated by column chromatography after 15 min of reaction time and it's purity was judged by HPLC (inset, Fig. 2) and ^{1}H NMR (refer supplementary data). Several control reactions were performed to learn the effects of amounts of catalyst and UHP on the product formation and the same is summarized in Table 1. No oxidation was noticed in absence of either catalyst or UHP and the same was also noticed by Sheikhshoaie et al. [18]. Increase in the reaction time or UHP concentration lead to over oxidation, forming the sulphone. Selective sulphone formation was observed when substrate to UHP concentration was taken in 1:5 ratio.

After confirming the oxidation of thioanisole in ethanol, several other solvent were tried and the results are summarized in Table 2. The maximum conversion of thioanisole into methyl phenyl sulphoxide was observed in ethanol, while minimum was in the toluene. Reaction proceeds with lower rate and yield in less polar solvents, while it is quicker and high yielding in alcohols.

The oxidation of thioanisole with rest of the complexes (2–4) was performed under optimized condition and the results are summarized in Table 3. In all the cases, reaction time was limited to 15 min to avoid the sulphone formation. Our results are comparable with the report where Sheikhshoaie et al. [18] have performed similar reaction using molybdenum complexes bearing ligand with ONO chelating site. The advantage of our method is that we got better yield in shorter time (15 min in compared to 30) using similar catalytic loading. Moreover, we have performed the reaction using glucose derived complexes, which open the door of chiral synthesis.

4 Conclusion

We have synthesized a series of 4,6-*O*-ethylidene-*β*-D-glucopyranosylamine derived ligands, their Mo (VI) complexes and application of the latter in the oxidation of thioanisole. This is the first report, where sugar derived Mo (VI) complexes has been used in sulfide oxidation reaction. Various aspects of the reaction like role of solvent, reaction time, necessities of catalyst and UHP has been explored. These preliminary success is getting extrapolated in the chiral synthesis as our metal complex will act as chiral catalyst due to its sugar origin.

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