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Journal of Coordination Chemistry
Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gcoo20

Selective oxidation of sulfides and olefins by a manganese(III) complex containing an N,O-type bidentate oxazine ligand

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Published online: 29 Jan 2013.

To cite this article: Mojtaba Amini, Mojtaba Bagherzadeh, Zeinab Moradi-Shoeili, Davar M. Boghaei, Arkady Ellern & L. Keith Woo (2013) Selective oxidation of sulfides and olefins by a manganese(III) complex containing an N,O-type bidentate oxazine ligand, Journal of Coordination Chemistry, 66:3, 464-472, DOI: 10.1080/00958972.2012.761339

To link to this article: http://dx.doi.org/10.1080/00958972.2012.761339

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Journal of Coordination Chemistry
Vol. 66, No. 3, 10 February 2013, 464–472

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Selective oxidation of sulfides and olefins by a manganese (III) complex containing an N,O-type bidentate oxazine ligand

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(*Received 15 October 2012; in final form 30 November 2012*)

A new manganese(III) complex [(N–O)$_2$Mn(OAc)] was synthesized using 2-(2′-hydroxyphenyl)-5,6-dihydro-1,3-oxazine (N–O) as a bidentate O, N donor. The complex has been characterized by elemental analysis, IR, UV–vis spectroscopy, and X-ray structure analysis. Oxidation of sulfides and epoxidation of olefins, respectively, to their corresponding sulfoxides and epoxides were conducted by this catalyst using urea hydrogen peroxide as oxidant at room temperature under air. The catalyst is efficient in oxidation reactions giving high yields and selectivities.

Keywords: Manganese(III) complex; Oxazine; Crystal structure; Olefin epoxidation; Sulfide oxidation

1. Introduction

Transition metal complex catalyzed oxidation of olefins and sulfides is of increasing significance in synthetic chemistry offering effective synthesis of important compounds that have industrial and medical applications [1–4]. Manganese complexes by virtue of their range of stable and chemically accessible oxidation states have been subject of much research as oxygen atom transfer reactions [5]; many manganese complexes oxidize hydrocarbons [6, 7].

Oxazoline-based and oxazine-based ligands are easily synthesized and form complexes with almost all metal ions [8, 9]. Many complexes with these ligands show excellent catalytic activity in various reactions and there have been many reports on applications in homogeneous and heterogeneous catalysis [10–12]. The presence of N and O donor sites in oxazolines and oxazines may modify significantly the steric and electronic properties of the ligand. We reported highly efficient methods for oxidation of olefins and sulfides by using two manganese(III)–oxazoline complexes [Mn(phox)$_2$(CH$_3$OH)$_2$]ClO$_4$ and Mn (phox)$_3$ (Hphox = 2-(2′-hydroxylphenyl)oxazoline) [11, 12]. There are no reports of manganese complexes with oxazine-based ligands as catalysts for oxidation.

Herein, we describe the synthesis of a new Mn(III) complex [(N–O)$_2$Mn(OAc)] ((N–O) =2-(2′-hydroxyphenyl)-5,6-dihydro-1,3-oxazine) and use of this complex as a catalyst in

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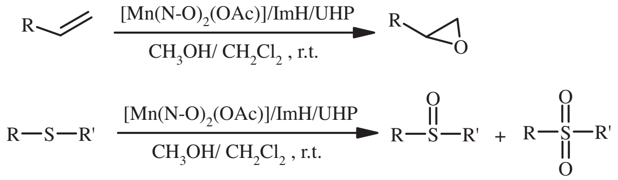
Journal of Coordination Chemistry
ISSN 0095-8972 print/ISSN 1029-0389 online
http://dx.doi.org/10.1080/00958972.2012.761339

epoxidation of olefins and oxidation of sulfides in the presence of urea hydrogen peroxide (UHP) as oxidant under air at room temperature (scheme 1). This is the first report on the catalytic activity of an oxazine–Mn(III) complex in oxidation reactions.



Scheme 1. Epoxidation of olefins and oxidation of sulfides in the presence of $[Mn(N–O)_2(OAc)]$.

2. Experiment

Chemicals and solvents were purchased from Fluka and Merck Chemical companies. 2-(2′-Hydroxyphenyl)-5,6-dihydro-1,3-oxazine (N–O) was synthesized according to a published procedure [8].

Carbon, hydrogen, and nitrogen analyses 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. UV–vis spectra were recorded by a CARY 100 Bio VARIAN UV–vis spectrophotometer in methanol. Products of oxidation reactions were determined and analyzed by an HP Agilent 6890 gas chromatograph equipped with a HP-5 capillary column (phenyl methyl siloxane 30 m · 320 lm · 0.25 lm) and a flame-ionization detector.

2.1. *Synthesis of $[(N–O)_2Mn(OAc)]$*

To a solution of $Mn(OAc)_2 \cdot 4H_2O$ (0.173 g, 1.00 mmol) in 15 mL of methanol was added a solution of 2-(2′-hydroxyphenyl)-5,6-dihydro-1,3-oxazine (N–O), (0.355 g, 2.00 mmol) in 10 mL of methanol. After 2 h stirring at room temperature, the solution was filtered and evaporated under reduced pressure to give the complex. Recrystallization from acetonitrile yielded the complex as brown crystals. Yield: 0.253 g, 48%. Anal. Calcd for $C_{22}H_{23}MnN_2O_6$: C, 56.66; H, 4.97; N, 6.00. Found: C, 56.51; H, 4.83; N, 6.11. Selected IR frequency (KBr disk, cm^{-1}): 1608 ($\nu_{C=N}$). Electronic absorption spectrum in MeOH λ_{max} (nm) (ε ($M^{-1}\,cm^{-1}$)): 298 (1.8×10^4), 243 (2.2×10^4).

2.2. *General procedure for epoxidation of olefins*

To a solution of olefin (0.2 mmol), imidazole(ImH) (0.2 mmol), chlorobenzene (0.2 mmol) as internal standard, and $[(N–O)_2Mn(OAc)]$ (0.01 mmol) in (1 : 1) mixture of CH_3OH/CH_2Cl_2 (1 mL) was added 0.6 mmol UHP as oxidant. The mixture was stirred at room temperature and the reaction progress monitored by gas chromatography (GC). Assignments of products were made by comparison with authentic samples.

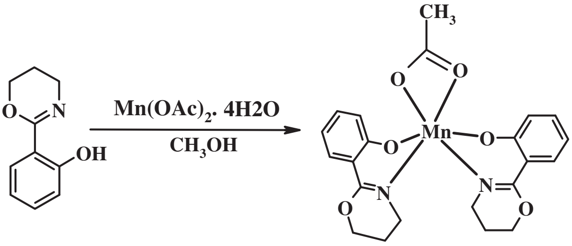
2.3. *General procedure for sulfide oxidation*

To a solution of sulfide (0.2 mmol), imidazole(ImH) (0.2 mmol) as axial ligand, chlorobenzene (0.2 mmol) as internal standard, and $[(N–O)_2Mn(OAc)]$ (0.01 mmol) in a 1 : 1 mixture of CH_3OH/CH_2Cl_2 (1 mL) was added 0.4 mmol UHP as oxidant. The mixture was stirred at room temperature and the reaction progress monitored by GC. Assignments of products were made by comparison with authentic samples.

3. Results and discussion

3.1. *Complex characterization*

The complex was obtained by reaction of 2 equiv. of ligand (N–O) and $Mn(OAc)_2{\cdot}4H_2O$ (scheme 2). The fast color change from a colorless to a green solution during the synthesis indicated that the manganese ion was oxidized by air in the presence of ligand [10].



Scheme 2. Synthesis of $[(N–O)_2Mn(OAc)]$.

Elemental analyses and spectral data confirm the assigned composition of the complex $[(N–O)_2Mn(OAc)]$. IR spectra of the complex clearly showed coordination of the ligands, as exemplified by the shift of the C=N absorption [13]. The complex has a similar absorption pattern in the UV–vis spectra, showing ligand-to-metal charge transfer (LMCT) and $\pi \rightarrow \pi^*$ transition with absorption maxima around 380 and 253 nm [10, 13].

The structure of complex was determined by X-ray crystallography and an ORTEP view of the complex is shown in figure 1. Single crystals of the complex were obtained with slow evaporation from Me–CN solution. Single-crystal X-ray structure refinement data are shown in table 1. The Mn(III) complex crystallizes in the monoclinic crystal system, space group $P2_1/c$, and consists of a Jahn–Teller distorted octahedral MnN_2O_4 chromophore. The manganese is ligated by four donors [N(1), N(2), O(3) and O(4)] from two chelating ligands (N–O) and two oxygens [O(1) and O(2)] of one acetate. The structure of the complex is mononuclear, consisting of discrete monomeric units of $[(N–O)_2Mn(OAc)]$, in which acetate is equatorial, with bond distances of 2.2834(16) and 2.0939(14) Å, respectively, for the $Mn_1–O_1$ and $Mn_1–O_2$ bonds, while two donors of each oxazine occupy one equatorial and one axial position. The rather short $Mn_1–O_3$ and $Mn_1–O_4$ distances of 1.864(11) and 1.868(11) Å compared to the equatorial coordination distances indicate compressed Jahn–Teller effect. Axial compressed distortion in this complex is very unusual, since most other Mn(III)-containing oxazoline moieties exhibit axial elongation [10, 13].

3.2. *Catalytic reactivity*

To compare catalytic reactivity of $[(N–O)_2Mn(OAc)]$ with $[Mn(phox)_2(CH_3OH)_2]ClO_4$ and $Mn(phox)_3$, we used the same optimization conditions from our previous work [11, 12].

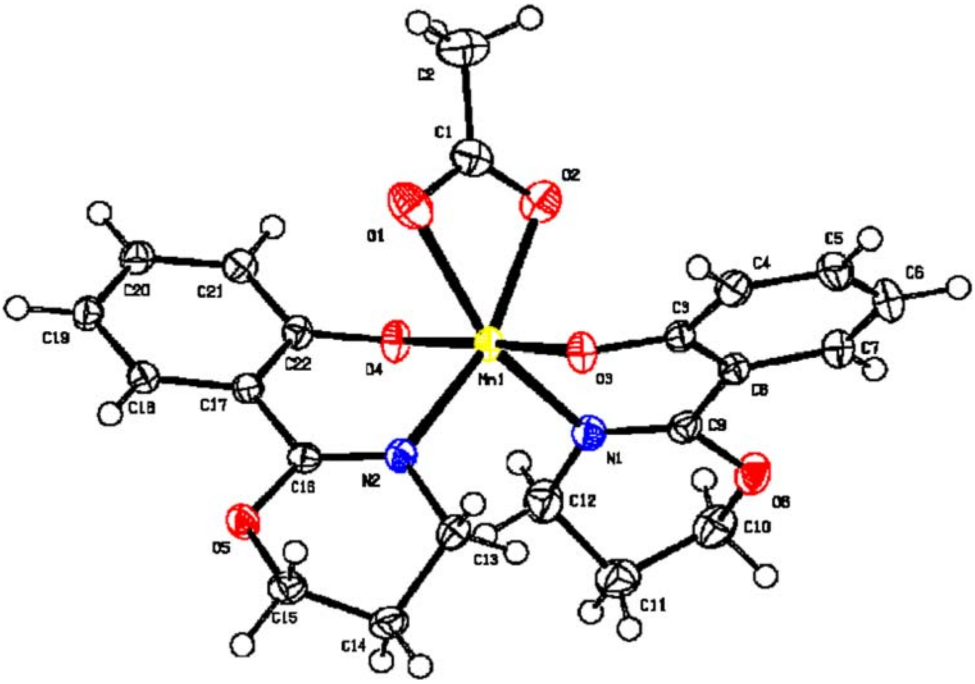


Figure 1. ORTEP diagram of [(N–O)$_2$Mn(OAc)] with thermal ellipsoids drawn at the 50% probability level.

Oxidation of various sulfides was investigated and the details of catalytic activity with respect to oxidation of sulfides are recorded in table 2. When oxidation of methylphenylsulfide was performed in the absence of catalyst, but with imidazole, no reaction occurred, whereas the [(N–O)$_2$Mn(OAc)]/ImH/UHP oxidizing system oxidized a wide variety of sulfides at room temperature and in short reaction times (5 min). As shown in table 2, sulfides were effectively oxidized to the corresponding sulfoxides as major products. The highest and the lowest yields were obtained for methylphenylsulfide and diphenylsulfide, respectively (table 2, entries 1 and 4). Benzylic sulfides oxidized to the corresponding sulfoxides without affecting the benzylic C–H bond (table 2, entries 3 and 4). Replacement of one or both alkyl groups in dialkylsulfide with phenyl or benzyl to probe electronic effects displayed no regular trends in the yields; the results in table 2 show that aromatic sulfides are slightly more reactive than alkyl and aryl sulfides. Minor amounts of byproducts resulting from overoxidation to sulfone were found (table 2, entries 1–8). As shown in table 2, catalytic oxidation of sulfides showed good efficiency in both yield and selectivity.

Due to the importance of epoxides as useful intermediates in organic syntheses, some of which play key roles in the production of biologically active compounds and pharmaceutical agents, epoxidation of olefins to epoxides has been extensively studied [14–16]. Therefore, this study was extended to epoxidation of several different linear and cyclic olefins (table 3).

[(N–O)$_2$Mn(OAc)] catalyzed epoxidation of olefins to the corresponding epoxides in 29–67% yield and 100% selectivity. The highest and lowest yields were obtained for styrene and 1-hexene, respectively. As the data in figure 2 indicate, electron-rich olefins were more reactive than electron-poor ones. For instance, 1-methylcyclohexene and cyclohex-2-en-1-ol with methyl and hydroxyl group as electron-donating substituents gave

Table 1. Crystal data and structure refinement for [(N–O)$_2$Mn(OAc)].

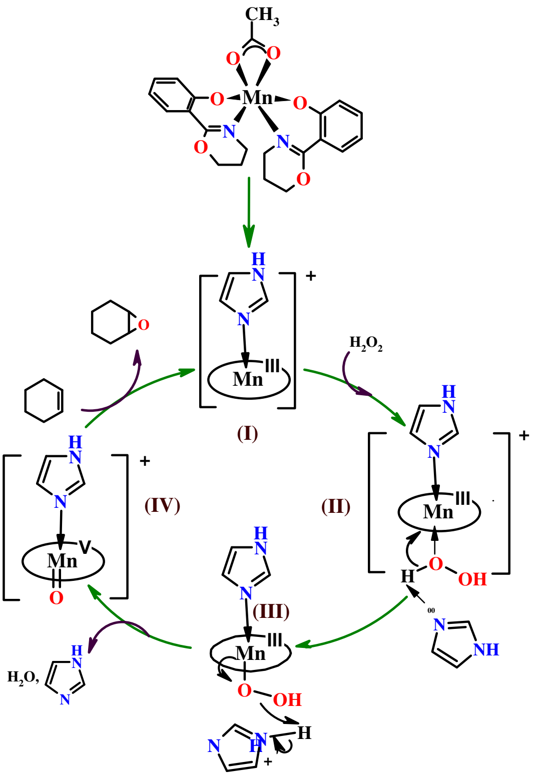
	[(N–O)$_2$Mn(OAc)]
Empirical formula	$C_{22}H_{23}MnN_2O_6$
Formula weight	466.36
Temperature	173(2) K
Wavelength	0.71073 Å
Crystal system	Monoclinic
Space group	*P 1 21/c 1*
Unit cell dimensions	$a = 14.0607(7)$ Å
	$\alpha = 90°$
	$b = 9.7785(5)$ Å
	$\beta = 108.1090(10)°$
	$c = 15.3720(8)$ Å
	$\gamma = 90°$
Volume	2008.85(18) Å^3
Z	4
Density (calculated)	1.542 Mg cm^{-3}
Absorption coefficient	0.701 mm^{-1}
F(0 0 0)	968
Crystal size	$0.12 \times 0.24 \times 0.24$ mm^3
Theta range for data collection	1.52° to 28.32°
Index ranges	$-18 \le h \le 18,\ -13 \le k \le 13,\ -20 \le l \le 20$
Reflections collected	20,219
Independent reflections	4994 [R(int) = 0.0313]
Completeness to theta = 28.70°	96.6%
Absorption correction	Multiscan
Max. and min. transmission	0.9332 and 0.7965
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	4994/0/218
Goodness-of-fit on F^2	1.020
Final R indices [$I > 2\mathrm{sigma}(I)$]	$R1 = 0.0335$, w$R2 = 0.0844$
R indices (all data)	$R1 = 0.0436$, w$R2 = 0.0902$
Largest diff. peak and hole	0.396 and −0.421 eÅ^{-3}

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

higher epoxidation yield than cyclohexene. In general, aromatic and cyclic olefins are slightly more reactive than linear olefins.

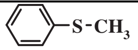
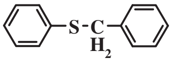
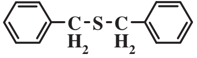
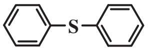
Turnover numbers are significantly increased if the reactions are performed in the presence of a relatively small amount of a heterocyclic base such as imidazole that is both as a ligand to Mn(III) and a base catalyst for efficient alkene epoxidation [17, 18]. According to our knowledge about the role of imidazole for catalytic reactivity of Mn(III) complex and by comparison with literature reports, we proposed a catalytic cycle for epoxidation of various olefins to epoxides using [(N–O)$_2$Mn(OAc)] (scheme 3).

To support the mechanism of catalytic reaction, electronic spectra of the complex were recorded in the presence of base, oxidant, and substrate. The role of imidazole is illustrated in figure 2. An absorption band near $\lambda = 380$ nm is assigned to the ligand-to-metal charge transfer [19] which decreased significantly by addition of imidazole to solution of [(N–O)$_2$Mn(OAc)], possibly due to the cleavage of axial Mn–O bond in the complex to form the first intermediate (I). The presence of imidazole could be important to increase the concentration of Mn–O–O–H species by proton abstraction from H_2O_2 (scheme 2) [10]



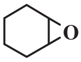
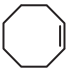
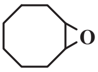
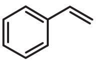
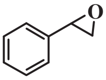
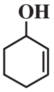
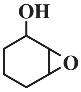
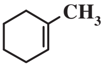
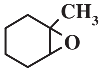
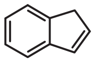
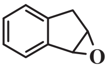
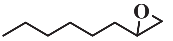
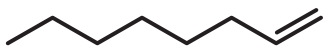
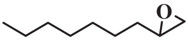
Scheme 3. Proposed catalytic cycle for olefin oxidation with UHP catalyzed by [(N–O)$_2$Mn(OAc)] and imidazole.

Table 2. Oxidation of sulfides catalyzed by [(N–O)$_2$Mn(OAc)]/ImH/UHP[a].

Entry	Substrate	Conversion (%)[b] (TON)[c]	Selectivity (%)[d]
1		71 (14.2)	77
2		66 (13.2)	79
3		63 (12.6)	76
4		59 (11.8)	73
5		54 (10.8)	77
6	$CH_3CH_2–S–CH_2CH_3$	61 (12.2)	75
7	$CH_3(CH_2)_2–S–(CH_2)_2CH_3$	60 (12)	77
8	$CH_3(CH_2)_7–S–(CH_2)_7CH_3$	57 (11.4)	79

[a]The molar ratios for [(N–O)$_2$Mn(OAc)] : imidazole : substrate : oxidant are 1 : 20 : 20 : 40. The reactions were performed in (1 : 1) mixture of CH_2Cl_2/CH_3OH (1 mL) under air at room temperature within 5 min.
[b]The GC yield (%) are measured relative to the starting sulfide.
[c]TON = (mmol of sulfoxide + mmol of sulfone)/mmol of catalyst.
[d]Selectivity to sulfoxide = (sulfoxide%/(sulfoxide% + sulfone%)) × 100.

Table 3. Details of the catalytic oxidation of olefins catalyzed by [(N–O)$_2$Mn(OAc)]/ImH/UHP[a].

Entry	Substrate	Product	Conversion (%) (TON)
1			37(7.4)
2			51(10.2)
3			67(13.4)
4			41(8.2)
5			45(9.0)
6			61(12.2)
7			29(5.8)
8			30(6.0)

[a]The molar ratios for [(N–O)$_2$Mn(OAc)] : imidazole : substrate : oxidant are 1 : 20 : 20 : 60. The reactions were performed in (1 : 1) mixture of CH_2Cl_2/CH_3OH (1 mL) under air at room temperature.

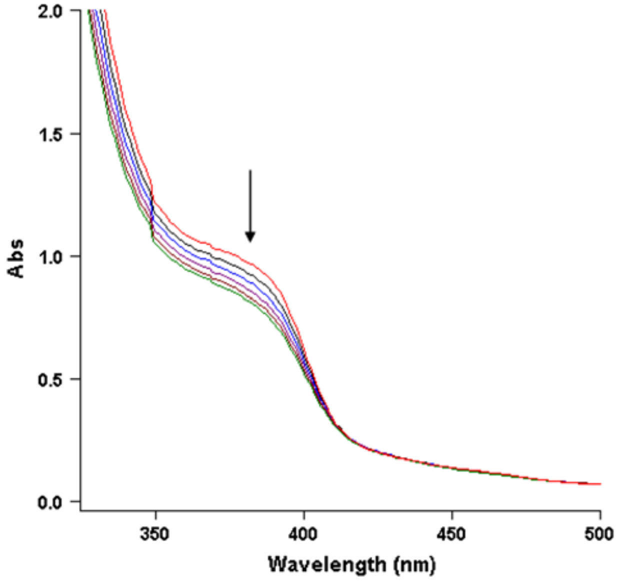


Figure 2. Visible spectra changes observed upon addition of imidazole to Mn(III) complex [(N–O)$_2$Mn(OAc)] in (1 : 1) mixture of CH_3OH/CH_2Cl_2, at 25 °C.

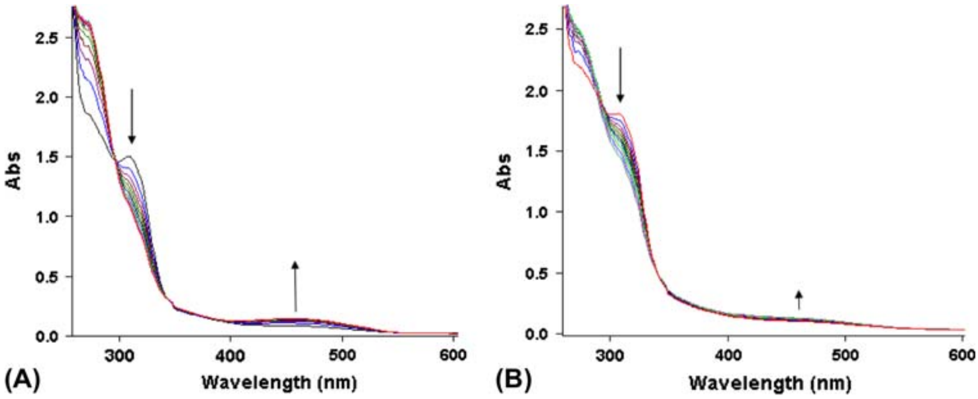


Figure 3. UV–vis spectral changes of a $CH_3OH–CH_2Cl_2$ (1/1 v/v) solution of Mn(III) complex $[(N–O)_2Mn(OAc)]$, adding (A) imidazole and UHP (B) imidazole, UHP, and cyclohexene (with 1.5 min time interval, at 25 ° C).

as well to favor the heterolytic cleavage of the O–O bond of Mn–O–O–H species (leading to $Mn^V=O$ and H_2O) over a possible homolytic cleavage (leading to OH formation and free radical oxidation reactions) [20, 21].

Formation of $Mn^V=O$ in the catalytic cycle is evidenced by addition of UHP and imidazole to a clear brown solution of Mn(III) complex in (1 : 1) mixture of CH_3OH/CH_2Cl_2 (figure 3(A)). A new band immediately appears at $\lambda_{max} = 457$ nm and repeated scanning reveals successive increase in intensity of this band that may be due to $Mo^V=O$ through a heterolytic cleavage of the O–O bond. This was accompanied by shifting the LMCT absorption to $\lambda_{max} = 310$ nm followed by gradual decrease in this band intensity. Decay of the absorption band at $\lambda_{max} = 457$ nm in the presence of olefin (figure 3(B)) indicates that the $Mn^V=O$ species is involved in oxygen transfer. This process was well illustrated by previous reports [18, 22–24]. It has been proposed that imidazole favors hydrocarbon monooxygenation over H_2O_2 dismutation at the level of an imidazole-$Mn^V=O$ intermediate (scheme 2) [18]. The initial brown color of the mixture attributed to Mn(III) complex is converted gradually to pale yellow, coincidental with decrease in catalytic activity. This observation indicates that catalytic activity is limited by complex degradation [13, 25].

4. Conclusion

$[(N–O)_2Mn(OAc)]$ has been synthesized and characterized by physico-chemical methods. We have demonstrated the effectiveness of this complex as a catalyst for oxidation of sulfides and epoxidation of olefins to their corresponding sulfoxides and epoxides, respectively. Easy preparation, mild reaction conditions, high yields of the products, short reaction time, high selectivity, and low cost make this catalytic system a useful method for oxidation.

Supplementary material

The CIF file of crystal structure complex $[(N–O)_2Mn(OAc)]$ has been deposited with the CCDC, No. 885686. 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.

Acknowledgements

M.A., M.B., and L.K.W. thank, respectively, the Research Council of University of Maragheh, Sharif University of Technology and the National Science Foundation (CHE-0809901) for funding of this work.

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