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Rapid and Efficient Aromatization of Hantzsch 1,4-Dihydropyridines with Potassium Peroxomonosulfate Catalyzed by Manganese(III) Schiff Base Complexes

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Rapid and efficient oxidation of Hantzsch 1,4-dihydropyridine with Potassium peroxomonosulfate is reported. The Mn(III)-salophen/monopersulfate catalytic system converts 1,4-dihydropyridines to their corresponding pyridine derivatives at room temperature in a 1:1, CH_3CN/H_2O mixture. The ability of various Schiff base complexes in the oxidation of 1,4-dihydropyridine was also investigated.

Keywords: Biomimetic oxidation, Manganese(III) Schiff base, Oxone, 1,4-Dihydropyridine

INTRODUCTION

The development of efficient catalytic systems for oxidation reactions that mimic the action of cytochrome P-450 dependent monooxygenases has attracted much attention in recent years [1]. The heme-containing monooxygenases cytochrome P-450 is known to play a key role in the oxidative metabolism of drugs and other environmental products, allowing their elimination from living organisms [1]. Therefore, numerous studies have been performed on the mimic of this enzyme [2,3]. Synthetic manganese porphyrins and related Schiff base complexes have also been shown to be versatile catalysts for the oxidation of a wide variety of organic substrates [4,5].

Iron and manganese porphyrins proved to be able to catalyze oxidation reactions using various single oxygen atom donors such as PhIO, ClO^-, H_2O_2, ROOH or IO_4^- [6]. The high efficiency of some of these systems makes them potentially useful for preparative oxidations in organic synthesis. On the other hand, metal complexes of salen and salophen ligands also aroused the interest of synthetic chemists as model compounds for the active site of cytochrome P-450, since they have features in common with metalloporphyrins with respect to their electronic structure and catalytic activity. The electronic and steric nature of the metal complex can be tuned by introducing electron-withdrawing and electron-releasing substituents and bulky groups in the ligand. Manganese, chromium, nickel, and cobalt Schiff base complexes have been used as catalysts for oxidation reactions [7].

Hantzsch 1,4-dihydropyridines are widely used as calcium channel blockers for the treatment of cardiovascular disorder including angina, hypertension and cardiac arrhythmias [8]. 1,4-Dihydropyridines are calcium antagonists [9], anti-tubercularagents [10], and neuropeptide Y Y1 receptor antagonists [11]. They possess neuroprotective [12], platelet antiaggregation [13], and antidiabetic activities [14]. Aromatization of 1,4-dihydropyridines has received considerable attention owing to the fact that in the human body, these compounds are oxidized to pyridine derivatives by the action of cytochrome P-450 in the liver [15]. These oxidized compounds are largely devoid of the pharmacological activity of the parent compounds. In addition,

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the corresponding pyridine derivatives show antihypoxic and antiischemic activities [16]. Additionally, dihydropyridines are often produced in a synthetic sequence and have to be oxidized to pyridines [17].

Numerous reagents and procedures have been recommended for this purpose, such as ferric or cupric nitrates on a solid support (clayfen or claycop) [18], manganese dioxide or 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) [19], pyridinium chlorochromate (PCC) [20], *tetrakis*-(pyridine) cobalt(II) dichromate (TPCD) [21], nicotinium dichromate [22], N_2O_4 complex of 18-crown-6 [23], diphenylpicrylhydrazyl and benzoyl peroxide as free radical oxidizing agents [24], $KMnO_4$ [25], silica gel supported ferric nitrate (silfen) [16], photochemical oxidation [26], inorganic acidic salts, sodium nitrite or nitrate [27], and Mn porphyrin/IO_4^- and Mn(salophen)/IO_4^- catalytic systems [28].

Potassium peroxomonosulfate is an inexpensive and readily accessible oxidizing agent. It is commonly used as Oxone ($2KHSO_5$-$KHSO_4$-K_2SO_4) and is a versatile oxidant for the transformation of a wide range of functional groups [29]. Recent reports have dealt with the use of a triple salt of potassium peroxomonosulfate, which can be used for the oxidation of alkenes [30], arenas [31], amines [32], imine [33], sulfides [34], selenides [35], α-amino acids [36], and acetals [37]. Oxone is a relatively stable peroxygen, and loses less than 1% of its activity per month when stored under appropriate conditions. However, like all other peroxygens, Oxone undergoes very slow decomposition in storage, with liberation of oxygen gas and a small amount of heat.

In this paper, we report the room temperature oxidation of 1,4-dihydropyridines with potassium peroxomonosulfate (Oxone) to their corresponding pyridine derivatives catalyzed by Mn-Salophen (Table 1) in CH_3CN/H_2O as solvent (Scheme 1). We have chosen the salophen ligand because it is similar to porphyrin, and the electronic and steric nature of the metal complex can be tuned by introducing electron-withdrawing and electron-releasing substituents and bulky groups in the ligand.

EXPERIMENTAL

Schiff base complexes 1-10 (Table 1) were prepared as described by Boucher [39] or by the more recently modified methods [7,40,41]. All Hantzsch 1,4-dihydropyridines were synthesized by the reported procedures [42]. ^{1}H NMR spectra were recorded on a Brucker AW80 (80 MHz) and a Bruker-Arance AQS 300 MHz spectrometers.

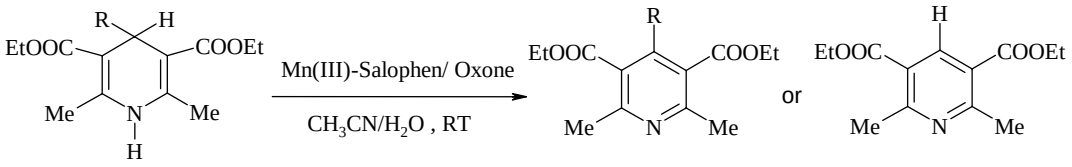
General Procedure for Oxidation of Hantzsch 1,4-Dihydropyridines to their Corresponding Pyridine Derivatives

All reactions were carried out at room temperature in a 25 ml flask equipped with a magnetic stirring bar. A solution of Oxone (2 mmol in 5 ml H_2O) was added to a mixture of Hantzsch 1,4-dihydropyridine (1 mmol), Mn-salophen (0.067 mmol) in CH_3CN (5 ml). The progress of the reaction was monitored by TLC. After reaction completion, the reaction mixture were extracted with CH_2Cl_2 (2 × 10 ml) and purified on a silica gel plate or a silica gel column (eluent: CCl_4-Et_2O). The identities of products were confirmed by m.p., IR, and ^{1}H NMR spectral data.

Diethyl 2,6-dimethyl-3,5-pyridinedicarboxylate (2a). Pale yellow solid; m.p.: 70-72 ºC (lit. [43c] 71-72 ºC). ^{1}H NMR ($CDCl_3$): δ (ppm) = 1.45 (t, 6H, J = 7.0 Hz), 2.82 (s, 6H), 4.40 (q, 4H, J = 7.0 Hz), 8.65 (s, 1H).

Diethyl 2,4,6-trimethyl-3,5-pyridinedicarboxylate (2b). Pale yellow oil (lit. [43c]). ^{1}H NMR ($CDCl_3$): δ (ppm) = 1.42 (t, 6H, J = 7.3 Hz), 2.28 (s, 3H), 2.49 (s, 6H), 4.40 (q, 4H, J= 7.3 Hz).

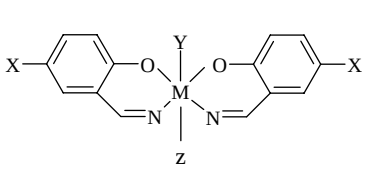
Diethyl 4-phenyl-2,6-dimethyl-3,5-pyridinedicarboxylate (2c). Pale yellow solid; m.p.: 60-62 ºC (lit. [43c] 60-61 ºC). ^{1}H NMR ($CDCl_3$): δ (ppm) = 0.95 (t, 6H, J = 7.2 Hz),

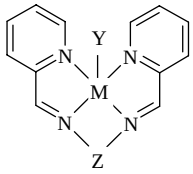


1 **2** **3**

Scheme 1

Table 1. Transition metal Schiff Base Complexes Used





	M	Z	X	Y	Yield (%)[a]
1	Fe	C_6H_4	H	Cl	7
2	Co	C_6H_4	H	-	9
3	Mn	C_6H_4	H	Cl	96
4	Mn	C_6H_4	NO_2	Cl	38
5	Ni	C_6H_4	H	-	6
6	Mn	$(CH_2)_2$	H	Cl	17
7	Fe	$(CH_2)_2$	H	Cl	12
8	Fe	C_6H_4	H	Cl	5
9	Mn	$(CH_2)_2$	H	Cl	22
10	Fe	$(CH_2)_2$	H	Cl	10

[a]Isolated yield for oxidation of 4-phenyl derivative of 1,4-dihydropyridines by various metal Schiff base complexes by Oxone.

2.58 (s, 6H), 4.03 (q, 4H, J = 7.2 Hz), 7.2-7.5 (m, 5H).

Diethyl 4-(2-chlorophenyl)-2,6-dimethyl-3,5-pyridinedicarboxylate (2d). Pale yellow solid; m.p.: 60-62 ºC (lit. [43d] 62 ºC).^{1}H NMR ($CDCl_3$): δ (ppm) = 0.97 (t, 6H, J = 7.1 Hz), 2.62 (s, 6H), 4.07 (q, 4H, J = 7.1 Hz), 7.20-7.41 (m, 4H).

Diethyl 4-(4-chlorophenyl)-2,6-dimethyl-3,5-pyridinedicarboxylate (2e). Pale yellow solid; m.p.: 65-67 ºC (lit. [43a] 65-66 ºC).^{1}H NMR ($CDCl_3$): δ (ppm) = 0.94 (t, 6H, J = 7.3 Hz), 2.59 (s, 6H), 4.05 (q, 4H, J = 7.3 Hz), 7.21 (d, 2H, J = 8.5 Hz), 7.32 (d, 2H, J = 8.4 Hz).

Diethyl 4-(4-nitrophenyl)-2,6-dimethyl-3,5-pyridinedicarboxylate (2f). Pale yellow solid; m.p.: 113-115 ºC (lit. [43c] 115-116 ºC). ^{1}H NMR ($CDCl_3$): δ (ppm) = 1.04 (t, 6H, J = 6.9 Hz), 2.64 (s, 6H), 4.08 (q, 4H, J = 6.9 Hz), 7.42 (d, 2H, J = 9.0 Hz), 8.29 (d, 2H, J = 9.0 Hz).

Diethyl 4-(2-nitrophenyl)-2,6-dimethyl-3,5-pyridinedicarboxylate (2g). Pale yellow solid; m.p.: 75-77 ºC (lit. [18] 75-76 ºC).^{1}H NMR ($CDCl_3$): δ (ppm) = 0.96 (t, 6H, J = 7.2 Hz), 2.60 (s, 6H), 3.98 (q, 4H, J = 7.2 Hz), 7.22 (s, 1H), 7.56 (t, 2H, J = 5.4 Hz), 8.23 (m, 1H).

Diethyl 4-(3-nitrophenyl)-2,6-dimethyl-3,5-pyridinedicarboxylate (2h). Pale yellow solid; m.p.: 59-61 ºC (lit. [43e] 61-62 ºC). ^{1}H NMR ($CDCl_3$): δ (ppm) = 0.90 (t, 6H, J = 7.0 Hz), 2.62 (s, 6H), 4.08 (q, 4H, J = 7.0 Hz), 7.50-7.75 (m, 2H), 8.12-8.27 (m, 2H).

Diethyl 4-(2-methoxyphenyl)-2,6-dimethyl-3,5-pyridine dicarboxylate (2j). Pale yellow solid; m.p.: 55-57 ºC (lit. [43f] 57-58 ºC). ^{1}H NMR ($CDCl_3$): δ (ppm) = 0.90 (t, 6H, J = 7.2 Hz), 2.65 (s, 6H), 3.74 (s, 3H), 4.04 (q, 4H, J = 7.2 Hz), 6.95-7.36 (m, 4H).

Diethyl 4-(4-methoxyphenyl)-2,6-dimethyl-3,5-pyridine dicarboxylate (2k). Pale yellow solid; m.p.: 49-50 ºC (lit. [43b] 51-53 ºC). ^{1}H NMR ($CDCl_3$): δ (ppm) = 0.91 (t, 6H, J = 7.4 Hz), 2.52 (s, 6H), 3.69 (s, 3H), 3.95 (q, 4H, J = 7.4 Hz), 6.80 (d, 2H, J = 6.9 Hz), 7.08 (d, 2H, J = 6.9 Hz).

Diethyl 4-(2-pyridyl)-2,6-dimethyl-3,5-pyridinedicarboxylate (2m). Pale yellow solid; m.p.: 89-91 ºC (lit.[43g] 88 ºC). ^{1}H NMR ($CDCl_3$): δ (ppm) = 0.92 (t, 6H, J = 7.4 Hz), 2.62 (s, 6H), 4.05(q, 4H, J = 7.4 Hz), 7.0-7.8 (m, 3H), 8.52 (d, 1H, J = 5.0 Hz).

Diethyl 4-(2-furyl)-2,6-dimethyl-3,5-pyridinedicarboxylate (2n). Pale yellow solid; m.p.: 37-39 ºC (lit. [43d] 38-41

ºC). ^{1}H NMR ($CDCl_3$): δ (ppm) = 1.18 (t, 6H, J = 7.0 Hz), 2.49 (s, 6H), 4.21 (q, 4H, J = 6.9 Hz), 6.37 (d, 1H, J = 3.5 Hz), 6.57 (d, 1H, J = 3.5 Hz), 7.42 (br, s, 1H).

RESULTS AND DISCUSSION

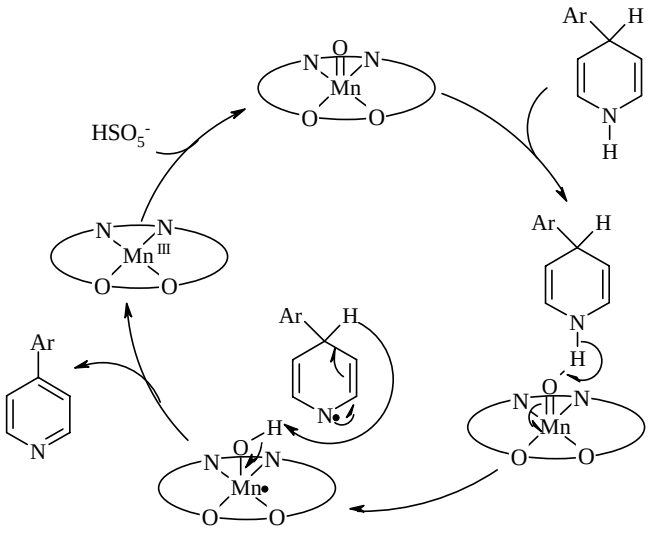
Oxidation of 1,4-Dihydropyridine with Oxone Catalyzed by Mn(III)-salophene

Initially, in order to show the monopersulfate anion activation by the Schiff base complex, the catalytic oxidation of 4-phenyl derivative of 1,4-dihydropyridine with Oxone in the CH_3CN/H_2O was investigated. The obtained results showed that Mn(III)-salophen is an efficient catalyst in the oxidation of 4-phenyl derivative of 1,4-dihydropyridine with Oxone at room temperature. The manganese(III) salophen/Oxone catalytic system can be used for oxidation of a wide variety of 1,4-dihydropyridine derivatives bearing an alkyl or an aryl group to their corresponding pyridine derivatives in excellent yields at room temperature. A simple proposed catalytic system shown in Scheme 2.

As shown in Table 2, oxidation of 4-isopropyl and 1-methyl benzyl derivatives (alkyl moiety may be responsible for generating stable carbocation) was accompanied by leaving of this substituent and gave dealkylated pyridine derivatives (entries 9 and 12) which was previously reported by Ortiz de Montellano in the oxidation of 1,4-dihydropyrines by cytochrome P-450 [38]. This approach shows that this synthetic model behaves as cytochrome P-450. All reactions were completed during the appropriate time and gave only the corresponding pyridine derivative. The results are summarized in Table 1. In the absence of Mn(III) salophen catalyst, Oxone has poor ability to oxidize 1,4-dihydropyridines at room temperature (6-10% yields).

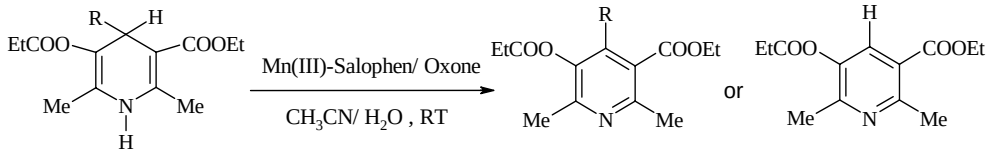
Oxidation of 1,4-Dihydropyridine Derivatives with Different Metal-Schiff Base Complexes

In order to show the peroxomonosulphate anion activation by metal Schiff base complexes, we decided to investigate the activity of various Schiff base complexes of Fe, Mn, Ni, and Co as metal ions. The obtained results on catalytic oxidation of 4-phenyl derivative of 1,4-dihydropyridine with Oxone in the presence of different Schiff base complexes (Table 1) indicated that the nature of the metal ion has an important role on the catalytic activity of Schiff base complexes. The iron, cobalt, and nickel complexes resulted a small amount of the corresponding pyridine derivative in the oxidation of 4-phenyl derivative of 1,4-dihydropyridine. In the case of nickel and cobalt, these metal ions are not capable of forming the high oxidation state oxo species. However, the use of manganese(III) complexes give a higher oxidized product in



Scheme 2. A proposed catalytic cycle for oxidation of 1,4-DHP's by the manganese(III) salophen/ Oxone system.

Table 2. Oxidation of Hantzsch 1,4-Dihydropyridines with Oxone Catalyzed by Mn(III)-Salophen[a]



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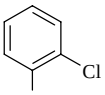
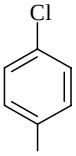
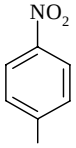
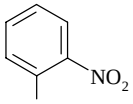
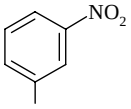
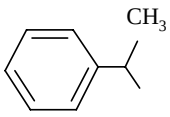
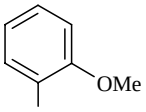
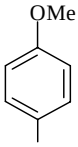
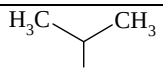
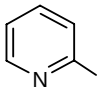
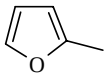
Entry	DHP's	R	Time (min)	Yields (%)[c]
1	**1a**	H	5	92
2	**1b**	CH_3	50	95
3	**1c**		30	94
4	**1d**		70	96
5	**1e**		45	92
6	**1f**		60	91
7	**1g**		130	93
8	**1h**		120	95
9	**1i**		80[b]	93
10	**1j**		140	94
11	**1k**		20	92

Table 2. Continued

12	**1l**		60[b]	96
13	**1m**		120	94
14	**1n**		90	95

[a]All products were identified by comparison with authentic samples (IR, ^{1}H NMR, m.p.). Reaction conditions are Oxone (2 mmol in 5 ml H_2O), 1,4-dihydropyridine (1 mmol), Mn-salophen (0.067 mmol) in CH_3CN (5 ml). [b]The product is a dealkylated pyridine derivative. [c]Isolated yield.

the oxidation of 4-phenyl derivative of 1,4-dihydropyridine.

Effect of Solvent on the Oxidation of 4-Phenyl Derivative of 1,4-Dihydropyridine

Among the 1:1 mixture of methanol, ethanol, acetone, acetonitrile (single-phase systems), chloroform, and carbon tetrachloride with water (two-phase systems), the 1:1 acetonitrile/water mixture was chosen as the reaction medium, because the metal Schiff base complexes are highly soluble in this solvent and higher pyridine derivative yields were obtained (Table 3).

Table 3. Effect of Solvent on the Oxidation of 4-Phenyl Derivative of 1,4-Dihydropyridine

Solvent	Yield (%)[a] after 60 min
CH_3CN/H_2O	97
CH_3COCH_3/H_2O	75
CH_3OH/H_2O	58
CH_3CH_2OH/H_2O	42
$CHCl_3/H_2O$	15
CH_2Cl_2/H_2O	33
CCl_4/H_2O	10

[a]Isolated yield.

Effect of Axial Ligand on the Oxidation of 4-Phenyl Derivative of 1,4-Dihydropyridine

When using metalloporphyrins and Schiff base complexes as catalysts, addition of an axial base in biomimetic systems is necessary to obtain high catalytic activity. This system shows a higher catalytic activity in the absence of imidazole. When imidazole is added as axial ligand to this catalytic system, the reaction times become longer for the oxidation of 1,4-dihydropyridines. For example, the oxidation of 4-phenyl and 4-nitrophenyl derivatives was completed in 30 and 60 min, respectively. Addition of imidazole as co-catalyst led to longer reaction times of 60 and 90 min, respectively, for 4-phenyl and 4-nitrophenyl derivatives. These observations show that the 1,4-dihydropyridines can play the axial ligand role.

CONCLUSIONS

Mn(III)-salophen/Oxone catalytic system has the following advantages in the oxidation of Hantzsch 1,4-dihydropyridines to their corresponding pyridine derivatives: (i) properly reaction time, (ii) high efficiency for oxidation of Hantzsch 1,4-dihydropyridines to their corresponding pyridine derivatives, (iii) mild reaction conditions, and (iv) cheapness and stability of the oxidant. Therefore, the present method could be a useful addition to the available methods in organic synthesis.

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