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Synthesis, physicochemical studies and aerobic enantioselective epoxidation of non functionalized olefins catalyzed by new Co(II) chiral salen complexes

R.I. Kureshy *, N.H. Khan, S.H.R. Abdi, A.K. Bhatt, P. Iyer

Central Salt and Marine Chemicals Research Institute, Bhavnagar - 364 002, India

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Abstract

Co(II) chiral salen complexes **1**–**3** derived from α-naphthyl salicylaldehyde with 1*S*,2*S* (+) diaminocyclohexane, 1*R*,2*R* (−) diaminodiphenylethane and *S*(+) 1,2-diaminopropane have been prepared. The characterization of the complexes was done by microanalysis, magnetic moment, IR-, UV/Vis-, CD spectral studies, optical rotation, conductance measurements and cyclic voltammetry. Epoxidation of non-functionalized prochiral olefins viz. styrene, *trans* 3-nonene and *trans* 4-octene was achieved by the combined use of an atmospheric pressure of molecular oxygen and sacrificial reductant isobutyraldehyde catalyzed by the above synthesized Co(II) chiral salen complexes with and without pyridine *N*-oxide as cooxidant. Good yields of the desired epoxide were obtained with the substrate *trans* 3-nonene and *trans* 4-octene by GLC. Enantiomeric excess of the epoxide were evaluated by ^{1}H NMR using chiral shift reagent Eu(hfc)$_3$ and by chiral capillary column.

Keywords: Aerobic epoxidation; Enantioselective; Non-functionalized olefins; Cobalt; Chiral Schiff base

1. Introduction

Enantioselective epoxidation of prochiral olefins is one of the most challenging area in organic synthesis [1–3] because the epoxide formed are very useful synthetic intermediates for complexed chiral bioactive molecules or as end product which also have biological activity such as gypsy moth insect pheromones [4]. Much attention has been paid towards the development of direct and selective epoxidation of olefins by use of molecular oxygen and a suitable reductant which can accept one oxygen atom from molecular oxygen to perform the reaction [1–3,5–9]. Several reports have been published about the utility of primary alcohol [10], aldehydes [6] or cyclic ketones [3] as efficient reductant in the aerobic oxidation of olefins catalyzed by transition metal complexes [11–13].

In continuation of our earlier work on enantioselective epoxidation of non functionalized olefins using Ru(II), Ru(III) and Mn(III) chiral Schiff base complexes [14–16] and to explore

* Corresponding author. Fax: +91-278-426970; e-mail: general@cscsmcri.ren.nic.in.

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detailed mechanistic analysis of an efficient catalytic system, we are reporting here the synthesis and characterization of new chiral salen Co(II) complexes derived from α-naphthyl salicylaldehyde with 1*S*,2*S*(+) diaminocyclohexane, 1*R*,2*R*(−) diaminodiphenylethane and *S*(+) 1,2-diaminopropane and the use of these catalysts for enantioselective epoxidation of *trans* 3-nonene, *trans* 4-octene and styrene using molecular oxygen with isobutyraldehyde as reductant with and without pyridine *N*-oxide as cooxidant.

2. Experimental

Cobalt acetate (Sisco), nitroaniline, naphthalene, zinc chloride, zinc dust, sodium nitrite, tin metal, sodium hydroxide, potassium hydroxide, pyridine *N*-oxide, acidic aluminum oxide (National), benzene, petroleum ether (40–60°C), acetone, sulfuric acid, chloroform (AR grade), styrene, *trans* 4-octene and *trans* 3-nonene, $Eu(hfc)_3$, 1*S*,2*S*(+) cyclohexanediamine and 1*R*,2*R*(−) diphenyldiaminoethane (Aldrich) were used as received. *S*(+) 1,2 diaminopropane were resolved from racemic mixture by literature procedure [17].

2.1. Synthesis of new chiral salen ligand

Before salen ligand synthesis L_1–L_3, 3(1-naphthyl)salicylaldehyde was prepared as described in Scheme 1.

Diazotization of *o*-nitroaniline in presence of zinc chloride gave *o*-nitrobenzene diazonium tetrazincate as light cream solid referred as **1**. The arylation of naphthalene in dry acetone with **1** gave nitrophenylnaphthalene in the form of two isomers (**a**) and (**b**). These isomers were chromatographed through acidic alumina in 9:1 petroleum ether and benzene to give 2(1-naphthyl) nitro benzene, (**b**) in the form of pale yellow needles as major isomer. Reduction of 2-(1-naphthyl) nitrobenzene by tin metal and concentrated hydrochloric acid gave 2(1-naphthyl) aniline **2**. It was further diazotized followed by hydroxylation reaction forming 2(1-naphthyl) phenol **3**. The phenolic compound **3** further undergoes Riemer Tiemann reaction producing the requisite **4** i.e., 3(1-naphthyl) salicylaldehyde. All the intermediates **1** to **4** were also purified and characterized by elemental analysis, IR and ^{1}H NMR. The condensation of 1*S*,2*S*(+) cyclohexane diamine, 1*R*,2*R*(−) diphenyl diaminoethane and *S*(+)1,2 diaminopropane with **4** gave the chiral schiff base ligands L_1–L_3. The chiral schiff bases were characterized by microanalysis, IR-, ^{1}H and $^{13}C\{^1H\}$ NMR spectra. The data is given below.

8*S*,8′*S*(+) bis{3 (1′-naphthyl) salicylidenecyclohexanediamine} (*S*-NAPHEX SAL)-(L_1)-yield 65%, m.p. 76°C, ^{1}H NMR ($CDCl_3$): δ1.64–1.87 [m, H_{10}, $(CH_2)_4$], 2.16–2.24 [m, H_9, $(CH_2)_4$], 2.8 (m, H_8, CH), 7.24–8.01 (m, aromatic phenyl and naphthyl), 9.04 (s, H_7, N=CH) and 13.44 (bs, H_2, OH), $^{13}C\{^1H\}$ NMR (CH_2Cl_2), δ, 24.71 (C_{10}), 33.43 (C_9), 58.23 (C_8), 191.39 (C–H=N), 116.04–135.81 (16 lines) (phenyl and naphthyl), calculated for $C_{40}H_{34}N_2O_2$: C, 83.59; H, 5.96; N, 4.87. Found C, 83.09; H, 5.93; N, 4.84, IR(KBr): ν(C=N) 1625 cm^{-1}.

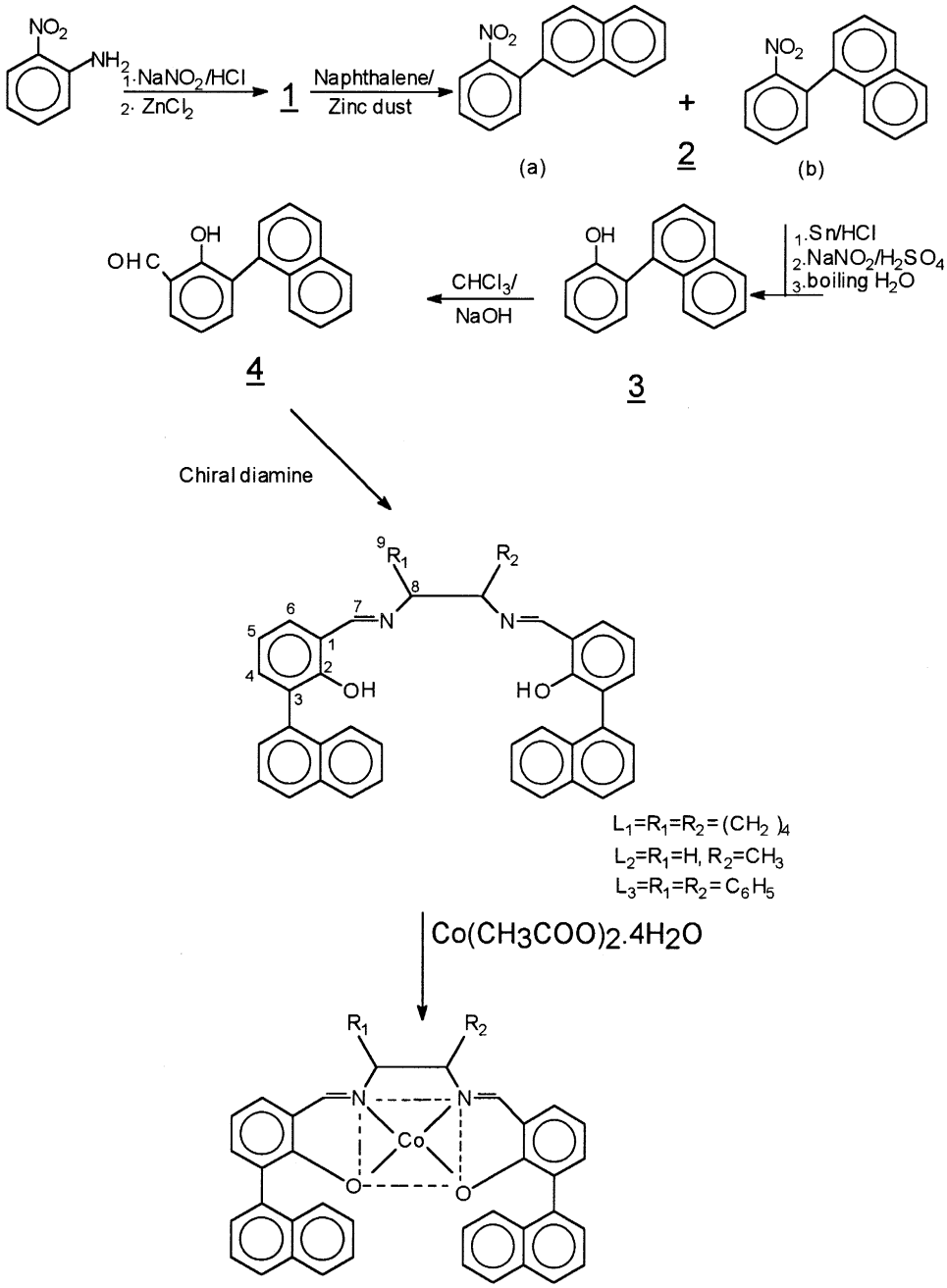
S(+)bis{3(1′-naphthyl)salicylidenepropylenediamine} (*S*-NAPROSAL)-(L_2)-yield 60%, m.p. 94°C, ^{1}H NMR ($CDCl_3$) 1.26 (d, CH_3), 2.16 (d, CH_2), 2.4 (m, H_8, CH), 7.12–8.24 (m, aromatic phenyl and naphthyl), 9.04 (s, H_7, N=CH) and 13.20 (bs, H_2, OH), $^{13}C\{^1H\}$ NMR (CH_2Cl_2), δ, 13.8 (C_9) 24.6 (CH_2), 28.7 (C_8), 190.11 (C–H=N), 116–136.24 (16 lines, phenyl, naphthyl), calculated for $C_{37}H_{30}N_2O_2$:C, 83.10: H, 5.65; N, 5.24. Found C, 82.96; H, 5.62; N, 5.21, IR(KBr): ν(C=N) 1630 cm^{-1}.

8*R*,8′*R*(−) bis{3(1′-naphthyl)salicylidenediphenyldiamine} (*R*-NAPDAMSAL)-(L_3)-yield 60%, m.p. = 124°C, ^{1}H NMR ($CDCl_3$); 2.60 (s, H_8, CH), 7.33–7.94 (m, aromatic phenyl and naphthyl) 10.09 (s, H_7, N=CH) and 12.80 (bs, H_2, OH), $^{13}C\{^1H\}$ NMR (CH_2Cl_2), δ,

26.08 (C_8), 199.54 (C–H=N), 110–135.54 (20 lines, phenyl and naphthyl). Calculated for $C_{48}H_{36}N_2O_2$: C, 85.68; H, 5.39; N, 4.16. Found: C, 85.46; H, 5.08; N, 4.22, IR(KBr): ν(C=N) 1630 cm^{-1}.

2.2. Synthesis of the catalysts *1–3*

Ethanolic solution of appropriate amount of chiral schiff bases L_1, L_2 and L_3 (0.001 mol) containing KOH 0.5 M (4 ml) was stirred vigor-



Scheme 1.

ously at reflux in argon atmosphere. Cobalt acetate (0.001 mol) was then added and the resulting solution was refluxed for 10–12 h. The completion of reaction was checked on TLC and the solution was filtered, concentrated on rotaevaporator and precipitated by petroleum ether. The complexes were filtered again and dried in vacuo and recrystallized from acetonitrile/CH_2Cl_2.

The analytical data for the complexes are given below: 8*S*,8′*S*(+) bis{3(1′-naphthyl)salicylidene cyclohexane diiminato cobalt(II) (*S*-NAPHEXSAL Co(II)} entry **1** — Micro analysis calculated for $C_{40}H_{32}N_2O_2$ Co: C, 76.06; H, 5.11; N, 4.44. Found: C, 76.00; H, 5.09; N, 4.36. IR(KBr) cm^{-1} 1580 (H–C=N), 1260 ν(C–O). UV/vis (nm)(MeOH) λ_{max} (ϵ), 288 (9996), 350 (9996). CD (MeOH) λ_{max} ($\Delta\epsilon$) 380(+2.5) 425 (−0.5), 480(+2.9), 550 (−4.5); $[\alpha]_D^t = +53.82$; Configuration (*S*); Λ_M (MeOH) 5 mho cm^{-1} mol^{-1}, μ_{eff} (BM) 2.4; $\Delta Ep_c = -1.38$ V.

S(+) bis{3(1′-naphthyl)salicylidene 1,2-diamino propanato cobalt (II) (*S*-NAPROSAL Co(II)) entry **2** — Microanalysis calculated for $C_{37}H_{28}N_2O_2$ Co: C, 75.12; H, 4.77; N, 4.74. Found: C, 75.06; H, 4.70; N, 4.72. IR(KBr) cm^{-1} 1585 ν(H–C=N), 1265 ν(C–O), UV/vis (nm)(MeOH) λ_{max} (ϵ), 218 (7660), 288 (26040), 348 (1180). CD (MeOH) λ_{max} ($\Delta\epsilon$) 398 (+1.6), 460 (+2.2), 570(+2.2); $[\alpha]_D^t = +37.18$; Configuration (*S*); Λ_M (MeOH) 4 mho cm^{-1} mol^{-1}, μ_{eff} (BM) 2.35; $\Delta Ep_c = -1.40$ V.

8*R*,8′*R*(−) bis{3(1′-naphthyl)salicylidene diphenyl diiminato Cobalt(II) (*R*-NAPDAMSAL Co(II)) entry **3** — Microanalysis calculated for $C_{48}H_{34}N_2O_2$ Co: C, 79.00; H, 4.70; N, 3.83. Found: C, 78.94; H, 4.65; N, 3.80. IR(KBr) Far IR (Nujol mull) cm^{-1} 1580 ν(H–C=N), 1260 ν(C–O), UV/Vis (nm) (MeOH) λ_{max} (ϵ), 273 (9812), 280(9006), 351^{sh} (2468), 357^{sh} (2600) nm. CD (nm) (MeOH) λ_{max} ($\Delta\epsilon$) 325 (+0.4), 350 (−2.5), 450 (+1.5), $[\alpha]_D^t = -19.23$; Configuration (*R*); $\Lambda_M = 4$ mho cm^{-1} mol^{-1}, μ_{eff} (BM) 2.4; $\Delta Ep_c = -1.40$ V.

3. Aerial enantioselective epoxidation of styrene, *trans* 3-nonene and *trans* 4-octene by the catalyst 1–3

Enantioselective epoxidation of styrene, *trans* 3-nonene and *trans* 4-octene by the catalyst entry **1**–**3** with molecular oxygen was carried out by the following procedure: The chiral catalyst (0.006 mmol), styrene, *trans* 3-nonene and *trans* 4-octene (2 mmol), pyridine *N*-oxide (0.24 mmol) dissolved in 5 ml dichloromethane was stirred in the presence of molecular oxygen at 4°C in the dark. After each interval of 30 min an aliquot was taken from the reaction mixture and analyzed by GLC. After the reaction was completed the solvent was removed and the product styrene oxide, *trans* 3-nonene oxide and *trans* 4-octene oxide was separated from the reaction mixture using short column of basic alumina in hexane:dichloromethane (9:1) as eluent. Evaluation of enantiomeric excess was done by chiraldex GTA. Besides, the product was taken in $CDCl_3$ for ^1H NMR using chiral shift reagent Eu(hfc)$_3$ for further evaluation of enantiomeric excess.

4. Methods

Microanalysis of the complexes was done on a Carlo Erba Analyser Model 1106. Molar conductance was measured at room temperature on a Digisun electronic conductivity bridge DI-909. The IR spectra were recorded on Carl Zeiss Specord M-80 spectrophotometer in KBr/nujol mull. Electronic spectra were recorded on Shimadzu UV/Visible recording spectrophotometer Model 160. ^1H NMR 99.55 MHz and $^{13}C\{^1H\}$ NMR 24.99 MHz were done on Jeol FX-100 NMR spectrometer in $CDCl_3$ and CH_2Cl_2. The magnetic moment measurements were done at 298 K by the Gouy method using Hg{Co(SCN)$_4$} as calibrant and experimental susceptibilities were corrected for diamagnetism. Cyclic voltammetry, differential pulse voltammogram were recorded with a Princeton Applied Research (PAR) instrument using tetra-

butylammonium-tetrafluoroborate as supporting electrolyte in dichloromethane. The optical rotation of the complexes in methanol was measured by polarimeter Atago, Japan. The CD spectra were recorded in methanol by Jasco Machine Model J-20 Japan. The purity of the solvent, substrate and analysis of the product was determined by GLC using Shimadzu GC 14B coupled with PC using 2 M long, 3 mm I-D, 4 mm O.D. stainless steel column packed with SE30, 5% mesh size 60 to 80 with FID detector. Column temperature programmed between 70 to 170°C and injection temperature 200°C with nitrogen carrier gas flow 30 ml/min. Synthetic standards of the product were used to determine yields by comparison of peak height and area. The optical yield of the product was determined by chiraldex GTA type column.

5. Results and discussion

The complexes **1**–**3** have been established as neutral low spin square planar complexes (μ_{eff} 2.4 BM) derived from α-naphthylsalicylaldehyde with 1*S*,2*S* (+) diaminocyclohexane, 1*R*,2*R*(−) diaminodiphenylethane and *S*(+) 1,2-diaminopropane. The details of synthesis is given in Section 2 (Scheme 1).

In the IR spectra a strong band at 3320 cm^{-1} in all chiral Schiff bases is assigned to ν(O–H). After complexation with metal ions this band disappeared showing the replacement of hydrogen by metal atom. In such cases the ν(C–O) at 1280 cm^{-1} in all chiral Schiff bases show a red shift in the frequency of the complexes. The bands near 1590–1580 cm^{-1} in all the complexes are due to co-ordinated azomethine nitrogen. These bands lie at higher wave number in free chiral Schiff bases.

The electronic spectra of the complexes in methanol show high intensity charge transfer band near 288 ($\epsilon = 9996$) nm while the MLCT band lie at 350 ($\epsilon = 1980$) nm.

CD spectra of the complexes were recorded in methanol which shows that the complex **2** is

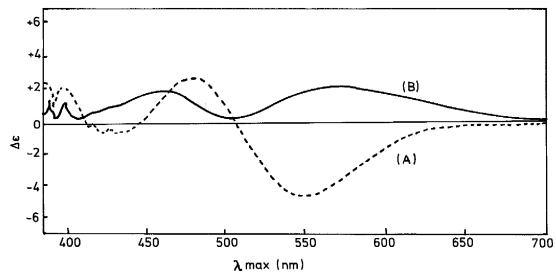


Fig. 1. CD spectra of A (---) S-NAPHEXSAL Co(II) and B (——) S-NAPROSAL Co(II) in methanol.

stereospecifically co-ordinated to cobalt so that the gauch chelate ring is located in λ conformation while the complexes **1** and **3** are in the equilibrium mixture of the two isomers δ and λ. Two representative CD spectra (A) *S*-NAPHEXSAL Co(II) and (B) *S*-NAPROSAL Co(II) are shown in Fig. 1. The preferred conformation and configuration depend on the steric interaction between the substituents at asymmetric center [18,19] and the size of the metal ions. In the ligand field region bands near 550 (−4.5) and 570(−1.6) nm are assigned to dd bands and spin forbidden ligand bands while $d \rightarrow \pi^*$ bands lie between 450(−1.25) to 480(+2.9) nm. The high intensity $\pi \rightarrow \pi^*$ transitions are seen at 350(−2.5) and 398(+1.6) nm.

The cyclic voltammogram of the complexes **1**–**3** were recorded in dichloromethane using tetrabutyl ammonium tetrafluoroborate as supporting electrolyte with a reference electrode Ag\AgCl under nitrogen showing a cathodic–anodic peak in the range of −0.59 to −0.63 V due to Co(III)/Co(II) couple while CV of all the complexes show irreversible behavior of Co(II)/Co(I) couple with ΔEp_c near −1.30 to −1.40 V respectively. These redox values are also reported earlier [20].

6. Aerobic enantioselective epoxidation

The catalyst entry **1**–**3** were first screened for aerobic enantioselective epoxidation of prochiral non functionalized olefins viz. *trans* 3-nonene, *trans* 4-octene and styrene using molecular oxygen in presence of isobutyraldehyde as sac-

Table 1
Data for aerobic enantioselective epoxidation of prochiral non functionalized olefins catalyzed by chiral Co(II) salen complexes without pyridine *N*-oxide

Catalyst	Substrate	Time (h)	Conversion (%) [b]	ee [c]	Configuration
1	styrene	24	35	28	*R*
	t-3 nonene	24	80	30	*R*
	t-4 octene	24	50	24	*R*
2	styrene	24	30	22	*R*
	t-3 nonene	24	75	28	*R*
	t-4 octene	24	70	20	*R*
3	styrene	24	36	20	*S*
	t-3 nonene	24	60	25	*S*
	t-4 octene	24	35	18	*S*

[a] Reaction conditions: Substrate (2 mmol), catalyst (0.006 mmol),isobutyraldehyde (6 mmol) solvent 5.0 ml dichloromethane, 1 atm. O_2 at 4°C.
[b] Determined by GC analysis.
[c] Determined by Chiraldex GTA and by ^{1}H NMR using Eu(hfc)$_3$.

rificial reductant, with and without pyridine *N*-oxide as cooxidant by GLC to give corresponding epoxide and the carboxylic acid as coproduct. Data regarding enantioselectivity is given in Tables 1 and 2. It is reasonable to assume that isobutyraldehyde behaves as an effective reductant to accept one oxygen atom of molecular oxygen in the present reaction system. The reaction involved during epoxidation is shown in Scheme 2A, B.

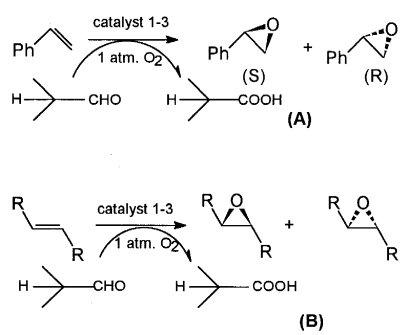
Table 2
Data for aerobic enantioselective epoxidation of prochiral non functionalized olefins catalyzed by chiral Co(II) salen with pyridine *N*-oxide

Catalyst	Substrate	Time (h)	Conversion (%) [b]	ee [c]	Configuration
1	styrene	24	45	45	*R*
	t-3 nonene	24	90	55	*R*
	t-4 octene	24	65	40	*R*
2	styrene	24	38	40	*R*
	t-3 nonene	24	82	42	*R*
	t-4 octene	24	78	38	*R*
3	styrene	24	36	37	*S*
	t-3 nonene	24	72	42	*S*
	t-4 octene	24	48	52	*S*

[a] Reaction conditions: Substrate (2 mmol), catalyst (0.006 mmol), isobutyraldehyde (6 mmol) solvent 5.0 ml dichloromethane, Pyridine *N*-oxide (0.24 mmol), 1 atm. O_2 at 4°C.
[b] Determined by GC analysis.
[c] Determined by Chiraldex GTA and by ^{1}H NMR using Eu(hfc)$_3$.

In absence of pyridine *N*-oxide *trans* 3-nonene gave very good conversion with catalyst **1** and **2** (75–80%) while better conversion was obtained with *trans* 4-octene with catalyst **2** (70%). Moderate to low conversion was obtained in case of styrene with these catalyst **1**–**3** (20–30%). When reaction was conducted in presence of pyridine *N*-oxide, the epoxide conversion was improved in all the cases (Table 2).

The enantiomeric excess for the resulting epoxide separated by short column of basic alumina. was evaluated by chiral capillary column (Chiraldex GTA) and also by ^{1}H NMR using chiral shift reagent Eu(hfc)$_3$. In the case of styrene the ^{1}H NMR show a set of three peaks with a doublet of doublet and two triplets between 2.6–3.2 ppm assigned to the product epoxide. The peak for the H *trans* to the phenyl ring appearing at 3 ppm was shifted down field and splitted into two sets of triplets between δ 3.8–4.4 ppm when treated with several equivalents of chiral shift reagents Eu(hfc)$_3$. The more downfield shifted the $R(+)$ isomer peak which was confirmed by the increase in the respective peak when enantiomerically pure $R(+)$ or $S(-)$ styrene oxide was added to the test sample respectively. For *trans* 3-nonene and *trans* 4-octene epoxide a broad triplet near δ 2.6 ppm in ^{1}H NMR is shifted to 3.6 to 3.9 ppm and 4.78–5.03 ppm as two sets of triplets on addition of several equivalents of Eu(hfc)$_3$ respectively and is used to evaluate the enantiomeric excess.



Scheme 2.

Furthermore, it is interesting to point out that the presence of catalytic amount of pyridine *N*-oxide improves the enantioselectivity without any change in configuration. In all the cases on employment of *S* form of the catalyst resulted in *R* form of the product as a dominant enantiomer. This trend was already seen by Jacobsen [21] and Katsuki [22] for Mn(III) salen complexes using iodosyl benzene as terminal oxidant while the trend was reversed with combined use of molecular oxygen with aldehyde by Yamada [23] in presence of *N*-methyl imidazole.

7. Conclusion

In this paper we have elucidated the aerobic enantioselective epoxidation of styrene, *trans* 3-nonene and *trans* 4-octene using Co(II) chiral schiff base complexes in presence of molecular oxygen using isobutyraldehyde as sacrificial reductant. The catalyst **1** gave very good conversion with *trans* 3-nonene while catalyst **2** favors the formation of *trans* 4-octene oxide in good yield. Moderate to low conversion was obtained with styrene in all the cases. However, in the presence of pyridine *N*-oxide both conversion and enantioselectivity was improved by maintaining the absolute configuration. Further investigation on the present reaction and development of more effective ligands are under process.

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