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Research Note

Chiral macrocyclic salen Mn(III) complexes catalyzed enantioselective epoxidation of non-functionalized alkenes using NaOCl and urea H_2O_2 as oxidants

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ABSTRACT

Two new chiral Mn(III) macrocyclic salen complexes **1a** and **1b** were prepared for the enantioselective epoxidation of non-functionalized alkenes. A 5 mol% loading of these catalysts in the presence of pyridine *N*-oxide as an axial base and sodium hypochlorite or urea hydrogen peroxide adduct as oxidant worked well to give respective epoxides in high yields and ee (up to >95% in selected cases). The catalyst **1b** with urea hydrogen peroxide adduct as an oxidant was recovered by precipitation with hexane and was reused up to four times with retention of enantioselectivity.

1. Introduction

Method development for the preparation of enantiomerically pure epoxides continues to be one of the most exciting fields of asymmetric catalysis [1–5]. Among several catalytic strategies for the synthesis of optically active epoxides, the asymmetric epoxidation of non-functionalized alkenes catalyzed by chiral Mn(III) salen complexes, initially developed by Katsuki and Jacobsen, are considered to be the most effective catalysts discovered in the last 25 years [2–4]. Although the classic chiral Mn(III) salen catalyst flaunts high enantioselectivity for the asymmetric epoxidation of Z and tri-substituted prochiral alkenes under homogeneous condition where catalyst recyclability is a major concern. To make Mn(III) salen catalyst recyclable, strategies like its immobilization on organic polymers [6], inorganic supports [6–14], encapsulation in zeolite cavities [15] and physical entrapment in siloxane membranes [16] have been reported. Very recently, Martinez et al. [17,18] reported multi-step synthesis of chiral Mn(III) salen complexes incorporated in a macrocycle bearing aliphatic polyether linker at 3,3′ positions of salicylidene moieties for enantioselective epoxidation of *cis*-olefins. However, 3,3′ positions of the salen unit are known to be very sensitive towards the enantioselectivity and any change from *t*-butyl group has invariably resulted in the loss of enantioselectivity [19].

In continuation of our earlier strategy [20–23] of making the catalyst recyclable by fine-tuning the solubility of the catalyst, we have developed a simple scheme for the synthesis of new macrocyclic recyclable chiral salen ligands **1a**′, **1b**′ (Scheme 1) linked through 5,5′ positions of salen unit in fewer steps. Mn(III) salen complexes **1a**, **1b**, derived from these ligands were used as catalysts for the enantioselective epoxidation of styrene (STR), indene (IND) and chromenes using oxidants e.g., sodium hypochlorite (NaOCl) and urea hydrogen peroxide (UHP) in the presence of pyridine *N*-oxide (PyNO) as an axial base at 0–5 °C. These catalysts were recovered by precipitation with hexane after each catalytic run and were reused several times with retention of enantioselectivity when UHP was used as an oxidant.

2. Experimental

The complexes **1a** and **1b** were prepared by the reaction of Mn(II) acetate with respective macrocyclic ligands **1a**′ and **1b**′, which were synthesized by the reaction of chiral diamine with a dialdehyde **3** as shown in Scheme 1 (experimental details are given in Supporting information).

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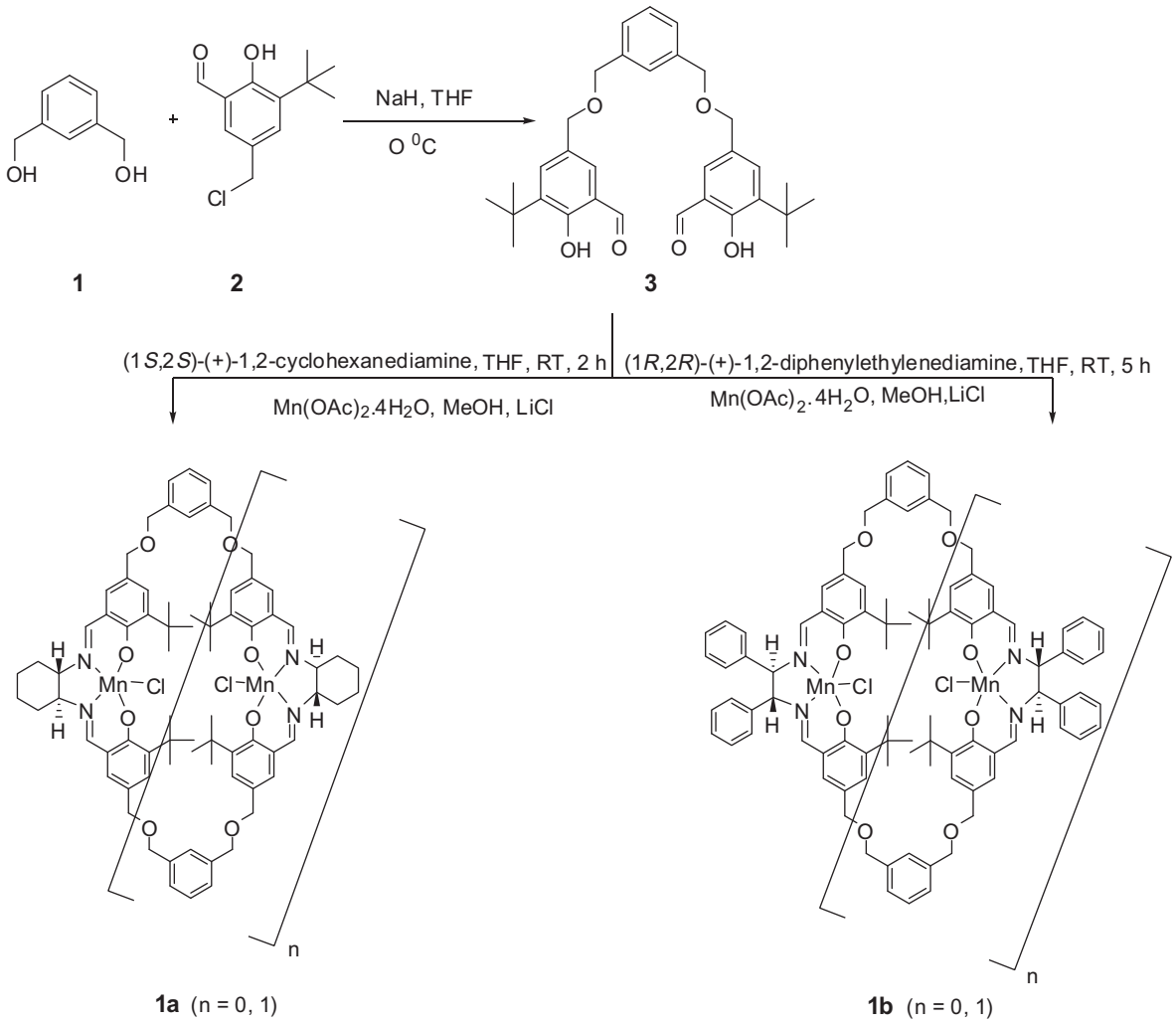


Scheme 1. Synthesis of chiral macrocyclic Mn(III) salen complexes **1a** and **1b**.

2.1. General procedure for enantioselective epoxidation of non-functionalized alkenes

2.1.1. NaOCl oxidant system

Enantioselective epoxidation reaction of different alkenes (0.625 mmol) viz. 2,2-dimethylchromene (CHR), 6-cyano-2,2-dimethylchromene (CN-CHR), 6-nitro-2,2-dimethylchromene (NO_2-CHR), 6-methoxy-2,2-dimethylchromene (MeO-CHR), spiro[cyclohexane-1,2-[2H][1]chromene] (Cy-CHR), IND, and STR was performed by using complex **1a**/**1b** (5 mol%) in CH_2Cl_2 (1 ml) in the presence of PyNO (11.5 mg, 0.12 mmol) as an axial base and buffered NaOCl (pH 11.3; 1.5 mmol added in four equal parts) as an oxidant at 0 °C. The epoxidation reaction was monitored by GC with *n-tri*decane (0.1 mmol) as GLC internal standard for product quantification. After the reaction, the product was extracted with CH_2Cl_2, washed with water, and dried over anhydrous sodium sulfate. The catalyst was separated from the product epoxide by precipitation with hexane (2 ml) and used as such for further catalytic runs. Epoxides were purified by flash chromatography through neutral alumina using ethyl acetate and hexane (9:1) as eluent.

2.1.2. UHP oxidant system

Enantioselective epoxidation of CHR, CN-CHR, NO_2-CHR, MeO-CHR, Cy-CHR, IND and STR (0.625 mmol) was performed by using **1a**/**1b** (5 mol%) as catalyst in 1:1 dichloromethane:methanol (1.0 ml) in the presence of PyNO (11.5 mg, 0.12 mmol) with UHP adduct (70 mg, 0.725 mmol, in six equal portions) as an oxidant at 3 °C, and the reaction was monitored on GC. After completion of the reaction, the solvent was removed and the residue was extracted with CH_2Cl_2, washed with water, and dried over anhydrous sodium sulfate. The catalyst was separated from the product epoxide by precipitation with hexane (2 ml) and was used as such for further catalytic runs. Epoxides were purified by flash chromatography through neutral alumina using ethyl acetate and hexane (9:1) as eluent.

3. Results and discussion

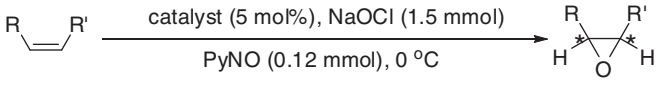
The synthesis of catalysts **1a** and **1b** is presented in Scheme 1. The reaction of dimethanol benzene **1** with aldehyde **2** yielded the desired dialdehyde **3** in rather low yield (maximum 32%) after chromatographic purification because of the presence of free OH group in aldehyde **2** resulting in the formation of side products. The macrocyclic chiral Schiff base ligands **1a**′ and **1b**′ were synthesized by reacting stoichiometric amount of dialdehyde **3** with corresponding diamines. Remarkably, the dialdehyde **3** reacted completely (within 2 h) with (*1S,2S*)-(+)-1,2-diaminocyclohexane in THF to give mostly monomeric macrocyclic ligand **1a**′ ($n = 0$). Whereas the reaction of dialdehyde **3** with (*1R,2R*)-(+)-1,2-diphenylethylenediamine in THF was tricky as longer reaction time (>5 h) resulted in the formation of polymeric compound. After several attempts on its synthesis in various solvents, it was realized that at

about 5-h reaction time in THF, formation of the product **1b′** is optimum (~75%). The ^{1}H NMR and LCMS data for ligand **1b′** show the presence of monomer (n = 0), dimer (n = 1) and trimeric species in 80%, 16% and 4%, abundance, respectively. These mono-, di- and trimeric salen species were separated by neutral alumina column chromatography with benzene and acetone (7:3) as eluent. Absence of aldehyde peak in NMR indicates that all the three species were cyclic. This mixture of species was used as such for making the chiral macrocyclic Mn(III) salen complex, as complexes of above separated ligands gave nearly similar performance. The monomeric **1a′** gave single crystals suitable for X-ray structure determination from acetone in 2–3 days, which further established its cyclic structure (Fig. S1) [24]. The ligand **1a′**/**1b′** on reaction with $Mn(OAc)_2{\cdot}4H_2O$ and subsequent air oxidation yielded the desired complex **1a**/**1b** in quantitative yield. Numerous attempts to get single crystals of complexes **1a** and **1b** failed; hence, we explored the probable structure of **1a** using energy minimization option (Fig. S2) in Material Studio (version 4.2). The crystallographic data of ligand **1a′** were used for simulating the structure of complex **1a**, which lacked C_2-symmetry (or distorted C_2-symmetry) as against stepped conformation [25] of the Jacobsen's Mn(III) salen complex. The cause of this distortion is due to the shorter length of the linker group. Both the complexes **1a** and **1b** (5 mol%) were used to catalyze the enantioselective epoxidation of STR as a representative substrate using NaOCl as an oxidant in the presence of PyNO as an axial base, and the results are given in Table S1. Excellent yield (>99%) of styrene oxide was achieved in 3 h with both the catalyst. However, the ee of styrene oxide was superior with catalyst **1b** (Table S1, entry 17). Epoxidation of STR was conducted with 2.5–10 mol% catalyst loading and the results indicated that 5 mol% (Table S1, entries 1–3, 17–19) is optimum at 0 °C. Epoxidation of STR over a temperature range of −5 to 20 °C at 5 mol% catalyst loading concluded that 0 °C is optimum reaction temperature (Table S1, entries 5–7, 21–23). Having optimized catalyst loading and reaction temperature next, we optimized reaction medium, as solvent is known to play a crucial role in the enantioselective epoxidation of non-functionalized alkenes [17,22]. In view of this, the effect of solvents viz. CH_2Cl_2, dichloroethane, $CHCl_3$ and mixture of (CH_2Cl_2 + CH_3CN) on the epoxidation of STR as representative substrate was carried with catalysts **1a** and **1b** under identical reaction conditions (Table S1, entries 1, 8–10, 17, 24–26). Among these, CH_2Cl_2 (Table S1, entries 1 and 17) was found to be the best solvent. It is reported in the literature [20,22] that pyridine *N*-oxide derivatives greatly influence catalyst turnover and enantioselectivity in Mn(III) salen catalyzed epoxidation of alkenes with NaOCl. According to these studies, *N*-oxides prevent the formation of catalytically inactive Mn–O–Mn species [26] as well as improve the transportation of HOCl from aqueous phase to the organic phase. Hydrophobic *N*-oxides e.g., 4-phenylpyridine *N*-oxide (4-PPyNO) and 4-(3-phenylpropyl) pyridine *N*-oxide (4-PPPyNO) have been reported to stabilize the reactive Mn=O intermediate [27]. In our optimized condition for the epoxidation of styrene, the use of 4-PPyNO and 4-PPPyNO as axial bases with catalysts **1a** and **1b** (Table S1, entries 11, 12, 27, 28) had no obvious advantage over the simple and inexpensive PyNO. This finding is in contrast to the results obtained with the use of Mn(III) salen complex [27] but is in line with the use of built-in phase transfer Mn(III) salen complexes reported by us [28,29]. A control experiment for the epoxidation of STR conducted without PyNO gave only 40–48% styrene oxide in 3 h with low enantioselectivity (Table S1, ee 18–22%; entries 13, 29), demonstrated the positive role of PyNO as an axial base.

We also explored the oxidant-UHP in the epoxidation of STR with catalysts **1a** and **1b** in the presence of different *N*-oxides viz., PyNO, 4-PPyNO, and 4-PPPyNO at 3 °C in CH_2Cl_2:CH_3OH 1:1 (Table S1, entries 14–16, 30–32). Excellent conversion (>99%) to styrene oxide was achieved in 5–6 h with both the catalysts, however, catalyst **1b** imparted better ee (~59%) when compared to the catalyst **1a** (ee, ~41%), which fared better with UHP than NaOCl.

The optimized epoxidation condition (as per entries 1 and 2; Table 1) was applied for other non-functionalized alkenes, to see the general applicability of catalysts **1a** and **1b** using PyNO as an axial base with NaOCl as an oxidant. Excellent conversion to epoxides (>99%) and ee (Table 1) was observed for all the substrates (entries 1–14) in 3–8 h at 0 °C. As far as enantioselectivity is in concern, the catalyst **1b** worked much better for STR (ee, 59%) and IND (ee, 91%) than the catalyst **1a**, which gave ee 33% and 75%, respectively. However, in the case of chromenes both the catalysts imparted

Table 1
Asymmetric epoxidation of alkenes catalyzed by **1a** and **1b** with NaOCl as an oxidant.



Entry[a]	Catalyst	Substrate	% Yield[b]	Time (h)	ee (%)	Configuration	TOF × 10^{-3} s^{-1}[f]
1	**1a**	STR	>99	3	33[c]	*S*	1.83
2	**1b**		>99	3	59[c]	*R*	1.83
3	**1a**	IND	>99	4.5	75[d]	*1S,2R*	1.22
4	**1b**			4	91[d]	*1R,2S*	1.38
5	**1a**	Cy-CHR	>99	6	86[e]	*3S,4S*	0.92
6	**1b**		>99	5	84[e]	*3R,4R*	1.10
7	**1a**	CHR	>99	6	93[e]	*3S,4S*	0.92
8	**1b**		>99	5	92[e]	*3R,4R*	1.10
9	**1a**	MeO-CHR	>99	8	73[e]	*3S,4S*	0.69
10	**1b**		>99	7	77[e]	*3R,4R*	0.79
11	**1a**	NO_2-CHR	>99	6	90[e]	*3S,4S*	0.92
12	**1b**		>99	5	91[e]	*3R,4R*	1.10
13	**1a**	CN-CHR	>99	6	93[e]	*3S,4S*	0.92
14	**1b**		>99	5	95[e]	*3R,4R*	1.10
15	**Jacobson's catalyst**	CN-CHR	60	9	92	*3S,4S*	0.37
16	**Katsuki's catalyst**	CHR	75	2	99	*3S,4S*	4.17

[a] Reaction condition: catalyst (5 mol% in 1 ml CH_2Cl_2), substrate (0.625 mmol), pyridine *N*-oxide (0.12 mmol), NaOCl (1.5 mmol).
[b] Determined on GC.
[c] Chiral capillary column GTA-type.
[d] Chiral HPLC OB column.
[e] Chiral HPLC OD column.
[f] Turn over frequency is calculated by the expression [product]/[catalyst] × time (s^{-1}).

Table 2
Epoxidation of alkenes catalyzed by **1a** and **1b** with UHP adduct as an oxidant.

Entry[a]	Catalyst	Substrate	% Yield[c]	Time (h)	ee (%)	Configuration	TOF × 10^{-3} s^{-1}[g]
1	**1a**	STR	>99	6	41[d]	*S*	0.91
2[b]	**1b**		>99	8	59[d]	*R*	0.69
3[h,b]	**1b**		>92	8	59[d]	*R*	0.63
4[i,b]	**1b**		>80	8	58[d]	*R*	0.55
5[j,b]	**1b**		>75	8	59[d]	*R*	0.52
6	**1a**	IND	93	8	93[e]	*1S,2R*	0.65
7	**1b**		99	10	62[e]	*1R,2S*	0.55
8	**1a**	Cy-CHR	95	22	84[f]	*3S,4S*	0.24
9	**1b**		90	22	86[f]	*3R,4R*	0.23
10	**1a**	CHR	>99	22	90[f]	*3S,4S*	0.25
11	**1b**		>99	22	93[f]	*3R,4R*	0.25
12	**1a**	MeO-CHR	90	8	89[f]	*3S,4S*	0.62
13	**1b**		98	10	93[f]	*3R,4R*	0.54
14	**1a**	NO_2-CHR	48	22	91[f]	*3S,4S*	0.12
15	**1b**		96	22	92[f]	*3R,4R*	0.24
16	**1a**	CN-CHR	44	24	91[f]	*3S,4S*	0.10
17	**1b**		35	24	80[f]	*3R,4R*	0.08

[a] Reaction condition: catalyst (5 mol% in 1 ml CH_2Cl_2), substrate (0.625 mmol), pyridine *N*-oxide (0.12 mmol), UHP (1.5 eq. with respect to substrate) at 3 °C.
[b] Reaction condition: catalyst (5 mol% in 2 ml CH_2Cl_2), substrate (1.15 mmol), pyridine *N*-oxide (0.23 mmol), UHP (1.5 eq. with respect to substrate) at 3 °C.
[c] Determined on GC.
[d] Chiral capillary column GTA-type.
[e] Chiral HPLC OB column.
[f] Chiral HPLC OD column.
[g] Turn over frequency is calculated by the expression [product]/[catalyst] × time (s^{-1}).
[h] 2nd run with the recovered catalyst.
[i] 3rd run with the recovered catalyst.
[j] 4th run with the recovered catalyst.

nearly similar enantioselectivites (Table 1). Nevertheless, ee with substrates CHR and chromenes having electron-withdrawing groups (NO_2-CHR and CN-CHR) (entries 11–14) were better than Cy-CHR and electron-rich MeO-CHR (entries 5, 6, 9, 10). For comparison, epoxidation of CN-CHR was conducted under the above optimized reaction condition with the monomeric Jacobsen Mn(III) salen complex in the presence of PyNO, which gave the corresponding epoxide in 60% yield with 92% ee in 9 h (entry 15, Table 1), while complexes **1a** and **1b** gave >99% epoxide yield with 93–95% ee in 5–6 h (entries 13 and 14). Further, Katsuki Mn(III) salen catalyst gave 75% conversion to 2,2-dimethylchromene oxide with 99% ee in presence of 4-PPyNO as an axial base [2], while catalysts **1a** and **1b** gave better conversion (>99%) to epoxide with ~93% ee. Additionally, catalysts **1a** and **1b** have the advantage of recyclability over classical Jacobsen and Katsuki catalysts.

Enantioselective epoxidation of above-mentioned substrates were also conducted with catalysts **1a** and **1b** using UHP as an oxidant in the presence of PyNO as an axial base at 3 °C, and results are presented in Table 2. Turn over frequency (TOF) values calculated for these reactions indicate that UHP took longer reaction time than NaOCl to give high conversions to their respective epoxides (entries 1–17, Table 2). Nevertheless, styrene, indene and electron-rich chromene (MeO-CHR) reacted readily (entries 1–2, 6–7, 12–13, Table 2) to give respective epoxides in 41–93% ee. On the other hand, Cy-CHR and CHR (entries 8–11) and electron-depleted chromenes like CN-CR and NO_2-CHR reacted sluggishly (22–24 h; entries 14–17) but the enantioselectivity was reasonably high (ee, 84–93%). In all catalytic runs, both the catalysts **1a** and **1b**, irrespective of the oxidant used (NaOCl and UHP) the configuration of the dominant enantiomer of the product was the same as that of the catalyst. These results indicate that transition species (via $L^*Mn=O$, L^* = chiral macrocyclic ligand) with both oxidants are common during oxygen atom transfer to substrate [22].

For reusability experiments, epoxidation of STR with **1b** was conducted under the optimized reaction condition using UHP as an oxidant. After first catalytic run (entry 2, Table 2), the macrocyclic complex was precipitated from the reaction mixture by the addition of hexane. The precipitated complex was washed with hexane, dried in vacuum, and used for the subsequent catalytic run without further purification. The enantioselectivity of the recovered complex did not change (entries 2–5, Table 2) during four reuse experiments. However, a decrease in epoxide yield was observed, due to minor physical loss of the catalyst during its recovery process. The same reaction when conducted with NaOCl, the recovered catalyst turned light brown in color from original dark brown possibly due to its oxidative degradation hence failed to further catalyze the epoxidation reaction. An indication of this is apparent in the IR and UV–Vis data of recovered catalyst, which is correlated for the degradation of imine and phenoxide moieties in the catalyst (Figs. S3 and S4).

4. Conclusions

In conclusion, we have demonstrated a simple synthesis method for the preparation of two new Mn(III) salen complexes **1a** and **1b** incorporated in macrocyclic framework having benzylic ether bridge linked to 5,5′ positions of the salicylidene moieties. These complexes worked well as catalyst in the enantioselective epoxidation of non-functionalized alkenes with NaOCl and UHP as oxidants in the presence of inexpensive PyNO as an axial base at 0–3 °C. The catalyst **1b** could be recycled couple of times with the retention of ee for the epoxidation of styrene with UHP as an oxidant, however, with NaOCl as an oxidant, catalyst degraded after one cycle.

Acknowledgments

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.jcat.2010.10.002.

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