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Kinetic resolution of terminal epoxides with phenols promoted by heterometallic Co–Al and Co–Ga salen complexes

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The binuclear chiral (salen) Co complexes bearing Lewis acid salt (Al and Ga) and 4-nitrobenzene sulfonic acid (NBS) catalyze the enantioselective ring opening of terminal epoxides with phenol derivatives. The easily prepared complexes exhibited very high catalytic reactivity and enantioselectivity for the asymmetric ring opening of terminal epoxides with phenol and consequently provide enantiomerically enriched corresponding α-aryloxy alcohols (up to $>99\%$ ee). The two unit of salen complexes combined by Lewis acid exhibited co-operative catalysis during the ring opening reaction.

KEY WORDS: heterometallic chiral complex; kinetic resolution; α-aryloxy alcohols; terminal epoxides.

1. Introduction

The area of kinetic resolution has grown tremendously during the last few years [1–6]. Remarkable progress had been made in the use of chiral catalysts to synthesize valuable enantioenriched intermediates of the academic and pharmaceutical interests [7–12]. A wide range of synthesis and application of chiral catalysts as dimeric [13], polymeric [14], oligomeric [15,16], dendrimeric [17] and supported [8,14] forms have been reported as a practical and commercial advantage. The oligomeric (salen) Co catalyst exhibited very high reactivity and enantioselectivity for the terminal epoxides ring opening with phenols *via* a kinetic resolution process, however multi-step synthesis was required for such oligomeric cobalt–salen catalysts [15,16].

Pursuant to our own efforts directed towards the designing of the di- and multimeric chiral (salen) Co catalysts, dinuclear chiral (salen) Co–MX_3 catalyst has shown the excellent activity in the enantioselective kinetic resolution of terminal epoxides with H_2O and asymmetric cyclization reaction in our previous work [18–21]. As a continuation of our research we tried to extend this methodology to direct synthesis of optically active α-aryloxy alcohols by using mainly heterometallic (salen) Co–$AlCl_3$/$GaCl_3$ complex (Scheme 1).

Herein, we report the highly effective methods for the asymmetric ring opening reaction catalyzed by the new chiral heterometallic (salen) Co complexes as a monomer or linked dimmer type. Especially, the oxidation of chiral salen complexes from Co^{II} to Co^{III} with 4-nitrobenzenesulfonic acid (NBS) in air has displayed higher reactivity and selectivity through the mechanism involving cooperative bimetallic catalysis [16].

In this work, the addition of phenol derivatives to epicholorohydrin (ECH) was examined mainly as a model reaction to test the efficiency of new chiral salens with different structures. The salen complex having C_3–^tBu group in the structure has never been used as a catalyst in the ring opening of terminal epoxides to obtain the variety of chiral intermediates, particularly in the reaction using phenol derivatives as a nucleophile.

2. Experimental

2.1. Materials and methods

All ^1H-NMR and ^{13}C-NMR were recorded using 400 MHz FT-NMR spectrometer (VARIAN UNITYINOVA400) at ambient temperature. Optical rotation measurements were conducted using a JASCO DIP 370 digital polarimeter. Gas chromatographic analyses were performed on Hewlett-Packard 5890 Series II instrument equipped with a FID detector using a chiral column (CHIRALDEX G-TA and A-TA, 20 m × 0.25 mm id (Astec) and a HP 3396 integrator with HP Chem Station software for data analysis. Chiral HPLC analyses were performed on a YOUNGLIN instrument using a Chiralcel® OD column (24 cm × 0.46 cm i.d.; Chiral Technologies, Inc.) and Regis (*S*,*S*) Whelk-O1 at 254 nm. UV spectra were recorded on UV–Vis spectrophotometer (Optizen 2120 UV) interfaced with a PC using Optizen view 3.1 software for data analysis. The IR spectra were measured with a PERKIN-ELMER Spectrum 2000 Explorer. ESCA spectra were obtained with SIGMA PROBE

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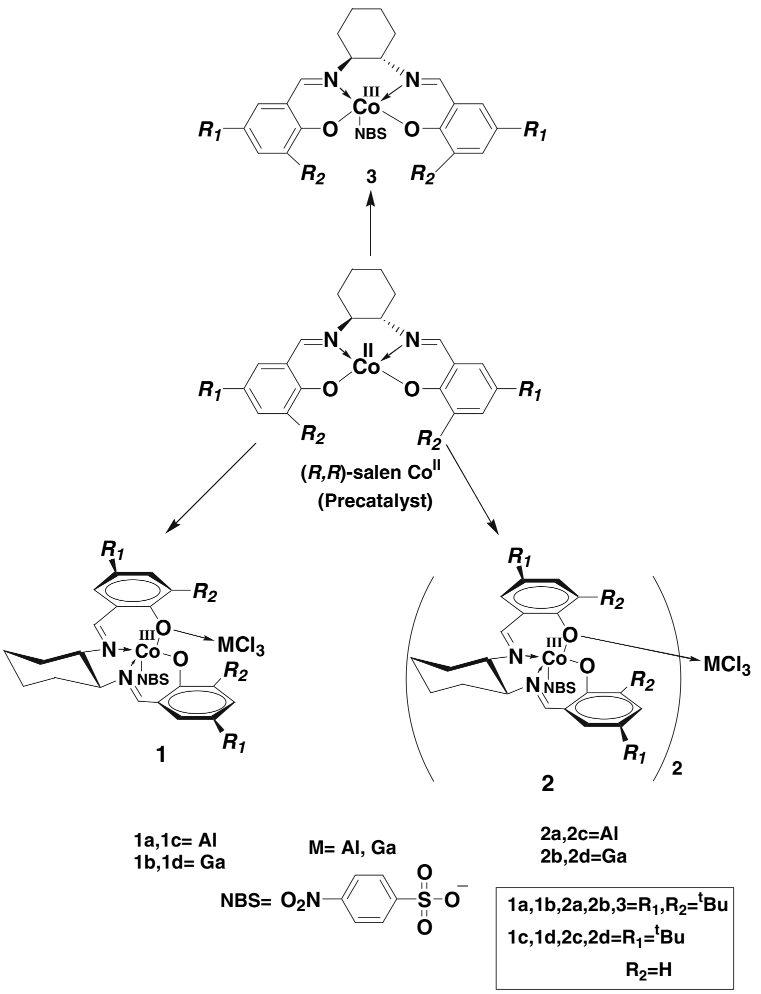


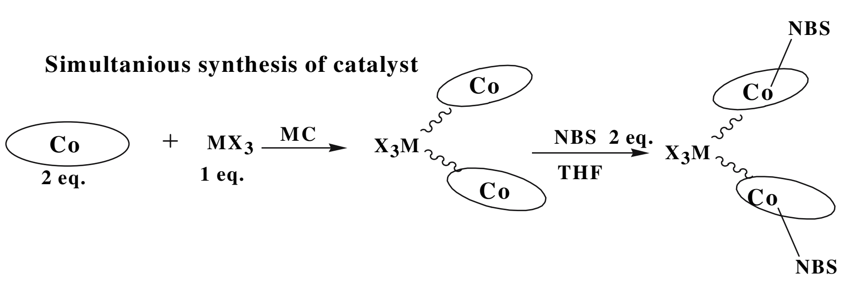
Scheme 1. Heterometallic chiral (salen) Co–MCl_3 complexes.

(Thermo VG, UK) Spectrometer using Mg Kα radiation as an excitation source ($h\ \nu$ = 1253.6 eV). Binding energy was compared with a reference of cobalt salt such as $Co^{II}Cl_2$, $Co^{III}Cl_3$, Co^{II} salen and Jacobsen catalyst from Aldrich. All reagents and solvents were purchased from Aldrich, Fluka and TCI and they were used without further purification.

2.2. Catalyst synthesis

Two synthetic methods were applied to obtain dimeric catalysts. As one method, the precatalyst **(R,R)**-salen Co^{II} and Lewis acid (2:1) was dissolved in methylene chloride (MC) simultaneously at ambient temperature and stirred in open atmosphere for 3 h. The color of the reaction mass changed from red to olive green. The solvent was evaporated up to dryness under reduced pressure to offer the dark brown solid powder. Then the catalyst was treated by NBS (2 equivalents based on Co unit) under stirring in THF for 2 h. The solvent was evaporated and catalyst was purified by recrystallization in MC.

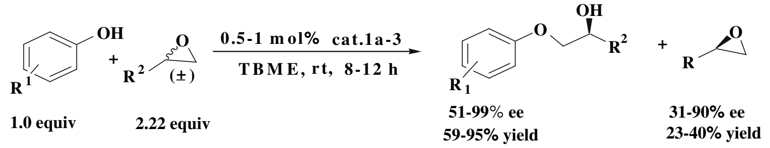
In another method, the **(R,R)**-salen Co^{II} and Lewis acid (1:1) was dissolved in MC simultaneously at



ambient temperature and stirred in the open atmosphere for 3 h. The solvent was evaporated up to dryness under reduced pressure and the residue was collected. After solvent evaporation the residue was dissolved in MC together with 1 equivalent (**R,R**)-salen Co^{II} and then stirred for 2 h. The catalyst was collected after solvent evaporation up to dryness under reduced pressure. Then the catalyst was treated by NBS (2 equivalents based on Co unit) under stirring in THF for 2 h. The solvent was evaporated and catalyst was purified by recrystallization in MC.

The catalyst **3** was synthesized using a mixture of (*R,R*)-*N,N′*-bis (3,5-di-*tert*-butylsalicylidene)-1,2-cyclohexane diamino cobalt (II) and 4-Nitrobenzene sulfonic acid in THF was stirred at room temperature in air for 1 h. The solvent was removed under reduced pressure

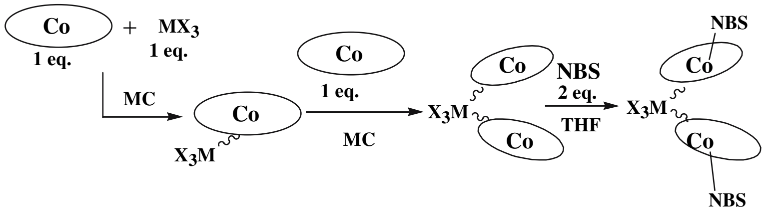
Table 1
Asymmetric ring opening of terminal epoxides with phenols catalyzed by heterometallic complexes



Entry	R^1	R^2	Catalyst	Co (mol%)[a]	Time (h)	Yield (%)[b]	ee (%)[c]	Yield (%)[d]	ee (%)[e]
1	H	CH_2Cl	3	1.0	9	84	57	30	41
2	H	CH_2Cl	1a	1.0	9	86	88	33	53
3	H	CH_2Cl	1b	1.0	9	82	85	23	65
4	H	CH_2Cl	1c	1.0	12	88	96	29	68
5	H	CH_2Cl	1d	1.0	12	86	94	31	62
6	H	CH_2Cl	2a	0.5	9	91	96	31	75
7	H	CH_2Cl	2b	0.5	9	88	93	40	66
8	H	CH_2Cl	2c	0.5	12	95	95	28	75
9	H	CH_2Cl	2d	0.5	12	88	93	35	69
10	H	CH_3	2a	0.5	11	93	95	29	53
11	H	C_2H_5	2a	0.5	10	86	92	31	71
12	H	CH_3–O–CH_2	2a	0.5	12	91	93	31	49
13	3-Cl	CH_2Cl	3	1.0	11	59	61	35	32
14	3-Cl	CH_2Cl	1a	1.0	11	88	92	38	47
15	3-Cl	CH_2Cl	1b	1.0	11	84	90	26	55
16	3-Cl	CH_2Cl	1c	1.0	12	86	95	33	65
17	3-Cl	CH_2Cl	1d	1.0	12	86	95	27	56
18	3-Cl	CH_2Cl	2a	0.5	9	95	98	35	68
19	3-Cl	CH_2Cl	2b	0.5	11	93	94	38	67
20	3-Cl	CH_2Cl	2c	0.5	12	95	96	31	75
21	3-Cl	CH_2Cl	2d	0.5	12	91	95	28	79
22	3-Cl	CH_3	2a	0.5	10	93	95	25	85
23	3-Cl	C_6H_5–O–CH_2	2a	0.5	12	84	96	29	66
24	3-Cl	CH_3–O–CH_2	2a	0.5	11	88	95	23	76
25	3-CH_3	CH_2Cl	3	1.0	8	62	51	25	39
26	3-CH_3	CH_2Cl	1a	1.0	8	86	91	31	70
27	3-CH_3	CH_2Cl	1b	1.0	8	82	89	30	65
28	3-CH_3	CH_2Cl	1c	1.0	12	86	95	28	71
29	3-CH_3	CH_2Cl	1d	1.0	12	86	95	24	63
30	3-CH_3	CH_2Cl	2a	0.5	10	91	99	33	90
31	3-CH_3	CH_2Cl	2b	0.5	10	91	96	32	89
32	3-CH_3	CH_2Cl	2c	0.5	12	95	96	29	75
33	3-CH_3	CH_2Cl	2d	0.5	12	91	95	33	67
34	3-CH_3	C_2H_5	2a	0.5	9	95	95	25	55
35	3-CH_3	CH_3–O–CH_2	2a	0.5	11	88	93	28	60

[a] Catalyst loading on a per Co basis relative to racemic epoxide.
[b] Isolated yield on the basis of phenol.
[c] ee % of product was determined by chiral HPLC/chiral GC. Products were characterized by ^{1}H- and ^{13}C-NMR and found to be similar to reported [14,15].
[d] GC yield.
[e] ee % of recovered epoxides was determined by chiral HPLC/chiral GC.

Step-by-step synthesis of catalyst



and the residue was purified by crystallization in MC and filtration gave brown color powder. The oxidation state of Co^{III} was checked by ESCA (Electron Spectroscopy for Chemical Analysis).

2.3. General procedure for kinetic resolution

In a representative reaction of epichlorohydrin (ECH) and phenol (table 1, entry 6), the catalyst **(R,R)-2a** (867 mg, 0.05 mmol, 0.5 mol%,), *tert*-butyl methyl ether (TBME) and (±)-ECH (0.93 g, 10 mmol) were charged in an oven dried 25 mL flask and the reaction mixture was stirred in open atmosphere at ambient temperature. Phenol (0.42 g, 4.5 mmol 0.45 equiv.) was added after complete dissolution of the catalyst. The resultant solution was stirred for 9 h and monitored by chiral GC and HPLC. The purification of the products was performed by flash column chromatography. The products were identified/characterized by IR, ^{1}H-NMR, ^{13}C-NMR and found to be similar to that reported.

2.4. Procedure for kinetic measurements

In a representative kinetic reaction, a 20-mL vial was charged with a stir bar and 0.2 mol (340 mg) catalyst **2a**. The catalysts were dissolved in TBME (5.00 mL). The racemic epichlorohydrin (ECH) (3.70 g, 40 mmol) and bromobenzene (250 μL; as an internal standard) were added to the dark brown solution. After the reaction mixture had been stirred for 30 min at 20 °C, 3-chlorophenol (2.30 g) was added. The rate of the reaction was monitored by GC analysis of 20 μL aliquots withdrawn periodically from the reaction mixture.

2.5. Non-linear effects studies

Catalyst **(R,R)-2a** (86.7 mg, 5.0×10^{-5} M) was dissolved in a solution of ECH (4.00 M) in TBME (1.00 mL) and stirred for 5 h. Relative concentrations of the catalysts were confirmed by diluting aliquots (20 μL × 3) with THF and measuring the characteristics absorbance at 365 nm by UV-Vis spectrophotometer. Reactions were carried out by mixing the solutions of enantiomeric catalysts in appropriate ratios and then adding a solution of 3-chlorophenol in TBME (1.0 M) such that $[ECH]_0 = 1.0$ M, [3-chlorophenol] = 0.45 and [catalyst] = 0.5 M. All reactions approached completion within 12 h. The ee% of the ring-opened product was determined by chiral HPLC using Regis Whelk-O1 column at 254 nm.

3. Results and discussion

The results obtained in the asymmetric ring opening of terminal epoxides with phenol as nucleophile catalyzed by (salen) Co–$AlCl_3$ and (salen) Co–$GaCl_3$ are summarized in table 1. Both the electron-poor or rich phenols reacted with ECH enantioselectively to provide the corresponding α-aryloxy alcohols in good yield and high ee%. As can be seen from table 1, a series of terminal epoxides reacted with a various phenol derivatives to offer corresponding optically enriched alcohols (92–99%). These reactions were regioselective and occurred in good yield when low loading (0.5 mol%) of catalyst was employed. The catalyst **2a--2d** (entry 6–12, 18–24 and 30–35) containing $AlCl_3$ and $GaCl_3$ exhibited higher ee's (93–99%) than catalyst **3** (entry 1, 13 and 25). The synthesis procedure for catalyst **3** was described in catalyst synthesis section. Additionally, the monomer catalyst **1a--1d** (entry 2–5, 14–17 and 26–29) exhibits the similar enantioselectivity like dimeric catalyst whereas it was examined that the dimer catalyst displayed superior activity with the half amount of catalyst as compared to monomer catalyst.

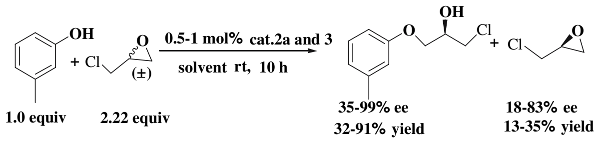
In table 1 it is evident that the catalyst **2a** exhibits the highest activity and selectivity among others. The order of reactivity of catalysts was observed as follows; **2a--2d** > **1a--1d** > 3. The kinetic resolution of terminal epoxides with phenols proved to be a useful reaction, requiring only 0.5 mol% catalyst (entry 6, 10, 11 and 12). 3-Chlorophenol acts as an excellent nucleophile for the kinetic resolutions of ECH, phenyl glycidyl ether, propylene oxide and butylene oxide (entry 18, 22, 23 and 25). Overall, the phenols with wide range of electronic properties participated in the ring opening reaction with good yield and ee's [14,15].

It was found that the solvent plays crucial role in this reaction. In the typical example during the reaction of ECH with 3-methyl phenol, the effect of polar and nonpolar solvents were examined and nonpolar solvents such as TBME was found to be the most effective as in table 2.

The identity of the counter anion and the oxidation state of cobalt for this new chiral salen complex proved to be an important in this work, displaying the superior activity with the NBS anion. As Co^{II} salen was oxidized

Table 2

Effect of solvent on the kinetic resolution of epichlorohydrin with 3-methyl phenol



Entry	Solvent	Catalyst	Co (mol%)[a]	Time (h)	Yield (%)[b]	ee (%)[c]
1	TBME	2a	0.5	10	91	99
2	CH_3CN	2a	0.5	10	82	88
3	CH_2Cl_2	2a	0.5	10	32	81
4	1,4-Dioxane	2a	0.5	10	59	87
5	THF	2a	0.5	10	59	87
6	n-Hexane	2a	0.5	10	51	80
7	TBME	3	1.0	10	62	51
8	CH_3CN	3	1.0	10	44	43
9	CH_2Cl_2	3	1.0	10	39	39
10	1,4-Dioxane	3	1.0	10	42	43
11	THF	3	1.0	10	42	43
12	*n*-Hexane	3	1.0	10	37	35

[a] Catalyst loading on a per Co basis relative to racemic epoxide.
[b] Isolated yield on the basis of phenol.
[c] ee % was determined by chiral HPLC or chiral GC.

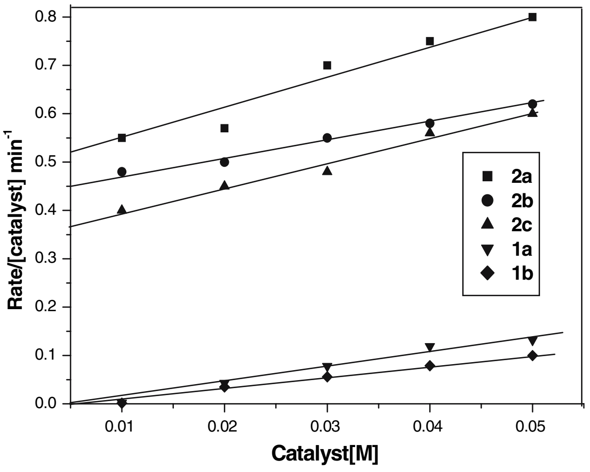


Figure 1. Initial rate kinetic study of 3-chlorophenol with ECH catalyzed by **1a**, **1b**, **2a**, **2b** and **2c**.

to Co^{III} by treatment of NBS in air, it became very active and enantioselective. Camphorsulfonic acid or *p*-toluenesulfonic acid treated chiral salen catalyst was less enantioselective in this reaction [16]. The heterometallic complex of chiral (salen) Co–$AlCl_3$ showed more active than that of chiral (salen) Co–$GaCl_3$ possibly due to the higher Lewis acidity.

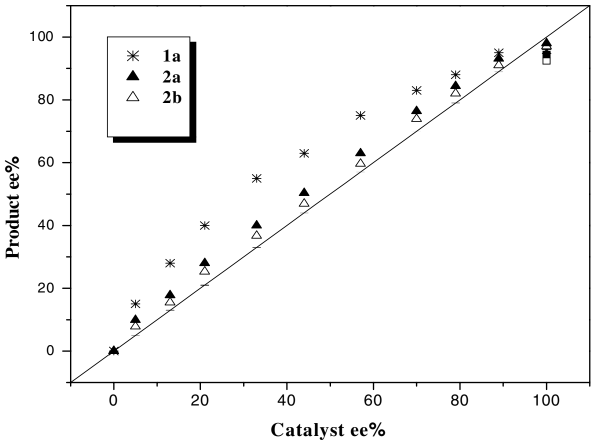


Figure 2. Nonlinear effects of the 3-chlorophenol with ECH catalyzed by **1a**, **2a** and **2b**. Catalyst amount for **1a** = 1.0 mol% and **2a**, **2b** = 0.5 mol% based on [Co].

Considering the two-term rate equation involving both intra- and intermolecular components of product carried out kinetic studies of the reaction of racemic ECH with 3-chlorophenol

$$\text{rate} \propto K_{\text{intra}}[\text{catalyst}] + K_{\text{inter}}[\text{catalyst}]^2$$

Plots of rate/[catalyst] vs. [catalyst] should be linear with slopes equal to $K_{\text{inter}}$and y-intercepts corresponding to K_{intra} [13,17].

Analysis of such plots with rate data obtained with dimmer catalyst **2a--2d** depicted linear correlation with positive slope and nonzero y-intercepts, consistent with both inter- and intramolecular pathways in the ring opening of epoxides with phenol as shown in figure 1. Similar analysis of rate data obtained with monomeric catalyst **1a** and **1b** revealed zero y-intercepts, reflecting

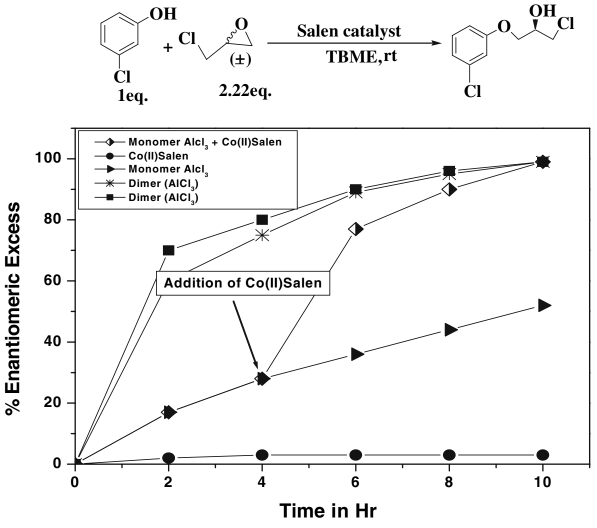
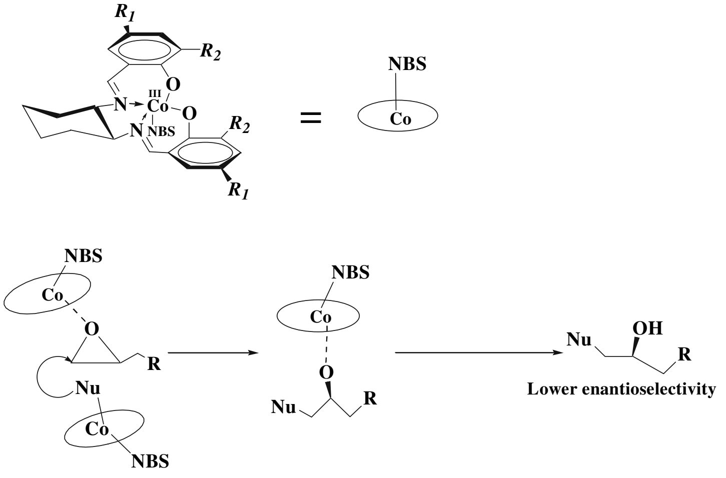
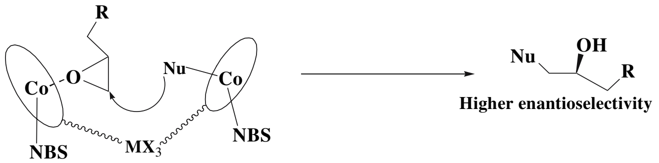


Figure 3. Effect of (salen) Co^{II} addition to the catalyst during the reaction: (a) –■– Using dimmer ($AlCl_3$) synthesized by simultaneous method explained in experimental section. (b) –✳– Using dimmer ($AlCl_3$) synthesized through step-by-step method.



Cooperative Catalysis in assymetric ring opening catalysed by monomeric metal salen complex



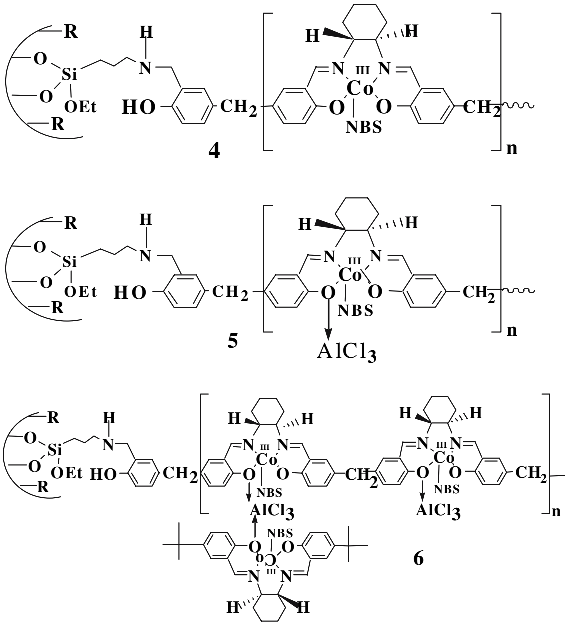
Cooperative Catalysis in assymetric ring opening catalysed by dimeric metal salen complex

Scheme 2. Possible working model for the kinetic resolution of terminal epoxides with phenols catalyzed by Co–Al and Co–Ga complexes.

absence of first-order pathway of these complexes. With dimer catalyst **2a**, a maximum value of intra- and intermolecular rate constant was obtained showing the highest reactivity and enantioselectivity. Thus, the dimer catalyst provides bimetallic cooperative action to reinforce the reactivity and selectivity relative to monomeric catalyst.

A further indication of the participation of intramolecular bimetallic pathway for the dimeric catalysts was provided by examination of the dependence of the reaction enantioselectivity on the enantiomeric purity of the catalysts. The catalyst **1a** displays a distinct positive nonlinear relationship between the enantiomeric excess of the product and the enantiomeric purity of the catalyst. The catalysts **2a** and **2b** revealed linear relationships with participation of first and second-order pathway in figure 2. This result suggests intramolecular cooperative catalysis in the ring opening of terminal epoxides with phenol.

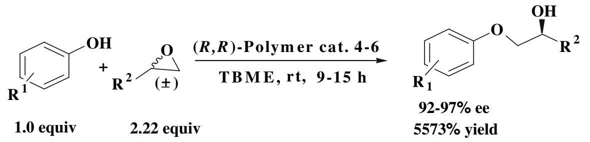
Figure 3 shows the effect of catalyst structure such as monomeric and dimeric form on the enantioselectivity in the ECH ring opening reaction with 3-chlrophenol. The (**R,R**)-salen Co^{II} itself has no activity in the kinetic resolution of epoxides. The catalyst having monomeric structure gave lower activity than dimeric one. The dimer catalyst synthesized by the simultaneous treatment between the (*R,R*) salen Co^{II} and metal salt (in the mole ratio of 2:1) has offered remarkably enhanced reactivity and significant higher enantioselectivity as compared to monomer catalyst as can be seen in figure 3. However, the catalyst prepared by the sequential treatment of monomeric salen with extra equivalent (*R,R*)-salen Co^{II} has shown the almost same catalytic activity as the catalyst which was synthesized



Scheme 3. Polymer salen catalyst complexes.

Table 3

Asymmetric ring opening of ECH with phenol derivatives catalyzed by polymer salen complexes catalysts



Entry	R^1	R^2	Catalyst[a]	Time (h)	Yield (%)[b]	ee (%)[c]
1	H	CH_2Cl	4	15	68	94
2	H	CH_2Cl	5	12	68	96
3	H	CH_2Cl	6	9	73	97
4	3-CH_3	CH_2Cl	4	15	66	92
5	3-CH_3	CH_2Cl	5	12	68	96
6	3-CH_3	CH_2Cl	6	9	68	96
7	H	CH_2Cl	6 cycle 1	15	64	97
8	H	CH_2Cl	6 cycle 2	12	55	95
9	H	CH_2Cl	6 cycle 3	9	64	97

[a] The catalyst structure is shown in scheme 3.

[b] The isolated yield on the basis of phenol.

[c] ee % was determined by chiral HPLC or chiral GC.

by simultaneous treatment between Co^{II} ligand and $AlCl_3$ in the mole ratio of 2:1.

In addition, when the inactive Co^{II} salen ligand was added into the reaction system in which the ring opening reaction was started in the presence of monomeric salen catalyst initially, the accelerated reaction rates and high enantioselectivity to aryloxy alcohol product could be found after introduction of additional Co^{II} salen ligand during the reaction.

Based on these experimental results, the mechanism involving cooperative bimetallic catalysis, simultaneous activation of both epoxides and phenol by different cobalt salen units happens in the dimeric form. The enhanced reactivity of dimeric form under the low loading of catalyst on the basis of cobalt unit indicates that kinetic resolution of epoxides using dimeric catalyst takes place *via* a cooperative action of two cobalt sites present in the intra-framework as shown in scheme 2 [13,17].

The construction of salen polymers consisting of repeating C_2 salen units was carried out to investigate whether the additional monomeric salen might link to the polymer backbone as shown in scheme 3. The attachment of monomeric salen complexes to the backbone of linear polymer chain can be conformed by increase in weight of sample after sufficient washing and drying [18]. It has been investigated that there was no leaching of the monomer units from the polymer backbone during the kinetic resolution after subsequent washing with solvent. The liquid mixture containing the product was transparent during the reaction and it could be easily separated by filtration. The greatly increased activities and recyclability of catalysts were investigated on the polymer salen catalyst and the obtained results are summarized in table 3. Thus these results strongly support the idea that units of salen complexes are linked together as a dimeric form. In all cases the dimeric catalyst was exhibited the higher reactivity than the salen having monomeric structure.

The ring opening of phenyl glycidyl ether with 3-chlorophenol is monitored by IR spectroscopy and the result is shown in figure 4. The disappearance of reactant 3-chlorophenol can be identified by the observation of decrease in phenolic OH peak (3200–3500 cm^{-1}) and appearance of new aliphatic OH peak (2800–3000 cm^{-1}) during the reaction. For this analysis the insoluble

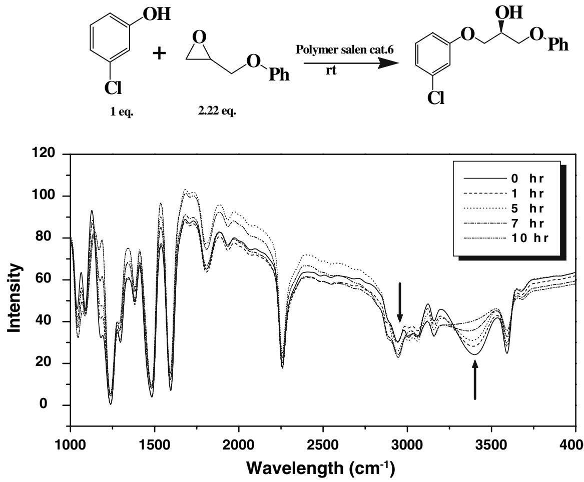


Figure 4. IR Spectra change during the ring opening reaction of phenyl glycidyl ether with 3-chlorophenol.

polymeric salen was used as a catalyst. The liquid type reactant and product could be easily recovered and separated from solid catalyst.

4. Conclusion

In summary, we have synthesized the new type of heterometallic chiral salen complexes and it has been found that group 13 metal salts combined to Co^{III} salen unit played the crucial role in the kinetic resolution of epoxides. More importantly, the Lewis acidic center of the chiral (salen) Co bonded to another Lewis acid center ($AlCl_3$ and $GaCl_3$) enabled the smooth and efficient asymmetric kinetic resolution of terminal epoxides to afford the valuable chiral building blocks. We hope these heterometallic nature of the complex would catalyze other important asymmetric reaction in high yield with excellent enantioselectivity.

Acknowledgment

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