Quaternary onium tribromide catalyzed cyclic carbonate synthesis from carbon dioxide and epoxides

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Abstract

Novel and efficient organic catalysts of quaternary onium tribromide and the related metal-containing catalysts of SalenCoX/PTAT were developed to catalyze the coupling reaction of carbon dioxide and epoxides generating relevant cyclic carbonates including chiral propylene carbonate under extremely mild condition.
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Keywords: Carbon dioxide; Epoxides; Quaternary onium tribromide; Coupling reaction; Chiral propylene carbonate

1. Introduction

Carbon dioxide fixation has received much attention in decades by reason of carbon dioxide is the most inexpensive and infinite carbon resource [1]. The cyclic carbonates are important products of carbon dioxide fixation and are widely used as organic synthesis and pharmaceutical intermediates, aprotic solvents, and raw materials for plastics [2–5]. Various catalyst systems were developed for the coupling reaction of carbon dioxide with epoxides in the so-called carbon dioxide fixation process [6–15]. Without metal involving, the coupling reaction of carbon dioxide and epoxides catalyzed by organic catalyst was rare reported [16–18]. We report, herein, a convenient method of cyclic carbonates synthesis from coupling of carbon dioxide and epoxides utilizing novel and efficient organic catalysts of quaternary onium tribromide (QOTB) and the related metal-containing catalysts of SalenCoX/phenyltrimethylammonium tribromide (PTAT, one of QOTB compounds) under extremely mild condition (Scheme 1).

2. Experimental

2.1. Materials and facilities

^{1}H and ^{13}C NMR spectra were recorded on a Bruker AM-400, AM-300, AM-200 spectrometer using TMS as an internal standard. Elemental analyses were determined with a Carioel elemental analyzer. MS spectra were measured with a ZAB-HS spectrometer. Melting points were determined on a XT-4 melting point apparatus without calibration. GC analyses of cyclic carbonates were carried out on Varian CP 3800 gas chromatograph equipped with FID detectors. For the determination of enantiomeric excess, Supelco-DEX series (225) chiral columns were used.

All known compounds were identified by comparison of their physical and spectral data with those presents in the literatures. Propylene oxide was distilled from CaH_2. Other epoxides and PTAT were purchased from Aldrich and used without further purification. QOTB **4** and **6** were prepared by using literature method [19,20].

2.2. Syntheses of QOTB **2**, **3** and **5**

2.2.1. Preparation of N-benzyl(4-dimethylamino) pyridinium tribromide (**2**)

A solution of 1.22 g of 4-(dimethylamino)pyridine (DMAP) (10 mmol) and 1.20 mL of benzyl bromide (10 mmol) in 10 mL
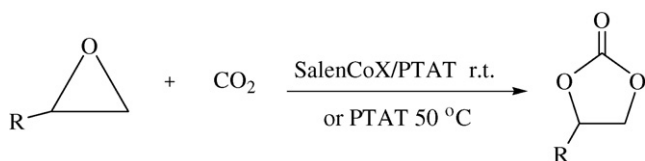
Scheme 1. Catalytic syntheses of cyclic carbonates.

ethanol was heated to reflux for 10 h. After being cooled, the reaction mixture was poured into diethyl ether. The brown precipitates were filtered and washed thoroughly with ether and dried in vacuo. This crude bromide salt 1.5 g was obtained without further purification. Yield = 70.4%.

A solution of 1.5 g of above bromide salt (7 mmol) and 0.36 mL of bromine (7 mmol) in 10 mL water was stirred for 6 h at room temperature. The yellow solid of tribromide 2.3 g was obtained. Yield = 99%, m.p. = 119–120 °C.

^{1}H NMR (acetone-d_6): δ (ppm) = 3.31 (s, 6 H), 5.62 (s, 2 H), 7.13 (d, J = 7.8 Hz, 2 H), 7.35 (dd, J = 4.8 Hz, 2.2 Hz, 3 H), 7.44 (d, J = 4.8 Hz, 2 H), 8.57 (d, J = 7.8 Hz, 2 H). ^{13}C NMR (acetone-d_6): δ (ppm) = 39.8, 60.3, 108.2, 128.4, 128.5, 128.8, 128.9, 129.2, 142.2. MS(FAB): m/z [M]$^+$ calcd. for $C_{14}H_{17}N_2$: 213.14; found: 213.1. Anal. calcd. for $C_{14}H_{17}N_2Br_3$: C, 37.12; H, 3.78; N, 6.18. Found: C, 36.86; H, 3.81; N, 6.00.

2.2.2. Preparation of N-methyl(4-dimethylamino) pyridinium tribromide (3)

To a solution of DMAP (2.44 g, 20 mmol) in 20 mL toluene, a fresh distilled dimethylsulfate (1.9 mL, 20 mmol) was added dropwise within 20 min at 40 °C under stirring condition. The reaction temperature was held constant for 1.5 h and then rose to 100 °C for another 1 h. After being cooled, the product was filtered, washed with 20 mL toluene, and dried in vacuo to give a white solid (8.6 g).

To a solution of above white solid (8.6 g) and 10 mL 40% aqueous hydrobromic acid in 10 mL water, bromine (2.15 mL, 41.8 mmol) was added dropwise within 10 min. The mixture was stirred for 1 h at room temperature. A yellow solid of tribromide 12.5 g was obtained. Yield = 83.6%, m.p. = 122–124 °C.

^{1}H NMR (acetone-d_6): δ (ppm) = 3.32 (s, 6 H), 4.12 (s, 3 H), 7.12 (d, J = 4.2 Hz, 2 H), 8.34 (s, J = 4.2 Hz, 2 H). ^{13}C NMR (acetone-d_6): δ (ppm) = 39.7, 44.4, 107.9, 143.1, 153.5. MS(FAB): m/z [M]$^+$ calcd. for $C_8H_{13}N_2$: 137.11; found: 137.3. Anal. calcd. for $C_8H_{13}N_2Br_3$: C, 25.49; H, 3.48; N, 7.43. Found: C, 25.79; H, 3.26; N, 7.55.

2.2.3. Preparation of P-benzyltriphenylphosphonium tribromide (5)

To a solution of triphenylphosphine (3.93 g, 15 mmol) in 20 mL toluene, 1.80 mL of benzyl bromide (15 mmol) was added dropwise within 20 min at room temperature. The mixture was then heated to reflux for 2 h. After being cooled, the product was filtered, washed with 20 mL toluene, and dried in vacuo to give a white solid (6.6 g).

To a solution of above white solid (1.2 g) in 10 mL CH$_2$Cl$_2$, bromine (0.13 mL, 2.5 mmol) was added. The mixture was

stirred for 1 h at room temperature. A yellow solid of tribromide 1.35 g was obtained. Yield = 84.4%, m.p. = 142.5–145 °C.

^{1}H NMR (CDCl$_3$): δ = 7.84–7.56 (m, 16 H), 7.27–7.20 (m, 2 H), 7.10–6.96 (m, 2 H), 4.87 (d, 2 H). ^{13}C NMR (CDCl$_3$): δ = 135.5, 134.2, 131.2, 130.6, 129.2, 128.9, 117.9, 116.2, 59.2. MS(FAB): m/z [M]$^+$ calcd. for $C_{25}H_{22}P^+$: 353.15; found: 353.1.

2.3. General procedure for coupling reaction of CO$_2$ and epoxide catalyzed by QOTB

A solution of quaternary onium tribromide in epoxide (100 mmol) was introduced into a 250 mL stainless steel autoclave. It was purged three times with carbon dioxide, and then filled CO$_2$ to 100 psi, stirred, and heated to 50 °C. When the pressure of CO$_2$ fell down to 0–15 psi, the reactor was cooled and was then vented. The remained mixture was distilled under reduced pressure or recrystallized with ethanol to obtain the pure cyclic carbonate.

2.4. General procedure for cyclic carbonates synthesis using SalenCoX/PTAT as catalyst

A solution of fresh-made SalenCo(III)X (0.1 mmol) and 76 mg of PTAT (0.2 mmol) in 100 mmol of epoxide was introduced into a 250 mL stainless steel autoclave. It was purged three times with carbon dioxide, and then filled CO$_2$ to 100 psi, stirred at 25 °C. After a proper time, the reactor was vented. The generated carbonate was distilled under reduced pressure or recrystallized from ethanol.

2.5. Characterization of bis((2-oxo-1,3-dioxolan-4-yl) methyl)cyclohex-4-ene-1,2-di carboxylate (13)

A pale yellow oil. ^{1}H NMR (CDCl$_3$): δ = 2.38–2.47 (s, 4 H) 3.16 (s, 2 H), 4.2–4.5 (m, 6 H), 4.61 (t, 2 H), 5.03 (t, 2 H), 5.71 (s, 2 H). ^{13}C NMR (CDCl$_3$): δ = 25.2, 39.4, 63.1, 65.8, 73.8, 124.7, 154.5, 172.8. MS(FAB): m/z [M]$^+$ calcd. for $C_{16}H_{18}O_{10}$: 370.09; found: 370.9.

3. Results and discussion

We have previous reported that the SalenSn complexes can be used as efficient catalysts for coupling reaction of carbon dioxide and epoxides in the presence of DMAP [21]. On the other hand, many salts have been used as catalysts in this coupling reaction including inorganic salts, organic salts, ionic liquid, etc. [22–27]. These investigations reveal that the mechanism of coupling reaction of epoxides and CO$_2$ must involve two catalytic center, Lewis acid and Lewis base center [28–30]. The salts, being make up of two parts: cation (Lewis acid, electrophile) and anion (Lewis base, nucleophile), have catalytic activity under a generally harsh condition. Taking notice of Caló's results [16], it can be seen that tetrabutylammonium chloride (TBAC) has no activity because a tight ion pair cannot release cation to be a catalytic center of Lewis acid, and that tetrabutylammonium iodide (TBAI) is a good catalyst by reason of an incompact ion pair in which the cation can acts as a Lewis acidic center and the

large anion I$^-$ acts as a Lewis base center simultaneously. Very recently, Lau and coworkers reported their coupling results of epoxides and CO_2 using the organic salts $(PPh_3)_2N^+X^-$ as efficient catalyst in which the large cation act as a Lewis acid center [18]. Accordingly, we envisioned that QOTB salts would provide the synergistic combination of the activation of carbon dioxide and epoxides under mild condition since these are incompact ion pairs too.

3.1. The effect of cation of QOTB in the coupling reaction of propylene oxide and CO_2

As prediction, when the tetrabutylammonium tribromide (TBATB, Table 1, entry 4) was only used as catalyst instead of tetrabutylammonium bromide (TBAB, entry 7), the coupling reaction of carbon dioxide and propylene oxide (PO) took place very well in more mild condition. To test this procedure, the scope of the reaction was then investigated with various synthesized QOTB salts. It can be seen that PTAT give the best catalytic result under this reaction condition (entry 1), and that PyH$^+$Br$_3^-$ and TBAB salts (entries 6 and 7) have lowest catalytic activity due to the less Lewis acidity of smaller radius of cations.

3.2. The cyclic carbonates synthesis from various epoxides and CO_2 catalyzed by PTAT

To further extend the scope of the reaction, several other epoxides were utilized as substrates in the coupling reaction using PTAT as catalyst. The results were summarized in Table 2. It can be seen that the cyclic carbonates were prepared with moderate to excellent yield. Some polymerization sensitive epoxides can also reacted very well (entries 4 and 7)

with carbon dioxide under this reaction condition to yield the corresponding cyclic carbonates with a little brominated by-product. The glycidyl methacrylate (GMA) was converted into the cyclic carbonate in 15 h without polymerization (entry 4). Comparing with the literature results, our new organic catalysts of QOTB have higher activity and can catalyze the coupling reaction of carbon dioxide and epoxides under more mild condition.
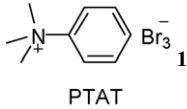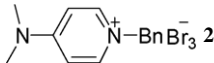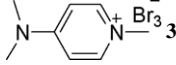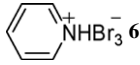
3.3. The co-catalyst effect of SalenCo(III)X on synthesis of propylene carbonate

Very recently, bifunctional catalyst systems of Co(Salen)X/Bu$_4$NY [8,31,32] were found to be the most efficient catalyst systems for this coupling reaction. To extend the scope of PTAT catalyst, the SalenCo(III)X complexes (Scheme 2) and PTAT were used as a new bifunctional catalyst in the coupling reaction of carbon dioxide and PO at room temperature. The results were summarized in Table 3. The screening of ratio of SalenCo(OAc)/PTAT was also achieved and showed that the best ratio is 1/2 (entries 1–4). The catalyst SalenCo(Cl$_3$CCO$_2$)(**1g**)/PTAT gave the high yield and highest TOF (706 mol/mol h, entry 9) within 1 h. In contrast, the **1g**/TBAB catalyst can only get the lower yield (49%) and TOF value (245 mol/mol h, entry 10) in literature.

3.4. The anion effect of SalenCo(III)X on synthesis of propylene carbonate

To optimize and select the best catalyst, the screening of counterion of catalysts SalenCo(III)X was investigated. The order of activity is as follows: X = Cl$_3$CCO$_2^-$ > Cl$^-$ > OAc$^-$ > Br$^-$ > (NO$_2$)$_3$C$_6$H$_2$O$^-$ > OTs$^-$ (Table 4).

Table 1
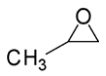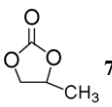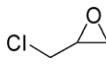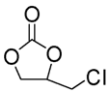Cation effect of QOTB in the coupling reaction of PO and CO_2[a]
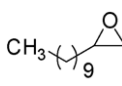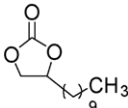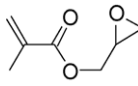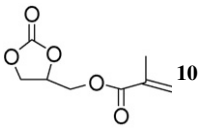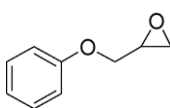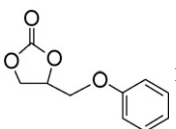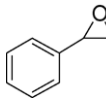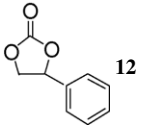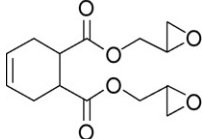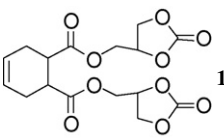
Entry	Organic catalyst	Time (h)	Yield (%)[b]	TON
1	PTAT **1**	6, 1.5[c]	90, 83.1	180, 16.6
2	**2**	6, 1.5[c]	77, 81.4	154, 16.3
3	**3**	6, 1.5[c]	69, 64.7	137, 12.9
4	n-Bu$_4^+$NBr$_3^-$ **4**	6, 1.5[c]	51, 61	103, 12.2
5	Bn$^+$PPh$_3$Br$_3^-$ **5**	6, 1.5[c]	37, 78.4	74, 15.7
6	NHBr$_3$ **6**	6, 1.5[c]	0, 24.3	0, 4.9
7	n-Bu$_4^+$NBr$^-$	6, 1.5[c]	0, 13.2	0, 2.6

[a] Reaction condition: catalyst (0.5 mmol), epoxide (5.8 g, 100 mmol), CO_2 (100 psi), 50 °C.
[b] Isolated yield.
[c] Catalyst (5 mmol).

Table 2
The coupling results of various epoxides and CO_2 catalyzed by PTAT[a]

Entry	Substrate	Time (h)	Product	Yield (%)[b]
1	CH$_3$	6	**7**	90
2	Cl	2, 6	**8**	52, 62
3[c]	CH$_3$(...)$_9$	10	**9**	53
4[d]		15	**10**	60
5[e]		1.5, 3.5	**11**	50, 76
6		20	**12**	86
7[e]		5	**13**	74

[a] Reaction conditions: PTAT (188 mg, 0.5 mmol), epoxide (100 mmol), CO_2 (100 psi), 50 °C.
[b] Isolated yield.
[c] A 10 ml DMF was added as solvent.
[d] A little brominated by-product were detected by GC–MS.
[e] A 20 ml acetone was added as solvent.

3.5. The framework effect of SalenCo(OAc) on synthesis of propylene carbonate

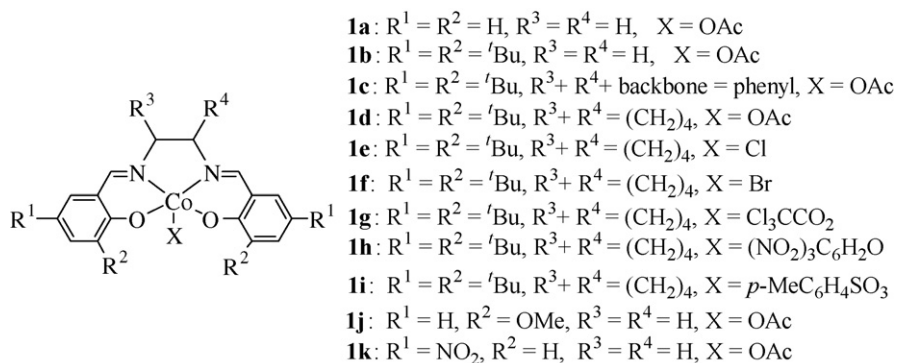To optimize the effect of framework of SalenCo(OAc), several SalenCo(OAc) complexes with different backbone/or different substituted groups were synthesized [33,34], characterized, and used to the coupling reaction of PO and CO_2 (Table 5). The catalysts containing ethylene backbone with different substituted groups have various catalytic activities (entries 1, 2, 5, 6): the catalyst **1k** with electron withdrawing group has lower

1a: R^1 = R^2 = H, R^3 = R^4 = H, X = OAc
1b: R^1 = R^2 = ^{t}Bu, R^3 = R^4 = H, X = OAc
1c: R^1 = R^2 = ^{t}Bu, R^3+ R^4+ backbone = phenyl, X = OAc
1d: R^1 = R^2 = ^{t}Bu, R^3+ R^4 = (CH$_2$)$_4$, X = OAc
1e: R^1 = R^2 = ^{t}Bu, R^3+ R^4 = (CH$_2$)$_4$, X = Cl
1f: R^1 = R^2 = ^{t}Bu, R^3+ R^4 = (CH$_2$)$_4$, X = Br
1g: R^1 = R^2 = ^{t}Bu, R^3+ R^4 = (CH$_2$)$_4$, X = Cl$_3$CCO$_2$
1h: R^1 = R^2 = ^{t}Bu, R^3+ R^4 = (CH$_2$)$_4$, X = (NO$_2$)$_3$C$_6$H$_2$O
1i: R^1 = R^2 = ^{t}Bu, R^3+ R^4 = (CH$_2$)$_4$, X = p-MeC$_6$H$_4$SO$_3$
1j: R^1 = H, R^2 = OMe, R^3 = R^4 = H, X = OAc
1k: R^1 = NO$_2$, R^2 = H, R^3 = R^4 = H, X = OAc

Scheme 2. SalenCo(III)X complex catalyst.

Table 3
The co-catalyst effect of SalenCo(III)X on synthesis of propylene carbonate[a]

Entry	Catalyst	Co-catalyst/equivalent	Time (h)	Yield (%)[b]	TOF (h^{-1})
1	SalenCo(OAc) (**1d**)	PTAT/1	4	91.2	228
2	SalenCo(OAc) (**1d**)	PTAT/2	4	>99	250
3	SalenCo(OAc) (**1d**)	PTAT/4	4	93.1	233
4	SalenCo(OAc) (**1d**)	PTAT/8	4	92.2	230
5	SalenCo(OAc) (**1d**)	PTAT/2	1.5	50.7	338
6[c]	SalenCo(OAc) (**1d**)	Bu$_4$NBr/1	2.5	52.4	210
7	SalenCo(Cl$_3$CCO$_2$)	PTAT/2	4	>99	250
8	SalenCo(Cl$_3$CCO$_2$)	PTAT/2	1.5	90.2	611
9	SalenCo(Cl$_3$CCO$_2$)	PTAT/2	1	70.6	706
10[c]	SalenCo(Cl$_3$CCO$_2$)	Bu$_4$NBr/1	2	49	245

[a] Reaction conditions: SalenCo(III)X (0.1 mmol), PTAT (76 mg, 0.2 mmol), epoxide (100 mmol), $T = 25\,°C$, CO$_2$ (100 psi).
[b] Isolated yield.
[c] Result of Ref. [9].

Table 4
The anion effect of SalenCo(III)X on synthesis of propylene carbonate[a]

Entry	Catalyst	Time (h)	Yield (%)[b]	TOF (h^{-1})
1	**1d**	4	>99	250
2	**1d**	1.5	50.7	338
3	**1e**	4	>99	250
4	**1e**	1.5	55.7	371
5	**1f**	4	88.2	221
6	**1g**	4	>99	250
7	**1g**	1.5	90.2	611
8	**1h**	4	84.3	211
9	**1i**	4	64.7	162

[a] Reaction conditions: SalenCo(III)X (0.1 mmol), PTAT (76 mg, 0.2 mmol), epoxide (100 mmol), $T = 25\,°C$, CO$_2$ (100 psi).
[b] Isolated yield.

activity (entry 6), the catalyst **1b** with more electron donating groups has higher activity (entry 2). The catalyst **1j** with methoxy groups (EDG) has less activity than that of catalyst **1a** without substituted group (entry 1). We might conclude that the activity of catalyst is affected not only by electron effect of backbone but also by steric effect of backbone. This conclusion would be supported by the following investigations: the catalyst **1c** containing phenyl backbone with *tert*-butyl substituted groups has good catalytic activity (entry 3), and the catalyst **1d** (Jacobsen catalyst) containing cyclohexane backbone with *tert*-butyl substituted groups (EDG) has the highest catalytic activity (entry 4).

Table 5
The framework effect of SalenCo(OAc) on synthesis of propylene carbonate[a]

Entry	Catalyst	Time (h)	Yield (%)[b]	TOF (h^{-1})
1	**1a**	4	20.6	51.5
2	**1b**	4	90	225
3	**1c**	4	90.2	225
4	**1d**	4	>99	250
5	**1j**	4	11.8	29.5
6	**1k**	4	7.8	19.5

[a] Reaction conditions: SalenCo(III)(OAc) (0.1 mmol), PTAT (76 mg, 0.2 mmol), epoxide (100 mmol), $T = 25\,°C$, CO$_2$ (100 psi).
[b] Isolated yield.

3.6. The cyclic carbonates synthesis from various epoxides and CO$_2$ catalyzed by SalenCo(Cl$_3$CCO$_2$)PTAT

To further extend the scope of the reaction, several other epoxides were utilized as substrates in the coupling reaction in the presence of the best catalyst system SalenCo(Cl$_3$CCO$_2$)/PTAT. The results were summarized in Table 6. It can be seen that the cyclic carbonates were synthesized with the yields from moderate to excellent except GMA carbonate (entry 5). To enhance the yields of high viscousness (**9**) or solid cyclic carbonates (**11–13**), some diluting solvent was added in the reaction mixture. 3-Chloropropylene oxide having higher boiling point had been run in a three-neck flask under atmospheric pressure to give a reasonable yield within **4h** (entry 3).
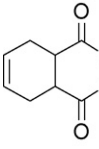
3.7. The chiral propylene carbonate synthesis from PO and CO$_2$

To further extend the scope of the catalysts, several chiral catalysts, (*R,R*)-1,2-cyclohexanediamino-*N,N'*-bis(3,5-di-*tert*-butylsalicylidene)Co(III)X (X = OAc (**1d′**), (NO$_2$)$_3$C$_6$H$_2$O (**1h′**), *p*-MeC$_6$H$_4$SO$_3$ (**1i′**)), were utilized as catalyst in the asymmetric coupling reaction of PO and CO$_2$. The results were summarized in Table 7. It can be seen that the *ee* values of chiral propylene carbonate are similar to that of Lu's result [9].

3.8. The proposed mechanism for coupling reaction of PO and CO$_2$

Jacobsen and coworkers have demonstrated that coordination of the epoxide to the SalenCr(III) complex is a necessary step to activate the former toward nucleophilic ring opening by the SalenCr(III)–azide complex [35]. Considering the mechanisms of coupling reaction of epoxide and CO$_2$ described by Lu and Lau in literatures [9,18], respectively, a proposed general mechanism is illustrated in Scheme 3, in which the epoxide be activated by coordinating firstly to a Lewis acidic center, and then be attacked by nucleophilic reagent Br$_3^-$ to produce the requisite metal/heteroatom alkoxide intermediates suffering insertion of CO$_2$ to lead cyclic carbonates. This mechanism insists that both Lewis acidic center and Lewis base

Table 6
The coupling results of various epoxides and CO_2 catalyzed by SalenCo(Cl$_3$CCO$_2$)/PTAT[a]

Entry	Substrate	Time (h)	Product	Yield (%)[b]	TOF (h^{-1})
1		1.5	**7**	90.2	611
2		4	**8**	57	143
3[c]		4	**8**	25.2	63
4[d]		10	**9**	72.8	73
5		5	**10**	29	58
6[d]		3	**11**	98	327
7[d]		10	**12**	67	67
8[e]		3	**13**	72.7	242

[a] Reaction conditions: SalenCo(III) (Cl$_3$CCO$_2$) (0.1 mmol), PTAT (76 mg, 0.2 mmol), epoxide (100 mmol), $T = 25\,°C$, CO_2 (100 psi).
[b] Isolated yield.
[c] CO_2 atmospheric pressure.
[d] A 1 mL DMF was added to dissolved the product.
[e] A 10 mL acetone was used to dilute the substrate.

center have the same importance when the coupling reaction takes place. The higher activity of these catalytic systems, SalenCoX/PTAT, is attributed to both good nucleophilicity and leaving group of large anion Br$_3^-$ except for their appropriate Lewis acidity. The PTAT is a good organic catalyst since it can release both Lewis acid and Lewis base centers in the epoxide solution, which is consistent with the Lau's result [18].
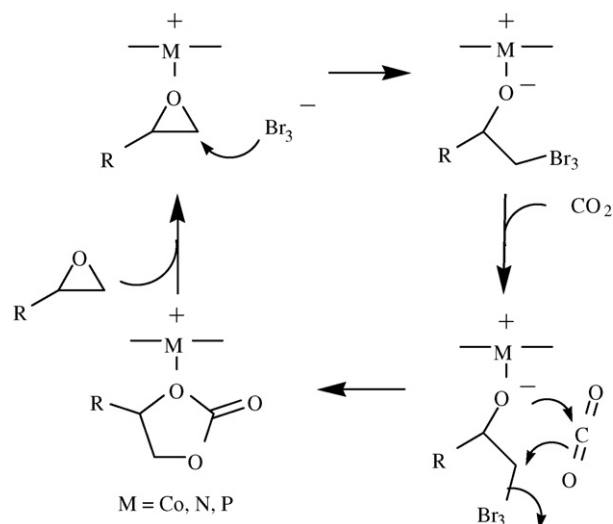
Table 7
The asymmetric coupling reaction of CO_2 and propylene oxide[a]

Entry	Catalyst	Time (h)	Yield (%)[b]	ee %
1[c]	**1d′**/2TBAB	4	45.1	34.1
2	**1d′**/2PTAT	10	57.7	30.2
3	**1h′**/2PTAT	15	31.4	45.5
4	**1i′**/2PTAT	10	43.1	48.7

[a] Reaction conditions: (Salen)CoX (0.1 mmol), PTAT (76 mg, 0.2 mmol), epoxide (100 mmol), CO_2 (100 psi), $T = -5\,°C$.
[b] Isolated yield.
[c] $T = 25\,°C$.

M = Co, N, P

Scheme 3. Proposed mechanism.

4. Conclusions

In conclusion, this methodology investigation for synthesis of cyclic carbonates shows that SalenCo(III)(Cl$_3$CCO$_2$)/PTAT is an excellent catalyst system for the coupling of carbon dioxide with various epoxides to yield cyclic carbonates under extremely mild condition. The PTAT itself can also act as an efficient organic catalyst in this coupling reaction under atmospheric pressure at 50 °C.

Acknowledgments

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