



Communication

Enantioselective carboxylative cyclization of propargylic alcohol with carbon dioxide under mild conditions

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ABSTRACT

An enantioselective carboxylative cyclization of propargylic alcohols and CO₂ was realized under mild conditions, based on a kinetic resolution strategy, which enabled the synthesis of chiral cyclic carbonates and propargylic alcohols with promising yield and enantioselectivity simultaneously.

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The utilization of carbon dioxide (CO₂) as a renewable, abundant and nontoxic C1 feedstock for chemical synthesis has received considerable attentions [1]. As it not only provides an attractive complement to carbon capture and storage, but also presents an important way to realize the green carbon science [2]. Over the past decades, a series of reactions have been developed for CO₂ chemical transformation [3], among which, the carboxylative cyclization of propargylic alcohols and CO₂ is very attractive [4], as the thus obtained α -alkylidene cyclic carbonates have wide applications in both organic synthesis and industrial chemistry [5]. However, duo to the well-known challenge associate with enantioselective transformation of CO₂, such as thermodynamic stability and kinetic inertness, the catalytic asymmetric carboxylative cyclization of propargylic alcohols and CO₂ is largely undeveloped [6]. The only two successful reports were realized by Yoshida & Ihara and Yamada respectively [7]. As early as 2003, Yoshida & Ihara developed a palladium-catalyzed cascade reaction of 4-methoxycarbonyloxy-2-butyne-1-ols with phenols and CO₂, which involved a CO₂ elimination-fixation process (Scheme 1A) [7a]. In 2010, Yamada and co-workers reported an enantioselective carboxylative cyclization of propargylic alcohols with CO₂ via a chiral silver catalyzed desymmetrization process

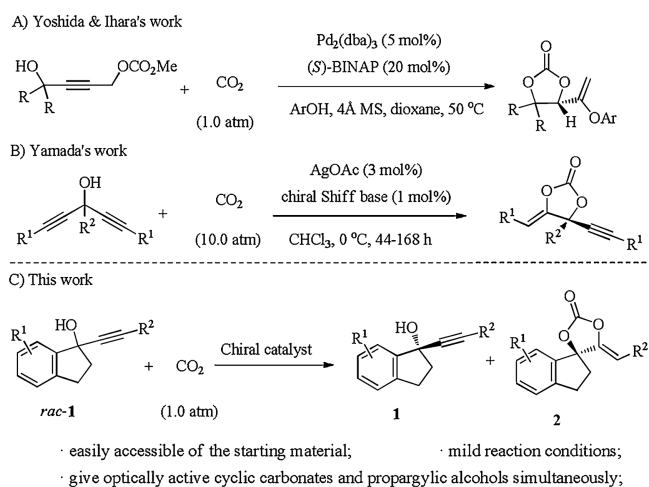
(Scheme 1B) [7b]. Distinct from those researches, we envisioned that enantioselective kinetic resolution would be another promising strategy for the development of enantioselective carboxylative cyclization of propargylic alcohols and CO₂ (Scheme 1C) [8]. The attractive features of this approach include the easy accessibility of various types of propargylic alcohols and, more importantly, the chiral cyclic carbonates and optically active propargylic alcohols could be obtained simultaneously under mild conditions.

The optically active propargylic alcohols are highly important as versatile building blocks for the construction of biologically active natural products as well as pharmaceuticals [9]. Enormous efforts have been devoted to the development of highly efficient catalytic asymmetric methods for propargylic alcohol synthesis [10]. However, most of these reports are aim to the chiral secondary propargylic alcohols, the access to optically active tertiary propargylic alcohols is much more challenging [11]. In this context, the development of new synthetic methods to optically active tertiary propargylic alcohols is highly desirable.

In light of these facts, together with our continue efforts in the enantioselective construction of chiral tertiary alcohols [12] and the chemical fixation of CO₂ for value-added products [13], herein, we wish to report our preliminary research in developing a catalytic asymmetric carboxylative cyclization of propargylic alcohols and CO₂ for the synthesis of optical active tertiary propargylic alcohols and chiral cyclic carbonates simultaneously, based on a kinetic resolution strategy.

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Scheme 1. Asymmetric carboxylative cyclization of propargylic alcohol and CO₂.

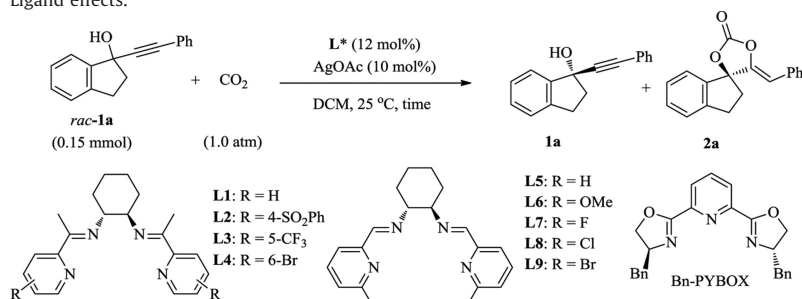
The 1-indanone derived propargylic alcohol **1a** was utilized for the investigation of the reaction, as it could give the novel spirocyclic carbonates and the chiral cyclic propargylic alcohols simultaneously, both compounds have a range of applications. We initiated our research by studying the kinetic resolution under 1 atm pressure of CO₂ in the presence of 10 mol% of AgOAc with various chiral ligands (for detail, see Supporting information). To our delight, the 1-(2-pyridinyl)ethanone derived chiral Schiff base **L1**, which had been used by Yamada in the desymmetric carboxylative cyclization [7b], could promoted the reaction smoothly in dichloromethane (DCM) under room temperature, to give the desired chiral carbonate **2a** in 49% yield and 30% *ee*, with the recovery of optical active **1a** in 46% yield and 43% *ee* (entry 1, Table 1). Other chiral ketimine ligand, such as **L2**, **L3** and **L4** bearing a 4-sulfonyl, 5-trifluoromethyl or 6-bromo group in pyridyl ring, gave slightly lower yield and enantioselectivity respectively (entries

2-4). Then, we turned attention to the 2-pyridinecarboxyaldehyde derived Schiff base ligand, and found that the substituents on the pyridyl ring had great influence on the reaction. Although the simple chiral aldimine ligand **L5** gave very poor results, the enantioselectivity increased gradually when the 6-substituted chiral ligands **L5-L9** was employed (entries 5-9). Finally, chiral ligand **L8** with an electron-withdrawing 6-chloro-substituted group was demonstrated to be the best one, affording the chiral **2a** in 53% yield and 36% *ee*, with the recovery of chiral **1a** in 46% yield and 60% *ee* (entry 8). The performance of other types of chiral ligand such as Bn-PYBOX was also studied, but only 3% *ee* for both **1a** and **2a** were obtained (entry 10).

To further improve the reaction outcomes, various silver salts and solvents were briefly investigated by using Schiff base **L8** as chiral ligand. As shown in Table 2, the silver salts had dramatic effects on the reaction. Apart from AgOAc and AgOBz, other ones failed to catalyze the reaction. While AgOBz gave slightly diminished enantioselectivity than AgOAc (entries 1-2, Table 2). The solvent effects were also investigated, but no better results could be obtained (entries 3-5). Therefore, the substrate scope was then examined by using **L8**/AgOAc complex as catalyst, DCM as solvent, under 1 atmosphere of CO₂ pressure.

As shown in Table 3, most of the 1-indanone derived propargylic alcohols bearing different substituents on the alkynyl moiety or phenyl ring were viable substrates for this kinetic resolution, with moderate enantioselectivity achieved. Initially, propargylic alcohols with different substituents (R²) on the alkynyl moiety were investigated. As expected, with the R² group varying from phenyl to *para*-halogenated phenyl groups, the reaction rates increased to a certain degree, and the carbonates **2b-2d** could be obtained smoothly, with the recovery of the chiral propargylic alcohols **1b-1d** in 40%-50% yields and 50%-59% *ee* values (entries 2-4). For the reaction of *para*-methylphenyl substituted propargylic alcohols, affected by the electron-donating effect, a slightly higher 0.5 MPa CO₂ pressure was needed (entry 5). By verifying the substituent in the phenyl ring from *para*-position to *meta*-position, the reaction could also proceed well to give the corresponding

Table 1
Ligand effects.



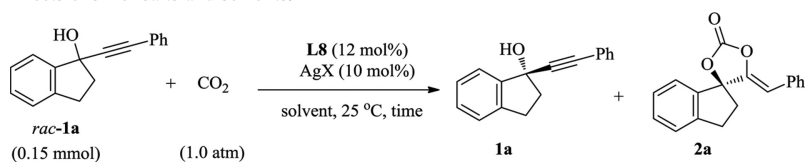
Entry	L*	Time (h)	1a		2a		s factor ^c
			Recovery (%) ^a	<i>ee</i> (%) ^b	Yield (%) ^a	<i>ee</i> (%) ^b	
1	L1	3.0	46	43	49	30	3
2	L2	5.0	43	18	45	14	2
3	L3	7.5	40	38	50	18	2
4	L4	13.0	57	20	41	29	2
5	L5	3.0	58	15	40	10	2
6	L6	72.0	42	35	40	20	2
7	L7	2.5	48	49	52	34	4
8	L8	4.5	46	60	53	36	6
9	L9	4.0	50	50	49	39	5
10	Bn-PYBOX	4.5	43	3	41	3	1

^a The recovery of **1a** and yield of **2a** were determined by ¹HNMR using CH₂Br₂ as internal standard.

^b *ee* was determined by chiral HPLC analysis.

^c $s = \ln[(1-C)(1-ee)] / \ln[(1-C)(1+ee)]$; C refers to the conversion of (*rac*)-**1a** [1-(the recovery of **1a**)].

Table 2
Effects of silver salts and solvents.



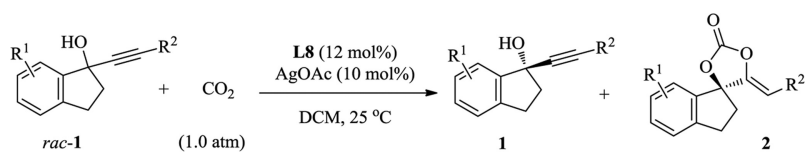
Entry	AgX	Solvent	Time (h)	1a		2a		s factor ^c
				Recovery (%) ^a	ee (%) ^b	Yield (%) ^a	ee (%) ^b	
1	AgOAc	DCM	4.5	46	60	53	36	6
2	AgOBz	DCM	8.0	53	46	41	38	5
3	AgOAc	CH ₃ CN	4.5	44	54	46	25	4
4	AgOAc	CHCl ₃	24	55	34	33	24	3
5	AgOAc	DCE	17	27	50	54	12	2

^a The recovery of **1a** and yield of **2a** were determined by ¹H NMR using CH₂Br₂ as internal standard.

^b ee was determined by chiral HPLC analysis.

^c $s = \ln[(1-C)(1-ee)]/\ln[(1-C)(1+ee)]$; C refers to the conversion of (*rac*)-**1a** [1-(the recovery of **1a**)].

Table 3
The substrate scope.



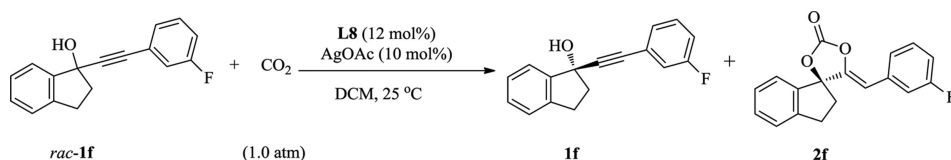
Entry	Compd.	R ¹	R ²	Time (h)	1		2		s factor ^c
					Recovery (%)	ee (%) ^a	Yield (%)	ee (%) ^a	
1	a	H	Ph	4.5	46	60	53	36	6
2	b	H	4-FC ₆ H ₄	3.5	50	50	49	34	5
3	c	H	4-ClC ₆ H ₄	2.0	40	59	51	23	4
4	d	H	4-BrC ₆ H ₄	4.3	46	56	53	20	5
5 ^b	e	H	4-MeC ₆ H ₄	1.5	44	57	55	20	5
6	f	H	3-FC ₆ H ₄	1.5	43	54	49	24	4
7	g	H	3-ClC ₆ H ₄	1.5	54	43	40	22	5
8	h	H	3-MeC ₆ H ₄	9.0	59	37	38	43	5
9	i	H	2-FC ₆ H ₄	4.5	45	59	55	23	5
10	j	H	2-ClC ₆ H ₄	5.0	46	45	54	31	3
11	k	4-Br	Ph	9.0	42	45	58	25	3
12 ^b	l	5-F	Ph	1.5	51	47	49	14	5

^a ee was determined by chiral HPLC analysis.

^b The reaction was carried out under 0.5 MPa CO₂ pressure.

^c $s = \ln[(1-C)(1-ee)]/\ln[(1-C)(1+ee)]$; C refers to the conversion of (*rac*)-**1** [1-(the recovery of **1**)].

Table 4
The monitor of the reaction course.

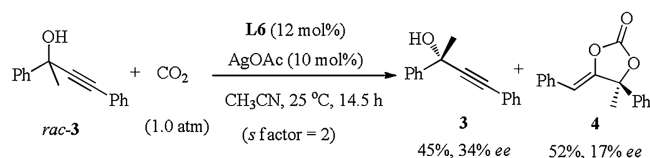


Entry	Time (h)	Conv. (%) ^a	ee of 1f (%) ^b	ee of 2f (%) ^b	s factor ^c
1	0.5	10	3	16	2
2	1.0	38	17	22	2
3	1.5	57	54	24	4
4	2.0	78	69	17	3
5	2.5	86	88	10	3

^a Determined by ¹H NMR.

^b determined by chiral HPLC analysis.

^c $s = \ln[(1-C)(1-ee)]/\ln[(1-C)(1+ee)]$; C refers to the conversion of (*rac*)-**1f** [1-(the recovery of **1f**)].



Scheme 2. The kinetic resolution of non-cyclic propargylic alcohol.

chiral carbonates **2f–2h** in 38%–49% yield, with the recovery of chiral alcohols **1f–1h** in 43%–59% yield and 37%–54% *ee* value (entries 6–8). When *ortho*-fluoro- or *ortho*-chloro-phenyl substituted propargylic alcohol **2i** or **2j** was applied to the reaction, a slightly lower reaction rate was observed, probably due to the steric hindrance effect (entries 9–10). Then, the reaction of propargylic alcohols **1k** and **1l** bearing 4-bromo and 5-fluoro substituent at the phenyl ring were studied, which could deliver the corresponding chiral carbonate **2k** and **2l** in 58% and 49% yield, along with the recovery of enantioenriched propargylic alcohol **1k** and **1l** in 42% and 51% yield with 45% and 47% *ee* respectively (entries 11 and 12). Further attempts to use the alkyl substituted or terminal alkyne derived propargylic alcohols were nevertheless unsuccessful, and no desired products could be detected. The absolute configuration of chiral alcohol **1a** was determined to be *S* by comparing its optical rotation value with the one synthesized by the asymmetric nucleophilic addition reaction [14]. Based on this, the absolute configuration of other chiral propargylic alcohols and carbonates were tentatively deduced by analogy (for detail, see Supporting information).

It should be pointed out that in most cases of the reaction, there is a notable difference between the *ee* value of recovered alcohols and chiral carbonates, even though the conversions are about 50% and the transformations are very clean. Theoretically, the *ee* value of both chiral compounds should be very similar to each other. To figure out the reason, the reaction of racemic-**1f** was monitored over time. As shown in Table 4, with the reaction went on, the conversion and the *ee* value of **1f** increased gradually, but the *ee* value of **2f** went up to a certain value, then dropped down. These results indicated that the chiral carbonate might be racemized during the reaction cause, and the best results could be obtained at around 50% conversion.

Finally, the reaction of the non-cyclic propargylic alcohol **3** with CO_2 was studied (Scheme 2), by using the **L6**/ AgOAc as chiral catalyst and CH_3CN as solvent, the chiral carbonate **4** was obtained in 52% yield, with the recovery of chiral propargylic alcohols **3** in 45% yield and 34% *ee* value.

In conclusion, we have developed a novel catalytic asymmetric carboxylative cyclization of propargylic alcohols with CO_2 based on a kinetic resolution process. Under mild reaction conditions, both optically active propargylic alcohols and chiral cyclic carbonates could be obtained simultaneously with considerable yield and moderate enantioselectivity by simple operation. Further investigation including the exploitation of new catalytic systems to improve the catalytic efficiency along with the development of other catalytic asymmetric reactions using CO_2 as C1 synthon are now in progress in our laboratory.

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Appendix A. Supplementary data

Supplementary material related to this article can be found, in the online version, at doi:<https://doi.org/10.1016/j.ccl.2019.05.060>.

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