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Lewis Acid Catalyzed Synthesis of Cyclic Carbonates, Precursors of 1,2- and 1,3-Diols

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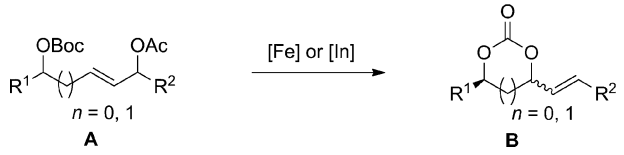
An eco-friendly synthesis of cyclic carbonates through a Lewis acid catalyzed cyclization of *tert*-butyl carbonates is described. These cyclic carbonates are precursors of 1,2- and 1,3-diols, and the developed method was applied to a short synthesis of a diarylheptanoid, (3*S*,5*S*)-alpinikatin.

Introduction

As 1,2- and 1,3-diols are ubiquitous units in biologically active compounds such as polyketide derivatives, a myriad of methods allowing the diastereoselective preparation of 1,2- and 1,3-diols has been developed[1] and utilized in the synthesis of natural products.[2] Among them, the stereoselective cyclization of allylic and homoallylic *tert*-butyl carbonates has emerged as a powerful strategy relying on asymmetric induction,[3,5] and the resulting cyclic carbonates can easily be converted into the corresponding diols under basic conditions. Since the pioneering work of Bartlett et al. in 1982,[4a] halogeno-cyclization providing iodo- or bromocarbonates has been widely used in total synthesis to introduce 1,2- as well as 1,3-diol moieties.[6] In contrast, examples of metal-catalyzed cyclization of allylic and homoallylic carbonates are still scarce and, to the best of our knowledge, most of them involve palladium catalysis.[7–9] Thus, an atom-economic and eco-friendly process is highly desirable to access 1,2- and 1,3-diols via cyclic carbonates. Our group has been involved in iron-catalyzed reactions[10] and particularly in the diastereoselective synthesis of a variety of heterocycles such as piperidines, tetrahydropyrans, or isoxazolidines.[10b–10d] Heterocycle formation implies an iron-induced activation of allylic and/or benzylic acetate derivatives. Herein, we would like to report an iron- and/or indium-catalyzed cyclization of *tert*-butyl carbonates **A** providing cyclic carbonates **B** (Scheme 1).



Scheme 1. Formation of cyclic carbonates from *tert*-butyl carbonates.

Results and Discussion

The cyclization of **1a** was first examined, and a variety of Brønsted and Lewis acids were screened (Table 1). In the presence of 10 mol-% PTSA·H_2O (PTSA = *p*-toluenesulfonic acid) or HCl (2 M in dioxane) in CH_2Cl_2, the starting material was completely recovered, whereas upon treatment with 10 mol-% TfOH, degradation was observed with no trace of the desired product (Table 1, entries 1–3). Lewis acids appeared more powerful than Brønsted acids, as the use of 10 mol-% $FeCl_3$·$6H_2O$, $La(OTf)_3$, $Cu(OTf)_2$, or $Zn(OTf)_2$ in CH_2Cl_2 yielded the cyclic carbonate **2a** in 56–69% NMR yield with a diastereomeric ratio of 70:30 in favor of the *cis*-isomer (Table 1, entries 4–7).[11] To our delight, when CH_2Cl_2 was replaced by CH_3CN, upon treatment with 10 mol-% $FeCl_3$·$6H_2O$,[12] **2a** was formed in 74% isolated yield and in a diastereomeric ratio of 73:27 (Table 1, entry 8). The best result was obtained in the presence of 10 mol-% $InCl_3$ as a 92% yield in cyclic carbonate **2a** was reached (Table 1, entry 9).[13,14] Thus, these two Lewis acids were selected to evaluate the scope and limitation of the cyclization of *tert*-butyl carbonates.

The optimized conditions were then applied to the cyclization of homoallylic *tert*-butyl carbonates **1b**–**1o** (Table 2). A phenylallylic substituent (R^2 = Ph) was found to be essential to the cyclization, as no conversion was observed for primary acetate **1b** (R^2 = H) or for homoallylic carbonate **1c** (R^2 = C_5H_{11}), regardless of the catalytic sys-

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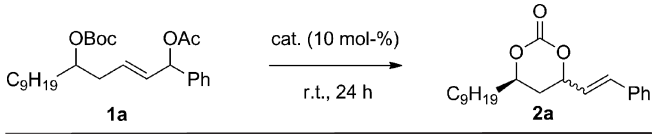
Table 1. Optimization of the cyclization conditions.



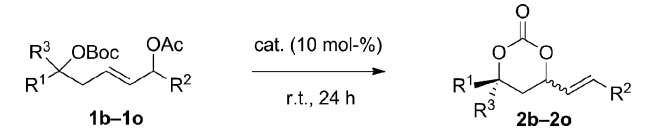
1a 2a

Entry	Solvent	Catalyst	NMR yield[a]	*cis*/*trans* ratio[b]
1	CH_2Cl_2	PTSA·H_2O	0%	–
2	CH_2Cl_2	TfOH	0%	–
3	CH_2Cl_2	HCl[c]	0%	–
4	CH_2Cl_2	$FeCl_3$·$6H_2O$	56%	70:30
5	CH_2Cl_2	$La(OTf)_3$	59%	70:30
6	CH_2Cl_2	$Cu(OTf)_2$	63%	70:30
7	CH_2Cl_2	$Zn(OTf)_2$	69%	70:30
8	CH_3CN	$FeCl_3$·$6H_2O$	94% (74%)	73:27
9	CH_3CN	$InCl_3$	100% (92%)	73:27

[a] Isolated yields in parentheses. [b] Determined by ^{1}H NMR spectroscopy on the crude reaction mixture. [c] 2 M in dioxane.

tem used (Table 2, entries 1 and 2).[15] When alkyl-substituted homoallylic *tert*-butyl carbonates **1d** (R^1 = Cy) and **1e** (R^1 = Me) were treated with 10 mol-% $FeCl_3$·$6H_2O$, the corresponding cyclic carbonates were isolated in good yields (82 and 89%, respectively), however with moderate diastereoselectivities (*dr* = 75:25 and 71:29, respectively) (Table 2, entries 3 and 4). A phenyl group can also be tolerated, as cyclic carbonate **2f** was obtained in 76% yield and in a 75:25 diastereomeric ratio (Table 2, entry 5). Interestingly, when homoallylic carbonate **1g**, derived from a tertiary alcohol, was treated with $FeCl_3$·$6H_2O$, the expected cyclic carbonate was formed with a good yield of 77%, albeit with low diastereoselectivity (*dr* = 60:40) (Table 2, entry 6).

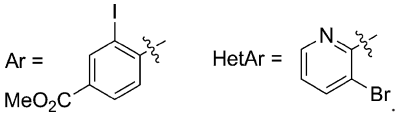
Table 2. Synthesis of a variety of six-membered-ring cyclic carbonates.



1b–1o 2b–2o

Entry	1	R^1	R^2	R^3	Cat.[a]	2, yield % (*dr*)[b,c]
1	**1b**	Ph	H	H	[Fe], [In]	**2b**, 0% (n.d.)
2	**1c**	Ph	C_5H_{11}	H	[Fe], [In]	**2c**, 0% (n.d.)
3	**1d**	Cy	Ph	H	[Fe]	**2d**, 82% (75:25)
4	**1e**	Me	Ph	H	[Fe]	**2e**, 89% (71:29)
5	**1f**	Ph	Ph	H	[Fe]	**2f**, 76% (75:25)
6	**1g**	C_4H_9	Ph	Me	[Fe]	**2g**, 77% (60:40)
7	**1h**	C_3H_6OH	Ph	H	[In]	**2h**, 0% (n.d.)
8[d]	**1i**	C_3H_6OAr	Ph	H	[In]	**2i**, 79% (70:30)
9[d]	**1j**	$C_3H_6OHetAr$	Ph	H	[In]	**2j**, 82% (73:27)
10	**1k**	C_3H_6Br	Ph	H	[In]	**2k**, 79% (75:25)
11	**1l**	CO_2Et	Ph	H	[In]	**2l**, 54% (70:30)
12	**1m**	$C_3H_6NHPhth$	Ph	H	[In]	**2m**, 78% (75:25)

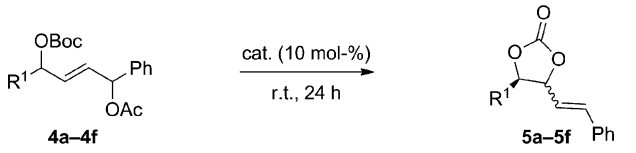
[a] [Fe] = $FeCl_3$·$6H_2O$, [In] = $InCl_3$. [b] Isolated yields. [c] Determined by ^{1}H NMR spectroscopy on the crude mixture. [d]



The presence of an hydroxy group was found to be detrimental to the cyclization, as no trace of the desired product was observed when **1h** was treated with $InCl_3$ (Table 2, entry 7).[16] In contrast, an aryl ether was compatible with the reaction conditions as the $InCl_3$-catalyzed cyclization of **1i** proceeded smoothly, affording the expected carbonate in 79% yield with a 70:30 diastereomeric ratio (Table 2, entry 8).[17] More surprisingly, the presence of a pyridyl ether did not disrupt the cyclization (Table 2, entry 9). In both previous examples, it should be highlighted that halogen atoms were well tolerated, thus offering opportunities for further functionalization. In addition, an alkyl bromide was also a suitable substituent, as carbonate **2k** was formed in 79% yield (Table 2, entry 10). Gratifyingly, in the presence of $InCl_3$, cyclic carbonates **1l** and **1m**, incorporating an ester and a phthalimide moiety, respectively, were formed in moderate to good yields of 54 and 78%, respectively, in a 70:30 and 75:25 *cis*/*trans* ratio (Table 2, entries 11 and 12), respectively.

In order to access 1,2-diols, we then turned our attention to the formation of five-membered-ring carbonates through allylic carbonate cyclization (Table 3). Upon treatment with 10 mol-% $FeCl_3$·$6H_2O$, allylic carbonates possessing an alkyl substituent such as **4a** (R^1 = C_9H_{19}) or **4b** (R^1 = Cy) delivered the corresponding cyclic carbonates with excellent yields (98 and 81% for **4a** and **4b**, respectively) but with moderate diastereoselectivity in favor of the *trans*-diastereomer (*dr* = 63:37 and 65:35, respectively) (Table 3, entries 1 and 2). However, the two diastereomers were separated by flash chromatography on silica gel. Disappointingly, the presence of a phenyl substituent (**4c**, R^1 = Ph) led to the formation of the deprotected and/or the isomerized products with no trace of the expected cyclized derivative. Switching from $FeCl_3$·$6H_2O$ to $InCl_3$ did not bring any improvement (Table 3, entry 3). This result could be explained by the presence of two benzylic positions in **4c**, both of which could be activated in the presence of a Lewis acid to yield a complex mixture of products. When a hydroxy group was present, such as in **4d**, the expected cyclic carbonate was formed (37%) (Table 3, entry 4) together with tetra-

Table 3. Synthesis of a variety of five-membered-ring cyclic carbonates.



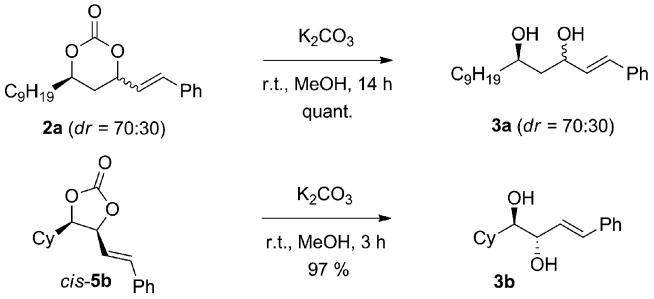
4a–4f 5a–5f

Entry	4	R^1	Cat.[a]	5, yield % (*dr*)[b],[c]
1	**4a**	C_9H_{19}	[Fe]	**5a**, 98% (63:37)
2	**4b**	Cy	[Fe]	**5b**, 81% (65:35)
3	**4c**	Ph	[Fe], [In]	**5c**, 0% (n.d.)
4	**4d**	C_3H_6OH	[In]	**5d**, 37% (75:25), **5′d**[18]
5	**4e**	C_3H_6OPMB	[In]	**5e**, 62% (70:30)
6	**4f**	$C_3H_6NTsBoc$	[In]	**5f**, 83% (64:36)

[a] [Fe] = $FeCl_3$·$6H_2O$, [In] = $InCl_3$. [b] Isolated yields. [c] Determined by ^{1}H NMR spectroscopy on the crude mixture.

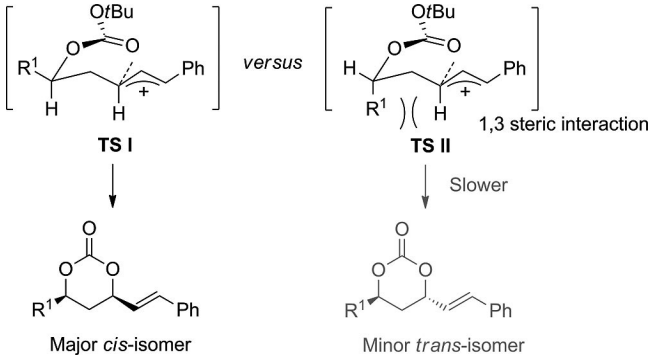
hydropyran **5′d** resulting from the nucleophilic attack of the alcohol (25%, *dr* = 60:40) (Table 3, entry 4).[18] Protection of the alcohol as a PMB ether (**4e**) significantly improved the yield of the cyclic carbonate (62%) (Table 3, entry 5). A protected amine is also tolerated under these reaction conditions, as carbonate **5f** was isolated in 83% yield with a diastereomeric ratio of 64:36 (Table 3, entry 6).

In order to ensure the easy formation of 1,3- and 1,2-diols from cyclic carbonates, **2a** (*dr* = 70:30) was treated with K_2CO_3 in MeOH. As expected, the corresponding 1,3-diol **3a** was isolated in quantitative yield with no modification of the diastereomeric ratio (Scheme 2). Similarly, carbonate *cis*-**5b** was easily transformed into 1,2-diol **3b** (Scheme 2).



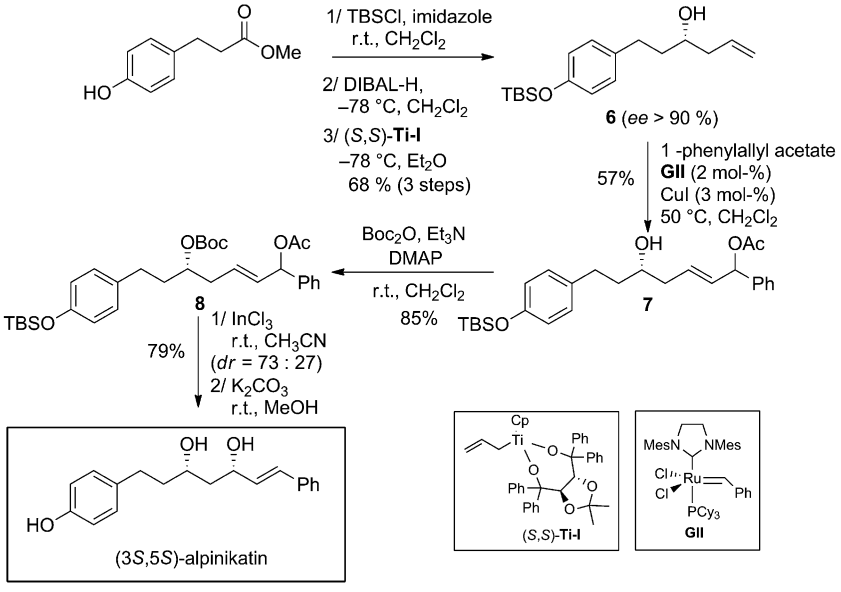
Scheme 2. Preparation of 1,3- and 1,2-diols.

Considering our previous mechanistic studies on iron-catalyzed heterocyclizations,[10] we suggest that an allylic carbocation intermediate is formed during the reaction. When the $InCl_3$-catalyzed cyclization of **1e** was monitored by ^1H NMR spectroscopy, no change in the diastereomeric ratio was observed, which suggests a kinetic control.[19] The diastereoselectivities could be explained by the minimization of the steric interactions in the transition state as shown in Scheme 3 for the cyclization of homoallylic carbonates (**TS I** vs. **TS II**). However, the ability of $FeCl_3{\cdot}6H_2O$ to induce the epimerization of 2,6-piperidines or 2,6-tetrahydropyrans by a re-opening process has already been highlighted,[9] and, as a consequence, the hypothesis of a thermodynamic control could not be completely ruled out.



Scheme 3. Hypothetical origin of diastereoselectivity under kinetic control.

In order to illustrate the potential of our method, we embarked on a short synthesis of (3*S*,5*S*)-alpinikatin (Scheme 4), which was recently extracted from the seeds of *Alpinia katsumadai*.[20] (3*S*,5*S*)-Alpinikatin is part of the diarylheptanoid family, which includes more than 300 molecules exhibiting attractive biological properties.[21] From a structural point of view, (3*S*,5*S*)-alpinikatin possesses a 1,3-diol motif that could come from a homoallylic carbonate through the developed indium-catalyzed cyclization followed by methanolysis. The synthesis started with the protection and subsequent reduction of methyl 3-(4-hydroxyphenyl)propionate to provide the corresponding



Scheme 4. Total synthesis of (3*S*,5*S*)-alpinikatin.

aldehyde. An enantioselective allyltitanation with the Duthaler–Hafner complex (*S*,*S*)-**Ti-I**[22] was then performed to afford the expected homoallylic alcohol **6** with good yield and enantiomeric excess (*ee* > 90%). A cross-metathesis with 1-phenylallyl acetate in the presence of the Grubbs II catalyst (**GII**) and CuI[23] furnished **7**, which was transformed into carbonate **8** upon treatment with Boc_2O. When this homoallylic carbonate was treated with 10 mol-% $InCl_3$, the corresponding cyclic product was isolated with a good yield of 81% albeit with a moderate diastereoselectivity of 73:27 in favor of the *cis*-isomer. Flash chromatography on silica gel allowed a partial separation of the two diastereomers, and a pure sample of the *cis*-compound was isolated. Finally, the use of K_2CO_3 in MeOH provided (3*S*,5*S*)-alpinikatin through carbonate methanolysis and deprotection of the phenolic group.

Conclusions

In summary, we have developed a new approach to five- and six-membered-ring cyclic carbonates by a Lewis acid catalyzed cyclization of allylic and homoallylic *tert*-butyl carbonates. In most examples, the reaction proceeds with high yields and moderate diastereoselectivities. The cheap $FeCl_3{\cdot}6H_2O$ with its low toxicity can be used as a catalyst for the cyclization, but $InCl_3$ was preferred when functional groups were present. The synthesized carbonates can easily be transformed to the corresponding 1,2- and 1,3-diols, and the method has been successfully applied to the total synthesis of a natural product, (3*S*,5*S*)-alpinikatin.

Experimental Section

Typical Procedure for Lewis Acid Catalyzed Cyclization of Allylic and Homoallylic Carbonates: To a solution of the *tert*-butyl carbonate **A** (1 equiv.) in CH_3CN (0.1 M) was added $FeCl_3{\cdot}6H_2O$ (0.1 equiv.) or $InCl_3$ (0.1 equiv.). The resulting mixture was stirred for 24 h at room temp., and the crude mixture was concentrated under reduced pressure. Flash chromatography on silica gel afforded the desired cyclic carbonates **B**.

Supporting Information (see footnote on the first page of this article): Experimental procedures, characterization data and copies of ^1H NMR and ^{13}C NMR spectra.

Acknowledgments

Agence Nationale de la Recherche (ANR) is thanked for financial support.

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[11] The same results were obtained with anhydrous $FeCl_3$ and $FeCl_3{\cdot}6H_2O$, and the latter was preferred as it was easier to handle.

[12] $FeCl_3{\cdot}6H_2O$ was purchased from Sigma–Aldrich (purity 97%, Cu < 0.003%, Zn < 0.003%).

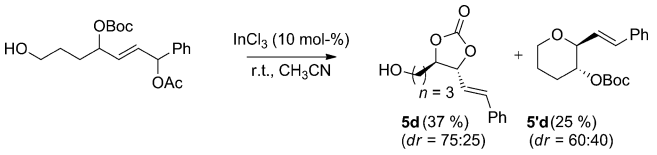
[13] $InCl_3$ was purchased from Sigma–Aldrich (98%).

[14] Surprisingly, in CH_2Cl_2, the use of $InCl_3$ (10 mol-%) led to poor conversion of **1a** (17%).

[15] The reactions became sluggish, and the sole new product observed resulted from a cleavage of the Boc group. Increasing the temperature to 50 or 120 °C under microwave irradiation did not improve the result.

[16] Nucleophilic attack of the hydroxy group took place, delivering the seven-membered ring with no diastereoselectivity in 50% yield; see Supporting Information for more details.

[17] Compound **2j** was formed in 36% yield in the presence of $FeCl_3 \cdot 6H_2O$.
[18] See Supporting Information for details.



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