CHEMISTRY
A EUROPEAN JOURNAL

ChemPubSoc
Europe

DOI: 10.1002/chem.201204386

N-Heterocyclic Carbene (NHC)-Catalyzed/Lewis Acid Mediated Conjugate Umpolung of Alkynyl Aldehydes for the Synthesis of Butenolides: A Formal [3+2] Annulation

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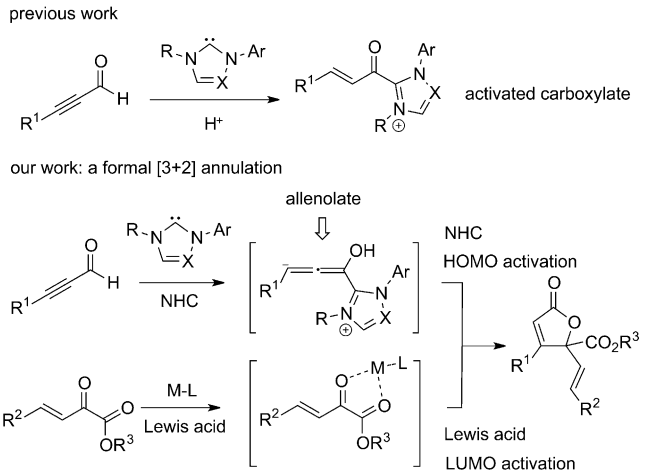
Umpolung reactions catalyzed by N-heterocyclic carbenes (NHCs) have become a fast growing field in the past decades. This polarity reversal strategy is widely used to provide unconventional access to various target molecules.[1] The typical research on this strategy is mostly focused on a^1-to-d^1 umpolung (benzoin condensation)[2] and a^3-to-d^3 umpolung (homoenolate equivalent).[3] In contrast to the large number of publications on nonconjugated aldehydes and enals, the conjugate umpolung, the β-position of alkynyl aldehydes, has received extremely little consideration.[4] In 2006, Zeitler demonstrated the elegant work of a carbene-mediated stereoselective redox esterification of alkynyl aldehydes to give *E*-configurated α,β-unsaturated carboxylic esters.[4a] More recently, Bode[4b] and Xiao[4c] independently reported a NHC-promoted reaction of alkynyl aldehydes with various enols to give functionalized 3,4-dihydropyranones. In their elegant work, the "allenolate" intermediate was trapped with a proton to afford an activated carboxylate for further reactions with different nucleophiles. Intriguingly, however, the addition reactions between the "allenolate" equivalents and electrophilic C=X systems (a formal [3+2] annulation) still pose significant challenges and remain underdeveloped. This can be partly attributed to the fact that the nucleophilic activity of the "allenolate" intermediate is weak and it is difficult to react with an electrophile reagent for the carbon–carbon bond formation. As a long-term research program, our group has devoted itself to the development of new carbon–carbon bond-forming reactions based on a NHC-catalyzed umpolung strategy, and recently we have focused on reactions between this unique "allenolate" equivalent and various electrophile reagents.[5]

The NHC/Lewis acid cooperative catalysis strategies developed by Scheidt's group have been used to provide direct access to new carbon–carbon bond-forming reactions with excellent levels of enantio- and diastereoselectivity.[6] Very recently, Scheidt and co-workers reported the use of lithium chloride as a mild Lewis acid in conjunction with NHC-catalyzed additions of enal homoenolates to isatins, which showed significant enhancement on the level of enantioselectivity in the resultant spirooxindole products.[6f] In all this elegant work, the NHC-catalyzed umpolung reactions give unusual nucleophiles, in which the energy of the HOMO is raised, whilst at the same time the optimal Lewis acid activates electrophiles by lowering the energy of the LUMO. Inspired by all these advances, we have turned our attention to the activation of electrophiles with a suitable Lewis acid to overcome the problems mentioned above. This cooperative catalysis strategy may allow direct access to new carbon–carbon bond formation. In our strategy, we reported the efficient NHC-catalyzed addition of an "allenolate equivalent" to β,γ-unsaturated α-ketoesters to generate butenolides. (Scheme 1)



Scheme 1. NHC-catalyzed/Lewis acid mediated allenolate addition strategy.

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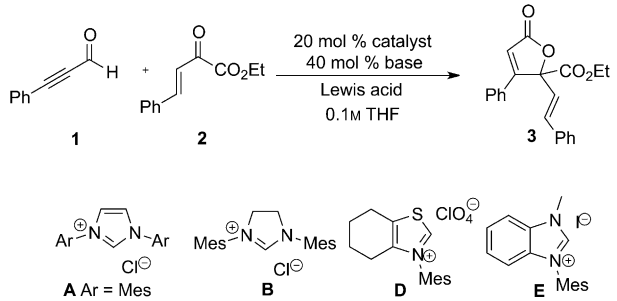
Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/chem.201204386.

We initiated our studies by combining 3-phenylpropiolaldehyde **1** with β,γ-unsaturated α-ketoester **2** in the presence of 20 mol% imidazolium salt **A** and 40 mol% 1,8-diazabicyclo[5.4.0]undec-7-ene (DBU). Under these conditions, butenolide **3** was obtained in a very low yield along with incomplete conversion of the starting materials after 48 h (<5%, Table 1, entry 1). We then focused our attention on the use of a NHC/Lewis acid cooperative catalysis strategy to improve this reaction and lithium chloride was selected as a potential mild Lewis acid additive. Gratifyingly, the addition of one equivalent of lithium chloride provided butenolide **3** with an increased yield of 15% (entry 2). The use of bicyclic guanidine 1,5,7-triazabicyclo[4.4.0]dec-5-ene (TBD) as the base gave a modest increase in yield (entry 3). During our investigation, we found that the utilization of lithium salts as bases was essential for the NHC-catalyzed annulation of the "allenolate" with β,γ-unsaturated α-ketoesters. After an extensive survey of alkali metal salts, the use of 40 mol% lithium *tert*-butoxide improved the reaction significantly (entries 4–10). After having established that lithium *tert*-butoxide was the optimal base, various NHCs were examined to enhance the reaction. The imidazolium salts **C**, **E**, and thiazolium salt **D** were proved to be ineffective. However, the imidazolium salt **B** furnished the desired product in good yield (entry 10). Notably, the addition of one equivalent of LiCl promoted a much faster transformation and the starting materials were consumed completely in just five minutes. With these initial results, we next investigated the effect of different Lewis acids on this reaction. No conversion of the starting materials was observed with magnesium chloride, zinc trifluoromethanesulfonate, and titanium(IV) isopropoxide. Other lithium salts, such as $LiBF_4$ and LiOAc, afforded butenolide **3** in very low yields. The variation of the reaction components, such as stoichiometry, temperature, and solvent also did not improve the yield significantly. When this reaction was performed under our optimized conditions in the absence of the Lewis acid, butenolide **3** was generated in a trace amount (entry 17). This result illustrates the importance of this Lewis acid as a key component.

Table 1. Optimization of the reaction conditions.[a]



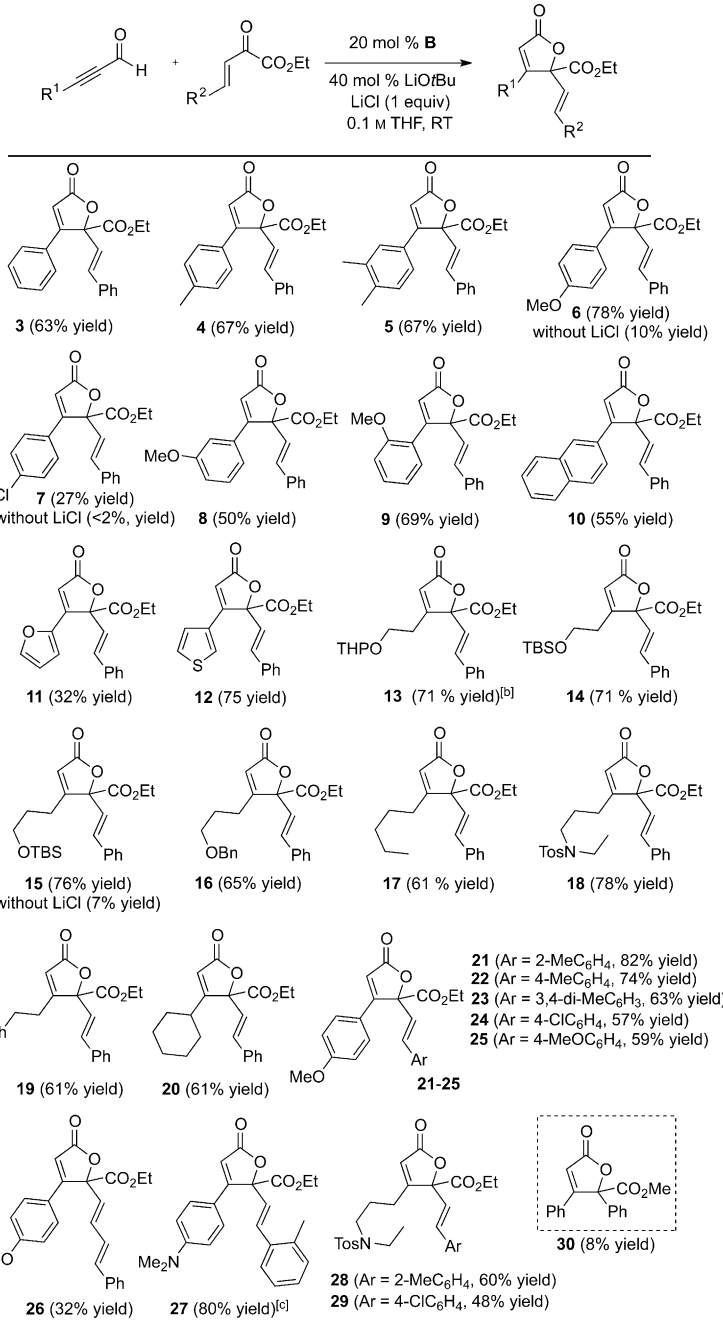
Entry	Catalyst	Base	Lewis acid	Yield [%][b]
1	**A**	DBU	–	<5
2	**A**	DBU	LiCl	15
3	**A**	TBD	LiCl	27
4	**A**	LiHMDS	LiCl	33
5	**A**	KO*t*Bu	LiCl	trace
6	**A**	*n*BuLi	LiCl	27
7	**A**	LDA	LiCl	29
8	**A**	LiO*t*Bu	LiCl	43
9	**A**	Li_2CO_3	LiCl	0[c]
10	**B**	LiO*t*Bu	LiCl	63
11	**C**–**E**	LiO*t*Bu	LiCl	0[c]
12	**B**	LiO*t*Bu	$LiBF_4$	<10
13	**B**	LiO*t*Bu	LiOAc	<10
14	**B**	LiO*t*Bu	$MgCl_2$	0[c]
15	**B**	LiO*t*Bu	$Ti(OiPr)_4$	0[c]
16	**B**	LiO*t*Bu	$Zn(OTf)_2$	0[c]
17	**B**	LiO*t*Bu	–	trace

[a] Reactions conducted with **1** (0.3 mmol) and **2** (0.45 mmol) at room temperature. [b] Yield of isolated product after purification by column chromatography. [c] Most of the starting material was recovered after 24 h. LDA = lithium diisopropylamide, LHMDS = lithium hexamethyldisilazide.

With these optimized reaction conditions in hand, we next turned to a systematic examination of the reaction scope (Table 2). A variety of β-substituted alkynyl aldehydes were evaluated with β,γ-unsaturated α-ketoester **2**. For alkynyl aldehydes bearing β-aryl substituents, electron-withdrawing and -donating substituents on the aromatic ring afforded different results. The substrate with a weak electron-withdrawing group (Cl) on the aromatic ring only gave the desired product in 27% yield, with the starting materials decomposed (**7**). However, electron-donating substrates were well accommodated and afforded the products in moderate to good yields (**4**–**6**, **8**, **9**). The naphthyl-derived alkynyl aldehyde was also tolerated, but the yield was a little lower in this case (**10**). In addition, the 3-furyl-substituted alkynyl aldehyde, which was problematic in earlier umpolung reactions was also accommodated in this transformation (**11**). Gratifyingly, the 3-thieny-substituted alkynyl aldehyde was well tolerated under these conditions and provided the desired product in 75% yield (**12**). To our great delight, the 3-aliphatic-substituted alkynyl aldehyde, which always gave no or low conversion in typical NHC-homoenolate reactions, underwent a smooth reaction to afford the desired products in good yields, significantly expanding the scope of this novel strategy. It is worth noting that the formation of the products **13**–**16** and **18** demonstrates that a heteroatom is accepted in this reaction. Moreover, the employment of alkynyl aldehydes containing *tert*-butyldimethylsilyl (TBS)-/Bn-protected alcohol substituents and a Tos-protected nitrogen substituent also provided another site for further functionalization.

Structural modification of the β,γ-unsaturated α-ketoesters was also explored. Substrates with either electron-withdrawing or -donating groups on the aromatic ring were tested, furnishing the desired products in moderate to good yields (**21**–**25**). When the aryl group of the ester was changed to a vinyl substituent, the yield decreased significantly (**26**). Unfortunately, replacing the aryl substituent with an aliphatic chain gave no conversion of the starting material even with a prolonged reaction time (results not

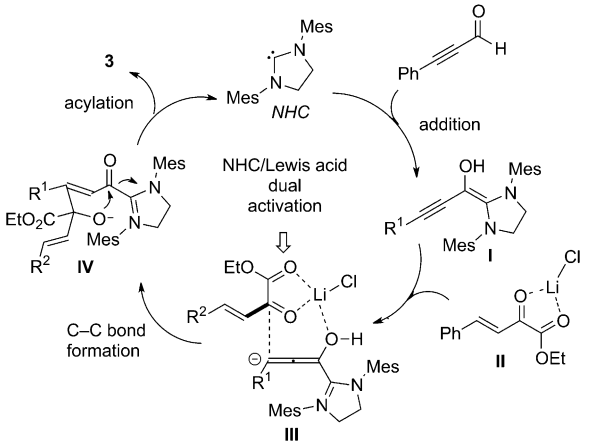
Table 2. Reaction scope.[a]



[a] All reactions were performed on a 0.3 mmol scale. Yield of isolated product after chromatography. [b] A 70% conversion of the aldehyde was observed and the yield is based on recovered material. [c] A 65% conversion of the aldehyde was observed and the yield is based on recovered material. THP = tetrahydropyranyl.

shown). Currently, methyl benzoylformate does not participate well in the reaction.[7] To further identify the importance of Lewis acids in our strategy several experiments without LiCl were also carried out (**6**, **7**, and **15**) and a significant decrease in the yields was observed. These results demonstrated that the Lewis acid effect is general for other substrates.

Our current understanding of the reaction pathway is shown in Scheme 2. The imidazolium precatalyst is deprotonated by the base (LiO*t*Bu) and the resultant carbene



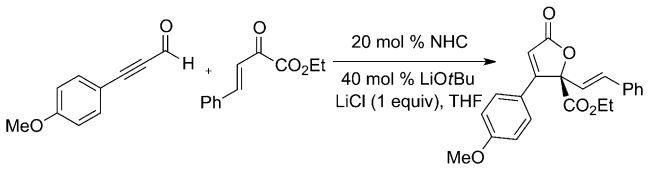
Scheme 2. Proposed catalytic pathway.

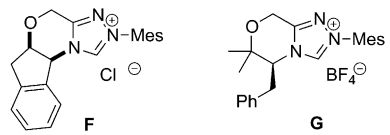
(NHC) adds to the alkynyl aldehyde **1** and induces the formation of the Breslow intermediate I.[8] The Lewis acid (LiCl) concurrently coordinates to the α,β-unsaturated α-ketoester to give II, thereby activating the α-ketoester and promoting the following 1,2-addition. The subsequent coordination of the intermediate II to the Breslow intermediate I brings the α-ketoester electrophile in close proximity to the β-carbon of the "allenolate" intermediate as shown in III (NHC/Lewis acid dual activation). By following the carbon–carbon bond formation and dissociation to give the intermediate IV, this intermediate rapidly undergoes an acylation reaction to afford the butenolide **3** and the NHC catalyst is regenerated.

The asymmetric catalytic version of this formal [3+2] cyclization reaction has also been roughly screened by using commercially available chiral carbenes **F** and **G** as catalysts and the results are depicted in Table 3. Reducing the reaction temperature to −20 °C provided a lower yield but largely improved the enantioselectivity (53.1%, Table 3, entry 3). Although the yields and the enantioselectivities are not high and synthetically practical at the present stage, these results demonstrate that our strategy may provide new opportunities for the stereoselective construction of a privileged heterocyclic system.

With the ability to engage a wide range of alkynyl aldehydes in this novel annulation reaction, we sought to apply the resulting butenolides to other synthetic transformations. For example, treatment of the product **15** with HCl to cleave the silyl protecting group provided the alcohol **31**. Subsequent base-promoted intramolecular oxa-Michael addition afforded the spiro compound **32** in moderate conversion and yield. The chemoselective ozonolysis of the double bond was also tested. Interestingly, the ozonolysis of the double bond afforded compound **33**.[9] This transformation demonstrates that the electron-rich double bond, which is

Table 3. Enantioselective studies of this methodology.[a]



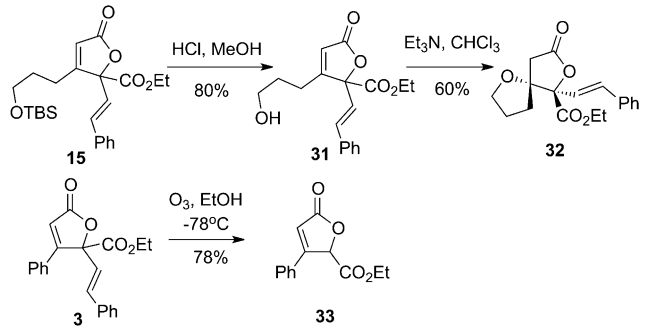


F G

Entry	Catalyst	*T*	Yield [%][b]	*ee* [%][c]
1	**F**	RT	38	21.5
2	**G**	RT	48	33.7
3	**G**	−20 °C	36	53.1

[a] All reactions were performed on a 0.3 mmol scale. [b] Yield of isolated product after chromatography. [c] Enantiomeric excess (*ee*) was determined by HPLC analysis with a chiral stationary phase.

necessary in this novel process, can be easily cleaved for the next synthetic transformations (Scheme 3).



Scheme 3. Synthetic transformations.

In summary, a new formal [3+2] reaction combining alkynyl aldehydes and β,γ-unsaturated α-ketoesters has been first disclosed by using a NHC-catalyzed/Lewis acid mediated strategy. The use of LiCl as a mild Lewis acid is essential for activation of the electrophilic reagents and promotion of the conjugate addition. This novel strategy should lead to many more powerful and highly efficient bond-forming reactions. Meanwhile, this approach allows the synthesis of butenolides with a broader substrate scope in moderate to good yields. In addition, we have demonstrated that using a chiral carbene catalyst in this reaction can lead to the corresponding butenolides with promising levels of enantioselectivity. Moreover, the products derived from the "allenolate-addition" reaction can be easily transformed into other functionalized compounds, which are useful intermediates in contemporary organic synthesis. Studies toward enhancing asymmetric induction and application to other electrophiles are currently ongoing.

Experimental Section

General experimental conditions: Imidazolium salt **B** (21 mg, 0.06 mmol, 0.2 equiv), LiO*t*Bu (10 mg, 0.12 mmol, 0.4 equiv), LiCl (13 mg, 0.3 mmol, 1 equiv), and dry THF (1 mL) were added to a flame-dried round-bottomed flask equipped with a stir bar under argon. The solution was stirred at room temperature for 15 min. Then, a solution of 3-phenylpropiolaldehyde **1** (48 mg, 0.3 mmol, 1.0 equiv) and β,γ-unsaturated α-ketoesters **2** (98 mg, 0.45 mmol, 1.5 equiv) in THF (2 mL) was added to the reaction mixture. After 5 min, the reaction mixture was filtered through a short plug of SiO_2 and eluted with CH_2Cl_2. The solution was concentrated under reduced pressure and purified by flash chromatography (silica gel, 10% EtOAc/petroleum ether) to afford the corresponding product.

Acknowledgements

We are grateful for the generous financial support from the MOST (2010CB833200), PCSIRT: IRT1138, the NSFC (21125207, 21102062, 21072086), and program 111.

Keywords: allenolates · Lewis acids · N-heterocyclic carbenes · organocatalysis · umpolung

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[7] We also tested other electrophiles, such as 4-bromobenzaldehyde and methyl pyruvate in our strategy, but we did not obtain the desired products. When 4-bromobenzaldehyde was used as the electrophile under our conditions, the starting materials were fully recovered. The use of methyl pyruvate as the electrophile lead to a very complex reaction mixture (with or without Lewis acid) and only trace amounts of the product were detected (yield $<2\%$, cannot be isolated). Further investigations in this area are ongoing.

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Received: December 10, 2012
Published online: February 27, 2013

