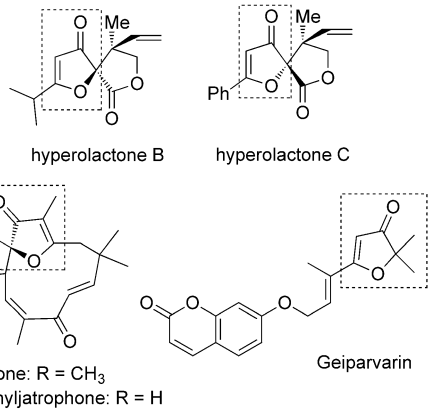
DOI: 10.1002/chem.201102796

From the Feist–Bénary Reaction to Organocatalytic Domino Michael–Alkylation Reactions: Asymmetric Synthesis of 3(2*H*)-Furanones

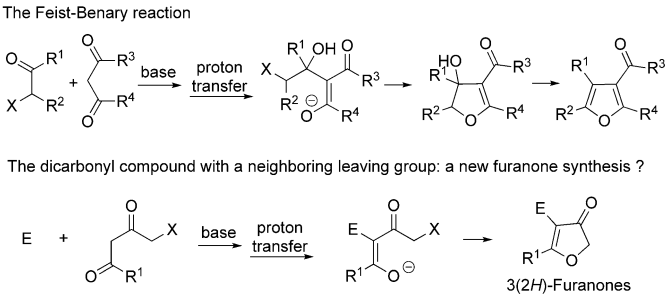
Xiaowei Dou, Xiaoyu Han, and Yixin Lu*[a]

3(2*H*)-Furanones are structural motifs that are widely present in natural products and medicinally important agents (a few examples are illustrated here).[1] Over the past



few decades, a number of approaches toward the synthesis of 3(2*H*)-furanones have been established, including metal-mediated cyclizations of alkynyl substrates,[2a–e] transformations from furans,[2f,g] cyclization of dienes or alkynes,[2h–k] and cycloisomerization of allenes.[2l] However, most of the above reactions require the employment of specific substrates and the reaction conditions are often harsh. Moreover, to the best of our knowledge, an organocatalytic asymmetric synthesis of chiral furanone derivatives has not been reported to date. Thus, there clearly exists a need to devise an efficient and mild synthetic strategy to access this important class of compounds in an optically enriched form.

The Feist–Bénary reaction is a base-catalyzed condensation between α-halogen ketones and 1,3-dicarbonyl compounds for the preparation of substituted furans.[3] The Feist–Bénary synthesis can also be viewed as a domino aldol–alkylation reaction, in which the ketone electrophile plays a key role in the proton transfer, and the presence of the halogen atom is crucial for the cyclization step. Since furanones are furan derivatives, a modified Feist–Bénary reaction may provide a straightforward method for the synthesis of furanones. By employing a γ-halogenated-β-dicarbonyl compound and a suitable electrophile, a modified Feist–Bénary reaction through a Michael–alkylation cascade sequence[4] is anticipated to generate 3(2*H*)-furanones (Scheme 1).



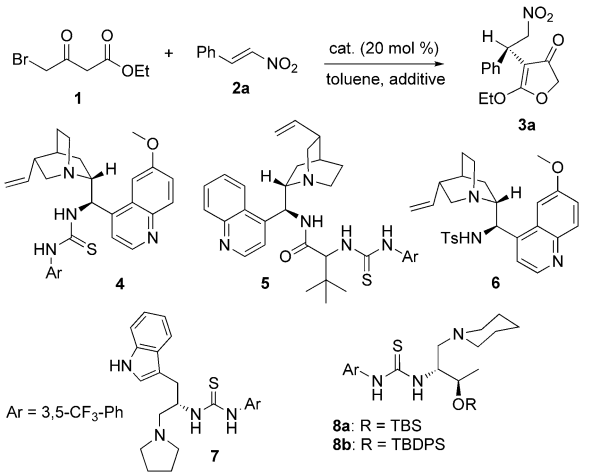
Scheme 1. Synthesis of furanones through a modified Feist–Bénary reaction.

To test the feasibility of our proposal, we chose nitroolefins as potential electrophiles, since they are readily available and versatile electrophiles in conjugate addition.[5,6h,i,l] For the selection of the catalysts, we focused on bifunctional amino catalysts containing a Brønsted acid moiety;[6] promotion of the modified Feist–Bénary reaction could be realized through the interactions of the catalysts with both substrates in a cooperative manner. The domino reaction between ethyl 4-bromoacetoacetate (**1**) and nitrostyrene (**2a**) was selected as a model reaction,[7] and the results are summarized in Table 1. When quinidine-derived tertiary amine/thiourea catalyst **4** was used, the reaction proceeded with very low conversion (entry 1). This result is not surprising, and it suggested that the catalyst was probably quenched by HBr generated during the reaction. To circumvent this problem, we next introduced a stoichiometric amount of base as an additive to capture HBr released. The inclusion of Et_3N or K_2CO_3 into the reaction system proved to be beneficial and the desired products were obtained in very high yields; however, the enantioselectivities were very poor (entries 2 and 3). Suspecting the above bases were strong enough to induce undesired background reactions, $(NH_4)_2CO_3$ was

[a] X. Dou, X. Han, Prof. Dr. Y. Lu
Department of Chemistry & Medicinal Chemistry Program
Life Sciences Institute
National University of Singapore
3 Science Drive 3, Singapore 117543 (Singapore)
Fax: (+65) 6779-1691
E-mail: chmlyx@nus.edu.sg

Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/chem.201102796.

Table 1. Domino Michael–alkylation reaction for the synthesis of 3(2*H*)-furanones.[a]



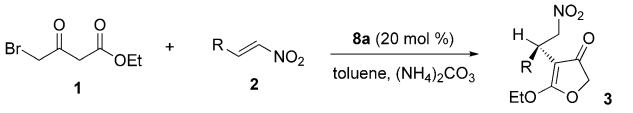
	Catalyst	Additive	*t* [h]	Yield [%][b]	*ee* [%][c]
1	**4**	None	24	< 10	–
2	**4**	Et_3N	0.5	95	−10
3	**4**	K_2CO_3	1	86	−16
4	**4**	$(NH_4)_2CO_3$	16	73	−65
5	**5**	$(NH_4)_2CO_3$	8	82	75
6	**6**	$(NH_4)_2CO_3$	24	60	−61
7	**7**	$(NH_4)_2CO_3$	24	84	85
8	**8a**	$(NH_4)_2CO_3$	24	90	90
9	**8b**	$(NH_4)_2CO_3$	24	85	90

[a] Reactions were performed with **1** (0.05 mmol), **2a** (0.05 mmol), the additive (0.05 mmol) and the catalyst (0.01 mmol) in toluene (0.5 mL) at room temperature. [b] Isolated yield. [c] Determined by HPLC analysis on a chiral stationary phase.

then introduced as a HBr scavenger. Gratifyingly, the desired furanone **3a** was obtained in good yield and with moderate enantioselectivity in the presence of $(NH_4)_2CO_3$ (entry 4). To further improve the enantioselectivity of the reaction, we turned our attention to different types of tertiary amine/thiourea catalysts with an amino acid structural scaffold.[8] Our group recently demonstrated tryptophan-derived tertiary amine/thiourea catalysts[9] and trifunctional catalysts[10] incorporating amino acids were very useful organic catalysts. In addition to these catalysts, we also prepared novel tertiary amine/thiourea catalysts based on L-threonine (catalysts **8a**,**b**, Table 1), a structural scaffold found to be remarkably powerful in a number of asymmetric reactions.[11] While cinchonidine-derived trifunctional catalyst **5** and quinidine-derived sulfonamide[12] **6** gave moderate enantioselectivity (entries 5 and 6), tryptophan-based **7** turned out to be an excellent catalyst, furnishing the desired product with very good enantioselectivity (entry 7). The L-threonine-derived tertiary amine/thiourea catalysts **8** proved to be the most efficient catalysts, and the desired 3(2*H*)-furanone **3a** was obtained in excellent yield and with very high enantioselectivity (entries 8 and 9).

The substrate scope for this novel domino Michael–alkylation reaction was subsequently examined (Table 2). Consistently high yields and excellent enantioselectivities were

Table 2. Enantioselective synthesis of 3(2*H*)-furanones via an **8a**-catalyzed domino Michael–alkylation reaction.[a]



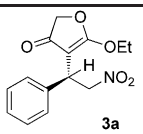
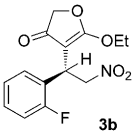
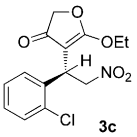
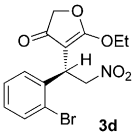
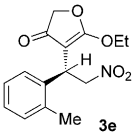
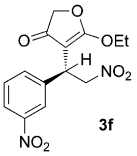
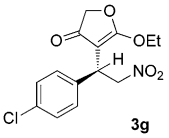
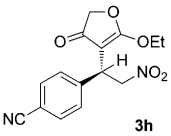
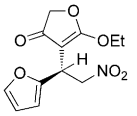
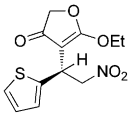
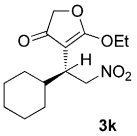
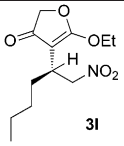
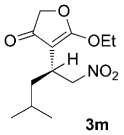
	Product	*t* [h]	Yield [%][b]	*ee* [%][c]
1	 **3a**	24	90	90
2	 **3b**	24	89	94
3	 **3c**	24	88	96
4	 **3d**	24	83	96
5	 **3e**	30	85	94
6	 **3f**	24	86	88
7	 **3g**	24	78	91
8	 **3h**	24	84	95
9	 **3i**	24	85	91
10	 **3j**	30	75	90
11	 **3k**	48	72	87

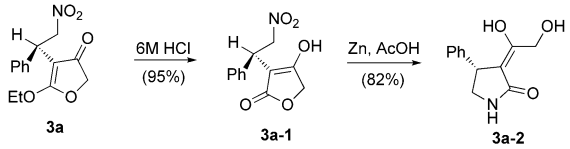
Table 2. (Continued)

	Product	*t* [h]	Yield [%][b]	*ee* [%][c]
12	 **3l**	48	81	91
13	 **3m**	48	78	93

[a] Reaction were performed with **1** (0.05 mmol), **2** (0.05 mmol), $(NH_4)_2CO_3$ (0.05 mmol) and **8a** (0.01 mmol) in toluene (0.5 mL) at room temperature. [b] Isolated yield. [c] Determined by HPLC analysis on a chiral stationary phase.

observed for a wide range of aryl nitroolefins (entries 1–10). Moreover, alkyl nitroolefins were also suitable substrates, and high yields and *ee* values were attained (entries 11–13). The absolute configurations of the 3(2*H*)-furanones were determined on the basis of the X-ray crystal structure of **3d** (see the Supporting Information for details).

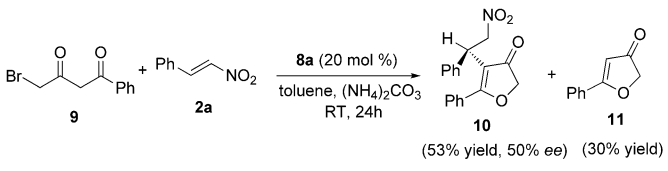
The 3(2*H*)-furanone products **3** are valuable molecules due to the importance of the furanone core in natural product and medicinal chemistry.[1] Moreover, such structures are also valuable synthetic intermediates. As illustrated in Scheme 2, treatment of furanone **3a** with HCl readily gave



Scheme 2. Preparation of tetronic acid and γ-lactam from furanone **3a**.

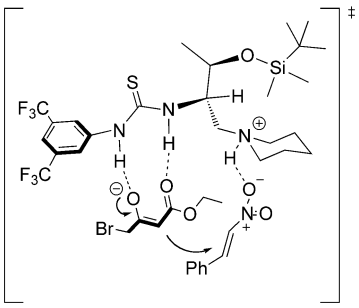
tetronic acid **3a-1**,[13] a core structural skeleton that has been found in many bioactive natural products.[14] Furthermore, reducing the nitro group of the tetronic acid resulted in spontaneous formation of α-alkylidenelactam **3a-2**,[15] an intriguing structural motif possessing a wide variety of biological activities.[16]

In an attempt to broaden the substrate scope, bromodiketone **9** was examined, as illustrated in Scheme 3. Under the optimized reaction conditions established in our previous experiments, the desired furanone **10** was obtained in moderate yield and with only 50 % *ee*. Meanwhile, furanone **11**, which resulted from the self-cyclization of **9**, was obtained in 30 % yield. When **9** was treated with triethylamine alone (2 equiv), product **11** was isolated in 89 % yield, which clearly demonstrated the high tendency of such substrates to undergo rapid cyclization reactions.



Scheme 3. Synthesis of furanone employing bromodiketone instead of bromoketoester

The detailed mechanism of the reaction is not investigated at this stage. A plausible transition state model (**TS**) is shown here. The tertiary amine group of the catalyst deprotonates the β-ketoester to form an enolate, which engages in hydrogen-bonding interactions with the thiourea moiety of the catalyst. The ionic interaction between the positively charged ammonium with the nitroolefin moiety is believed to be important for the substrate binding. Such a proposal is consistent with the theoretical studies carried out in the literature for similar systems.[17]



TS

In summary, we have designed a modified Feist–Bénary reaction by employing a domino Michael–alkylation sequence for the enantioselective preparation of 3(2*H*)-furanones, synthetically useful structural motifs with significant biological importance. We have also prepared L-threonine-derived tertiary amine/thiourea catalysts for the first time; such catalysts promoted the designed domino Michael–alkylation reactions efficiently, affording 3(2*H*)-furanones in high yields and with excellent enantioselectivities. Further development of practical synthetic methods based on the Feist–Bénary reaction to access biologically significant molecules is currently ongoing in our laboratory.

Experimental Section

General procedure: Ethyl 4-bromoacetoacetate **1** (10.5 mg, 0.05 mmol) was added to a mixture of **2a** (7.5 mg, 0.05 mmol), $(NH_4)_2CO_3$ (4.8 mg, 0.05 mmol) and **8a** (5.6 mg, 0.01 mmol) in anhydrous toluene (0.5 mL) in a sample vial. The vial was then sealed and the resulting mixture was stirred at room temperature for 24 h. The reaction mixture was concentrated in vacuo, and the crude was purified by flash column chromatography (ethyl acetate/hexane = 1/5 to 1/1) to afford furanone **3a** (12.5 mg, 90 % yield) as a light yellow oil.

Acknowledgements

We are grateful for the generous financial support from the National University of Singapore (R-143-000-469-112).

Keywords: asymmetric synthesis · domino reactions · Feist–Bénary reaction · furanones · organocatalysis

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Received: September 7, 2011
Published online: December 12, 2011

