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Vilsmeier-Type Reaction of Dimethylaminoalkenoyl Cyclopropanes: One-Pot Access to 2,3-Dihydrofuro [3,2-*c*]pyridin-4(5*H*)-ones

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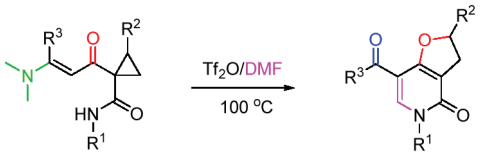
ABSTRACT



R^1: Aryl, Alkyl; R^2: H, Ph; R^3: H, Me; 12 examples, 71-92% yield

A domino reaction of readily available 1-carbamoyl-1-dimethylaminoalkenoylcyclopropanes in the presence of triflic anhydride (Tf_2O) in *N, N*-dimethylformamide (DMF) is described, which provides a facile one-pot access to 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones *via* tandem formylation (Vilsmeier-type reaction), intramolecular cyclization, and ring-enlargement sequences.

2,3-Dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones constitute the core structure of many natural products and synthetic compounds along with a broad range of bioactivities.[1,2] Additionally, 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones can be used as versatile intermediates in organic transformations to furo[3,2-*c*]pyridin-4(5*H*)-ones and other heterocyclic systems.[3] Their pharmacological and synthetic importance has intrigued researchers in search of novel furopyridones from natural products or synthetic approaches for the construction of the skeleton of this type of heterocycle.[4] Sakemi et al. isolated many furopyridones, e.g. CJ-16,170, from the fermentatiom broth of fungus *Cladobotryum varium* CL12284, as a new type of antibiotics (Figure 1).[5] Fukuda et al. reported a series of citridones A, B, B′, and C, isolated from the culture broth of *Penicillium* sp. FKI-1938, which potentiate miconazole activity against *C. albicans* (wide type).[6] Very recently, Liang et al. reported the synthesis of dihydrofuro[3,2-*c*]pyridines from 1-carbamoyl-1-propenoyl cyclopropanes

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via a non-Brook rearrangement and halonium-initiated cascade process, respectively.[7]

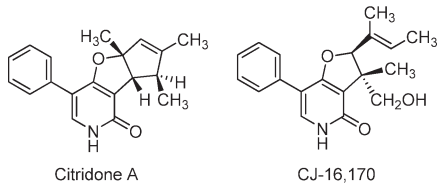


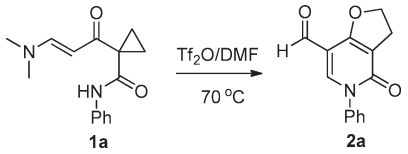
Figure 1. Structures of selected natural products.

During the course of our studies on β-oxo amide derivatives in the synthesis of carbo- and heterocycles, we developed convenient syntheses of substituted phenols, cyclohexenones, 2,3-dihydro-4-pyridones, pyrrolin-4-ones, pyrazolin-5-ones, 2*H*-pyrans, 4*H*-pyrans, and pyridin-2(1*H*)-ones.[8] In our recent work, we achieved the divergent synthesis of dihydrofurans and halogenated pyridin-2(1*H*)-ones from 1-aminopropenoyl-1-carbamoylcyclopropanes derived from β-oxo amides.[9]

In connection with these studies and following with our interest in the synthesis of highly valuable heterocycles, we envisioned that under appropriate conditions a tandem ring enlargement and intramolecular cyclization of 1-aminoalkenoyl-1-carbamoylcyclopropanes may be realized. Thus, the reactions of 1-carbamoyl-1-dimethylaminoalkenoyl cyclopropanes with Vilsmeier reagents were investigated. As a result of these studies, we developed a facile one-pot synthesis of 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones. Herein, we wish to report our experimental results and a proposed mechanism involved in the domino reactions.

The Vilsmeier–Haack reaction, due to its mild reaction conditions, commercial viability of reagents, and the improved understanding of its reaction mechanism, has been widely used for the formylation, halogenation, and dehydroxylation of activated aromatic compounds or carbonyl compounds.[10,11] In our recent work, we have demonstrated the utilization of Vilsmeier–Haack reaction in the synthesis of functionalized pyridin-2(1*H*)-ones, 1*H*-pyrazoles, quinolines, and pyrimidin-4(3*H*)-ones.[12] In the present work, the reaction between 1-aminopropenoyl-1-carbamoylcyclopropane **1a** and a Vilsmeier reagent, $POCl_3$/DMF, was first attempted at rt, in which 4-chloro-3-(2-chloroethyl)-1-phenylpyridin-2(1*H*)-one was obtained as the main product. Yet, a complex mixture was formed when the reaction was performed at elevated temperature, e.g. 100 °C. Of note was that when **1a** was treated with another Vilsmeier reagent, i.e. triflic anhydride (Tf_2O)/DMF, at 70 °C,[13] a product was obtained and characterized as 7-formyl-5-phenyl-2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-one **2a** (Scheme 1).

Scheme 1. Reaction of Aminopropenoyl Cyclopropane **1a** with Tf_2O/DMF



The results encouraged us to investigate the domino reaction of **1a** with other anhydrides in DMF. No reaction was observed by subjecting **1a** to acetic anhydride in DMF at 100 °C, whereas a complex mixture was formed when trifluoroacetic anhydride was employed as indicated by TLC. The reaction of **1a** with phosphorus(V) oxide did proceed, but the conversion to **2a** was very low. A series of experiments revealed that the optimal results were obtained when the reaction of **1a** and 1.5 equiv of Tf_2O was performed in anhydrous DMF at 100 °C for 0.5 h, whereby the yield of **2a** reached 88% (Table 1, entry 1).

Under the optimal conditions as in the case for **2a** in Table 1, a range of reactions of substrates **1** were carried

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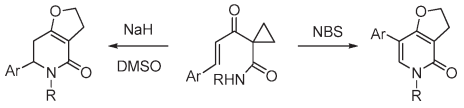

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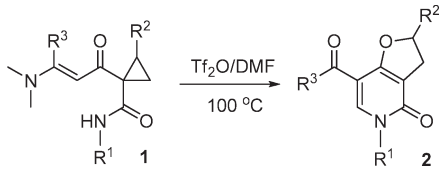
Table 1. Synthesis of 2,3-Dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones **2** from **1**[a]



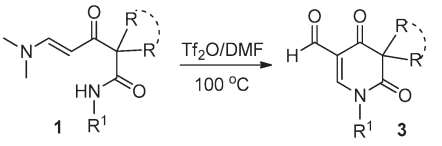
entry	1	R^1	R^2	R^3	2	yield (%)[b]
1	**1a**	Ph	H	H	**2a**	88
2	**1b**	2-MeC_6H_4	H	H	**2b**	86
3	**1c**	4-MeC_6H_4	H	H	**2c**	90
4	**1d**	2,4-$Me_2C_6H_3$	H	H	**2d**	85
5	**1e**	2-$MeOC_6H_4$	H	H	**2e**	90
6	**1f**	4-$MeOC_6H_4$	H	H	**2f**	92
7	**1g**	2-ClC_6H_4	H	H	**2g**	85
8	**1h**	4-ClC_6H_4	H	H	**2h**	87
9	**1i**	4-$CF_3C_6H_4$	H	H	**2i**	82
10	**1j**	Bn	H	H	**2j**	83
11	**1k**	4-MeC_6H_4	Ph	H	**2k**	85
12	**1l**	Ph	H	Me	**2l**	71

[a] Reagents and conditions: **1** (1.0 mmol), Tf_2O (1.5 mmol), DMF (anhydrous, 5.0 mL), 100 °C, 0.5–1.0 h. [b] Isolated yields.

out aiming to determine the scope of the furo[3,2-*c*]pyridin-4(5*H*)-one synthesis, and some of the results are summarized in Table 1. It was found that the reactions of **1b**–**j** bearing variable aryl and alkyl amide groups could proceed efficiently to afford the corresponding 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones **2b**–**j** in high yields (Table 1, entries 2–10). In the case of substrate **1k** with a phenyl group on its cyclopropane ring, the reaction furnished the corresponding 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-one **2k** as a single regioisomer, which suggested that the ring enlargement of the cyclopropyl moiety occurred in a high regioselective manner (Table 1, entry 11). The versatility of this furo[3,2-*c*]pyridin-4(5*H*)-one synthesis was further evaluated by performing the reaction of 1-aminobutenoyl-1-carbamoyl cyclopropane **1l** under the identical conditions (Table 1, entry 12). It was worth noting that 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-one **2l** with an acetyl group on the pyridone ring rather than a formyl group was exclusively obtained in good yield, which also gave a piece of evidence for the mechanism of the domino reaction. The results shown above have demonstrated the efficiency and interest of the cyclization reaction for the synthesis of 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones **2** with respect to substrates **1** bearing variable substituted groups, i.e. R^1, R^2, and R^3. Therefore, we have provided a facile synthesis of substituted furo[3,2-*c*]pyridin-4(5*H*)-one of type **2**.

The above findings inspired us to examine the reaction behavior of analogous 1-aminopropenoyl-1-carbamoyl cyclopentanes **1** under identical conditions. Thus, carbamoylcyclopentane **1m** and Tf_2O (1.5 equiv) were subjected to anhydrous DMF at 100 °C. To our delight, the reaction proceeded smoothly and furnished a product, which was characterized as 7-azaspiro[4.5]dec-8-ene-6,10-dione **3a** (Table 2, entry 1). In the same fashion, a range of reactions of 1-aminopropenoyl-1-carbamoylcyclopentanes **1** bearing variable *N*-aryl and alkyl groups were carried out, and the corresponding 7-azaspiro[4.5]dec-8-ene-6,10-diones **3b**–**g** were obtained in high yields (Table 2, entries 2–7). In the cases of substrates 5-amino-2,2-dialkyl-3-oxo-pent-4-enamides **1t** and **1u**, pyridine-2,4(1*H*,3*H*)-diones **3h** and **3i** were obtained, respectively (Table 2, entries 8 and 9). These results suggested that the cyclization of **1m**–**u** might proceed in the same manner as 1-alkenoyl-1-carbamoylcyclopropanes **1a**–**l** did in the presence of Tf_2O in DMF.

Table 2. Synthesis of Pyridine-2,4(1*H*,3*H*)-diones **3**[a]



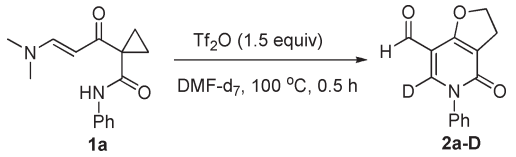
entry	1	R^1	R (or R⋯R)	3	yield (%)[b]
1	**1m**	Ph	$(CH_2)_4$	**3a**	91
2	**1n**	2-MeC_6H_4	$(CH_2)_4$	**3b**	87
3	**1o**	4-MeC_6H_4	$(CH_2)_4$	**3c**	90
4	**1p**	2-$MeOC_6H_4$	$(CH_2)_4$	**3d**	86
5	**1q**	2-ClC_6H_4	$(CH_2)_4$	**3e**	83
6	**1r**	4-ClC_6H_4	$(CH_2)_4$	**3f**	87
7	**1s**	Bn	$(CH_2)_4$	**3g**	82
8	**1t**	Ph	Me	**3h**	85
9	**1u**	Ph	Et	**3i**	84

[a] Reagents and conditions: **1** (1.0 mmol), DMF (anhydrous, 5.0 mL), Tf_2O (1.5 mmol), 100 °C, 0.5–1.0 h. [b] Isolated yields.

To gain insight into the mechanism of the furo[3,2-*c*]pyridin-4(5*H*)-one synthesis from 1-aminoalkenoyl-1-carbamoylcyclopropanes, a separated reaction of **1a** and Tf_2O (1.5 equiv) was conducted in DMF-d_7 at 100 °C,[14] and deuterated product **2a-D** and **2a** were obtained in a 86% yield with a molar ratio of 9:1 (Scheme 2; see Supporting Information). The structure of **2a-D** was established by comparison of its NMR spectra with those of **2a**. In the ^1H NMR spectrum, **2a** displayed two single peaks at 9.81 and 8.02 ppm, which were assigned to formyl-H and 6-H from the furo[3,2-*c*]pyridin-4(5*H*)-one ring, respectively. With respect to **2a-D**, the peak at 8.02 ppm for 6-H disappeared, indicating the formylation should occur on the N-atom of the amide moiety. In the ^{13}C NMR spectrum, the formyl-C and 6-C of **2a** were indicated at 184.6 and 145.6 ppm, respectively. As for **2a-D**, there was almost no change on the chemical shifts of these two peaks, but the intensity of the peak at 145.6 became weaker, which is consistent with the above ^1H NMR results. The fact that **2a** appeared within this system might be attributed to the small amount of nondeuterated DMF in the reaction medium.

(14) Reagents and conditions: **1a** (0.5 mmol), Tf_2O (0.75 mmol), DMF-d_7 (99.5%, 1.0 mL), 100 °C, 0.5 h.

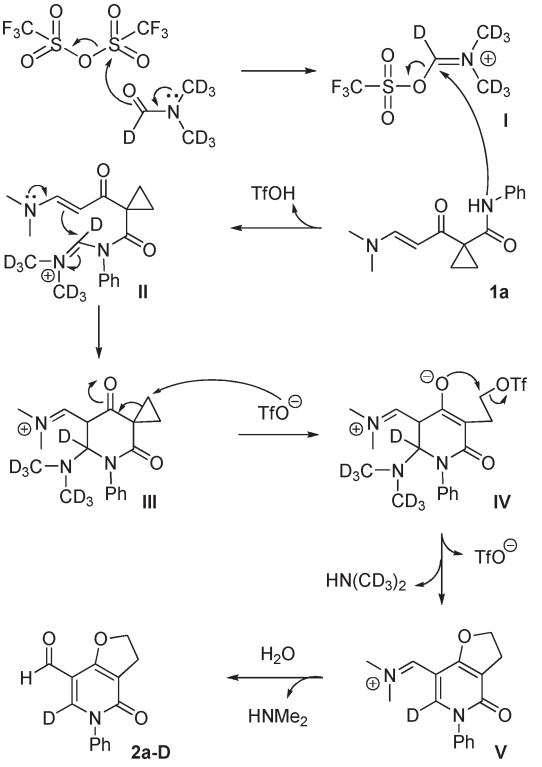
Scheme 2. Reaction of Aminopropenoyl Cyclopropane **1a** with Tf_2O/DMF-d_7



On the basis of the above experimental results together with some literature reports, a plausible mechanism for the synthesis of 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones **2** is proposed as depicted in Scheme 3 (with the reaction of **1a** in DMF-d_7 as an example). The secondary amide DMF-d_7 is activated with Tf_2O to generate a very reactive iminium triflate **I**,[15−17] which can be regarded as a Vilsmeier-type reagent. Mediated by **I**, substrate **1a** undergoes formylation to afford iminium salt **II**. Followed by a tandem intramolecular cyclization reaction[10,12a,12b] and ring-enlargement reaction,[18] **II** sheds dimethylamine to give rise to iminium intermediate **V**, which is finally converted into 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-one **2a-D** upon treatment with water.

In summary, a facile and efficient access to substituted 2,3-dihydrofuro[3,2-*c*]pyridin-4(5*H*)-ones **2** is developed *via* a domino reaction of readily available 1-aminoalkenoyl-1-carbamoylcyclopropanes in the presence of Tf_2O in DMF. The Vilsmeier-type reaction was further utilized for the synthesis of spiro or substituted pyridine-2,4(1*H*,3*H*)-diones **3** from

Scheme 3. Plausible Mechanism for the Domino Reaction of **1** with Tf_2O/DMF



1-aminoalkenoyl-1-carbamoylcyclopentanes and 5-amino-2,2-alkyl-3-oxo-pent-4-enamides, respectively. The one-pot protocol is associated with readily available substrates, mild conditions, high yields, and a wide range of products with synthetic potential. Further work on the reaction mechanism and extension of the scope of the present protocol is currently underway in our laboratory.

Acknowledgment. Financial support of this research by the National Natural Science Foundation of China (20872136 and 21172211) is greatly acknowledged.

Supporting Information Available. Experimental details, full characterization data, and copies of NMR spectra for compounds **2** and **3**. This materials are available free of charge via the Internet at http://pubs.acs.org.

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