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Advanced Synthesis & Catalysis

Synthesis of 2-Alkenyl-4*H*-3,1-Benzoxazin-4-Ones through HFIP-Mediated Decarboxylative [4+2]-Annulation of Isatoic Anhydrides with Cyclopropenones under Silver Catalysis

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Abstract: We herein describes an HFIP-mediated [4+2]-cycloaddition reaction from simple and easily available isatoic anhydrides and cyclopropenones under silver catalysis. This transformation involves the tandem decarboxylative esterification, intermolecular addition, intramolecular substitution, small ring opening and isomerization processes, which allows the rapid assembly of versatile 2-diarylalkenyl-4*H*-3,1-benzoxazin-4-ones.

Keywords: isatoic anhydride; cyclopropenone; hexafluoro-2-propanol; cycloaddition; 4*H*-3,1-benzoxazin-4-one

4*H*-3,1-Benzoxazin-4-ones represent a class of important *N*-heterocycles of considerable interest, and have been used as versatile and valuable precursors for the preparation of pharmaceutically active compounds.[1] As a particularly branch, 2-alkenyl-substituted 4*H*-3,1-benzoxazin-4-ones and their derivatives exhibit a variety of biological activities such as human leukocyte elastase inhibitor, acaricide, fibrosis inhibitor and antagonist, and therefore have received much attention in recent years (Figure 1).[2]

In view of their high biological activity, the construction of common moieties such as alkyl or aryl substituents at C2 position on the 4*H*-3,1-benzoxazin-4-one framework were extensively investigated in the past decades,[3] whereas the synthesis of 2-alkenyl-substituted 4*H*-3,1-benzoxazin-4-ones has been comparatively less studied. In this context, one general approach to 2-alkenyl-4*H*-3,1-benzoxazin-4-ones was successively reported via palladium-catalyzed cyclocarbonylation reaction of *o*-haloanilines with unsaturated halides or triflates under an atmosphere of carbon monoxide (Scheme 1a).[4] The other route to access to 2-alkenyl-4*H*-3,1-benzoxazin-4-ones was recently achieved from *N*-(2-carboxyphenyl)- and *N*-(2-formylphenyl)acrylamides through the intramolecular cyclization strategy (Scheme 1b).[5] Despite the progress, the availability of substrates and required reaction conditions (e.g. CO atmosphere, external oxidant, strong acidic or basic medium, microwave) makes these synthetic methods of limited utility to some extent. Based on our ongoing interest in versatile nitrogen-containing heterocyclic constructions,[6] we herein wish to present an intermolecular synthesis of 2-alkenyl-substituted 4*H*-3,1-benzoxazin-4-ones from easily available isatoic anhydrides and cyclopropenones.[7] Notably, isatoic anhydrides underwent decarboxylative esterification in the presence of hexafluoro-2-propanol

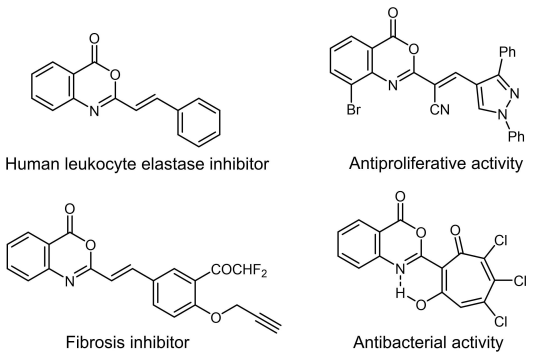
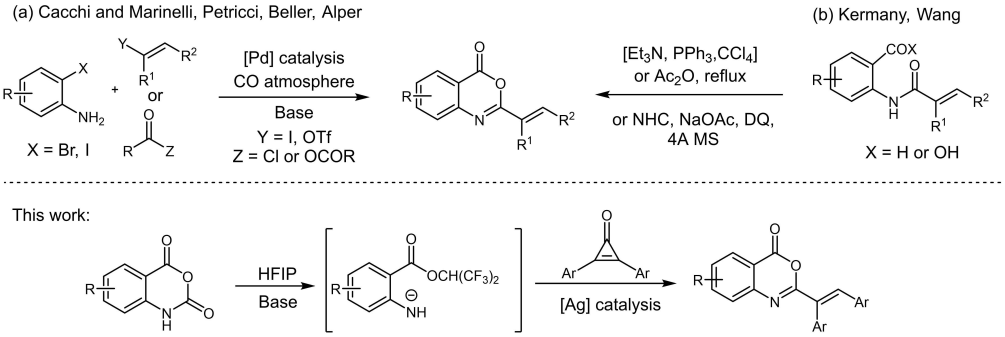


Figure 1. The representative 2-alkenyl-4*H*-3,1-benzoxazin-4-ones and their related derivatives of biological interest.

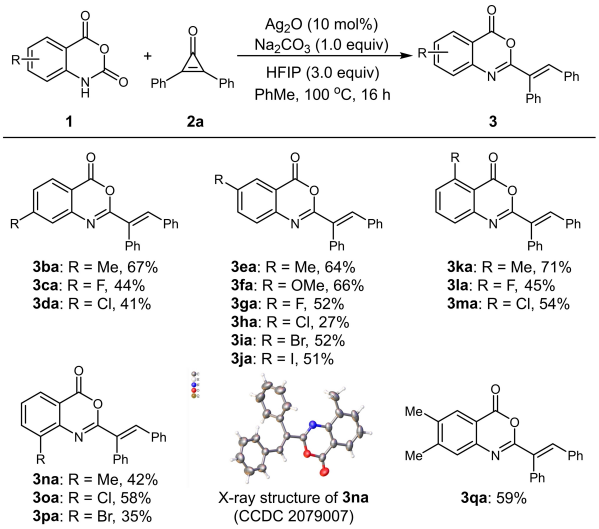


Scheme 1. Synthetic protocols of 2-alkenyl-4*H*-3,1-benzoxazin-4-ones.

(HFIP) to generate the nitrogen anion intermediate in this transformation,[8] which is prone to the [4+2] cycloaddition with cyclopropenones through nucleophilic attack under silver catalysis to allow the assembly of versatile 2-diarylalkenyl-4*H*-3,1-benzoxazin-4-one products (Scheme 1).

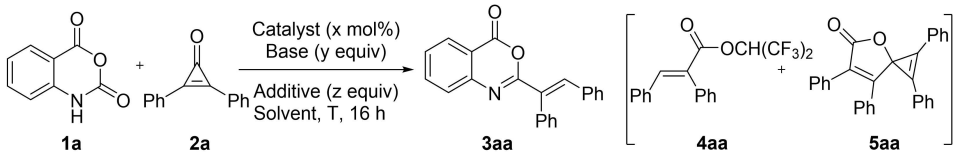
Our initial experiments were carried out by using isatoic anhydride (**1a**, 0.1 mmol) and diphenylcyclopropenone (**2a**, 0.1 mmol) as the model substrates, as shown in Table 1. No reaction was observed in the presence of Ag_2O as catalyst and Na_2CO_3 as base in PhMe at 120 °C for 16 h (entry 1), and only trace amounts of the target product **3aa** was obtained without silver catalysis (entry 2). To our delight, the current decarboxylative cycloaddition proceed smoothly when HFIP was loaded as the efficient additives,[8] which delivered the 2-alkenyl-4*H*-3,1-benzoxazin-4-one product **3aa** in 27% isolated yield (entry 3). At the same time, the undesired α,β-unsaturated ester product **4aa** from the nucleophilic addition of cyclopropenone **2a** with HFIP and the spirolactone product **5aa** were observed from dimerization of cyclopropenone **2a** in 7% and 5% yields, respectively, and the similar results were observed in the following reaction condition screenings as well as in the substrate scope investigations, which reduced the efficiency of the desired [4+2] cyclization processes to some extent. Furthermore, the examination of other alcohol additives such as *i*-PrOH, *t*-BuOH and Me_3COH only generated **3aa** in no more than 10% yields (entries 4–6). Besides, the yield of **3aa** was no further improvement by either increasing or decreasing the loadings of HFIP, or even in place of PhMe as the reaction solvent (entries 7–9). Besides, investment of silver and other metal salts, such as AgOTf, CF_3COOAg, AgOAc, $AgNO_3$, Ag_2CO_3, $Cu(OTf)_2$, $Cu(OAc)_2$, CuI, $Sc(OTf)_3$ and $FeCl_3$ proved Ag_2O to be the best (entry 3 *vs* entries 10–19). With Ag_2O as catalyst, various inorganic and organic bases were next tested (entries 3 and 20–25), and the experimental results showed that the current transformation efficiently proceeded in the presence of Na_2CO_3 (entry 3). Furthermore, we screened different kinds of solvents including PhMe, $PhCF_3$, 1,4-dioxane and MeCN, and found that the expected product **3aa** could be obtained only under the conditions of PhMe and $PhCF_3$ (entries 26–30). Finally, the best performance of the cycloaddition was observed in 84% yield when 3.0 equivalents of diphenylcyclopropenone (**2a**) was loaded into the reaction system in three times at intervals of 2 h (entries 31–32).

Having acquired the optimal reaction conditions (Table 1, entry 32), we investigated the cycloaddition reaction of isatoic anhydrides **1** with diphenylcyclopropenone (**2a**), as shown in Scheme 2. Generally, isatoic anhydrides **1** bearing electron-donating and electron withdrawing substituents in different positions of the phenyl rings were converted into the 2-diphenylalkenyl-substituted 4*H*-3,1-benzoxazin-4-one products **3** in moderate to good yields, in which substrates **1** with electron-donating groups behave better reactivity than those with electron-withdrawing groups. Moreover, the reaction of diphenylcyclopropenone (**2a**) with a variety



Scheme 2. Substrate scope of isatoic anhydrides **1**.

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

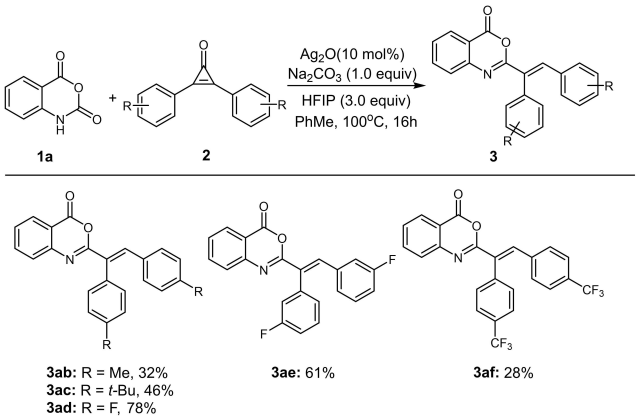


Entry	Catalyst (x)	Base (y)	Additive (z)	Solvent	T [°C]	**3aa** [%][b]
1	Ag_2O (10)	Na_2CO_3 (2)	–	PhMe	120	N.R.
2	–	Na_2CO_3 (2)	HFIP (3)	PhMe	120	Trace
3	Ag_2O (10)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	27
4	Ag_2O (10)	Na_2CO_3 (2)	*i*-PrOH (3)	PhMe	120	5
5	Ag_2O (10)	Na_2CO_3 (2)	*t*-BuOH (3)	PhMe	120	10
6	Ag_2O (10)	Na_2CO_3 (2)	Me_3COH (3)	PhMe	120	4
7	Ag_2O (10)	Na_2CO_3 (2)	HFIP (2)	PhMe	120	18
8	Ag_2O (10)	Na_2CO_3 (2)	HFIP (4)	PhMe	120	21
9	Ag_2O (10)	Na_2CO_3 (2)	–	HFIP	120	N.R.
10	AgOTf (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	23
11	CF_3CO_2Ag (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	8
12	AgOAc (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	10
13	$AgNO_3$ (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	15
14	Ag_2CO_3 (10)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	10
15	$Cu(OTf)_2$ (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	N.R.
16	$Cu(OAc)_2$ (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	Trace
17	CuI (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	4
18	$Sc(OTf)_3$ (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	6
19	$FeCl_3$ (20)	Na_2CO_3 (2)	HFIP (3)	PhMe	120	N.R.
20	Ag_2O (10)	K_2CO_3 (2)	HFIP (3)	PhMe	120	Trace
21	Ag_2O (10)	Cs_2CO_3 (2)	HFIP (3)	PhMe	120	Trace
22	Ag_2O (10)	NaOAc (2)	HFIP (3)	PhMe	120	Trace
23	Ag_2O (10)	*t*-BuONa (2)	HFIP (3)	PhMe	120	N.R.
24	Ag_2O (10)	Et_3N (2)	HFIP (3)	PhMe	120	Trace
25	Ag_2O (10)	DBU (2)	HFIP (3)	PhMe	120	Trace
26	Ag_2O (10)	Na_2CO_3 (1)	HFIP (3)	PhMe	120	42
27	Ag_2O (10)	Na_2CO_3 (1)	HFIP (3)	$PhCF_3$	120	42
28	Ag_2O (10)	Na_2CO_3 (1)	HFIP (3)	1,4-Dioxane	120	N.R.
29	Ag_2O (10)	Na_2CO_3 (1)	HFIP (3)	MeCN	120	Trace
30[c]	Ag_2O (10)	Na_2CO_3 (1)	HFIP (3)	PhMe	120	56
31[d]	Ag_2O (10)	Na_2CO_3 (1)	HFIP (3)	PhMe	120	76
32[d]	Ag_2O (10)	Na_2CO_3 (1)	HFIP (3)	PhMe	100	84

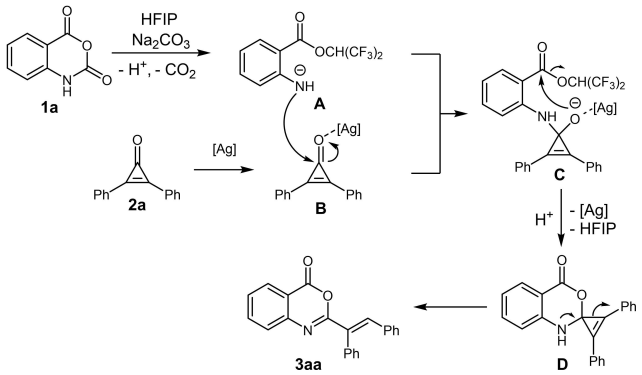
[a] Reaction conditions: **1a** (0.1 mmol), **2a** (0.1–0.3 mmol), Catalyst (10–20 mol%), Base (1.0–2.0 equiv.), HFIP (2.0–5.0 equiv.) and PhMe (1.0 mL) under N_2 for 16 h.
[b] Isolated yields.
[c] **2a** (0.3 mmol).
[d] **2a** (0.3 mmol) was added three times at intervals of 2 h, and then further reacted for 10 h. HFIP = Hexafluoro-2-propanol. DBU = 1,8-Diazabicyclo[5.4.0]undec-7-ene. N.R. = No reaction.

of the halo-substituted isatoic anhydrides **1** steadily delivered the corresponding products **3**, which might allow for the late-stage modifications through transition-metal-catalyzed couplings. Unfortunately, heterocyclic-type substrates, such as 2*H*-pyrido[2,3-*d*][1,3]oxazine-2,4(1*H*)-dione (**1r**), 2*H*-pyrido[3,4-*d*][1,3]oxazine-2,4(1*H*)-dione (**1s**) and 2*H*-thieno[2,3-*d*][1,3]oxazine-2,4(1*H*)-dione (**1t**) were failed to deliver the desired annulation products **3ra**–**ta** under the standard reaction conditions. The structure of compound **3na** were unambiguously confirmed by X-ray single crystal diffraction.[9]

In order to further evaluate the scope of this transformation, several substituted cyclopropenones **2** were tested. As shown in Scheme 3, substrates **2** bearing alkyl, fluoro and trifluoromethyl substituents could proceed smoothly with isatoic anhydrides **1** under the standard conditions. In contrast to isatoic anhydrides **1**, cyclopropenones **2d** and **2e** with fluoro groups behaved better reactivity than those with alkyl groups (**2b** and **2c**). To our disappointment, dithio-



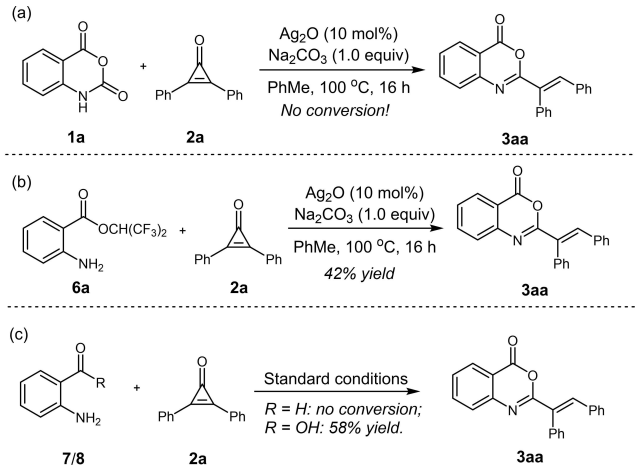
Scheme 3. Substrate scope of cyclopropenones **2**.



Scheme 5. Proposed mechanism.

phene-substituted cyclopropenone **2g** only delivered the corresponding annulation product **3ag** in 4% yield.

To gain insight into the mechanism of this cycloaddition reaction, preliminary experiments were conducted. As mention above, no target product **3** was formed in the model reaction of **1a** and **2a** without HFIP additives in this catalytic system (Scheme 4a), and meanwhile, intermediate **6a** was always detected in the presence of HFIP. Therefore, intermediate **6a** was isolated and was further loaded into the cycloaddition with cyclopropenone **2a** under silver catalysis in the absence of HFIP. Thus, the target product **3aa** was successfully obtained with a yield of 42%, indicating **6a** as a key intermediate in the present transformation (Scheme 4b). Besides, the assumptive intermediates 2-aminobenzoic acid (**7**) and 2-aminobenzaldehyde (**8**) instead of isatoic anhydride (**1a**) were introduced into the standard reaction conditions, and the desired product **3aa** was only obtained with **8** in 58% yield, indicating the unique of the present synthetic protocol from isatoic anhydrides **1** (Scheme 4c).



Scheme 4. Preliminary mechanistic studies.

On the basis of the preliminary experimental results and the previous literature reports,[5] a plausible reaction mechanism is presented in Scheme 5. First, isatoic anhydride **1a** underwent decarboxylative esterification with HFIP to generate intermediate **A** under basic conditions. Subsequently, an intermolecular nucleophilic attack from intermediate **A** to the carbonyl group of diphenylcyclopropenone **2a** with the assistance of silver catalyst gave intermediate **C**, which immediately underwent an intramolecular nucleophilic substitution to accomplish the [4+2] cyclization. Finally, the three-membered-ring opening and isomerization processes from the thus formed intermediate **D** afforded the desired 2-alkenyl substituted 4*H*-3,1-benzoxazin-4-one product **3aa**.

In order to explore the application of the 2-alkenyl-4*H*-3,1-benzoxazin-4-one products **3**, their fluorescence emission characteristics were preliminarily investigated. Unfortunately, all the obtained compounds **3** were failed to exhibit characteristics of aggregation-induced emission (AIE, for selected examples see the supporting information).[10]

In summary, we have developed an HFIP-mediated [4+2]-cycloaddition reaction from easily available isatoic anhydrides and cyclopropenones under silver catalysis. The reaction involves the successive transformations of decarboxylative esterification of HFIP to isatoic anhydrides, intermolecular 1,2-addition to cyclopropenones, intramolecular substitution, small ring opening and isomerization to afford the 2-alkenyl-4*H*-3,1-benzoxazin-4-one products.

Experimental Section

The representative procedure for the preparation of compounds 3

Isatoic anhydride **1** (0.1 mmol, 1.0 equiv.), HFIP (0.3 mmol, 3.0 equiv.), Ag_2O (0.01 mmol, 10 mol%) and Na_2CO_3

(0.1 mmol, 1 equiv.) were added sequentially to a flame-dried Schlenk tube and was dissolved in PhMe (1.0 mL) at room temperature under N_2 atmosphere. Then, cyclopropenone **2** (0.3 mmol, 3.0 equiv.) was added three times at intervals of 2 h at 100 °C. After cyclopropenone **2** was completely added, the mixture was further stirred at the same temperature for 10 h. After reaction completion, the solvent was removed under reduced pressure and the resulting residue was purified by column chromatography on silica gel with petroleum ether/ethyl acetate (v/v) afforded the corresponding product **3**.

Acknowledgements

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[9] CCDC 2079007 (**3 na**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre.
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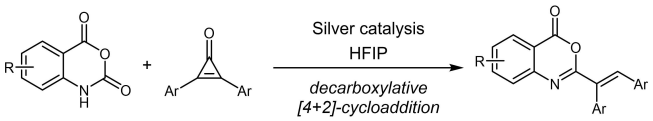
Synthesis of 2-Alkenyl-4*H*-3,1-Benzoxazin-4-Ones through HFIP-Mediated Decarboxylative [4 + 2]-Annulation of Isatoic Anhydrides with Cyclopropenones under Silver Catalysis



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