Efficient and Highly Selective Method for the Synthesis of Benzo(naphtho)quinoline Derivatives Catalyzed by Iodine

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A mild, efficient, and highly selective method for the synthesis of pyranoquinoline and furoquinoline derivatives via a three-component reaction of aromatic aldehyde, naphthalen-2-amine or anthracen-2-amine and 2,3-dihydrofuran, or 3,4-dihydro-2*H*-pyran catalyzed by iodine is described. It should be noted that *exo*-isomer was obtained with high selectivity in good yields, which was confirmed by X-ray diffraction analysis. The 3-arylbenzo(naphtha)[*f*]quinolines were isolated in high yields when the chain *n*-butylvinyl ether was used as reactant, with the unexpected loss of the butoxy group.

1. Introduction

Control of selectivity, for example, chemo-, stereo, and regioselectivity, is among the most important objectives in organic chemistry. Many different process parameters such as temperature, pressure, solvent, as well as catalyst type, and other factors can be utilized to modulate the selectivity of synthetic transformations.[1] Multicomponent reactions (MCRs) play an important role in modern synthetic organic chemistry since they generally occur in a single pot and exhibit a high atom-economy and selectivity. They also deliver fewer byproducts compared to classical stepwise synthetic routes.[2] They provide a powerful tool toward the one-pot synthesis of diverse and complex compounds as well as small and drug-like heterocycles.[3] Owing to their convergence and productivity, the MCRs have attracted considerable attention from the point of view of combinatorial chemistry.[4] For multicomponent reactions involving the simultaneous molecular interaction of three or more components, the issue of selectivity is of particular significance because of the high probability of several potential parallel reaction pathways leading to different product classes.[5]

Pyranoquinoline derivatives are found to possess a wide spectrum of biological activities, such as psychotropic, antiallergenic, anti-inflammatory and estrogenic activity.[6] The imino-Diels−Alder reaction provides easy access to the preparation of pyrano and furoquinolines. The imines derived from aromatic amines act as heterodienes and undergo imino-Diels−Alder reaction with various dienophiles in the presence of acid catalysts.[7−10] However, many of these synthetic protocols reported so far suffer from disadvantages, such as harsh reaction conditions, multistep reaction, use of metals and expensive reagents, poor stereoselectivity, and so forth. Especially for the latter, most of the reported procedures always give a mixture of *endo* and *exo*-isomers[11] using 2,3-dihydrofuran or 3,4-dihydro-2*H*-pyran as starting materials. Therefore, the development of a system with reagent economy, metal free, one-pot, and good stereoselectivity is desirable.

In our previous paper, we have synthesized series of benzo[*f*]quinoline derivatives through three-component reactions of aromatic aldehyde, various ketones, and naphthalen-2-amine.[12] It was found that naphthalen-2-amine involved-reaction always gave the desired products in high yields and good stereoselectivity for its higher activity. In connection with our previous research on this iodine-catalyzed reaction and for the challenge of stereoselectivity of above-mentioned imino-Diels−Alder reaction, naphthalen-2-amine or anthracen-2-amine was selected as similar aromatic amine to react with aromatic aldehyde and 2,3-dihydrofuran or 3,4-dihydro-2*H*-pyran. It was noteworthy that *exo*-4-arylbenzo[*f*]furo [3,2-*c*]quinoline, pyrano[3,2-*c*]quinoline, naphtho[2,3-*f*]furo[3,2-*c*]quinoline or naphtho[2,3-*f*] pyrano[3,2-*c*]quinoline derivatives were obtained with high selectivity in good yields. 3-Arylbenzo[*f*]quinolines or 3-arylnaphtho[2,3-*f*]quinolines were obtained in high yields when the chain *n*-butylvinyl ether was selected as dienophile, with the unexpected loss of the butoxy group.

2. Results and Discussion

Treatment of aromatic aldehyde **1**, naphthalen-2-amine **2**, and 2,3-dihydrofuran **3** in tetrahydrofuran (THF) in the presence of 10 mol % iodine at reflux condition afforded the corresponding *exo*-4-arylbenzo[*f*]furo[3,2-*c*]quinoline derivatives **4** in good yields with high stereoselectivity (Scheme 1). Obviously, these results were different from *endo*- and *exo*-isomers catalyzed by PMA, $SbCl_3$−HAP,

Scheme 1. Reaction of **1**, **2** and 2,3-Dihydrofuran

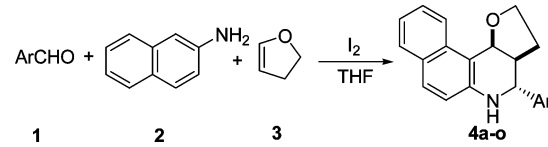

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Table 1. Synthetic Results of **4a** under Different Reaction Conditions[a]

entry	temp. (°C)	catalyst (mol %)	solvent	yields (%)[b]
1	r.t.	0	THF	0
2	reflux	0	THF	0
3	r.t.	I_2(5)	THF	trace
4	50	I_2(5)	THF	68
5	reflux	I_2(5)	THF	84
6	reflux	I_2(10)	THF	83
7	reflux	I_2(20)	THF	83
8	reflux	I_2(5)	CH_3CN	72
9	reflux	I_2(5)	benzene	82
10	80	I_2(5)	DMF	75
11	reflux	I_2(5)	$CHCl_3$	76
12	r.t.	I_2(15)	CH_2Cl_2	72(68:32)[c]
13	r.t.	$H_3PMO_{12}O_{40}$(1)	none	92(8:12)[d]
14	r.t.	$SbCl_3$(10)	CH_3CN	90(28:72)[e]

[a] Reagents and conditions: 4-nitrobenzaldehyde **1a** (0.302 g, 2 mmol), **2** (0.286 g, 2 mmol), **3** 2,3-dihydrofuran (0.210 g, 3 mmol), solvent (10 mL). [b] Isolated yields. [c] Reference 11e, benzaldehyde (1 mmol), aniline (1 mmol), 2,3-dihydrofuran (2 mmol), CH_2Cl_2, r.t. the 68:32 is the ratio of *exo*/*endo*. [d] Reference 11a, benzaldehyde (1.5 mmol), aniline (1.5 mmol), 2,3-dihydrofuran (2 mmol), solvent free, r.t. the 8:12 is the ratio of *exo*/*endo*. [e] Reference 11c, benzaldehyde (2 mmol), aniline (2.2 mmol), 2,3-dihydrofuran (2.6 mmol), CH_3CN, r.t. the 28:72 is the ratio of *exo*/*endo*.

$SbCl_3$ or iodine using simple aromatic amines as reactants (Table 1, Entries 12−14).[11]

In our initial study, the reaction of 4-nitrobenzaldehyde **1a**, naphthalen-2-amine **2**, and 2,3-dihydrofuran **3** was used as a model reaction to optimize the reaction conditions. The reaction was first carried out in THF in the absence of I_2. It was found that no reaction occurred at room temperature or reflux condition (Table 1, entries 1 and 2). Similar reactions were attempted in the presence of 5, 10, and 20 mol % of I_2. The results from Table 1 (entries 5−7) show that 5 mol % I_2 at reflux in THF is sufficient to initiate the reaction. Higher loading of the catalyst had no significant influence on the reaction yield. To find the optimum reaction temperature, the reaction was carried out with 5 mol % of I_2 at room temperature, 50 °C and reflux temperature, resulting in the isolation of **4a** in trace amount, 68%, and 84% yields (Table 1, entries 3−5), respectively. Thus, 5 mol % of I_2 and a reaction temperature at reflux were optimal conditions. In addition, CH_3CN, benzene, *N*,*N*-dimethylformamide (DMF), and $CHCl_3$ (Table 1, entries 8−11) were also tested as the solvents. In these cases, product **4a** was formed in slightly lower yields (Table 1, entries 8−11).

According to the optimized conditions, various aromatic aldehydes **1** were then subjected to react with **2** and 2,3-dihydrofuran **3** to generate a library of *exo*-benzo[*f*]furo[3,2-*c*]quinoline derivatives **4** (Table 2). For aldehyde **1**, the yields of **4** were not sensitive to the electronic properties of the aromatic ring in the presence of electron-withdrawing groups (such as halide and nitro) or electron-donating groups (such as alkoxyl group) (Table 2). The sole product was observed in the solution of the reaction system monitored by TLC. Subsequently it was separated by filtration before crystallization, and was also confirmed to be the *exo*-isomer by ^{1}H NMR. Furthermore, the naphthalen-2-amine **2** also gives high regioselectivity in position 1 rather than position 3 for its high activity, because the position 1 is not only the ortho position of the amino group, but also the α-position or benzyl position of another benzene ring in the naphthalene ring

Table 2. Synthetic Results of **4a**−**o** Catalyzed by Iodine in THF[a]

entry	Ar	products	time (h)	yields (%)[b]
1	4-$NO_2C_6H_4$	**4a**	10	84
2	4-ClC_6H_4	**4b**	10	92
3	3-$NO_2C_6H_4$	**4c**	11	88
4	3,4-$(MeO)_2C_6H_3$	**4d**	15	90
5	2-thienyl	**4e**	14	78
6	C_6H_5	**4f**	14	89
7	4-BrC_6H_4	**4g**	14	90
8	3-ClC_6H_4	**4h**	15	82
9	3,4-$Cl_2C_6H_3$	**4i**	12	84
10	4-FC_6H_4	**4j**	10	88
11	3-FC_6H_4	**4k**	12	83
12	2-$MeOC_6H_4$	**4l**	14	90
13	4-$MeOC_6H_4$	**4m**	18	93
14	3-BrC_6H_4	**4n**	13	83
15	3,4-$(MeO)_2C_6H_3$	**4o**	18	79

[a] Reagents and conditions: **1** (2 mmol), **2** (0.286 g, 2 mmol), **3** (0.210 g, 3 mmol), I_2 (0.1 mmol, 0.026 g), THF (10 mL). [b] Isolated yields.

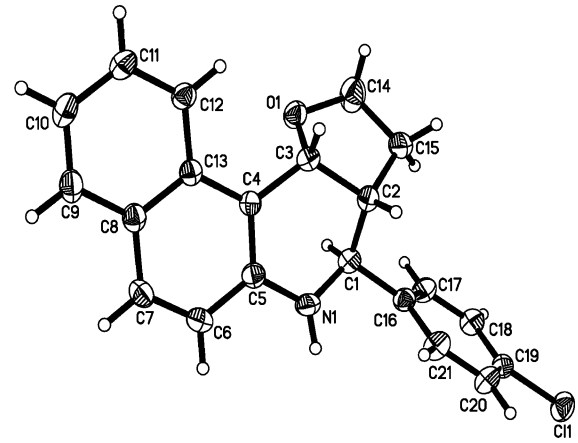
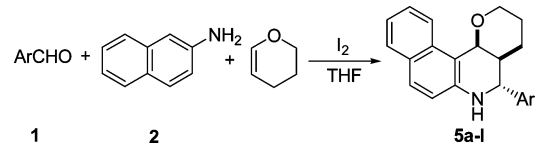


Figure 1. Crystal structure of product **4b** indicating the *exo*-structure.

Scheme 2. Reaction of **1**, **2** and 3,4-Dihydro-2*H*-pyran



moiety. The *exo*-structure of **4b** was further confirmed by X-ray diffraction analysis. Figure 1.

As expected, the substrate of 2,3-dihydrofuran could be extended to 3,4-dihydro-2*H*-pyran, which was also chosen to react with the aromatic aldehyde, naphthalen-2-amine (Scheme 2), and were found to generate the corresponding *exo*-5-arylbenzo[*f*]pyrano[3,2-*c*]quinoline derivatives (**5a**−**l**). The results are summarized in Table 3.

These three-component reactions with good stereoselectivity inspire us to choose other similar moieties to expand their application, especially for chain vinyl ethers. As a representative, *n*-butylvinyl ether was chosen as reactant to react with aromatic aldehyde, and naphthalen-2-amine to obtain *trans*-1-butoxy-1,2,3,4-tetrahydro-3- arylbenzo[*f*]quinoline derivatives **6′**. To our surprise, the desired reaction proceeded smoothly, but the butoxy group was not found in the ^{1}H NMR with aromatized 3-arylbenzo[*f*]quinolines **6a**−**i** being obtained in high yields (Scheme 3). The results are summarized in Table 4.

Table 3. Synthetic Results of **5a**−**l** Catalyzed by Iodine in THF[a]

entry	Ar	products	time (h)	yields (%)[b]
1	4-$NO_2C_6H_4$	**5a**	12	82
2	4-$MeOC_6H_4$	**5b**	18	84
3	4-ClC_6H_4	**5c**	16	82
4	3,4-$(MeO)_2C_6H_3$	**5d**	19	76
5	2-Thienyl	**5e**	16	74
6	C_6H_5	**5f**	14	86
7	4-BrC_6H_4	**5g**	16	86
8	3-ClC_6H_4	**5h**	14	80
9	3,4-$Cl_2C_6H_3$	**5i**	13	89
10	4-FC_6H_4	**5j**	16	80
11	4-MeC_6H_4	**5k**	18	78
12	3,4-$(MeO)_2C_6H_3$	**5l**	18	81

[a] Reagents and conditions: **1** (2 mmol), **2** (0.286 g, 2 mmol), 3,4-dihydro-2*H*-pyran (0.252 g, 3 mmol), I_2 (0.1 mmol, 0.026 g), THF (10 mL). [b] Isolated yields.

Scheme 3. Reaction of **1**, **2** and *n*-Butylvinyl Ether

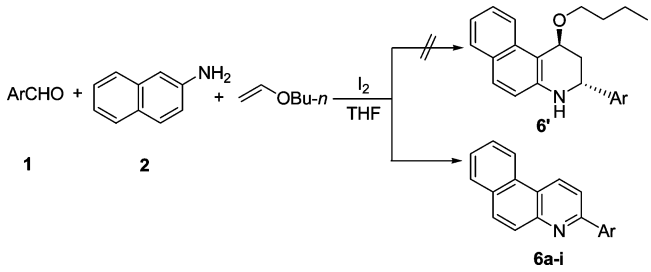
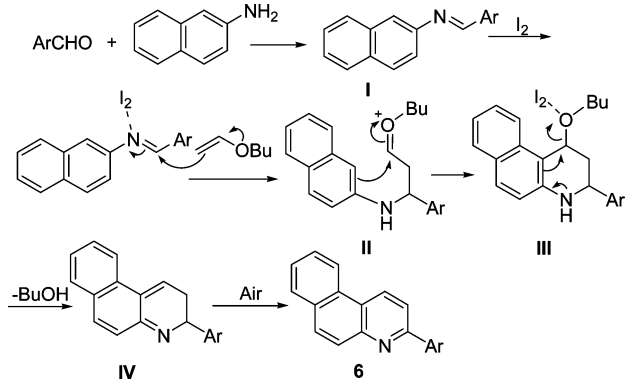


Table 4. Synthetic Results of **6a**−**i** Catalyzed by Iodine in THF[a]

entry	Ar	products	time (h)	yields (%)[b]
1	C_6H_5	**6a**	10	92
2	4-FC_6H_4	**6b**	12	88
3	4-$MeOC_6H_4$	**6c**	12	92
4	4-$NO_2C_6H_4$	**6d**	8	93
5	4-BrC_6H_4	**6e**	12	90
6	3-BrC_6H_4	**6f**	11	87
7	3,4-$Cl_2C_6H_3$	**6g**	10	84
8	3,4-$OCH_2OC_6H_3$	**6h**	12	86
9	3,4-$(MeO)_2C_6H_3$	**6i**	12	82

[a] Reagents and conditions: **1** (2 mmol), **2** (0.286 g, 2 mmol), *n*-butylvinyl ether (0.300 g, 3 mmol), I_2 (0.1 mmol, 0.026 g), THF (10 mL). [b] Isolated yields.

Scheme 4. Possible Mechanism for the Formation of Products **6**



On the basis of the literature,[12c,13] we think that iodine catalyzes the reaction as a mild Lewis acid. The mechanism was tentatively proposed as shown in Scheme 4. The Schiff base **I** may be formed by the reaction of aromatic aldehyde and naphthalen-2-amine first. And then the vinyl ether attacks iodine-activated Schiff base to form intermediate **II**, followed by an intramolecular Friedel−Crafts cyclization to give the product **III**. The unexpected loss of BuOH induced by iodine results in dihydroquinoline **IV**, which is further oxidized by air to afford aromatized 3-arylbenzo[*f*]quinolines **6**.

Scheme 5. Reaction of **1**, **3** and Anthracen-2-amine

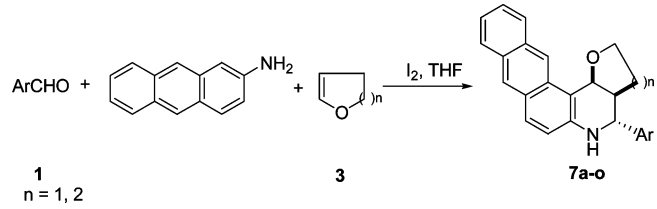


Table 5. Synthetic Results of **7a**−**o** Catalyzed by Iodine in THF[a]

entry	Ar	*n*	products	time (h)	yields (%)[b]
1	4-BrC_6H_4	1	**7a**	12	82
2	4-ClC_6H_4	1	**7b**	11	84
3	4-MeC_6H_4	1	**7c**	13	78
4	4-$MeOC_6H_4$	1	**7d**	13	80
5	4-FC_6H_4	1	**7e**	10	87
6	2-thienyl	1	**7f**	10	86
7	3,4-$(MeO)_2C_6H_3$	1	**7g**	14	83
8	3,5-$(MeO)_2C_6H_3$	1	**7h**	14	84
9	3,4-$OCH_2OC_6H_3$	1	**7i**	14	79
10	4-BrC_6H_4	2	**7j**	12	78
11	4-FC_6H_4	2	**7k**	8	80
12	4-MeC_6H_4	2	**7l**	12	82
13	4-$MeOC_6H_4$	2	**7m**	10	80
14	3,4-$(MeO)_2C_6H_3$	2	**7n**	10	83
15	3,4-$OCH_2OC_6H_3$	2	**7o**	14	84

[a] Reagents and conditions: **1** (2 mmol), anthracen-2-amine (0.386 g, 2 mmol), 2,3-dihydrofuran or 3,4-dihydro-2*H*-pyran (3 mmol), I_2 (0.1 mmol, 0.026 g), THF (10 mL). [b] Isolated yields.

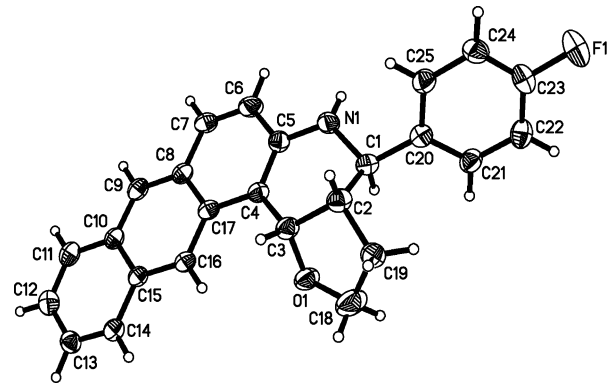


Figure 2. Crystal Structure of Product **7e** Indicating the *exo*-Structure.

This sequence is also extended from naphthalen-2-amine to other amine, such as anthracen-2-amine (Scheme 5). The desired three-component reactions of aromatic aldehyde, anthracen-2-amine and 2,3-dihydrofuran, 3,4-dihydro-2*H*-pyran took place successfully and also selectively afforded *exo*-naphtho[2,3-*f*]furo(pyrano) [3,2-*c*]quinoline derivatives (**7a**−**o**) in good yields at reflux in THF (Table 5). The *exo*-structure of **7e** is also confirmed by X-ray diffraction analysis, and its crystal structure is shown in Figure 2.

As expected, the formation of aromatized arylbenzo[*f*]quinolines lacking the butoxy fragment is also applied to anthracen-2-amine (Scheme 6). The aromatized 3-arylnaphtho[2,3-*f*]quinoline derivatives **8a**−**j** were obtained in high yields at reflux in THF (Table 6), when the 2,3-dihydrofuran was replaced by *n*-butylvinyl ether in above-mentioned reaction.

Scheme 6. Reaction of **1**, *n*-Butylvinyl Ether, and Anthracen-2-amine

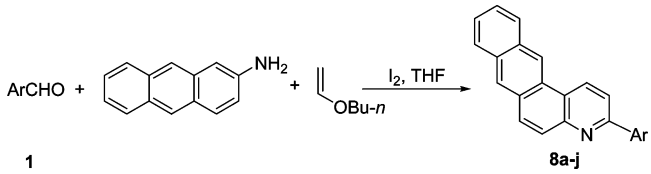


Table 6. Synthetic Results of **8a**−**j** Catalyzed by Iodine in THF[a]

entry	Ar	products	time (h)	yields (%)[b]
1	4-MeC_6H_4	**8a**	9	87
2	4-$NO_2C_6H_4$	**8b**	6	89
3	2,4-$Cl_2C_6H_3$	**8c**	7	93
4	3,4-$OCH_2OC_6H_3$	**8d**	10	92
5	3,4-$(MeO)_2C_6H_3$	**8e**	10	83
6	4-$MeOC_6H_4$	**8f**	8	84
7	3,4-$Cl_2C_6H_3$	**8g**	7	88
8	4-FC_6H_4	**8h**	7	87
9	3,4-$(MeO)_2C_6H_3$	**8i**	10	89
10	4-ClC_6H_4	**8j**	8	89

[a] Reagents and conditions: **1** (2 mmol), anthracen-2-amine (0.386 g, 2 mmol), *n*-butylvinyl ether (0.300 g, 3 mmol), I_2 (0.1 mmol, 0.026 g), THF (10 mL). [b] Isolated yields.

3. Conclusion

In conclusion, we found a mild and efficient method for the synthesis of *exo*-pyranoquinoline and *exo*-furoquinoline derivatives via three-component reactions of aromatic aldehyde, naphthalen-2-amine or anthracen-2-amine and 2,3-dihydrofuran, or 3,4-dihydro-2*H*-pyran catalyzed by iodine. The 3-arylbenzo(naphtha)[*f*]quinolines were obtained in high yields when the chain *n*-butylvinyl ether was used as reactant, with the unexpected loss of the butoxy group. The features of this procedure are mild reaction conditions, good to high yields, operational simplicity, and high stereoselectivity.

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Supporting Information Available. Representative experimental procedures, spectral data of compounds **4a**−**o**, **5a**−**l**, **6a**−**i**, **7a**−**o**, and **8a**−**j**, crystallographic information files (CIF) of **4b** and **7e**. This material is available free of charge via the Internet at http://pubs.acs.org.

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