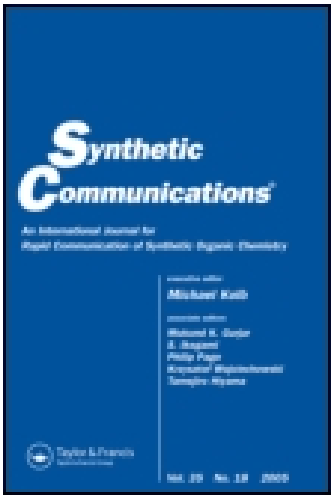

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One-Pot Synthesis of 2,3-Diarylbenzofurans via Sequential Iodocyclization and Pd-Catalyzed Suzuki Coupling Reactions of 2-Alkynylanisoles with Boronic Acids in Water

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ONE-POT SYNTHESIS OF 2,3-DIARYLBENZOFURANS VIA SEQUENTIAL IODOCYCLIZATION AND Pd-CATALYZED SUZUKI COUPLING REACTIONS OF 2-ALKYNYLANISOLES WITH BORONIC ACIDS IN WATER

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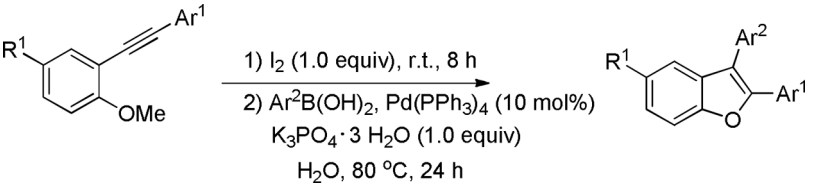
GRAPHICAL ABSTRACT



Abstract *An efficient approach for the one-pot synthesis of 2,3-diarylbenzofurans via sequential iodocyclization and Pd-catalyzed Suzuki coupling reaction of 2-alkynylanisoles with boronic acids in water is reported. The protocol utilizes water as the solvent and there is no need to isolate the intermediate 3-iodine-2-arylbenzofurans, which exemplifies the ideal of green chemistry. Various 2-alkynylanisoles and boronic acids can participate in the reactions, providing a series of 2,3-diarylbenzofurans for drug discovery in moderate to good yields.*

Keywords 2-Alkynylanisole; boronic acid; 2,3-diarylbenzofuran; iodocyclization; palladium; Suzuki coupling reaction

INTRODUCTION

Benzofuran derivatives are an important class of heterocyclic compounds and they are found as a key structural unit in many natural products, pharmaceuticals, and electroactive materials. For example, Anigopreissin A (Fig. 1) is the naturally

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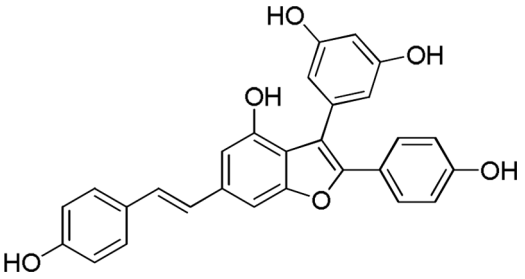


Figure 1. Anigopreissin A.

occurring resveratrol dimer isolated from root cultures of *Anigozanthos preissii* and from rhizomes of *Musa cavendish* plants.[1] Viniferifuran[2] and its congener gnetuhainin B[3] (Fig. 2) were first derived from *Vitis vinifera* Kyohou and lianas of *Gnetum hainanense*, respectively. In addition, many benzofurans exhibit a wide variety of pharmacological activities including anticancer, anti-inflammatory, antifungal, anti-HIV, and antibacterial activities.[4]

Owing to their potential utility, there have been enduring efforts to develop general and versatile methods for the synthesis of diversity-oriented benzofuran derivatives, such as 3-aryl-2-benzoylbenzofurans,[5] 3-chalcogen benzofurans,[6] 3-aryl-2-arylamidobenzofurans,[7] 3-halo- and 3-cyano benzofurans,[8] benzofuran-2-carboxylic acid,[9] 2-arylbenzofurans,[10] and so on. As part of our ongoing efforts devoted to constructing benzofuran scaffold, our interest was focused on the synthesis of 2,3-diarylbenzofurans.

Initially, 2,3-diarylbenzofurans can be synthesized through the reductive cyclization of 2-benzyloxybenzophenone by low-valent titanium reagents,[11] which were then improved by Kraus using the hindered phosphazene base P_4-t-Bu in benzene.[12] These two methods are limited to the use of uncommercial 2-benzyloxybenzophenone reactants, unstable titanium reagents, and the solvent of benzene. In 2005, Churruca et al. prepared a series of 2,3-diarylbenzofurans starting from

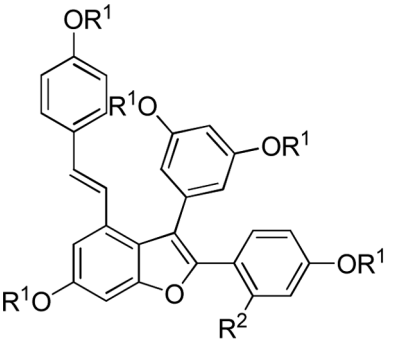


Figure 2. Viniferifuran and gnetuhainin B.

1,2-diarylethanones and 1,2-dibromoarenes by means of both homogeneous and polymer-anchored palladium catalysts,[13] but the high temperature (165 °C) also limited the application of this method.

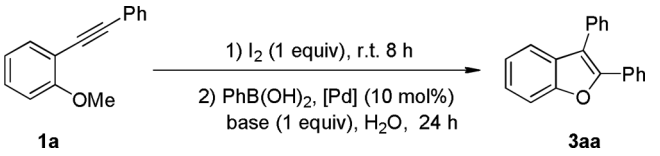
The alternative synthesis of 2,3-diarylbenzofurans is the metal-catalyzed annulation of *o*-alkynylphenol. For example, Hu et al. described the palladium/bpy-catalyzed annulation of o-alkynylphenol with various aryl halides to generate diversified 2,3-diarylbenzofurans.[14] The cyclization of a metal 2-ynyl phenolate to a 3-metallobenzoheterole intermediate that reacts further with an electrophile to afford the 2,3-diarylbenzofurans was reported by Nakamura et al.[15] Moreover, a one-pot method for the synthesis of 2,3-disubstituted benzofurans from commercially available 2-iodophenols, terminal acetylenes, and aryl iodides has been developed utilizing Sonogashira reaction conditions by Markina et al.[16] These excellent works have the merits of good yields and wide substrate scopes as well as the shortcomings of the environmental problems owing to the use of toxic solvent CH_3CN or the equivalent $BuLi/ZnCl_2$. Some novel approaches for the 2,3-diarylbenzofurans have been developed via the Pd-catalyzed arylation of 2-arylbenzofuran (benzofuran).[17] However, these methods required drastic conditions to realize the C-H bond functionalization and the yield is relatively poor. At present, 2,3-diarylbenzofurans were traditionally obtained via Pd-catalyzed Suzuki cross-coupling reactions of 3-iodo-2-arylbenzofuran or 2,3-dibromobenzofuran with boronic acids.[18] These reactions are dependent on forming the substrates of benzofuran halide, which require several extra synthetic steps and generate waste. The overall process is neither atom economical nor green. Therefore, there is still a great need for developing mild and environmentally benign reaction conditions to provide the 2,3-diarylbenzofurans in good yields.

During our previous work,[6c] the by-products 3-iodo-2-arylbenzofurans were discovered from the iodocyclization of 2-alkynylanisoles. So we envisioned that 2,3-diarylbenzofurans can be obtained via sequential iodocyclization and Pd-catalyzed Suzuki coupling reaction of 2-alkynylanisoles with boronic acids in one pot. After a series of trials, we are pleased to find that the reaction can proceed smoothly when adopting water as the solvent in mild conditions. In this article, we describe our results in detail.

RESULTS AND DISCUSSION

Initially, the reactants 2-phenylethynylanisole and iodine were added in a Schlenk tube. After stirring for 8 h at room temperature, 2-phenylethynylanisole was totally transformed to the intermediate 3-iodo-2-phenylbenzofurans by gas chromatography–mass spectrometry (GC-MS). Then phenylboronic acid (2 equiv), H_2O (2 mL) different base (1 equiv), and catalyst (10 mol%) were added to screen the optimal reaction conditions. It was found that $Pd(PPh_3)_4$ (Table 1, entry 4) gave the best result among the screened Pd catalysts, such as $PdCl_2$, $Pd(OAc)_2$, $Pd(PPh_3)_2Cl_2$, $Pd(TFA)_2$, $Pd(dba)_2$, and $Pd_2(dba)_3$ (entries 1–3 and 5–7) when using $K_3PO_4 \cdot 3H_2O$ as base at 80 °C. Subsequently, other bases including Na_2CO_3, Cs_2CO_3, KOH, CsF, and t-BuOK were investigated, affording the 2,3-diphenylbenzofuran (**3aa**) in less yield than $K_3PO_4 \cdot 3H_2O$ (entries 8–12). The structure of **3aa** was also unambiguously confirmed by X-ray crystallography (Fig. 3). Controlled

Table 1. Reaction condition screening for the synthesis of 2,3-diphenylbenzofuran[a]



1a **3aa**

Entry	Catalyst (10 mol%)	Base (1 equiv)	T (°C)	Yield[b] (%)
1	$PdCl_2$	$K_3PO_4 \cdot 3H_2O$	80	62
2	$Pd(OAc)_2$	$K_3PO_4 \cdot 3H_2O$	80	58
3	$Pd(PPh_3)_2Cl_2$	$K_3PO_4 \cdot 3H_2O$	80	84
4	$Pd(PPh_3)_4$	$K_3PO_4 \cdot 3H_2O$	80	95
5	$Pd(TFA)_2$	$K_3PO_4 \cdot 3H_2O$	80	65
6	$Pd(dba)_2$	$K_3PO_4 \cdot 3H_2O$	80	80
7	$Pd_2(dba)_3$	$K_3PO_4 \cdot 3H_2O$	80	85
8	$Pd(PPh_3)_4$	Na_2CO_3	80	91
9	$Pd(PPh_3)_4$	Cs_2CO_3	80	85
10	$Pd(PPh_3)_4$	KOH	80	53
11	$Pd(PPh_3)_4$	CsF	80	35
12	$Pd(PPh_3)_4$	t-BuOK	80	78
13	$Pd(PPh_3)_4$	$K_3PO_4 \cdot 3H_2O$	50	65
14	$Pd(PPh_3)_4$	$K_3PO_4 \cdot 3H_2O$	100	96

[a]Reaction conditions: 2-phenylethynylanisole (0.5 mmol) and iodine (0.5 mmol) were added in a Schlenk tube and stirred for 8 h at room temperature; then phenylboronic acid (1 mmol), base (0.5 mmol), catalyst (10 mol%), and H_2O (2 mL) were added and stirred for another 24 h under the corresponding temperature.
[b]The yields were detected by GC-MS analysis.

experiments showed that no obvious improvement of the yield was observed by increasing the temperature to 100 °C, whereas the yield was dramatically decreased to 65% at 50 °C. Therefore, the optimal condition was identified as follows: boronic acid (2 equiv), $K_3PO_4 \cdot 3H_2O$ (1 equiv), $Pd(PPh_3)_4$ (10 mol%), and H_2O (2 mL), 80 °C, 24 h under air.

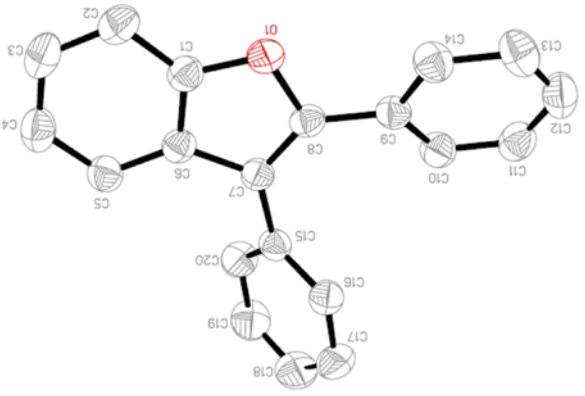
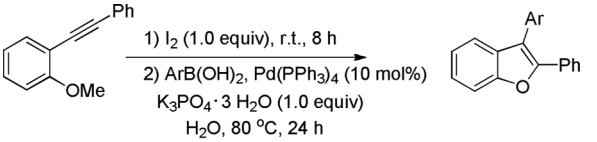


Figure 3. X-ray crystallography of **3aa**.

Table 2. Scope of boronic acids for the synthesis of 2,3-diarylbenzofurans[a]



1a **ArB(OH)$_2$ (2a-2m)** **3aa-3am**

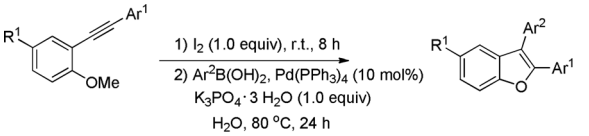
Entry	Ar	Products	Yield (%)[b]
1	C_6H_5	**3aa**	84
2	4-$CF_3C_6H_4$	**3ab**	74
3	4-FC_6H_4	**3ac**	79
4	4-ClC_6H_4	**3ad**	80
5	4-MeC_6H_4	**3ae**	90
6	4-EtC_6H_4	**3af**	86
7	4-Ph-C_6H_4	**3ag**	55
8	3,5-diFC_6H_3	**3ah**	38
9	3,5-diMeC_6H_3	**3ai**	74
10	2-FC_6H_4	**3aj**	41
11	2-ClC_6H_4	**3ak**	55
12	2-MeC_6H_4	**3al**	66
13	3-MeC_6H_4	**3am**	84

[a]Conditions: 2-phenylethynylanisole (0.5 mmol) and iodine (0.5 mmol) were added in a Schlenk tube and stirred for 8 h at room temperature; then boronic acid (1 mmol), $K_3PO_4 \cdot 3H_2O$ (0.5 mmol), $Pd(PPh_3)_4$ (10 mol%), and H_2O (2 mL) were added and stirred for another 24 h at 80 °C.
[b]Isolated yields.

With the optimized reaction in hand, the scope of arylboronic acids with 2-phenylethynylanisole was next discussed (Table 2). The results showed that the reaction seemed to be sensitive to the electronic effects on the aromatic ring of arylboronic acids. For example, arylboronic acids with 4-Me and 4-Et moieties gave greater yields than their 4-CF_3, 4-F, and 4-Cl equivalents (entries 2–6). Similarly, substrates with 3,5-diMe and 2-Me (entries 9, 12) generated their corresponding product in greater yields than those with 3,5-difluoro, 2-F, and 2-Cl groups (entries 8, 10, and 11), respectively. In addition, steric hindrance from arylboronic acids affected the efficiency dramatically. It is evident that arylboronic acids bearing either electron-donating or electron-withdrawing substituents on C2, C3, or C3,5 positions (entries 8–13) afforded the respective target molecules in lower yields than phenylboronic acid (entry 1). To our delight, treatment of 4-biphenylboronic acid with 2-phenylethynylanisole also provided the compound **3ag** in 55% yield (entry 7).

Subsequently, the scope of the 2-alkynylanisoles **1b–i** for this reaction with boronic acids was evaluated (Table 3) and various substituents of 2-alkynylanisoles have slight influence on the reaction. 2-Alkynylanisoles that had electron-donating groups at the terminal of the 1-ethynyl-2-methoxybenzene or on the anisole moiety favored the coupling reactions more than those with electron-withdrawing groups. For example, 4-methyl-2-phenylethynylanisole (**1b**), 2-(4-methylphenyl)ethynylanisole (**1c**), and 4-methyl-2-(4-methylphenyl)ethynylanisole (**1d**) (entries 1–9) gave their

Table 3. Scope of 2-arylethynylanisoles and boronic acids for the synthesis of 2,3-diarylbenzofurans[a]



1b-1i **$Ar^2B(OH)_2$ (2a, 2c, 2f, 2g)** **3ba-3ig**

Entry	R^1	Ar^1	Ar^2	Products	Yield (%)[b]
1	CH_3	C_6H_5	C_6H_5	**3ba**	87
2	CH_3	C_6H_5	4-FC_6H_4	**3bc**	81
3	CH_3	C_6H_5	4-EtC_6H_4	**3bf**	90
4	H	4-MeC_6H_4	C_6H_5	**3ca**	85
5	H	4-MeC_6H_4	4-FC_6H_4	**3cc**	80
6	H	4-MeC_6H_4	4-EtC_6H_4	**3cf**	87
7	CH_3	4-MeC_6H_4	C_6H_5	**3da**	88
8	CH_3	4-MeC_6H_4	4-FC_6H_4	**3dc**	80
9	CH_3	4-MeC_6H_4	4-EtC_6H_4	**3df**	89
10	CH_3	4-FC_6H_4	C_6H_5	**3ea**	80
11	CH_3	4-FC_6H_4	4-FC_6H_4	**3ec**	69
12	CH_3	4-FC_6H_4	4-EtC_6H_4	**3ef**	82
13	Cl	C_6H_5	C_6H_5	**3fa**	75
14	Cl	4-MeC_6H_4	C_6H_5	**3ga**	80
15	Cl	4-FC_6H_4	C_6H_5	**3ha**	60
16	H	4-Ph-C_6H_4	C_6H_5	**3ia**	90
17	H	4-Ph-C_6H_4	4-Ph-C_6H_4	**3ig**	60

[a]Conditions: 2-phenylethynylanisole (0.5 mmol) and iodine (0.5 mmol) were added in a Schlenk tube and stirred for 8 h at room temperature; then boronic acid (1 mmol), $K_3PO_4 \cdot 3H_2O$ (0.5 mmol), $Pd(PPh_3)_4$ (10 mol%), and H_2O (2 mL) were added and stirred for another 24 h at 80 °C.
[b]Isolated yields.

corresponding products in slightly greater yields than 4-methyl-2-(4-fluorophenyl) ethynylanisole (**1e**) and 4-chloro-2-arylethynylanisole (**1f, 1g, 1h**) (entries 10–15). The steric hindrance at the terminal of the 1-ethynyl-2-methoxybenzene does not affect the efficiency. The reaction of 4-biphenylethynylanisole (**1i**) with phenylboronic acid and biphenylboronic acid can proceed smoothly, giving compounds **3ia** and **3ig** in 90% and 60% yields, respectively (entries 16 and 17).

CONCLUSIONS

In conclusion, we have developed a convenient and environmentally benign protocol for the construction of 2,3-diarylbenzofurans via sequential iodocyclization and Pd-catalyzed Suzuki coupling reaction of 2-alkynylanisoles with boronic acids in water. The protocol utilizes water as the solvent, does not need isolation of the intermediate 3-iodine-2-arylbenzofurans, and exemplifies the ideal of green chemistry. Various 2-alkynylanisoles and boronic acids can participate in the reactions, providing a series of 2,3-diarylbenzofurans for drug discovery in moderate to good yields.

EXPERIMENTAL

Chemicals were either purchased or purified by standard techniques without special instructions. ^{1}H NMR and ^{13}C NMR spectra were measured on 500-MHz Brucker spectrometer, using $CDCl_3$ as the solvent with tetramethylsilane (TMS) as the internal standard at room temperature. Chemical shifts are given in δ relative to TMS, and the coupling constants J are given in hertz. Thin-layer chromatography was performed using commercially prepared 60-mesh silica-gel plates, and visualization was effected with short wavelength UV light (254 nm). All melting points are uncorrected. All reagents were used directly as obtained commercially unless otherwise noted. Column chromatography was performed using EM silica gel 60 (300–400 mesh). Known compounds are identified by comparison with authentic samples.

Typical Procedure for the Sequential Iodocyclization and Pd-Catalyzed Suzuki Coupling Reaction of 2-Alkynylanisoles with Boronic Acids in Water

2-Phenylethynylanisole (0.5 mmol) and iodine (0.5 mmol) were added in a Schlenk tube and stirred for 8 h at room temperature; then boronic acid (1 mmol), $K_3PO_4 \cdot 3H_2O$ (0.5 mmol), $Pd(PPh_3)_4$ (10 mol%), and H_2O (2 mL) were added and stirred for another 24 h at 80 °C. After cooling to room temperature and extracting twice with EtOAc, the combined organic phase was washed with brine, dried over $MgSO_4$, filtered, and evaporated under reduced pressure. The crude product was purified by flash chromatography over silica gel with petroleum ether to afford the desired product 2,3-diphenylbenzofuran **(3aa)** in 84% yield, mp 123–124 °C. ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.65–7.67 (m, 2H), 7.55–7.56 (d, $J = 5$ Hz, 1H), 7.45–7.51 (m, 5H), 7.38–7.42 (m, 1H), 7.28–7.34 (m, 4H), 7.22–7.25 (m, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 154.0, 150.5, 132.9, 130.7, 130.3, 129.8, 129.0, 128.4, 128.4, 127.6, 127.0, 124.7, 122.9, 120.0, 117.5, 111.1. Crystallographic data for **3aa**: $C_{20}H_{14}O$, white prism, formula weight 270.31, orthorhombic, P 21 21 21, $a = 7.9454(18)$ Å, $b = 10.384(2)$ Å, $c = 17.007(4)$ Å, $V = 1403.2(5)$ Å^3, $Z = 4$, $D_{calcd} = 1.280$ Mg/m^3, $R_{(int)} = 0.0172$, $R_1 = 0.0300$, $wR_2 = 0.0874$, GOF = 1.218. CCDC No. 876668.

FUNDING

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SUPPORTING INFORMATION

Supplemental data for this article can be accessed on the publisher's website.

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