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Microwave-assisted efficient synthesis of 2-arylbenzo[b]furans and 2-ferrocenylbenzo[b]furans from readily prepared propargylic alcohols and *o*-iodophenols

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Abstract. A simple, efficient and expeditious method for synthesis of 2-arylbenzo[b]furans and 2-ferrocenylbenzo[b]furans from readily prepared propargylic alcohols, *o*-iodophenols and silica gel with the catalyst of $PdCl_2(PPh_3)_2$ (2 mol%)/CuI (2 mol%) and microwave-promoted Sonogashira coupling/cyclization reaction is developed. The methodology can produce good to excellent yields. In addition, this method can also be completed in one-pot with iodobenzene, 2-methyl-3-butyn-2-ol and 2-iodo-4-methylphenol as reactants.

Keywords. Benzo[b]furan; *o*-iodophenol; propargylic alcohols; microwave irradiation.

1. Introduction

Benzo[b]furans and their derivatives are ubiquitous in the realms of pharmacologically active agents and isolated natural products which have important biological properties.[1] For example, they are widely used as antitumor drugs,[2] inhibitors of 5-lipoxygenase,[3] blood coagulation factor Xa inhibitors,[4] and antimicrobial.[5] Known methods for the synthesis of benzo[b]furan and its derivatives commonly employ the intramolecular cyclization of a suitably substituted benzene.[1,6] Benzo[b]furans are the focus of many recent reports on palladium-based cross-coupling reactions with copper iodide as a co-catalyst. In most of these cases the process is accomplished through a tandem Sonogashira coupling/5-endo-dig cyclization starting from either *o*-alkynylphenols or *o*-iodophenols. These one-pot palladium-based protocols have advantages over the multi-step traditional methods in terms of functional group tolerability and yields of the desired benzofuran. Recently, the Venkataraman group,[7] Buchwald group,[8] and others[9] have been interested in developing copper-catalysed cross-coupling reactions; copper-based methods have an economic attractiveness. However, these methods have several drawbacks in terms of different reaction conditions, they need expensive acetylenes, or their substrate preparation needs tedious operation and expensive reagents, long reaction time or low yield.

Microwave (MW)-promoted reactions are widely known and can be utilized as an alternative energy source for organic reactions ordinarily fulfilled by heating since it was found to be able to accelerate a wide variety of transformations.[10] Kabalka group has reported microwave-enhanced Sonogashira coupling and cyclization reactions on alumina,[10c] but his method needs expensive trimethylsilyl-acetylene as monoprotected acetylene derivative, large amount of catalyst, and low yield.

2. Experimental

NMR spectra were performed on a Mercury 4N-PEG-300 (^{1}H: 300 MHz; ^{13}C: 75 MHz) spectrometer, using $CDCl_3$ as a solvent and TMS as the internal standard. IR spectra recorded on Nicolet Nexus 670 FT-TR spectrophotometer as KBr pellets or KBr film. Mass spectra were recorded by the EI method on a HP 5998 mass spectrometer.

2.1 *5-Methyl-2-phenylbenzo[b]furan (**3a**); Typical procedure*

All reactions were performed on a 0.5 mmol scale relative to 1a. A round-bottom side-arm flask (10 mL) containing $PdCl_2(PPh_3)_2$ (0.01 mmol), CuI (0.01 mmol)

*For correspondence

was subjected to the Schlenk-line procedures of evacuation and purging of argon for three cycles. 2-iodo-4-methylphenol (1a, 0.5 mmol), 2-methyl-4-phenylbut-3-yn-2-ol (2a, 0.5 mmol), *t*-BuOK (1.5 mmol), silica gel (200 mg) and DMSO (2 ml) were successively added, and then the mixture was stirred magnetically at room temperature for 1 min to mix thoroughly. The flask was then exposed to microwave irradiation (intermittently at 1 min intervals; 110°C) for 2 min. After cooling to r.t, the crude product was extracted with ethyl acetate (3 × 10 mL). The combined organic phases were washed with brine (2 × 5 mL), dried over anhydrous $MgSO_4$ and concentrated *in vacuo*. The residue was subjected to flash column chromatography with hexanes/EtOAc (10/1) as eluent to obtain the desired **3a** (92.6 mg, 89% yield).

White solid; mp: 131°C (lit[11] 131°C); ^{1}H NMR (300 MHz, $CDCl_3$): δ = 7.82 (d, J = 7.5 Hz, 2 H), 7.45–7.30 (m, 5 H), 7.06 (d, J = 8.1 Hz, 1 H), 6.93 (s, 1 H), 2.43 (s, 3 H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 153.97, 153.31, 132.48, 130.60, 129.18, 128.72, 128.40, 125.50, 124.82, 120.73, 110.62, 101.08, 21.31; MS (EI): m/z = 208 [M^+]; Anal. Calcd for $C_{15}H_{12}O$: C, 86.51; H, 5.81. Found: C, 86.57; H, 5.78.

2.2 *5-Methyl-2-ferrocenylbenzo[b]furan* ***(3r)***

Red solid; mp 144–145°C; IR (KBr) ν: 3107, 2919, 2861, 1606, 1482, 1445, 1267, 1078, 1008, 931, 804, 731, 479 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.45 (s, 3H), 4.13 (s, 5H), 4.37 (t, J = 1.8 Hz, 2H), 4.77 (t, J = 1.8 Hz, 2H), 6.56 (s, 1H), 7.06 (d, J = 7.8 Hz, 1H), 7.30 (s, 1H), 7.36 (d, J = 8.1 Hz, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 21.32, 66.19, 69.19, 69.61, 75.21, 99.41, 110.28, 119.85, 124.31, 129.64, 132.11, 153.01, 157.01; FAB-MS: m/z = 316 [M^+]; Anal. Calcd for $C_{19}H_{16}FeO$: C, 72.18; H, 5.10. Found: C, 72.20; H, 5.21.

2.3 *2-Ferrocenylbenzo[b]furan* ***(3s)***

Red solid; mp 124–125°C; IR (KBr) ν: 3085, 2920, 2851, 1606, 1458, 1438, 1253, 1164, 1104, 1076, 1006, 933, 812, 746, 479 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.13 (t, J = 2.7 Hz, 5H), 4.37 (t, J = 1.5 Hz, 2H), 4.78 (t, J = 1.2 Hz, 2H), 6.62 (s, 1H), 7.18–7.24 (m, 2H), 7.47–7.51 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 66.29, 69.26, 69.65, 75.08, 99.66, 110.79, 119.95, 122.71, 123.14, 129.57, 154.61, 156.98; FAB-MS: m/z = 302 [M^+]; Anal.Calcd for $C_{18}H_{14}FeO$: C, 71.55; H, 4.67. Found: C, 71.59; H, 4.60.

2.4 *5-Chloro-2-ferrocenylbenzo[b]furan* ***(3t)***

Red solid; mp 159–160°C; IR (KBr) ν: 3110, 3089, 2919, 2851, 1603, 1462, 1438, 1260, 1159, 1059, 1009, 935, 910, 875, 800, 693, 478 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 4.13 (s, 5H), 4.39 (t, J = 1.8 Hz, 2H), 4.77 (d, J = 1.8 Hz, 2H), 6.56 (s, 1H), 7.20 (dd, J = 2.4 Hz, 9.0 Hz, 1H), 7.38 (d, J = 8.7 Hz, 1H), 7.46 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 66.37, 69.53, 69.69, 74.27, 99.08, 111.62, 119.44, 123.14, 128.27, 130.95, 152.94, 158.80; FAB-MS: m/z = 336 [M^+]; Anal.Calcd for $C_{18}H_{13}ClFeO$: C, 64.23; H, 3.89. Found: C, 64.15; H, 3.76.

2.5 *5-Tert-butyl-2-ferrocenylbenzo[b]furan* ***(3u)***

Red solid; mp 138–139°C; IR (KBr) ν: 3089, 2954, 2924, 2861, 1607, 1478, 1447, 1361, 1268, 1160, 1077, 1005, 924, 876, 807, 748, 643, 483 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 1.38 (s, 9H), 4.13 (s, 5H), 4.37 (s, 2H), 4.77 (s, 2H), 6.56 (s, 1H), 7.29 (d, J = 7.8 Hz, 1H), 7.38 (d, J = 8.1 Hz, 1H), 7.49 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 31.88, 34.67, 66.26, 69.29, 69.77, 75.20, 99.97, 110.01, 116.27, 121.08, 129.18, 145.70, 152.88, 156.95; FAB-MS: m/z = 358 [M^+]; Anal Calcd for $C_{22}H_{22}FeO$: C, 73.76; H, 6.19. Found: C, 73.71; H, 6.10.

2.6 *5,7-Dimethyl-2-ferrocenylbenzo[b]furan* ***(3v)***

Red solid; mp 134–135°C; IR (KBr) ν: 3096, 3014, 2920, 2859, 1607, 1439, 1412, 1320, 1279, 1204, 1137, 1106, 1079, 1007, 933, 867, 844, 819, 747, 510, 484 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$): δ = 2.40 (s, 3H), 2.52 (s, 3H), 4.11 (s, 5H), 4.38 (s, 2H), 4.79 (s, 2H), 6.58 (s, 1H), 6.82 (s, 1H), 7.16 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$): δ = 15.03, 21.27, 66.27, 69.13, 69.67, 75.63, 99.76, 117.29, 120.37, 125.54, 129.07, 132.08, 152.03, 156.58; FAB-MS: m/z = 330 [M^+]; Anal Calcd for $C_{20}H_{18}FeO$: C, 72.75; H, 5.49. Found: C, 72.58; H, 5.55.

3. Results and discussion

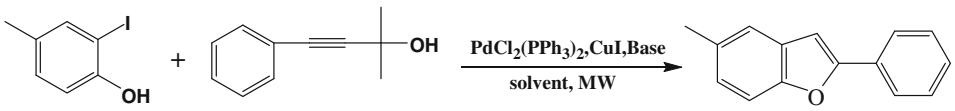
Previously, András Kotschy group has reported tandem Sonogashira coupling using the cheap reagent 2-methyl-3-butyn-2-ol.[12] An elegant procedure developed by Chow, realizing the simultaneous removal of acetone and the Sonogashira coupling of the released terminal acetylene with a second aryl halide under phase transfer conditions.[13] Very recently our team

has developed an efficient one-pot synthesis of 2-aryl-substituted benzo[b]furans from easily prepared aryl iodides, *o*-iodophenols and the inexpensive reagent 2-methyl-3-butyn-2-ol.[14] To extend the scope of this protocol, we report here a simple, efficient and expeditious method for the preparation of 2-arylbenzo[b]furans via $PdCl_2(PPh_3)_2$/CuI co-catalysed coupling reaction of *o*-iodophenols and propargylic alcohols under the microwave irradiation.

Our initial study began with the reaction of 1 equiv. of 2-iodo-4-methylphenol (1a, 0.5 mmol), 1 equiv. of 2-methyl-4-phenylbut-3-yn-2-ol (2a), and 3 equiv. of *t*-BuOK with 2 mol% of $PdCl_2(PPh_3)_2$ and 2 mol% of CuI as the co-catalyst in DMF under argon, the reaction vessel was then exposed to microwave irradiation for 2 min in an unmodified household microwave oven (Midea W7022J, 700 W). The desired product **3a** was isolated in a 21% yield (table 1, entry 1). Further optimization of the reaction conditions revealed that DMSO was more effective than other solvents, such as DMF, toluene, MeOH, EtOH, THF, CH_3CN (entries 1–3, 6–11). Silica gel played an important role in this system. Other additives, such as alumina, 4 Å MS, were tested less effectively (entries 4–6). From these evidence, we found silica gel to be a particular important reagent in this system. Silica gel apparently acts as a temperature moderator. Without silica gel, the liquid reactants can react uncontrollably. The effect of the base on the coupling/cyclization reaction was examined with *t*-BuOK as the most effective one (entries 6, 12–16). Decreasing or increasing the reaction time did not result in a higher yield (entries 17 and 18). On the basis of these control experiments, we chose the $PdCl_2(PPh_3)_2$/CuI, *t*-BuOK, silica gel, DMSO system as the standard protocol to synthesize 2-arylbenzo[b]furans from *o*-iodophenols and propargylic alcohols.

Under the optimized reaction conditions above, a series of 2-arylbenzo[b]furans can be prepared in good to excellent yields (table 2, entries 1–8). It can be seen that the yield decreases with the increasing electron withdrawing of the functional group (table 2, entries 3–5, 8). The presence of *ortho* group also gives low yield of the product (table 2, entry 7). According to the results in table 2 (table 2, entries 9 and 10, 12 and 13), it can be seen that the position of aryl's functional group has a distinct effect on the yield. It is likely that the

Table 1. Reaction of 2-iodo-4-methylphenol with 2-Methyl-4-phenylbut-3-yn-2-ol under various conditions.

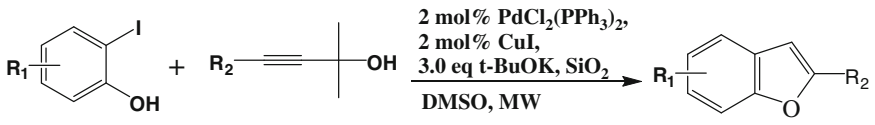


1a **2a** **3a**

Entry	Additive	Base	Solvent	Time (min)	Yield (%)[a]
1	–[b]	*t*-BuOK	DMF	2	21
2	–	*t*-BuOK	DMSO	2	38
3	–	*t*-BuOK	toluene	2	11
4	Alumina[c]	*t*-BuOK	DMSO	2	67
5	4Å MS[d]	*t*-BuOK	DMSO	2	58
6	**Silica gel**	***t*-BuOK**	**DMSO**	**2**	**89**
7	Silica gel	*t*-BuOK	DMF	2	56
8	Silica gel	*t*-BuOK	MeOH	2	n.r.[e]
9	Silica gel	*t*-BuOK	EtOH	2	n.r.
10	Silica gel	*t*-BuOK	THF	2	n.r.
11	Silica gel	*t*-BuOK	CH_3CN	2	n.r.
12	Silica gel	KOH	DMSO	2	48
13	Silica gel	K_2CO_3	DMSO	2	n.r.
14	Silica gel	Et_3N	DMSO	2	18
15	Silica gel	NaOAc	DMSO	2	n.r.
16	Silica gel	K_3PO_4	DMSO	2	12
17	Silica gel	*t*-BuOK	DMSO	1	78
18	Silica gel	*t*-BuOK	DMSO	5	80

[a]Isolated yield after column chromatography, [b]no additive was used.
[c]The amount of additives is 200 mg for all the reactions. [d]4Å molecular sieves (freshly activated)
[e]n.r. = no reaction

Table 2. Synthesis of 2-Arylbenzo[b]furans and 2-ferrocenylbenzo[b]furans.[a]

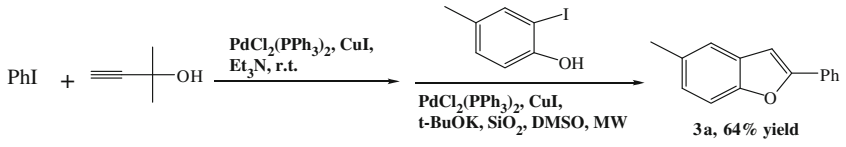


1 2 3

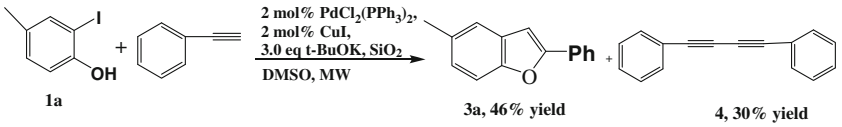
Entry	R_1[b]	R_2	Product	Yield[c]
1	4-CH_3	Phenyl	**3a**	89
2	H	–	**3b**	82
3	4-Cl	–	**3c**	69
4	4-NO_2	–	**3d**	55
5	4-Br	–	**3e**	52
6	4 -tert-butyl	–	**3f**	81
7	4,6-dimethyl	–	**3g**	68
8	4-Phenyl	–	**3h**	63
9	4-CH_3	4-CH_3Phenyl	**3i**	93
10	–	2-CH_3Phenyl	**3j**	67
11	–	3,4-dimethylPhenyl	**3k**	79
12	–	4-CH_3OPhenyl	**3l**	90
13	–	2-CH_3OPhenyl	**3m**	56
14	–	4-ethoxyPhenyl	**3n**	69
15	–	4-ClPhenyl	**3o**	73
16	–	3-ClPhenyl	**3p**	76
17	–	2-ClPhenyl	**3q**	79
18	–	Fc[d]	**3r**	88
19	H	–	**3s**	74
20	4-Cl	–	**3t**	76
21	4-tert-butyl	–	**3u**	73
22	4,6-dimethyl	–	**3v**	69
23	4-NO_2	–	**3w**	trace

[a]The reaction was carried out with 1 (0.5 mmol), 2 (0.5 mmol), SiO_2 (200 mg), *t*-BuOK (3 equiv) and DMSO (2 ml) in the presence of $PdCl_2(PPh_3)_2$ (2 mol%)/CuI (2 mol%) promoted by microwave for 2 min under argon.
[b]4-Substituted-o-iodophenols were synthesized from readily available phenols following the method reported by Edgar and Falling.[15]
[c]Isolated yields after column chromatography.
[d]Fc = ferrocene

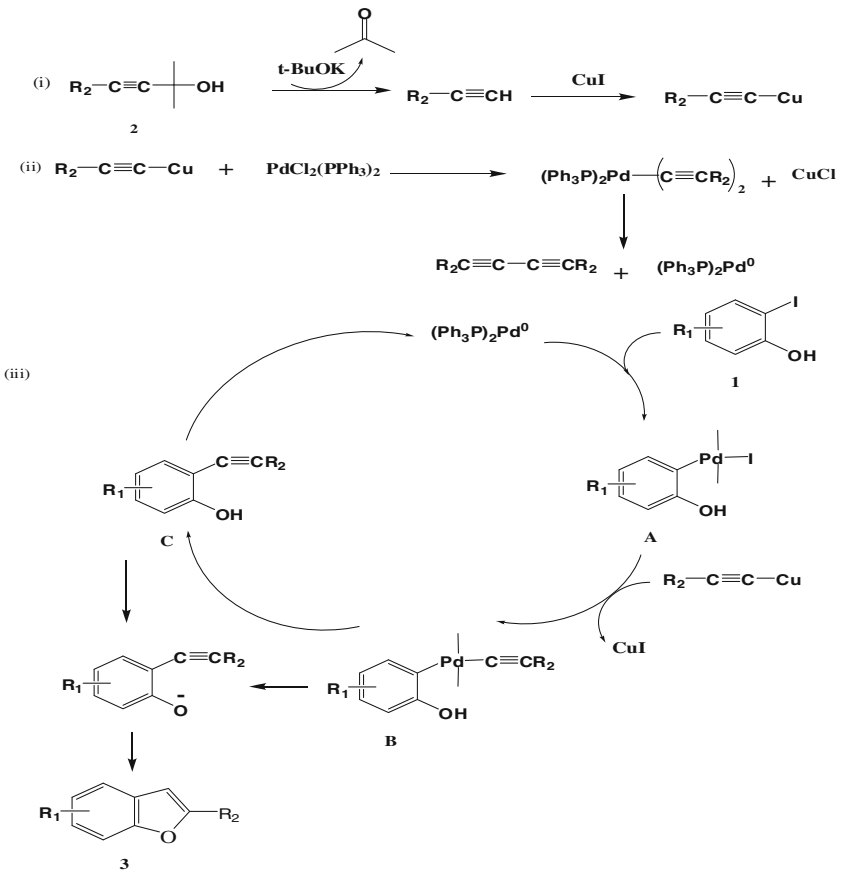
steric hindrance of the *ortho* group led to corresponding yield decrease. Upon further studies, we successfully coupled a variety of *o*-iodophenols with 2-methyl-4- ferrocenylbut-3-yn-2-ol in good to excellent yields, but 2-iodo-4-nitrophenol was ineffective (table 2, entries 18–23). Finally, we applied our synthetic protocol to synthesize **3a** in one-pot (scheme 1). This one-pot process needs no isolation of the intermediate, which greatly simplifies the operational steps, but the desired product **3a** was obtained with lower yield (64%) comparatively. To prove the benefit of the use of alcohol instead of alkyne,[13] we employed phenylacetylene instead of **2a** to synthesize **3a** under our optimized reaction conditions. We found the homo-coupling product 4 was a prominent side reaction product (scheme 2). In contrast, because of a better



Scheme 1. One-pot sequential synthesis of 5-methyl-2-phenylbenzo[b]furan (**3a**).



Scheme 2. Coupling-cyclization reaction between 2-iodo-4-methylphenol and phenylacetylene.



Scheme 3. Plausible mechanism.

selectivity toward the formation of the cross-coupling product, the benzo[b]furans were obtained with little contamination.

A plausible mechanism for the formation of 2-substituted benzofurans by a single-step palladium-catalysed reaction of *o*-iodophenol with propargylic alcohols is illustrated in scheme 3. Removal of acetone from propargylic alcohols can release terminal acetylene derivatives in step (i). The formation of Pd^0 from the interaction of *bis*(triphenylphosphine) palladium(II) chloride and cuprous acetylide as shown in step (ii). We have found evidence in favour of this from this work on the dimerisation of monosubstituted alkynes to 1,4-disubstituted 1,3-diynes by palladium–copper catalysis. Oxidative addition of *o*-iodophenol to a Pd^0 complex gives a σ-arylpalladium(II) complex (A) which then *trans*-metallates with cuprous acetylide to generate the arylalkynylpalladium(II) species (B). Through reductive elimination of Pd^0 then affords acyclic products, e.g., 2-alkynylphenols (C). The latter on cyclization in the presence of *t*-BuOK where the phenoxide ion made an attack on the triple bond resulted in the formation of the benzofurans.

4. Conclusions

In summary, we have developed a microwave-promoted, simple, and efficient method for synthesis of various substituted benzo[b]furans starting from

o-iodophenols and propargylic alcohols. Reagent 2-methyl-4-arylbut-3-yn-2-ols are more easily prepared than aryl acetylenes to synthesize benzo[b]furans. The most attractive features of this method were short reaction time, good yields, low-cost and easy preparation.

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