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Synthesis of Aryl Substituted Quinones as β-Secretase Inhibitors: Ligand-Free Direct Arylation of Quinones with Aryl Halides[1]

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Abstract—The simple ligand-free direct arylation of quinones with aryl halides applying $Pd(OAc)_2$ as a catalyst in accordance with Heck reaction was studied. This reaction provided a simple and efficient synthetic approach to efficient inhibitors of β-secretase aryl-substituted quinones.

Keywords: ligand-free, quinones, inhibitors, arylation, Heck reaction

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INTRODUCTION

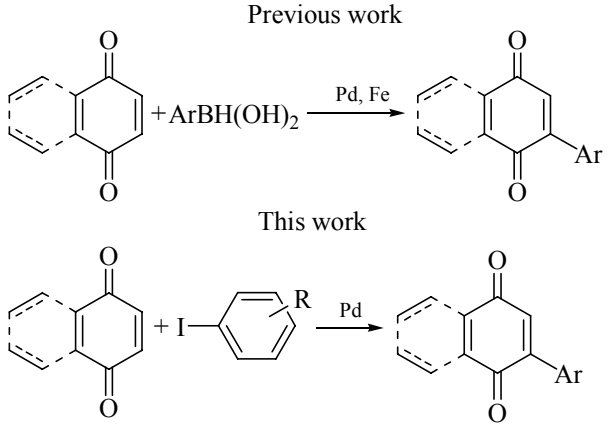
Aryl substituted quinones and naphthoquinones are important building blocks and efficient precursors of numerous biologically active natural compounds and pharmaceuticals [1–7]. Due to their unique structure, aryl substituted quinones exhibit various important biological activities [8–11], such as antibiotic, antiviral, antitumor, antimalaria, antidiabetic, and anticancer. Aryl substituted quinones are efficiant inhibitors of β-secretase (BACE1) [12–14]. Bermejo-Bescós and coworkers [15] reported the reaction of several arylquinones and their derivatives with the amyloidogenic compounds. However, synthesis of aryl substituted quinones by cross-coupling of quinones with naphthoquinones was highly limited as well as methods of synthesis of aryl substituted quinones and naphthoquinones [16–24]. Diazonium derivatives [25] and boronic acids are examples are typical examples of starting materials [26–32]. In 2009 Molina et al. [26] described the coupling reaction of quinones with arylboronic acids that was leading to aryl quinones. Several more studies were also devoted to such kind of processes (Scheme 1) [26–32].

Over recent decades, palladium-catalyzed Heck reaction or cross coupling reactions of aryl halides with nucleophiles were successfully used in organic synthesis [33–34]. Surprisingly, there were no publications on the synthesis of aryl substituted quinones based on Heck reaction though that involved aryl halides. The latter are much more cost efficient than corresponding aryl boronic acids. The The direct arylation of quinones involving aryl halides and catalysis by $Pd(OAc)_2$ in accordance with Heck reaction is presented in the current paper.

RESULTS AND DISSCUSION

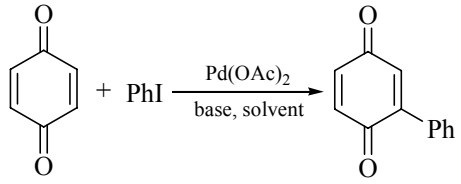
Based on our earlier data [35–39], we had selected the common $Pd(OAc)_2$ as a catalyst of the reaction of quinone with iodobenzene. The targeted product was separated with 36% yield (Table 1, entry 1). Based on

Scheme 1. The direct arylation of quinones with aryl iodide



[1] The text was submitted by the authors in English.

Table 1. Reaction conditions[a]



I **IIa** **IIIa**

Entry	Base	Sovlent	Yield[b], %
1	Na_2CO_3	CH_2Cl_2	36
2	Na_2CO_3	Toluene	<5
3	Na_2CO_3	Benzene	<5
4	Na_2CO_3	DMF	21
5	Na_2CO_3	THF	18
6	Na_2CO_3	Dioxane	14
7	Na_2CO_3	DCE	56
8	$NaHCO_3$	DCE	41
9	KOH	DCE	16
10	*t*-BuOK	DCE	52
11	Cs_2CO_3	DCE	81
12	K_2CO_3	DCE	53
13	*t*-BuONa	DCE	47

[a] Conditions: **I** (0.5 mmol, 1.0 equiv.), **IIa** (1.5 equiv.), $Pd(OAc)_2$ (5 mol %), base (1.5 equiv.), 2 mL solvent, 24 h, reflux. [b] Yields base on **I**.

the resulting data, activity of various solvents was evaluated (Table 1, entries 1–7). Superiority of 1,2-dichloroethane over other solvents was established. Influence of bases on the process conditions was studied. The results have proven Cs_2CO_3 as the most efficient one in this reaction.

Upon establishing the optimum conditions, namely $Pd(OAc)_2$ (5%), Cs_2CO_3 as the base in DCE media, the progress of Pd-catalyzed direct arylation of quinones was studied (Table 2). In most cases the corresponding aryl substituted quinones were separated with good to excellent yields. Application of meta-substituted iodobenzene resulted in moderate yield of the product (Table 2, entry 8).

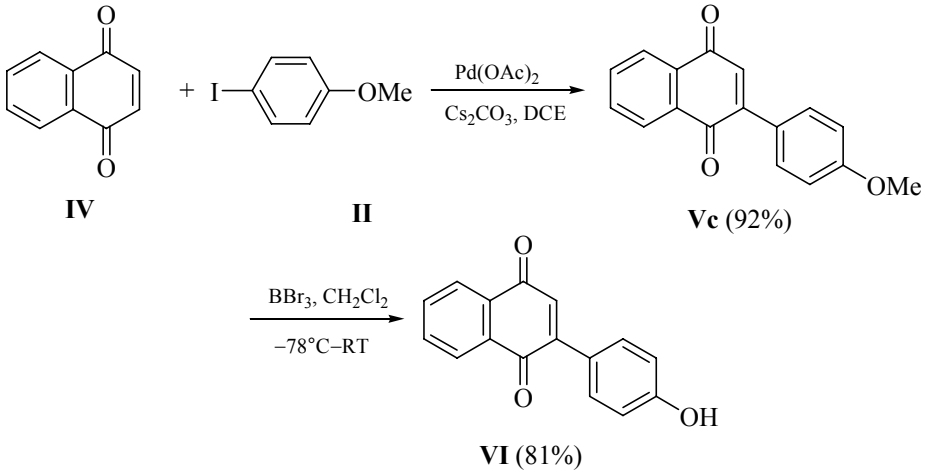
Synthesis of naphthoquinones under the optimum conditions was studied. In general, the reaction could proceed with moderate to good yield. It is noteworthy that 1-(2-chloroetoxy)-4-iodobenzene was also introduced in this reaction (Table 3, entry 9).

This method of Pd-catalyzed direct arylation of quinones with aryl halides provided an efficient route to synthesis of the inhibitors of β-secretase (BACE1) based on cost efficient quinones. For example, 2-phenylnaphthalene-1,4-dione (**Va**) was synthesized according to this method with the yield 82% (Table 3, entry 1). 2-(4-Hydroxyphenyl)naphthalene-1,4-dione (**VI**) was synthesized by the following deprotection reaction (Scheme 2) [41].

EXPERIMENTAL

General procedure. 1,4-Quinone, 1,4-naphthoquinone, palladium acetate and other chemicals were obtained from commercial resources and used without

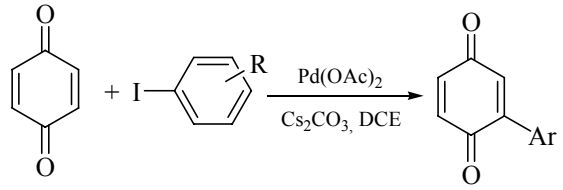
Scheme 2. Synthesis of BACE1 inhibitors B



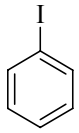
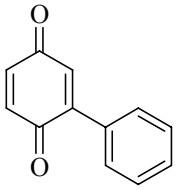
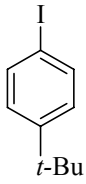
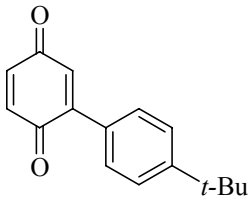
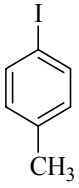
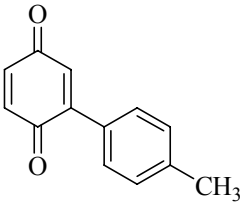
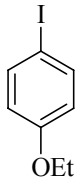
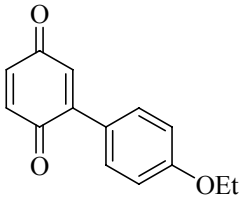
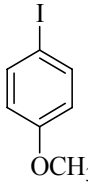
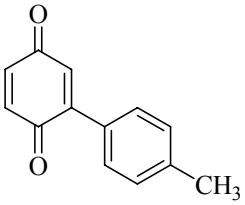
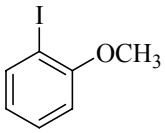
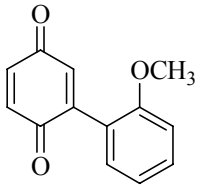
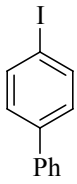
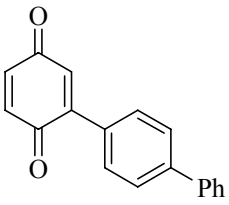
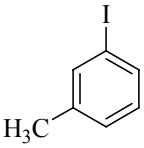
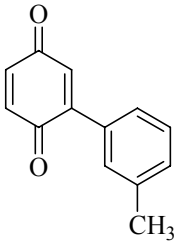
IV **II** **Vc** (92%)

VI (81%)

Table 2. Substrate expansion of quinones[a]



I **II** **III**

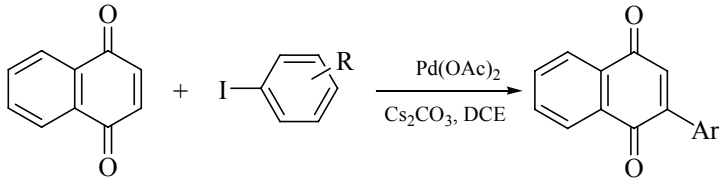
Entry	Compound	Product	Yield[b], %	Entry	Compound	Product	Yield[b], %
1			81 (**IIIa**)	5			82 (**IIIe**)
2			83 (**IIIb**)	6			83 (**IIIf**)
3			94 (**IIIc**)	7			65 (**IIIg**)
4			91 (**IIId**)	8			53 (**IIIh**)

[a] Conditions: **I** (0.5 mmol, 1.0 equiv.), **II** (1.5 equiv.), $Pd(OAc)_2$ (5%), Cs_2CO_3 (1.5 equiv.), 2mL DCE, 24 h, reflux. [b] Yields base on **I**.

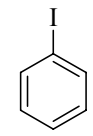
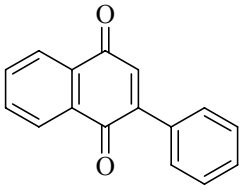
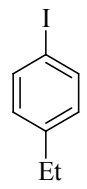
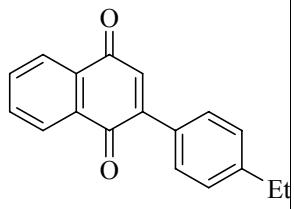
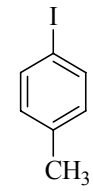
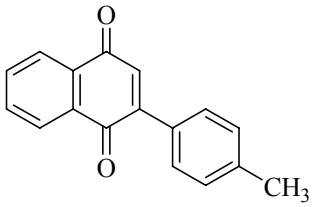
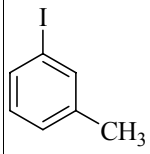
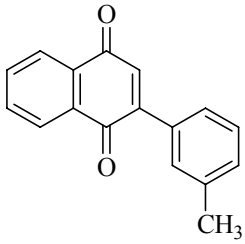
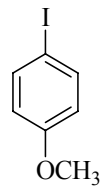
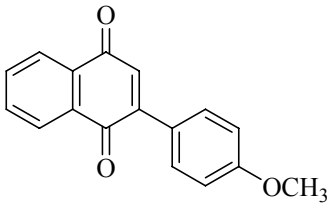
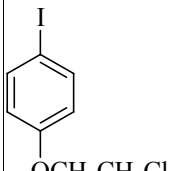
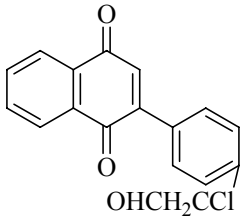
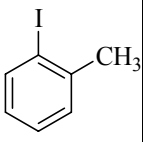
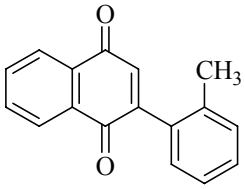
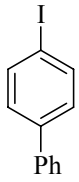
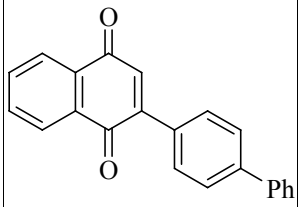
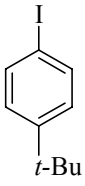
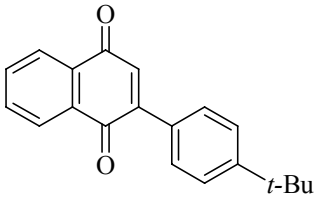
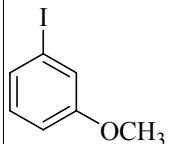
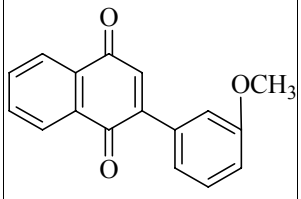
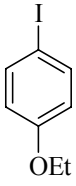
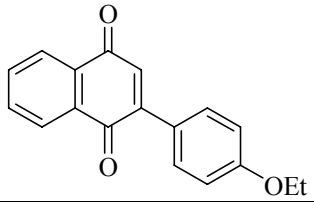
further purification. All solvents were dried, purified and freshly distilled under nitrogen. The products were isolated by column chromatography on silica gel (200–300 mesh or 100–200 mesh) using petroleum ether (60–90°C) and ethyl acetate as the eluent. The yields presented herein are based on the results of column chromatography. Reaction progress and product mixtures were routinely monitored by TLC on SiO_2. ^1H NMR spectra were recorded with a Bruker AVANCE III 400 spectrometer in $CDCl_3$ using tetramethylsilane (TMS) as the reference.

General procedure for synthesis of IIIa from quinone and iodobenzene. A mixture of 1,4-quinone (**I**) (54.0 mg, 0.50 mmol), iodobenzene (**IIa**) (153.0 mg, 0.75 mmol), $Pd(OAc)_2$ (5.6 mg, 5 mol%), and Cs_2CO_3 (244.4 mg, 1.5 equiv.), in DCE (2.0 mL) was stirred at 90°C for 24 h. Upon completion, the solvent was eva-

Table 3. Substrate expansion of naphthoquinones[a]



IV **II** **V**

Entry	Compound	Product	Yield[b], %	Entry	Compound	Product	Yield[b], %
1			82 (**Va**)	7			85 (**Vg**)
2			86 (**Vb**)	8			81 (**Vh**)
3			92 (**Vc**)	9			76 (**Vi**)
4			82 (**Vd**)	10			88 (**Vj**)
5			80 (**Ve**)	11			85 (**Vk**)
6			90 (**Vf**)				

[a] Conditions: **IV** (0.5 mmol, 1.0 equiv.), **II** (1.5 equiv.), $Pd(OAc)_2$ (5%), Cs_2CO_3 (1.5 equiv.), 2 mL DCE, 24 h, reflux. [b] Isolated yields based on **IV**.

porated. The residue was purified by column chromatography on silica gel (ethyl acetate/petroleum ether = 1 : 10, v : v) to give the corresponding product **IIIa**.

General method of synthesis of the compounds VI. The compound **Vc** (0.1 mmol) was dissolved in dry CH_2Cl_2 (2.0 mL), cooled to –78°C and 0.32 mL of BBr_3 (1.0 M in CH_2Cl_2) was added drop wise upon stirring. The cooling bath was removed and following reaction mixture stirring for ca 8 h heated it up to room temperature. The reaction was terminated by slow addition of water. The layers were separated, the organic phase washed with H_2O, and the combined aqueous layers evaporated to dryness. The residue was separated in RP-18 column (MeOH : H_2O = 35 : 65) to give brown-violet solids **VI**.

2-Phenyl[1,4]benzoquinone (IIIa). Faint yellow solid (81%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.52–7.42 m (5H), 6.84–6.90 m (3H).

2-*p*-Tolyl-[1,4]benzoquinone (IIIb). Faint yellow solid (83%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.39 d (*J* = 8.2 Hz, 2H), 7.26 d (*J* = 8.0 Hz, 2H), 6.88–6.77 m (3H), 2.40 s (3H).

2-(4-Methoxyphenyl)[1,4]benzoquinone (IIIc). Faint yellow solid (94%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.48 d (*J* = 8.8 Hz, 2H), 6.97 d (*J* = 8.8 Hz, 2H), 6.88–6.77 m (3H), 3.86 s (3H).

2-Biphenyl-4-yl[1,4]benzoquinone (IIId). Faint yellow solid (91%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.77–7.59 m (6H), 7.50 t (*J* = 7.5 Hz, 2H), 7.41 t (*J* = 7.5 Hz, 1H), 7.03–6.85 m (3H).

2-(4-*tert*-Butyl-phenyl)[1,4]benzoquinone (IIIe). Faint yellow solid (82%).^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.47 d (*J* = 8.5 Hz, 2H), 7.42 d (*J* = 8.5Hz, 2H), 6.87–6.79 m (3H), 1.34 s (9H).

2-(4-Ethoxy-phenyl)-[1,4]benzoquinone (IIIf). Faint yellow solid (83%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.53 d (*J* = 8.6 Hz, 1H), 7.47 d (*J* = 8.7 Hz, 1H), 7.36 s (1H), 7.13 m (1H), 6.97–6.77 m (3H), 4.09 q (*J* = 6.9 Hz, 2H), 1.44 t (*J* = 7.0 Hz, 1H).

2-(2-Methoxyphenyl)[1,4]benzoquinone (IIIg). Faint yellow solid (65%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.42 m (1H), 7.16 d.d (*J* = 7.5, 1.6 Hz, 1H), 7.02 t (*J* = 7.5 Hz, 1H), 6.97 d (*J* = 8.3 Hz, 1H), 6.87–6.79 m (3H), 3.78 s (3H).

2-*m*-Tolyl-[1,4]benzoquinone (IIIh). Faint yellow solid (53%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 7.35–7.24 m (4H), 6.88–6.80 m (3H), 2.40 s (3H).

2-Phenyl[1,4]naphthoquinone (Va). Faint yellow solid (82%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.22–8.17 m (1H), 8.16–8.10 m (1H), 7.82–7.74 m (2H), 7.61–7.56 m (2H), 7.51–7.46 m (3H), 7.09 s (1H).

2-*p*-Tolyl-[1,4]naphthoquinone (Vb). Faint yellow solid (86%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.19 m (1H), 8.12 m (1H), 7.80–7.75 m (2H), 7.49 d (*J* = 8.1 Hz, 2H), 7.29 d (*J* = 8.0 Hz, 2H), 7.07 s (1H), 2.42 s (3H).

2-(4-Methoxyphenyl)[1,4]naphthoquinone (Vc). Faint yellow solid (92%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.17 m (1H), 8.10 m (1H), 7.79–7.72 m (2H), 7.58 d (*J* = 8.5 Hz, 2H), 7.04 s (1H), 6.99 d (*J* = 8.6 Hz, 2H), 3.87 s (3H).

2-*o*-Tolyl[1,4]naphthoquinone (Vd). Faint yellow solid (82%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.20–8.11 m (2H), 7.81–7.76 m (2H), 7.36 t (*J* = 7.4 Hz, 1H), 7.30–7.25 m (2H), 7.17 d (*J* = 7.5 Hz, 1H), 6.93 s (1H), 2.23 s (3H).

2-(4-*tert*-Butylphenyl)[1,4]naphthoquinone (Ve). Faint yellow solid (80%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.19 m (1H), 8.12 m (1H), 7.77 m (2H), 7.52 d.d (*J* = 17.4, 8.4 Hz, 4H), 7.08 s (1H), 1.36 s (9H).

2-(4-Ethoxyphenyl)[1,4]naphthoquinone (Vf). Faint yellow solid (90%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.17 m (1H), 8.14–8.08 m (1H), 7.80–7.73 m (2H), 7.57 d (*J* = 8.9 Hz, 2H), 7.05 s (1H), 6.98 d (*J* = 8.8 Hz, 2H), 4.10 q (*J* = 7.0 Hz, 2H), 1.45 t (*J* = 7.0 Hz, 3H).

2-(4-Ethylphenyl)[1,4]naphthoquinone (Vg). Yellow solid (85%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.19 m (1H), 8.12 m (1H), 7.80–7.75 m (2H), 7.52 d (*J* = 8.1 Hz, 2H), 7.31 d (*J* = 8.1 Hz, 2H), 7.07 s (1H), 2.72 q (*J* = 7.6 Hz, 2H), 1.28 t (*J* = 7.6 Hz, 3H).

2-*m*-Tolyl[1,4]naphthoquinone (Vh). Faint yellow solid (81%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.17–8.18 m (1H), 8.10–8.12 m (1H), 7.76–7.78 m (2H), 7.29–7.37 m (4H), 7.06 s (1H), 2.42 s (3H).

2-[4-(2-Chloroethyl)phenyl][1,4]naphthoquinone (Vi). Faint yellow solid (76%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.20–8.15 m (1H), 8.11 m (1H), 7.80–7.74 m (2H), 7.58 d (*J* = 8.5 Hz, 2H), 7.05 s (1H), 7.01 d (*J* = 8.5 Hz, 2H), 4.30 t (*J* = 5.8 Hz, 2H), 3.85 t (*J* = 5.8 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$), δ, ppm: 185.17 s, 184.73 s, 147.28 s, 133.97 s, 133.80 s,

132.55 s, 132.14 s, 131.18 s, 127.04 s, 126.41 s, 125.93 s, 114.72 s, 68.07 s, 41.70 s. Calculated, %: C 72.85; H 4.42; Cl 11.95; O 10.78. $C_{18}H_{13}ClO_2$. Found, %: C 72.79; H 4.40; Cl 11.99; O 10.82.

2-Biphenyl-4-yl[1,4]naphthoquinone (Vj). Yellow solid (88%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.24–8.19 m (1H), 8.16–8.12 m (1H), 7.81–7.78 m (2H), 7.73–7.64 m (6H), 7.48 t (J = 7.5 Hz, 2H), 7.39 t (J = 7.4 Hz, 1H), 7.14 s (1H).

2-(3-Methoxyphenyl)[1,4]naphthoquinone (Vk). Faint yellow solid (85%). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.17–8.19 m (1H), 8.11–8.12 m (1H), 7.77–7.79 m (2H), 7.36–7.40 m (1H), 7.10–7.15 m (1H), 7.07 s (1H), 7.01–7.03 m (2H), 3.86 s (3H).

2-(4-Hydroxyphenyl)[1,4]naphthoquinone (VI). ^{1}H NMR (400 MHz, $CDCl_3$), δ, ppm: 8.12–8.07 m (1H), 8.05–8.01 m (1H), 7.73–7.65 m (2H), 7.49–7.41 m (2H), 6.96 s (1H), 6.90–6.82 m (2H), 5.69 s (1H).

CONCLUSIONS

The ligand-free direct arylation of quinones by aryl halides with $Pd(OAc)_2$ as a catalyst according to Heck was studied. The reaction products were accumulated with moderate to good yields. This reaction provided an easy and convenient method of synthesis of aryl-substituted quinones. Further study of the process with a broader range of compounds is in progress.

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