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An oxy-Michael addition: 2,5-dihydroxy-1,4-benzoquinone-assisted synthesis of 1-[ethoxy(phenyl)methyl]-2-naphthol and 5-[ethoxy(phenyl)methyl]-6-hydroxyquinoline derivatives

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Abstract The 2,5-dihydroxy-1,4-benzoquinone-assisted oxy-Michael addition reaction between an aromatic aldehyde, EtOH and 2-naphthol or 6-hydroxyquinoline in the presence of HCl to afford the 1-[ethoxy(aryl)methyl]-2-naphthol and 5-[ethoxy(phenyl)methyl]-6-hydroxyquinoline derivatives at room temperature is described.

Keywords Oxy-Michael addition · *o*-Naphthoquinone methide · Multi-component reactions · 2-Naphthol · 6-Hydroxyquinoline

Introduction

Naphthalene and quinoline nucleus are commonly found in pharmaceutical and agricultural agents [1–6]. For example, hydroxyquinoline and styrylquinoline derivatives have been reported as potential HIV-1 integrase inhibitors [7] and antifungal agents [8]. Also, some of them have herbicidal and antineoplastic activities [9]. These compounds showed a good antifungal activity similar to some novel compounds. So, hydroxyl-functionalized derivatives such as 2-naphthols are one of the interesting scaffolds with bactericide and antioxidant activity [10].

o-Quinone methides (oQMs) are reactive α,β-unsaturated ketones and important intermediates in many chemical and biological processes [11–13]. These intermediates are efficient dDNA alkylating and cross-linking agents [14, 15], and are believed to be responsible for the cytotoxicity of antitumor antibiotics of the mitomycin C and anthracycline families [16, 17]. These reactive species are generated by photochemical dehydration of *o*-hydroxybenzyl alcohols and its analogues [18–20]. High electrophilicity of the methide C- atom and also the Diels–Alder reaction with electron-rich alkenes to oQMs are two of their important

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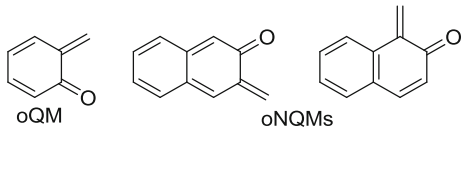
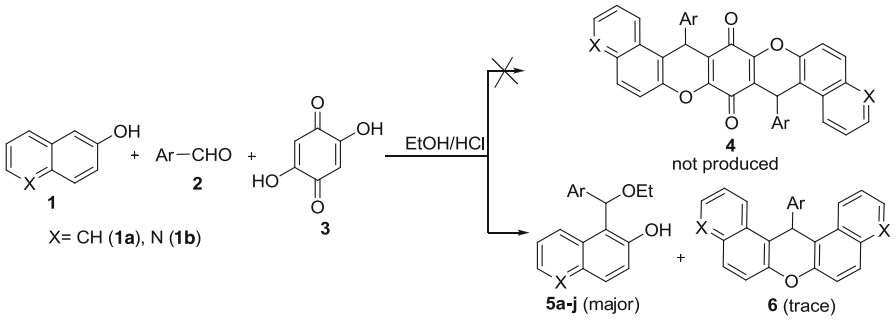


Fig. 1 Structures of oQM and oNQMs



Scheme 1 Expected (**4**) and observed (**5, 6**) reaction products

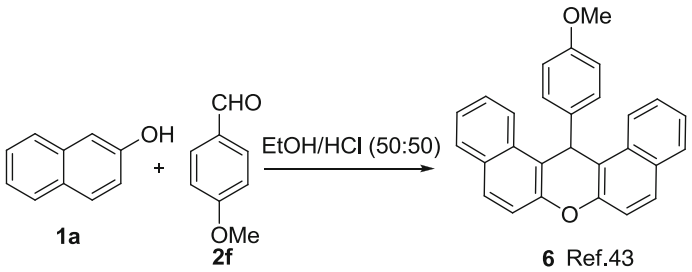
synthetic features [18–22]. Recently, the photochemical generation and the reactivity of *o*-naphthoquinone methides (oNQMs) were studied [23]. Irradiation of 3-hydroxy-2-naphthalenemethanol and 2-hydroxy-1-naphthalenemethanol followed by efficient dehydration led to the formation of isomeric oNQMs (Fig. 1).

β-Alkoxy ketones are valuable building blocks in a variety of natural products [24]. These compounds are usually prepared by the aldol reaction and the subsequent alkylation of the resulting hydroxyl group [25, 26]. The oxy-Michael addition of the *O*-nucleophiles to electron-deficient *α*,*β*-unsaturated ketones has been a significant challenge in organic synthesis, owing to the low reactivity coupled with the reversibility of the reaction [27–33]. Some of the electron-deficient olefins could undergo conjugate addition of the *O*-nucleophiles in the presence of various acids and amines [30, 34–36].

In continuation of our study on multi-component reactions [37–40], 2,5-dihydroxy-1,4-benzoquinone [41], and Michael additions [42], we investigated the reaction of 2-naphthol (**1a**) or 6-hydroxyquinoline (**1b**), aromatic aldehydes **2**, and 2,5-dihydroxy-1,4-benzoquinone (**3**) in the mixture of HCl (37 %) and ethanol (1:1) at ambient temperature. In the event, the desired product **4** was not obtained, instead the reaction afforded the corresponding products **5a**–**j** in good to moderate yields assisted with 2,5-dihydroxy-1,4-benzoquinone (**3**). The xanthene **6** is produced in traces as a by-product (Scheme 1).

Results and discussion

In a pilot experiment, 2-naphthol (**1a**) (1 mmol), 2-nitrobenzaldehyde (**2c**) (1 mmol), and **3** (1 mmol) were stirred in EtOH/HCl (1:1) for 24 h at room temperature. On completion, the reaction mixture was filtered, and the precipitate



Scheme 2 The reaction of 2-naphthol (**1a**) and 3-methoxybenzaldehyde (**2f**) in the absence of 2,5-dihydroxy-1,4-benzoquinone (**3**)

washed with H_2O/EtOH (1:2). The crude product (**5c**) was purified by recrystallization from CH_2Cl_2/*n*-hexane (1:2) and was obtained in 82 % yield.

To illustrate the role of **3**, the reaction of **1a** and 3-methoxybenzaldehyde (**2f**) was studied in the absence of **3**. However, 14-phenyl-14*H*-dibenzo[*a*,*j*]xanthene (**6**) was produced instead of the expected product **5** via a condensation reaction of **2f** with two molecules of **1a** (Scheme 2) [44].

As indicated in Table 1, the optimum amount of **3** in the reaction of 2-naphthol (**1a**) and 3-nitrozaldehyde (**2a**) was examined in EtOH/HCl (37 %) (1:1) at room temperature for 24 h. The highest yield of **5a** was obtained with 100 % of **3**. A further increase in the amount of **3** up to 150 % did not have significant effects on the product yield.

To explore the scope and limitations of the reaction, we extended our studies to various aldehydes and 6-hydroxyquinoline (**1b**) instead of 2-naphthol (**1a**). As indicated in Fig. 2, aromatic aldehydes **2** containing electron-donating or electron-withdrawing groups, and **1a** or **1b** in EtOH in the presence of **3** undergo a smooth 1:1:1 addition reaction at room temperature to produce 1-[ethoxy(aryl)methyl]-2-naphthol and 5-[ethoxy(phenyl)methyl]-6-hydroxyquinoline derivatives **5a–j** in good to moderate yields.

The structure of compounds **5a–j** was deduced from their IR, ^{1}H NMR, ^{13}C NMR data, and elemental analyses. The ^{1}H NMR spectrum of **5c** consisted of a triplet for the Me group (0.96 ppm, $^3J_{HH} = 7.0$ Hz), two multiplets for CH_2O (3.23 and 3.30 ppm, $^3J_{HH} = 7.0$ Hz), a singlet for CH (3.98 ppm), a multiplet for aromatic H-atoms (6.71–7.96 ppm), and a broad singlet for the OH group (10.12 ppm). The ^{1}H decoupled ^{13}C NMR spectrum of **5c** showed 19 distinct resonances; partial assignment of these resonances is given in the experimental section.

Table 1 Optimization of the amounts of 2,5-dihydroxy-1,4-benzoquinone (**3**)

Entry	1	2	3	4	5	6
Catalyst **3** (mol%)	5	25	50	75	100	150
Yield (%)[a]	Trace	21	42	63	86	87

Reaction conditions: 2-naphthol (**1a**) (1 mmol), 4-nitrobenzaldehyde (**2a**) (1 mmol), EtOH/HCl (37 %) (1:1) (10 mL), room temperature, 24 h

[a] Isolated yield

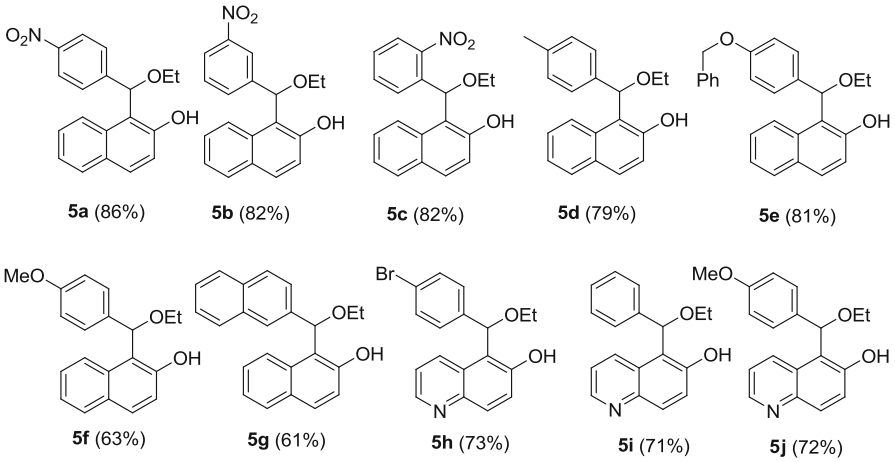


Fig. 2 Structures of products **5a–j**

Further investigations into the scope, mechanism, and synthetic applications of this reaction are now in progress.

Conclusions

A simple and efficient one-pot multi-component reaction for the synthesis of 1-[ethoxy(phenyl)methyl]-2-naphthol and 5-[ethoxy(phenyl)methyl]-6-hydroxy-quinoline derivatives via an oxy-Michael addition reaction has been elaborated. We developed a new route for oNQM generating and trapping it via ethoxy group nucleophilic addition. To the best of our knowledge, this is the first reaction promoted by 2,5-dihydroxy-1,4-benzoquinone as the auxiliary.

Experimental

Melting points were measured on an Electrothermal 9100 apparatus and are uncorrected. IR spectra were recorded in KBr on a Shimadzu IR-470 spectrometer. ^{1}H NMR Spectra were recorded on a Bruker DRX-300 Avance spectrometer at 300.13 MHz. The ^{13}C NMR spectra were recorded at 75.47 MHz; for all NMR spectra data, chemical shifts (δ scale) are reported in parts per million (ppm). The elemental analyses were performed with an Elementar Analysensysteme VarioEL. The chemicals used in this work were purchased from Merck and Fluka Chemical.

Typical procedure for preparation of 1-[Ethoxy(4-nitrophenyl)methyl]-2-naphthol (**5a**)

To a magnetically stirred solution of 2-naphthol (**1a**, 0.14 g, 1.0 mmol) in EtOH/HCl (37 %) (1:1) (10 ml), 2,5-dihydroxy-1,4-benzoquinone (**3**, 0.14 g, 1 mmol) and

4-nitrobenzaldehyde (**2a**, 0.15 g, 1.0 mmol) was added, respectively. The reaction mixture was stirred for 24 h at room temperature. After completion of the reaction (monitored by TLC method), the residue was filtered off and crystallized from CH_2Cl_2/*n*-hexane (1:2) and the product **5a** was obtained as a brown powder.

1-[Ethoxy(4-nitrophenyl)methyl]-2-naphthol (**5a**)

Brown powder (0.28 g, yield 86 %). Mp > 300 °C. IR: 3,272, 3,123, 2,911, 2,850, 1,637, 1,601, 1,571, 1,337 cm^{-1}. ^1H NMR (300 MHz, DMSO) δ: 0.94 (t, $^3J_{HH} = 6.9$, 3H, CH_3), 3.19-3.21 (m, 1H, OCH_2), 3.27-3.30 (m, 1H, OCH_2), 3.94 (s, 1H, CH), 6.69–7.94 (m, 10H, Ar), 10.25 (br s, 1H, OH). ^{13}C NMR (75 MHz, DMSO) δ: 15.1 (CH_3), 61.3 (CH_2), 69.7 (CH), 113.9, 118.2, 123.0, 125.1, 125.3, 126.3, 128.7, 129.0, 129.4, 129.7, 131.3, 133.1, 133.4, 134.7, 150.1, 154.9. Anal. Calcd. for $C_{19}H_{17}NO_4$: C, 70.58; H, 5.30; N, 4.33; Found: C, 70.64; H, 5.39; N, 4.23.

1-[Ethoxy(3-nitrophenyl)methyl]-2-naphthol (**5b**)

Brown powder (0.26 g, yield 82 %). Mp 292–293 °C. IR: 3,235, 3,097, 2,951, 2,869, 1,643, 1,611, 1,543, 1,347 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 1.18 (t, $^3J_{HH} = 7.0$, 3H, CH_3), 3.42–3.44 (m, 1H, OCH_2), 3.54–3.57 (m, 1H, OCH_2), 4.00 (s, 1H, CH), 7.20–8.32 (m, 10H, Ar), 10.20 (br s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 15.7 (CH_3), 64.2 (CH_2), 74.1 (CH), 117.0, 118.3, 120.5, 122.1, 124.6, 126.5, 129.2, 129.5, 130.2, 130.6, 130.7, 132.3, 132.6, 145.9, 148.2, 154.4. Anal. Calcd. for $C_{19}H_{17}NO_4$: C, 70.58; H, 5.30; N, 4.33; Found: C, 70.59; H, 5.37; N, 4.21.

1-[Ethoxy(2-nitrophenyl)methyl]-2-naphthol (**5c**)

Brown powder (0.26 g, yield 82 %). Mp > 300 °C. IR: 3,357, 3,111, 2,925, 2,847, 1,648, 1,627, 1,545, 1,350 cm^{-1}. ^1H NMR (300 MHz, DMSO) δ: 0.96 (t, $^3J_{HH} = 6.9$, 3H, CH_3), 3.21–3.24 (m, 1H, OCH_2), 3.29–3.31 (m, 1H, OCH_2), 3.98 (s, 1H, CH), 6.71–7.96 (m, 10H, Ar), 10.12 (br s, 1H, OH). ^{13}C NMR (75 MHz, DMSO) δ: 15.1 (CH_3), 65.0 (CH_2), 72.9 (CH), 113.8, 118.2, 123.0, 124.4, 125.3, 126.3, 128.9, 129.0, 129.2, 129.3, 131.0, 132.1, 133.1, 134.4, 150.7, 155.2. Anal. Calcd. for $C_{19}H_{17}NO_4$: C, 70.58; H, 5.30; N, 4.33; Found: C, 70.57; H, 5.35; N, 4.27.

1-[Ethoxy(*p*-tolyl)methyl]-2-naphthol (**5d**)

Brown powder (0.23 g, yield 79 %). Mp > 300 °C. IR: *v* 3,239, 3,009, 2,987, 2,911, 1,673, 1,605, 1,587 cm^{-1}. ^1H NMR (300 MHz, DMSO) δ: 1.15 (t, $^3J_{HH} = 6.5$, 3H, CH_2CH_3), 1.96 (s, 3H, CH_3), 4.00–4.04 (m, 3H, OCH_2 and CH), 7.21–8.03 (m, 10H, Ar), 9.98 (br s, 1H, OH). ^{13}C NMR (75 MHz, DMSO) δ: 15.1 (CH_3), 34.9 (CH_3), 55.3 (CH_2), 78.1 (CH), 117.2, 119.1, 119.8, 122.2, 123.3, 125.1, 126.1, 126.7, 127.1,

127.7, 128.0, 128.7, 129.5, 143.3, 146.9, 152.8. Anal. Calcd. for $C_{20}H_{20}O_2$: C, 82.16; H, 6.89; Found: C, 81.07; H, 6.93.

1-[(4-(Benzyloxy)phenyl)(ethoxy)methyl]-2-naphthol (**5e**)

Brown powder (0.31 g, yield 81 %). Mp > 300 °C. IR: 3,330, 3,067, 2,931, 1,659, 1,621, 1,537 cm^{-1}. ^1H NMR (300 MHz, DMSO) δ: 1.17 (t, $^3J_{HH} = 6.8$, 3H, CH_3); 3.40–3.43 (m, 2H, OCH_2Ph); 3.49 (br, 3H, CH and OCH_2CH_3); 7.18–8.20 (m, 15H, Ar); 10.27 (br s, 1H, OH). ^{13}C NMR (75 MHz, DMSO) δ: 15.6 (CH_3), 64.2, 64.3, 72.9 (CH), 117.0, 118.3, 120.5, 122.0, 122.9, 123.0, 124.4, 124.5, 126.4, 128.9, 129.2, 130.2, 130.7, 132.3, 132.6, 145.9, 148.2, 154.4. Anal. Calcd. for $C_{26}H_{24}O_3$: C, 81.22; H, 6.29; Found: C, 81.29; H, 6.17.

1-[Ethoxy(4-methoxyphenyl)methyl]-2-naphthol (**5f**)

Brown powder (0.19 g, yield 63 %). Mp > 300 °C. IR: 3,231, 3,123, 2,917, 2,833, 1,641, 1,539, 1,537 cm^{-1}. ^1H NMR (300 MHz, DMSO) δ: 1.20 (br, 3H, CH_3), 3.61 (br, 3H, CH and OCH_2), 3.67 (s, 3H, OCH_3), 6.74–8.11 (m, 10H, Ar), 9.62 (br s, 1H, OH). ^{13}C NMR (75 MHz, DMSO) δ: 31.1 (CH_3), 55.3, 56.1, 70.1 (CH), 113.4, 113.5, 113.6, 119.4, 121.0, 122.5, 124.4, 126.2, 128.8, 129.1, 129.5, 130.0, 134.6, 136.1, 153.1, 157.7. Anal. Calcd. for $C_{20}H_{20}O_3$: C, 77.90; H, 6.54; Found: C, 77.79; H, 6.57.

1-[Ethoxy(naphthalen-2-yl)methyl]-2-naphthol (**5g**)

Brown powder (0.20 g, yield 61 %). Mp > 300 °C. IR: 3,267, 3,019, 2,857, 1,639, 1,603, 1,531 cm^{-1}. ^1H NMR (300 MHz, DMSO) δ: 1.05 (t, $^3J_{HH} = 7.0$, 3H, CH_3), 3.43–3.46 (m, 1H, OCH_2), 3.90–3.94 (m, 2H, OCH_2 and CH), 7.19–8.05 (m, 13H, Ar), 10.13 (br s, 1H, OH). Anal. Calcd. for $C_{23}H_{20}O_2$: C, 84.12; H, 6.14; Found: C, 84.23; H, 6.09.

5-[(4-Bromophenyl)(ethoxy)methyl]-6-hydroxyquinoline (**5h**)

Dark yellow powder (0.26 g, yield 73 %). Mp 260–263 °C. IR: 3,233, 3,037, 2,928, 1,641, 1,601, 1,573 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 1.30 (br, 3H, CH_3), 3.86 (s, 1H, CH), 3.95–3.98 (m, 2H, OCH_2), 6.74–7.88 (m, 9H, Ar), 9.87 (br s, 1H, OH). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 36.2 (CH_3), 54.5 (CH_2), 56.2 (CH), 112.5, 114.3, 114.4, 114.5, 115.8, 116.0, 128.5, 130.0, 130.1, 130.5, 130.6, 133.0, 157.7. Anal. Calcd. for $C_{18}H_{16}BrNO_2$: C, 60.35; H, 4.50; N, 3.91; Found: C, 60.53; H, 4.56; N, 3.79.

5-[Ethoxy(phenyl)methyl]-6-hydroxyquinoline (**5i**)

Gray powder (0.20 g, yield 71 %). Mp 230–233 °C. IR: 3,209, 3,102, 2,937, 1,631, 1,607, 1,551, 1,403 cm^{-1}. ^1H NMR (300 MHz, $CDCl_3$) δ: 1.31 (br, 3H, CH_3), 3.98–4.01 (m, 2H, OCH_2), 4.48 (s, 1H, CH), 7.10–7.84 (m, 9H, Ar), 9.99 (br s, 1H, OH).

^{13}C NMR (75 MHz, $CDCl_3$) δ: 14.9 (CH_3), 57.3 (CH_2), 73.9 (CH), 118.4, 119.1, 119.3, 122.2, 123.3, 125.2, 127.1, 127.8, 128.1, 128.7, 128.9, 143.3, 150.9, 156.1. Anal. Calcd. for $C_{18}H_{17}NO_2$: C, 77.40; H, 6.13; N, 5.01; Found: C, 77.46; H, 6.23; N, 4.89.

5-[Ethoxy(4-methoxyphenyl)methyl]-quinolin-6-ol (**5j**)

Gray powder (0.21 g, yield 72 %). Mp 270–272 °C. IR: 3,321, 3,152, 3,019, 2,956, 1,672, 1,615, 1,532, 1,471 cm^{-1}. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 1.29 (br, 3H, CH_3), 3.86 (s, 3H, OCH_3), 3.96–3.99 (m, 2H, OCH_2), 4.31 (s, 1H, CH), 7.12–7.93 (m, 10H, Ar), 10.02 (br s, 1H, OH). Anal. Calcd. for $C_{19}H_{19}NO_3$: C, 73.77; H, 6.19; N, 4.53; Found: C, 73.83; H, 6.21; N, 4.47.

14-(4-Methoxyphenyl)-14*H*-dibenzo[*a*,*j*]xanthene (**6**) [43]

White powder (0.28 g, yield 73 %). Mp 242–245 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ: 3.76 (s, 3H, CH_3), 6.83 (s, 1H, CH), 6.87–8.00 (m, 16H, Ar). ^{13}C NMR (75 MHz, $CDCl_3$) δ: 41.7 (CH), 55.2 (CH_3), 114.5, 119.2, 119.8, 122.6, 123.1, 127.1, 128.9, 129.3, 129.5, 129.6, 132.8, 133.9, 153.1, 158.4.

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