

An efficient three-component reaction involving [3 + 1 + 1] furannulation leading to furanonaphthoquinones in water

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Abstract An efficient and clean green synthesis of highly substituted linear naphtho[2,3-*b*]-furan-4,9-dione derivatives, starting from 2-hydroxy-1,4-naphthoquinone, alkyl isocyanides and a variety of aldehydes, is described. This new method provides the first example of an efficient regioselective synthetic method for the synthesis of linear naphtho[2,3-*b*]-furan-4,9-dione ring systems by formation of three bonds. This [3 + 1 + 1] furannulation strategy affords furanonaphthoquinones in moderate to high yields, using water as a cheap, non-toxic, environmentally friendly solvent, in a one-step reaction, without the need of complicated work-up procedures.

Keywords Isocyanide; Multicomponent reaction; Naphtho[2,3-*b*]-furan-4,9-dione; Water.

Introduction

The toxic, hazardous, and volatile nature of many organic solvents, particularly chlorinated hydrocarbons and benzene, which are widely used in organic synthesis procedures, has posed a serious threat to the environment. In recent years, studies of low waste routes and clean reaction media for enhanced selectivity and energy minimization are among the

key interests of synthetic organic chemists [1, 2]. The use of aqueous medium in organic synthesis has attracted much attention for environmental, economical, safety reasons and is distinguished by showing unique reactivities and selectivities which are not observed for reactions in organic media [3, 4]. On the other hand, multi-step syntheses produce considerable amounts of waste mainly due to a series of complex isolation procedures often involving environmentally unfavourable solvents after each step. Unlike the stepwise formation of individual bonds in the target molecule, the advantageous attribute of multicomponent reactions is the inherent formation of several bonds in one operation without isolating the intermediates, changing the reaction conditions, or adding any further reagents [5]. Thus, the combination of steps into a multi-step, one-pot multicomponent reaction sequence can be economically and environmentally very advantageous as long as the overall yield and efficiency are not adversely affected. In other words, the environmental acceptability of the process is improved when a multicomponent strategy is applied. In addition, the use of water as a green solvent in combination with multicomponent approaches represents a powerful green chemical technology procedures.

Among various classes of fused annulated furans, furanonaphthoquinones have attracted widespread interest in view of their presence in natural products, and their pharmacological activities [6]. A great

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number of furanonaphthoquinones, are natural products exhibiting a broad spectrum of biological activity [7]. The diversity of their natural origins and biological activities has both motivated efforts towards their syntheses or to find pathways to structural analogues [8–20]. Many of the synthesis protocols reported so far suffer from disadvantages, such as relying on multi-step reactions [8b, d, 10a], difficulties in controlling regiochemistry [8i, 13a], generating by-products [8f, 14], low yields [8g, 14], use of metal-containing reagents [8e, 11–13a, 16], and special starting materials [16]. Therefore, the development of new, efficient methods for the preparation of linear naphtho[2,3-*b*]-furan-4,9-dione derivatives is still strongly desirable.

Results and discussion

This communication shows that one-pot three-component reactions of isocyanides with various aldehydes in the presence of 2-hydroxy-1,4-naphthoquinone can successfully take place in water as a cheap, non-toxic green solvent at 75°C within 2 h.

As far as we know, there is no report concerning the synthesis of naphtho[2,3-*b*]-furan-4,9-dione ring systems by concomitant formation of three bonds. As part of our research to develop green chemistry methods by synthesis of target molecules in aqueous medium [21], in the present work 2-hydroxy-1,4-naphthoquinone was used for the formation of novel furanonaphthoquinone derivatives based on a linear naphtho[2,3-*b*]-furan-4,9-dione skeleton (Scheme 1).

The one-pot three-component condensation reactions of 2-hydroxy-1,4-naphthoquinone **1** with vari-

ous aldehydes **2** in the presence of alkyl isocyanides **3** proceeded spontaneously in water at 75°C and were complete after 2 h to afford 2-(alkylamino)-3-alkylnaphtho[2,3-*b*]-furan-4,9-diones **4**, in moderate to good yields. ¹H and ¹³C NMR spectra of the crude products clearly indicated the formation of fused naphthofuroquinone **4**. All the products were characterized by FT-IR, ¹H, and ¹³C NMR spectra and elemental analyses.

The ¹H NMR spectrum of **4a** consists of multiplet signals for cyclohexyl ring ($\delta = 1.24$ – 2.34 ppm) and the N–CH group ($\delta = 3.65$ ppm). A fairly broad doublet ($\delta = 4.97$ ppm, ³*J*_{HH} = 7.3 Hz) is observed for the NH group. The presence of an amine proton is confirmed by exchange with D₂O indicating an exchangeable proton. The aromatic hydrogens give rise to characteristic signals in the aromatic region of the spectrum.

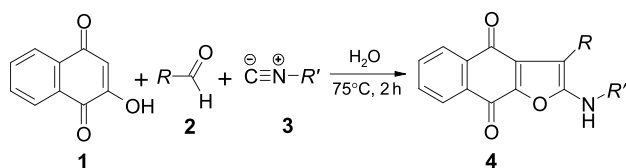
The ¹H decoupled ¹³C NMR spectrum of **4a** shows twenty distinct resonances in agreement with the suggested structure. Partial assignment of these resonances is given in the Experimental section.

The structural assignments made on the basis of the ¹H and ¹³C NMR spectra of **4a** was supported by measurement of its IR spectra. The IR spectrum of **4a** showed absorptions at $\bar{\nu} = 1678$ and 1645 cm^{-1} due to the carbonyls and the amino group at $\bar{\nu} = 3287\text{ cm}^{-1}$ as a weak broad band.

The ¹H and ¹³C NMR spectra of **4b–4i** are similar to those of **4a** and the results are summarized in the Experimental section.

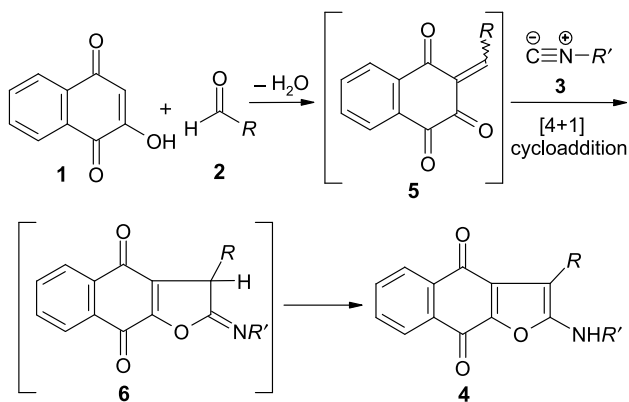
The scope of the reaction regarding the aldehydes was examined and found that aliphatic aldehydes, the substituted groups of aromatic aldehydes, such as electron-withdrawing groups and electron-donating groups can tolerate the reaction conditions with good yields. To explore the scope of this reaction regarding the isocyanides, we have extended it to various alkyl isocyanides. We have found that the reaction proceeds very efficiently with various alkyl groups.

The synthesis of linear naphtho[2,3-*b*]-furan-4,9-dione derivatives **4** can be rationalized by initial formation of a conjugated electron-deficient heterodiene **5** by a *Knoevenagel* condensation of the cyclic 2-hydroxy-1,4-naphthoquinone **1** and the aldehyde **2**. The next step of this mechanism could involve the [4 + 1] cycloaddition reaction of the electron-deficient heterodiene moiety of adduct **5** with the isocyanide to afford an iminolactone intermediate **6**.



	R	R'	Yield %
4a	4-Nitrophenyl	Cyclohexyl	75
4b	2,6-Dichlorophenyl	Cyclohexyl	64
4c	Phenyl	Cyclohexyl	70
4d	Phenyl	<i>tert</i> -Octyl	61
4e	3-Hydroxyphenyl	<i>tert</i> -Butyl	60
4f	4-(<i>N,N</i> -Dimethyl)phenyl	Cyclohexyl	63
4g	2,5-Dimethoxyphenyl	<i>tert</i> -Octyl	69
4h	Methyl	Cyclohexyl	71
4i	Propyl	<i>tert</i> -Butyl	68

Scheme 1



Scheme 2

The subsequent isomerization of iminolactone **6** leads to formation of product **4** (Scheme 2).

In conclusion, the paper describes a facile and efficient environmentally friendly process for the synthesis of biologically interesting highly functionalized linear naphtho[2,3-*b*]-furan-4,9-dione derivatives starting from easily available reagents. The reaction proceeds along a rather complex pathway but is very simple from the experimental point of view and allows the creation of a fused naphthofurquinone ring with concomitant formation of two new C–C bonds and one C–O bond in a single operation. In other words, this reaction results in high bond efficiency. The notable features of this procedure are its neutral and mild reaction conditions, improved yields, easy work-up, being environmentally friendly and use of water as an ideal reaction medium.

Experimental

Melting points were measured on a Büchi 535 apparatus. Elemental analyses were performed using a Heraeus CHN–O–Rapid analyzer and their results agreed favourably with calculated values. FT-IR Spectra were recorded on a Bruker Equinox-55 spectrometer. ^1H and ^{13}C NMR spectra were recorded on a Bruker DRX-400 Avance spectrometer at 400.13 and 100.77 MHz, respectively, with CDCl_3 as solvent. The solvents, aldehydes and 1,1,3,3-tetramethylbutyl isocyanides used in this work were purchased from Merck and the *tert*-butyl isocyanide was obtained from Fluka (Buchs, Switzerland). The 2-hydroxy-1,4-naphthoquinone was obtained from Aldrich. All reagents were used without further purification.

Typical procedure for the preparation of **4a**

To a magnetically stirred suspension of 0.174 g 2-hydroxy-1,4-naphthoquinone (1.0 mmol) and 0.151 g 4-nitrobenzaldehyde (1.0 mmol) in 30 cm^3 water were added 0.110 g

cyclohexyl isocyanide (1.0 mmol) via a syringe and heated for 2 h at 75°C. After cooling to room temperature, the resulting black precipitate was filtered off and washed with 20 cm^3 water. The solid residue was dried and crystallized from diethyl ether:*n*-hexane (1:4) to yield 0.313 g **4a** as blue crystals (75%).

2-(Cyclohexylamino)-3-(4-nitrophenyl)naphtho[2,3-*b*]furan-4,9-dione (**4a**, $\text{C}_{24}\text{H}_{20}\text{N}_2\text{O}_5$)

Mp 246–249°C; FT-IR (KBr): $\bar{\nu}_{\text{max}}$ = 3287 (N–H), 1678, 1645 (C=O), 1585 (C=C) cm^{-1} ; ^1H NMR (CDCl_3 , 400.1 MHz): δ = 1.24–2.34 (m, 5 CH_2), 3.87 (m, N–CH), 4.97 (d, $^3J_{\text{HH}}$ = 7.3 Hz, NH), 7.66, 8.28 (2d, $^3J_{\text{HH}}$ = 8.7 Hz, $\text{C}_6\text{H}_4\text{NO}_2$), 7.62, 7.70, 8.01, 8.15 (4 m, C_6H_4) ppm; ^{13}C NMR (CDCl_3 , 100.7 MHz): δ = 24.71, 25.25, 33.83 (5 CH_2 of cyclohexyl), 52.23 (N–CH), 96.18 (C=C–NH), 124.05, 126.24, 126.58, 129.88, 130.52, 132.67, 132.87, 133.01, 133.97, 137.68, 143.91, 146.41 (arom. carbons, C=C), 158.94 (C=C–NH), 169.92, 181.72 (2C=O) ppm.

2-(Cyclohexylamino)-3-(2,6-dichlorophenyl)naphtho[2,3-*b*]furan-4,9-dione (**4b**, $\text{C}_{24}\text{H}_{19}\text{Cl}_2\text{NO}_3$)

Blue crystals (0.282 g, 64%); mp 239–241°C; FT-IR (KBr): $\bar{\nu}_{\text{max}}$ = 3263 (N–H), 1648 (C=O), 1598 (C=C) cm^{-1} ; ^1H NMR (CDCl_3 , 400.1 MHz): δ = 1.16–2.06 (m, 5 CH_2), 3.75 (m, N–CH), 4.40 (d, $^3J_{\text{HH}}$ = 7.4 Hz, NH), 7.26–4.43 (m, $\text{C}_6\text{H}_3\text{Cl}_2$), 7.58, 7.67, 7.96, 8.16 (4m, C_6H_4) ppm; ^{13}C NMR (CDCl_3 , 100.7 MHz): δ = 24.79, 25.29, 34.04 (5 CH_2 of cyclohexyl), 52.57 (N–CH), 92.65 (C=C–NH), 126.23, 126.33, 128.21, 130.17, 132.29, 132.67, 132.75, 133.38, 133.77, 137.05, 143.22 (arom. carbons, C=C, CH=CH), 158.48 (C=C–NH), 169.65, 181.50 (2C=O) ppm.

2-(Cyclohexylamino)-3-phenylnaphtho[2,3-*b*]furan-4,9-dione (**4c**, $\text{C}_{24}\text{H}_{21}\text{NO}_3$)

Blue crystals (0.260 g, 70%); mp 245–248°C; FT-IR (KBr): $\bar{\nu}_{\text{max}}$ = 3226 (N–H), 1676, 1640 (C=O), 1583 (C=C) cm^{-1} ; ^1H NMR (CDCl_3 , 400.1 MHz): δ = 1.18–2.34 (m, 5 CH_2), 3.85 (m, N–CH), 4.95 (br, s, NH), 7.15–7.50 (m, C_6H_5), 7.58, 7.67, 8.00, 8.15 (4m, C_6H_4) ppm; ^{13}C NMR (CDCl_3 , 100.7 MHz): δ = 24.73, 25.33, 33.90 (5 CH_2 of cyclohexyl), 52.30 (N–CH), 98.83 (C=C–NH), 126.08, 126.41, 127.68, 128.85, 129.33, 130.12, 131.20, 132.20, 133.22, 133.38, 133.67, 143.26 (10 arom. carbons, C=C), 159.15 (C=C–NH), 169.25, 181.91 (2C=O) ppm.

3-Phenyl-2-[(1,1,3,3-tetramethylbutyl)amino]naphtho[2,3-*b*]furan-4,9-dione (**4d**, $\text{C}_{26}\text{H}_{27}\text{NO}_3$)

Blue crystals (0.245 g, 61%); mp 262–264°C; FT-IR (KBr): $\bar{\nu}_{\text{max}}$ = 3287 (N–H), 1673, 1643 (C=O), 1582 (C=C) cm^{-1} ; ^1H NMR (CDCl_3 , 400.1 MHz): δ = 0.98 (s, $\text{C}(\text{CH}_3)_3$), 1.52 (s, $\text{C}(\text{CH}_3)_2$), 1.78 (s, CH_2), 5.06 (br, s, NH), 7.46 (m, C_6H_5), 7.58, 7.67, 8.00, 8.15 (4m, C_6H_4) ppm; ^{13}C NMR (CDCl_3 , 100.7 MHz): δ = 30.26 (CMe_2), 31.39 (CMe_3), 31.44 (CH_2), 53.46 (CMe_3), 57.67 (CMe_2), 100.07 (C=C–NH), 126.03, 126.39, 127.76, 128.87, 129.38, 130.15, 132.14, 133.27, 133.45, 133.59, 133.66, 143.74 (arom. carbons, C=C), 159.40 (C=C–NH), 169.03, 181.90 (2C=O) ppm.

2-[(*Tert*-butyl)amino-3-(3-hydroxyphenyl)naphtho[2,3-*b*]-furan-4,9-dione (**4e**, C₂₄H₂₁NO₄)

Blue crystals (0.232 g, 60%); mp 241–244°C; FT-IR (KBr): $\bar{\nu}_{\max}$ = 3338 (N–H, O–H), 1689, 1640 (C=O), 1585 (C=C) cm⁻¹; ¹H NMR (CDCl₃, 400.1 MHz): δ = 1.45 (s, C(CH₃)₃), 5.08 (br, s, NH), 7.33–7.41 (m, C₆H₄OH), 7.57, 7.66, 7.97, 8.15 (4H, 4m, C₆H₄), 9.93 (1H, s, OH) ppm; ¹³C NMR (CDCl₃, 100.7 MHz): δ = 29.85 (CMe₃), 54.14 (CMe₃), 100.57 (C=C–NH), 115.03, 116.50, 121.15, 122.12, 123.01, 126.17, 126.56, 130.30, 131.36, 132.33, 133.87, 137.80, 143.54, 156.76 (arom. carbons, C=C), 159.99 (C=C–NH), 169.12, 181.90 (2C=O) ppm.

2-(Cyclohexylamino)-3-[4-(dimethylamino)phenyl]-naphtho[2,3-*b*]furan-4,9-dione (**4f**, C₂₆H₂₆N₂O₃)

Blue crystals (0.261 g, 63%); mp 248–251°C; FT-IR (KBr): $\bar{\nu}_{\max}$ = 3299 (N–H), 1636 (C=O), 1579 (C=C) cm⁻¹; ¹H NMR (CDCl₃, 400.1 MHz): δ = 1.18–2.07 (m, 5CH₂), 2.97 (s, NMe₂), 3.85 (m, N–CH), 4.96 (d, ³J_{HH} = 8.3 Hz, NH), 6.77, 7.34 (4H, 2d, ³J_{HH} = 8.7 Hz, C₆H₄NMe₂), 7.57, 7.65, 8.00, 8.14 (4m, C₆H₄) ppm; ¹³C NMR (CDCl₃, 100.7 MHz): δ = 24.74, 25.37, 33.90 (5CH₂ of cyclohexyl), 40.37 (NMe₂), 52.23 (N–CH), 99.93 (C=C–NH), 112.41, 117.10, 125.97, 126.32, 130.08, 131.24, 131.85, 133.32, 133.51, 133.75, 142.84, 149.84 (10 arom. carbons, C=C), 159.43 (C=C–NH), 168.48, 182.04 (2C=O) ppm.

3-(2,4-Dimethoxyphenyl)-2-[(1,1,3,3-tetramethylbutyl)-amino]naphtho[2,3-*b*]furan-4,9-dione (**4g**, C₂₈H₃₁NO₅)

Blue crystals (0.319 g, 69%); mp 251–254°C; FT-IR (KBr): $\bar{\nu}_{\max}$ = 3295 (N–H), 1666, 1641 (C=O), 1573 (C=C) cm⁻¹; ¹H NMR (CDCl₃, 400.1 MHz): δ = 0.97 (s, C(CH₃)₃), 1.51 (s, C(CH₃)₂), 1.80 s, CH₂), 3.77, 3.79 (2s, 2OCH₃), 5.03 (br, s, NH), 6.89–6.98 (m, C₆H₃OMe₂), 7.56, 7.65, 7.99, 8.15 (4m, C₆H₄) ppm; ¹³C NMR (CDCl₃, 100.7 MHz): δ = 30.39 (CMe₂), 31.43 (CMe₃), 31.76 (CH₂), 53.66 (CMe₃), 55.79, 56.63 (2OCH₃), 57.23 (CMe₂), 97.05 (C=C–NH), 113.14, 114.61, 117.64, 119.63, 123.54, 125.95, 126.39, 131.33, 131.99, 133.36, 133.51, 144.01, 150.64, 153.61 (arom. carbons, C=C), 159.41 (C=C–NH), 168.71, 181.63 (2C=O) ppm.

2-(Cyclohexylamino)-3-methylnaphtho[2,3-*b*]furan-4,9-dione (**4h**, C₁₉H₁₉NO₃)

Blue crystals (0.220 g, 71%); mp 192–195°C; FT-IR (KBr): $\bar{\nu}_{\max}$ = 3425 (N–H), 1642 (C=O), 1588 (C=C) cm⁻¹; ¹H NMR (CDCl₃, 400.1 MHz): δ = 1.24–2.08 (m, 5CH₂), 2.19 (s, CH₃), 3.81 (m, N–CH), 4.97 (d, ³J_{HH} = 7.3 Hz, NH), 7.57, 7.66, 8.03, 8.13 (4H, 4m, C₆H₄) ppm; ¹³C NMR (CDCl₃, 100.7 MHz): δ = 15.28 (CH₃), 24.78, 25.21, 33.54 (5CH₂ of cyclohexyl), 53.53 (N–CH), 96.12 (C=C–NH), 122.66, 126.01, 126.75, 131.52, 133.29, 134.10, 141.29, 146.36 (arom. carbons, C=C), 158.87 (C=C–NH), 164.60, 182.97 (2C=O) ppm.

2-(*Tert*-butylamino)-3-propylnaphtho[2,3-*b*]furan-4,9-dione (**4i**, C₁₉H₂₁NO₃)

Blue crystals (0.212 g, 68%); mp 197–199°C; FT-IR (KBr): $\bar{\nu}_{\max}$ = 3344 (N–H), 1675, 1640 (C=O), 1582 (C=C) cm⁻¹;

¹H NMR (CDCl₃, 400.1 MHz): δ = 0.94 (t, ³J_{HH} = 7.3 Hz, CH₃), 1.45 (s, C(CH₃)₃), 1.58 (m, –CH₂CH₃), 2.54 (t, ³J_{HH} = 7.3 Hz, =C–CH₂), 4.28 (br, s, NH), 7.57, 7.65, 8.01, 8.12 (4m, C₆H₄) ppm; ¹³C NMR (CDCl₃, 100.7 MHz): δ = 13.88 (CH₃), 22.61 (–CH₂CH₃), 24.62 (=C–CH₂), 30.17 (CMe₃), 53.93 (CMe₃), 101.77 (C=C–NH), 126.01, 126.15, 131.89, 131.97, 133.07, 133.62, 133.75, 143.73 (arom. carbons, C=C), 159.73 (C=C–NH), 168.82, 182.94 (2C=O) ppm.

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