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Cyclization of 2-(3-R-prop-2-ynyloxy)-4-phenyliminonaphthalen-1(4*H*)-ones into 2-R-methylidene-6-phenylamino-2,3-dihydronaphtho[2,1-*b*]-1,4-dioxines

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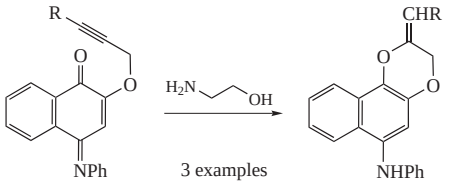
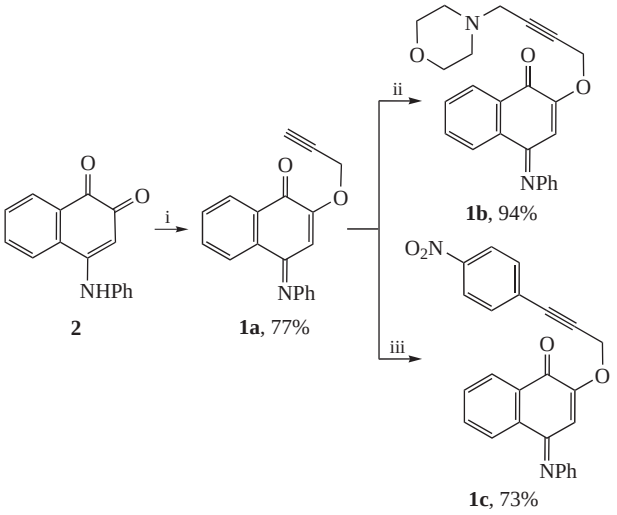
2-(3-R-prop-2-ynyloxy)-4-phenyliminonaphthalen-1(4*H*)-ones on heating with ethanolamine undergo cyclization into 2-R-methylidene-6-phenylamino-2,3-dihydronaphtho[2,1-*b*]-1,4-dioxines.



The benzo-1,4-dioxine moiety is found in some biologically active substances, including the natural ones. Among those are antihypertensive preparations (doxazosin, piperoxan)[1] and antidepressants (idazoxan, fluparoxan).[2] A number of useful derivatives, such as eusiderin,[3] haedoxan A,[4] purpurenol,[5] silybin,[6] and isosilybin[7] were isolated from vegetable feed.

This work offers a novel method to synthesize the benzo-1,4-dioxine derivatives, namely, 2-R-methylidene-6-phenylamino-2,3-dihydronaphtho[2,1-*b*]-1,4-dioxines. The transformations studied are the cyclizations of the 2-propargyloxy derivatives of 1,4-naphthoquinone. It is worth noting that the precedents of preparing naphtho-1,4-dioxines are few in number.[8]

Quinones containing substitutes with a triple bond, are the convenient building blocks for the synthesis of various fused heterocyclic compounds.[9,10] A new type of such a substrate **1a** was obtained from naphthoquinone **2** and propargyl chloride (Scheme 1). The Mannich and Sonogashira reactions were used to extend the series of starting compounds and to obtain alkynes **1b**,**c** with substitutes of various origin (Scheme 1).†



Scheme 1 *Reagents and conditions*: i, $ClCH_2C{\equiv}CH$, K_2CO_3, DMF, 55 °C, 2 h; ii, bis(morpholino)methane, CuCl, 1,4-dioxane, room temperature, 1 h; iii, 4-$NO_2C_6H_4I$, $PdCl_2(PPh_3)_2$, CuI, Na_2CO_3, Py, H_2O, 80 °C, 0.5 h.

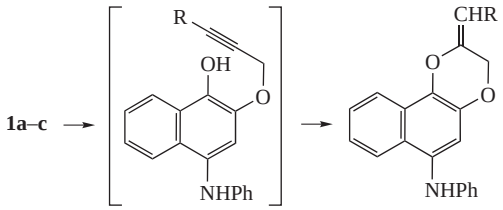
† Elemental analysis was performed with a CHN-analyzer (Model 1106, Carlo Erba). The NMR spectra were recorded on a Bruker AV 400 spectrometer (400.13 MHz) in $CDCl_3$. Melting points were determined with a Kofler apparatus. Mass spectra were obtained on a Thermo Electron Corporation DFS mass spectrometer (70 eV) using direct injection, the temperature of the ionization chamber was 220–270 °C. The IR spectra were recorded on a Shimadzu IRTracer-100 instrument with GS10802-X Quest ATR ZnSe Accessory (Specac). Column chromatography was performed on alumina (50–150 μm, TU 6-09-3916-75) and Kieselgel 60 plates (Merck) were used for TLC analysis.

Quinone **2** was produced by the reported method.[15]

*4-Phenylimino-2-(prop-2-ynyloxy)naphthalen-1(4*H*)-one* **1a**. A mixture of 4-phenylamino-1,2-naphthoquinone **2** (4.3 g, 17.3 mmol), propargyl chloride (1.9 g, 25.5 mmol), K_2CO_3 (2.4 g, 17.3 mmol) in DMF (35 ml) was stirred at 55 °C for 2 h, then cooled and treated with cold water. The precipitate was collected by filtration, washed with water and air-dried. Yield: 3.8 g (77%), mp 134–135 °C (ethanol). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.54 (t, 1H, ≡CH, *J* 2.2 Hz), 4.60 (d, 2H, CH_2, *J* 2.2 Hz), 6.58 (s, 1H, H_{Ar}), 6.94 (m, 2H, *o*-H_{Ph}), 7.20 (m, 1H, *p*-H_{Ph}), 7.41 (m, 2H, *m*-H_{Ph}), 7.65 (td, 1H, H_{Ar}, *J* 1.3, 7.5 Hz), 7.73 (td, 1H, H_{Ar}, *J* 1.3, 7.5 Hz), 8.22 (dd, 1H, H_{Ar}, *J* 1.3, 7.8 Hz), 8.50 (dd, 1H, H_{Ar}, *J* 1.3, 7.8 Hz). ^{13}C NMR ($CDCl_3$, 100 MHz) δ: 56.1 (CH_2), 76.5 (–C≡), 77.5 (≡CH), 104.4 (CH), 120.5 (2CH), 124.9 (CH), 125.4 (CH), 126.6 (CH), 129.1 (2CH), 131.2 (CH), 133.5 (CH), 134.7 (C), 150.4 (C), 154.1 (C), 154.7 (C), 180.2 (C=O). IR (ν/cm^{-1}): 1668 (C=O), 2118 (C≡C), 3264 (≡C–H). Found (%): C, 79.15; H, 4.65; N, 5.01. Calc. for $C_{19}H_{13}NO_2$ (%): C, 79.43; H, 4.56; N, 4.88.

*2-(4-Morpholinobut-2-ynyloxy)-4-phenyliminonaphthalen-1(4*H*)-one* **1b**. A solution of bis(morpholino)methane (325 mg, 1.74 mmol) in 1,4-dioxane (3 ml) was added under argon to a mixture of alkyne **1a** (500 mg, 1.74 mmol) and CuCl (30 mg) in 1,4-dioxane (12 ml) and the mixture was stirred for 1 h at room temperature. Water was added, and the precipitate was collected by filtration, washed with water and air-dried. Yield: 630 mg (94%), mp 125–126 °C (light petroleum–toluene). ^{1}H NMR ($CDCl_3$, 400 MHz) δ: 2.44 (t, 4H, 2CH_2, *J* 4.6 Hz), 3.25 (t,

Compounds **1** were cyclized into dioxines **3** under the action of excess ethanolamine upon heating in pyridine or ethanol (Scheme 2).‡ Selectivity of the process and the yields of products with various R substituents depended on the solvent employed. For example, the yield of compound **3a** in ethanol (73%) was higher than in pyridine (44%). An opposite effect was observed in the synthesis of **3b** when pyridine appeared more suitable and the use of ethanol resulted in the formation of a great amount of side products that brought difficulties to isolation. The complete conversion of terminal alkyne **1a**, depending on the solvent, took



a R = H
b R = morpholinomethyl
c R = 4-$NO_2C_6H_4$

Scheme 2 *Reagents and conditions*: $NH_2CH_2CH_2OH$, Py or EtOH, reflux, 1–6 h.

3–6 h. For disubstituted derivatives **1b**,**c**, the reaction time was 1–1.5 h. In the absence of ethanolamine, alkyne **1a** was not converted into dioxine **3a** at reflux but was slowly decomposed.

Probably, the quinones **1** are cyclized into dioxines **3** under the action of ethanolamine through the formation of hydroquinone **4** followed by 6-*exo*-*dig* cyclization (see Scheme 2). The stage of ring closing is similar to the well-known cyclization of 2-propargyloxyphenols into benzo-1,4-dioxines catalyzed by the metal complex Pd^{II}/Cu^{I} or HgO.[11,12] Note that prolongation of synthesis of compound **1c** causes its destruction and the formation of small amounts of compound **3c** (8–10%).

Intermediate N-heterocyclic compounds in the reaction between alkynes **1** and ethanolamine were not isolated. Apparently, ethanolamine served as a reducing agent[13] rather than nucleophile. The conversion of 2,3-dimethyl-5-phenylethynyl-1,4-naphthoquinone into 9,8-dimethyl-7-hydroxynaphtho[1,8-*bc*]pyrane in the presence of hydrazine exemplifies a similar reductive cyclization among ethynylquinones.[14]

The structures of compounds **1** and **3** were confirmed by a set of spectral data (IR, ^1H, ^{13}C NMR, mass spectra) and elemental analysis. The molecular structure of dioxine **3a** was confirmed by X-ray diffraction (Figure 1).§

Conformation of the 1,4-dioxinic cycle is close to the envelope with C(3) atom deviation by 0.610(2) Å from the plane of rest atoms (Figure 1). The same conformation is observed in several compounds containing benzo[1,4]dioxin-2-ylidene fragment, for example in 2-(benzo[1,4]dioxin-2-ylidene)-*N*,*N*-diethylacetamide.[11(*c*)] The angle between phenyl and naphthalene planes is

2H, C≡CCH$_2$N, *J* 1.8 Hz), 3.69 (t, 4H, 2CH$_2$, *J* 4.6 Hz), 4.62 (t, 2H, OCH$_2$C≡C, *J* 1.8 Hz), 6.55 (s, 1H, H$_{Ar}$), 6.92 (m, 2H, *o*-H$_{Ph}$), 7.18 (m, 1H, *p*-H$_{Ph}$), 7.40 (m, 2H, *m*-H$_{Ph}$), 7.65 (m, 1H, H$_{Ar}$), 7.73 (m, 1H, H$_{Ar}$), 8.21 (m, 1H, H$_{Ar}$), 8.49 (m, 1H, H$_{Ar}$). ^{13}C NMR (CDCl$_3$, 100 MHz) δ: 47.4 (CH$_2$N), 52.4 (2CH$_2$), 56.6 (CH$_2$O), 66.9 (2CH$_2$), 78.0, 84.2 (C≡C), 104.1 (CH), 120.4 (2CH), 124.8 (CH), 125.4 (CH), 126.6 (CH), 129.1 (2CH), 131.2 (CH), 133.5 (CH), 134.7 (C), 150.5 (C), 154.4 (C), 154.8 (C), 180.3 (C=O). IR (ν/cm^{-1}): 1667 (C=O). Found (%): C, 74.60; H, 5.71; N, 7.22. Calc. for $C_{24}H_{22}N_2O_3$ (%): C, 74.59; H, 5.74; N, 7.25.

2-[3-(4-Nitrophenyl)prop-2-ynyloxy]-4-phenyliminonaphthalen-1(4H)-one **1c**. A mixture of alkyne **1a** (500 mg, 1.74 mmol), 1-iodo-4-nitrobenzene (480 mg, 1.7 mmol), $PdCl_2(PPh_3)_2$ (20 mg, 0.028 mmol), CuI (10 mg, 0.052 mmol) in pyridine (20 ml) was stirred under argon and heated to 60 °C. Then, a hot solution of Na_2CO_3 (320 mg, 3.0 mmol) in H_2O (5 ml) was added, and the mixture was refluxed for 0.5 h. After that, toluene (100 ml) was added. The organic layer was separated, washed with water (3×200 ml), dried over $MgSO_4$ and evaporated to dryness on a rotary evaporator. A mixture product **1c** and 1-iodo-4-nitrobenzene were separated by chromatography on Al_2O_3 (elution with dichloromethane). Yield: 300 mg (73%), mp 125–126 °C (light petroleum–toluene) ^1H NMR (CDCl$_3$, 400 MHz) δ: 4.86 (s, 2H, CH$_2$), 6.55 (s, 1H, H$_{Ar}$), 6.89 (m, 2H, *o*-H$_{Ph}$), 7.15 (m, 1H, *p*-H$_{Ph}$), 7.29 (m, 2H, *m*-H$_{Ph}$), 4.48 (dt, 2H, *m*-H, *J* 2.1, 9.0 Hz), 7.67 (m, 1H, H$_{Ar}$), 7.75 (m, 1H, H$_{Ar}$), 8.20 (dt, 2H, *o*-H, *J* 2.1, 9.0 Hz), 8.23 (m, 1H, H$_{Ar}$), 8.51 (m, 1H, H$_{Ar}$). ^{13}C NMR (CDCl$_3$, 100 MHz) δ: 56.8 (CH$_2$), 86.8, 86.9 (C≡C), 104.2 (CH), 120.3 (2CH), 123.7 (2CH), 124.9 (CH), 125.5 (CH), 126.7 (CH), 128.5 (C), 129.1 (2CH), 131.2 (C), 131.3 (CH), 132.7 (2CH), 133.6 (CH), 134.7 (C), 147.8 (C), 150.5 (C), 154.4 (C), 154.7 (C), 180.2 (C=O). IR (ν/cm^{-1}): 1661 (C=O), 1339, 1514 (NO$_2$). Found (%): C, 73.43; H, 4.04; N, 6.73. Calc. for $C_{25}H_{16}N_2O_4$ (%): C, 73.52; H, 3.95; N, 6.86.

‡ *Compounds* **3** *(general procedure)*. A mixture of alkyne **1** (1.74 mmol), and ethanolamine (1.06 g, 17.4 mmol) in ethanol (or in pyridine) (5 ml) was refluxed for 1–6 h. Toluene (100 ml) and water (100 ml) were then added, the organic layer was separated, dried over $MgSO_4$ and evaporated to dryness under reduced pressure. The crude product was purified by column chromatography on Al_2O_3 (elution with dichloromethane). Subsequent recrystallization gave pure products **3**.

2-Methylidene-6-phenylamino-2,3-dihydronaphtho[2,1-b]-1,4-dioxine **3a**. Yield 370 mg (73%), mp 99–100 °C (light petroleum). ^1H NMR (CDCl$_3$, 400 MHz) δ: 4.47 (d, 1H, =CH, *J* 1.7 Hz), 4.62 (s, 2H, CH$_2$), 4.93 (d, 1H, =CH, *J* 1.8 Hz), 5.71 (br. s, 1H, NH), 6.87 (m, 3H, H$_{Ph}$), 7.06 (br. s, 1H, H$_{Ar}$), 7.23 (m, 2H, H$_{Ph}$), 7.38 (m, 1H, H$_{Ar}$), 7.53 (m, 1H, H$_{Ar}$), 7.94 (m, 1H, H$_{Ar}$), 8.18 (m, 1H, H$_{Ar}$). ^{13}C NMR (CDCl$_3$, 100 MHz) δ: 65.1 (CH$_2$), 91.8 (=CH$_2$), 110.5 (CH), 116.4 (2CH), 120.0 (CH), 120.6 (CH), 122.5 (CH), 124.5 (CH), 125.2 (C), 125.3 (C), 126.6 (CH), 129.5 (2CH), 132.7 (C), 133.1 (C), 139.2 (C), 145.6 (C), 150.2 (C). Found (%): C, 79.18; H, 5.23; N, 4.87. Calc. for $C_{19}H_{15}NO_2$ (%): C, 78.87; H, 5.23; N, 4.84.

2-(2-Morpholinoethylidene)-6-phenylamino-2,3-dihydronaphtho-[2,1-b]-1,4-dioxine **3b**. Yield 418 mg (62%), mp 128–130 °C (light petroleum). ^1H NMR (CDCl$_3$, 400 MHz) δ: 2.64 (br. s, 4H, 2CH$_2$), 3.42 (br. d, 2H, CH$_2$N, *J* 6.6 Hz), 3.78 (br. s, 4H, 2CH$_2$), 4.98 (br. s, 1H, =CH, *J* 6.6 Hz), 5.74 (s, 1H, NH), 6.88 (m, 3H, H$_{Ph}$), 7.05 (s, 1H, H$_{Ar}$), 7.23 (m, 2H, H$_{Ph}$), 7.40 (t, 1H, H$_{Ar}$, *J* 7.1 Hz), 7.55 (t, 1H, H$_{Ar}$, *J* 7.1 Hz), 7.95 (d, 1H, H$_{Ar}$, *J* 7.3 Hz), 8.13 (d, 1H, H$_{Ar}$, *J* 7.3 Hz). ^{13}C NMR (CDCl$_3$, 100 MHz) δ: 52.6 (CH$_2$), 53.6 (2CH$_2$), 65.5 (CH$_2$), 67.0 (2CH$_2$), 103.3 (=CH), 110.2 (CH), 116.6 (2CH), 120.2 (CH), 120.4 (CH), 122.5 (CH), 124.6 (CH), 125.1 (C), 125.4 (C), 126.7 (CH), 129.5 (2CH), 132.3 (C), 133.4 (C), 139.4 (C), 145.4 (C), 146.0 (C). Found (%): C, 74.04; H, 6.48; N, 7.24. Calc. for $C_{24}H_{24}N_2O_3$ (%): C, 74.21; H, 6.23; N, 7.21. HRMS, *m*/*z*: 388.1776 (calc. for $C_{24}H_{24}N_2O_3$, *m*/*z*: 388.1781 [M]$^+$).

2-(4-Nitrobenzylidene)-6-phenylamino-2,3-dihydronaphtho[2,1-b]-1,4-dioxine **3c**. Yield 435 mg (61%), mp 154–155 °C (light petroleum–toluene). ^1H NMR (CDCl$_3$, 400 MHz) δ: 4.74 (s, 2H, CH$_2$), 5.74 (s, 1H, =CH), 5.81 (s, 1H, NH), 6.94 (m, 3H, H$_{Ph}$), 7.08 (s, 1H, H$_{Ar}$), 7.26 (m, 2H, H$_{Ph}$), 7.46 (m, 1H, H$_{Ar}$), 7.64 (m, 1H, H$_{Ar}$), 7.93 (m, 2H, H$_{Ar}$), 8.00 (d, 1H, H$_{Ar}$, *J* 8.5 Hz), 8.19 (d, 1H, H$_{Ar}$, *J* 8.2 Hz), 8.28 (m, 2H, H$_{Ar}$). ^{13}C NMR (CDCl$_3$, 100 MHz) δ: 66.3 (CH$_2$), 105.3 (CH), 108.8 (CH), 117.2 (2CH), 120.2 (CH), 120.7 (CH), 122.6 (CH), 124.0 (2CH), 124.8 (C), 124.9 (CH), 125.2 (C), 127.4 (CH), 129.3 (2CH), 129.6 (2CH), 131.9 (C), 134.8 (C), 140.0 (C), 141.4 (C), 144.8 (C), 146.0 (C), 147.0 (C). Found (%): C, 73.60; H, 4.26; N, 6.84. Calc. for $C_{25}H_{18}N_2O_4$ (%): C, 73.16; H, 4.42; N, 6.83. HRMS, *m*/*z*: 410.1257 (calc. for $C_{25}H_{18}N_2O_4$, *m*/*z*: 410.1261 [M]$^+$).

§ *Crystal data for compound* **3a**. $C_{19}H_{15}NO_2$, *M* = 289.32, orthorhombic, space group *Pbca*, *a* = 9.4653(4), *b* = 9.1161(3) and *c* = 34.7687(16) Å, *V* = 3000.1(2) Å^3, *T* = 296 K, *Z* = 8, d_{calc} = 1.281 g cm^{-3}, μ(MoKα) = = 0.083 mm^{-1}, 17774 reflections collected, 2654 independent, final *R* indexes [2140 $I > 2\sigma(I)$]: R_1 = 0.0420, wR_2 = 0.1213, *S* = 1.085. Data were collected on a Bruker Kappa Apex II CCD diffractometer using graphite monochromated MoKα radiation. All usual procedures were performed with SHELX-97 program set.

CCDC 1469105 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* http://www.ccdc.cam.ac.uk.

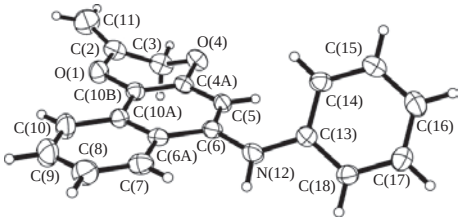


Figure 1 Molecular structure of dioxine **3a**.

equal to 54.61(8)°. The molecules in crystal are connected into chains along *b* axis by the hydrogen bonds N–H···O(4) [H···O 2.24(3) Å, ∠N–H…O 170(2)°].

In conclusion, 2-(3-R-prop-2-ynyloxy)-4-phenyliminonaphthalen-1(4*H*)-ones are subjected to reductive cyclization into 2-R-methylene-6-phenylamino-2,3-dihydronaphtho[2,1-*b*]-1,4-dioxines under the action of ethanolamine. The reaction is typical of such alkynes **1** and may be used to obtain angular naphtho-1,4-dioxines **3** with different sets of substitutes. The diversity of products may be enhanced additionally by applying various anilines to synthesize the starting 4-arylamino-1,2-naphthoquinones.

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