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Oximation of 2-(R^1-Amino)-4-(R^2-imino)naphthalen-1(4*H*)-ones

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Abstract—The oximation of 2-(R^1-amino)-4-(R^2-imino)naphthalen-1(4*H*)-ones with hydroxylamine hydrochloride in pyridine afforded 2-(R-amino)-4-(hydroxyimino)naphthalen-1(4*H*)-ones.

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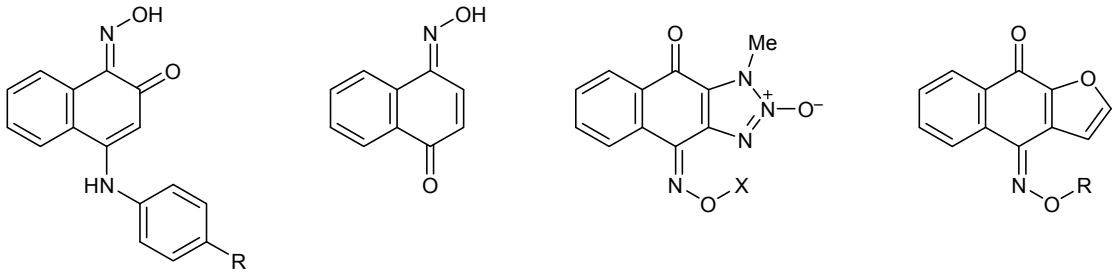
1,4-Naphthoquinone monooximes exhibit various biological activities. For example, (*Z*)-4-(arylamino)-1-(hydroxyimino)naphthalen-2(1*H*)-ones **1a** and **1b** showed cytotoxic activity [1], unsubstituted (4*E*)-4-(hydroxyimino)naphthalen-1(4*H*)-one (**2**) displayed antiparasitic properties [2], and naphthoquinone oximes **3** and **4** with a triazole or furan ring fused to the naphthalene system are promising as antitumor agents due to their pronounced cytotoxicity against human cancer cell lines [3, 4]. Replacement of one carbonyl group in polycyclic quinones by hydroxyimino or imino group was found to reduce their potential cardiotoxicity [4, 5].

Quinone monooximes can be synthesized by nitrosation of phenols or oximation of quinones. The conditions of the latter reaction are determined by the substrate nature. 1,4-Benzoquinone reacts with hydroxylamine hydrochloride in water at room temperature, the oximation of 1,4-naphthoquinone requires heating in boiling ethanol [6], whereas 9,10-anthraquinone reacts only at 180°C [7]. The presence of substituents in the initial quinone molecule affects not only the rate of oximation but also its direction. The oximation of 4-acetylamino-1,2-naphthoquinone yields 4-acetylamino-2-(hydroxyimino)naphthalen-1(2*H*)-one [8], while (*Z*)-4-(arylamino)-1-(hydroxyimino)naphthalen-2(1*H*)-ones **1a** and **1b** were obtained by oximation of 4-(arylamino)-1,2-naphthoquinone [1]. In the latter case, prolonged heating of the reactants in 2-ethoxyethanol was necessary. The oximation of naphtho[2,3-*b*]furan-4,9-dione also required harsh conditions [4]. There are no published data on reactions of 1,4-naphthoquinone derivatives having two nitrogen-containing substituents in the quinoid fragment.

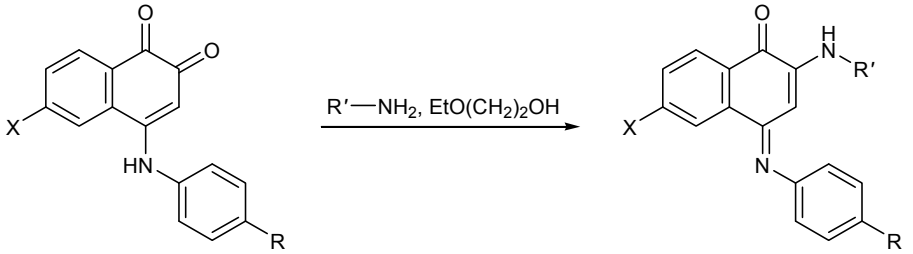
We have synthesized a series of 2-(R^1-amino)-4-(R^2-imino)naphthalen-1(4*H*)-ones **6a**–**6e** and **6h** by



1a, **1b** **2** **3a**, **3b** **4a**, **4b**

1, R = H (**a**), OMe (**b**); **3**, X = H (**a**), COAc (**b**); **4**, R = H (**a**), Me (**b**).

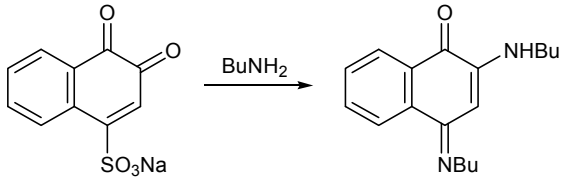
Scheme 1.



5a–5c **6a–6e**, **6h**

5, X = H, R = H (**a**), Me (**b**); X = Br, R = Me (**c**); **6**, X = H: R = H, R′ = $PhCH_2$ (**a**), Bu (**b**), Ph (**c**); R = Me, R′ = *i*-Bu (**d**), 4-MeC_6H_4 (**e**); X = Br, R = Me, R′ = Bu (**h**).

Scheme 2.



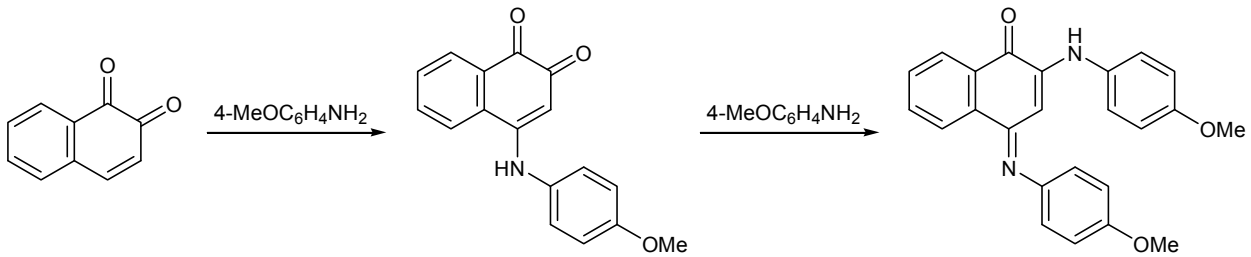
7 **6f**

reacting 4-(arylamino)-1,2-naphthoquinones **5a**–**5c** with primary aromatic or aliphatic amines (Scheme 1) and studied their reaction with hydroxylamine.

Compounds **5** failed to react with arylamines in boiling ethanol, and the corresponding 2-amino derivatives were obtained by heating the reactants in 2-ethoxyethanol (cf. [9]). Aliphatic amines reacted with **5a–5c** under milder conditions. Compound **6f** was synthesized by treatment of sodium 3,4-dioxonaphthalene-1-sulfonate (**7**) with butan-1-amine at 40–50°C (Scheme 2).

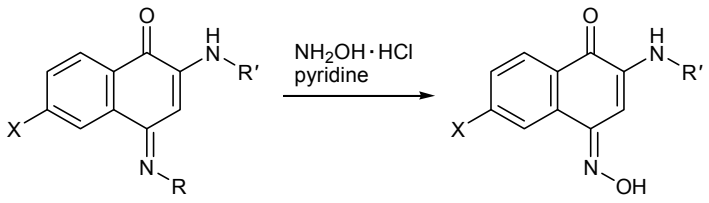
The reaction of 1,2-naphthoquinone with *p*-anisidine in boiling 2-ethoxyethanol afforded 2-(4-methoxyanilino)-4-[(4-methoxyphenyl)imino]naphthalen-1(4*H*)-one (**6g**) (Scheme 3). In contrast to published data [10], 1,2-naphthoquinone reacted with *p*-anisidine

Scheme 3.



5d **6g**

Scheme 4.



6a–6h **8a–8g**

6, X = H: R = Ph, R′ = $PhCH_2$ (**a**), Bu (**b**), Ph (**c**); R = 4-MeC_6H_4, R′ = *i*-Bu (**d**), 4-MeC_6H_4 (**e**); R = R′ = Bu (**f**), 4-$MeOC_6H_4$ (**g**); X = Br, R = 4-MeC_6H_4, R′ = Bu (**h**); **8**, X = H, R′ = $PhCH_2$ (**a**), Bu (**b**), Ph (**c**), *i*-Bu (**d**), 4-MeC_6H_4 (**e**), 4-$MeOC_6H_4$ (**f**); X = Br, R′ = Bu (**g**).

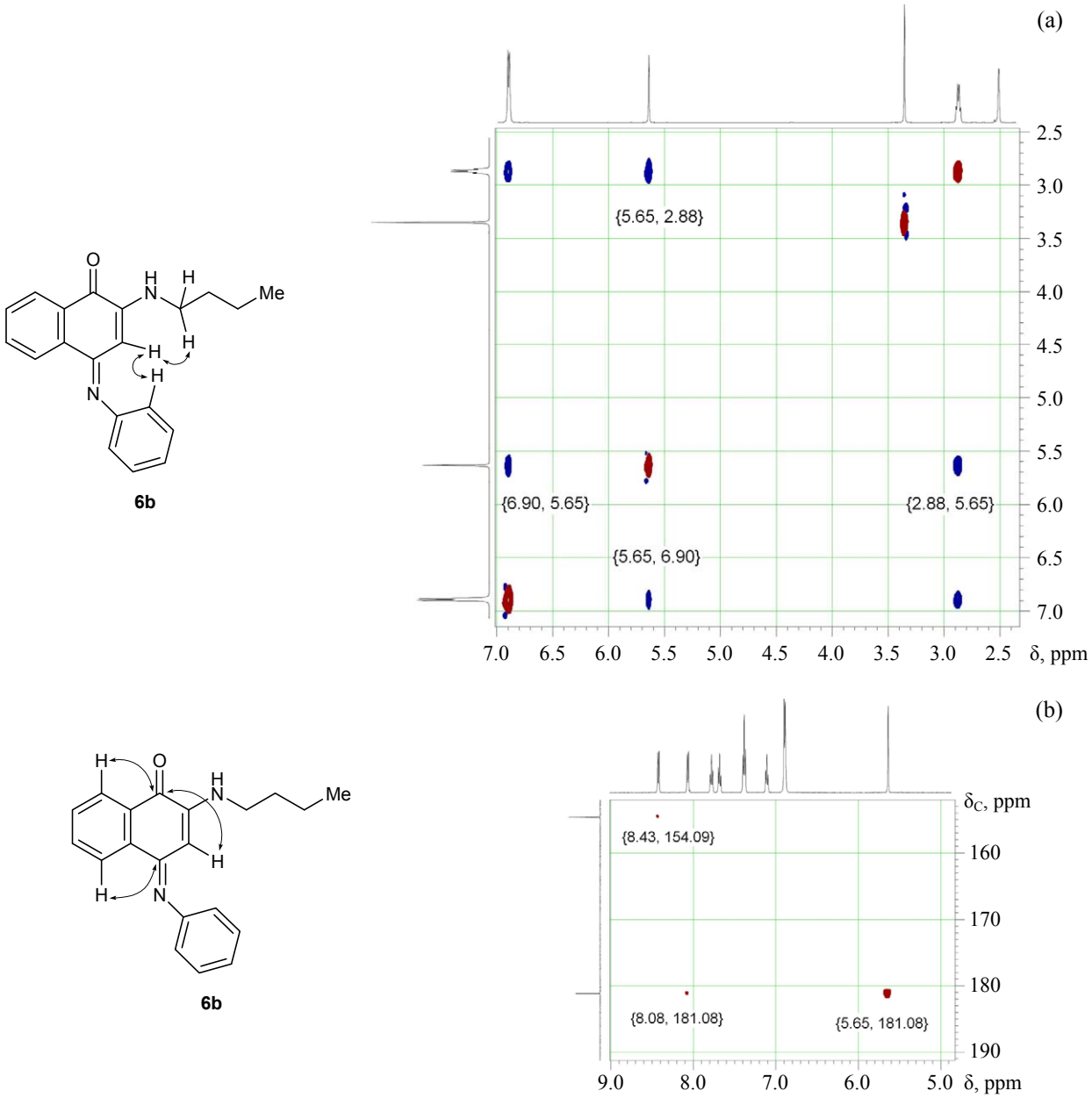


Fig. 1. Fragments of the (a) ^{1}H–^{1}H NOESY and (b) ^{1}H–^{13}C HMBC spectra of (4*E*)-2-(butylamino)-4-(phenylimino)naphthalen-1(4*H*)-one (**6b**).

in boiling methanol to give 4-(4-methoxyanilino)-1,2-naphthoquinone (**5d**), whereas no imine **6g** was obtained in methanol at room temperature.

The oximation of quinones **6a**–**6h** with hydroxylamine hydrochloride on heating for a short time in pyridine or in ethanol in the presence of pyridine was regioselective (Scheme 4), and only the corresponding 4-hydroxyimino derivatives **8a**–**8g** were formed, which is consistent with published data [11] indicating higher reactivity of the 4-position toward other nucleophiles, such as water.

The structure of 2-(R^{1}-amino)-4-(R^{2}-imino)naphthalen-1(4*H*)-ones **6a**–**6h** and 2-(R-amino)-4-(hydroxyimino)naphthalen-1(4*H*)-ones was confirmed by spectral methods, including two-dimensional NMR experiments (NOESY, HMBC, HSQC) which allowed us to reliably assign signals in the ^{1}H and ^{13}C NMR spectra.

In the ^{1}H NMR spectra of **6** and **8**, the 5-H proton resonated in a weaker field than 8-H. Obviously, anisotropic effect of the imino group on the *peri* position is stronger than the effect of the carbonyl group. Protons

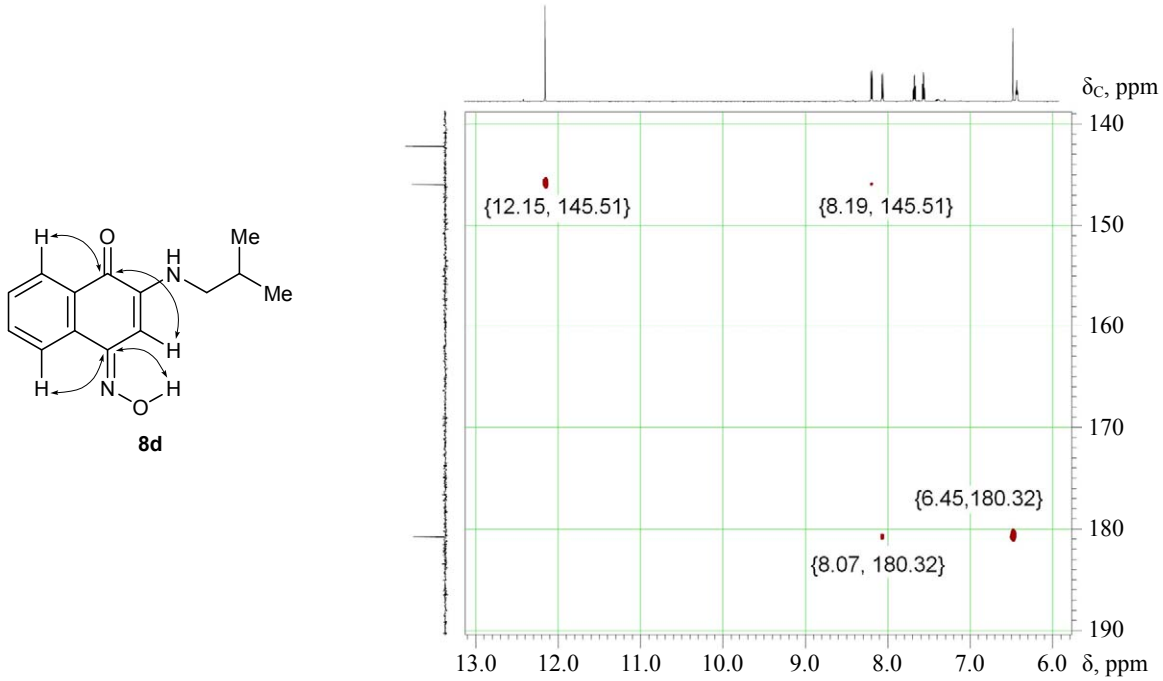


Fig. 2. A fragment of the ^{1}H–^{13}C HMBC spectrum of (4*E*)-4-(hydroxyimino)-2-(2-methylpropylamino)naphthalen-1(4*H*)-one (**8d**).

of the methylene group linked to the nitrogen atom of the 2-alkylamino substituent of **6** give a triplet signal in the ^{1}H NMR spectra, and the signal from protons of the neighboring methylene group has the corresponding multiplicity. These data indicate 1,4-quinone imine structure of compounds **6**, which is also confirmed by two-dimensional NMR data.

The NOESY spectrum of **6b** (Fig. 1a) showed cross-peaks due to coupling of 3-H (δ 5.65 ppm) with the 2-NHC**H**$_2$ protons (δ 2.88 ppm) and *ortho* protons of the phenylimino group (δ 6.90 ppm). In the HMBC spectrum of the same compound (Fig. 1b) we observed correlations between the carbonyl carbon atom (δ_C 181.08 ppm) and protons in positions *3* (δ 5.65 ppm) and *8* (δ 8.08 ppm) and between the imino carbon atom (C^4, δ_C 154.09 ppm) and proton in the *peri* position (5-H, δ 8.43 ppm).

Likewise, oxime **8d** displayed HMBC cross-peaks (Fig. 2) between the carbonyl carbon atom (C^1, δ_C 180.32 ppm) and protons in the 3- (δ 6.45 ppm) and 8-positions (δ 8.07 ppm), as well as cross peaks between the "oxime" carbon atom (C^4, δ_C 145.51 ppm) and *peri* proton (5-H, δ 8.19 ppm) and proton of the oxime hydroxy group (δ 12.15 ppm).

The structure of 4-(hydroxyimino)-2-(4-methylanilino)naphthalen-1(4*H*)-one (**8e**) was determined by X-ray analysis (Fig. 3). The unit cell of **8e** includes two independent molecules differing by the orientation of the tolyl group. The torsion angles $C^3C^2N^2C^{11}$ and $C^2N^2C^{11}C^{16}$ are −16.5(9), −28.2(8) and −10.0(9), 39.4(8)°, respectively. The bond lengths in molecule **8e** are similar to the corresponding bond lengths in 2-(4-methoxyanilino)-4-[(4-methoxyphenyl)imino]-naphthalen-1(4*H*)-one [10] and 5-(hydroxyimino)quinolin-8(5*H*)-one [11]. Molecules **8e** in crystal are linked to form bands along the *a* axis through the intermolec-

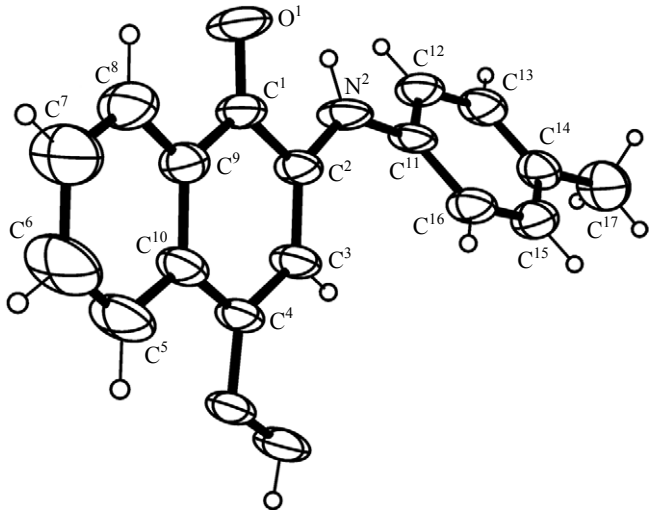


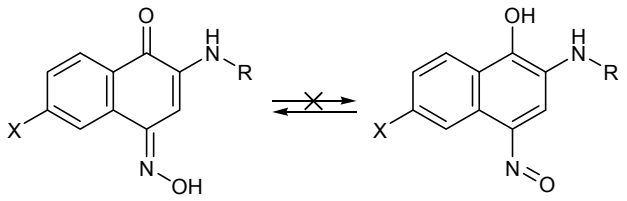
Fig. 3. Structure of the molecule of 4-(hydroxyimino)-2-(4-methylanilino)naphthalen-1(4*H*)-one (**8e**) according to the X-ray diffraction data.

ular hydrogen bonds NO–H···NOH (H···N 2.04, 2.10 Å, ∠OHN 148, 146°) and N–H···OC (H···O 2.25, 2.17 Å, ∠NHO 160, 160°).

The regioselectivity of the oximation of compounds **6a**–**6h** (at the 4-position) is consistent with the available data [12] indicating higher reactivity of that position toward other nucleophiles, e.g., water.

According to the X-ray diffraction and UV spectral data, compounds **8a**–**8g** both in crystal and in solution exist as oxime rather than nitroso tautomers (Scheme 5). The UV spectra of **8a**–**8g** lacked long-wave absorption band assignable to n–π^* transition in the nitroso group [13].

Scheme 5.



8a–8g

We plan to perform further functionalization of 2-(R-amino)-4-(hydroxyimino)naphthalen-1(4*H*)-ones synthesized in this work with the goal of obtaining biologically active compounds.

EXPERIMENTAL

The IR spectra were recorded in KBr on a Shimadzu IR Affinity-1 spectrometer. The ^{1}H and ^{13}C NMR spectra were measured on a Bruker DRX spectrometer at 500 and 125 MHz, respectively, using DMSO-d_6 as solvent and tetramethylsilane as internal standard. The UV spectra were recorded on an Evolution 300 spectrophotometer from solutions in ethanol with a concentration of 10^{-4} M using 10-mm path length cells. The mass spectra (electron impact, 70 eV) were obtained with a Finnigan MAT 8200 instrument. The elemental compositions were determined on a Euro EA 3000 analyzer. The melting points were measured on a Boetius micro hot stage. The progress of reactions and the purity of products were monitored by TLC on Silufol UV-254 plates (eluent toluene–acetone, 10:1).

The X-ray diffraction data for compound **8e** were obtained with a Bruker Kappa Apex II diffractometer (Mo K_α radiation, graphite monochromator, 296 K). All calculations were performed using SHELX97. The positions of hydrogen atoms were refined according to the riding model. The crystallographic data were deposited to the Cambridge Crystallographic Data Centre (CCDC entry no. 1558392) and are available at *www.ccdc.cam.ac.uk/data_request/cif* upon requiest.

6-Bromo-4-(4-methylanilino)naphthalene-1,2-dione (5c). A solution of 3.55 g (15 mmol) of 6-bromo-1,2-naphthoquinone in 30 mL of ethanol was mixed with a solution of 1.6 g (15 mmol) of *p*-toluidine in 15 mL of ethanol. The mixture was stirred for 20 min at 18–20°C, and the precipitate was filtered off and washed with ethanol. Yield 2.75 g (54%), red crystals, mp 289–290°C (from DMF). UV spectrum, λ_{max}, nm (logε): 208 (4.38), 257 (4.23), 296 (4.06), 479 (3.49). Found, %: C 60.08; H 3.37; Br 23.32; N 3.92. $C_{17}H_{12}BrNO_2$. Calculated, %: C 59.67; H 3.53; Br 23.35; N 4.09.

(4*E*)-4-Anilino-2-(benzylamino)naphthalen-1(4*H*)-one (6a). A solution of 1.65 g (15 mmol) of benzylamine in 10 mL of ethanol was added with stirring to a solution of 3 g (12 mmol) of 4-anilino-1,2-naphthoquinone in 30 mL of ethanol and 20 mL of dioxane. The mixture was refluxed for 2 h and cooled to 60°C, 50 mL of water was added, and the mixture was heated to the boiling point and cooled. The precipitate was filtered off and washed with water, aqueous ethanol, and ethanol. Yield 2.6 g (77%), red crystals, mp 155–157°C (from EtOH–DMF, 1:1). UV spectrum, λ_{max}, nm (logε): 206 (4.44), 239 (4.41), 332 (3.70), 452 (3.77). IR spectrum, ν, cm^{-1}: 3371 (N–H), 1657 (C=N), 1609 (C=O), 1347 (N–C). ^{1}H NMR spectrum, δ, ppm: 4.17 d (2H, CH_2, J = 5.4 Hz), 5.63 s (1H, 3-H), 6.63 d (2H, *o*′-H, J = 7.7 Hz), 7.06 d (2H, *o*-H, J = 6.7 Hz), 7.12 t (3H, *m*′-H, *p*′-H, J = 7.1 Hz), 7.28 t (3H, *m*-H, *p*-H, J = 7.1 Hz), 7.63 t (1H, NH, J = 5.4 Hz), 7.70 t (1H, 6-H, J = 7.2 Hz), 7.78 t (1H, 7-H, J = 7.2 Hz), 8.09 d (1H, 8-H, J = 7.8 Hz), 8.37 d (1H, 5-H, J = 7.8 Hz). ^{13}C NMR spectrum, δ_C, ppm: 45.41 (CH_2), 94.65 (C^3), 120.54 ($C^{o'}$), 123.41 ($C^{p'}$), 124.85 (C^p), 125.73 (C^5), 126.99 (C^8), 127.18 (C^o), 128.44 (C^m), 128.80 ($C^{m'}$), 130.14 (C^{4a}), 130.63 (C^6), 133.44 (C^7), 135.24 (C^{8a}), 137.84 (C^i), 143.74 (C^2), 151.09 ($C^{i'}$), 154.26 (C^4), 181.13 (C^1). Mass spectrum, m/z (I_{rel}, %): 338 (100) $[M]^+$, 246 (54), 91 (97.4). Found, %: C 81.76; H 5.14; N 8.33. $C_{23}H_{18}N_2O$. Calculated, %: C 81.63; H 5.36; N 8.28. M 338.41.

(4*E*)-2-(Butylamino)-4-(phenylimino)naphthalen-1(4*H*)-one (6b). Butylamine, 0.5 mL (5 mmol), was added with stirring to a solution of 1.25 g (5 mmol) of 4-anilino-1,2-naphthoquinone in a mixture

of 10 mL of ethanol and 10 mL of dioxane. The mixture was refluxed for 2 h and cooled to 60°C, 20 mL of water was added, and the mixture was heated to the boiling point and cooled. The precipitate was filtered off and washed with water, aqueous ethanol, and ethanol. Yield 1.28 g (84%), orange crystals, mp 122–123°C (from EtOH–DMF, 1:1). UV spectrum, λ_{max}, nm (log ε): 205 (4.41), 240 (4.42), 334 (3.71), 456 (3.79). IR spectrum, ν, cm^{-1}: 3332 (N–H), 1654 (C=N), 1605 (C=O), 1335 (N–C). ^{1}H NMR spectrum, δ, ppm: 0.78 t (3H, CH_3, *J* = 7.3 Hz), 1.15–1.23 m (2H, C**H**$_2$CH$_3$), 1.38–1.45 m (2H, CH_2), 2.88 q (2H, C**H**$_2$NH, *J* = 6.0 Hz), 5.65 s (1H, 3-H), 6.90 d (2H, *o*-H, *J* = 7.6 Hz), 6.93 t (1H, NH, *J* = 6.0 Hz), 7.12 t (1H, *p*-H, *J* = 7.6 Hz), 7.40 t (2H, *m*-H, *J* = 7.6 Hz), 7.71 t (1H, 6-H, *J* = 7.4 Hz), 7.81 t (1H, 7-H, *J* = 7.7 Hz), 8.08 d (1H, 8-H, *J* = 7.7 Hz), 8.43 d (1H, 5-H, *J* = 7.7 Hz). ^{13}C NMR spectrum, δ_C, ppm: 13.53 (CH_3), 19.64 (**C**H$_2$CH$_3$), 29.28 (**C**H$_2$CH$_2$CH$_3$), 41.21 (NHCH$_2$), 92.63 (C^3), 120.45 (C^o), 123.36 (C^p), 124.88 (C^5), 125.74 (C^8), 128.25 (C^m), 130.09 (C^{4a}), 130.58 (C^6), 133.43 (C^7), 135.40 (C^{8a}), 144.00 (C^2), 151.59 (C^i), 154.96 (C^4), 181.01 (C^1). Mass spectrum, *m*/*z* (I_{rel}, %): 304 (100) $[M]^+$, 261 (22.1), 232 (24.1), 204 (24.4), 77 (31.7). Found, %: C 79.13; H 6.51; N 9.25. $C_{20}H_{20}N_2O$. Calculated, %: C 78.92; H 6.62; N 9.20. *M* 304.40.

2-Anilino-4-(phenylimino)naphthalen-1(4*H*)-one (6c). Aniline, 5 mL (55 mmol), was added to a solution of 7.5 g (30 mmol) of 4-anilino-1,2-naphthoquinone in 80 mL of 2-ethoxyethanol. The mixture was refluxed for 3 h and cooled to room temperature, and the precipitate was filtered off and washed with water, aqueous ethanol, and ethanol. Yield 7.25 g (75%), red crystals, mp 184–186°C (from EtOH–DMF, 1:1). UV spectrum, λ_{max}, nm (log ε): 208 (4.45), 254 (4.27), 283 (4.32), 472 (3.77). IR spectrum, ν, cm^{-1}: 3346 (N–H), 1648 (C=N), 1599 (C=O), 1347 (N–C). ^{1}H NMR spectrum, δ, ppm: 6.50 s (1H, 3-H), 6.93 d (2H, *o*-H, *J* = 7.5 Hz), 7.03 t (1H, *p*-H, *J* = 7.5 Hz), 7.11 t (2H, *m*-H, *J* = 7.5 Hz), 7.23 d (2H, *o*′-H, *J* = 7.8 Hz), 7.27 t (2H, *m*′-H, *J* = 7.8 Hz), 7.38 t (1H, *p*′-H, *J* = 7.8 Hz), 7.76 t (1H, 6-H, *J* = 7.8 Hz), 7.84 t (1H, 7-H, *J* = 7.8 Hz), 8.16 d.d (1H, 5-H, *J* = 7.8, 1.1 Hz), 8.45 d.d (1H, 8-H, *J* = 7.8, 1.1 Hz), 8.74 s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 324 (100) $[M]^+$, 323 (35.8), 247 (22.0), 77 (34.0). Found, %: C 81.39; H 4.86; N 8.47. $C_{22}H_{16}N_2O$. Calculated, %: C 81.46; H 4.97; N 8.64. *M* 324.39.

(4*E*)-4-[(4-Methylphenyl)imino]-2-(2-methylpropyl)naphthalen-1(4*H*)-one (6d). Isobutylamine, 1.6 g (22 mmol), was added with stirring to a solution of 2.63 g (10 mmol) of 4-(4-methylanilino)-1,2-naphthoquinone in 50 mL of ethanol. The mixture was refluxed for 2 h and cooled to 60°C, 40 mL of water was added, and the mixture was heated to the boiling point and cooled. The precipitate was filtered off and washed with water and ethanol. Yield 2.43 g (76%), red crystals, mp 144–145°C (from EtOH–DMF, 1:1). UV spectrum, λ_{max}, nm (log ε): 207 (4.45), 242 (4.44), 335 (3.71), 468 (3.81). IR spectrum, ν, cm^{-1}: 3351 (N–H), 1654 (C=N), 1596 (C=O), 1338 (N–C). ^{1}H NMR spectrum, δ, ppm: 0.81 d (6H, CH_3, *J* = 6.7 Hz), 1.78–1.85 m (1H, CH), 2.33 s (3H, CH_3), 2.73 t (2H, CH_2, *J* = 6.2 Hz), 5.73 s (1H, 3-H), 6.81 d (2H, *m*-H, *J* = 8.1 Hz), 6.89 t (1H, NH, *J* = 6.2 Hz), 7.21 d (2H, *o*-H, *J* = 8.1 Hz), 7.70 t (1H, 6-H, *J* = 7.6 Hz), 7.79 t (1H, 7-H, *J* = 7.6 Hz), 8.08 d (1H, 8-H, *J* = 7.6 Hz), 8.43 d (1H, 5-H, *J* = 7.6 Hz). ^{13}C NMR spectrum, δ_C, ppm: 20.29 [CH(**C**H$_3$)$_2$], 20.58 (CH_3), 26.77 [**C**H(CH$_3$)$_2$], 49.31 (NCH$_2$), 92.95 (C^3), 120.65 (C^o), 124.86 (C^5), 125.73 (C^8), 129.36 (C^m), 130.04 (C^{4a}), 130.54 (C^6), 132.47 (C^{8a}), 135.42 (C^7), 135.48 (C^p), 144.13 (C^2), 148.81 (C^i), 181.08 (C^1). Mass spectrum, *m*/*z* (I_{rel}, %): 318 (100) $[M]^+$, 275 (40.8), 246 (19.5). Found, %: C 79.38; H 6.90; N 8.88. $C_{21}H_{22}N_2O$. Calculated, %: C 79.21; H 6.96; N 8.80. *M* 318.42.

2-(4-Methylanilino)-4-[(4-methylphenyl)imino]-naphthalen-1(4*H*)-one (6e). *p*-Toluidine, 5.35 g (50 mmol), was added to 7.89 g (30 mmol) of 4-(4-methylamino)-1,2-naphthoquinone in 80 mL of 2-ethoxyethanol. The mixture was refluxed for 3 h and cooled to room temperature, and the precipitate was filtered off and washed with water, aqueous ethanol, and ethanol. Yield 9.5 g (90%), red–brown crystals, mp 175–177°C (from EtOH–DMF, 1:1). UV spectrum, λ_{max}, nm (log ε): 209 (4.44), 254 (4.22), 281 (4.26), 480 (3.73). IR spectrum, ν, cm^{-1}: 3332 (N–H), 1640 (C=N), 1594 (C=O), 1337 (N–C). ^{1}H NMR spectrum, δ, ppm: 2.24 s (3H, CH_3), 2.30 s (3H, CH_3), 6.40 s (1H, 3-H), 6.84 d (2H, *m*-H, *J* = 8.0 Hz), 6.93 d (2H, *o*-H, *J* = 8.0 Hz), 7.08 d (2H, *m*′-H, *J* = 8.5 Hz), 7.12 d (2H, *o*′-H, *J* = 8.5 Hz), 7.75 t (1H, 6-H, *J* = 7.6 Hz), 7.84 t (1H, 7-H, *J* = 7.6 Hz), 8.15 d.d (1H, 5-H, *J* = 7.9, 1.2 Hz), 8.44 d.d (1H, 8-H, *J* = 7.9, 1.2 Hz), 8.66 s (1H, NH). Mass spectrum, *m*/*z* (I_{rel}, %): 352 (100) $[M]^+$, 351 (22.8), 261 (12.41). Found, %: C 81.42; H 5.58; N 7.93. $C_{24}H_{20}N_2O$. Calculated, %: C 81.79; H 5.72; N 7.95. *M* 352.44.

(4*E*)-2-(Butylamino)-4-(butylimino)naphthalen-1(4*H*)-one (6f). Sodium 1,2-dioxo-1,2-dihydronaph-

thalene-4-sulfonate (**7**), 10.5 g (40 mmol), was dissolved in 300 mL of water, 4.5 g of potassium carbonate, 20 mL of butylamine, and 20 mL of water were added with stirring, and the mixture was stirred for 1 h at 40–50°C. The precipitate was filtered off and washed with water, aqueous ethanol, and ethanol. Yield 4.3 g (38%), yellow crystals, mp 92–94°C (from EtOH–DMF, 1:1). UV spectrum, λ_{max}, nm (logε): 205 (4.35), 243 (4.33), 277 (4.12), 439 (3.65). IR spectrum, ν, cm^{-1}: 3338 (N–H), 1654 (C=N), 1595 (C=O), 1331 (N–C). ^{1}H NMR spectrum, δ, ppm: 0.92 t (3H, CH_3, J = 7.4 Hz), 0.96 t (3H, CH_3, J = 7.4 Hz), 1.32–1.41 m (2H, C**H**$_2$CH$_3$), 1.43–1.52 m (2H, C**H**$_2$CH$_3$), 1.54–1.64 m (2H, C**H**$_2$CH$_2$CH$_3$), 1.70–1.78 m (2H, C**H**$_2$CH$_2$CH$_3$), 3.15 q (2H, C**H**$_2$NH, J = 6.7 Hz), 3.71 t (2H, CH_2N=, J = 6.9 Hz), 6.03 s (1H, 3-H), 6.55 t (1H, NH, J = 6.7 Hz), 7.60 t (1H, 6-H, J = 7.9 Hz), 7.70 t (1H, 7-H, J = 7.9 Hz), 8.01 d.d (1H, 8-H, J = 7.9, 1.1 Hz), 8.34 d (1H, 5-H, J = 7.7 Hz). ^{13}C NMR spectrum, δ_C, ppm: 11.53 (=NCH$_2$CH$_2$CH$_2$**C**H$_3$), 11.38 (NHCH$_2$CH$_2$CH$_2$**C**H$_3$), 13.74 (NHCH$_2$CH$_2$**C**H$_2$CH$_3$), 13.98 (=NCH$_2$CH$_2$**C**H$_2$CH$_3$), 19.86 (NHCH$_2$**C**H$_2$), 20.41 (=NCH$_2$**C**H$_2$), 41.41 (NHCH$_2$), 50.04 (=NCH$_2$), 90.94 (C^3), 124.29 (C^5), 125.27 (C^8), 129.54 (C^6), 133.06 (C^7), 134.13 (C^{8a}), 136.35 (C^{4a}), 142.90 (C^2), 154.00 (C^4), 181.35 (C^1). Mass spectrum, m/z (I_{rel}, %): 284 (68.8) $[M]^+$, 255 (45.2), 241 (100). Found, %: C 76.47; H 8.09; N 9.94. $C_{18}H_{24}N_2O$. Calculated, %: C 76.02; H 8.51; N 9.85. M 284.40.

(4*E*)-2-(4-Methoxyanilino)-4-[(4-methoxyphenyl)imino]naphthalen-1-(4*H*)-one (6g). *p*-Anisidine, 1.4 g (11 mmol), was added to a solution of 0.8 g (5 mmol) of 1,2-naphthoquinone in 10 mL of 2-ethoxyethanol. The mixture was refluxed for 1.5 h and cooled to room temperature, and the precipitate was filtered off and washed with water, aqueous ethanol, and ethanol. Yield 1.23 g (64%), red–brown crystals, mp 234–235°C (from EtOH–DMF, 1:1). UV spectrum, λ_{max}, nm (logε): 205 (4.29), 246 (4.09), 274 (4.09), 496 (3.62). IR spectrum, ν, cm^{-1}: 3325 (N–H), 1656 (C=N), 1604 (C=O), 1339 (N–C), 1238 (C–O–C). ^{1}H NMR spectrum, δ, ppm: 3.72 s (3H, CH_3), 3.76 (3H, CH_3), 6.85–7.30 m (8H, H_{arom}), 6.40 s (1H, 3-H), 7.73 br.t (1H, 6-H, J = 7.3 Hz), 7.82 br.t (1H, 7-H, J = 7.3 Hz), 8.14 d (1H, 8-H, J = 7.3 Hz), 8.44 d (1H, 5-H, J = 7.7 Hz), 8.55 s (1H, NH). Mass spectrum, m/z (I_{rel}, %): 384 (100) $[M]^+$, 369 (65). Found, %: C 75.05; H 5.21; N 7.28. $C_{24}H_{20}N_2O_3$. Calculated, %: C 75.00; H 5.21; N 7.29. M 384.

6-Bromo-2-(butylamino)-4-[(4-methylphenyl)imino]naphthalen-1(4*H*)-one (6h). A solution of 0.9 mL (9 mmol) of butylamine in 10 mL of ethanol was added to a solution of 1.54 g (4.5 mmol) of 6-bromo-4-(4-methylanilino)-1,2-naphthoquinone in 35 mL of ethanol. The mixture was refluxed for 2 h and cooled, and the precipitate was filtered off and washed with ethanol. Yield 1.49 g (83%), dark red crystals, mp 187–189°C (from EtOH–DMF, 1:1). UV spectrum, λ_{max}, nm (logε): 206 (4.17), 226 (4.22), 252 (4.32), 294 (4.04), 477 (3.72). IR spectrum, ν, cm^{-1}: 3283 (N–H), 1656 (C=N), 1589 (C=O), 1325 (N–C). ^{1}H NMR spectrum, δ, ppm: 0.80 t (3H, C**H**$_3$CH$_2$, J = 7.4 Hz), 1.17–1.26 m (2H, C**H**$_2$CH$_3$), 1.43 quint (2H, CH_2, J = 7.4 Hz), 2.32 s (3H, CH_3), 2.91 q (2H, C**H**$_2$NH, J = 6.7 Hz), 5.73 s (1H, 3-H), 6.83 d (2H, *m*-H, J = 8.1 Hz), 7.22 d (2H, *o*-H, J = 8.1 Hz), 7.88 d.d (1H, 7-H, J = 8.4, 2.0 Hz), 7.95 t (1H, NH, J = 6.7 Hz), 7.98 d (1H, 8-H, J = 8.4 Hz), 8.51 d (1H, 5-H, J = 2.0 Hz). Mass spectrum, m/z (I_{rel}, %): 398 (100) $[M]^+$, 396 (99.7), 353 (24.7), 247 (40.4). Found, %: C 63.86; H 5.12; Br 19.90; N 7.08. $C_{21}H_{21}BrN_2O$. Calculated, %: C 63.48; H 5.33; Br 20.11; N 7.05. M 397.32.

(4*E*)-2-(Benzylamino)-4-(hydroxyimino)naphthalen-1(4*H*)-one (8a). Hydroxylamine hydrochloride, 0.695 g (10 mmol), was added to a solution of 3.38 g (10 mmol) of (4*E*)-2-(benzylamino)-4-(phenylimino)-naphthalen-1(4*H*)-one (**6a**) in a mixture of 30 mL of ethanol and 10 mL of pyridine. The mixture was refluxed for 20 min and poured into 400 mL of an ice–water mixture, and the precipitate was filtered off and washed with ethanol. Yield 2.667 g (96%), orange crystals, mp 232–233°C (from EtOH). UV spectrum, λ_{max}, nm (logε): 205 (4.35), 244 (4.50), 279 (3.98), 367 (3.68), 418 (3.75). IR spectrum, ν, cm^{-1}: 3370 (N–H), 3237 (O–H), 1647 (C=N), 1601 (C=O), 1346 (N–C), 961 (N–O). ^{1}H NMR spectrum, δ, ppm: 4.41 d (2H, CH_2, J = 6.3 Hz), 6.41 s (1H, 3-H), 7.17 t (1H, NH, J = 6.3 Hz), 7.23 t (1H, *p*-H, J = 4.3 Hz), 7.32 t (2H, *m*-H, J = 4.3 Hz), 7.33 d (2H, *o*-H, J = 4.3 Hz), 7.57 t (1H, 6-H, J = 7.3 Hz), 7.67 t (1H, 7-H, J = 7.3 Hz), 8.08 d (1H, 8-H, J = 7.8 Hz), 8.16 d (1H, 5-H, J = 7.8 Hz), 12.08 s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 45.32 (CH_2), 90.61 (C^3), 122.35 (C^5), 125.79 (C^8), 126.92 (C^{4a}), 128.49 (C^m), 128.74 (C^p), 128.84 (C^6), 132.72 (C^7), 134.10 (C^{8a}), 138.39 (C^o), 141.47 (C^i), 145.41 (C^2), 149.66 (C^4), 180.40 (C^1). Mass spectrum, m/z (I_{rel}, %): 278 (55.1) $[M]^+$, 91 (100), 79 (22.0). Found, %: C 73.65; H 4.90; N 10.11. $C_{17}H_{14}N_2O_2$. Calculated, %: C 73.37; H 5.07; N 10.07. M 278.31.

(4*E*)-2-(Butylamino)-4-(hydroxyimino)naphthalen-1(4*H*)-one (8b). Hydroxylamine hydrochloride,

0.8 g (11.5 mmol), was added to a solution of 1.52 g (5 mmol) of (4*E*)-2-(butylamino)-4-(phenylimino)-naphthalen-1(4*H*)-one **6b** in a mixture of 30 mL of ethanol and 10 mL of pyridine. The mixture was refluxed for 20 min and cooled to 60–70°C, 100 mL of water was added, the mixture was heated again and cooled, and the precipitate was filtered off and washed with ethanol. Yield 1 g (82%), yellow crystals, mp 177–178°C (from EtOH). UV spectrum, λ_{max}, nm (logε): 204 (4.03), 244 (4.50), 281 (3.98), 366 (3.68), 428 (3.77). IR spectrum, ν, cm^{-1}: 3356 (N–H), 3217 (O–H), 1651 (C=N), 1601 (C=O), 1352 (N–C), 951 (N–O). ^{1}H NMR spectrum, δ, ppm: 0.92 t (3H, CH_3, J = 7.4 Hz), 1.32–1.40 m (2H, C**H**$_2$CH$_3$), 1.60 quint (2H, CH_2, J = 7.4 Hz), 3.13 q (2H, C**H**$_2$NH, J = 6.6 Hz), 6.43 t (1H, NH, J = 5.7 Hz), 6.48 s (1H, 3-H), 7.57 t (1H, 6-H, J = 7.6 Hz), 7.68 t (1H, 7-H, J = 7.6 Hz), 8.07 d (1H, 8-H, J = 7.6 Hz), 8.20 d (1H, 5-H, J = 7.6 Hz), 12.15 s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 13.73 (CH_3), 19.86 (**C**H$_2$CH$_3$), 29.62 (**C**H$_2$CH$_2$CH$_3$), 41.60 (NHCH$_2$), 89.39 (C^3), 122.37 (C^5), 125.77 (C^8), 128.75 (C^6), 132.65 (C^7), 134.23 (C^{8a}), 141.63 (C^2), 145.54 (C^4), 180.34 (C^1). Mass spectrum, m/z (I_{rel}, %): 244 (69) $[M]^+$, 201 (100), 130 (23), 102 (23), 41 (27). Found, %: C 69.13; H 6.39; N 11.43. $C_{14}H_{16}N_2O_2$. Calculated, %: C 68.83; H 6.60; N 11.47. M 244.30.

(4*E*)-4-(Hydroxyimino)-2-anilinonaphthalen-1(4*H*)-one (8c). Hydroxylamine hydrochloride, 0.8 g (11.5 mmol), was added to a solution of 1.62 g (5 mmol) of 2-anilino-4-(phenylimino)naphthalen-1(4*H*)-one (**6c**) in a mixture of 50 mL of ethanol and 10 mL of pyridine. The mixture was refluxed for 15 min and cooled to 60–70°C, 100 mL of water was added, the mixture was heated again and cooled, and the precipitate was filtered off and washed with ethanol. Yield 1.1 g (83%), red crystals, mp 211–213°C (from EtOH). UV spectrum, λ_{max}, nm (logε): 208 (4.40), 264 (4.32), 292 (4.28), 368 (3.74), 437 (3.68). IR spectrum, ν, cm^{-1}: 3335 (N–H), 3219 (O–H), 1650 (C=N), 1602 (C=O), 1304 (N–C), 962 (N–O). ^{1}H NMR spectrum, δ, ppm: 7.11 t (1H, *p*-H, J = 7.0 Hz), 7.30 s (1H, 3-H), 7.38 t (2H, *o*-H, J = 8.2 Hz), 7.41 t (2H, *m*-H, J = 8.2 Hz), 7.63 t (1H, 6-H, J = 7.4 Hz), 7.73 t (1H, 7-H, J = 8.2 Hz), 8.14 d (1H, 8-H, J = 8.2 Hz), 8.21 d (1H, 5-H, J = 7.4 Hz) 12.42 s (1H, OH). Mass spectrum, m/z (I_{rel}, %): 264 (59) $[M]^+$, 130 (38), 102 (36), 77 (100), 51 (76). Found, %: C 72.50; H 4.42; N 10.52. $C_{16}H_{12}N_2O_2$. Calculated, %: C 72.72; H 4.58; N 10.60. M 264.29.

(4*E*)-4-(Hydroxyimino)-2-(2-methylpropyl-amino)naphthalen-1(4*H*)-one (8d). Hydroxylamine hydrochloride, 0.8 g (11.5 mmol), was added to a solution of 1.6 g (5 mmol) of (4*E*)-2-(2-methylpropyl-amino)-4-[(4-methylphenyl)imino]naphthalen-1(4*H*)-one (**6d**) in a mixture of 30 mL of ethanol and 10 mL of pyridine. The mixture was refluxed for 20 min and cooled to 60–70°C, 140 mL of water was added, the mixture was heated agained and cooled, and the precipitate was filtered off and washed with ethanol. Yield 0.998 g (81%), orange crystals, mp 182–184°C (from EtOH). UV spectrum, λ_{max}, nm (logε): 206 (4.36), 246 (4.51), 283 (4.01), 368 (3.70), 429 (3.78). IR spectrum, ν, cm^{-1}: 3364 (N–H), 3217 (O–H), 1653 (C=N), 1601 (C=O), 1263 (N–C), 953 (N–O). ^{1}H NMR spectrum, δ, ppm: 0.93 s (6H, CH_3), 1.96–2.03 m (1H, CH), 2.96 t (2H, CH_2, J = 6.5 Hz), 6.45 t (1H, NH, J = 6.5 Hz), 6.48 s (1H, 3-H), 7.57 t (1H, 6-H, J = 7.9 Hz), 7.68 t (1H, 7-H, J = 7.9 Hz), 8.07 d.d (1H, 8-H, J = 7.9, 1.2 Hz), 8.19 d.d (1H, 5-H, J = 7.9, 1.2 Hz), 12.15 s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 20.37 (CH_3), 26.60 (CH), 49.56 (CH_2), 89.45 (C^3), 122.09 (C^5), 125.80 (C^8), 128.71 (C^{4a}), 129.31 (C^6), 132.70 (C^7), 134.19 (C^{8a}), 141.75 (C^2), 145.51 (C^4), 180.32 (C^1). Mass spectrum, m/z (I_{rel}, %): 244 (55) $[M]^+$, 201 (100), 41 (21). Found, %: C 68.99; H 6.38; N 11.48. $C_{14}H_{16}N_2O_2$. Calculated, %: C 68.83; H 6.60; N 11.47. M 244.30.

(4*E*)-4-(Hydroxyimino)-2-[(4-methylphenyl)-amino]naphthalen-1(4*H*)-one (8e). Hydroxylamine hydrochloride, 5 g (70 mmol), was added to a solution of 6.7 g (20 mmol) of 2-(4-methylanilino)-4-[(4-methylphenyl)imino]naphthalen-1(4*H*)-one (**6e**) in a mixture of 120 mL of isopropyl alcohol and 30 mL of pyridine. The mixture was refluxed for 10 min and cooled to 60°C, 20 mL of water was added, the mixture was heated again and cooled, and the precipitate was filtered off and washed with ethanol. Yield 4.39 g (86%), orange crystals, mp 244–245°C (from EtOH). UV spectrum, λ_{max}, nm (logε): 208 (4.11), 263 (4.35), 291 (4.33), 367 (3.77), 443 (3.69). IR spectrum, ν, cm^{-1}: 3312 (N–H), 3222 (O–H), 1653 (C=N), 1597 (C=O), 1310 (N–C), 956 (N–O). ^{1}H NMR spectrum, δ, ppm: 2.5 s (3H, CH_3), 7.19 s (1H, 3-H), 7.21 d (2H, *o*-H, J = 8.3 Hz), 7.25 d (2H, *m*-H, J = 8.3 Hz), 7.61 t (1H, 6-H, J = 7.6 Hz), 7.72 t (1H, 7-H, J = 7.6 Hz), 8.13 d (1H, 8-H, J = 7.8 Hz), 8.19 d (1H, 5-H, J = 7.8 Hz), 8.34 s (1H, NH), 12.35 s (1H, OH). Mass spectrum, m/z (I_{rel}, %): 278 (100) $[M]^+$, 130 (69.7), 102 (33.2), 91 (32.3), 77 (26.6), 65 (33.1). Found, %:

C 73.36; H 4.84; N 10.04. $C_{17}H_{14}N_2O_2$. Calculated, %: C 73.38; H 5.04; N 10.07. *M* 278.31.

X-Ray diffraction data for compound 8e. Monoclinic crystal system, space group $P2_1/n$; $C_{17}H_{14}N_2O_2$, *M* 278.30; Unit cell parameters: a = 15.8398(11), b = 7.6654(6), c = 22.5750(17) Å; β = 94.094(3)°; V = 2734.0(4) Å^3; Z = 8; d_{calc} = 1.352 g/cm^3. Number of independent reflections 4854 (θ_{max} = 25°), including 2772 reflections with $I > 2\sigma(I)$. Final divergence factor R = 0.0794 for reflections with $I > 2\sigma(I)$; goodness of fit S = 0.948.

(4*E*)-4-(Hydroxyimino)-2-(4-methoxyanilino)-naphthalen-1(4*H*)-one (8f). Hydroxylamine hydrochloride, 0.52 g (7.5 mmol), was added to a solution of 0.96 g (2.5 mmol) of (4*E*)-2-(4-methoxyanilino)-4-[(4-methoxyphenyl)imino]naphthalen-1-(4*H*)-one (**6g**) in a mixture of 10 mL of ethanol and 5 mL of pyridine. The mixture was refluxed for 30 min and cooled to 60–70°C, 5 mL of water was added, the mixture was heated again and cooled, and the precipitate was filtered off and washed with ethanol. Yield 0.6 g (82%), orange crystals, mp 249–250°C (from *i*-PrOH–DMF, 1:1). UV spectrum, λ_{max}, nm (log ε): 205 (4.34), 254 (4.35), 287 (4.31), 364 (3.79). IR spectrum, ν, cm^{-1}: 3311 (N–H), 3222 (O–H), 1648 (C=N), 1600 (C=O), 1347 (N–C), 1250 (C–O–C), 959 (N–O). ^{1}H NMR spectrum, δ, ppm: 3.77 s (3H, CH_3), 7.00 d (2H, *m*-H, J = 8.8 Hz), 7.04 s (1H, 3-H), 7.28 d (2H, *o*-H, J = 8.8 Hz), 7.61 t (1H, 6-H, J = 7.4 Hz), 7.71 t (1H, 7-H, J = 7.2 Hz), 8.13 d (1H, 8-H, J = 7.8 Hz), 8.20 d (1H, 5-H, J = 7.9 Hz), 8.26 s (1H, NH), 12.98 s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 55.28 (CH_3), 91.57 (C^3), 114.57 (C^m), 122.30 (C^5), 124.71 (C^o), 125.95 (C^8), 128.81 (C^{4a}), 128.98 (C^6), 132.29 (C^i), 132.87 (C^7), 133.90 (C^{8a}), 139.85 (C^2), 145.50 (C^4), 156.01 (C^p), 180.38 (C^1). Mass spectrum, *m*/*z* (I_{rel}, %): 294 (100) $[M]^+$, 279 (31), 130 (29), 15 (36). Found, %: C 69.37; H 4.58; N 9.42. $C_{17}H_{14}N_2O_3$. Calculated, %: C 69.39; H 4.76; N 9.52. *M* 294.31.

6-Bromo-2-(butylamino)-4-(hydroxyimino)naphthalen-1(4*H*)-one (8g). Hydroxylamine hydrochloride, 0.5 g (7 mmol), was added to a solution of 0.99 g (2.5 mmol) of 6-bromo-4-(4-methylanilino)-1,2-naphthoquinone in a mixture of 10 mL of ethanol and 10 mL of pyridine. The mixture was heated for 1 h at 60°C, 10 mL of water was added, the mixture was cooled, and the precipitate was filtered off and washed with ethanol. Yield 0.7 g (87%), red crystals, mp 218–220°C (from EtOH). UV spectrum, λ_{max}, nm (log ε): 206 (4.35), 256 (4.46), 293 (4.03), 356 (3.62), 436 (3.72). IR spectrum, ν, cm^{-1}: 3345 (N–H), 3237 (O–H), 1653 (C=N), 1597 (C=O), 1339 (N–C), 961 (N–O). ^{1}H NMR spectrum, δ, ppm: 0.92 t (3H, CH_3, J = 7.4 Hz), 1.32–1.40 m (2H, C**H**$_2$CH$_3$), 1.59 quint (2H, CH_2, J = 7.4 Hz), 3.12 q (2H, NHC**H**$_2$), 6.44 s (1H, 3-H), 6.51 t (1H, NH, J = 5.7 Hz), 7.75 d.d (1H, 7-H, J = 8.5, 2.0 Hz), 7.98 d (1H, 8-H, J = 8.5 Hz), 8.28 s (1H, 5-H), 12.34 s (1H, OH). ^{13}C NMR spectrum, δ_C, ppm: 13.74 (CH_3), 19.86 (**C**H$_2$CH$_3$), 29.54 (**C**H$_2$CH$_2$CH$_3$), 41.61 (NCH_2), 89.32 (C^3), 124.81 (C^5), 126.84 (C^{4a}), 127.65 (C^6), 128.10 (C^8), 131.68 (C^7), 141.59 (C^2), 144.62 (C^4), 179.58 (C^1). Mass spectrum, *m*/*z* (I_{rel}, %): 322 (35), 281 (94), 279 (100), 41 (30). Found, %: C 52.35; H 4.50; Br 24.70; N 8.66. $C_{14}H_{15}BrN_2O_2$. Calculated, %: C 52.03; H 4.68; Br 24.72; N 8.67. *M* 323.19.

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