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On the Reaction of 2-Benzylamino-1,4-naphthoquinones with Nitrosylsulfuric Acid

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Abstract—The reaction of 2-benzylamino-1,4-naphthoquinone with nitrosylsulfuric acid in acetic acid results in preferential formation of 2,1- and 2,3-heterocyclization products: (*E*)-4-(hydroxyimino)-2-phenylnaphtho[2,1-*d*]oxazol-5(4*H*)-one and 2-phenyl-1-hydroxy-1*H*-naphtho[2,3-*d*]imidazole-4,9-dione. In addition, 2-phenylnaphtho[2,1-*d*]oxazol-4,5-dione and *N*-(3-nitro-1,4-dioxo-1,4-dihydronaphthalen-2-yl)benzamide are also formed. The reaction of 2-benzylamino-3-chloro-1,4-naphthoquinone with nitrosylsulfuric acid in acetic acid gives 3-diazonapthalene-1,2,4(3*H*)-trione and benzaldehyde.

Keywords: 2-benzylamino-1,4-naphthoquinone, hydroxyimidazole, naphtho-1,2-oxazole, oximes, 2,3-diazido-1,4-naphthoquinone, nitrosylsulfuric acid

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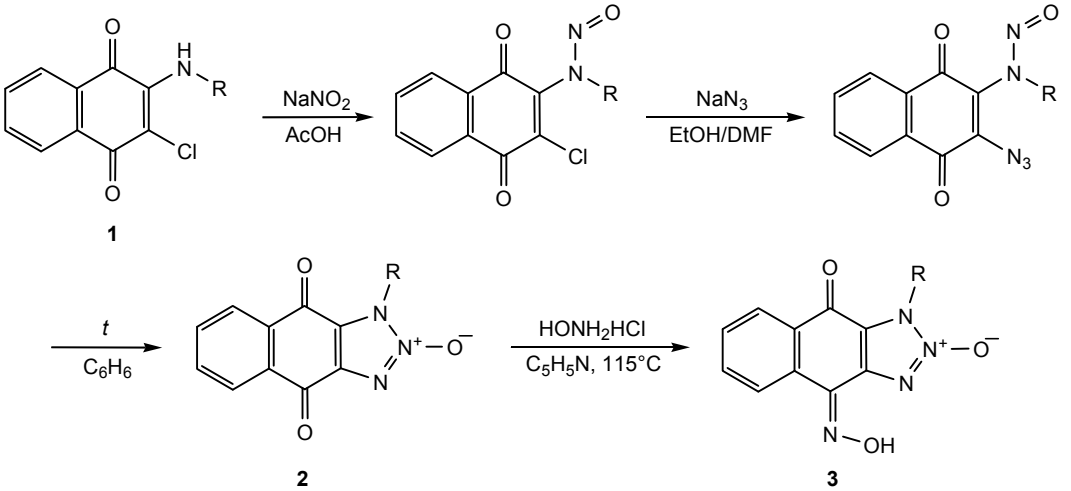
2-Amino-1,4-naphthoquinones exhibit diverse biological activity [1–3]. Of interest are also nitrogenous heterocycles derived from 2-amino-1,4-naphthoquinones. For example, the substitution of the chlorine in 2-acetylamino-3-chloro-1,4-naphthoquinone by an amino group followed by cyclization gave 1,2-substituted 1*H*-naphtho[2,3-*d*]imidazole-4,9-diones [4]. Such fused imidazoles showed antitumor effects [5, 6]. 2-Alkyl-amino-3-chloro-1,4-naphthoquinones **1** was used as the starting materials to prepare 1-alkyl-4,9-dioxo-1*H*-naphtho[2,3-*d*][1,2,3]-triazole 2-oxides **2** and their oximes **3**, which were found to be efficient cytostatics [7, 8] (Scheme 1).

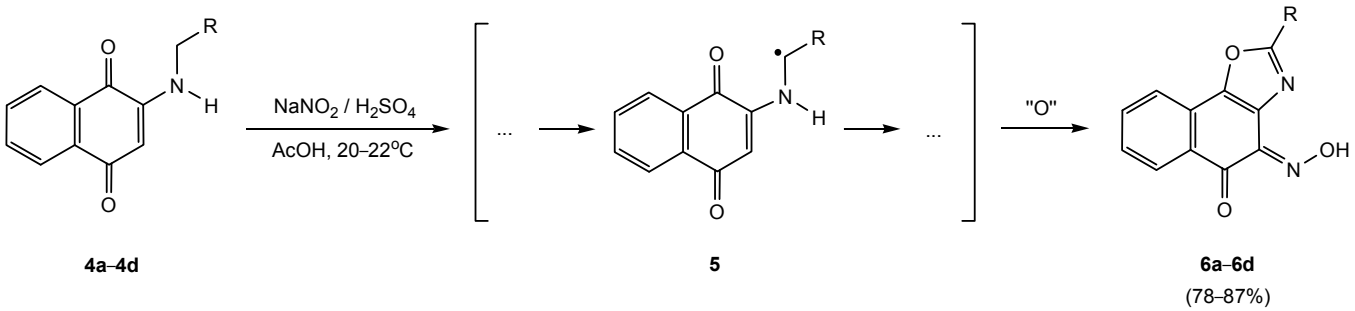
Scheme 1.



1 **2** **3**

R = Me, Bu, Bn.

Scheme 2.



R = Me, Et, Pr, *i*-Pr.

We earlier found that 2-alkylamino-1,4-naphthoquinones **4a–4d** react with nitrosylsulfuric acid to form 2-alkylnaphtho[2,1-*d*][1,3]oxazole-4,5-dione 4-oximes **6a–6d** in high yields [9] (Scheme 2).

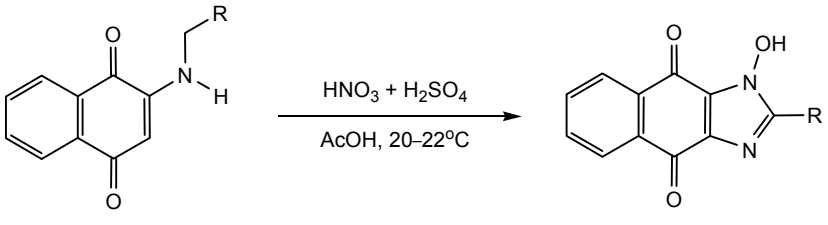
At the same time, treatment of 2-benzylamino- and 2-alkylamino-1,4-naphthoquinones **4a–4g** with nitration mixture in acetic acid afforded 2-R-1-hydroxy-1*H*-naphtho[2,3-*d*]imidazole-4,9-diones **7a–7g** as the main products [10, 11] (Scheme 3).

The reaction of 2-benzylamino-1,4-naphthoquinones with nitrosylsulfuric acid has never been studied. We found that 2-benzylamino-1,4-naphthoquinone **4e** reacts with nitrosylsulfuric acid in acetic acid to form products **7–10** (Scheme 4).

Under these conditions, like in the case of treatment of 2-benzylamino-1,4-naphthoquinone **4e** with nitration mixture, the main reaction product is 2-phenyl-1-hydroxy-1*H*-naphtho[2,3-*d*]imidazole-4,9-dione (**7e**) rather than 2-alkylnaphtho[2,1-*d*][1,3]-oxazole-4,5-dione 4-oxime (**8**) (Scheme 2). We suggest that the transformations of compound **4e**, shown in Scheme 4, occur by a radical cation mechanism involving intermediates **11a–11d** (Scheme 5). Apparently, the higher stability of radical **11d** (Scheme 5) compared to radical **5** (Scheme 2) predetermines changes toward reactions of 2-benzylamino-1,4-naphthoquinone (**4e**) and 2-alkylamino-1,4-naphthoquainones **4a–4d**.

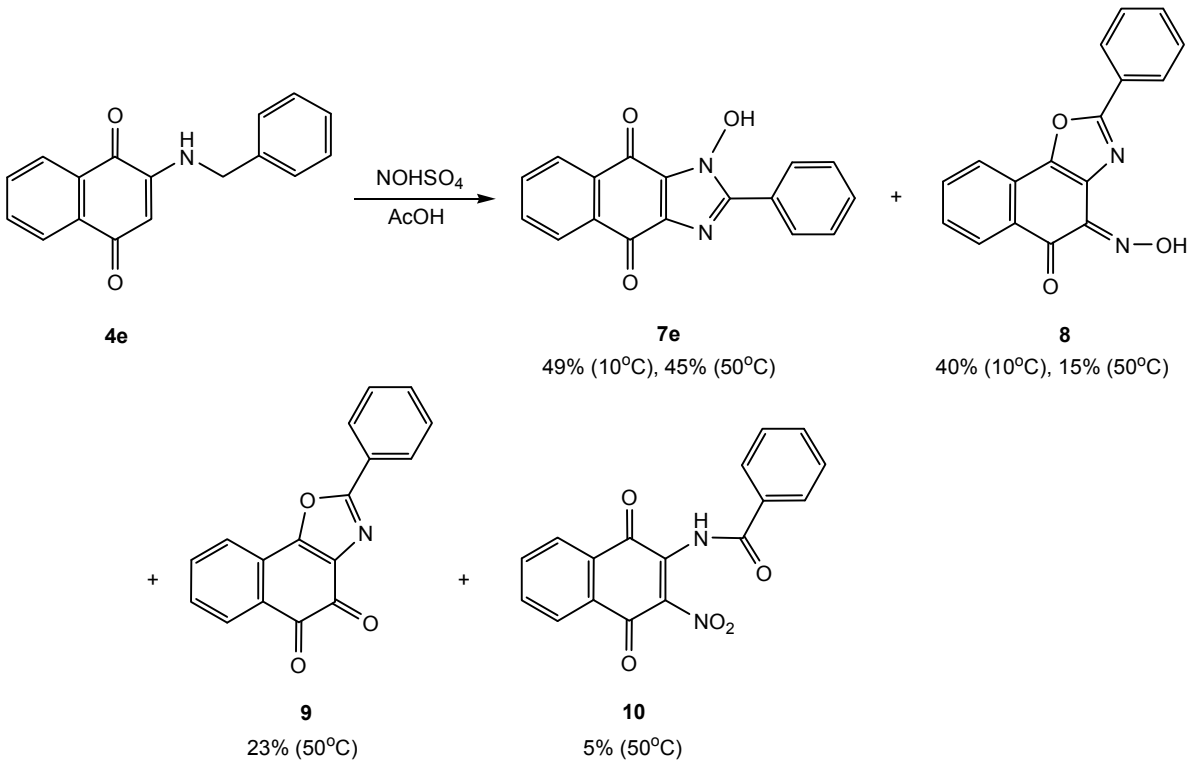
We established that 1-hydroxy-2-phenyl-1*H*-naphtho[2,3-*d*]imidazole-4,9-dione (**7e**) and (*E*)-4-(hydroxyimino)-2-phenylnaphtho[2,1-*d*]oxazole-5(4*H*)-one (**8**) are formed in a high total yield on treatment of 2-benzylamino-1,4-naphthoquinone (**4e**) with nitrosylsulfuric acid at 10–12°C. Treatment of hydroxyimidazole **7e** or oxazole **8** with sulfuric acid in acetic acid at 20–60°C does not lead to their interconversions. Consequently, hydroxyimidazole **7e** and oxazole **8** are formed from intermediates **11** concurrently by independent pathways. Treatment of 2-benzylamino-1,4-naphthoquinone (**4e**) with nitrosylsulfuric acid at 40–50°C produces, along with compounds **7e** and **8**, small amounts of 2-phenylnaphtho[2,1-*d*]oxazole-4,5-dione (**9**) and *N*-(3-nitro-1,4-dioxo-1,4-dihydronaphthalen-2-yl)benzamide (**10**). These compounds are not formed by the reaction of hydroxyimidazole **7e** or oxazole **8** with sulfuric acid in acetic acid. Apparently, compounds **9** and **10** are formed directly from precursors **11** or azoles **7e** and **8**, when they are treated with nitrogen oxides at an elevated temperature (50°C).

Scheme 3.



R = Me, Et, Pr, *i*-Pr, Ph, 4-Me-Ph, 4-Cl-Ph.

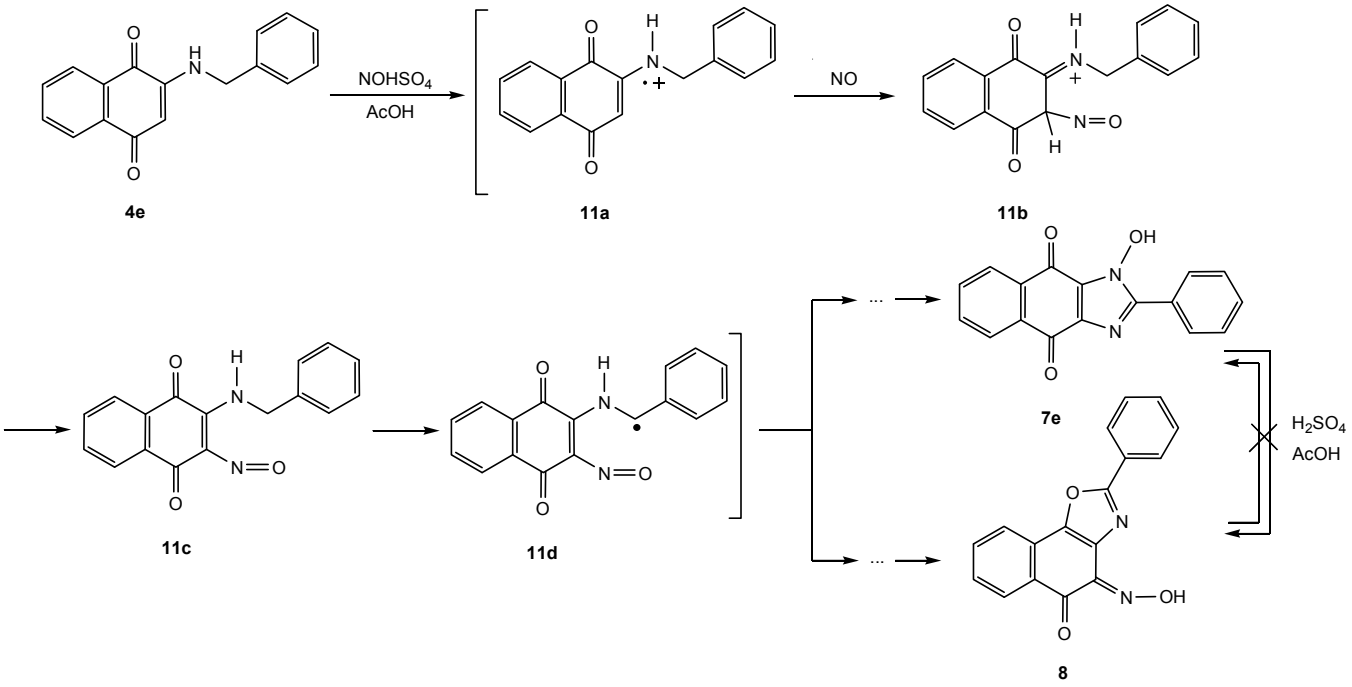
Scheme 4.

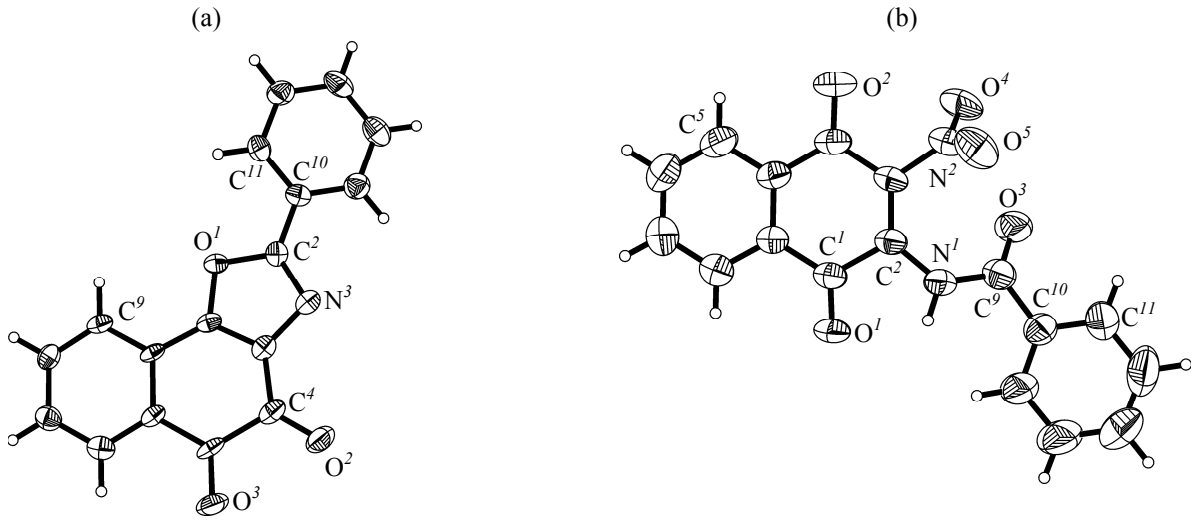


The products of these reactions were characterized by spectral methods and X-ray diffraction (XRD) analysis. The crystal structures of compounds **9** and **10** are shown in the figure.

The bond lengths in the naphthalenedione fragment in compound **9** are close to those in 6-methoxy-2-methylnaphtho[1,2-*b*]furan-4,5-dione [12]. The dihedral angle between the naphthalene core and

Scheme 5.





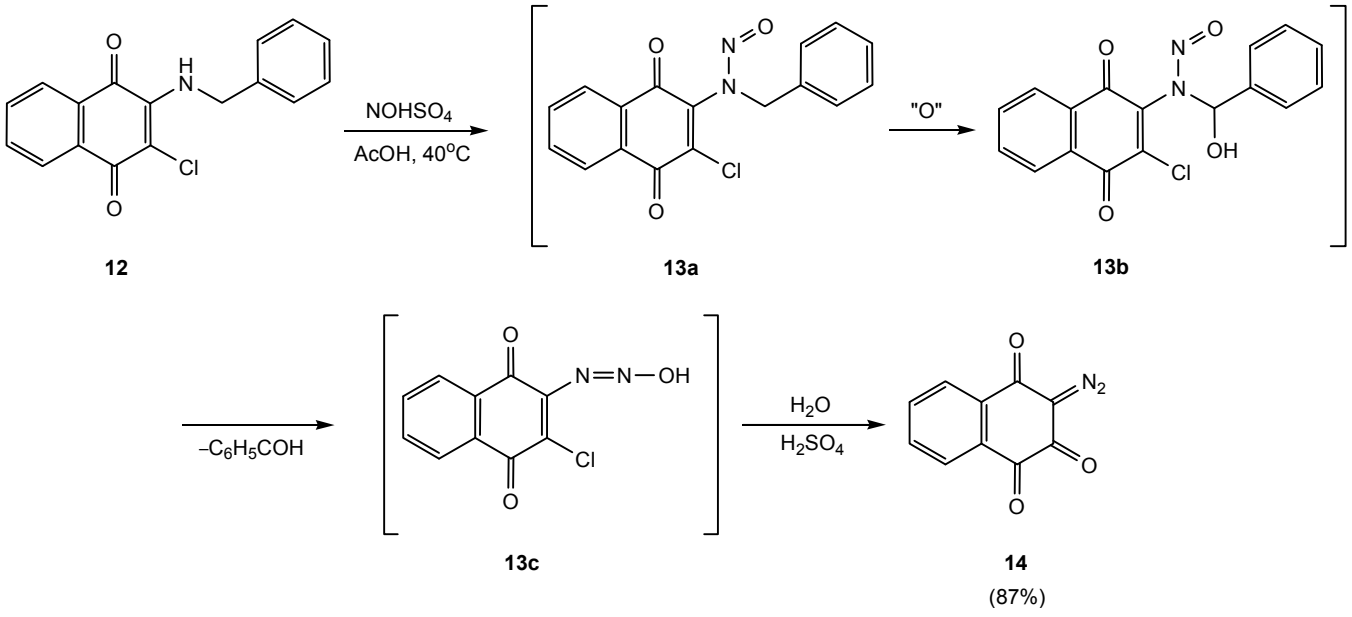
Structures of (a) 2-phenylnaphtho[2,1-*d*]oxazole-4,5-dione (**9**) and (b) *N*-(3-nitro-1,4-dioxo-1,4-dihydronaphthalen-2-yl)benzamide (**10**) by XRD data (50% thermal ellipsoid drawing).

phenyl planes is 14.5(3)°. The bond lengths in molecule **10** are close to the respective bond lengths in *N*-(1,4-dioxo-1,4-dihydronaphthalen-2-yl)benzamide [13]. The nitro and amido groups are turned out of the nathphalene core plane. The corresponding dihedral angles are 62.4(1) and 30.7(1)°. In the absence of the nitro group 13], the amido group is almost coplanar to the naphthalene core plane (angle 4.0°).

The reaction of 2-benzylamino-3-chloro-1,4-naphthoquinone (**12**) with nitrosylsulfuric acid proceeds in a different way. We found that the reaction gives 3-diazonaphthalene-1,2,4(3*H*)-trione (**14**). As the by-product we isolated benzaldehyde (yield 76%). In view of such results, we can propose a mechanism of the formation of compound **14**, which involves the oxidation of the *N*-nitroso compound to an alcohol with nitrogen oxides (Scheme 6).

Note that other 2-alkylamino-3-chloro-1,4-naphthoquinones did not react with nitrosylsulfuric acid in acetic acid under such conditions (~40°C). These results can suggest that for the 1,2-annelation of the oxazole ring of 2,3-annelation of the hydroxy-

Scheme 6.



imidazole ring to 2-R-amino-1,4-naphthoquinones **4a–4g**, the *3* position in the latter should be free (Schemes 2–4).

Apparently, 2-benzylamino-3-chloro-1,4-naphthoquinone (**12**) transforms into diazoquinone **14** by a pathway involving *N*-nitrosation followed by oxidation of the methylene group. The resulting *N*-nitrosamine **13b** eliminates a molecule of benzaldehyde to form 3-chloro-2-diazohydrate **13c**, which hydrolizes to diazo quinone **14**. Note that the first convenient synthetic approach to diazoquinone **14** via the reaction of 2-amino-3-chloro-1,4-naphthoquinones with nitrosylsulfuric acid was described by Mosby and Silva [14]; other authors [15–16] reported other methods of synthesis of this compound.

Thus, we found that 2-benzylamino-1,4-naphthoquinone and 2-benzylamino-3-chloro-1,4-naphthoquinone differently react with nitrosylacetic acid in acetic acid, even though these two reactions have a common feature, specifically, increased activity of the benzyl methylene group.

EXPERIMENTAL

The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker DRX spectrometer (500 and 125 MHz) in DMSO-d_6, internal reference TMS. The UV spectra were obtained on a Thermo Evolution 300 spectrophotometer (cells 10 mm) in 10^{-4} M toluene solutions. The IR spectrum of compound **14** was measured on a Thermo Nicolet iN10 spectrophotometer. The mass spectra were obtained on a Finnigan MAT 8200 instrument (EI, 70 eV). The high-resolution mass spectra of compounds **10** and **14** were obtained on a Bruker microOTOF II instrument in the positive mode with electrospray ionization (capillary voltage 4500 V) [17]. Scan range *m/z* 50–3000. The samples were injected in acetonitrile solutions, flow rate 3 μL/min. Nebulizer gas nitrogen (4 L/min), interface temperature 180°C. Elemental analysis was performed on an EURO EA 3000 automatic CHNS analyzer. The melting points were determined on a Boetius hot stage. Reaction progress and purity of compounds were controlled by TLC on Silufol UV-254 plates (eluent toluene–acetone, 4 : 1).

The XRD analysis of compounds **9** and **10** was performed on a Bruker Kappa Apex II diffractometer (MoK_α radiation, graphite monochromator). All calculations were performed using SHELX97 program suite. The hydrogen atoms were refined riding on their carrier atoms. The hydrogen atom of the amido group in compound **10** was located by difference synthesis and refined with isotropic displacement parameters with bond length constraints. Absorption correction was applied using SADABS program. The crystal data were deposited in the Cambridge Crystallographic Data Center (CCDC 1853605 and 1853606 for **9** and **10**, respectively) and can be requested by www.ccdc.cam.ac.uk/data_request/cif. The starting aminonaphthoquinones **4e** and **12** were prepared as described in [18, 19].

Reactions of 2-(benzylamino)-1,4-naphthoquinone 4e and 2-(benzylamino)-3-chloro-1,4-naphthoquinone 12 with nitrosylsulfuric acid (*general procedure*). *a*. Nitrosylsulfuric acid (10 mL), prepared from $NaNO_3$ (0.80 g) and 92% H_2SO_4 (8 mL), was added to a stirred suspension of 1.32 g (5 mmol) of 2-(benzylamino)-1,4-naphthoquinone (**4e**) in CH_3COOH (25 mL) at 20°C. As this took place, the reaction mixture warmed up to 50°C and was held at this temperature for 1 h, after which it was cooled to 20°C and poured into 500 g of ice with water. The orange precipitate that formed was filtered off, washed with water, and dried. The resulting dry material, 1.22 g, was added to 40 mL of chloroform, and the mixture was refluxed for 20 min, cooled to 20°C, and filtered. The precipitate on the filter was practically pure 1-hydroxy-2-phenyl-1*H*-naphtho-[2,3-*d*]imidazole-4,9-dione (**7e**). Yield 0.60 g (45.3%). The filtrate was chromatographed on silica gel (eluent chloroform) to isolate 0.20 g (15.2%) of (*E*)-4-(hydroxyimino)-2-phenylnaphtho-[2,1-*d*]oxazol-5(4*H*)-one (**8**), 0.30 g (22.7%) 2-phenylnaphtho[2,1-*d*]oxazole-4,5-dione (**9**), and 0.07 g (5.3%) of *N*-(3-nitro-1,4-dioxo-1,4-dihydronaphthalen-2-yl)benzamide (**10**).

b. The reaction of 1.32 g (5 mmol) of 2-benzylamino-1,4-naphthoquinone (**4e**) in CH_3COOH (30 mL) with nitorsylsulfuric acid prepared from $NaNO_2$ (0.80 g) and 92% H_2SO_4 (8 mL) for 10 min with external heating (10–12°C) and stirring for 20 min followed by the workup similar to that described in procedure *a* gives 0.64 g of hydroxyimidazole **7e** (48.5%) and 0.53 g of oxazole **8** (40.2%).

c. 2-Benzylamino-3-chloro-1,4-naphthoquinone (**12**), 5.95 g (20 mmol), was added to CH_3COOH (100 mL), and nitrosylsulfuric acid prepared from $NaNO_2$ (3 g) and 94% H_2SO_4 (30 mL) was added with stirring to the resulting suspension. The reaction mixture warmed up to 40–45°C and was stirred at this temperature for 2 h.

The solution changed color from red to pale yellow. It was poured into a mixture of ice with water (800 g). The pale yellow precipitate that formed was filtered off, washed with water, and dried to obtain 3.5 g (87.5%) of 3-diazonaphthalene-1,2,4(3*H*)-trione (**14**). The filtrate was extracted with chloroform (3 × 100 mL), and the extract was evaporated to isolate 1.62 g (76.4%) of benzaldehyde.

1-Hydroxy-2-phenyl-1*H*-naphtho[2,3-*d*]imidazole-4,9-dione (7e) was identical to the product synthesized in [11]. Yield 0.64 g (48.5%) at 10°C and 0.60 g (45.3%) at 50°C, pale yellow crystals; mp 248–250°C. UV spectrum (toluene), λ_{max} (log ε), nm: 286 (4.39), 343 (3.60), 544 (3.19). ^{1}H NMR spectrum, δ, ppm: 7.55–7.63 m (3H, H$^{3',4',5'}$), 7.86 s [1H, H$^{5(8)}$], 7.85 s [1H, H$^{5(8)}$], 8.10 s (2H, H^{6,7}), 8.20 s (2H, H$^{2',6'}$), 13.25 br.s (1H, OH). ^{13}C NMR spectrum, δ, ppm: 178.02 [1C, C$^{4(9)}$], 173.87 [1C, C$^{4(9)}$], 147.69 (1C, C^2), 138.01 [1C, C$^{9a(3a)}$], 134.01 [1C, C$^{5(8)}$], 133.86 [1C, C$^{5(8)}$], 133.02 [1C, C$^{4a(8a)}$], 132.62 [1C, C$^{4a(8a)}$], 130.75 [1C, C$^{9a(3a)}$], 130.67 (1C, C$^{4'}$), 128.84 (2C, C$^{3',5'}$), 128.24 (2C, C$^{2',6'}$), 127.24 (1C, C$^{1'}$), 126.45 [1C, C$^{6(7)}$], 126.09 [1C, C$^{6(7)}$]. Found, %: C 70.81; H 3.48; N 9.60. $C_{17}H_{10}N_2O_3$. Calculated, %: C 70.34; H 3.47; N 9.65. *M* 290.28.

(*E*)-4-(Hydroxyimino)-2-phenylnaphtho[2,1-*d*]-oxazol-5(4*H*)-one (8). Yield 0.53 g (40.2%) at 10°C and 0.20 g (15.2%) at 50°C, yellow orange crystals; mp 206–208°C. UV spectrum (toluene), λ_{max} (log ε), nm: 313 (4.25), 429 (3.46). ^{1}H NMR spectrum, δ, ppm: 13.67 s (NOH), 8.18–8.15 m (2H, H$^{2',6'}$), 8.10 d (1H, H^6, *J* 7.5 Hz), 7.92 d (1H, H^9, *J* 7.5 Hz), 7.84 d.t (1H, H^8, *J* 7.5, *J* 1.2 Hz), 7.63–7.60 m (3H, H$^{3',5',4'}$), 7.58 d.t (1H, H^7, *J* 7.5, *J* 1.2 Hz). ^{13}C NMR spectrum, δ, ppm: 180.61 (1C, C^5), 160.90 (1C, C^2), 146.81 (1C, C^{9b}), 141.83 (1C, C^4), 134.91 (1C, C^8), 131.42 (1C, C$^{4'}$), 129.61 (1C, C$^{1'}$), 129.50 (1C, C^{9a}), 129.31 (1C, C^7), 129.26 (2C, C$^{3'}$), 128.48 (1C, C^6), 126.59 (2C, C$^{2'}$), 126.43 (1C, C^{3a}), 126.12 (1C, C^{5a}), 121.56 (1C, C^9). Mass spectrum, *m/z* (I_{rel}, %): 290 (30.83) [*M*]$^+$, 274 (2.90), 158 (5.31), 129 (7.71), 114 (11.31), 106 (10.81), 105 (100), 103 (10.11), 102 (5.01), 101 (13.01), 77 (21.52), 76 (8.91), 75 (6.51), 30 (6.51). Found, %: C 70.48.; H 3.47; N 9.20. $C_{17}H_{10}N_2O_3$. Calculated, %: C 70.34; H 3.47; N 9.65. *M* 290.28.

2-Phenylnaphtho[2,1-*d*]oxazole-4,5-dione (9). Yield 0.30 g (22.7%), mp 138–140°C. UV spectrum (toluene), λ_{max} (log ε), nm: 296 (4.25), 438 (3.08). ^{1}H NMR spectrum, δ, ppm: 8.21–8.19 m (2H, H$^{2',6'}$), 8.03 d (1H, H^6, *J* 7.2 Hz), 7.96 d (1H, H^9, *J* 7.2 Hz), 7.83 d.t (1H, H^8, *J* 7.6 Hz), 7.67–7.63 m (4H, H$^{3',4',5',7}$). ^{13}C NMR spectrum, δ, ppm: 178.83 (1C, C^5), 172.35 (1C, C^4), 161.50 (1C, C^2), 157.53 (1C, C^{9b}), 135.02 (1C, C^{3a}), 134.98 (1C, C^8), 132.08 (1C, C$^{4'}$), 130.99 (1C, C^7), 130.19 (1C, C$^{1'}$), 129.58 (1C, C^6), 129.44 (2C, C$^{3',5'}$), 126.88 (2C, C$^{2',6'}$), 125.44 (1C, C^{5a}), 125.38 (1C, C^{9a}), 122.84 (1C, C^9). Mass spectrum, *m/z* (I_{rel}, %): 275 (37.14), 247 (22.02), 172 (6.01), 114 (6.61), 104 (27.93), 89 (20.72), 88 (41.44), 77 (38.24), 76 (100), 74 (10.21), 63 (20.52), 62 (26.23), 51 (23.22), 50 (33.43). Found, %: C 74.63; H 3.68; N 4.68. $C_{17}H_9NO_3$. Calculated, %: C 74.18; H 3.30; N 5.09. *M* 275.26.

Rhombic crystals, temperature 200 K, *a* 24.505(8), *b* 6.881(3), *c* 7.350(4) Å, *Z* 4, space group *Pca*2$_1$, d_{calc} 1.475 g/cm^3, μ 0.103 mm^{-1}, θ ≤ 25.2°, measured reflections 2143 (unique 1726), *R* 0.0346 on 1332 reflections with $I > 2\sigma(I)$, wR_2 0.0880 on all reflections.

***N*-(3-Nitro-1,4-dioxo-1,4-dihydronaphthalen-2-yl)benzamide (10).** Yield: 0.07 g (5.3%), light yellow crystals; mp 217–218°C. UV spectrum (toluene), λ_{max} (log ε), nm: 286 (4.05). ^{1}H NMR spectrum, δ, ppm: 10.4 s (NH), 8.13 d (1H, H^8, *J* 6.8 Hz), 8.11 d (1H, H^5, *J* 6.8 Hz), 8.00 d (2H, H$^{2',6'}$, *J* 7.6 Hz), 7.97 t (1H, H^7, *J* 7.4 Hz), 7.94 t (1H, H^6, *J* 7.4 Hz), 7.72 t (1H, H$^{4'}$, *J* 7.5 Hz), 7.6 t (2H, H$^{3',5'}$, *J* 7.4 Hz). ^{13}C NMR spectrum, δ, ppm: 179.79 (1C, C^1), 175.42 (1C, C^4), 165.71 (1C, CONH), 137.76 (1C, C^3), 135.68 (1C, C^7), 134.78 (1C, C^6), 133.75 (1C, C^2), 133.63 (1C, C$^{4'}$), 131.93 (1C, C$^{1'}$), 130.13 [1C, C$^{4a(8a)}$], 130.10 [1C, C$^{4a(8a)}$], 128.88 (2C, C$^{3',5'}$), 128.68 (2C, C$^{2',6'}$), 126.82 (1C, C^8), 126.60 (1C, C^5). Mass spectrum, *m/z* (I_{rel}, %): 276 (8.31), 106 (7.51), 105 (100), 77 (46.25), 76 (9.21), 51 (11.91). High-resolution mass spectrum, *m/z*: 345.0474 [*M* + Na]$^+$. Found, %: C 63.67; H 3.17; N 8.51. $C_{17}H_{10}N_2O_5$. Calculated, %: C 63.36; H 3.13; N 8.69. *M* 322.27, *M* + Na 345.0482.

Monoclinic crystals, temperature 296 K, *a* 5.2751(3), *b* 13.4961(9), *c* 20.5594(12) Å, β 97.109(2)°, *Z* 4, space group *P*2$_1$/*n*, d_{calc} 1.474 g/cm^3, μ 0.111 mm^{-1}, θ ≤ 26.1°, measure reflections 25189 (unique 2877), *R* 0.0466 on 2015 reflections with $I > 2\sigma(I)$, wR_2 0.1648 on all reflections.

3-Diazonaphthalene-1,2,4(3*H*)-trione (14). Yield: 3.5 g (87.5%), pale yellow crystals; mp 119–121°C ($CHCl_3$) (121–122°C [16]). UV spectrum (toluene), λ_{max} (log ε), nm: 284 (3.90). IR spectrum, ν, cm^{-1}: 2120–2200 (N=N). ^{1}H NMR spectrum, δ, ppm: 8.13

d.t [1H, $H^{6(7)}$, *J* 7.6, *J* 1.3 Hz], 8.08–8.11 m [1H, $H^{5(8)}$], 7.95 d.t [1H, $H^{6(7)}$, *J* 7.6, *J* 1.3 Hz], 7.89–7.92 m [1H, $H^{5(8)}$]. ^{13}C NMR spectrum, δ, ppm: 177.54 (1C, C^{1}), 176.82 (1C, C^{4}), 172.36 (1C, C^{2}), 142.81 (1C, C^{3}), 135.34 (1C, C^{6}), 135.04 (1C, C^{8a}), 134.85 (1C, C^{7}), 128.46 (1C, C^{8}), 127.45 (1C, C^{4a}), 126.56 (1C, C^{5}). Mass spectrum, *m/z* (I_{rel}, %): 200 (3.40) $[M]^+$, 172 (69.07), 104 (89.19), 88 (21.12), 77 (13.51), 76 (100), 74 (18.12), 68 (10.31), 62 (19.92), 50 (41.64). High-resolution mass spectrum, *m/z* 223.0111 $[M + Na]^+$. Found, %: C 59.95; H 2.02; N 13.68. $C_{10}H_4N_2O_3$. Calculated, %: C 60.01; H 2.01; N 14.00. *M* 200.15, *M* + Na 223.0114.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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