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HETEROCYCLIC ANALOGS OF 5,12-NAPHTHACENE-QUINONE. 3.* SYNTHESIS OF 4,11-DIAMINONAPHTHO-[2,3-*f*]INDOLE-5,10-DIONE AND CERTAIN OF ITS DERIVATIVES

A. E. Shchekotikhin[1], Yu. N. Luzikov[1], V. N. Buyanov[2], and M. N. Preobrazhenskaya[1]

Nucleophilic substitution of methoxy groups in 4,11-dimethoxynaphtho[2,3-f]indole-5,10-dione by the action of primary and secondary alkylamines, or arylamines leads to the formation of N-alkyl or N-aryl derivatives of 4,11-diaminonaphtho[2,3-f]indole-5,10-dione respectively. 4,11-Diamino-1H-naphtho-[2,3-f]indole-5,10-dione is obtained by the dealkylation of 4,11-bis[(1-phenylethyl)amino]-1H-naphtho[2,3-f]indole-5,10-dione in the presence of a Lewis acid (BBr_3).

Keywords: 4,11-diamino-1H-naphtho[2,3-*f*]indole-5,10-dione, 4,11-dimethoxynaphtho[2,3-*f*]indole-5,10-dione, dealkylation of alkylamino groups, nucleophilic aromatic substitution, solvatochromism.

Previously we synthesized 4,11-dimethoxynaphtho[2,3-*f*]indole-5,10-dione [2] and some of its derivatives and showed that demethylation of the methoxy groups is a convenient method of synthesizing 4,11-dihydroxynaphtho[2,3-*f*]indole-5,10-dione (pyrroloquinizarin) and its derivatives [3, 4]. The present work is devoted to the development of a method of synthesizing 4,11-diaminonaphtho[2,3-*f*]indole-5,10-dione, derivatives of which are promising in the search for biologically active substances but have not been obtained up to the present time.

The traditional approach to the synthesis of 1,4-diaminoanthraquinone derivatives from leucocompounds, derivatives of 1,4-dihydroxyanthraquinone (quinizarin) for the synthesis of 4,11-diaminonaphtho[2,3-*f*]indole-5,10-dione, proved to be of no use since we were unsuccessful in obtaining the leucocompound of pyrroloquinizarin. Another approach to the synthesis of 1,4-diaminoanthraquinone derivatives is based on nucleophilic aromatic substitution reactions. In the anthraquinone series not only are good [5] leaving groups (halogens, sulfo, nitro groups) substituted by N-nucleophiles, but also even poor leaving groups (hydroxy, aryloxy, alkoxy groups, and hydride ion) [6]. The rarely encountered aromatic substitution reaction of methoxy groups by amines was used in several cases for the synthesis of amino derivatives of anthraquinone [7-9]. We have established that the analogous reaction in the methoxynaphtho-[2,3-*f*]indole-5,10-dione series proceeds somewhat more readily than in the anthraquinone series and may be used as a preparative method for the synthesis of various aminonaphtho[2,3-*f*]indole-5,10-diones.

4,11-Dimethoxynaphtho[2,3-*f*]indole-5,10-dione interacts under mild conditions with primary alkylamines. For example, 4,11-bis[(2-hydroxyethyl)amino]naphtho[2,3-*f*]indole-5,10-dione (**2**) is obtained by the interaction of naphthoindoledione **1** with monoethanolamine on boiling in THF.

* For Part 2 see [1].

[1] G. F. Gauze Institute of New Antibiotics, the Russian Academy of Medical Science Moscow 119021, Russia. [2] Russian D. I. Mendeleev University of Chemical Technology, Moscow 125190; e-mail: chekotikhin@mtu-net.ru. Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 6, pp. 854-861, June, 2006. Original article submitted March 21, 2005.

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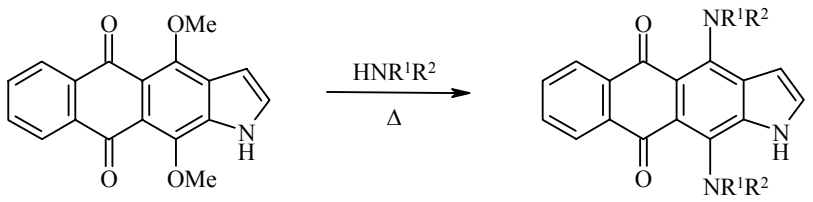
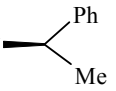


1 **2– 5**

2 NR^1R^2 = $HN(CH_2)_2OH$ (75%); **3** NR^1R^2 = HNPh (57%); **4** NR^1R^2 = piperidino (63%),

5 NR^1R^2 = pyrrolidinyl (70%), **6** NR^1R^2 =  (75%)

Under more forcing conditions the methoxy groups in naphthoindole **1** may be substituted by a primary arylamine residue. 4,11-Bis(phenylamino)naphtho[2,3-*f*]indole-5,10-dione (**3**) is formed on boiling **1** in aniline. In addition, naphthoindole **1** also reacts analogously with secondary aliphatic amines. For example, it reacts with piperidine somewhat more slowly than with primary alkylamines, giving on boiling 4,11-di(piperidino)naphtho[2,3-*f*]indole-5,10-dione (**4**). Reaction with pyrrolidine occurs under practically the same conditions as with ethanolamine with the formation of 4,11-di(1-pyrrolidinyl)naphtho[2,3-*f*]indole-5,10-dione (**5**). It should be noted that the derivatives of secondary amines **4** and **5**, unlike the monosubstituted amines **2** and **3,** dissolve readily in dilute mineral acid and form salts, such as the yellow hydrochlorides. An analogous increase in basicity is observed for N-disubstituted derivatives of α-aminoanthraquinones and is explained by the emergence of steric difficulties and by the deviation of the geometry of the molecule from planarity. This leads to a reduction in the intensity of the long-wave absorption of the free bases compared with the spectra of the N-monosubstituted analogs [10].

The singlet signals of the methoxy group protons are absent from the ^{1}H NMR spectra of the obtained derivatives of 4,11-diaminonaphtho[2,3-*f*]indole-5,10-dione **2-5** and signals appear for the appropriate substituents, *viz*. 2-hydroxyethyl, phenyl, piperidino, and pyrrolidinyl fragments respectively. In the low field portion of the spectra of compounds **2** and **3** at 12.5-13.5 ppm triplets (with J = 6.2 Hz) are observed for compound **2** and singlets for compound **3** for the signals of the NH group protons, located in the α-position to the carbonyl groups of the anthraquinone fragment.

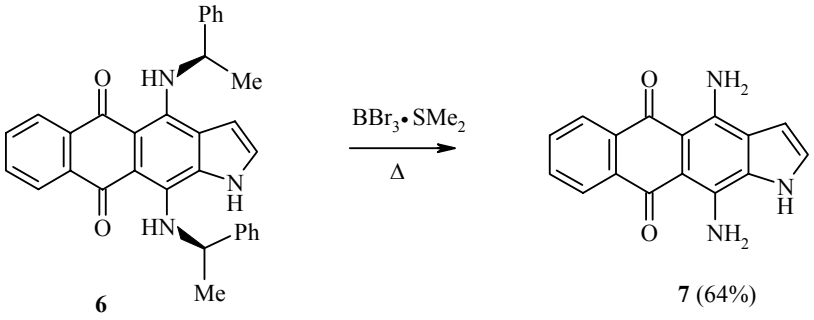
In the high field region of the spectrum of compound **2** singlet signals were observed for the hydroxyl group protons at 5.93 and 4.97 ppm. The difference in the chemical shifts of the hydroxyl group proton signals is probably explained by the effect of the NH group proton of the pyrrole fragment on the hydroxyl group of the aminoethanol substituent at position 11. In addition, compared with the spectrum of the initial compound **1** and of amino derivatives **2**, **4**, and **5**, in the spectrum of diphenylamino derivative **3** significant displacements were observed for the signals of the H-2 and H-3 protons of the pyrrole fragment in the low field region to 6.78 and 5.77 ppm, which is caused by the effect of the magnetic anisotropy of the side phenyl rings (ASIS effect).

In the IR spectrum of ethanolamino derivative **2** an intense broad band was observed at 3300-3500 cm^{-1} for the absorption of the N–H and O–H groups. The absorption band of the stretching vibrations of the C=O groups of the anthraquinone fragment of the molecule are found in the region of 1590 cm^{-1}.

In spite of the fact that the methoxyl groups in naphthoindole **1** are replaced by residues of primary and secondary alkylamines and even weaker nucleophiles, arylamines, we were unsuccessful when carrying out an analogous substitution by the action of ammonia. Naphthoindole **1** withstood heating for several hours in a sealed ampul with ammonia and dioxane and no substitution products were detected by us.

In certain cases the dealkylation of α-monoalkylaminoanthraquinones has been used for the synthesis of primary α-aminoanthraquinones [6]. Since dealkylation occurs in the presence of Brönsted or Lewis acids by a cationic mechanism, then under the mildest conditions anthraquinones containing α-alkylamino groups are

dealkylated giving stable carbocations, such as *tert*-butyl, benzyl, or 1-phenylethylamino groups [11]. Consequently by the action of (1*R*)-phenylethylamine on naphthoindole **1**, we obtained 4,11-bis{[(1*R*)-1-phenylethyl]amino}naphtho[1,3-*f*]indole-5,10-dione (**6**), containing readily eliminatable alkyl groups in the side chains. Subsequent dealkylation of naphthoindole **6** under the action of $BBr_3 \cdot SMe_2$ by boiling in dichloroethane leads to the formation of 4,11-diaminonaphtho[2,3-*f*]indole-5,10-dione (**7**).



6 **7** (64%)

Intense peaks were observed in the mass spectra of compounds **2-7** for the molecular ions with M^+ 365 (**2**), 429 (**3**), 413 (**4**), 385 (**5**), 485 (**6**), and 277 (**7**), which correspond to their molecular masses.

According to the data of electronic absorption spectra replacement of the methoxyl groups in naphthoindole **1** by an amino group in compounds **2-7** causes a bathochromic shift of 100-125 nm in the long-wave absorption maxima (Fig. 1). In the spectra of 4,11-di(alkylamino)- and 4,11-diaminonaphtho[2,3-*f*]indole-5,10-diones **2, 3, 5-7** three absorption bands were observed in the long-wave region, displayed as two intense maxima and a short-wave inflection. Such double-peaked absorption is characteristic of $\pi_1,\pi^*\alpha_1$-absorption bands for derivatives of 1,4-diamino- and 1-amino-4-hydroxyanthraquinones [12]. In addition, less intense short-wave absorption bands were observed in the spectra of aminonaphthoindoles **3-7** in the 355-400 nm region, practically coinciding precisely with the position of the $\pi_1,\pi^*\alpha_2$-band in the spectra of the analogous derivatives of 1,4-diaminoanthraquinone [10]. The absorption spectra of compounds **4** and **5** in ethanol differed significantly in the position and structure of the long-wave absorption maximum. The long-wave absorption maximum of the pyrrolidine derivative **5** was shifted bathochromically by 25 nm compared with the spectrum of the piperidine analog **6**, and has a marked double-peak structure and practically coincides with the spectrum of the diphenyl derivative **3**.

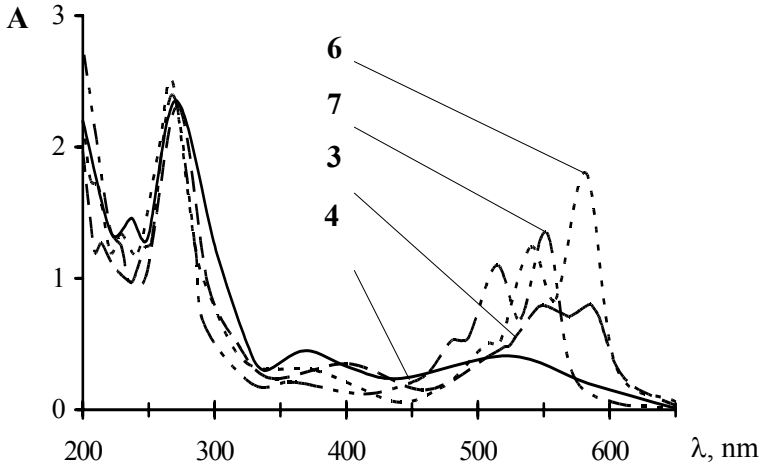


Fig. 1. Electronic absorption spectra of compounds **3, 4, 6, 7** in ethanol.

Dealkylation of the diaminonaphthoindole **6** causes a hypsochromic shift of ~30 nm of the long wave absorption maxima of 4,11-diaminonaphtho[2,3-*f*]indole-5,10-dione (**7**). A somewhat larger effect was observed on monoalkylation of the amino groups of 1,4-diaminoanthraquinone causing a shift of the absorption maximaum of ~40-50 nm [10]. Comparison of the electronic absorption spectra of naphthoindoles **2, 3, 6, 7** and the 1,4-disubstituted analogs in the anthraquinone series shows that annelation of the pyrrole fragment to the anthraquinone chromophore causes a hypsochromic shift of the long-wave absorption maxima in the spectra of derivatives of 4,11-diaminonaphtho[2,3-*f*]indole-5,10-dione of ~70 nm. The increase of color, in accordance with the Stepanov rules [13], indicates the intense interaction between the annelated pyrrole fragment and the carbonyl groups leading to a disengaging of the conjugation chain between the amino and carbonyl groups.

The absorption bands in the electronic absorption spectra of compound **7** and its N-monosubstituted derivatives **2**, **3**, and **6** had little sensitivity towards a change of solvent. In contrast to this the derivatives of secondary amines **4** and **5** possessed marked positive solvatochromism (Fig. 2), since the position of both long-

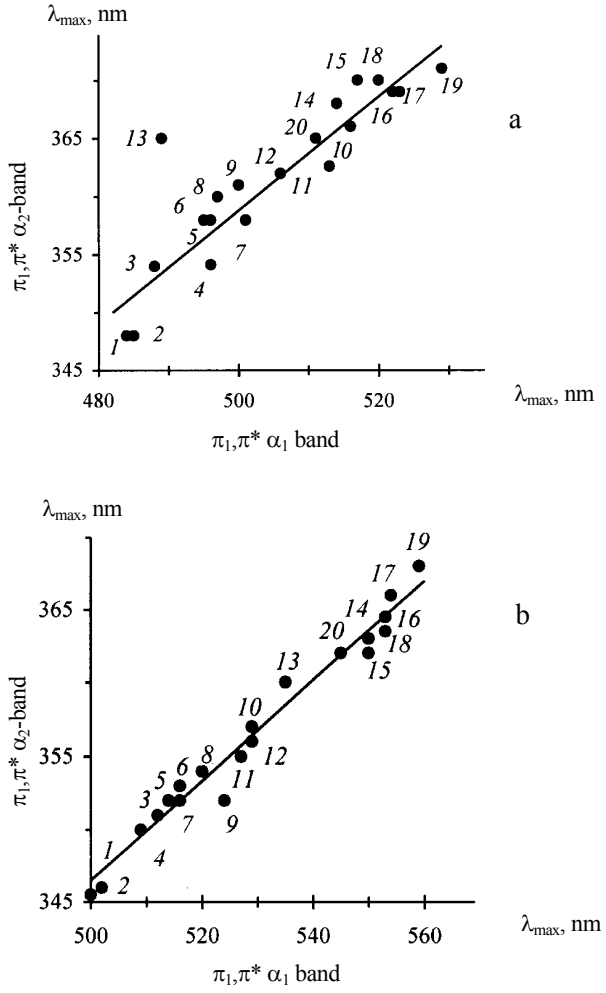


Fig. 2. Sensitivity to solvent of the long-wave bands in the absorption spectra of naphthoquinones **4** (a) and **5** (b). Solvents in order of increasing solvatochromic parameter π* [14] were 1) hexane, 2) cyclohexane, 3) diethyl ether, 4) carbon tetrachloride, 5) toluene, 6) ethyl acetate, 7) benzene, 8) THF, 9) acetone, 10) chloroform, 11) methylene chloride, 12) acetonitrile, 13) DMF, 14) DMSO, 15) 2-propanol, 16) propanol, 17) ethanol, 18) methanol, 19) water (compounds **4** and **5** as dihydrochlorides), 20) acetic acid.

wave absorption maxima changed practically in proportion to the increase in the solvatochromic parameter for π*-aprotic solvents [14]. In addition, as already mentioned above, the long-wave absorption band for compound **5** in ethanol has a discontinuity and two maxima. A similar structure for the long-wave absorption band is retained in all the protic and low polarity (hexane and cyclohexane) solvents, while solutions of the pyrrolidine derivative **5** in the remaining solvents have only one long-wave absorption maximum. In conclusion it should be noted that the absorption spectra of the hydrochlorides of naphthoindoles **4** and **5** in water are close to the absorption spectra in other protic solvents, while in acid medium (pH <1) they differ substantially. In 0.01 N HCl solution the absorption spectrum of the piperidino derivative **4** is close to the absorption spectrum of the initial naphthoindole **1** and is characterized by a small hypsochromic shift of the long-wave maximum (416 nm). The absorption spectrum of the pyrrolidinyl derivative **5** in 0.01 N HCl solution differs from the spectrum of the initial naphthoindole **1** by the bathochromic shift of the long-wave maximum (453 nm).

EXPERIMENTAL

The ^{1}H NMR spectra were recorded on a Varian VXR 400 (400 MHz) spectrometer, internal standard was TMS. The mass spectra were recorded on a Finnigan-MAT SSQ 710 chromato-mass spectrometer, energy of ionizing voltage 70 eV, direct insertion of sample into the ion source, sample heated to 350°C, temperature of ionization chamber 150°C. The IR spectra were obtained on a Perkin-Elmer 599 spectrometer in KBr disks. Absorption spectra were recorded on a Hitachi U2000 spectrometer. A check on the progress of reactions and the purity of compounds was effected by TLC on Silufol UV 254 plates.

4,11-Bis[(2-hydroxyethyl)amino]-1H-naphtho[2,3-*f*]indole-5,10-dione (2). 2-Aminoethanol (1.0 ml, 15 mmol) was added to 4,11-dimethoxynaphtho[2.3-*f*]indole-5,10-dione (**1**) (0.1 g, 0.33 mol) in THF (3.0 ml), and the mixture was boiled in an atmosphere of argon until complete disappearance of the starting material from the reaction mixture (according to TLC). The violet reaction mass was cooled to room temperature, ethyl acetate (20 ml) was added, and the mixture poured into water. The organic phase was washed with dilute HCl (1%), with water, dried over $MgSO_4$, and evaporated in vacuum. The residue was purified chromatographically (silica gel, benzene–acetone, 3:1), recrystallized from methanol, and compound **2** (0.09 g, 75%) was obtained as needle-like crystals of a dark violet color with mp 210-213°C. IR spectrum, ν, cm^{-1}: 3300-3500 (NH, OH), 1590 (C=O). ^{1}H NMR spectrum, $CDCl_3$), δ, ppm (*J*, Hz): 13.15 (1H, t, *J* = 6.2, NH); 12.68 (1H, t, *J* = 6.2, NH); 12.37 (1H, s, NH); 8.32 (2H, m, H-6, 9); 7.64 (2H, m, H-7, 8); 7.35 (1H, m, H-2); 7.03 (1H, m, H-3); 5.93 (1H, s, OH); 4.97 (1H, s, OH); 3.92 [8H, m, (-CH_2-)$_2$]. Mass spectrum, *m/z* (I_{rel}, %): 365 (100), 313 (32), 279 (21). Found, %: C 65.30; H 5.53; N 11.57. $C_{20}H_{19}N_3O_4$. Calculated, %: C 65.74; H 5.24; N 11.50.

4,11-Bis(phenylamino)-1H-naphtho[2,3-*f*]indole-5,10-dione (3). A solution of naphthoindole **1** (0.1 g, 0.33 mmol) in aniline (2 ml, 22 mmol) was boiled in an atmosphere of argon until complete disappearance of the starting material from the reaction mixture (by TLC). The mixture was cooled to room temperature, ethyl acetate (20 ml) was added to the violet reaction mixture, which was then poured into water. The organic phase was washed three times with dilute (1%) HCl, with water, dried over $MgSO_4$, and evaporated in vacuum. The residue was purified chromatographically (silica gel, benzene–acetone, 3:1), recrystallized from toluene, and naphthoindole **3** (0.08 g, 57%) obtained as dark violet crystals of mp 226-229°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 13.35 (1H, s, NH); 12.81 (1H, s, NH); 8.38 (2H, m, H-6, 9); 8.08 (1H, br. s, NH); 7.72 (2H, m, H-7, 8); 7.22-7.38 (10H, m, NC_6H_5); 6.78 (1H, m, *J* = 3.2, H-2); 5.77 (1H, m, *J* = 3.2, H-3). Mass spectrum, *m/z* (I_{rel}, %): 429 (100) [M]$^+$, 354 (7), 336 (5). Found, %: C 78.39; H 4.59; N 9.55. $C_{28}H_{19}N_3O_2$. Calculated, %: C 78.31; H 4.46; N 9.78.

4,11-Di(piperidino)-1H-naphtho[2,3-*f*]indole-5,10-dione (4). A solution of naphthoindole **1** (0.1 g, 0.33 mol) in piperidine (2.0 ml, 20 mmol) was boiled in an atmosphere of argon for 3 h. The mixture was cooled to room temperature, ethyl acetate (20 ml) was added to the violet reaction mixture, which was then poured into

water. The organic phase was washed three times with water, dried over $MgSO_4$, and evaporated in vacuum. The residue was purified chromatographically (silica gel, benzene–acetone, 3:1), recrystallized from a toluene–heptane mixture, and naphthoindole **4** (0.08 g, 63%) was obtained as claret colored crystals of mp 208-210°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 8.82 (1H, br. s, NH); 8.19 (2H, m, H-8, 9); 7.66 (2H, m, H-7, 8); 7.28 (1H, t, *J* = 3.0, H-2); 6.78 (1H, t, *J* = 3.0, H-3); 3.40 [4H, br. s, $N(CH_2)_2$]; 3.15 [4H, br. s, $N(CH_2)_2$]; 1.76 (12H, br m, CH_2). Mass spectrum, *m/z* (I_{rel}, %): 413 (94) $[M]^+$, 396 (100) $[M\text{-}OH]^+$, 327 (7), 313 (45), 297 (19). Found, %: C 75.91; H 6.92; N 10.25. $C_{26}H_{27}N_3O_2$. Calculated, %: C 75.52; H 6.58; N 10.16.

Dihydrochloride of 4 was obtained by adding a 2% ethereal HCl solution to a solution of **4** free base in MeOH. The salt was precipitated with ether, filtered off, washed with ether, and with hexane. The yellow substance was collected rapidly, and dried in vacuum; mp 185-190°C (decomp.).

4,11-Di(1-pyrrolidinyl)-1H-naphtho[2,3-*f*]indole-5,10-dione (5). A solution of naphthoindole **1** (0.1 g, 0.33 mmol) in THF (3 ml) and pyrrolidine (2.0 ml, 20 mmol) was boiled in an atmosphere of argon for 1 h. The mixture was cooled to room temperature, ethyl acetate (50 ml) was added to the violet colored reaction mixture, which was then poured into water. The organic phase was washed three times with water, dried over $MgSO_4$, and evaporated in vacuum. The residue was recrystallized twice from a toluene–dioxane mixture and naphthoindole **5** (0.089 g, 70%) was obtained as violet crystals of mp 238-240°C. ^{1}H NMR spectrum (DMSO-D_6, 30°C), δ, ppm (*J*, Hz): 11.51 (1H, br. s, NH); 8.30 (2H, m, H-6, 9); 7.69 (2H, m, H-7, 8); 7.36 (1H, t, *J* = 2.8, H-2); 7.09 (1H, m, *J* = 2.8, H-3); 3.52 [4H, m, $N(CH_2)_2$]; 3.35 [4H, m, $N(CH_2)_2$]; 2.03 (4H, m, CH_2); 1.95 (4H, m, CH_2). Mass spectrum, *m/z* (I_{rel}, %): 385 (100) $[M]^+$, 368 (96) $[M\text{-}OH]^+$, 340 (22), 299 (59), 273 (34). Found, %: C 75.01; H 6.24; N 10.52. $C_{24}H_{23}N_3O_2$. Calculated, %: C 74.78; H 6.01; N 10.90.

Dihydrochloride of **5** was obtained by adding 2% ethereal HCl solution to a solution of **5** free base in hot 2-methoxyethanol. The salt was filtered off, washed with ether, and with hexane. The orange product was collected and dried in vacuum; mp <200°C (decomp.).

4,11-Bis{[(1*R*)-1-phenylethyl]amino}-1H-naphtho[2,3-*f*]indole-5,10-dione. (6) was obtained analogously to naphthoindole **2** from compound **1** and (*R*)-1-phenylethylamine. Yield was 75%; mp 128-130°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm (*J*, Hz): 13.42 (1H, d, *J* = 6.8, NH); 12.68 (1H, s, *J* = 6.4, NH); 8.76 (1H, br. s, NH); 8.47 (2H, m, H-6, 9); 7.70 (2H, m, H-7, 8); 7.51 (2H, d, 2-C_6H_5); 7.41 (2H, t, 3-C_6H_5); 7.37 (2H, d, 2-C_6H_5); 7.32 (1H, m, 4-C_6H_5); 7.29 (2H, t, 3-C_6H_5); 7.19 (1H, m, 4-C_6H_5); 6.77 (1H, m, H-2); 6.62 (1H, m, H-3); 5.40 (1H, q, *J* = 6.8, NCH); 5.01 (1H, q, *J* = 6.4, NCH); 1.76 (3H, d, *J* = 6.4, CH_3); 1.73 (3H, d, *J* = 6.4, CH_3). Mass spectrum, *m/z* (I_{rel}, %): 485 (61) $[M]^+$, 380 (57), 276 (78), 105 (100). Found, %: C 78.91; H 5.79; N 8.53. $C_{32}H_{27}N_3O_2$. Calculated, %: C 79.15; H 5.60; N 8.65.

4,11-Diamino-1H-naphtho[2,3-*f*]indole-5,10-dione (7). A 1 M solution (3.0 ml, 3.0 mmol) of $BBr_3 \cdot SMe_2$ in dichloromethane was added to a solution of naphthoindole **6** (0.2 g, 0.41 mmol) in dichloroethane (25 ml) and the mixture was boiled for 5 h. The reaction mixture was evaporated in vacuum, water (50 ml) added, and the mixture extracted with hot ethyl acetate. The extract was washed with water, dried over $MgSO_4$, and evaporated in vacuum. The residue was recrystallized from DMF, washed with water, and dried. Yield of compound **7** was 0.073 g (64%) as claret-colored crystals; mp >260°C. ^{1}H NMR spectrum (DMSO-d_6), δ, ppm: 12.01 (1H, s, NH); 10.62 (1H, br. s, NH); 8.9 (2H, br. s, NH); 8.27 (2H, m, H-6, 9); 7.74 (2H, m, H-7, 8); 7.53 (1H, d, H-2); 7.17 (1H, d, H-3). Mass spectrum, *m/z* (I_{rel}, %): 277 (100) $[M]^+$, 261 (4) $[M\text{-}NH_2]^+$, 248 (3), 220 (5), 192 (2). Found, %: C 68.99; H 4.21; N 15.19. $C_{16}H_{11}N_3O_2$. Calculated, %: C 69.31; H 4.00; N 15.15.

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